OPTIMISATION OF PROCESS PARAMETERS FOR PREPARATION OF FLAVOURED INSTANT GREEN TEA

by

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DECLARATION

I hereby declare that this thesis entitled **"Optimisation of process parameters for preparation of flavoured instant green tea"** is a *bonafide* record of research work done by me during the course of research and the thesis has not previously formed the basis for the award of any degree, diploma, associateship, fellowship or other similar title of any other University or Society.

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Certified that this thesis entitled **"Optimisation of process parameters for preparation of flavoured instant green tea"** is a *bonafide* record of research work done independently by Ms. Sankalpa K. B. under my guidance and supervision and that it has not previously formed the basis for the award of any degree, fellowship or associateship to her.

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SYMBOLS AND ABBREVIATIONS

-	:	Minus
%	:	Percent
+	:	Plus
<	:	Less than
>	:	Greater than
±	:	Pluse or minus
ΔE	:	Colour difference
~	:	In the range of
°	:	Degree brix
°C	:	Degree centigrade
µM	:	Micrometer
a*	:	Greenness or redness
AF	:	Aluminum foil
ALF	:	Aluminium laminated polyethylene
ALP	:	Aluminium laminated polyethylene
ALPE	:	Aluminum laminated polyethylene
ANOVA	:	Analysis of variance
AOAC	:	Association of Official Analytical Chemists
a_w	:	Water activity
b*	:	Blueness or yellowness
BET	:	Brunauer-emmett-teller
BHT	:	Butylated hydroxy toluene
BMF	:	Broken mixed fannings

BOPP	:	Biaxially oriented polypropylene
C	:	Catechin
CE	:	Catechin equivalents
CFU/ml	:	Coloni forming unit per mili liter
CI	:	Carr's index
CO_2	:	Carbon dioxide
CRC	:	Colorectal cancer risk
d.b	:	Dry basis
DPPH	:	2,2-diphenyl-1-picrylhydrazyl
EC	:	Epicatechin
EDTA	:	Ethylene diamine tetraacetic acid
EGC	:	Epigallocatechin
EGCG	:	Epigallocatechin gallate
EMC	:	Equilibrium moisture content
ERH	:	Equilibrium relative humidity
et al.	:	And others
FRAP	:	Ferric reducing antioxidant power assay
FCRD	:	Factorial completely randomised design
Fig.	:	Figure
G	:	Grame
$g.cc^{-1}$	:	Grame per cubic centimeter
$g.cm^{-3}$.	:	Grame per centimeter cube
$g.ml^{-1}$	:	Grame per mililiter
g/100 g	:	Grame per hundred grame

g/kg	:	Grame per kilograme
g/ml	:	Grame per mililiter
GAB	:	Guggenheim-Anderson-de Boer
GAE	:	Gallic acid equivalents
GC	:	Gass chromatography
GCg	:	Gallocatechin gallate
GC-MS	:	Gass chromatography mass sprctroscopy
gl^{-1}	:	Grame per liter
GTE	:	Green tea extracts
H	:	Houre
HC	:	Hydroxychavicol
HDPP	:	Heigh density polyethylene
HPLC	:	Heigh performence liquied chromatograpy
HR	:	Hausner ratio
HV	:	High vacuum
ISO	:	International organization for standardization
KCAET	:	Kelappaji College of Agricultural Engineering and Technology
kg H_2O/kg	:	Kilograme water per kilograme
Kg	:	Killograme
$kg.m^{-2}$.day-1.Pa-1	:	Kilogram per square meter day Pascal
$kg.m^{-3}$	:	Killograme per meter qube
$kg.s^{-1}$	:	Kilogram per second
kJ/mol	:	Kilo jouls per mol

kPa	:	Kilopascal
L*	:	Lightness or darkness
LDPE	:	Low density polyethylene
Ltd.	:	Limited
$m\ s^{-1}$	:	Meter per second
M	:	Meter
M	:	Molar
m2	:	Square meter
m3 s-1	:	Cubic meter per second
$m^3.h^{-1}$	:	Meter cube per houre
MAE	:	Microwave assisted extraction
MCPP	:	Metallized co-extruded polypropylene
MD	:	Maltodextrin
MDC	:	Maltodextrin concentrations
mg GAE/100 g	:	Miligrame of gallic acid per gramme
$mg.g^{-1}$	:	Miligramme per grame
MIC	:	Minimum inhibitory concentration
Min	:	Minits
Ml	:	Mililiter
$ml.h^{-1}$	:	Mililiter per hour
$ml.min^{-1}$	:	Mililiter per minutes
ml/g	:	Mililiter per grame
ml/min	:	Mililiter per minutes
ml/ml	:	Mililiter per mili liter
mM	:	Mili molar
mmol/L	:	Milimol per liter

MS	:	Mass spectrometric
N	:	Normality
Na_2CO_3	:	Sodium corbonate
$NaNO_2$	:	Sodium hydroxide
Nm	:	Nano meter
P	:	Probabality
Pa	:	Pascal
PET	:	Polyethylene terephthalate
Pg*	:	Corresponding saturation pressure
POD	:	Peroxidase
PP	:	Polypropylene
PPO	:	Polyphenol oxidase
RH	:	Rhelative humidity
RO	:	Reverse osmosis
RP-HPLC	:	Reverse phase heigh performence liquied chromatography
Rpm	:	Revalution per minits
RSM	:	Response surface methodology
RTD	:	Ready-to-drink
S	:	Seconds
s-1	:	Per second
SE	:	Secondary electron
SEM	:	Scanning electron microscope
Sl.No.	:	Serial number
Std. Dev	:	Standard deviation
T	:	Temperature

T	:	Time
TBA	:	Thiobarbituric acid
TEAC	:	Trolox equivalent antioxidant capacity
Tg	:	Glas transition temperature
Tp	:	Storage temperature
TPTZ	:	2, 4 ,6- tripyridyl- s- triazine
Ts	:	Stickypoint temperature
UHPLC	:	Ultra heigh performence liquied chromatograpy
USA	:	United states of America
UV	:	Ultraviolet
V	:	Volume
v/v	:	Volume by volume
w.b.	:	Wetbulb
w/v	:	Weight by volume
WD	:	Working distance
Xpc	:	Critical moisture content
Xpc	:	Stickiness at moisture content
XRD	:	X-ray diffraction
Y	:	Response value predicted by the model
A	:	Alpa
B	:	Beeta
Γ	:	Gama
Δ	:	Delta
ΔE	:	Total colour difference
µg/ml	:	Microgramme per mililiter

CHAPTER I

INTRODUCTION

Beverages constitute an important part of overall dietary intake, resulting in more than one-fifth of daily energy intake. The Experts Committee of Nutrition reviewed and ranked six categories of beverage based on calorie delivered, contribution to intake of energy and essential nutrients. Among the six, water stands first followed by tea and coffee. Other four groups are milk and soy beverage, non-calorically sweetened beverage, caloric beverage with some nutrients and calorically sweetened beverage. Half of our fluid intake should come from water and one third may be tea or coffee and the remaining can be any other beverage. But high consumption of sweetened carbonated beverages has been linked to the increased body mass index and obesity epidemic (Johnson *et al*., 2010). Due to these facts beverage consumption patterns have changed in the past decade, with reductions in sugar-sweetened beverages, whole milk, increase in coffee and tea, energy drinks and sports drinks (Eisenberg *et al*., 2017). Among all the beverages, tea is valued more due to the bioactive compounds and associated health benefits. Due to these reasons tea consumption is well ahead of coffee, beer, wine and other carbonated soft drinks (Sinija and Mishra, 2008).

The tea plant belongs to the genus *Camellia* (*Camellia sinensis*), a member of the family Theaceae. Tea was originated in southwest China, then British introduced tea to India by starting commercial cultivation in Bengal. Presently, tea is cultivated in at least 30 countries around the globe (Sharma *et al*., 2007; Cooper *et al*., 2005). India is the second largest producer of tea in the world. Tea is indigenous to eastern and northern India, and was cultivated and consumed there for thousands of years. In the early 1820s, the British East India Company began large-scale production of tea in Assam. Tea is grown in 16 Indian States, of which Assam, West Bengal, Tamil Nadu and Kerala account for about 96 per cent of the total tea production (Arya, 2013). In 2016 total tea production in India was 1239 M. kg. Among the states Assam produces 642 M. kg of tea and stand in first position followed by west Bengal producing 357

M. kg. In south India Tamil Nadu is having the highest production of 145.41 M.kg followed by Kerala and Karnataka with production of 61.52 and 5.28 M.kg, respectively (Anon., 2017). Area under tea cultivation is 563,980 ha in India. Assam has the highest tea cultivating area of 304.40 ha followed by west Bengal with an area of 140 ha. In South India Tamil Nadu has the highest area of tea cultivation (69.62 ha) followed by Kerala (35.01 ha) and Karnataka (2.22 ha) (IBEF, 2016).

Tea can be categorized into three main types, depending on the level of oxidation, as green (unfermented), oolong (partially fermented) and black (fermented) tea (Senanayake, 2013; Lee *et al*., 2014). However, the most significant effect on human health has been observed with the consumption of green tea (Chacko *et al*., 2010). Green tea consumption helps in preventing cardiovascular diseases, cancer, diabetes, obesity. Its antioxidant nature of green tea inhibits the growth of cancer cells and also kills cancer cells without harming healthy tissue. It has also been effective in lowering low density lipoprotein cholesterol levels thereby minimizing the risks of heart attacks and strokes by inhibiting formation of abnormal blood clots (thrombosis), reduction of platelet aggregation, lipid regulation and inhibition of proliferation and migration of smooth muscle cells. The major and most chemo-preventive constituent in green tea responsible for these biochemical or pharmacological effects are catechins, epicatechin (EC), epigallocatechin (EGC), epicatechin gallate (ECG) and epigallocatechin gallate (EGCG). Epigallocatechin gallate is viewed as the most significant active component (Sinija and Mishra 2008).

Green tea processing involves steaming of leaves to inactivate polyphenol oxidase enzymes, then they undergo cooling, set of curling and drying steps until dry green tea leaves are obtained (Ozturk *et al*., 2015). The most severe oxidation employed during processing of black tea which decreases levels of monometric catechins to a much greater extent than the other teas (Ozturk *et al*., 2015). Green tea leaves are steamed, which prevents EGCG compound from being oxidized. In contrast, black and oolong tea are made from fermented leaves, which results in the EGCG being converted into other compounds that are not nearly as effective in

preventing and fighting various diseases. Health benefits of green tea are superior to black tea due to polyphenols which are responsible for the antioxidant activity, owing to higher content of EGCG.

Green tea catechin is Generally-Recognized-As-Safe (GRAS) for use in bottled teas, sports drinks, carbonated soft drinks, and juice as per recent reports from Japan. Nowadays demand is increasing for nonconventional tea products like instant tea (cold- and hot-soluble), decaffeinated tea and flavoured tea and also convenience products like canned or bottled teas, soluble tea mixes, frozen tea liquid, and tea tablets (Nagalakshmi., 2003). The use of instant tea powder will reduce the preparation time to a large extent. It saves time as well as the effort of tea preparation because of being soluble even in cold water. The instant tea gives a clear solution without any residue. Spray drying technique is the most widely used commercial method for producing instant food powders because of the very short time of heat contact and the high rate of evaporation giving a high quality product with relatively low cost. A dry powder product is highly desirable since it not only possesses long shelf-life, but also requires relatively low transportation cost and storage capacity (Jinapong *et al*., 2008). Green tea powder obtained by spray drying will be soluble in water without losing any nutritive value.

Commonly herbs and spices are used for flavouring many beverages. In addition to boosting the flavours, herbs and spices also imparts medicinal value to the tea beverage (Rani and Pragya, 2016).

Ginger (*Zingiber officinale Roscoe, Zingiberacae*) is a medicinal plant that has been widely used in Chinese, Ayurvedic and Tibb-Unani herbal medicines all over the world, since antiquity, for a wide array of unrelated ailments that include arthritis, rheumatism, sprains, muscular aches, pains, sore throats, cramps, constipation, indigestion, vomiting, hypertension, dementia, fever, infectious diseases and helminthiasis (Ali *et al*., 2008).

Cardamom (*Elettaria cardamomum* Maton) is one of the oldest known spices in the world. Evergreen forests of Western Ghats of South India are considered as the

centre of origin as well as natural habitat of cardamom (Ankegowda *et al*., 2015). The fruit are tri-ocular, ovoid, oblong or greenish-brown capsules containing about 15-20 reddish brown seeds. The cardamom seeds have a warm, slightly pungent and highly aromatic flavour. Cardamom oil is used in food, perfumery, liquor and in pharmaceutical industries as a flavour and a carminative. In medicine, it is used as a powerful aromatic, antiseptic, stimulant, carminative, stomachic, expectorant, anti-spasmodic and diuretic (Agaoglu *et al*., 2005).

Holy basil (*Ocimum sanctum*), Queen of Herbs, the Legendary, "Incomparable One" is one of the holiest and most cherished of the many healing and health-giving herbs. Tulsi is a widely grown, sacred plant belongs to the lamiaceae family. It is called by names like Rama Tulsi, Krishna Tulsi in Sanskrit and Holy Basil in English (Kayastha, 2014; Chandra, 2016). *Ocimum sanctum* is one such plant showing multifarious medicinal properties viz. analgesic activity, anti-ulcer activity, antiarthritic activity, immunomodulatory activity, antiasthmatic activity, antifertility activity, anticancer activity, anticonvulsant activity, antidiabetic activity, antihyperlipidemic activity, anti-inflammatory activity, antioxidant activity, anti-stress activity in addition to possessing useful memory enhancer and neuroprotective activity (Kadian and Parle, 2012). The essential oil of basil extracted via steam distribution from the leaves and flavouring tops are used to flavour foods, dental and oral products, in fragrances and in traditional rituals and medicines (Kayastha, 2014; Vishan and Srivastava, 2016). So these spices and herb was selected to enhance the flavour and to add the goodness of instant green tea.

Instant powder are hygroscopic and having less shelf life without proper packaging and storage. Packaging performance and specifications therefore vary and depend on variations in product characteristics, the ambient distribution environment, and the market environment (Sonneveld, 2000).

In this background the project entitled “Optimisation of process parameters for preparation of flavored instant green tea” was undertaken at Kelappaji College of Agricultural Engineering and Technology (KCAET), Tavanur, Kerala, India with the following objectives

- To optimize the extraction process parameters of green tea
- To optimize the process parameters for drying of the flavored green tea extract using different drying techniques
- To optimize the flavoring compounds to get better flavor for the instant green tea
- To conduct storage studies of flavored instant green tea powder packed in different packaging materials

CHAPTER II

II. REVIEW OF LITERATURE

In the present investigation, an attempt was made to optimisation of process parameters for preparation of flavoured instant green tea. This chapter deals with brief account of literature, which has direct and indirect bearing on the specific objectives of the investigation.

a) Importance and composition of green tea
b) Flavours for green tea
c) Production techniques for instant flavoured green tea
d) Physical and chemical properties of spray dried instant flavoured green tea
e) Packaging, sorption isotherm kinetic and storage studies for instant flavoured green tea

Tea is a commonly consumed beverage. An oriental evergreen tree that can reach a height of thirty feet in the wild, the tea plant is pruned to a height of about three feet to promote new growth and easy plucking. The tea plant (botanical name- *Camellia sinesis*) produces abundant foliage, a camellia like flower and berries containing one to two seeds. Only the two leaves and bud at the tip of each new shoot are picked for tea.

2.1 IMPORTANCE AND COMPOSITION OF GREEN TEA

Wang and Helliwell (2001) studied the flavonols content in green tea leaves and black tea infusion by high-performance liquid chromatography. Tea flavonols are potent antioxidant and make up 2-3% of the water-soluble solids from tea leaves. Aqueous ethanol was selected as the best solution for hydrolyzing flavonoids in tea leaves. The content of flavonols on a dry weight basis in green tea leaves ranged from 0.83-1.59, 1.79-4.05 and 1.56-3.31 $g.kg^{-1}$, and in black tea leaves from 0.24-0.52, 1.04-3.03 and 1.72-2.31 $g.kg^{-1}$ for myricetin, quercetin and kaempferol, respectively.

Kilmartin and Hsu (2003) conducted experiment on characterisation of polyphenols in green, oolong, and black teas, and in coffee. The green and oolong teas was dominated by epigallocatechin gallate, black tea by theaflavin and coffee by 5-O-caffeoylquinic acid. The level of phenolics increased with water temperature from 20 to 100°C, while lower levels were obtained for repeat infusions. The addition of milk lowered the active components more in the tea than in the coffee.

Perva-Uzunalic *et al*. (2006) investigated the effect of different solvent and extraction steps on efficiency of caffeine and catechins from green tea leaves at various extraction temperatures from 60 to 100°C between 5-240 min time. The major catechins content and Caffeine of green tea extracts varied from 61 to almost 100% and 62 to 76%, respectively. Average extraction yield was 30% with exceptions when using pure acetone and acetonitrile, where extraction yield was about 3%. Contents of

flavonols and proanthocyanidins were in the range of 6-20 and 12-19 $g.kg^{-1}$, respectively. Optimal condition for extraction with water was obtained at 80 °C after 20 min (97%) and at 95°C after 10 min of extraction (90%). Degradation of catechins was observed at higher extraction temperatures and with prolonged extraction times.

Yang *et al*. (2007) reported that, anticarcinogenic activity of tea and its constituents in *in vitro* animal studies. They evaluated association between colorectal cancer (CRC) risk and green tea consumption in 69,710 Chinese women. The reduction in risk was most evident among those who consistently reported to drink tea regularly at both the baseline and follow-up surveys. The inverse association with regular tea drinking was observed for both colon and rectal cancers. Result suggests that regular consumption of green tea may reduce CRC risk in women.

By consuming green tea it prevent cancer and cardiovascular diseases, the anti-inflammatory, antiarthritic, antibacterial, antiangiogenic, antioxidative, antiviral, neuroprotective and cholesterol-lowering effects (Chacko *et al*., 2010).

Sarah *et al*. (2010) studied the effect of green tea (*Camellia sinenses*) extract and onion (*Allium cepa*) juice on lipid degradation and sensory acceptance of Persian sturgeon fillets. Result of this experiment shows that, green tea extracts and onion juice in concentration upper than 1% (v/v) and 2.5% (v/v) respectively had more antioxidant characteristics and oxidative stability against lipid oxidation and shelf life enhancement.

Wu *et al*. (2012) determined the catechins and flavonol glycosides in Chinese tea varieties by high performance liquid chromatography combined with ultraviolet (UV) and mass spectrometric detection (MS). The catechins, flavonol and flavones glycosides, phenolic acids and purine alkaloids of 24 tea constituents were analysed and it was unique. The composition of catechins were lower in the tea varieties for Green tea manufacturing variety tea had lower catechins, while oolong tea

manufacturing variety had lowest myricetin glycosides. The content of phenolic compounds in among the selected tea varieties is highly variable that helpful to classify tea in to non fermented green and fermented oolong or black tea.

Vuong *et al*. (2013) produced caffeinated and decaffeinated green tea catechin powders from underutilised old tea leaves. The decaffeination was done by blanching tea leaves in water hot (100℃) water for 10 min which remove caffeine up to 80% by retaining 85% of the catechins. The 100% yields of extractable powder was obtained by extracting leaves in water at 80℃ and freeze drying while 20-25% lower yields of catechins was obtained by spray drying. Decaffeination and spray drying increased the conversion of epistructured to non-epistructured catechins and also old green tea leaves could be source for caffeinated and decaffeinated green tea catechin powders.

Cunha *et al*. (2012) evaluated the effects of green tea *Camellia sinensis* extract on proinflammatory molecules and lipolytic protein levels in adipose tissue of diet-induced obese mice. Green tea administered concomitantly with a high-fat diet increased HSL, ABHD5 and perilipin inmesenteric adipose tissue, and this was associated with reduced body weight and adipose tissue gain. Results show that green tea increases the lipolytic pathway and reduces adipose tissue and this may explain the attenuation of low-grade inflammation in obese mice.

Study was conducted to determine the relationship between the plucking periods and the major constituents and the antioxidant activity in green tea. Green tea was prepared from leaves plucked from the end of April to the end of May at intervals of one week or longer. The contents of theanine, theobromine, caffeine, catechin (C) and gallocatechin gallate (GCg) were significantly decreased, whereas those of epicatechin (EC), epigallocatechin gallate (EGCg) and epigallocatechin (EGC) were significantly increased along with the period of tea leaf plucking. The highest antioxidant activity was observed in relatively the oldest leaf. The *cis*-catechins

contents were the key factor affecting the antioxidant activity of green tea (Lee *et al*., 2014)

Jun (2009) extracted caffeine from green tea leaves assisted by high pressure processing. The effect of high hydrostatic pressure, different solvents, pressure holding time and liquid/solid ratio were studied for the optimal caffeine extraction from green tea leaves. The highest yields of 4.0 ± 0.22% were obtained at 50% ($ml.ml^{-1}$) ethanol concentration, liquid/solid ratio of 20:1 ($ml.g^{-1}$), and 500 MPa pressure applied for 1 min. High pressure processing resulted higher yields of caffeine, shorter extraction times and lower energy consumption than conventional method.

Bancirova (2010) compared the antioxidant capacity and the antimicrobial activity of green and black tea. The average value of antioxidant capacities of the non-fermented (green tea) and semi-fermented tea samples was 1.43 mM and the average value of Trolox equivalent antioxidant capacity (TEAC) of the fermented teas (black tea) samples was 1.43 mM. All samples were stored in freezer (-20°C) and the TEAC determination was repeated after a year. The average values of TEAC of non-fermented and semi-fermented tea samples were two fold lower in comparison to fermented tea samples and only 20% of average value of TEAC of the fresh tea infusion.

Dong *et al*. (2011) isolated antioxidant catechins from green tea by ethyl acetate, n-butanol and n-hexane and the catechins were decaffeinated using citric acid solution. The optimum extraction conditions were that 100 g tea was extracted in water at 80°C for 40 min and the catechins in the extracted solution were isolated using 1.5 l ethyl acetate for three times. The extracted catechins in the ethyl acetate phase was then decaffeinated by washing the organic phase with 1.5 l of 10 $g\ l^{-1}$ citric acid solution for three times. The obtained product contained 694.47 $mg.g^{-1}$ catehchins and 37.89 $mg.g^{-1}$ caffeine, with 78.8% caffeine being removed. The

method is considered to be an alternative to replace traditional chloroform decaffeination.

Vuong *et al*. (2012) developed improved method of extraction for green tea components from tea bags using the microwave oven. Brewing green tea bag in 200 ml freshly boiled water for 2-3 min were not sufficient to extract all the catechins and that a household microwave oven could be used to improve the extraction. To maximise the extraction of the catechins and caffeine, first teabag brewing by using microwave assisted extraction (MAE) for 0.5 min before irradiation for 1 min in a microwave oven (hot MAE).

El-Shahawi *et al*. (2012) investigated the catechins and caffeine in green tea by high performance liquid chromatography. The average concentrations of caffeine, catechin, EC, EGC, ECG and EGCG were found to be in the ranges 0.086–2.23, 0.113–2.94, 0.58–10.22, 0.19–24.9, 0.22–13.9 and 1.01–43.3 $mg.g^{-1}$, respectively. The contents of caffeine and catechins followed the sequence: EGCG > EGC > ECG > EC > C > caffeine.

Xi *et al*. (2012) studied the effects of green tea extract for increasing shelf life of oyster meats by reducing *Vibrio parahaemolyticus*. Tea extract reduce a mixture of five clinical *V. parahaemolyticus* strains in tryptic soy broth plus 1.5% NaCl from 4.5 log $CFU.ml^{-1}$ to non-detectable level (<1 log $CFU.ml^{-1}$) within 8 h. This study indicated that green tea treatment at oyster/tea extract ratio of approximate 0.7 $g.ml^{-1}$ could enhance reducing V. parahaemolyticus while retarding the growth of total bacteria in oysters during 5 ± 1°C storage. Therefore, green tea might be utilized as a natural antimicrobial agent to inactivate V. parahaemolyticus in oysters and extend the shelf life during refrigeration storage.

Carloni *et al*. (2013) studied the antioxidant activity of white, green and black tea. ‘Antioxidant profile’ of different tea was in the order of green≥low-caffeine

green>white≥black Orthodox>black CTC. Metal chelating activity, which was lowest in the green teas, does not correlate with antioxidant activity but appears to be influenced by theaflavins content.

Kanda *et al*. (2013) developed a new technique for green tea decaffeination involving ingredient extraction and drying of green tea leaves by using liquefied dimethyl ether as a safe extraction solvent. After hot water extraction with water content of 74.6-76.2%, green tea leaves were tested to verify the liquefied dimethyl ether extraction. Caffeine was completely removed from the green tea leaves. Approximately 25.2-56.0% of catechins remained in the residue after liquefied dimethyl ether extraction. In particular, 56.0% of epigallocatechin gallate, which has the greatest activity of all catechins remained in the residue.

Yu *et al*. (2014) identified thirty-nine non-volatile compounds in seven ready-to-drink (RTD) green tea samples and were analysed and quantified using liquid chromatography. Taste profiles of the reconstructed samples did not differ significantly from the RTD tea samples. Sensory evaluation revealed that the astringent and bitter-tasting (-)-epigallocatechin gallate, bitter-tasting caffeine and the umami-tasting L-glutamic acid were the main contributors to the taste of RTD green tea.

Ozturk *et al*. (2015) investigated the effect of process parameters on polyphenol oxidase (PPO), peroxidase (POD) and quality parameters of green tea. The best quality parameters were obtained in the “high rate steam-short time” application. Drum steam system provided higher inactivation rates than tunnel steam system at shorter time of steam treatment. The leaves were exposed to drying process after steaming and residual amounts of PPO were inactivated after completing the final drying step.

Jiang *et al*. (2015) determined flavonol glycosides in green tea, oolong tea and black tea. The total amounts of flavonol glycosides compounds in the tea samples were 2.32–5.67 $g.kg^{-1}$ dry weight and there is no significant difference for the total flavonol glycosides content among green tea, oolong tea and black tea. However, kaempferol glycosides are more abundant in green teas, while oolong tea has more quercetin and myricetin glycosides. In black tea quercetin glycosides were most abundant.

Sharpe *et al*. (2016) investigated the effects of brewing conditions on the antioxidant capacity of twenty four commercial green tea varieties. The results reveal that, the antioxidant capacity of green tea varied greatly depending on leaf preparation (growing location, harvest season, drying method, and whether tea is loose leaf or bagged) and brewing techniques (re-infusions, water temperature, brewing time, and time between re-infusions). The antioxidant capacity are not reflected in their prices and appear not to be affected by modification of tealeaves through decaffeination or pesticide use. Brewing temperature and time had direct relationship to antioxidant capacity, while time between re-brews up to one hour, and use of tap vs. de-ionized water showed no effect.

Zhang *et al*. (2016) conducted experiment for improving the sweet aftertaste of green tea infusion by hydrolyzing (-)-epigallocatechin gallate (EGCG) and (-)-epicatechin gallate (ECG) with tannase. EGC and EC were found to be the main contributors for the sweet aftertaste, based on a trial compatibility with EGCG, ECG, EGC, and EC monomers, and a synergistic action between EGC and EC to sweet aftertaste was notices. This helps to produce a tea beverage with excellent sweet aftertaste by hydrolyzing the green tea infusion with tannase.

2.2 FLAVOURS FOR GREEN TEA

2.2.1 Ginger

He *et al*. (1998) analyzed the pungent constituents of ginger by high-performance liquid chromatography–electrospray mass spectrometric. The pungent compounds were assigned as [6]-gingerol, [8]-gingerol, [10]-gingerol, [6]-shogaol, [8]-shogaol, [10]-shogaol and [6]-gingediol. Another eight minor compounds were tentatively identified as gingerol analogues.

Stoilova *et al*. (2007) studied the Antioxidant activity of a ginger extract (*Zingiber officinale*). The CO_2 extract of ginger is good scavenging of DPPH and reduced its reducing capacity due to high polyphenol. The extract can be used as an antioxidant during fat oxidation. The ginger extract showed an antioxidant activity at 37°C, and at a high temperature of 80°C. Polyphenols in the ginger extract also demonstrated a higher chelatoforming capacity with regard to Fe^{3+}, leading to the prevention of the initiation of hydroxyl radicals. The property of the ginger extract is compared with the synthetic antioxidant, which determine its potential as a natural preservative in the food and pharmaceutical industries.

Schwertner and Rios (2007) developed a high-performance liquid chromatographic (HPLC) method that is suitable for the analysis of 6-gingerol, 6-shogaol, 8-gingerol, and 10-gingerol in a wide variety of ginger-containing dietary supplements, spices, teas, mints, and beverages. Samples were extracted with ethyl acetate and analyzed by HPLC on a C-8 reversed phase column at 282 nm.

Maizura *et al*. (2010) conducted experiment on total phenolic content and antioxidant activity of kesum (polygonum minus), ginger (zingiber officinale) and turmeric (curcuma longa) extract. Among the tested sample Kesum had the highest total phenolic content (165.34 mg GAE.100 g^{-1}) followed by ginger (101.56 mgGAE.100 g^{-1}) and turmeric (67.89 mg GAE.100 g^{-1}). In case of antioxidant activity

kesum extract had the highest DPPH radical scavenging activity (82.6 ± 0.7%), followed by ginger extract (79.0 ± 0.6%) and turmeric extract (64.6 ± 2.4%).

Sasidharan and Menon (2010) analysed the volatile oils from fresh and dried ginger rhizomes. Zingiberene was the major compound in both ginger oils. Fresh ginger oil contained geranial (8.5%) as the second main compound and had more oxygenated compounds (29.2%) compared to dry ginger oil (14.4%). The dry ginger oil also contained ar-curcumene (11%), β-bisabolene (7.2%), sesquiphellandrene (6.6%) and δ-cadinene (3.5%). Ginger oil showed antimicrobial activity against microorganisms. The MIC values of the oils ranged from 10μg.ml^{-1} to 1 μg.ml^{-1}.

Sasidharan and Menon (2010) analysed the volatile oils of fresh and dried ginger rhizomes by GC and GC-MS. Zingiberene was the major compound present both ginger oils. Fresh ginger oil contained geranial (8.5%) as the second main compound and had more oxygenated compounds (29.2%) compared to dry ginger oil (14.4%). The dry ginger oil also contained ar-curcumene (11%), β-bisabolene (7.2%), sesquiphellandrene (6.6%) and δ-cadinene (3.5%).

Pawar *et al*. (2011) selected the twelve ginger variety from various agriclimatic zone and assess the phenolic content and antioxidant capacity. 6-gingerol had strongest free radical scavenging activities and was ranging from 0.1% to 0.2%. Among the twelve cultivars Rajasthan and Rio De Janero cultivars was found to be good source of 6-gingerol by high performance liquid chromatography (RP-HPLC) analysis.

Hasan *et al*. (2012) evaluated the crude extracts from *Zingiber officinale* for chemical composition and antimicrobial activity. Main components identified from the terpene family, most of them were sesquiterpene hydrocarbons among them zingiberene (9%, 6%), monoterpene hydrocarbons which is α-curcumene (14%, 0%), α-farnesne (11%, 7%), β-sesquiphellandrene (9%, 13%), β- bisabolene (4%, 5%) and

phenolic compounds which are gingerol (25%, 23%) and shogaol (18%, 25%) in methanol and n-hexane, respectively and ginger extracts were more effective against the Gram-positive bacteria.

Nagendrachari *et al*. (2013) extracted the 6-Gingerol which is the major bioactive constituent responsible for the anti-inflammatory, anti-tumour and antioxidant activities of ginger. Ethanol-extracted ginger oleoresin obtained from cellulase treatment exhibited good antioxidant activity which could be due to the presence of the bioactive compounds *viz*., 6-gingerol, shogaols, paradol and zingerone. Ginger oleoresin, with its nutraceutical constituents and a characteristic flavour profile, can be a potent ingredient in functional food formulations.

Yeh *et al*. (2014) revealed that, gingerols and shogaol is the major component in ginger. The essential oils exhibited volatile profiles of 60 to 65 compounds. Among the essential oils major components were zingiberene, camphene, afarnesene, sabinene, neral, a-curcumene, b-sesquiphellandrene, and geranial. Ginger ethanolic extracts exhibited higher antioxidant effect than aqueous extracts in trolox equivalent antioxidant capacity and ferric reducing ability of plasma. Ginger aqueous extracts were more effective in free radical scavenging activities and chelating abilities. Based on the results author concluded that, ginger could be used as a flavouring agent and a natural antioxidant.

Gingerols are the major pungent compounds present in the rhizomes of ginger (*Zingiber officinale Roscoe*) and are renowned for their contribution to human health and nutrition. Shogaols are important biomarkers used for the quality control of many ginger-containing products, due to their diverse biological activities. Inclusion of ginger or ginger extracts in nutraceutical formulations could provide valuable protection against diabetes, cardiac and hepatic disorders (Semwal *et al*., 2015).

Hamad *et al*. (2016) conducted study on chemical constituents and antibacterial activities of crude extract and essential oils of *Zingiber officinale*. The essential oil was analyzed by Gas Chromatography - Mass Spectroscopy. The abundant constituents of *Z. officinale* were cineole, 2,2-dimethyl-3-methylenenorbornane, α-curcumenene, β-sesquiphellandrene and rosefuran epoxide. Alpha-pinene, 2,2-dimethyl-3-methylenenorbornane, β-pinene, β-mircene, cineole, β-citral, α-citral, bornyl acetate, α-curcumene, α-zingiberene, β-sesquiphellandrene, and hexadecanoic acid also found in ginger rhizomes. Minimum Inhibitory Concentration (MIC) of essential oils and crude extracts of *Z. officinale* valuated against food borne bacteria *Bacillus subtilis, Escherichia coli, Staphylococcus aureus, Salmonella typhymurium and Vibrio cholera*, and it show relatively high MIC.

Hamad *et al*. (2016) conducted experiment on chemical constituents and antibacterial activities of crude extract and essential oils *Zingiber officinale*. The abondent constituents are cineole, 2,2-dimethyl-3-methylenenorbornane, α-curcumenene, β-sesquiphellandrene and rosefuran epoxide. Alpha-pinene, 2,2-dimethyl-3-methylenenorbornane, β-pinene, β-mircene, cineole, β-citral, α-citral, bornyl acetate, α-curcumene, α-zingiberene, β-sesquiphellandrene, and hexadecanoic acid.

2.2.2 Tulsi

Vasudevan *et al*., 1999 reported that eugenol (57-50%), caryophyllene (22-32%), methyl eugenol (6-14%) were the major constituents together comprising 85% of the oil. Eugenol was highest in leaf, methyl eugenol in stem, phenylprone glucosides and caryophyllene in inflorescence of tulsi.

Rai *et al*. (1997) studied the effect of tulasi (*Ocimum sanctum*) powder supplementation on glycaemic control, lipidaemic control, total amino acids and uronic acid on 27 non-insulin dependent diabetes mellitus patients. The results

indicate that significant lowering of the blood glucose (20.8%), glycated proteins (11.2%), total amino acids (13.5%) and uronic acid (13.7%) after one month of tulasi powder supplementation. The levels of total cholesterol (11.3%), low-density lipoprotein-cholesterol (14.0%), very low-density lipoprotein-cholesterol (16.3%) and triglycerides (16.4%) also reduced during the study.

Kelm *et al*. (2000) reported the phenolic compounds from *Ocimum sanctum*. Eugenol, a major component of the volatile oil, and compounds cirsilineol, isothymusin, isothymonin, and rosmarinic acid demonstrated good antioxidant activity at 10 µM concentrations. Anti-inflammatory activity or cyclooxygenase inhibitory activities of these compounds were observed. Eugenol demonstrated 97% cyclooxygenase-l inhibitory activity when assayed at 1000 µM concentrations.

Ozcan and Chalchat 2002 studied the essential oil composition of *Ocimum spp.* The oil of O. basilicum contained, as main components, methyl eugenol (78.02%), α-cubebene (6.17%), nerol (0.83%) and ε-muurolene (0.74%). Major compounds in the volatile oil of *O. minimum* were geranyl acetate (69.48%), terpinen-4-ol (2.35%) and octan-3-yl-acetate (0.72%). The essential oil of *O. basilicum* was characterised by its high content of methyl eugenol (78.02%), whereas the most important essential oil constituent of *O. minimum* was geranyl acetate (69.48%).

Kothari *et al*. (2005) conducted experiment on constituents of holy basil volatile oil obtained from different plant parts which grown in South India. Oil obtained by hydro-distillation from leaves, stems, inflorescence and whole herbs were analyzed by GC and GC/MS and retention indices. Methyl eugenol was the major constituent of all the oils *i.e*., 72.5% in whole herb, 75.3% leaf, 83.7% stem and 65.2% inflorescence. β-Caryophyllene was the second most dominant constituent and the concentration in each oil is 5.5%, 6.4%, 2.7% and 12.0% in whole herb, leaf, stem and inflorescence, respectively.

Vani *et al*. (2009) conducted a comparative study of volatile compounds from genus Ocimum. Volatile constituents of *Ocimum Sanctum* and *Ocimum Basilicum* were extracted using various solvents and their chemical constituents were identified and quantified by using GC-MS. The predominant species in *Ocimum Sanctum* and *Ocimum Basilicum* was found to be methyl eugenol and methyl chavicol, respectively, during different months of analysis.

Raina *et al*. (2013) evaluated for essential oil content and composition of Holy basil grown in India. Essential oil obtained by hydrodistillation was analyzed by GC and GC/MS for aroma compounds. Essential oil content in Holy basil germplasm showed wide range of variation from 0.13 to 0.45%. GC and GC/MS profile of holy Basil oil showed phenylpropanoids, mainly eugenol constituted the major proportion of essential oil. The range of major chemical constituents identified were eugenol (1.94-60.20%), methyl eugenol (0.87-82.98%), β-caryophyllene (4.13-44.60%), β-elemene (0.76-32.41%). Eugenol and methyl eugenol rich two chemo types were identified in *Ocimum tenuiflorum* germplasm.

Mahapatra and Roy 2014 studied the free radical scavenging and antioxidative potential of eugenol and *Ocimum gratissimum*. Free radical scavenging activity, antioxidant activity and reducing power was observed by *O. gratissimum* samples. Eugenol was more potential than *O. gratissimum* extract samples but lower potent antioxidant ascorbic acid and concluded that *O. gratissimum* is potentially valuable source of natural antioxidant and bioactive material.

Vieira *et al*. (2014) found new antifungal agents from five Brazilian Ocimum species essential oils. The main constituents for *O. americanum* oil were 1,8-cineole (25.9%) and (Z)-methyl cinnamate (29.4%), for *O. basilicum* var. purpurascens linalool (41.5%) and α-muurulol (11.8%), for *O. basilicum* var. minimum linalool (44%) and 1,8-cineole (15.5%), for *O. micranthum* and eugenol (64.11%) and β-caryophyllene (14.3%) and for *O. selloi*, linalool (16.8%) andanethole (52.2%).

Among the five plants *O. micranthum* and *O. selloi* showed the best results, with MIC ranging from 312.5 to 1250 µg ml^{-1}.

Mitra *et al*. (2014) reported antioxidant properties of aqueous tulsi leaf (*Ocimum sanctum*) extract. Tulsi leaf aqueous extract may be useful as a protective antioxidant supplement with promising antioxidant potential to combat oxidative stress-induced tissue damages in the areas where humans are exposed to cadmium occupationally or environmentally.

Suthar and Malik (2015) conducted the experiment on volatile compounds presented in *Ocimum sanctum* leaves essential oil by GC/MS. *Ocimum sanctum* were extracted using hydrodistilation and essential oil have allowed to identify 13 components, but methyl chavicol (34.12%), γ-muurolene (32.44%) and β-caryophyllene (24.33%) are the main component of *Ocimum sanctum.*

2.2.3 Cardamom

Muhmud (2008) reported the composition of essential oil of cardamom leaves. Sixteen components were identified by GC-MS. Components were monoterpenes (27.37%), oxygenated monoterpenes (63%), acetates (0.63%), sesquiterpenes (1.43%) and fatty acid ester (1.17%). Among these 4-terpineol (30.261%) and 1:8 cineol (25.74%) were found as major components whereas other components were found to be α-terpinolene (9.807%), p-cymene (5.3%) and α-terpinene (4.675%). α-Tujene (1.633%), α-pinene (1.165%), sabinene (2.069%), γ- Terpinene (2.675%), Linalool (2.675%), Menth-2-en-l-0l (0.754%), α-terpineol (3.44%) and endbornyl acetae (0.593%).

Gochev *et al*. (2012) conducted experiment on low temperature extraction of essential oil from cardamom (*Elettaria cardamomum (L.) Maton*) seeds. The major compounds reported were terpinyl acetate (36.8%), 1,8-cineole (29.2%), linalyl acetate (5.2%), sabinene (3.9%) and linalool (3.1%). The cardamom extract

demonstrated antimicrobial activity against pathogenic species *Staphylococcus aureus, S. epidermidis, Salmonella abony* and was inactive against *Pseudomonas aeruginosa*.

Baby and Ranganathan (2016) reported the quality of enzyme pre-treatment cardamom (*Elettaria cardamomum maton*.) volatile oil. GC-MS studies indicated that the flavour profile of cardamom oil contains the major flavour compounds *viz*., 1, 8 cineol and terpinyl acetate increased from 34.3 to 37.6% and 40.8–42.3% respectively with enzyme pre-treatment.

Ghosh *et al*. (2016) conducted experiment on food application of an encapsulated polyherbal mix of tulsi, bay and cardamom for shelf-life and frying stability of soybean oil. Polyherbal mix of 1:1:2 tulsi leaves, bay leaves and cardamom seeds having appreciable antioxidant potency was encapsulated using spray drying technology. Maltodextrin and gum arabic (60:40) were used as wall materials at an inlet temperature of 140℃ to obtain an encapsulated powder with appreciable encapsulation efficiency.

2.3 PRODUCTION TECHNIQUES FOR INSTANT FLAVOURED GREEN TEA

Meterc *et al*. (2007) conducted study on extraction of green tea and drying with a high pressure spray process. They extracted green tea with different solvent at different temperature and extraction times to obtain optimum condition and reported that water extraction at 80°C for more than 15 min time of extraction was optimum. Among different condition applied for PGSS drying process, high pre-expansion temperature causes degradation of polyphenols in powder. Maintaining temperature 130°C and lower gave satisfying results.

Sinija *et al*. (2007) developed a novel technique for the production of instant/soluble tea powder from the expressed fermented juice of green leaves. The

fermented juice is steamed, centrifuged and freeze-dried to get instant tea powder. The pressed leaf residue is subjected to fermentation and drying for preparation of tea granules. The instant tea produced is of good liquoring characteristics and various constituents are also in the acceptable range.

Danrong *et al*. (2009) conducted experiment on effect of water quality on the nutritional components and antioxidant activity of green tea extracts. Green tea extracts (GTE) were prepared with tap water, activated carbon adsorbed water, deionized water, distilled water, reverse osmosis water and ultra-pure water. The results indicated that there were statistically significant differences ($P < 0.05$) in the yield rate, the contents of polyphenols, catechins, caffeine, copper, lead and fluorine. Among them, deionized water gave the greatest yield rate and polyphenols, with low caffeine, distilled water increased the contents of non-ester catechins and activated carbon adsorbed water enhanced the concentrations of ester catechins. The contents of copper and lead in green tea extracts were highly correlated with those of the tested water. GTEs prepared with RO displayed the highest antioxidant activities among the six GTEs.

Nadeem *et al*. (2011) produced soluble mountain tea by extracting the mountain tea with water and by spray-dried by using different food hydrocolloids *viz*., β-cyclodextrin, arabic gum and maltodextrins as carrier materials. Powdered samples were then tested for product yield, water activity, moisture content, solubility, bulk density, colour, total phenolic content, SEM particle microstructure analyses, total antioxidant activity, turbidity and volatile compound analyses were performed on the reconstituted mountain tea samples. The mountain tea power yield increased with the increase in carrier materials whereas decreased at higher drying temperatures. For better retention of β-pinene inlet air temperature 155℃ was provided. Inlet air temperature, the type and concentration of the carrier materials affect the physicochemical properties of the spray-dried mountain tea samples ($P < 0.05$).

Vuonf *et al.* (2013) produced the caffeinated and decaffeinated green tea catechin powders from underutilized leaves. Freeze-dried and spray-dried decaffeinated green tea powders had caffeine levels of 6.42 and 6.11 mg/g, respectively which is below the maximum permitted for decaffeinated tea powder. The level of caffeine in the decaffeinated powders were less than in the powders which produced by other decaffeination methods.

Someswararao and Srivastav (2012) invented novel technology for production of instant tea powder from the existing black tea manufacturing process (withering, maceration, and fermentation). The tea juice was extracted by pressing the fermented leaves. The extract was converted to powder form through following steps: heating, centrifugation and vacuum drying. The remaining leaf residue was vacuum dried to produce low grade tea granules. From one kilogram of green tea leaves around 20 and 220 g of soluble tea and pressed cake tea, respectively was obtained. The theaflavi : thearubigin ratio for soluble tea and pressed cake tea was 0.084 and 0.140, respectively.

Pandey and Manimehalai (2014) conducted experiment on spray drying of instant tea powder. Dried black tea (Camellia sinensis) was extract by dissolved in hot water at temperature 65°C, 75°C, 85°C, 95°C and ratio of dried tea to water 1: 10 (w/v) with different time of extraction (10, 20, 30, 40, minutes). Tea extract was concentrated by using low temperature until reached 10-12° Brix of TSS, and dried with spray drier with condition inlet air temperatures were 180°C, 200°C, 220°C, 240°C, outlet air temperatures was controlled as 95°C and blower speed was adjusted in 2500 rpm. The feed flow at 20 $ml.min^{-1}$ and inlet temperature at 200°C was evaluated with the highest yields of physical, chemical results and the highest of kg water removal/hour by spray dryer. So it is recommended to brew black tea at 65°C for 50 min. for maximum yields the caffeine content and total polyphenol content should be 38.09 $mg.100\ gm^{-1}$ and 13.19 $mg.100\ gm^{-1}$.

Pasrija *et al*. (2015) conducted experiment on microencapsulation of green tea polyphenols and its effect on incorporated bread quality. Green tea extract was microencapsulated by freeze drying and spray drying technique using three different wall materials such as maltodextrin, β-cyclodextrin and combination of both. Freeze dried encapsulates exhibited higher encapsulation efficiency and antioxidant activity than spray dried encapsulates. Comparatively, MD encapsulates had higher encapsulation efficiency and antioxidant activity than other encapsulates. Hence spray dried and freeze dried MD encapsulates were incorporated into bread and evaluated for their quality characteristics. Green tea extract and encapsulates incorporated bread retained their quality in terms of volume and crumb firmness.

Perera *et al*. (2015) studied the effects of raw material on the chemical composition, organoleptic properties, antioxidant activity, physical properties and the yield of instant black tea. Experiment was conducted with dhool (particles resulting from rolling of green leaf) fermented for varying durations, black tea and the rejected fraction in black tea manufacture which is known as broken mixed fannings (BMF) as raw materials. Total polyphenols and theaflavins contents of instant black teas prepared by using fermented dhools were significantly higher than those of instant black teas prepared by using black tea and BMF. Further, trolox equivalent antioxidant activities of instant black teas prepared by using fermented dhools were significantly higher than those of instant black teas prepared by using black tea and BMF. Type of raw material did not affect significantly on the yield and fluoride and aluminium contents of instant black tea.

2.4 PHYSICAL AND CHEMICAL PROPERTIES OF INSTANT FLAVOURED GREEN TEA

Chegini and Ghobadian (2005) investigated the effect of feed ratio, atomizer speed and inlet air temperature on properties of spray-dried orange juice powders. The results indicated that with increase in inlet air temperature there was increase in

particle size, average time of wettability and insoluble solids and decrease in bulk density and moisture content of the powder. Increase in atomizer speed resulted in increase in the bulk density and average time of wettability of powder and decrease in particle size, moisture content and insoluble solids of powder. With increase in feed flow rate, there was increase in bulk density, particle size and moisture content of the powder and decrease in average time of wettability and insoluble solids of powder.

Nijdam and Langrish (2005) investigated the morphological changes that occurred to milk particles during the spray drying process. They have confirmed that the bulk density of spray-dried milk powder was greatly affected by the drying temperature, due to the strong influence of the latter on the porosity of the particles. The drying temperature affects the bulk density of the dried powder, because the degree of inflation and shrivelling of particles influences their porosity.

Kha *et al.* (2010) studied the effects of inlet drying air temperature (120/83°C, 140/94°C, 160/103°C, 180/112°C and 200/125°C) and maltodextrin addition (10, 20 and 30%) on the physico-chemical and antioxidant properties of the Gac aril powder. The drying air flow rate, compressor air pressure and feed rate were constant at 56 ± 2 ($m^3.h^{-1}$), 0.06 MPa gauge and 12 to 14 $ml.min^{-1}$, respectively. As maltodextrin concentration increased from 10 to 20%, the moisture content of samples significantly reduced from 4.87 to 4.06%. A similar trend was observed while increasing the drying temperature from 120 to 200°C, resulting in a significant drop in moisture content from 5.29 to 3.88% and increasing inlet air drying temperature resulted in decrease in bulk density. The values of pH, aw and water solubility index of the Gac powders in this study were not significantly affected by inlet air drying temperature and maltodextrin concentration ($p>0.05$).

Fazaeli *et al.* (2012) studied the effects of some processing parameters on moisture content, water activity, drying yield, bulk density and solubility of spray dried black mulberry juice powders. The drying yield obtained in the range of 45 to

82%. The highest drying yield (82%) and solubility (87%) found in powder obtained by blend of maltodextrin 6 DE and gum arabic. The lowest moisture content powders (1.5%) produced at the compressed air flow rate of 800 $l.h^{-1}$. The lowest water activity of powder ranged from 0.15 to 0.32. Eight percent maltodextrin (6 DE) and 9 DE found to have particle size of 5.06±0.10 and 6.65±0.15 µm, respectively. Particle produced with 8% maltodextrin (6 DE) with different compressed air flow rate at inlet air temperature of 130°C showed 0.3 to 0.4 $g.cm^{-3}$.

Goula and Adamopoulos (2010) invented a new technique for production of orange juice powder. Which mainly include spray drying of concentrated orange juice by dehumidified air as drying medium and maltodextrin (21 DE, 12 DE, and 6 DE maltodextrins) as drying agent. A pilot-scale spray dryer was employed for the spray drying process. The modification made to the original design consisted in connecting the dryer inlet air intake to an absorption air dryer. Concentrated orange juice was spray dried at 110, 120, 130, and 140°C of inlet air temperatures and 4, 2, 1, and 0.25 of concentrated orange juice solids/maltodextrin solids ratios. Resulted powder was tested for rehydration, moisture content, bulk density, hygroscopicity and degree of caking. After determining the powder propertied modification made by introducing the dehumidified air and combination of maltodextrin addition was proved to be an effective way producing orange juice powder.

Tee *et al*. (2012) optimised the spray drying process parameters for *Piper betle L* extract. The major properties which includes hydroxychavicol (HC) content, particle size distribution, moisture content, powder yield and hygroscopicity of spray dried *Piper betle L* powder. The experimental run and optimisation work were designed using Box-Behnken method of Response Surface Methodology. The optimum operation conditions for the highest HC content (229.29 ppm)with the lowest moisture content (6.99%), the smallest particle size (5.48 µm), highest powder yield (10.53 g)and lowest hygroscopicity (28.88%) were obtained at inlet air drying

temperature of 159.52°C, feed flow rate of 10.5 $ml.min^{-1}$ and aspirator rate of 98.33%.

Fernandes *et al*. (2013) optimised the spray drying process parameters for microencapsulation of rosemary oil with gum arabic as wall material. The independent parameters *viz.*, moisture content, hygroscopicity, wettability, solubility, bulk and tapped densities, particle density, flowability and cohesiveness were determined for the gum arabic concentration of 10-30%, spray drying temperature of 135-195°C, and flow rate of 0.5-1.0 $l.h^{-1}$ by employing 2^3 central composite rotational design. Bulk density was affected directly with gum concentration and affected inversely to the spray drying temperature. While, both gum concentration and spray drying temperature affected inversely to the particle density. The obtained data did not fit for the properties *viz.*, solubility, tapped density, flowability and cohesiveness. The gum concentration of 25%, inlet air temperature of 135°C, and feed flow rate of 0.7 $l.h^{-1}$ were observed with high quality yield of rosemary oil powder.

Caliskan and Dirim (2013) standardized the process parameters for the spray dried sumac extract powder. The inlet/outlet air temperatures were to 160/80, 180/90, and 200/100°C and the outlet air temperature varied based on feed flow rate. The total soluble solid content of sumac extract was 3.5% and was ranged between 10 to 25% (w/w) with maltodextrin addition. The physical, biochemical, functional and microstructure were analysed for the sumac extract powder. All the individual and the interactions of process parameters affected the properties of spray dried sumac powder.

Mishra *et al*. (2014) studied the effects of inlet air temperatures of 125, 150, 175 and 200°C and maltodextrin levels at 3, 5, 7 and 9% for production of amla juice powder. Amla juice was concentrated up to 40% in a rotary evaporator at 70°C temperature. Maltodextrin of varying concentration 5-9% (w/v) was added and stirred for about 15 min. The juice was fed into spray dryer. Results found that with increase

in drying temperature and maltodextrin concentration, free radical scavenging activity of the powder was decreased. Spray dried amla juice powder made with 7% maltodextrin and processed at 175°C inlet air temperature had less hygroscopicity, acceptable colour and potent free radical scavenging activity.

Sarabandi *et al*. (2014) reported the physical properties of spray dried grape syrup powder which is dried at inlet air temperatures of 150, and 170°C (maltodextrin)/(grape syrup solids) ratios of 4, 2, 1, and 0.5. The results showed that, by increasing inlet air temperature, moisture content, water activity, bulk density and hygroscopicity were decreased, while wettability and solubility were increased. Moisture content and water activity were significantly affected by the maltodextrin concentration. Bulk density, solubility, hygroscopicity and wettability were negatively influenced by maltodextrin concentration. Overall, increasing inlet air temperature and maltodextrin concentration led to reduced stickiness enhancing powder production yield.

Shishir *et al*. (2015) conducted the experiment on effect of maltodextrin concentrations at different drying temperatures on the physical and drying properties of the spray-dried pink guava powder. The most remarkable result was found in 15% of the MDC at 150ºC where lower particle size led to the highest bulk density 492 $kg.m^{-3}$) that was mostly desired, and the drying rate of about at 307 $gm.h^{-1}$ increased the powder yield by 15%. Lower colour change that indicates better colour retention than the others were found in 10% and 15% MDC at 150ºC. However, the powder produced with 15% of MDC at 150ºC was found to be more satisfactory because of the moderate moisture content within the target range of below 5%, the highest bulk density, maximum production of 60% and significant colour attributes.

Wang *et al*. (2015) revealed the impacts of spray-drying conditions on the physicochemical properties of soy sauce powders using maltodextrin as auxiliary drying carrier. Soy sauce was spray-dried using a pilot-scale spray-dryer with two-

fluid atomization nozzle. Maltodextrin (DE 10) was incorporated into liquid soy sauce as a drying aid, to form a uniform liquid of 40% w/v. The influence of spray-drying parameters including inlet drying air temperature, feed flow rate, and atomization air flow was examined. The product yield, bulk density, cohesiveness and particle size of the spray-dried powders were found to be influenced by the spray-drying conditions. Extremely cohesive soy sauce powders were obtained when the air-drying temperature reached 180°C and above.

A-sun *et al*. (2016) studied the effects of inlet air temperature (120°C, 150°C and 180°C) and maltodextrin concentrations (DE 9-12) at 10%, 20% and 30% (w/v) on physical properties of spray dried coconut sugar powder. Inlet air temperature of 180°C caused a significant decrease in the moisture content, water activity, bulk density and solubility of the spray dried product. The increased inlet air temperature made the product more flowability or wettability. In addition, increasing of maltodextrin concentrations resulted in increased bulk density (0.43-0.73 $g.ml^{-1}$) and flowability (36º-56º), decreased solubility (39-103 s) and wettability (21-55 s). However, there were no significant changes in the moisture content (1.92%-3.03%) and water activity (0.20-0.33) of the coconut sugar powder at all the maltodextrin concentrations.

2.5 PACKAGING, SORPTION ISOTHERM KINETIC AND STORAGE STUDIES FOR INSTANT FLAVOURED GREEN TEA

Kumar and Mishra (2004) tested the storage stability of mango soy fortified yoghurt powder which was packed in HDPP and ALP. The shelf life of mango soy fortified yoghurt was predicted on the basis of free flowness of product under accelerated storage condition (38±1°C, 90% relative humidity) and was calculated to be 45 and 54 days in HDPP and ALP, respectively. The storage stability of mango soy fortified yoghurt powder in terms of quality parameters free fatty acid, thiobarbituric acid, hydroxymethyl furfural, starter counts and colour change was

studied in both packaging materials. The magnitude of quality change of mango soy fortified yoghurt powder measured during storage suggests that ALP was better than HDPP.

Jaya and Das (2005) studied on accelerated storage, shelf-life and colour of mango powder. The mango powders were packed in aluminum foil laminated pouches and stored in an accelerated storage environment maintained at 90±1% relative humidity and 38±1°C. The shelf-life of the powder predicted from this consideration and the Guggenheim-Anderson-de Boer (GAB) model for the water activity moisture content relationship was 114.68 days, whereas the actual shelf-life was 105 days. The colour change of the powder during storage followed first-order reaction kinetics with a rate constant of 0.038 per day.

Shrestha *et al*. (2007) conducted experiment on glass transition and water sorption properties of lactose hydrolysed skim milk powder produced from spray dryer. The sorption isotherm data for skim milk powder with hydrolysed lactose fitted well with the BET and GAB models with monolayer moisture contents of 7.55 and 8.27 g.100 g^{-1}, respectively. The glass transition temperature of anhydrous SMPHL was 49°C. The critical water activity and moisture content for skim milk powder with hydrolysed lactose were 0.15 and 2.4 g/100 g dry solid, respectively. The low critical values indicated hydrolysis of lactose necessities maintenance of very low moisture of powder for its long-term stability.

Goula *et al*. (2007) determined the adsorption isotherms of tomato pulp spray dried in dehumidified air by gravimetric technique at six temperatures between 20 and 70°C. The data obtained were fitted to several models including two-parameter (BET, Halsey, Smith and Oswin), three-parameter (GAB), and four-parameter (Peleg) relationships. The isosteric heat of sorption was determined from the equilibrium adsorption data using the Clausius-Clapeyron equation. Isosteric heats of sorption were found to decrease exponentially with increasing moisture content.

Sinija and Mishra (2008) conducted experiment on moisture adsorption isotherms of green instant tea powder and green tea granules were determined at 20, 30, 40 and 50°C. A gravimetric static method was used under 0.11–0.90 water activity ranges for the determination of sorption isotherms that were found to be typical type II sigmoid. Experimental data were fitted to various mathematical models and found that Peleg model suits best in describing equilibrium moisture content–equilibrium relative humidity (EMC–ERH) relationships for instant green tea samples as well as green tea granules, over the entire range of temperatures. The net isosteric heat of sorption was determined from the equilibrium data at different temperatures. The isosteric heat of sorption varied between 48.54 and 44.71 $kJ.mol^{-1}$ at moisture levels varying between 1 and 9 $g.g^{-1}$ dry matter for instant green tea powder and 47.96 and 44.10 $kJ.mol^{-1}$ at moisture level varying between 0.2 and 1.4 $g.g^{-1}$ dry matter for green tea granules.

Ramachandra and Rao (2013) studied the shelf-life and colour change kinetics of Aloe vera gel powder under accelerated storage in three different packaging materials. The powder was packaged in the three different packaging materials *viz.*, laminated aluminum foil (AF), biaxially oriented polypropylene (BOPP) and polypropylene (PP). The shelf-life of the powder was predicted on the basis of free flowness of product under accelerated storage condition (38±1°C, 90±1% relative humidity) and was calculated to be 33.87, 42.58 and 51.05 days in BOPP, PP and AF, respectively. The magnitude of colour change of Aloe vera gel powder during storage suggests that AF was better than BOPP and PP.

Langova *et al.* (2012) studied the water sorption isotherms of skimmed milk powder within the temperature range of 5-20°C. The equilibrium moisture content (EMC) of skimmed milk powder samples was growing with an increase of water activity (a_w) at a constant temperature both for water adsorption and desorption. Isotherms were found to be type II of Brunauer-Emmett-Teller classification (BET).

Structural modifications of crystals were observed during adsorption. Critical value of EMC of tested samples corresponding to the a_w equal to 0.6 for adsorption was 6.50% MC (w.b.) at temperature 5°C, 9.15% MC (w.b.) at temperature 10°C, and 7.71% MC (w.b.) at temperature 20°C.

Langova and Stencl (2014) studied the moisture sorption isotherms of whole milk powder in the temperature range of 5-35°C and critical values of water activity (a_w) prediction. Critical values of the equilibrium moisture content (EMC) from the viewpoint of microorganism growth corresponding to a_w of 0.6 were calculated for the temperature range tested. The critical EMC was 7.82% and 8.51% (w.b.) for water adsorption and desorption, respectively, at the temperature of 20ºC. Sorption capacity of samples tested decreased as temperature increased, and vice versa.

Wong and Lim (2016) conducted experiment on storage stability of spray-dried papaya powder packaged in aluminium laminated polyethylene (ALP) and polyethylene terephthalate (PET) at accelerated storage (38 ± 2°C, 90% relative humidity) for 7 weeks. The final water activity (a_w) of the papaya powder was less than 0.6 for both packaging materials, which showed that the powder was shelf-stable. Packaging materials significantly ($p<0.05$) influenced spray-dried papaya powder's water activity, moisture content, water solubility index, hygroscopicity, degree of caking, flowability and colour. They concluded that ALP packaging with storage conditions of 38°C and RH 90% was better suited for keeping spray-dried papaya powder.

Bastioglu *et al*. (2016) conducted experiment on moisture sorption isotherms of yogurt powder, plain, and containing 5, 10, 20% candied chestnut puree at 25°C using the standard, static-gravimetric method. The experimental adsorption data of yogurt powders fitted to 14 sorption equations which are most widely used to fit experimental sorption data of various food materials. The GAB model gave the closest fit to the sorption data of freeze dried yogurt powders with candied chestnut

puree at 25°C. BET, Ferro Fanton, Henderson, Halsey, Oswin and Modified Oswin models are also acceptable for describing the adsorption isotherms for freeze dried yogurt with candied chestnut puree at 25°C.

Singh and Hathan (2017) investigated the effect of different packaging materials (HDPE and LAP) on moisture content, betalain content and colour during storage of beetroot powder. Both packaging material and storage period showed significant effect on powder properties. The magnitude of quality changes of beetroot powder measured during accelerated storage suggested that LAP was better choice than HDPE for long term storage of spray dried beetroot powder.

CHAPTER III

MATERIALS AND METHODS

This chapter deals with the materials and methodology used to obtain instant flavoured green tea, processing steps and process parameters adopted. It describes the standardised methods used to carry out the physical, functional, biochemical analysis of the developed instant flavoured green tea and storage characteristics of the three best instant flavoured green tea. In last section it deals with moisture sorption phenomenon of instant flavoured green tea powder with regard to temperature dependence and isotherm modeling.

3.1 PROCUREMENT OF RAW MATERIALS

Dried green tea leaf and dust green tea were procured from Kannan Devan Hills Plantations Company (P) Ltd. Kerala, India. Prior to the major experiments the quality parameters like polyphenols and extractable total soluble solids were estimated.

3.2 PROCESS PROTOCOL FOR PRODUCTION OF FLAVOURED INSTANT GREEN TEA

Processing steps involved in production of instant flavoured green tea includes extraction of green tea in water, concentrating the extract, addition of flavours to the concentrated extract, drying of concentrated extract and packaging of instant flavoured green tea. Processing technology for the production of instant flavoured green tea is given in Fig. 3.1

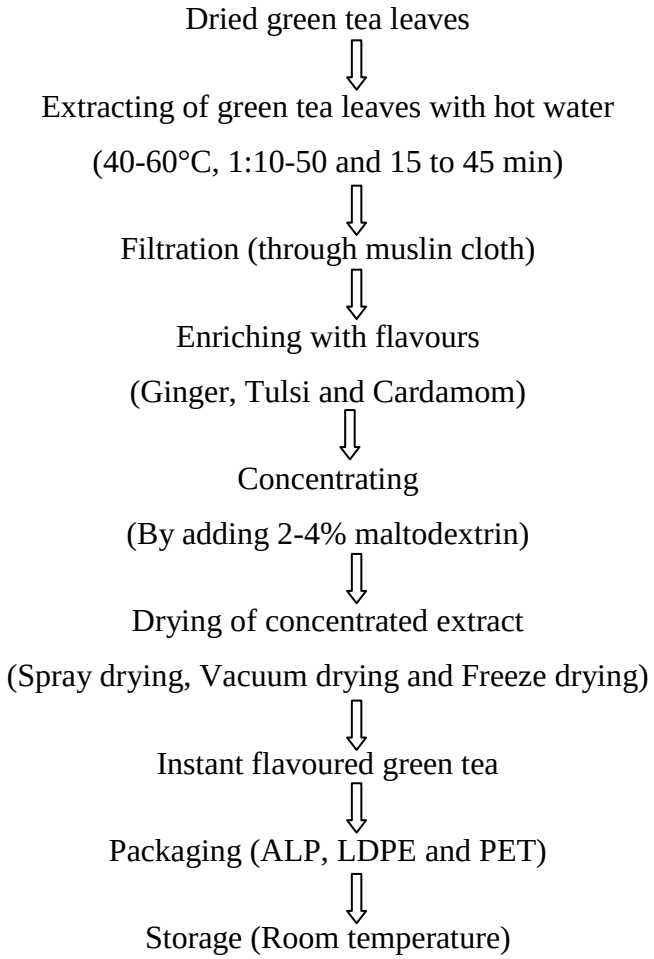


Fig. 3.1 Process flow chart for production of instant flavoured green tea

Experiment I

3.3 OPTIMISATION OF PROCESS PARAMETERS FOR THE EXTRACTION OF GREEN TEA

This study was carried out with dust green tea and dried green tea leaves. Major quality parameters like total polyphenols and extractable total soluble solids were estimated.

3.3.1 Extraction of Green Tea

Extraction was conducted with optimized type of green tea. Extraction trials was conducted with hot water in three different temperature, time and leaf-water ratio. The leaf-water ratio were 1:10, 1:30 and 1:50 for different extraction time 15, 30 and 45 min at three levels of temperature 40, 50 and 60°C. Extract was analysed for total polyphenols, total flavonoids, caffeine, tannins and antioxidant activity.

3.3.2 Experimental Design for Extraction of Green Tea

Response surface methodology (RSM) was adopted in the experimental design as it emphasizes the modeling and analysis of the problem in which response of interest is influenced by several variables, and the objective is to optimize this response (Montgomery, 2001). RSM is a statistical method based on the multivariate non-linear model that has been widely used method for optimisation process. It is useful in studying the interactions of the various parameters affecting the process. The main advantage of RSM is to reduce the number of experimental runs needed to provide sufficient information for statistically acceptable results.

Box-Behnken experimental design was employed for best experiment combination. The use of the Box–Behnken design is popular in industrial research because it is an economical design and requires only three levels for each factor where the settings are -1, 0 and 1. The independent variables considered were leaf-

water ratio, time and temperature. The coded and uncoded independent variables used in the RSM design were listed in Table 3.1. A 17-run Box–Behnken design with three factors and three levels, including five replicates at the centre point is given in Table 3.2. Statistical analysis was done with the aid of the Design-Expert software version 7.0.0.

The desired goals for each variable and response were chosen. The values of all the responses at operating conditions were converted to a desirability function. The desirability values of the minimum and maximum were configured as 0 and 1, respectively. All of the independent variables were kept within range, while the responses were either maximised or minimised. Numerical optimisation was applied for extraction of green tea, on the basis of quality parameters. The quadratic response surface analysis was based on multiple linear regressions taking into account linear, quadratic and interaction effects according to the equation below:

$$Y = b_0 + \sum a_i x_i + \sum a_{ij} x_i x_j + \sum a_{ii} x_i^2 \quad \text{... (3.1)}$$

Where Y is the response value predicted by the model; b_0 is offset value, a_i, a_{ij} and a_{ii} are main (linear), interaction and quadratic coefficients, respectively.

The adequacy of the models was determined using model analysis; lack-of fit test and coefficient of determination (R^2) analysis. For model to be suited, R^2 should be at least 0.80 for a good fitness of a response model (Mirhosseini *et al*., 2009).

3.3.3 Verification of Predicted and Actual Responses

To verify the predicted and actual responses of spray dried instant green tea powder, the mean relative per cent deviation modulus was calculated by using the following relation:

$$\text{Per cent relative deviation} = \frac{100}{N} \sum_{i=1}^{N} \frac{|e_i - p_i|}{p_i} \quad \text{… (3.2)}$$

Where,

N = Total number of observations

e_i = Experimental value

p_i = Predicted value

The best combination of time temperature and leaf to water ratio was selected based on the quality parameters of the extract and drying was proceeded with optimized extraction parameters.

Table 3.1 Uncoded and coded independent variables used in RSM design for extraction of green tea

Code	Independent variable	Coded levels		
		-1	0	+1
A	Leaf-water ratio	1:10	1:30	1:50
B	Time (min)	15	30	45
C	Temperature (°C)	40	50	60

Table 3.2 Box-Behnkan design with independent response variables for extraction of green tea

	Coded factors			Decoded factors		
Run	Water	Time (min)	Temperature (°C)	Water	Time (min)	Temperature (°C)
1	-1	-1	0	10	15	50
2	+1	-1	0	50	15	50
3	-1	+1	0	10	45	50
4	+1	+1	0	50	45	50
5	-1	0	-1	10	30	40
6	+1	0	-1	50	30	40
7	-1	0	+1	10	30	60
8	+1	0	+1	50	30	60
9	0	-1	-1	30	15	40
10	0	+1	-1	30	45	40

11	0	-1	+1	30	15	60
12	0	+1	+1	30	45	60
13	0	0	0	30	30	50
14	0	0	0	30	30	50
15	0	0	0	30	30	50
16	0	0	0	30	30	50
17	0	0	0	30	30	50

Experiment II

3.4 OPTIMISATION OF DRYING METHODS FOR INSTANT GREEN TEA POWDER

In order to obtain instant green tea powder, extract prepared with optimised condition is subjected to three drying methods *i.e*., freeze drying, vacuum drying and spray drying.

3.4.1 Freeze Drying

A freeze dryer (model-DPRG-110; make: LYODEL Advance) of 1.5 kg maximum ice capacity was used for drying of green tea extract sample which is shown in Plate 3.1. Before placing the green tea extract samples in a freeze dryer, it was placed in a deep freezer at a temperature of -20 °C for 5-6 hours to solidify the moisture present in the green tea extract and then again they were placed in a freeze dryer for 36 h. The drying process was carried out at a temperature of -47±1°C.

3.4.2 Vacuum Drying

The vacuum dryer (Make: Milk Tech Engineers, Model-GMP, 600×600 mm) consists of hot water generator, condensation unit, control unit, three pumps namely hot water pump, vacuum pump and cold water pump. Vacuum dryer used in present study is shown in Plate 3.2

Water was filled in the SS tank which feeds to water pump and hot water condenser. Then the drying time was set to minutes on digital process timer (0 – 999

minutes) and also the hot water generator was set as 60°C. After that the hot water pump, cold water pump and heater were switched on. The vacuum gauge showed 680 mm Hg vacuum after setting all these parameters. Once the drying was complete the vacuum pressure is released and then the product was taken out for the further analysis.

3.4.3 Spray Drying

Spray drying is a one-step, continuous process, allowing ease of scale-up. It is also useful for the processing of heat-sensitive materials due to very short exposure period to heat, which can range in the order of 5 to 10 s. The main factors that were optmised in spray drying were inlet air temperature, feed rate and feed concentration. Spray drying is a method where the result strongly depends upon the material properties and factors of spray drying in a combined system that influence the product parameters (Patel *et al*., 2009).

The spray dryer (Make: SM Scientech, Kolkata), a vertical co-current type with an evaporation rate of 1000 $ml.h^{-1}$, was used in the present study and it is shown in Plate 3.3 . The components of spray dryer are air filter, air heater, air distributor, two fluid nozzle, drying chamber, collection glass bottles, cyclone separator and air compressor. For present research work, distilled water was pumped into the spray dryer to adjust the spray drying temperatures. The dryer was run at this condition for about 20 min before the concentrated feed was introduced. The feed was introduced into the spray dryer through feed pipe after adjusting the inlet and outlet air temperatures. Spray drying involved spraying the concentrated green tea extract using two fluid nozzles at different inlet temperature and feed pump rate, and at fixed blower rpm.

Plate 3.1 Freeze dryer used for drying of green tea extract

Plate 3.2 Vaccum dryer used for drying of green tea extract

Plate 3.3 Spray dryer used for drying of green tea extract

3.4.3.1 Spray dryer parameters

The spray dryer operating conditions and response variables are very important to produce the highest powder efficiency and yield. These response variables were selected as they were important indicators of microsphere functionality and process efficiency. Factors that can significantly affect the spray drying process and product characteristics include the feed rate, atomizing wheel speed, dryer inlet and outlet temperatures and drying air humidity. The atomizing wheel speed would affect the size of the drying droplets and the final particle in an inverse relationship. Spray dryer inlet temperatures had a more direct effect on the drying process, with droplet drying rates positively related to inlet temperatures used.

Green tea extract was prepared with optimized extraction parameters and it was concentrated by adding maltodextrin at three different levels. The experimental parameters were selected based upon the different trials. Concentrated green tea extract was spray dried with three different temperature and flow rate. Three levels of maltodextrins were 2, 3 and 4% as M_1, M_2 and M_3, respectively, spray drier inlet temperature was 160, 170 and 180°C as T_1, T_2 and T_3, respectively and three levels of feed flow rate is 420, 672 and 924 $ml.h^{-1}$ as F_1, F_2 and F_3, respectively was considered for the drying of green tea.

Level of maltodextrins, %	:	2, 3 and 4%
Spray dryer inlet temperature, °C	:	160, 170 and 180°C
Feed flow rate, $ml.h^{-1}$	:	420, 672 and 924 $ml.h^{-1}$

3.4.3.2 Experimental design for spray drying of green tea extract

Box-Behnken experimental design was employed (Montgomery, 2001) for optimisation of experiment combination. The significance of all terms in the polynomial were judged statistically by computing the F-values at a probability (P) of 0.0001 or 0.05. A complete second order quadratic model was employed to fit the data and adequacy of the model was tested considering R^2 (the coefficient of multiple determination, a measure of the amount of variation around the mean explained by the model). The mathematical model is reliable with an R^2 value closer to 1 (The closer

the R^2 value to 1, the better is the model fit to the experimental data). Coefficient of determination, R^2 is defined as the ratio of the explained variation to the total variation and is a measure of the degree of fit (Haber and Runyon, 1977).

It is also the proportion of the variability in the response variables, which is accounted for the regression analysis (McLaren *et al*., 1977). For any term in the models, a large F-value and a small P-value would indicate a more significant effect on respective response variables (Yuan *et al*., 2008). The regression coefficients were then used to make statistical calculation to generate three-dimensional plots from the regression model. The models provide several comparative measures for model selection. R^2 statistics, which give a correlation between the experimental response and the predicted response and it should be high for a particular model to be significant. Adjusted R^2, which gives a similar correlation after ignoring the insignificant model terms, should have good agreement with predicted R^2 for the model to be fit (Gonnissen *et al*., 2008).

The experimental design was done with the aid of the Design-Expert software version 7.7.0 (Statease Inc., Minneapolis, USA) to identify optimum levels of three independent variables. Feed concentration (%), inlet air temperature (°C) and feed flow rate ($ml.h^{-1}$) were chosen as independent variables. Thirteen responses were selected as dependent variables such as moisture content, yield, colour, water activity, loose bulk density, tapped bulk density, Hausner Ratio (HR), Carr's Index (CI), solubility, wettability, total polyphenols, total flavonoids and caffeine. The coded and uncoded independent variables used in the RSM design were listed in Table 3.3. A 17-run Box–Behnken design with three factors and three levels, including five replicates at the centre point, experimental points used according to this design are shown in Table 3.4. The test of statistical significance was performed on the total error criteria, with a confidence level of 95%. The significant terms in the model were found by analysis of variance (ANOVA) for each response. The adequacy of the model was checked by calculating R^2 and adjusted-R^2.

Optimisation technique was followed as explained in 3.3.3. All of the independent variables were kept within range, while the responses were either maximised or minimised. Numerical optimisation was applied for spray dried instant green tea on the basis of quality and properties of instant green tea. The quadratic response surface analysis was based on multiple linear regressions taking into account. Verification of predicted and actual responses was done according section 3.3.4.

3.4.4 Standardisation of Water to Instant Green Tea Powder Ratio by Sensory Evaluation

After standardizing the spray drying process parameters, the optimum instant green tea powder required for better taste was standardised by sensory evaluation with nine point hedonic scale. Green tea was prepared by adding various quantity of instant green tea powder to both hot and cold water (Table 3.5). The reconstituted instant green tea was compared with the freshly prepared green tea.

Table 3.3 Uncoded and coded independent variables used in RSM design for spray drying of green tea extract

Code	Independent variable	Coded levels		
		-1	0	+1
A	Temperature (°C)	160	170	180
B	Concentration (%)	2	3	4
C	Flow rate ($ml.h^{-1}$)	420	672	924

Table 3.4 Box-Behnkan design with independent response variables for spray drying of green tea extract

	Coded factor			Decoded factor		
Run/ Treatment	Temperature (°C)	Maltodextrin (%)	Feed rate ($ml.h^{-1}$)	Temperature (°C)	Maltodextrin (%)	Feed rate ($ml.h^{-1}$)
T1	-1	-1	0	160	2	672
2	+1	-1	0	180	2	672
3	-1	+1	0	160	4	672
4	+1	+1	0	180	4	672
5	-1	0	-1	160	3	420
6	+1	0	-1	180	3	420
7	-1	0	+1	160	3	924
8	+1	0	+1	180	3	924
9	0	-1	-1	170	2	420
10	0	+1	-1	170	4	420
11	0	-1	+1	170	2	924
12	0	+1	+1	170	4	924
13	0	0	0	170	3	672
14	0	0	0	170	3	672
15	0	0	0	170	3	672
16	0	0	0	170	3	672
17	0	0	0	170	3	672

Table 3.5 Treatment combination for reconstituted instant green tea

	Sample code	Quantity of instant green tea powder in 100 ml of water
Fresh green tea	T_0	0
Sample reconstituted with hot water	HT_1	0.75 g
	HT_2	1.00 g
	HT_3	1.25 g
	HT_4	1.50 g
Sample reconstituted with cold water	CT_1	0.75 g
	CT_2	1.00 g
	CT_3	1.25 g
	CT_4	1.50 g

Experiment III

3.5 OPTIMISATION OF FLAVOURING COMPOUNDS IN THE INSTANT GREEN TEA POWDER

An attempt was made to see the effect of two spices mainly ginger and cardamom and one herb tulsi in instant green tea powder. These herbs and spices are used for flavouring the cuisines and naturally occurring home food. Some of them have natural volatile oils or other components that influence on flavour and taste of some drinks and foods.

3.5.1 Ginger Flavoured Instant Green Tea

Ginger flavour to green tea powder was tried with two methods, by addition of ginger power and fresh ginger extract.

Commercially available ginger after washing, peeling and extract was taken after crushing. The extract was directly added to green tea extract in 4 different levels as given in Table 3.6. This mixture was spray dried along with optimum spray drying condition and maltodextrin to get ginger flavoured instant green tea.

The commercially available ginger after washing and peeling was dried at 60°C and powdered in hammer mill. This powder was added to hot water along with the green tea, and then the extract was spray dried at optimum spray drying condition and maltodextrin to get ginger flavoured instant green tea.

Table. 3. 6 Treatment combination for ginger flavoured green tea extract

Treatment	Percent of ginger juice	Treatment	Ginger powder : Dried tea leaves
G1	2% ginger juice	G5	1: 10
G2	4% ginger juice	G6	2:10
G3	6% ginger juice	G7	3:10
G4	8% ginger juice	G8	4:10

3.5.2 Cardamom Flavoured Instant Green Tea

Cardamom flavour was tried to incorporate to green tea by soaking the crushed cardamom (along with pods) in green tea extract at different concentration for various time periods as given in table 3.7. Then the extract was filtered before spray drying. The obtained powder is reconstituted according to standardised powder concentration and the reconstituted flavoured green tea was subjected to sensory analysis to know the best concentration of cardamom powder and time of soaking.

Table 3.7 Treatment combination for cardamom flavoured green tea extract

Treatment	Quantity of cardamom in 100 ml of green tea	Time of extraction (min)

	extract	
C1	1	20
C2	2	20
C3	3	20
C4	1	30
C5	2	30
C6	3	30
C7	1	40
C8	2	40
C9	3	40

3.5.3 Tulsi Flavoured Instant Green Tea

Addition of tulsi flavour was attempted by extracting the dried tulsi leaves with green tea in hot water together. The fresh green tulsi was collected from KCAET campus. Fresh tulsi was washed with running water and dried in hot air dryer at a temperature of 50°C (Hassanpouraghdam *et al*., 2010; Kadam *et al*., 2011).

For producing the tulsi flavoured green tea, dried tulsi was extracted in hot water along with the dried green tea leaves with 4 different ratios of tulsi at a temperature of 52°C for 30 min. Since the flavour of tulsi is not prominent in powdered instant green tea powder the extraction time for tulsi leaves was increased. Extraction time of tulsi leaves was increased by extracting the tulsi for additional time and then by adding the green tea leaves to same extract and further extracting both for 30 min and it is shown in table 3.8.

Table 3. 8 Treatment combination for tulsi flavoured green tea extract

Treatment	Tulsi: Dried green tea leaves	Time of extraction (min)	Additional time of extraction for tulsi	Total time of extraction (min)

H1	1:10	30	-	30
H2	2:10	30	-	30
H3	3:10	30	-	30
H4	4:10	30	-	30
H5	1:10	30	10	40
H6	2:10	30	10	40
H7	3:10	30	10	40
H8	4:10	30	10	40

All the instant flavoured green tea sample were packed immediately after drying in aluminium laminated pouches and stored under refrigerated condition for quality analysis. Quality of these sample were analysed by total flavonoids, total polyphenols and caffeine content. Sensory anlalysis of the same samples was done with nine point hedonic scale.

3.5.4 Statistical Analysis for Flavoured Instant Green Tea

Factorial completely randomised design (FCRD) was used to analyse the data. After proper analysis, data were accommodated in the tables as per the needs of objectives for interpretation of results. Statistical significance was examined by analysis of variance (ANOVA) for each response. Statistical analysis was done with the aid of Design-Expert software version 7.7.0

3.6 QUALITY ANALYSIS FOR GREEN TEA EXTRACT, INSTANT GREEN TEA POWDER AND FLAVOURED INSTANT GREEN TEA POWDER

3.6.1 Biochemical Analysis

3.6.1.1 Total flavonoids

Total flavonoid content was measured by the aluminum chloride colorimetric assay described by Hajimahmoodi *et al*. 2008. Total flavonoid content was expressed as mg catechin equivalents (CE)/g dry mass.

3.6.1.2 Total polyphenol

Total phenol content in the tea infusions was determined using the Folin–Ciocalteu reagent (Carloni *et al.,* 2013). Absorbance was read at 760 nm and the results were expressed as Gallic Acid Equivalents (GAE) using the linear regression value obtained from the gallic acid calibration curve.

3.6.1.3 Antioxidant activity

The FRAP (Ferric reducing antioxidant power assay) procedure described by Hajimahmoodi *et al*. 2008 was followed. The principle of this method is based on the reduction of a ferric-tripyridyl triazine complex to it's ferrous coloured form in the presence of antioxidants. FRAP reagent contained 5 ml of a (10 mmol/L) TPTZ (2, 4 ,6- tripyridyl- s- triazine) solution in 40 mmol/L HCL plus 5 ml of $FeCl_3$ (20 mmol/L) and 50 ml of acetate buffer, (0.3 mol/L, pH=3.6) and was prepared freshly and warmed at 37ºC . Aliquots of 100 µL sample were mixed with 3 ml FRAP reagent and the absorbance of reaction mixture at 593 nm was measured spectrophotometrically after incubation at 37ºC for 10 min. For construction of calibration curve five concentrations of $FeSO_4$ $7H_2O$ (1000, 750, 500, 250, 125 µmol/L) were used and then absorbencies were measured as sample solution. The values were expressed as the concentration of antioxidants having a ferric reducing ability equivalent to that of 1 mmol/L $FeSO_4$.

3.6.1.4 Caffeine

Ten milliliter of hot water extract of tea samples were taken in separating funnels, and 10 ml of chloroform was added to each sample. The separating funnel was shaken vigorously for 5 min. The solutions were then allowed to separate for 10

min at room temperature. Lower chloroform layer was collected for further analysis. One milliliter of this chloroform layer was diluted with pure chloroform appropriately to read absorbance. Absorbance of these solutions was measured at 277 nm against pure chloroform as blank using UV-VIS spectrophotometer. A standard curve was prepared for caffeine estimation in the concentration range between 10 and 100 µg (Paradkar and Irudayaraj, 2002).

3.6.1.5 Tannins

Tannins in green tea extract was determined by AOAC method (AOAC, 1980).

3.6.2 Determination of Functional Properties of Instant Green Tea Powder and Flavoured Instant Green Tea Powder

Functional properties such as colour, water activity, loose bulk density, tapped bulk density, Hausner Ratio (HR) and Carr Index (CI) of instant green tea and instant flavoured green tea were determined using standard procedures as explained below.

3.6.2.1 Colour

Hunterlab colourimeter (made by: Hunter Associates Laboratory, Reston, Virginia, USA) was used for the measurement of colour of green tea extract, instant green tea, instant flavoured green tea powder and in rehydration powders. The colour was measured by using CIELAB scale at 10° observer at D_{65} illuminant. It works on the principle of focussing the light and measuring energy reflected from the sample across the entire visible spectrum. The 3-dimensional scale L^*, a^* and b^* was used. The L^* is the lightness coefficient, ranging from 0 (black) to 100 (white), a^* represents greenness and redness (+100 for red and -80 for green) while b^* represents yellowness and blueness (+70 for yellow and -80 for blue), and H represents hue angle. The hue angle values vary from 0° (pure red colour), 90° (pure yellow colour), 180° (pure green colour) to 270° (pure blue colour).

The instrument was standardised before placing the sample by placing black tile and white tile provided with the instrument. Determination of colour values of samples was carried out thrice for all the samples and the average value was considered as colour values of green tea extract, instant green tea, instant flavoured green tea powder and in rehydration powders in terms of L^*, a^* and b^*values. The colour difference (ΔE) was determined for stored samples using the following equation:

$$\text{Hue angle } (°) = \tan^{-1}(b^*/a^*) \quad \ldots(3.3)$$

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad \ldots (3.4)$$

The subscript "0" in the equation represents the colour value of samples at 0^{th} month..

3.6.2.2 Water activity (a_w)

The water activity of instant green tea and instant flavoured green tea was measured using water activity meter (model: Aqua lab, Decagon Devices Inc., Pullman (Wa), USA). Before measuring the water activity, the instrument was calibrated for its accuracy by measuring the water activity of distilled water.

3.6.2.3 Bulk density

Bulk density includes loose bulk density and tapped bulk density. Loose packing is defined as the state obtained by pouring a powder sample into a vessel without any consolidation and tapped packing is defined as the state obtained when the vessel containing the powder sample is to be repeatedly dropped from a specified distance at a constant drop rate until the apparent volume of sample in the vessel becomes almost constant.

The bulk density of instant green tea powder from different treatments was measured according to the procedure described by Caparino *et al.* (2012).

Determination of loose bulk density and tapped bulk density of samples was carried out at room temperature thrice for instant green tea and instant flavoured green tea powder and the average value was considered as loose bulk density and tapped bulk density. The bulk density of powder was computed using the following expression;

$$\text{Loose bulk density } (g.cc^{-1}) = \frac{\text{Weight of powder (g)}}{\text{Bulk powdered volume (cc)}} \quad \ldots(3.5)$$

$$\text{Tapped bulk density } (g.cc^{-1}) = \frac{\text{Weight of powder (g)}}{\text{Tapped powdered volume (cc)}} \quad \ldots(3.6)$$

3.6.2.4 Moisture content

The moisture content of spray dried instant green tea powder was determined by AOAC hot air oven method (No. 990.20; AOAC, 2005).

3.6.3 Determination of Flowability and Cohesiveness of Instant Green Tea Powder

The instant green tea powder and instant flavoured green tea powder were evaluated for their flowability and cohesiveness in terms of Carr Index (CI) and Hausner Ratio (HR), respectively. Both CI and HR were calculated from the loose bulk and tapped densities of the instant green tea powder, according to formula given by Olayemi *et al*. (2008). Table 3.9 illustrates the some specifications about the compressibility index and the Hausner ratio.

3.6.3.1 Carr's index (CI)

The compressibility index of the instant green tea powder and instant flavoured green tea powder was determined by Carr's Index (Compressibility Index). It is a simple test to evaluate the Carr's Index from bulk density and tapped density of instant green tea powder and instant flavoured green tea powder. The formula for Carr's index is as below;

$$\text{Carr's Index } (\%) = \frac{\text{Tapped bulk density } (g.cc^{-1}) - \text{Loose bulk density } (g.cc^{-1})}{\text{Tapped bulk density } (g.cc^{-1})} \times 100 \quad (3.7)$$

3.6.3.2 Hausner ratio (HR)

The Hausner Ratio is a number that is correlated to the flowability of instant green tea and instant flavoured green tea powder. It was calculated by using the following expression;

$$\text{Hausner Ratio} = \frac{\text{Tapped bulk density } (g.cc^{-1})}{\text{Loose bulk density } (g.cc^{-1})} \quad \text{... (3.8)}$$

Table 3.9 Specifications for Carr's Index and Hausner Ratio

Sl. No.	Flowability	Carr's Index (%)
1	Very good	<15
2	Good	15-20
3	Fair	20-35
4	Bad	35-45
5	Very bad	>45
Sl. No.	**Flowability**	**Hausner Ratio**
1	Low	<1.2
2	Intermediate	1.2-1.4
3	High	>1.2

Source: Jinapong *et al*. (2008)

3.6.4 Determination of Reconstitution Properties of Instant Green Tea and Flavoured Instant Green Tea Powder

The properties of instant green tea powder are very important criteria for its acceptability. Reconstitution properties such as solubility and wettability instant green

tea powder and instant flavoured green tea were determined using standard procedures as explained below.

3.6.4.1 Solubility

The solubility of the product was measured according to the method reported by Zhang *et al.* (2013) and Shittu and Lawal (2007). One gram of (m_1) powder was put into the 50 ml beaker and then 10 ml distilled water (30±2 °C) was added. The suspension was stirred intermittently for 30 min before it was finally centrifuged at 9000 rpm for 10 min and then the supernatant was completely drained into an evaporating dish and dried to constant weight in a hot air oven at 105±2 °C for 4 h. The samples were removed from the oven, cooled in a desiccator and weighted as m_2. The drying and weighing processes were repeated until constant weight was obtained. Determination of solubility was carried out thrice for instant green tea and instant flavoured green tea powder and the average value was considered as solubility of instant flavoured green tea powder. The solubility was calculated using the following expression:

$$\text{Solubility (\%)} = \frac{10.m_2}{m_1} \times 100 \qquad \ldots (3.9)$$

Where,

m_1 = Initial weight of sample, g

m_2 = Final weight of sample, g

3.6.4.2 Wettability

The wettability of a powder is the time necessary to achieve complete wetting of a specified amount of powder, when it is dropped into water at a specified temperature. Wettability of the powder sample was measured according to the method

reported by Jinapong *et al.* (2008). One hundred ml of distilled water at 30±2 °C was poured into a 250 ml beaker. A glass funnel held on a ring stand was set over the beaker with the height between the bottom of the funnel and the water surface of 10 cm. A glass rod was placed inside the funnel to block the lower opening of the funnel. The powder sample (1 g) was placed around the glass rod and then the glass rod was lifted while the stop watch was started at the same time. Finally, the time was recorded for the powder to become completely wetted (visually assessed as when all the powder particles penetrated the surface of the water). Determination of wettability was carried out thrice for instant green tea and instant flavoured green tea powder and the average value was considered wettability.

3.6.5 Sensory Evaluation of Flavoured Instant Green Tea

The instant green tea prepared from different flavours was sensory evaluated by untrained panel member. Sixteen judges in the age group 23–50 years from non-smokers and non-beetle-leaf chewers belonging to faculty and research scholars of the of Food and Agricultural process Engineering department of KCAET, Tavanur were selected on good health, average sensitivity, interests in sensory evaluation, and familiarity with tea drinks (Sinija and Mishra 2011). Quality attributes selected for sensory evaluation were colour, flavour, taste, astringency and overall acceptability. Five-point hedonic scale was used for sensory evaluation and score card was given to bring out the inherent characteristics. The score card model is given in appendix D.

3.6.5.1 Fuzzy logic

The sensory analysis done subjectively was ranked based on its attribute preference mathematically using fuzzy comprehensive model (Sana *et al.,* 2016). The sensory preference score given by judges were taken and their linguistic judgment was converted to numerical ranking using fuzzy model. The attributes were assigned with respective values based on the preference given by sensory panels. Scores assigned for the instant flavoured green tea were colour and appearance – 0.21,

flavour – 0.21, taste - 0.18, astringency– 0.2 and overall acceptability – 0.2. The sensory analysis using fuzzy logic involved formation of three sets.

I.	Factor set (F_f)	Quality attributes of green tea (Colour and appearance, , Taste, astringency, Flavour and Overall acceptability)
II.	Evaluation set (E_f)	Scale factors for quality attributes (Excellent (EX), Good (GD), Medium (MD), Fair (FR) and Not Satisfactory (NS))
III.	Transformation set (T_f)	Numerical values for the evaluation set (EX =1, GD = 0.9, MD = 0.7, FR = 0.4, NS = 0.1)

The fuzzy model for sensory analysis was done through the membership functions represented below

Fuzzy membership function (FMF) - Value obtained by dividing the added individual scale factors with total number of judges

Normalized Fuzzy membership function (NFMF) - It is the function obtained by multiplying FMF and scale factor allotted to respective membership function.

Normalized Fuzzy membership function matrix – It is the matrix formulated by adding NFMF with its respective scale factors

Judgment membership function matrix (JMFM) – This is the deciding matrix for ranking. It could be obtained by adding the column values of all matrix and divide with highest total column value

Judgment subset (JS) – It is the final ranking of samples evaluated along with attributes preference of judges.

Experiment IV

3.7 STORAGE STUDIES FOR INSTANT FLAVOURED GREEN TEA POWDER

After the quality evaluation, the best one sample each of ginger, cardamom and tulsi flavoured instant green tea were packed in PET, laminated aluminum pouches and LDPE of 400 gauge and kept for storage studies at room temperature. The quality was test at one month interval for six months.

The variation of quality parameters such as moisture content, water activity, bulk density, colour change, solubility, wettability, total flavonoids, total polyphenols and caffeine while storage is considered for study.

3.7.1 Statistical Analysis for Storage of Instant Flavoured Green Tea

Factorial completely randomised design (FCRD) was used to analyse the storage data. Statistical significance was examined by analysis of variance (ANOVA) for each response. Statistical analysis was done with the aid of Design-Expert software version 7.7.0

3.8 SHELF-LIFE PREDICTION OF INSTANT FLAVOURED GREEN TEA POWDER BASED ON MOISTURE GAIN

Instant flavoured green tea powder with an initial moisture content *Xi* = 0.003 kg water/kg dry solids was packaged in three packaging material (ALF, LDPE and PET) and placed in an environment maintained at 38±1°C and 90±1% RH. After intervals of 7 days, one of the pouches was removed from the control environment and its contents analyzed for moisture content *X* (kg water/kg dry solids) and water activity (Jaya and Das, 2005).

3.8.1 GAB Model Constants of Spray Dried Instant Flavoured Green tea Powder

The GAB equation relating water activity a_w (fraction) and moisture content *X* (kg water/kg dry solids) is given

$$X = \frac{MCKa_w}{(1-Ka_w)(1-Ka_w+CKa_w)} \quad \ldots(3.10)$$

Where *C*, *K* and *M* are the GAB constants. *M* (kg water/kg dry solids) is the monolayer moisture content of food. Eq. (3.10) can be written as,

$$\frac{a_w}{X} = \left[\frac{K}{M}\left(\frac{1}{C} - 1\right)\right] a_w^2 + \left[\frac{1}{M}\left(1 - \frac{2}{C}\right)\right] a_w \qquad \ldots(3.11)$$

Best-fit values of $K(1/C\text{-}1)$, $(1\text{-}2/C)/M$ and $1\text{-}(MCK)$ for instant flavoured green tea powder could be obtained from *awm*/*Xm* and *aw* data by fitting a second order polynomial type regression equation between them. Denoting,

$$b_1 = K(1/C - 1)/M \qquad \ldots(3.12)$$

$$b_2 = (1 - 2/C)/M \qquad \ldots(3.13)$$

$$b_3 = 1/(MCK) \qquad \ldots(3.14)$$

It should be able to estimate the values *C*, *K* and *M* from the following equation.

$$K = \frac{-b_2 + \left(b_2^2 - 4b_3 b_1\right)^{0.5}}{2b_3} \qquad \ldots(3.15)$$

$$M = \left(b_2 + 2b_3 K\right)^{-1} \qquad \ldots(3.16)$$

$$C = (b_3 MK)^{-1} \qquad \ldots(3.17)$$

Eq (3.10) could be expressed for *aw* (fraction) as explicit function of *X* (kg water/kg dry solids) by the following equation.

$$a_w = \frac{2 + \left(\frac{M_o - 1}{X}\right)C - \left[\left(2 + \left(\frac{M_o}{X} - 1\right)C\right)^2 - 4(1 - C)\right]^{0.5}}{2k(1 - C)} \qquad \ldots(3.18)$$

Relative deviation per cent *E* between actual *Xm* and predicted *X* (Eq. 3.10) moisture content of instant flavoured green tea powder is given by

$$E = \frac{100}{m}\sum_{i=1}^{m}\frac{\left|X - X_m\right|}{X} \qquad \ldots (3.19)$$

where, m is the number of observed data on the moisture content and water activity. A value of E less than 10 is considered as reasonably good fit between predicted and actual values.

3.8.2 Moisture Gain and Storage Life Prediction

A linear relationship of the type shown in Eq. 3.20 between sticky point temperature *Ts* (°C) and the corresponding moisture content *X* (kg water/kg dry solids) was developed.

$$X = a_1 + a_2 T_s \qquad \ldots(3.20)$$

where, a_1 is the intercept of best-fit straight line drawn between *Xsm* and *Tsm* and a_2 was the slope of the straight line.

When the storage temperature was *Tp* (°C), powder would develop stickiness at moisture content *Xpc* (kg water/kg dry solids), which was its critical moisture content. The value of *Xpc* can be obtained from Eq (3.20) by substituting *Tp* for *Ts* and *Xpc* for *X* in the equation.

3.8.3 Permeability of Packaging Material

Regression equation relating saturation temperature T_g ($0<T_g<150$ °C) and the corresponding saturation pressure P_g^* (Pa) of water was developed from steam table data available in Geankoplis (1997). The equation was given by,

$$P_g^* = \exp\left(23.0603 - \frac{3723.67}{222.857 + \mathrm{T_g}}\right) \qquad \ldots(3.21)$$

Since, silica gel was a strong water adsorbent, small amount of water adsorption by the gel that took place during permeability measuring experiment would not appreciably raise the vapour pressure of water present within the gel.

Under this condition, water vapour permeability *kg* (kg.m^{-2}.day^{-1}.Pa^{-1}) of the packaging material could be using the following equation

$$k_g = \frac{S}{2b_g l_g P_g^* R_{hg}} \qquad \ldots(3.22)$$

where, S (kg water.s^{-1}) is the slope of straight line plot between storage time θ_s (s) and weight *W*g (kg) of silica gel kept within the packaging material. b_g (m) and l_g (m) are the width and and length of packaging material respectively in which the silica gel is kept. P_g^* (Eq 3.21) is the saturation vapour pressure of water at temperature T_g of environment in which the packaging material is kept and R_{hg} (fraction) is the relative humidity of the environment.

3.8.4 Shelf life of Instant Flavoured Green Tea Powder

When instant flavoured green tea powder stored inside the packaging material at temperature T_p (°C), rate of change of moisture content $dx/d\theta_p$ of powder with time of storage θ_p (s) can be expressed as,

$$W_p \frac{dX}{d\theta_p} = 2k_g b_p l_p \left(R_{hp} P_p^* - a_w P_p^*\right) \qquad \ldots(3.23)$$

where, W_p (kg) is the dry weight of instant green tea powder kept inside the package, R_{hp} (fraction) is the relative humidity of storage environment, *kg* (kg water.m^{-2}.s^{-1}.Pa^{-1}) is the permeability of packaging material, b_p (m) and l_p (m) are respectively the width and length of the package and $2b_p l_p$ (m^2) is the surface area of packaging material through which water vapour permeation takes place. a_w is the water activity of powder and X is its equilibrium moisture content after θ_p (s) of storage at temperature T_p (°C) of storage environment.

p^*_p (Pa) is the saturation vapour pressure of water at temperature T_p (°C). Value of p^*_p (Pa) can be obtained from Eq (3.21) by substituting p^*_p for p^*_g and T_p and

T_g in the equation. The term ($R_{hg}\ p^*_p$) in Eq (3.23) is the vapour pressure of water outside the packaging material and aw p^*_p is the vapour pressure inside the material.

Relationship between storage time θ_{ps} and moisture content X of powder can be obtained by putting the expression for aw from Eq (3.18) into Eq (3.23) and integrating the resulting equation.

$$\theta_{Ps} = \int_0^{\theta_{ps}} d\theta_p = \frac{W_p}{2k_g b_p l_p P_p^*} \int_{X_t}^{X_{pc}} \frac{dX}{R_{hp} - a_w} \qquad \ldots(3.24)$$

The integral on the right hand side of Eq (3.24) can be evaluated numerically for X within limits X_i and X_{pc} where, X_i and X_{pc} are the initial and critical moisture contents of powder respectively. From the solution of Eq (3.24), graphical relationship between storage time θ_p (s) and powder moisture content X can be developed. Shelf life θ_{ps} (s) of powder can be obtained from the above graph at X = X_{pc} (Das, 2005).

3.9 DETERMINATION OF SORPTION ISOTHERMS FOR SPRAY DRIED INSTANT FLAVOURED GREEN TEA

3.9.1 Static Gravimetric Method

The equilibrium moisture contents (EMC) of spray dried instant flavoured green tea powder were determined at 20, 30 and 40 °C by static gravimetric method. The static gravimetric method uses saturated salt slurries inside a sealable container to control relative humidity values (Airtight desiccators were used in this study). In order to cover the entire relative humidity range, eight salts were chosen ($KC_2H_3O_2$, $MgCl_2{\cdot}6H_2O$, K_2CO_3, $MgNO_3{\cdot}6H_2O$, $SrCl_2$, NaCl, KCl and K_2SO_4) and their corresponding relative humidity values at are listed in Table 3.10 ranging from 20 to 98% (Greenspan, 1977). When the slurry was added into the container, the salt crystals covered the bottom of the container, but did not protrude above the top of the liquid saturated salt slurry layer. This was important so that when the sample absorbed moisture from the salt slurry and more salt came out of the slurry, the liquid

layer was not depleted and maintains its specified relative humidity. The extra salt crystals at the bottom of each container were critical for accurate control of relative humidity. This was because when the salt slurry absorbed moisture from a sample the extra salt crystals dissolved in the absorbed water to maintain the saturated concentration of the salt slurry and, thus, the specified relative humidity.

Table 3.10 Saturated salt solutions at different temperatures and relative humidities

Sl. No.	Chemical name	Chemical symbol	20°C	30°C	40°C
1	Potassium acetate	$KC_2H_3O_2$	23.16	21.61	21.00
2	Magnesium chloride hexahydrate	$MgCl_2.6H_2O$	33.07	32.44	31.60
3	Potassium carbonate	K_2CO_3	43.16	43.17	42.00
4	Magnesium nitrate hexahydrate	$Mg(NO_3).6H_2O$	54.38	51.40	48.42
5	Strontium chloride	$SrCl_2$	65.00	67.00	65.00
6	Sodium chloride	NaCl	75.47	75.09	74.68
7	Potassium chloride	KCl	85.11	83.62	82.32
8	Potassium nitrate	KNO_3	94.62	92.31	90.72

Source: Sinija and Mishra (2008)

3.9.2 Procedures for Static Gravimetric Method

a. Saturated salt slurries were made by mixing more than the soluble amount of salt with distilled water so that the accurate corresponding relative humidity could be reached and maintained.

b. After the eight saturated salt slurries were made, the desiccators with different salts were prepared by placing the saturated salt slurry on the bottom and

separated porous plate to place the sample above the slurry. One desiccator was used for each relative humidity. Triplicate samples were used for each sample at each relative humidity.

c. One gram samples each were weighed in a disposable sterilized plastic sample holder. Samples were weighed to the nearest 0.001 g and placed on the porous plate inside the desiccator.

d. After securing the lids, air was removed to create vacuum, the desiccators were placed at 20, 25 and 30 °C in an incubator correspondingly.

e. Samples were weighed at 1-day intervals until the weight of each sample changed less than 0.1 mg/g dry weight for two consecutive weeks (Bell and Labuza, 2000) to ensure reaching equilibrium.

f. The equilibrium moisture content (EMC) on the dry basis was calculated using the initial weight (W_i), the initial moisture content (M), and the final weight (W_f).

$$EMC= [(W_f-W_i)+W_i\times M]/W_i \quad \ldots. (3.25)$$

g. The triplicate EMC values were averaged and plotted against the relative humidity or a_w to construct an isotherm of that material.

3.9.3 Modelling of Sorption Isotherm

EMC and a_w data were fitted to eight different sorption models recommended by many researchers, models were Guggenhein-Anderson-de Boer (GAB), Brunauer-Emmett-Teller classification (BET), Oswin, Smith, Modified Mizrahi, Chung-Pfost, Halsey and Henderson models. The eight models are represented in Table 3.11 In these models, X and Mo represent equilibrium and monolayer moisture content of instant flavoured green tea powder (kg water/kg solid), a_w is the water activity (fraction), Cg and Kg are GAB model constants.

Table 3.11 Isotherm models used for experimental data fitting

Sl. No.	Model	Equation
1	GAB	$X = C_g K_g M_O a_w / \left[(1 - K_g a_w)(1 - K_g a_w + c_g K_g a_w)\right]$
2	BET	$X = (X_m C a_w)/(1 - a_w)(1 + (C - 1)a_w)$
3	Oswin	$X = a\left(a_w/(1 - a_w)\right)^b$
4	Smith	$X = a + \left(b * \log(1 - a_w)\right)$
5	Halsey	$X = A_1(-\ln a_w)^{A_2}$
6	Henderson	$X = A_1[\ln(1 - a_w)]^{A_2}$

3.10 PARTICLE SIZE ANALYSIS AND FLAVOURED INSTANT GREEN TEA POWDER

Particle size analysis was done in Nano science and technology department, Tamil Nadu Agricultural University, Coimbatore, India. The Zetasizer nano range (Horiba SZ100, Japan) of instruments provides the ability to measure three characteristics of particles or molecules in a liquid medium. These three fundamental parameters are Particle size, Zeta potential and molecular weight. By using Zetasizer system these three parameters can be measured over a wide range of concentrations. Zetasizer (nano series) was used in the study as dynamic light scattering apparatus. The range of particle size that can be determined is: 0.6 nm - 6 μm. The equipment requires the sample to be diluted. Computer system uses the Malvern software for control of the equipment and for showing the results.

Particle size measurements were performed on aqueous suspensions of instant green tea powders. Aqueous suspensions were prepared as follows: one milligram of micro structured powder was carefully weighed and then dispersed in 10 ml of ethanol. The suspension was sonicated for 5 minutes in order to break powder agglomerates resulting fine, colloidal particles dispersed in ethanol. A small portion of the sample was taken by a syringe. A polystyrene cuvette was filled and placed in

the instrument for measurements. Care was taken to avoid air bubbles in the sample when filling the cuvette.

3.11 SEM ANALYSIS OF INSTANT FLAVOURED GREEN TEA

SEM analysis was done in Nano science and technology department, TNAU, Coimbatore. The SEM (Scanning Electron Microscope) is an instrument that produces a largely magnified image by using electrons instead of light to form an image. The microstructure of the instant flavoured green tea powders was examined using a scanning electron microscope (Model: Quanta 250, FEI, Czech Republic). To obtain SEM images, small amounts of powder were taken from well mixed powder samples. Powders were attached to a double sided adhesive tape on SEM stubs. SEM was operated with working distance (WD) of 8.3 mm using SE (secondary electron) detector with 15 kV (EHT-Extra high tension) at magnification of 1000× and 5500× and scan speed of 7 under the LAB 6 aperture with HV (high vacuum) mode.

3.12 X-RAY DIFFRACTION

X-ray diffraction analysis was done in Nano science and technology department, TNAU, Coimbatore. X-ray diffraction (XRD) characteristics of instant green tea powders of different flavoured were investigated usinga Siemens D-500 diffractometer (Rigaku, Japan). X-ray diffraction of powers were carried out as per the procedure explained by Caparino *et al*., 2012. Diffractograms were taken between 5 and 50° (2θ) with a step angle of 0.02° and scan rate of 1 s per step.

3.13 ESTIMATION OF CATECHIN FRACTION

Catechin fraction was estimated in United Planters' Association of Southern India (UPASI), Tamil Nadu. For quantification of catechins, standard ISO method (ISO 142502-2:2005(E)) is followed.

3.14 COMPUTATION OF HEAT UTILIZATION EFFICIENCY OF SPRAY DRYER USING MATLAB (R2013a)

The wide application of spray drying in research of new products increases the need for engineers to better understand energy calculation with mass and heat balance concerning the spray drying process. With the scarcity of energy and its rising cost, it is important to evaluate energy consumption on the drying process (Kajiyama and Park, 2010). In the present investigation, the heat utilization efficiency of spray dryer was computed using MATLAB software (R2013a) with following considerations;

Heat utilization efficiency of spray dryer =

$$\eta = \frac{\text{Amount of heat energy utilized by liquid drops}}{\text{Amount of heat energy supplied to ambient air}}$$

$$\eta = \frac{M_w \lambda_{Tw}}{M_a (H_{Tx} - H_{Ta})} \qquad \ldots(3.26)$$

Where,

M_w = Water evaporation rate, kg water.s^{-1}

λ_{Tw} = Latent heat of water evaporation at the wet bulb temperature of mixed air, J.(kg water)$^{-1}$

H_{Tx} = Enthalpy of heated air, J.(kg dryair)$^{-1}$

H_{Ta} = Enthalpy of ambient air, J.(kg dryair)$^{-1}$

The heat utilization of spray dryer was determined for best combination with following design, operating and performance parameters of the dryer during drying of water and green tea extract. The MATLAB programme for computation of actual heat utilization efficiency of spray dryer is given in Appendix H.

3.14.1 Design Parameters

Inside diameter of drying chamber, Dd = 0.367 m

Length of drying chamber, Ld = 0.91 m

Diameter of pipe at blower outlet, Db = 0.056 m

3.14.2 Operating Parameters

Volumetric flow rate of atomizing air at atmospheric pressure and temperature, Q_a (m^3 s^{-1}) and diameter of nozzle, d_{nozzle} (m) were measured using screw gauge and velocity of atomizing air was measured using anemometer, V_a.

This Qa was measured with the following equation

$$Q_a = A_{nozzle} \times V_a \quad \ldots (3.27)$$

Where,

Volumetric flow rate of atomizing air (m^3 s^{-1}) = Q_a

Area of the nozzle (m^2) = $A_{nozzle} = \frac{\pi}{4} \times d^2_{nozzle}$

Diameter of the nozzle (m) = d_{nozzle}

Velocity of atomizing air (m s^{-1}) = V_a

Dry bulb and wet bulb temperatures of atmospheric air were determined using sling dry and wet bulb thermometer and velocity of air and water vapour mixture at the blower outlet was measured using anemometer,

Dry bulb temperature of atmospheric air = T_a (°C)

Wet bulb temperature of atmospheric air = T_{aw} (°C)

Temperature of air and water vapour mixture at the blower = T_b (°C)

Velocity of air and water vapour mixture at the blower out let, $V_{aw} - \frac{\pi}{4} \times D_b^{\,2} \times N$, m.$s^{-1}$

Where,

Diameter of pipe at blower outlet (m) = D_b

Blower speed, N = 2000 rpm

Concentration of green tea extract = Xm (fraction)

Pressure of atmospheric air, P*a* = 101325 Pa

3.14.3 Performance Parameters

Maximum water flow rate into the dryer = M_w ($kg.s^{-1}$)

Maximum green tea extract flow rate into the dryer = M_m ($kg.s^{-1}$)

Temperature of air at the bottom of drying chamber during water evaporation = T_f (°C)

Temperature of air at the bottom of drying chamber during green tea extract drying = T_{fm} (°C)

Temperature of air at the blower outlet = T_b (°C)

3.14.4 Computation of Parameters from the Data Obtained During Water Drops and Green Tea Extract

3.14.4.1 *During drying of water drops*

Temperature of mixed air at atomizer outlet = T_x°C

Wet bulb temperature of mixed air at atomizer outlet = T_{xw} °C

Wet bulb temperature of mixed air was found out through a trial and error method from the following procedure (Das, 2005; Geankoplis, 2004),

Dry bulb temperature (T_a) and absolute humidity of air (Y_a) were known and these values were calculated from the MATLAB programme.

$$P = \exp\left(23.0603 - \frac{3723.67}{222.857 + T}\right) \quad \ldots (3.28)$$

$$Y = \left(\frac{18p}{28.9(Pa - p)}\right) \quad \ldots (3.29)$$

$$\lambda_T = 2501000 + 1880T - 4186T \quad \ldots (3.30)$$

$$Y = \frac{Y_w\lambda_T - 1005(T_a - T_w)}{\lambda_T + 1880(T_a - T_w)} \quad \ldots (3.31)$$

$$T_{xw} = \left(\frac{\left(\lambda_T Y_a - Y_W \lambda_T + 1005_{Tx} Y_a\right)}{1005 + 1880 Y_a} \right) \quad \ldots (3.32)$$

Where

P = Saturation vapour pressure (kPa)

T = Saturation temperature (° C)

Y = Saturation absolute humidity (g.m^{-3})

p = Partial pressure (Pa)

λ_T = Latent heat of vaporisation (kJ.kg^{-1})

T_{xw} was assumed first. The value of saturation vapour pressure P was obtained from Eq. 3.28. After substituting T_{xw} for saturation temperature T, value of saturation absolute humidity Y was obtained from Eq. 3.32. After substituting P for partial pressure p, value of latent heat of vaporisation λ_T was obtained from Eq. 3.30. After substituting T_{xw} for T, finally T_{xw} was obtained after substituting λ_T, Y_w, T_x and Y_a and this T_x was very close to T_{xw} assumed earlier for each treatment.

3.14.5 Heat Utilization Efficiency of Spray Dryer During Drying of Green Tea Extract

Actual heat utilization efficiency of spray dryer during drying of green tea extract droplets = η_{wm}

$$\eta_{wm} = \frac{M_w \lambda_{Txw}}{M_b (H_x - H_a)} \quad \ldots (3.33)$$

Where,

M_w = Rate of water evaporation from green tea extract droplets (kg.s^{-1})

M_b = Net mass flow rate of air leaving dryer (kg.s^{-1})

Latent heat of vapourization of water at wet bulb temperature of mixed air,

$T_{xwb} = \lambda_{T\,xw}$

$$\lambda_{Txw} = 2501000 + 1880T_{xwb} - 4186\,T_{xwb} \qquad \ldots(3.34)$$

Enthalpy of mixed air at temperature of $T_x = H_x$

$$H_x = (1005 + 1880Y_a)T_x + 2501000Y_a \qquad \ldots(3.35)$$

Enthalpy of atmospheric air at dry bulb temperature, $T_a = H_a$

$$H_a = (1005 + 1880Y_a)T_a + 2501000Y_a \qquad \ldots(3.36)$$

3.15 ECONOMICS OF INSTANT FLAVOURED GREEN TEA POWDER

The cost of production of instant flavoured green tea powder was estimated by considering the fixed, variable costs and other related costs. The costs of operation were determined by estimating the fixed cost and variable cost. The estimation of cost of instant green tea and instant flavoured green tea is given in Appendix I.

CHAPTER IV
RESULTS AND DISCUSSION

This chapter deals with the results and discussion of the experiments conducted on production of flavoured instant green tea powder and quality analysis. Second phase of investigation include storage studies, shelf life prediction and moisture sorption phenomenon for instant flavoured green tea powder.

4.1 PHYSICAL PROPERTIES OF RAW MATERIALS

Physical properties such as true density, bulk density and colour of dried green tea leaf and dust green tea was estimated as per standard procedure and tabulated (Table 4.1). The data showed that, the average true density is of dried whole green tea leaf and dust green tea is 0.125 and 0.334 $g.cc^{-1}$, respectively. The average bulk density values are 0.077 and 0.282 $g.cc^{-1}$ for dried whole green tea leaf and dust

green tea, respectively. The average colour values *viz*., L*, *a** and *b** of the dried whole green tea leaf were 22.763, 1.863 and 4.673, respectively. The *L**, *a** and *b** values for dust green tea *were* 26.400, 3.116 and 7.516, respectively. The moisture content is 3.3 and 3.5% (w.b.) for dried whole green tea leaf and dust green tea, respectively.

Table 4.1 Physical properties of dried green tea leaf

Sl. No.	Properties		Whole green tea leaf	Dust green tea leaf
1	Bulk density ($g.cc^{-1}$)		0.0773	0.282
2	True density ($g.cc^{-1}$)		0.125	0.334
3	Colour values	*L**	22.763	26.400
		*a**	1.863	3.113
		*b**	4.673	7.516
4	Moisture content (w.b.)		3.3%	3.5%

Experiment I

4.2 OPTIMISATION OF PROCESS PARAMETERS FOR THE EXTRACTION OF GREEN TEA

Dried green tea leaf and dust green tea were extracted with water with three levels of leaf to water ratio at four different temperatures and three levels of extraction time from 15 to 45 minutes. The quality of the extract was accessed in terms of total polyphenols, total soluble solids, total flavonoids, caffeine, tannis and antioxidant activity.

4.2.1 Total Soluble Solids

Yield of instant green tea powder depends on total soluble solids. The effect of extraction parameters on total soluble solid is shown in Table 4.2, and it is revealed that all three parameters had significant effect on total soluble solids. Maximum value of 26.47% was observed for green tea leaves extracted at 70°C for 45 min

extraction time. In both the cases (dried green tea leaves and dust green tea) with the increase in temperature, time and material to water ratio there was significant increase in the total soluble solids. Whereas, for dust green tea the soluble solids is only 14.95% under the same extraction condition. Similar trend was also observed by Pandey and Manimehalai, 2014 in black tea extraction with different time and temperature combination. Among the three parameters, material to solid ratio was most significant for total soluble solids. From Table 4.2, for increase in temperature from 40 to 60°C there was a significant increase in total soluble solids but further increase in temperature had no significant effect. Due to appreciable increase in yield further experiment was conducted only in dried green tea leaves.

4.2.2 Total Polyphenols

Polyphenols, the major content of green tea was estimated according to the procedure given in section 3.7.2. Variation of total polyphenols is given in Table 4.3. A maximum total polyphenols content of 50.40 $mg.g^{-1}$ in dried green tea leaves and 42.55 $mg.g^{-1}$ in dust tea was recorded at 60°C, 30 min extraction time and higher solid to water ratio. Whereas minimum of 25.25 $mg.g^{-1}$ in dried green tea leaves and 23.69 $mg.g^{-1}$ in dust tea was recorded at 40°C, 15 min extraction time and 1:10 solid to water ratio. From the table it is clear that as the temperature increases from 40 to 60°C there will be an increase in extraction of polyphenols, but further increase in temperature results in decrease in total polyphenol content.

Table 4.2 Effect of time, temperature and solid to water ratio of total soluble solids (%)

		Total soluble solids (%)					
		Dried green tea leaf			**Dust green tea**		
		Green tea: water			Green tea: water		
Time (Min)	Temperature (°C)	1:10	1:30	1:50	1:10	1:30	1:50

15	40	18.27	19.20	21.90	9.89	11.12	12.30
30		18.99	20.33	22.78	10.14	11.68	12.70
45		19.32	21.34	23.91	10.69	11.93	13.09
15	50	20.65	19.53	22.30	10.01	12.23	13.45
30		20.99	21.73	23.20	10.78	12.69	13.89
45		21.05	22.42	24.75	11.31	12.93	14.09
15	60	21.59	20.41	23.86	11.42	13.00	14.28
30		22.38	22.86	24.52	11.89	13.12	14.41
45		23.01	24.51	26.36	12.46	13.40	14.52
15	70	21.56	20.46	22.90	11.54	13.02	14.34
30		22.41	22.99	24.67	11.98	13.41	14.63
45		23.05	24.63	26.47	12.63	13.86	14.95

Table 4.3 Effect of time, temperature and solid to water ratio of total polyphenol (%) content

		Total polyphenols ($mg.g^{-1}$)					
		Dried green tea leaf			**Dust green tea**		
		Green tea: water			Green tea: water		
Time (Min)	Temperature(°C)	1:10	1:30	1:50	1:10	1:30	1:50
15	40	25.25	34.30	42.38	23.69	30.57	36.53
30		32.5	36.61	44.5	27.31	32.80	38.13
45		33.2	37.83	46.45	29.5	33.67	39.59
15	50	28.48	37.33	43.93	26.11	32.74	37.29

30		33.2	41.80	45.08	29.99	36.44	38.08
45		34.2	44.93	45.78	30.78	38.93	39.48
15	60	36.8	40.98	48.58	32.13	35.48	41.18
30		34.35	45.53	50.40	29.69	38.97	42.55
45		33.72	42.45	47.48	31.66	36.59	40.36
15	70	35.88	38.73	45.55	30.66	33.79	37.91
30		31.28	41.23	44.58	28.43	36.12	36.18
45		27.93	37.05	39.85	25.69	32.61	34.64

This might be due to the oxidation and epimerisation of catechin which are the main component of tea polyphenols above the temperature of 60°C (Ananingsih *et al*., 2013). In case of dust green tea also same trend was observed but with lower percentage of total polyphenols. An increase in polyphenols was observed for an extraction period up to 30 min and further increase in time had no appreciable effect and this might be due to degradation of polyphenols. Similar result was found by Ahmed *et al.,* 2013 for an extraction of photochemicals on green tea.

Since these two factors are major components which reflects the quality of product, green tea leaves was selected for further experiment. It was also noticed that the temperature greater than 60°C affect the quality. So, maximum temperature of

60°C was selected for further studies. The other component like total flavonoid, caffeine, tannins and antioxidant activity was estimated only in case of green tea leaf extract.

Regression model was fitted to experimental results of total polyphenols for selected treatment which is given in Appendix A1. Following regression model was obtained to predict the total polyphenols extracted from green tea.

$$\text{Total polyphenols} = 46.05 + 6.78(A) + 1.47(B) + 2.38(C) - 1.18(A*B) + 1.01(A*C) - 0.51(B*C) - 3.31(A^2) - 4.86(B^2) - 2.31(C^2) \quad \ldots(4.1)$$

For total polyphenols the coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, B^2 (Time^2), C (Temperature) and A^2(leaf to water ratio^2) were significant model terms with P- values of less than 0.05. A (leaf to water ratio) had largest effect on total polyphenols with P value less than 0.0001. The p-value of the model was <0.0001, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9519, which showed that the regression model defined well the true behavior of the system. Surface graph shows interaction of independent parameters which is given in Fig.4.1.

4.2.3 Total Flavonoid

Flavonoid compounds in tea have very strong antioxidant and free radical scavenging activities, and are much more effective than vitamins C and E at protecting cells from free radical damage. The maximum total flavonoids of 26.50 mg.g^{-1} was recorded at 60°C, 30 min and 1:50 leaf to water ratio and was on par with treatment (25.0 mg.g^{-1}) 50°C temperature, 45 min time and 1:50 leaf to water ratio. The minimum total flavonoids (12.5 mg.g^{-1}) were observed for treatment in 50°C temperature, 15 min time and 1:10 leaf to water ratio. The data obtained for total flavonoid content at different extraction condition is given in Appendix A1 and Fig 4.2 (b-c). Here there is an increase in total flavonoids content with increase in

temperature up to 50°C, and it might be due to the permeable of cell with the increase in temperature. Whereas, further increase in temperature to 60°C, a slight decrease in total flavonoids which may be due to degradation (Vuong *et al*., 2011). Total flavonoids increases with increase in extraction time up to 30 min and further increase in extraction time shows a decreasing trend (Fig. 4.2a and 4.2c). This might be due to the stabilisation of catechins after 30 min (Labbe *et al*., 2006).

Regression model fitted to experimental results of total flavonoid and it is given in Appendix A3. Following regression model was obtained to predict the total flavonoid extracted from green tea.

Total flavonoid = 23.48 + 3.69(A) + 2.19(B) + 2.50(C) + 1.37(A*B) + 0.50 (A*C) + 0.5(B*C) - 1.55(A^2) -4.55(B^2) - 2.18(C^2) …(4.2)

The magnitude of p-value indicates that all linear terms (A, B, C), quadratic terms B^2 and C^2 were highly significant 1% level ($p < 0.0001$), interaction term A*B quadratic terms A^2 were significant 5% level ($p < 0.05$). But interaction term B*C and A*C had not significant at 5 percent level ($p > 005$). The p-value of the model was <0.0001. The best fit model was expressed by the coefficient of determination R^2, which was 0.9937, indicating that 99.37 per cent of the variability of the response could be explained by the model.

4.2.4 Caffeine

Caffeine belongs to a group of compounds known as alkaloids and these can be preferentially extracted using water or by organic solvent. Here the extracted caffeine content of green tea ranged from 4.95 to 23.75 $mg.g^{-1}$. The highest value was observed at 1:50 leaf to water ratio, 30 min and 60°C temperature whereas, lower value was observed for 1:10 leaf to water ratio, 30 min and 40°C temperature.

It is clear from the Fig. 4.3, all the process parameters have positive effect on extraction of caffeine. When the temperature of extraction increases from 40 to 50°C, extraction of caffeine increases significantly but further increase in temperature do not have much effect. It clear from Fig 4.3, that there is no significant increase in

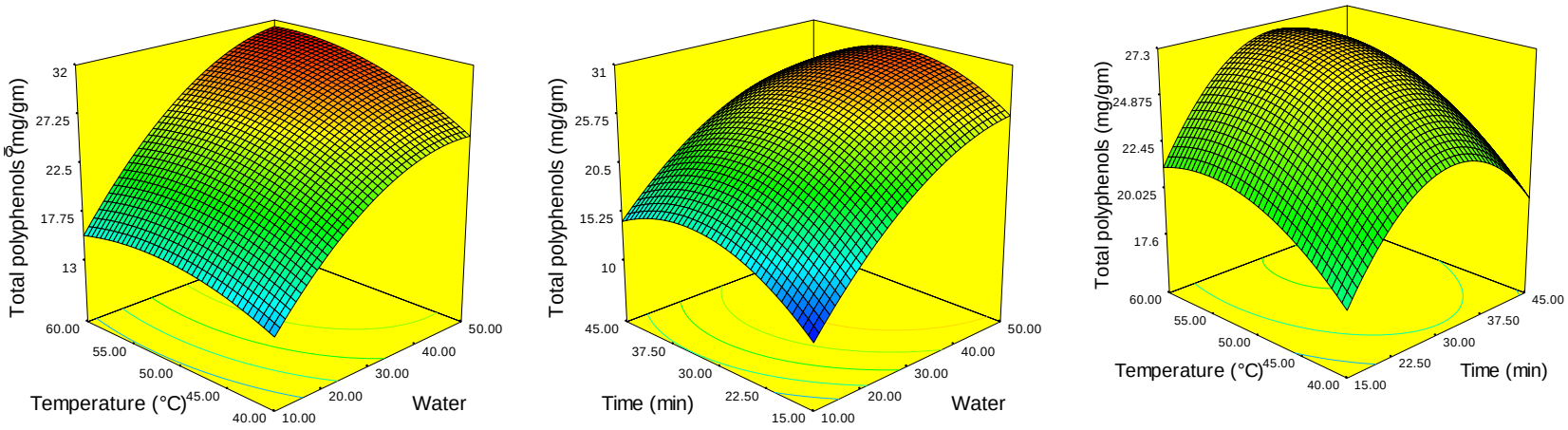
caffeine content with increase in time from 30 to 45 min of extraction. Same trend was also supported by Ziaedini *et al*., 2010; Labbe *et al*. (2006) and reported best time for caffeine extraction is 20-30 min.

The ANOVA for the response caffeine is given in Appendix A4. The second order non-linear regression equation was fitted between dependent and independent variables using the experimental values.

Regression model fitted to experimental results of extracted caffeine content and the following regression model was obtained to predict the caffeine extracted from green tea.

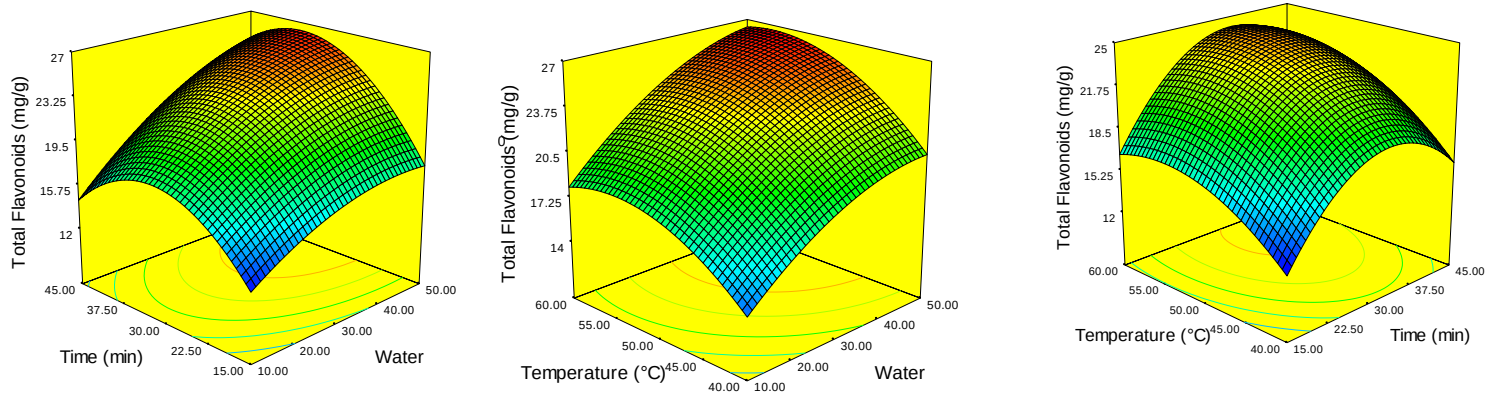
$$\text{Caffeine} = 15.96 + 3.98(A) + 1.10(B) + 5.23\ (C) + 0.76(A*B) + 0.12(A*C) + 0.012(B*C) - 1.13(A^2) - 3.75(B^2) - 0.60(C^2) \quad \ldots(4.3)$$

For caffeine the coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, A (leaf to water ratio) and B^2 ($Time^2$), were significant model terms with P- values of less than 0.05 and C (Temperature) was highly significant model terms with P- values of less than 0.0001.



a **b** **c**

Fig. 4.1 Response surface plots showing effect of extraction parameters on for total polyphenols



a **b** **c**

Fig. 4.2 Response surface plots showing effect of independent parameter on total flavonoids

The p-value of the model was 0.0007, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9533, which showed that the regression model defined well the true behaviour of the system.

4.2.5 Antioxidant Activity

Antioxidants are substances that considerably decrease the adverse effects of reactive oxygen species, reactive nitrogen species or both on normal physiological functions. Antioxidant activity of tea extracted in different condition ranged from 1.025-2.499 $mM.g^{-1}$. Green tea extracted at 1:50 leaf-water ratio for 30 min and 60°C had antioxidant activity of 2.499 $mM.g^{-1}$ which was on par with extract obtained at 1:50 leaf-water ratio, 45 min and 50°C (2.464 $mM.g^{-1}$). Extract obtained at 1:10 leaf-water ratio, 30 min and 40°C showed significantly lower antioxidant activity value of 1.025 $mM.g^{-1}$.

Antioxidant activity of tea extract increase with increment in all three parameters. High catechin levels have been positively correlated with tea radical scavenging properties (Tenore *et al*., 2015) and that might be the reason for high antioxidant activity at high temperature and time. Similar antioxidant value of green tea is also reported by Yashin *et al*., 2011, Hajimahmoodi *et al*. 2008 and Gimenez *et al*., 2013. Response surface generated for antioxidant activity as the function of two independent variables given in Fig. 4.4.

The ANOVA for the antioxidant activity is given in Appendix A5. The second order non-linear regression equation was fitted between dependent and independent variables using the experimental values. Regression model fitted to experimental results of green tea extract antioxidant activity and the following regression model was obtained to predict the antioxidant activity of green tea extract.

$$\text{Antioxidant activity} = 1.70 + 0.56(A) + 0.12(B) + 0.16(C) + 0.099(A*B) - 0.035(A*C) - 5.175E\text{-}003(B*C) + 0.079(A^2) - 0.11(B^2) + 9.050E\text{-}003(C^2) \quad \ldots(4.4)$$

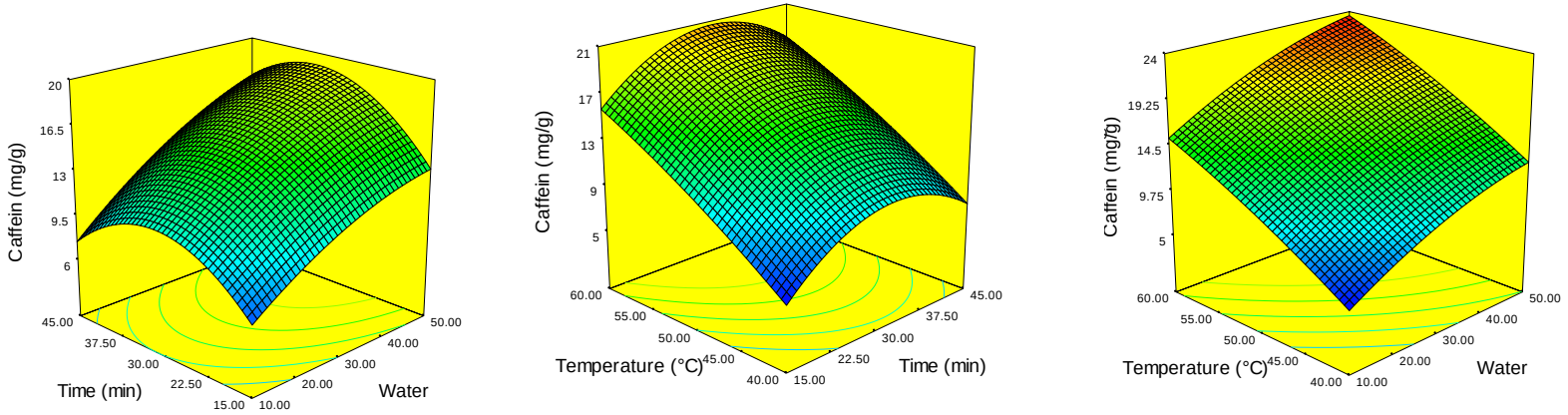


Fig. 4.3 Response surface plots showing influence of extraction process parameter on caffeine

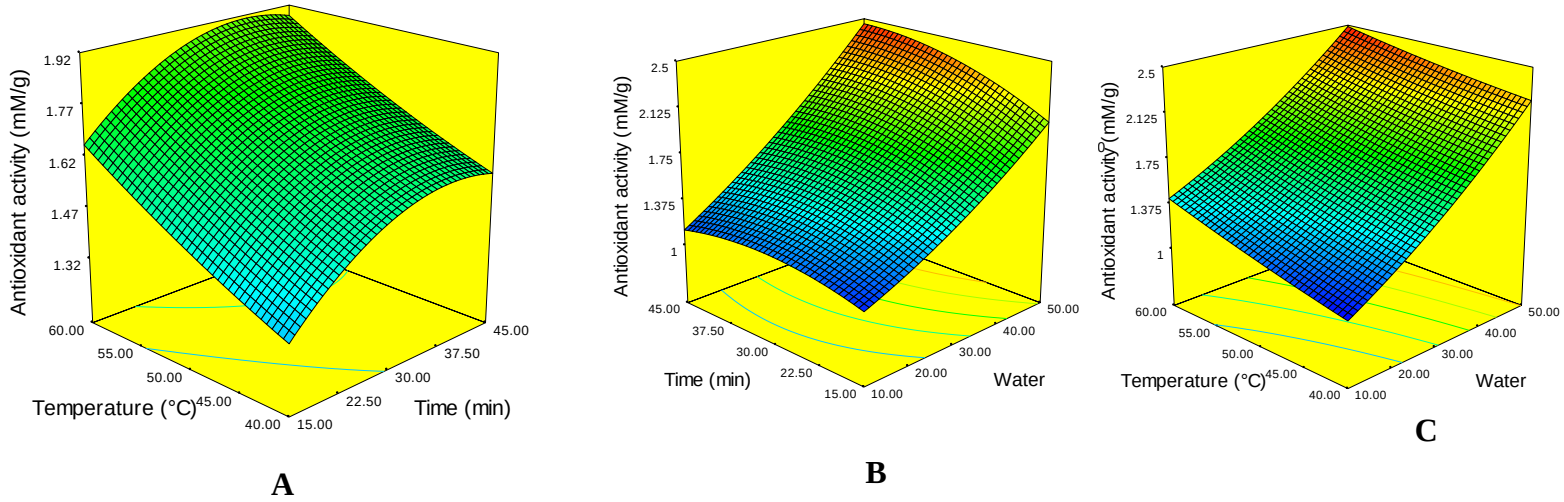


Fig. 4.4 Response surface plots showing effect of extraction process parameters on antioxidant activity

All linear coefficients (A, B and C), two quadratic coefficients (A^2 and B^2) and interaction factor of water and time had significant effect on antioxidant activity and among the six significant terms leaf-water ratio was highly significant with p value less than 0.0001. The mathematical model representing the antioxidant activity of green tea extract as a function of the independent variables within the region under investigation. The value of the determination coefficient for the equation for tea antioxidant activity is R^2 0.9895, which indicates that only 2% of the total variation is not explained by the model. The p-value of the model was less than 0.0001, which indicated that the model fitness was significant.

4.2.6 Tannin

Tea tannins are soluble in water and responsible for the typical bitter taste of tea. Extraction parameters have significant effect on tannins and it ranged from 1.181 to 3.964%. Green tea extracted at 50°C, 1:10 leaf-water ratio and 15 min had lowest tannin content and was on par with extract obtained at 1:10 leaf-water ratio, 30 min and 40°C which had 1.390% of tannins. Extract obtained at 1:50 leaf-water ratio, 30 min and 60°C showed significantly higher tannin value of 3.964%. Minimum tannins value of 1.181% was obtained at the lowest extraction time of 15 min, water ratio of 1:10 and 50°C temperature and it reached maximum of 3.964% at 60°C, 30min and 1:50 water ratio, similar tread was observed by Mache *et al*., 2015 for Khaya tea tannins and they reported that with increase in water volume, infusion time and temperature there will be increase in tannin extraction. Obtained values are in line with the findings of Kopjar *et al*., 2015 and Khasnabis *et al*., 2015 for commercially available green tea. Effect of two independent factors on tannins is given in Fig. 4.5.

ANOVA was performed to evaluate the significance of the coefficients of the quadratic polynomial models and it is given in Appendix A6. The second order non-linear regression equation was fitted for dependent and independent variables using the experimental values. Regression model fitted to experimental results of green tea

tannin content and the following regression model was obtained to predict the tannin extracted from green tea.

$$\text{Tannin} = = 2.15 + 0.82(A) + 0.46(B) + 0.51(C) + 0.19(A*B) + 0.20(A*C) + 0.20(B*C) + 0.041(A^2) - 0.046(B^2) + 0.32\,(C^2) \quad \ldots(4.5)$$

On the basis of the regression coefficients and the p-value, it was found that, all the interaction terms and the quadratic C^2 had significant effect on tannin content ($p<0.005$). All linear terms had greatest effect with $p<0.0001$. The p-value of the model was <0.0001, which indicated that the model fitness was significant. The model can fit well with the actual data when R^2 approaches unity. By analysis of variance, the R^2 value of this model was determined to be 0.9833.

Optimization of the three process variables namely; extraction temperature (40, 50 and 60ºC), time (15, 30 and 45 min) and green tea to water ratio (1:10, 1:30 and 1:50) was performed using the Box-Behnken design in Design Expert Software 7.7.0. In the present investigation, the independent variables were kept within the range and dependent variables were chosen as maximum and minimum. Appendix A7 shows response optimization constraints of experiment.

4.2.7 Verification of the Predicted Variables

The optimum response values were tested using the recommended optimum conditions of the variables and was also used to validate the experimental and predicted values of the responses. The experimental results had the optimum process conditions of 1:47 leaf-water ratio, 30 min extraction time and 52°C extraction temperature. The predicted, actual values of the responses and the percentage variation at the optimized condition of experiments are presented in Table 4.4.

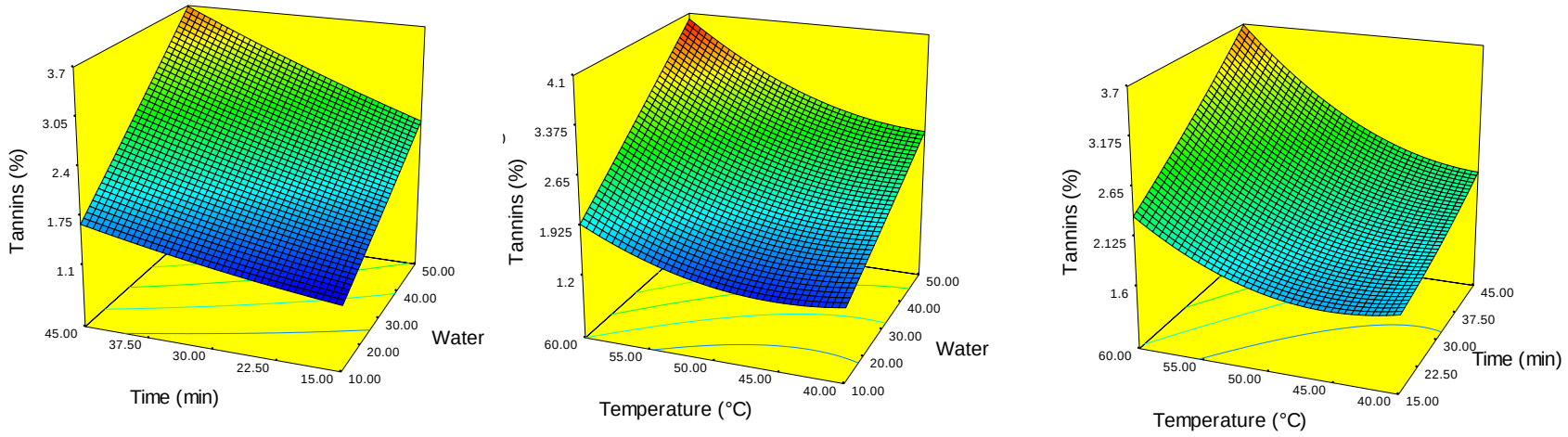


Fig. 4.5 Response surface plots showing influence of extraction process parameter on tannins

Table 4.4. Predicted and actual values of responses of the optimized condition

Sl. No.	Responses	Predicted value	Actual value	Variation (%)
1	Total Flavonoids ($mg.g^{-1}$)	26.40	26.87	+1.78
2	Total polyphenols ($mg.g^{-1}$)	50.16	50.26	+0.19
3	Caffeine ($mg.g^{-1}$)	19.20	19.22	+0.10
4	Antioxidant ($mM.g^{-1}$)	2.25	2.30	+2.22
5	Tannins (%)	2.92	2.89	-1.02

Experiment II

4.3 OPTIMISATION OF DRYING METHODS FOR INSTANT GREEN TEA POWDER

In order to optimise the drying method, the green tea extract was prepared with optimised parameters was dried in vacuum dryer, spray dryer and freeze dryer. The quality such as colour of instant powder and reconstituted sample along with overall acceptability by sensory analysis was as the criteria for the selection of drying method. The results are given in Table 4.5.

The extract was subjected to vacuum drying (60°C) with 3% maltodextrin, freeze drying condition with 3% maltodextrin and sprays dying (170°C). It is clear that, L* value of instant green tea powder obtained through vacuum drying is lower than freeze drying and spray drying, which indicates that vacuum drier sample is more darker. Hue value of reconstituted green tea indicates that, it is near to yellow and on par value can be noticed in spray dried tea. The overall acceptability of the spray dried tea sample (7.9) was higher than other samples and it is also comparable to the fresh green tea (8.4). Fig. 4.6 show the graphical representation of average sensory analysis score reconstituted green tea powder. Based on all these parameters spray drying was selected for further studies.

4.3.1 Optimisation of Spray Drying Process Parameters

The treatment combinations in the present experiment *viz*., maltodextrin concentrations of 2, 3 and 4%, inlet air drying temperatures of 160, 170 and 180°C and feed flow rate 420, 672 and 924 $ml.h^{-1}$ were used for instant green tea powder using spray dryer. The optimisation was carried out using Box-Behnken experimental design. The results of the effect of processing conditions on biochemical properties, functional properties, flow behaviour properties and reconstituted properties of spray dried instant green tea powder are presented in the following sections.

Table 4.5 Quality parameters of instant green tea powder obtained by different drying method

Sl. No.	Drying method	Time of drying	Colour of instant green tea powder			Colour of reconstituted green tea				Overall Acceptability
			*L**	a*	*b**	*L**	a*	*b**	Hue (°)	
1	Freeze drying	17	50.78	14.07	31.05	5.18	3.8	7.8	64.04	7.3
3	Vacuum drying	13	48.32	11.87	27.00	6.20	2.17	6.98	72.74	6.5
4	Spray drying	0.75	83.14	3.07	23.07	3.83	0.44	3.5	82.85	7.90
5	Fresh green tea					2.06	0.08	2.36	88.08	8.3

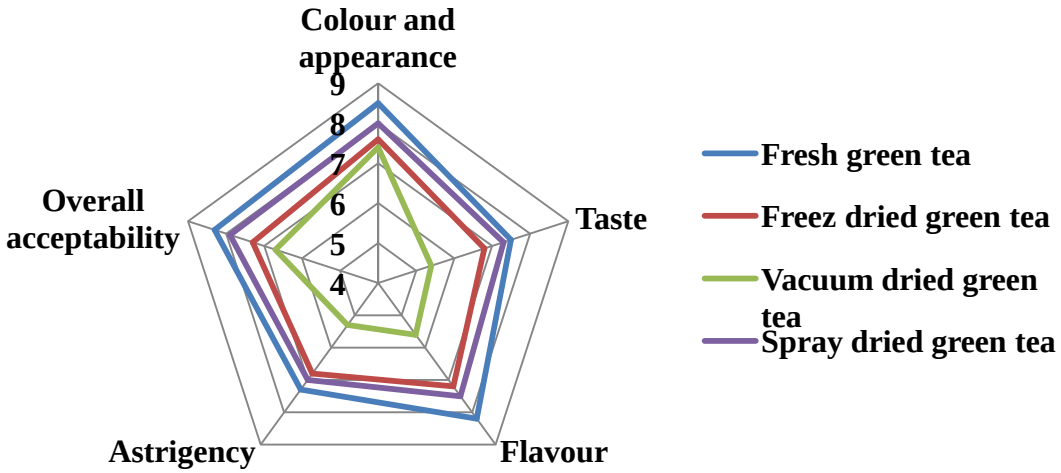


Fig. 4.6 Graphical representation of average sensory analysis score reconstituted green tea

4.3.1.1 Yield

The effect of various process parameters on yield is given in Appendix B1. The maximum yield was recorded at inlet air temperature of 170°C, 4% maltodextrin and feed flow rate of 420 $ml.h^{-1}$ followed by 35.529% at inlet air temperature of 170°C, 3% maltodextrin and feed flow rate of 672 $ml.h^{-1}$. The minimum yield was recorded at inlet air temperature of 160°C, 3% maltodextrin and 924 $ml.h^{-1}$ feed flow rate.

Yield increases significantly with increase in temperature from 160 to 170°C and further increase in temperature to 180°C result in decrease in yield (Fig. 4.7a-b). This reduction in yield might be due to the stickiness of powder when particles heated behind the glass transition temperature (Telang and Thorat, 2010). This tendency is good agreement with the findings of Nadeem *et al*., 2013 for instant soluble sage, Vuong *et al*., 2013 for decaffeinated green tea powder and Sarabandi *et al*., 2014 for spray dried grape powder. Effect of feed flow rate and its interaction with other drying parameters is shown in Fig.4.7a and 4.7c. Feed rate had negative effect on the yield of spray dried powder. This tendency is comparable with the study of Maskat *et al*., 2014 for spray dried hibiscus powder and Wang *et al*., 2015 for spray dried soy sauce powder. Effect of maltodextrin on yield of spray dried instant green tea powder is shown in Fig.4.7 (b-c). As the concentration of maltodextrin increases, product yield increases significantly, this is in good agreement with the findings of Nadeem *et al*. (2011) for spray dried mountain tea powders Nadeem *et al*., 2013 spray-dried sage powders and Fazaeli *et al*., 2014 for black mulberry juice powder.

Regression model fitted to experimental results of yield and it is given in Appendix B2. Following regression model was obtained to predict the moisture content of instant green tea powder.

$$\text{Yield} = 40.52+1.27(A)+1.32(B)-4.73\ (C)+0.92\ (A^*\ B)-0.99\ (A^*\ C)-3.27\ (B^*C)-3.71\ (A^2)-1.36\ (B^2)-9.82\ (C^2) \quad \ldots(4.6)$$

For moisture content the coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, A^2 (inlet air temperature^2) B

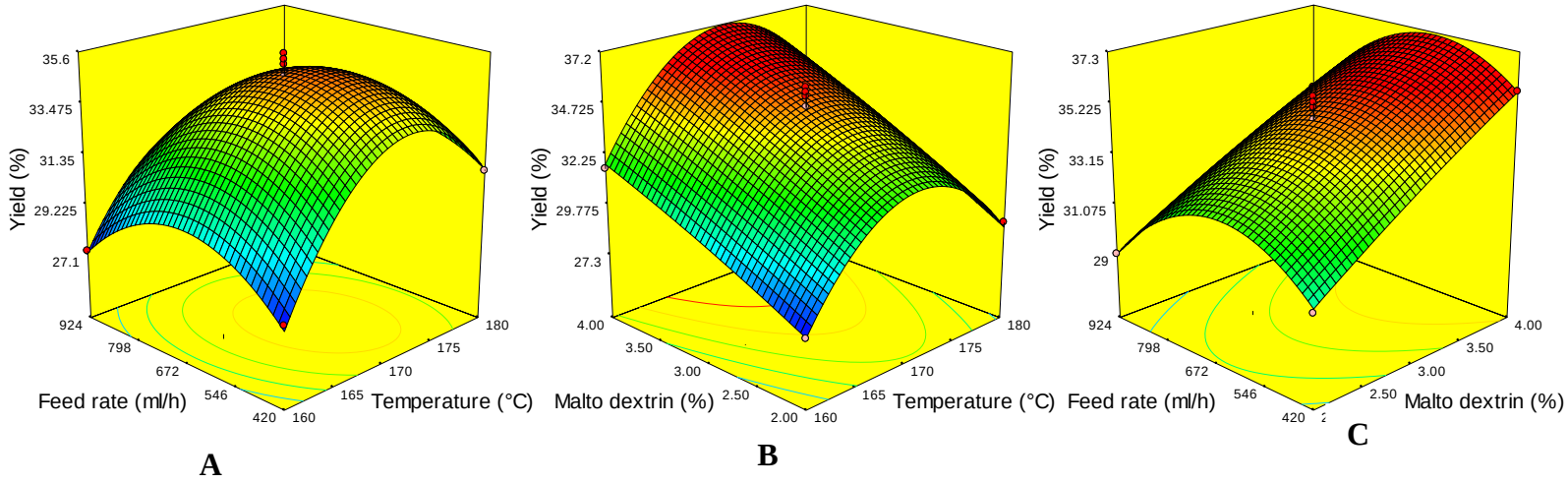


Fig. 4.7 Response surface plots showing effect of spray drying parameter on yield of instant green tea powder

(maltodextrin concentration) and were highly significant model terms with P- values of less than 0.0001. C (Feed flow rate), A (inlet air temperature) and C^2 (Feed flow rate^2) were significant model terms with P- values of less than 0.05.The p-value of the model was <0.0001, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9808, which showed that the regression model defined well the true behavior of the system.

4.3.1.2 Total flavonoid

The total flavonoid content of instant green tea ranged from 28.34 to 18.78 mg.g^{-1}. The total flavonoids content of instant green tea powder was found to be maximum at 170°C inlet temperature, 2% maltodextrin and feed rate of 924 ml.h^{-1}, whereas the minimum average total flavonoid content was recorded in treatment at 170°C inlet temperature, 4% maltodextrin and feed pump speed of 420 ml.h^{-1}. The total flavonoids of spray dried green tea powder from different treatments are presented in Appendix B3.

Fig. 4.8 depict the effect of inlet air temperature, maltodextrin concentration and feed flow rate on total flavonoid content. As the temperature increase from 160 to 180°C there was an increase in total flavonoid content of the samples but the variation is less (Fig. 4.8b-c). Similar trend was also observed by Couto *et al*., 2013 for spray dried Eugenia dysenterica extract powder. Vuong *et al*. 2013 also reported that, total flavonoids content in spray-dried caffeinated and decaffeinated tea is not affected by the inlet air temperature. Also, from the same figure it is clear that the feed flow rate show positive effect on total flavonoid content, and it might be due to the less exposure of particle due to the high feed rate. ANOVA was performed to evaluate the significance of the coefficients of the quadratic polynomial models and it is given in Appendix B4. The second order non-linear regression equation was fitted between dependent and independent variables using the experimental values. Regression model fitted to experimental results of total flavonoid content of instant green tea and

the following regression model was obtained to predict the total flavonoid content of instant green tea powder.

Total flavonoids=24.46+1.02(A)-2.07 (B)+2.74 (C)-0 (A* B)-0.17 (A* C)-0.12 (B*C)+0.42 (A^2)-0.84 (B^2)-0.88 (C^2) …(4.7)

For the total flavonoid content of instant green tea powder coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, B (Maltodextrin concentration) and C (Feed flow rate) was highly significant model terms with P- values of less than 0.0001. A (Inlet air temperature) and C^2 (Feed flow rate^2) were significant model terms with P- values of less than 0.05. The p-value of the model was less than 0.0001, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9740, which showed that the regression model defined well the true behaviour of the system.

4.3.1.3 Total polyphenols

The total polyphenols of instant green tea powder varied from 27 to 59 mg.g^{-1}. Variation of polyphenols at different spray drying condition is given in Appendix B3. The highest total polyphenols content was found at 170°C, 2% MD and 924 ml.h^{-1} feed rate. The lowest polyphenol content (27 mg.g^{-1}) was found in green tea powder with treatment inlet air temperature of 180˚C, 4% maltodextrin concentration in feed and 672 ml.h^{-1} feed flow rate. This result is more similar to the report of Nadeem *et al*. (2011). Fig. 4.9 depict the effect of inlet air temperature, maltodextrin concentration and feed flow rate on total polyphenol content. As the temperature increase from 160 to 170°C there was an increase in total polyphenol content of the samples and by increasing the inlet air temperature further to 180°C result in decreased phenolic content (Fig 4.9a-b). Similar trend was observed by Georgetti *et al*. (2008) with slight decrease in total polyphenol content in spray-dried soybean extract when the inlet air temperature is increased.

The effect of different feed flow rates on total polyphenolic contents is shown in Fig. 4.9b-c. In the present investigation, feed flow rate show positive effect on total polyphenol content. The highest retention of total polyphenols at higher feed flow rate might be due to less heating contact time (Loan *et al*., 2006). A reduced feed flow rate was related to increased contact time between samples with high temperature during spray drying. Similar observation was also reported by Muzaffar *et al*. (2016) for spray dried pomegranate juice powder. The total polyphenol content was decreased with the increase in maltodextrin concentration (Fig 4.9a-c), this decrease in total polyphenols is attributed to dilution of green tea extract at higher concentration of MD (Nadeem *et al*., 2011). Obtained value is comparable with the findings of Nadeem *et al*. (2011).

ANOVA was performed to evaluate the significance of the coefficients of the quadratic polynomial models and it is given in Appendix B5. The second order non-linear regression equation was fitted between dependent and independent variables using the experimental values. Regression model fitted to experimental results of total polyphenol content of instant green tea and the following regression model was obtained to predict the total polyphenol content of instant green tea powder.

Total Polyphenols= 44.00-5.50 (A)-7.63(B)+7.38 (C)+0.50 (A* B)-0.50 (A* C)-2.25 (B*C) -2.88(A^2) -2.13(B^2)-0.63(C^2) …(4.8)

For the total polyphenol content of instant green tea powder coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, A (Inlet air temperature) and A^2 (Inlet air temperature^2), were significant model terms with P- values of less than 0.05 and B (Maltodextrin concentration) and C (Feed flow rate) was highly significant model terms with P- values of less than 0.0001. The p-value of the model was 0.0002, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9659, which showed that the regression model defined well the true behaviour of the system.

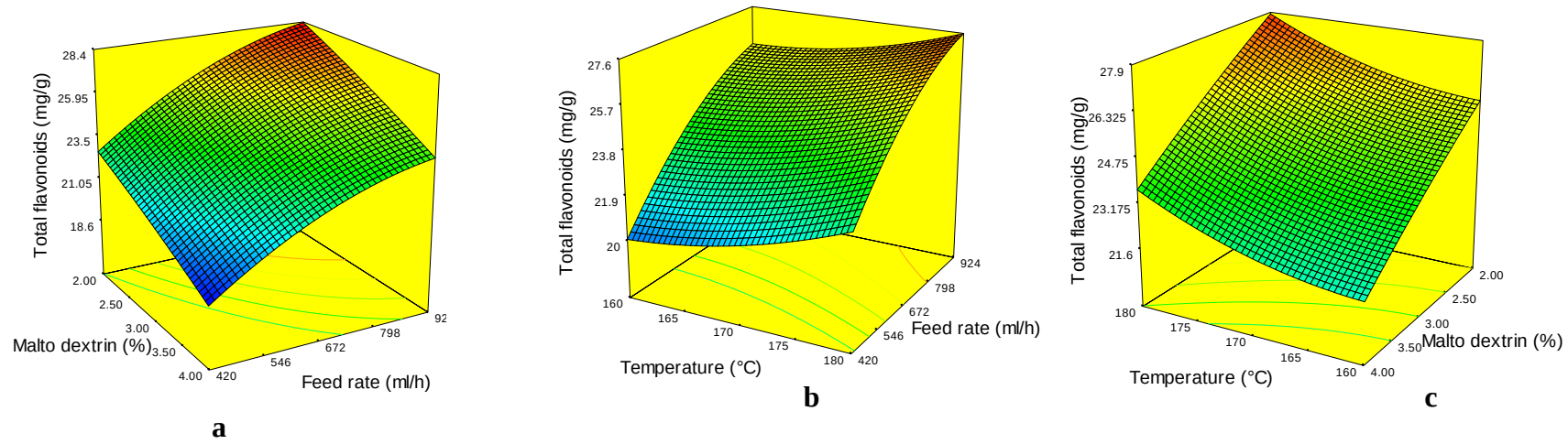


Fig. 4.8 Response surface plots showing effect of spray drying parameter on total flavonoids of instant green tea powder

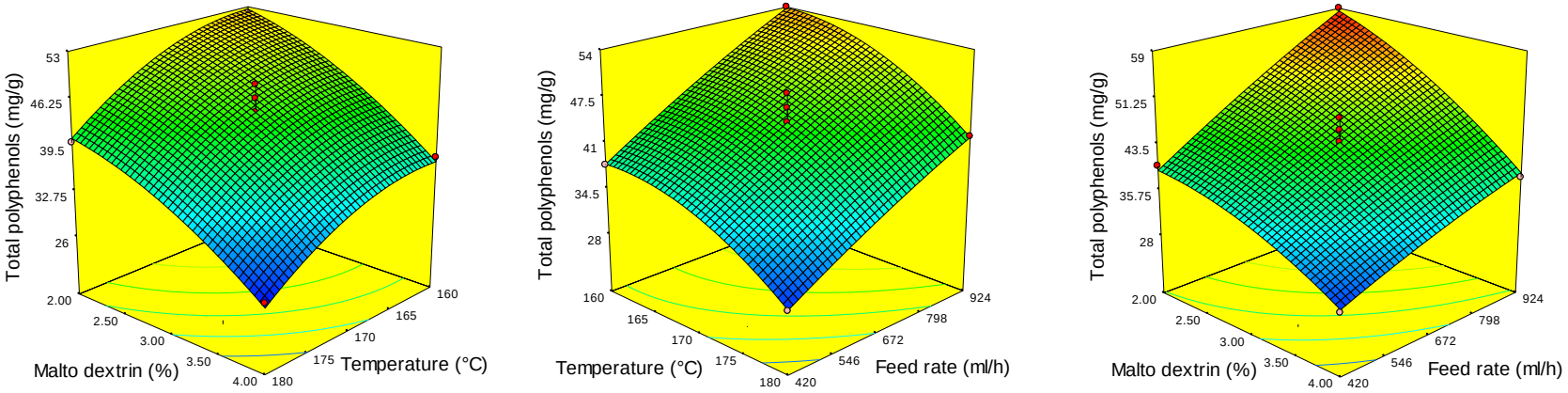


Fig. 4.9 Response surface plots showing effect of spray drying parameter on total polyphenols of instant green tea powder

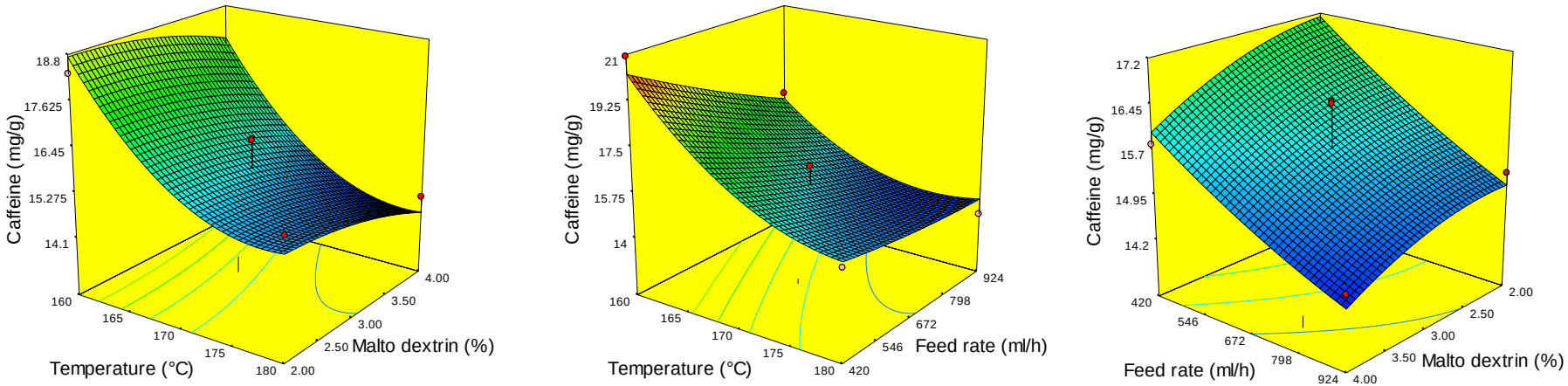
4.3.1.4 Caffeine

The caffeine content in instant green tea powder varied from 14.06-20.92 $mg.g^{-1}$. The highest value was is noticed for an inlet air temperature of 160°C, 3% MD and 420 $ml.h^{-1}$ feed rate and lowest value is for 180°C, 3% MD and 924 $ml.h^{-1}$ feed rate. Fig. 4.10 depict the effect of inlet air temperature, maltodextrin concentration and feed rate on caffeine content. It is evident from the Fig. 4.10 that all these parameters shows the negative effect on caffeine content, but the variation less significant ($p<0.05$). Similar trend was observed by Vuong *et al*., 2013 for spray drying of decaffeinated and high caffeine tea powders.

ANOVA was performed to evaluate the significance of the coefficients of the quadratic polynomial models and it is given in Appendix B6. Regression model fitted to experimental results of caffeine content of instant green tea and the following regression model was obtained to predict the caffeine content of instant green tea powder.

$$\text{Caffeine} = 15.68 - 1.80(A) - 0.47(B) - 0.99(C) - 0.05(A*B) + 0.62(A*C) + 0.10(B*C) + 1.06(A^2) - 0.22(B^2) + 0.12(C^2) \quad \ldots(4.9)$$

It is evident from the equation (4.9) that the caffeine content was in negative correlation with Inlet air temperature, maltodextrin concentration and feed flow rate. For the caffeine content of instant green tea powder coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, A (Inlet air temperature), C (Feed flow rate) and A^2 (Inlet air temperature^2), were significant model terms with P- values of less than 0.05. The p-value of the model was 0.0044, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9193, which showed that the regression model defined well the true behaviour of the system.



a **b** **c**

Fig. 4.10 Response surface plots showing effect of spray drying parameter on caffeine of instant green tea powder

4.3.1.5 Colour

L* value

The *L** values of spray dried green tea powder ranged from 81.71 to 88.15 at different spray drying parameters and is given in Appendix B7. It was revealed that, the highest *L** value was recorded at 160°C, 4% MD and 672 $ml.h^{-1}$ feed rate whereas, lower value was at 180°C, 2% MD and 672 $ml.h^{-1}$ feed rate.

From the Fig. 4.11 it was clear that as the temperature increases from 160 to 180°C there was a decrease in *L** value and it might be due the effect of higher temperature on the colour also there is a chance of non-enzymatic browning reactions on maltodextrin at high temperature (Quek *et al*., 2007; Rodriguez-Hernandez *et al*., 2005). Similar tendency was observed by Nadeem *et al*. (2011) for spray dried tea. It was observed from Fig 4.11a-b that the feed flow rate and maltodextrin concentration had positive effect on *L** value. Increase in L* value at higher feed rate was due to lower exposure of particle in drying medium (Maskat *et al*., 2014). Similar tendency was reported by Kha *et al*., 2010 for gac fruit aril powder and Caliskan and Dirim, 2013 sumac extract.

Regression model fitted to experimental results of *L** value and it is given in Appendix B8. Following regression model was obtained to predict the *L** value of instant green tea powder.

$$L^* = 83.91-1.69(A)+1.33(B)+0.70(C)-0.14(A^*B)+0.22(A^*C)-0.01(B^*C)+0.55(A^2)+0.34(B^2)+0.79(C^2) \quad \ldots(4.10)$$

The magnitude of p-value indicates that all linear terms (A, B, C), quadratic terms C^2 were significant 5% ($p < 0.005$). But interaction term A*B, B*C and A*C had not significant at 5 per cent level ($p > 005$). The p-value of the model was 0.0019, which indicated that the model fitness was significant. The model F-value of 11.65 implies that model is significant. The best fit model was expressed by the coefficient of determination R^2, which was 0.9374, indicating that 93.74 per cent of the variability of the response could be explained by the model.

a* value

The a^* values of green tea powder with various spray drying treatment is presented in Appendix B7. Temperature showed positive effect, MD and feed flow rate showed negative effect on redness value. From Fig. 4.12 it was clear that, with increase in temperature there was increase in redness value. This increase in redness might be either due to non enzymatic browning or by burning at higher temperature (Quek *et al*., 2007). As the MD concentration increases there was decrease in redness owing to inherent colour of MD.

Regression model fitted to experimental results of a* value and it is given in Appendix B9. Following regression model was obtained to predict the a* value of instant green tea powder .

$$a^* = 2.39 + 0.13(A) - 0.21(B) - 0.16(C) + 0.025(A^*B) + 0(A^*C) - 0.07(B^*C) + 0.0048(A^2) + 0.088(B^2) + 0.054(C^2) \quad \ldots(4.11)$$

The magnitude of p-value indicates that all linear terms were highly significant at 1% ($p < 0.0001$), interaction term B*C (Maltodextrin concentration* Feed flow rate) and all quadratic term had significant effect at 5% level ($p < 0.05$). The p-value of the model was <0.0010. The best fit model was expressed by the coefficient of determination R^2, which was 0.9866, indicating 98.66%of the variability of the response could be explained by the model.

b* value

The b^* values of spray dried green tea powder at different spray drying is presented in Appendix B7. It was inferred that, the highest *b** value of 22.65 was found at 170°C, 2% MD and 420 $ml.h^{-1}$ feed rate whereas, lowest of 15.25 at 170°C, 4% MD and 924 $ml.h^{-1}$ feed rate.

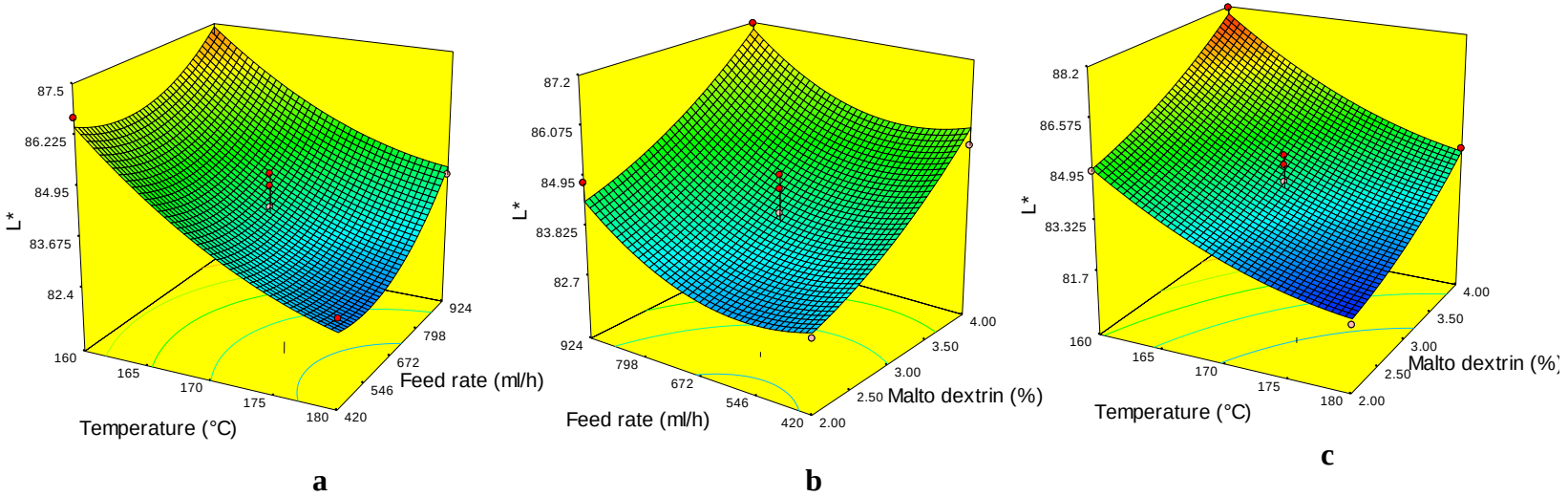


Fig. 4.11 Response surface plots showing effect of spray drying parameter on *L of instant green tea powder**

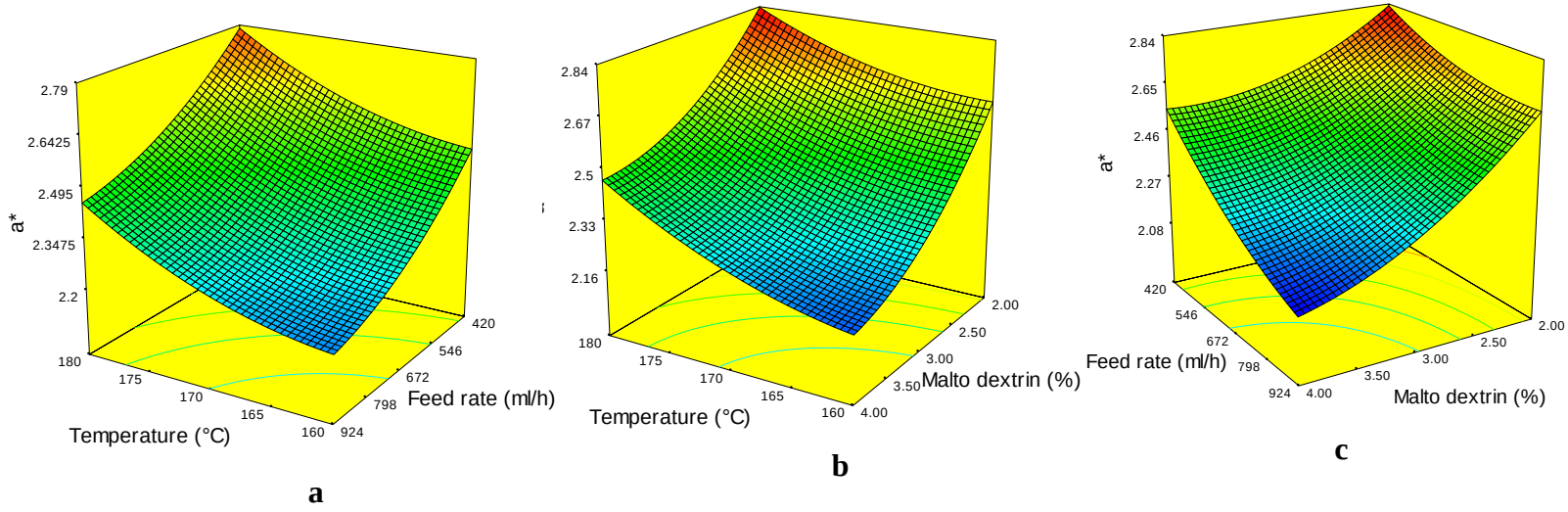


Fig. 4.12 Response surface plots showing effect of spray drying parameter on *a of instant green tea powder**

Effect of inlet air temperature, MD and feed flow rate on *b** value is depicted in Fig 4.13. As the temperature increases from 160 to 180°C there was increase in yellowness, as feed flow rate and maltodextrin increases there was reduction in *b** value. The lower value of yellowness at high concentration of MD was due to dilution of extract (Daza *et al*., 2016). Comparable tendency was observed by Maskat *et al*. (2014) in hibiscus extract and Caliskan and Dirim (2013) in sumac extract. Higher value of b* was observed at higher temperature and lower feed rate due to burning of particles. Similar trend was reported by Quek *et al*. (2007) in spray dried watermelon powder. The overall colour value indicated that powder was slight darker at higher drying temperatures and lower feed rate.

Regression model fitted to experimental results of *b** value and it is given in Appendix B10. Following regression model was obtained to predict the *b** value of instant green tea powder.

$$b^* = 19.23+1.69\,(A)-1.59(B)-1.48\,(C)+0.44(A^*B)-0.43(A^*C)+0.06\,(B^*C)+0.00\,(A^2)+0.18\,(B^2)-0.56\,(C^2) \quad \ldots(4.12)$$

The magnitude of p-value indicates that all linear terms A (inlet air temperature) B (Maltodextrin concentration) and C (Feed flow rate) were significant at 5% ($p < 0.005$). Remaining interaction terms and quadratic term are not significant at 5 per cent level ($p > 005$). The p-value of the model was 0.0006. The best fit model was expressed by the coefficient of determination R^2, which was 0.9556, indicating that 95.56 per cent of the variability of the response could be explained by the model.

Hue angle

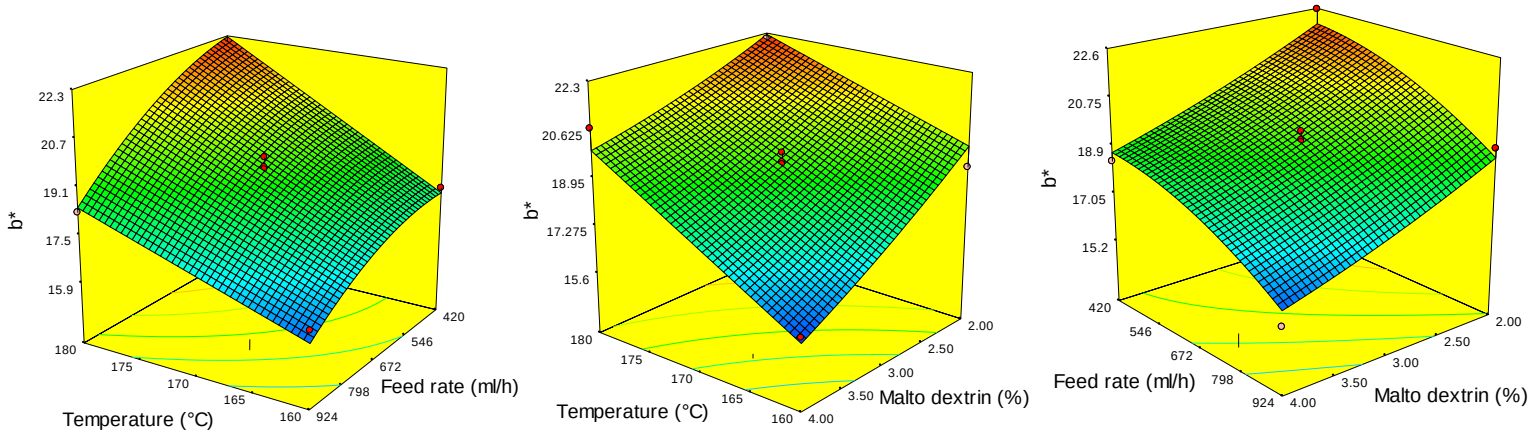
The hue angle of instant green tea powder from different treatments is presented in Appendix B7. According to hue angle values given in 3.6.2.1, colour of powder was near to the yellow. Hue angle was significantly affected by temperature and feed flow rate while, MD was insignificant to hue angle. Obtained values were inline with the finding of Caparino *et al*. (2012) for maltodextrin added spray dried mango powder.

Regression model fitted to experimental results of hue angle and it is given in Appendix B11. Following regression model was obtained to predict the hue angle of instant green tea powder.

$$Hue=82.94+0.28(A)-0.0\ (B)-0.088(C)+0.10\ (A*B)-0.16\ (A*C)+0.20\ (B*C)-0.15\ (A^2)-0.22(B^2)-0.38\ (C^2) \quad \ldots(4.13)$$

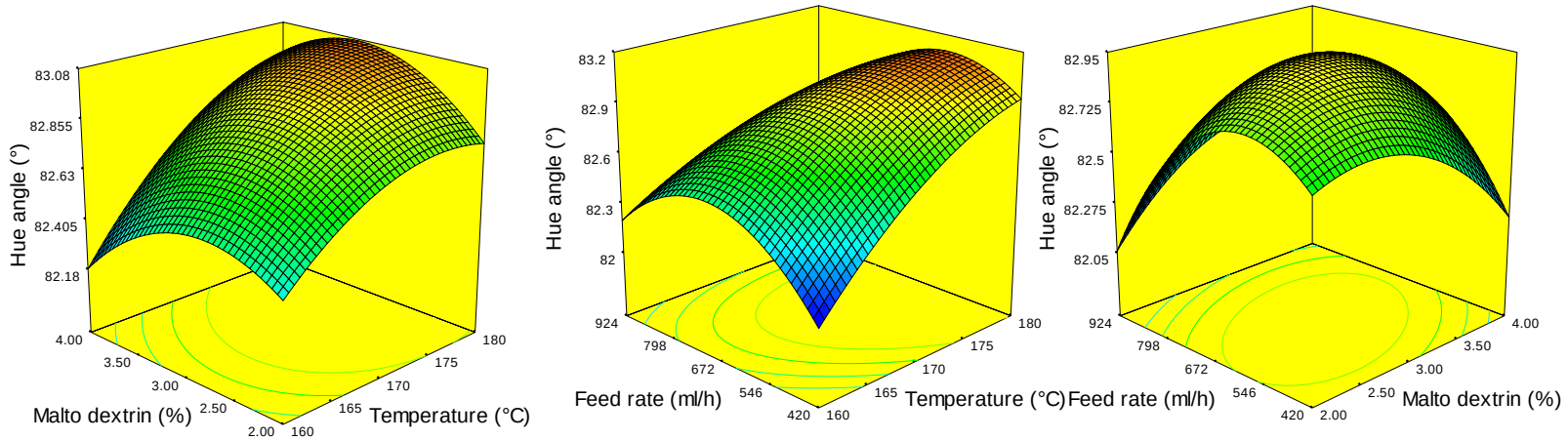
Following observations were made from the above equation, coefficients of C is negative and for A is positive. Therefore, increase in feed flow rate may reduce the hue angle, whereas increase in inlet air temperature increase the hue angle of instant green tea powder (Fig. 4.14). Kha *et al*., 2010 also observed the higher hue angles as a result of increasing the inlet drying temperature for spray dried Gac powder

For hue angle the coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, A (inlet air temperature) and C^2 (Feed flow rate^2) were significant model terms with P- values of less than 0.05. By analysis of variance, the R^2 value of this model was determined to be 0.788.



a **b** **c**

Fig. 4.13 Response surface plots showing effect of spray drying parameter on *b of instant green tea powder**



a **b** **c**

Fig. 4.14 Response surface plots showing effect of spray drying parameter on hue angle of instant green tea powder

4.3.1.6 Moisture content

The moisture content represents the water composition in a food system. It plays a key role in all food materials and is one of the most commonly measured properties of food materials. The information on moisture content is necessary to see the suitability for storage. The moisture content of instant green tea powder ranges from 3.24 to 8.32% (d.b.). Maximum was recorded at 160°C inlet air temperature, 2% MD and 672 $ml.h^{-1}$ feed rate whereas, minimum for 180°C inlet air temperature, 4% MD and 672 $ml.h^{-1}$ feed flow rate and it is given in Appendix B12.

Moisture content decreased significantly with increase in temperature (Fig.4.15a-b), this might be due to increase in water evaporation rate at higher temperature that result in fast drying of materials. At higher inlet temperature, the rate of heat transfer to the particle is greater, providing greater driving force for moisture evaporation. This is in good agreement with the findings of Fazaeli *et al*., 2012 for spray dried black mulberry juice powder, Loh *et al*. (2005) for spray dried pandan extract and Moreira *et al*. (2009) for spray dried acerola pomace extract.

It is clear from the Fig. 4.15, as the maltodextrin concentration increase there was reduction in moisture content of powder. Increased level of maltodextrin increased the level of feed solids and reduced the level of total moisture for evaporation (Kha *et al.,* 2010). This result was more similar to the report of Mishra *et al*, (2014) for amla juice powder, Abadio *et al.* (2004) for pineapple juice powder and Patil *et al*., 2014 for guava powder.

It is evident from the figure 4.15b-c that, as the feed flow rate increases there will be increase in powder moisture content. Under high feed rate the amount of water need to be evaporated is high, so contact time with drying air is not enough to cause evaporation. Reason for this is also explained by Maskat *et al*. (2014) for hibiscus powder and Wang *et al*., 2015 for spray dried soy sauce powders.

Regression model fitted to experimental results of moisture content and it is given in Appendix B13. Following regression model was obtained to predict the moisture content of instant green tea powder.

Moisture content= 3.37-1.45(A)-0.90+(B) 0.96 (C)+0.08(A* B)+0.01(A* C)+0.03 (B*C) +1.45(A^2) +0.88(B^2) +1.13(C^2) …(4.14)

For moisture content, the coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, all the linear term, A (inlet air temperature) B (Maltodextrin concentration), C (Feed flow rate), and all the quadratic term A^2 (inlet air temperature^2) B^2 (Maltodextrin concentration^2), C^2 (Feed flow rate^2) were significant model terms with P- values of less than and 0.0001. The p-value of the model was <0.0001, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9953, which showed that the regression model defined well the true behavior of the system.

4.3.1.7 Water activity

Water activity measures the availability of free water in a food system that is responsible for any biochemical reactions. High water activity indicates more free water available for biochemical reactions and hence the shorter shelf life. Water activity ranges from 0.132 to 0.355 and maximum water activity was recorded at inlet air temperature of 160°C, 3% maltodextrin and 924 ml.h^{-1} feed flow rate. Minimum water activity of 0.132 was observed in sample which dried at inlet air temperature of 180°C, 4% maltodextrin and 672 ml.h^{-1} feed rate. Reported water activity in agreement with the findings of Nadeem *et al*. (2013) for instant soluble sage and Caliskan and Dirim (2013) for sumac extract.

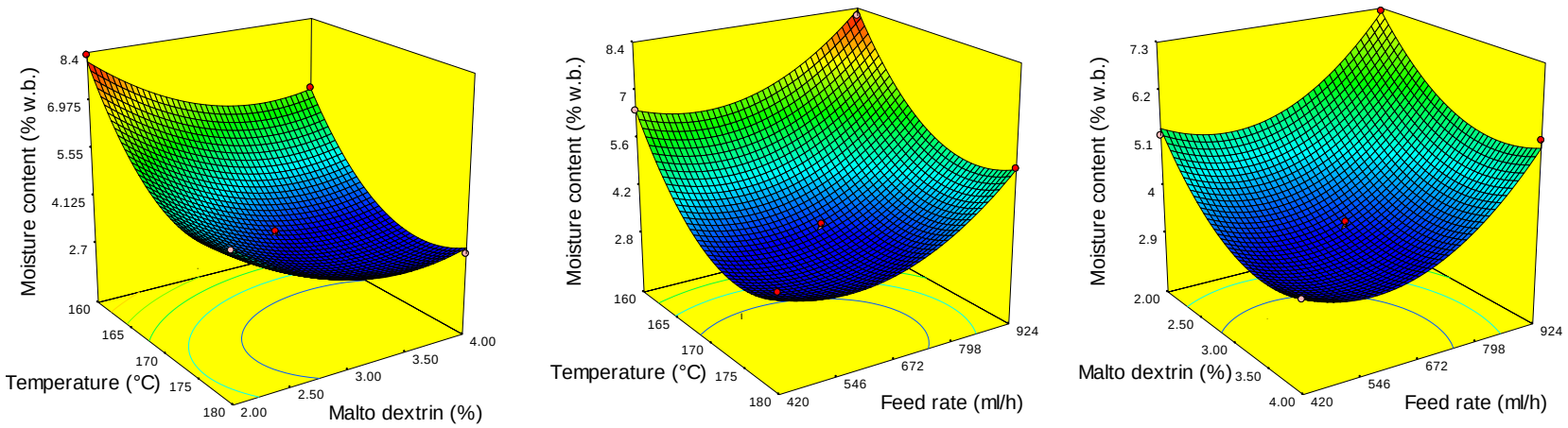
Water activity decreased significantly with increase in temperature and inlet air temperature is the sole factor that affects the moisture content and water activity**.** The effects of independent variables on water activity are found to be similar with moisture content. So as the moisture varies water activity also varies in similar manner. Effect of inlet air temperature, maltodextrin concentration and feed flow rate

on water activity of spray dried instant green tea powder is shown in Fig. 4.16. From the figure it can be observed that, lower water activity is achieved by increase in maltodextrin, in inlet air temperature and by decreasing the feed flow rate. Similar trend was observed by Fang and Bhandari (2011) in bayberry juice powder, Coralia *et al.* (2011) in guava powder, Fazaeli *et al.* (2012) in black mulberry juice powder.

Regression model fitted to experimental results of water activity and it is given in Appendix B14. Following regression model was obtained to predict the water activity of instant green tea powder.

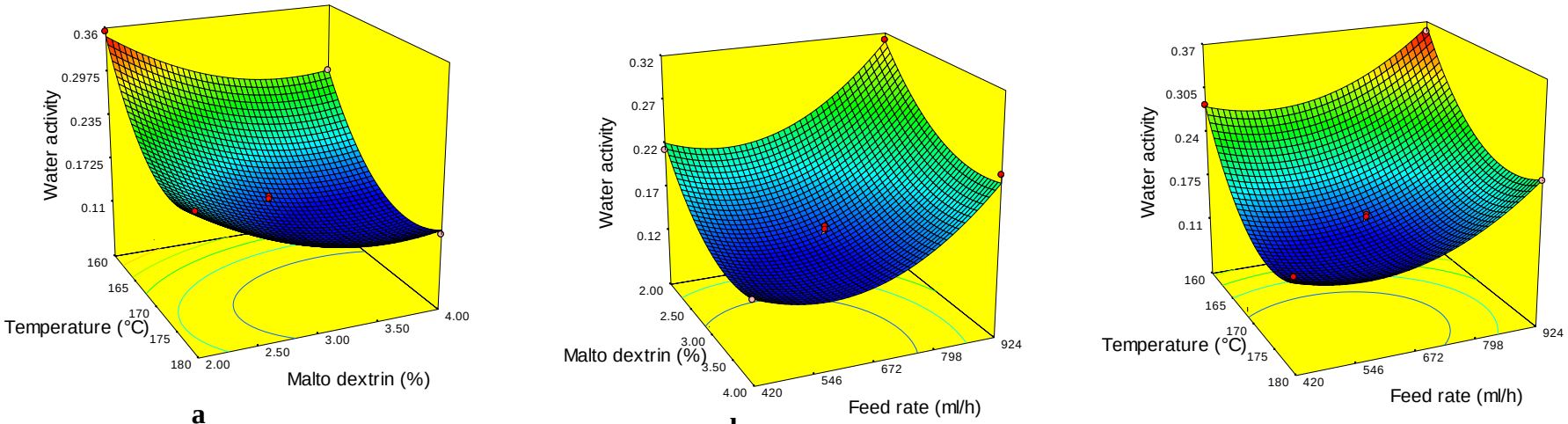
Water activity= 0.1436-0.0643(A)-0.0407 (B)+0.0416(C)+0.0016(A* B)+0.0005 (A* C)-0.0025(B*C) +0.0647(A^2)+0.0335(B^2)+0.0485(C^2) …(4.14)

Following observations can be made from the above equation, coefficients of A and B are negative but for C is positive. Therefore, increase in inlet air temperature and maltodextrin may reduce the water activity, whereas increase in feed flow rate increase the water activity of spray dried powder. For water activity the coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, all the linear term, A (inlet air temperature) B (Maltodextrin concentration), C (Feed flow rate), and all the quadratic term A^2 (inlet air temperature^2) B^2 (Maltodextrin concentration^2), C^2 (Feed flow rate^2) were significant model terms with P- values of less than and 0.0001. The p-value of the model was <0.0001, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9955, which showed that the regression model defined well the true behavior of the system.



a **b** **c**

Fig. 4.15 Response surface plots showing effect of spray drying parameter on moisture content of instant green tea powder



a **b** **c**

Fig. 4.16 Response surface plots showing effect of spray drying parameter on water activity of instant green tea powder

4.3.1.8 Bulk density

The loose and tapped bulk density of instant green tea powder under different spray drying inlet air temperatures, maltodextrin concentration along with varied feed flow rate was observed. It was revealed from the results that the of instant green tea powder had the highest loose bulk density value of 0.288 $g.cc^{-1}$ in inlet air temperature of 160°C, 3% maltodextrin and 924 $ml.h^{-1}$ feed flow and the lowest loose bulk density of 0.243 $g.cc^{-1}$ in inlet air temperature of 180°C, 2% maltodextrin and 672 $ml.h^{-1}$ feed flow. The highest tapped bulk density of 0.430 $g.cc^{-1}$ was recorded at inlet air temperature of 160°C, 4% maltodextrin and 672 $ml.h^{-1}$ feed flow and the lowest tapped bulk density of 0.324 $g.cc^{-1}$ at inlet air temperature of 180°C, maltodextrin level of 2 and 3%, and feed flow rate of 420 $ml.h^{-1}$ and 672 $ml.h^{-1}$. Fig. 4.17 and 4.18 depicts the three dimensional plot on effect of treatments on loose and tapped bulk densities of spray dried instant green tea powder.

Increasing spray drying inlet air temperature and decreasing feed flow rate generally produces an increase in particle size that lead to reduction in bulk density. At higher temperatures evaporation rates is faster that, lead products more dry and porous therefore lower density of the powder can be observed (Fazaeli *et al*., 2012). Increase in inlet air temperature often results in a rapid formation of dried layer at the droplet surface and particle size was due to skinning over or case-hardening of the droplets at the higher temperatures. This leads to the formation of vapour-impermeable films on the drop surface, followed by the formation of vapor bubbles and, consequently, droplet expansion (Reddy, 2013). Obtained values are also parallel to the findings of Susantikarn and Donlao (2016), Wang *et al*., 2015, Jinapong *et al*., 2008 and Nadeem *et al*., 2011 for spray dried powders. As concentration of maltodextrin increases both loose bulk density and tapped bulk density increases. The reason for this was that as occluded air content decreased, it led to decrease in particle volume, thus increasing particle density and bulk density (Reddy, 2013). Fernandes *et al*., 2013 reported that higher concentration, powder accommodates itself more easily

in the spaces among the particles, resulting in higher density. The obtained values are in line with the findings of Fazaeli *et al*., 2012 and Caliskan and Dirim, 2013 for spray dried mulberry juice powder and sumac extract, respectively. ANOVA for the loose bulk density is given in Appendix B16. In this case of loose bulk density, all linear terms, A (inlet air temperature) B (Maltodextrin concentration), C (Feed flow rate) are highly significant at p value less than 0.0001 and quadratic factors A^2 (Inlet air temperature^2) and C^2 (Feed flow rate^2) were significant at p value less than 0.05. The p-value of the model was <0.0001, which indicated that the model fitness was significant. The statistical analysis indicated that the proposed models fitted the experimental data with R^2 value of 0.9908, whereas the predictive model had an R^2 value of 0.9695 was in reasonable agreement with the "Adj R-Squared" of 0.9789. The optimized predictive model (in terms of coded factors) for loose bulk density of spray dried instant green tea powder is presented in equation 4.15.

Loose bulk density= 0.258-0.01125(A)+0.008625(B)+0.009125(C)+0(A* B)
0.0005(A* C)-0.00125 (B*C)+0.004625(A^2)+ 0.000375(B^2)
+0.003875(C^2) …(4.15)

ANOVA for the tapped bulk density is given in Appendix C15. In this case of tapped bulk density, terms A (inlet air temperature) is highly significant at p value less than 0.0001 and other factors B (Maltodextrin concentration), C (Feed flow rate) and A^2 (inlet air temperature^2) were significant at p value less than 0.05. The p-value of the model was <0.0001, which indicated that the model fitness was significant. The statistical analysis indicated that the proposed models fitted the experimental data with R^2 value of 0.9944, whereas the predictive model had an R^2 value of 0.9533 was in reasonable agreement with the "Adj R-Squared" of 0.9871. The optimized predictive model (in terms of coded factors) for tapped bulk density of spray dried instant green tea powder is presented in equation 4.16.

Tapped bulk density =0.3438-0.04475 (A)+0.006(B)+0.009(C)-0.0015(A* B)-0.001(A* C) 0.001(B*C) +0.0291(A^2) +0.0026(B^2) + 0.0011(C^2) …(4.16)

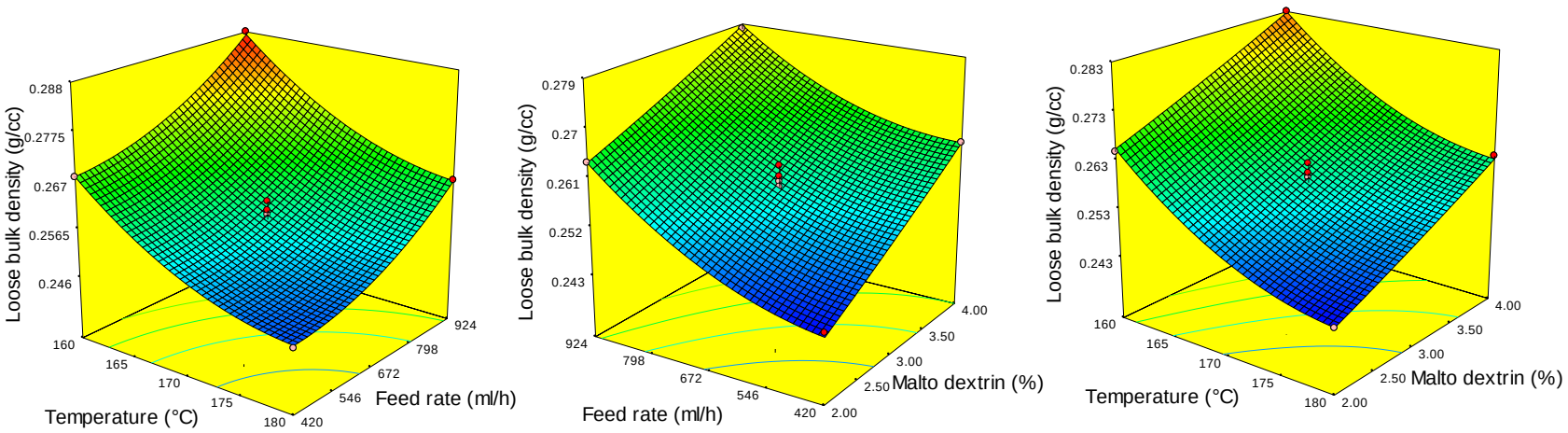


Fig. 4.17 Response surface plots showing effect of spray drying parameter on loose bulk density of instant green tea powder

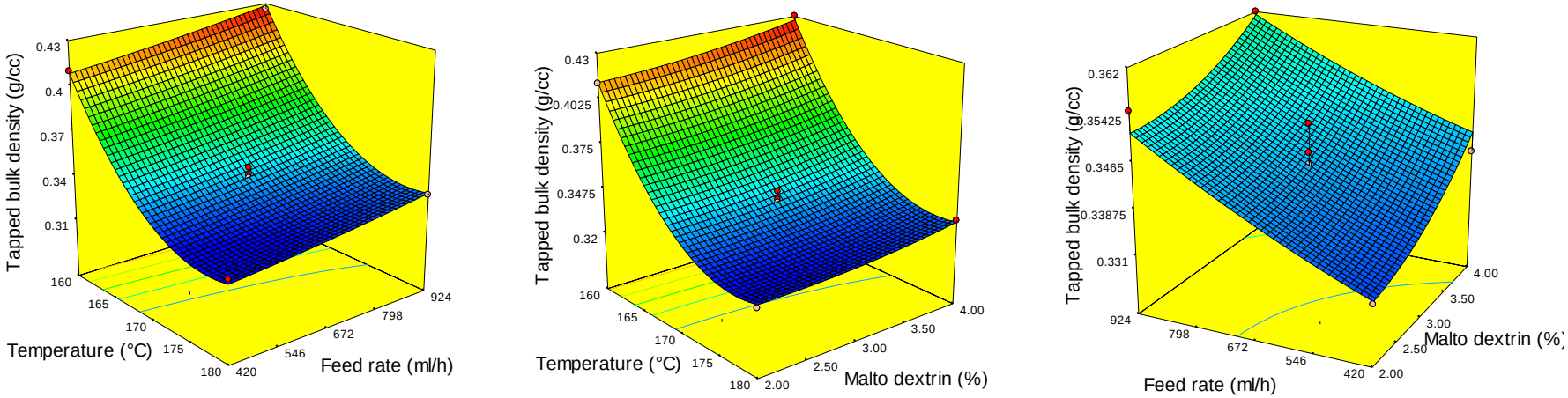


Fig. 4.18 Response surface plots showing effect of spray drying parameter on tapped bulk density of instant green tea powder

4.3.1.9 Wettability

Wettability is the ability of the powder particles to over-come the surface tension between themselves and water. Wettability value of spray dried instant green tea ranges from 42 to 224 s and maximum wettability was recorded at inlet air temperature of 170°C, 4% MD and 420 $ml.h^{-1}$ feed rate. Minimum wettability of 42 s was observed in sample which dried at inlet air temperature of 160°C, 3% maltodextrin and 924 $ml.h^{-1}$ feed rate (Appendix B17).

As represented in Fig. 4.19. it is clearly observed that, as increase in temperature, maltodextrin concentration and decrease in feed flow rate result in increase in wettability. The longest particle instantisation times occurred at greater inlet air temperature and carrier agent concentration. This fact can be explained due to the lower moisture content of the powders obtained under these circumstances. Caking, which usually occurs in powders with higher moisture, can contribute to wettability since the liquid penetrates into the pores more easily (Fernandes *et al*., 2013). Similar trend was observed by Caliskan and Dirim, 2013 for sumac extracts. Patil *et al*. (2014) for guava powder and Ferrari *et al*., 2013 for blackberry powder produced with higher maltodextrin and inlet temperature showed the lowest wettability. Patil *et al.* (2014) for wettability of guava powder and concluded that as the maltodextrin concentration increased from 7 to 12%, the wettability also increased.

Regression model fitted to experimental results of wettability and it is given in Appendix B18. Following regression model was obtained to predict the wettability of instant green tea powder.

Wettability= 161.2+25.5 (A)+21.875(B)-65.875 (C)-10.25(A* B)+0.25(A* C)-3.5(B*C)+8.65(A2)+10.9 (B2)-38.6 (C2) …(4.18)

Following observations can be made from the above equation, coefficients of A and B are positive but for C is negative. Therefore, increase in inlet air temperature and maltodextrin may enhance the wettability value *i.e*., increases time of

instantanization, whereas increase in feed flow rate reduces the wettability value of spray dried powder *i.e.*, decreases time of instantanization. For wettability the coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, C (Feed flow rate) is highly significant with P- values of less than and 0.0001 and A (inlet air temperature) B (Maltodextrin concentration), C^2 (Feed flow rate^2) were significant model terms with P- values of less than and 0.05. The p-value of the model was <0.0001, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9846, which showed that the regression model defined well the true behavior of the system. The Pred R^2 of 0.8861 is in reasonable agreement with the Adj R^2 of 0.9648.

4.3.1.10 Solubility

Solubility is the final step of powder dissolution and is considered as the key determinant of the overall reconstitution quality (Caliskan and Dirim, 2013). Solubility value of spray dried instant green tea was varied from 80.40 to 89.80%. maximum solubility was recorded at inlet air temperature of 170°C, 4% MD and 420 ml.h^{-1} feed rate and minimum solubility of 80.4% s was observed in sample which dried at inlet air temperature of 180°C, 2% maltodextrin and 672 ml.h^{-1} feed flow rate (Appendix B17). Obtained values are similar to the findings of Fazaeli *et al*., 2012, Nadeem *et al*., 2013 and Mishra *et al*., 2014 for black mulberry juice powder, instant soluble sage and amla juice powder.

As represented in Fig. 4.20. it is clearly observed that, as increase in temperature solubility increases initially and further increase in temperature causes decrease in solubility. Reason for this is explained by Quek *et al*. (2007), at lower inlet temperatures, spray dried powder have higher tendency of agglomeration which helps increasing the reconstitution of the powders, this might be the reason for achieving higher solubility at 170°C than the 180°C. As the concentration of maltodextrin increases solubility increases, this trend is in good agreement with the findings of Caliskan and Dirim, 2013 and Nadeem *et al*., 2013 for sumac powder and

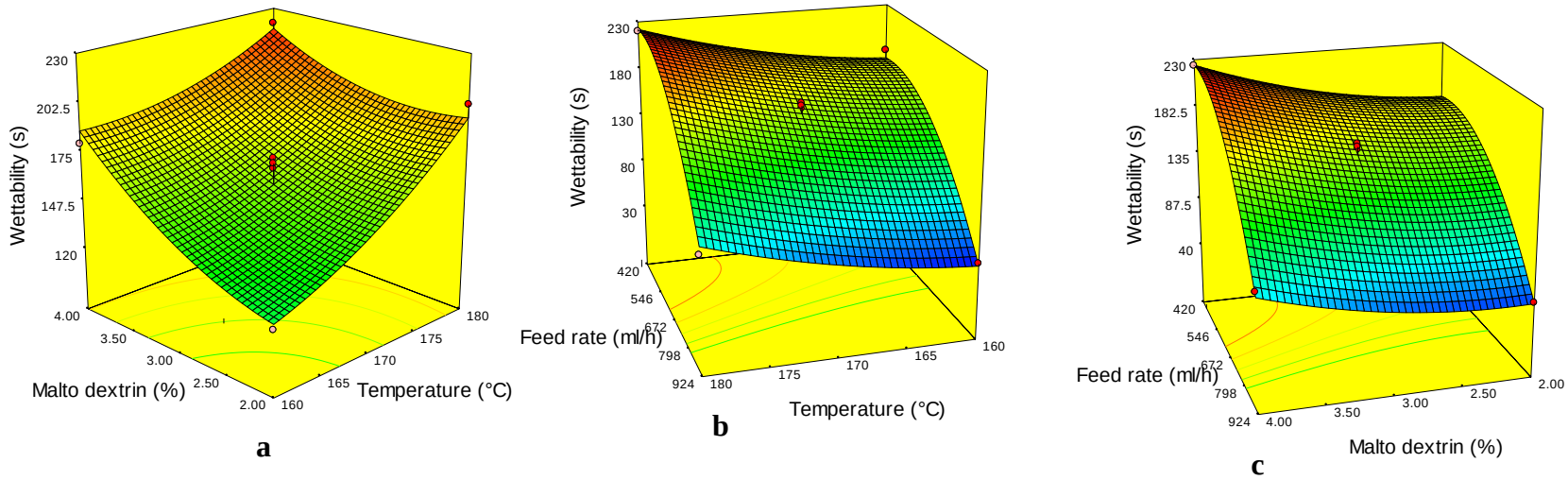


Fig. 4.19 Response surface plots showing effect of spray drying parameter on wettability of instant green tea powder

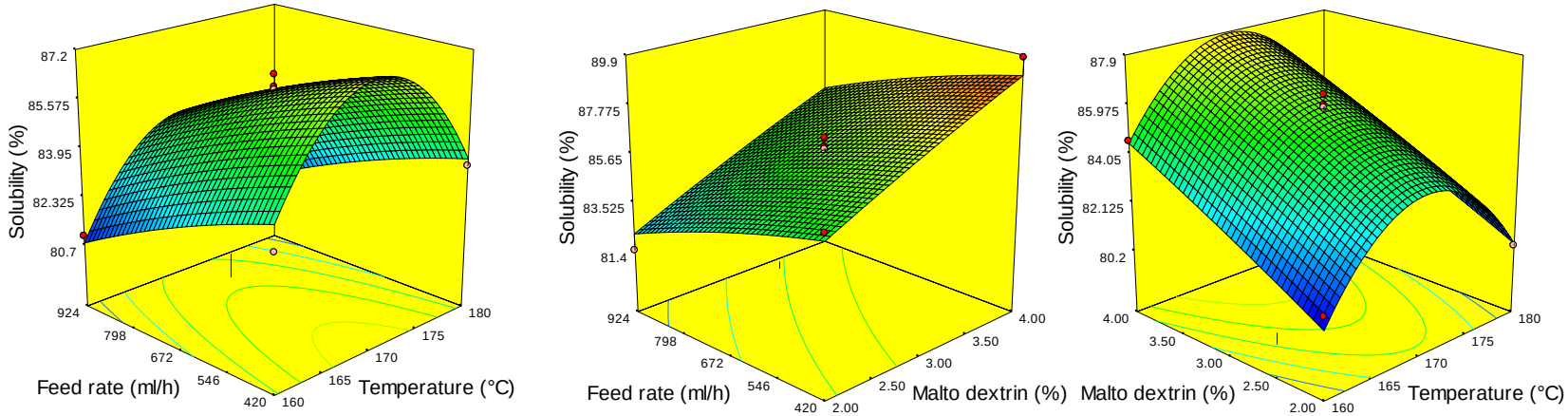


Fig. 4.20 Response surface plots showing effect of spray drying parameter on solubility of instant green tea powder

instant soluble sage powder. Cause for this may be attributed to the fact that maltodextrin has superior water solubility that result in increase in powder solubility with increase in maltodextrin. (Fazaeli *et al*., 2012). Chauca *et al*., 2005 also reported treatment with maltodextrin induces high degree of solubility. From Fig. 4.20(a-b) it can be observed that, as the feed flow rate increases solubility decreases, this might be due to increased in particle size at higher feed rate. Parallel tendency was observed by Banat *et al*., 2002 for spray dried tomato paste.

Regression model fitted to experimental results of solubility is given in Appendix B 19. The following regression model was obtained to predict the solubility of instant green tea powder.

Solubility= 85.94+0(A)+2.0125+(B)-1.4375(C)-0.1(A* B)+0.1 (A* C)+0.025+

(B*C)-3.4325 (A^2) -0.1075(B^2)-0.2075(C^2) …(4.19)

The following observations can be made from the above equation, coefficients of A and B are positive but for C is negative. Therefore, increase in inlet air temperature and maltodextrin may enhance the solubility, whereas increase in feed flow rate reduces the solubility of spray dried powder. For solubility the coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, B (Maltodextrin concentration) and A^2 (inlet air temperature^2) are highly significant with P- values of less than and 0.0001 and, C (Feed flow rate) is significant with P- values of less than and 0.05. The p-value of the model was <0.0001, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9757, which showed that the regression model defined well the true behavior of the system.

4.3.1.11 Carr index and Hausner ratio

Flow behaviour of instant green tea powder was explained through Carr's index and Hausner ratio, which was calculated by using loose and tapped bulk density values. Carr's index is a measure of powder bridge strength and stability and Hausner ratio is a measure of the interparticulate friction (Rakhi *et al.,* 2008).

It is reveled from the result (Appendix B20) that the Carr index and Hausner ratio value ranged between 1.273 to 1.555 and 21.429 to 35.680, respectively. Instant green tea powder had the highest Carr index and Hausner ratio value at inlet air temperature of 160°C, 2% maltodextrin and 672 $ml.h^{-1}$ feed flow. The lowest Carr index and Hausner ratio of 1.2727 and 21.4286 was obtained in inlet air temperature of 180°C, 3% maltodextrin and 924 $ml.h^{-1}$ feed rate. Fig. 4.21 and 4.22 depicts the three dimensional plot on effect of treatments on Carr index and Hausner ratio of spray dried instant green tea powder, respectively.

In terms of handling properties, the instant green tea powder obtained in the present study were considered as "fair" powders by their Hausner ratio (HR). This was in accordance with Carr index (CI) which indicated that their flowability was intermediate (Table 3.9). This might be due to smaller particle sizes and the presence of the fine particles that cause reduced flow characteristics as evidenced by their higher CI and HR values.

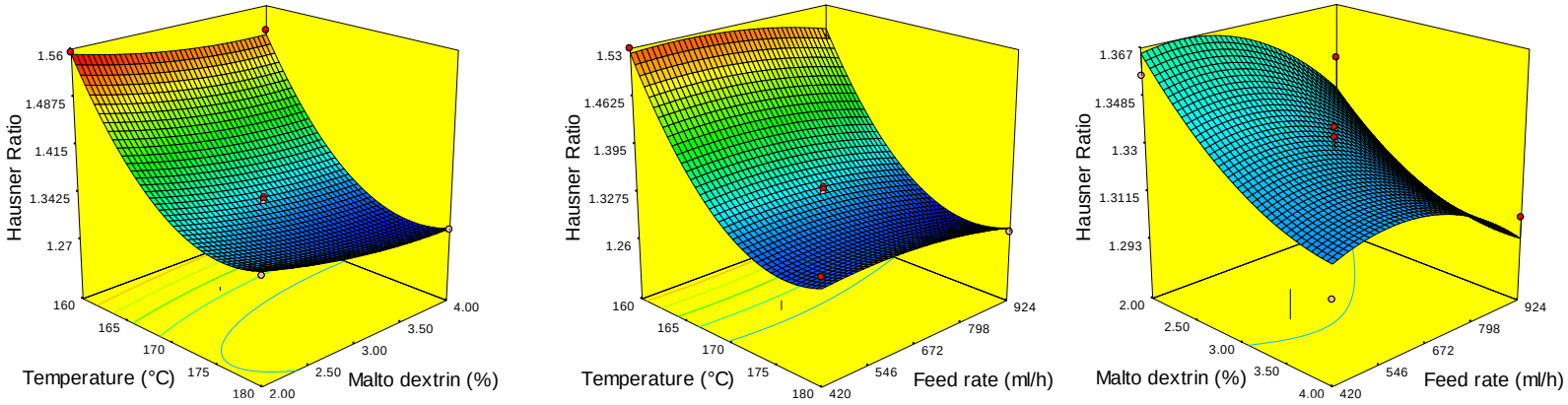
Similar values are reported by Jinapong *et al*., 2008 for atomized soy milk. Teunou *et al*. (1999). The presence of water in a powder could significantly affect its flowability, sticking and caking properties. In general, the greater the water content of a powder, there will be cohesion and will be more difficult to flow. Therefore higher moisture content samples had less flow properties.

Regression model fitted to experimental results of Carr index (Appendix B21) and Hausner ratio (Appendix B22). Following regression models was obtained to predict the Carr index and Hausner ratio of instant green tea powder.

Carr index= 24.73-5.51 (A)-1.22(B)-0.68 (C)-0.30 (A* B)-0.10 (A* C)+0.19 (B*C)+4.07 (A^2)+0.50 (B^2)-0.67 (C^2) …(4.20)

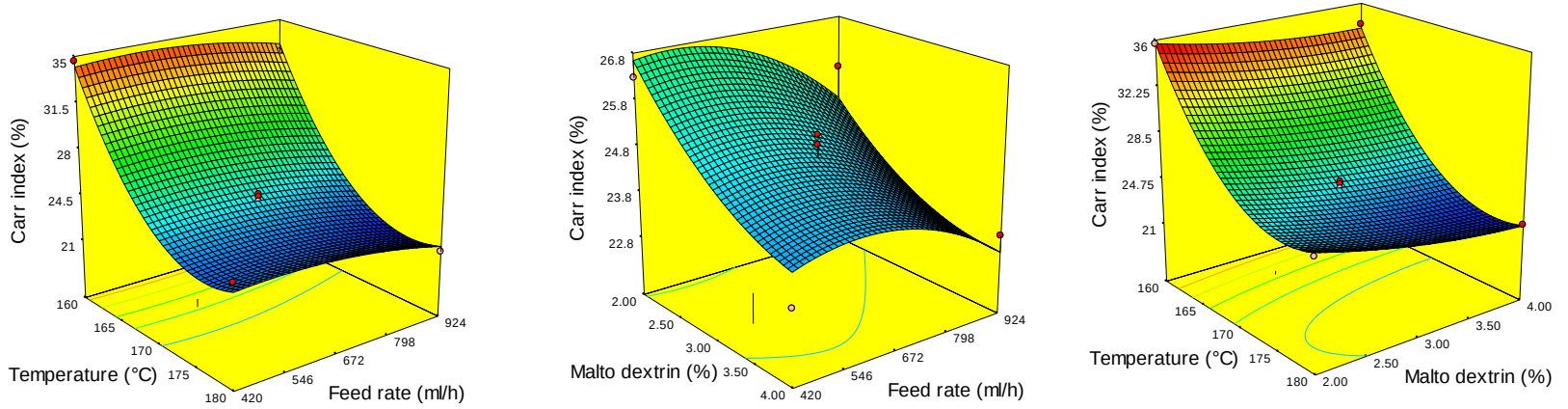
Hausnar ratio= 1.33 -0.11(A)-0.02(B)-0.01 (C)+0.00(A* B)+0.00(A* C)+0.00(B*C)+0.08(A^2)+0.01(B^2) -0.01(C^2) …(4.21)

Following observations can be made from the above equation, coefficients of A, B and C is negative. Therefore, increase in inlet air temperature, maltodextrin and feed flow rate will reduce the values of Carr index and Hausner ratio, *i.e.*, it increases the flow properties. For Carr index and Hausner ratio the coefficient estimates and the corresponding P-values suggest that, among the test variables used in the study, A (inlet air temperature) and A^2 (inlet air temperature^2) are highly significant with P-values of less than and 0.0001, B (Maltodextrin concentration) and C (Feed flow rate) is significant with P- values of less than and 0.05. The p-value of the model in both case was <0.0001, which indicated that the model fitness was significant. By analysis of variance, the R^2 value of this model was determined to be 0.9901 and 0.9905 for Carr index and Hausner ratio respectively, which showed that the regression model defined well the true behavior of the system.



a **b** **c**

Fig. 4.21 Response surface plots showing effect of spray drying parameter on hausner ratio of instant green tea powder



a **b** **c**

Fig. 4.22 Response surface plots showing effect of spray drying parameter on carr index of instant green tea powder

Optimization of the three process variables namely, spray drying inlet air temperature (160, 170 and 180ºC), maltodextrin concentration (2, 3 and 4%) and feed flow rate (420, 672 and 924 $ml.h^{-1}$) was performed using the Box-Behnken design in Design Expert Software 7.7.0. In the present investigation, the independent variables were kept within the range and dependent variables were chosen as maximum and minimum. Appendix B21 shows response optimization constraints for spray drying of green tea. The experimental results had the optimum process conditions of, inlet temperature 174ºC, maltodextrin concentration 2.7% and feed flow rate 671 $ml.h^{-1}$.

4.3.2 Verification of the Predicted Variables

The optimum response values were tested using the recommended optimum conditions of the variables and was also used to validate the experimental and predicted values of the responses. The predicted, actual values of the responses and the percentage variation at the optimized condition of spray drying are presented in Table 4.6. The predicted values of optimized treatment are comparable with the actual value. Powder obtained at optimized condition is shown in plate 4.1

4.3.1 Sensory Evaluation of Reconstituted Instant Green Tea

Spray dried instant green tea was reconstituted with hot and cold water with different ratio of powder to water. Then eight reconstituted green tea samples along with fresh green tea was given to sensory evaluation. The scale factors viz. excellent (EX), good (GD), medium (MD), fair (FR) and not satisfactory (NS) assigned to the quality factors viz**.,** color and appearence, taste, flavour, astringency and overall acceptability for the all the samples are given in chapter III. Fuzzy membership function (FMF) and normalized fuzzy membership function (NFMF) were then calculated and presented in the Appendix B22. Then the judgment membership functions (Appendix B23) are compared with the weightage value of quality attributes and the minimum value was selected to assign the quality ranking. Five samples of different concentration reconstituted with hot water are shown in plate 4.2.

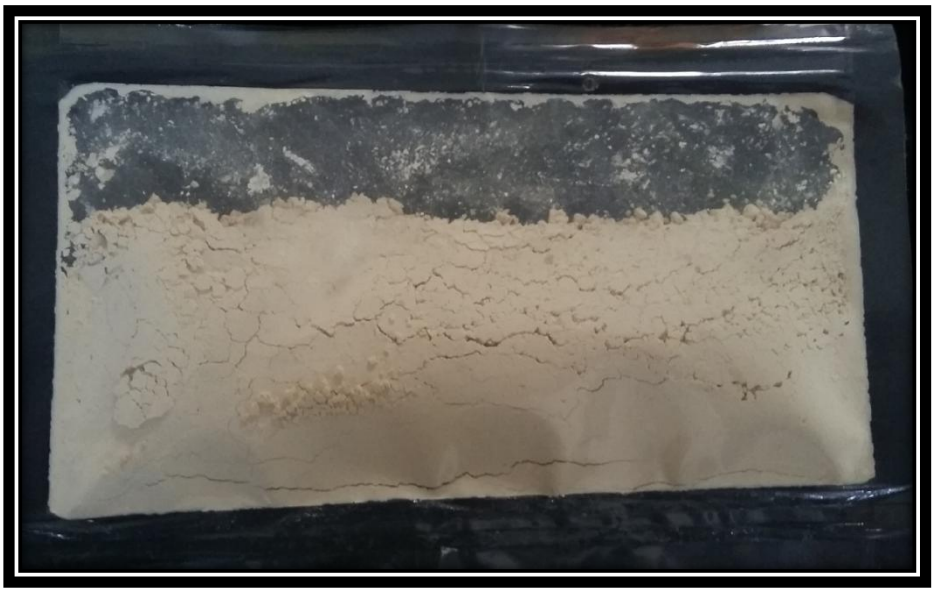


Plate 4.1 Spray dried instant green tea powder at optimised condition

Table 4.6 Predicted and actual values of responses of the optimized condition

Sl. No.	Responses	Predicted value	Actual value	Variation (%)
1	Yield (%)	33.416	32.590	-2.472
2	Loose bulk density ($g.cc^{-1}$)	0.251	0.259	3.268
3	Tapped bulk density ($g.cc^{-1}$)	0.329	0.338	2.861
4	Carr Index (%)	23.759	23.345	-1.742
5	Hausner Ratio	1.309	1.305	-0.301
6	Moisture content (% w.b.)	3.154	3.080	-2.334
7	Solubility (%)	84.573	87.970	4.016
8	Water activity	0.149	0.150	0.526
9	Wettability (s)	168.014	160	-4.760
10	L*	82.860	83.670	0.977
11	a*	2.541	2.581	1.574
12	b*	20.512	20.892	1.851
13	Total flavonoids ($mg.g^{-1}$)	25.735	26.670	3.632
14	Caffeine ($mg.g^{-1}$)	15.287	15.170	-0.765
15	Total polyphenols ($mg.g^{-1}$)	43.830	43.180	-1.482
16	Hue value	82.979	81.900	-1.300

T_0 **HT_1** **HT_2** **HT_3** **HT_4**

Plate 4.2 Reconstituted instant green tea

T_0- Fresh green tea

HT_1- 0.75 g of instant green tea powder in 100 ml hot water

HT_2- 1.00 g of instant green tea powder in 100 ml hot water

HT_3- 1.25 g of instant green tea powder in 100 ml hot water

HT_4- 1.50 g of instant green tea powder in 100 ml hot water

Based on the quality ranking T_0 samples score highest (0.210) value followed by HT_3 (0.209) and CT_4 (0.200) samples. It is observed from the table that the sample T_0 (control) gets the first rank based on the score obtained for colour and appearance. Sample HT_3 (1.25%) scored 0.209 based on its flavour and HT_4 get third rank based on colour and appearance.

Referring to Table 4.7 it is observed that the reconstituted HT_3 sample scored 0.204, 0.180, 0.209, 0.192 and 0.200 for colour, taste, flavour, astringency and overall acceptability respectively, whereas the corresponding weightage average of these quality attributes are 0.21, 0.18, 0.21, 0.2 and 0.20. Therefore HT_3 sample satisfied the quality criteria set for taste and overall acceptability but not the colour, astringency and flavour. Then the next highest score is 0.207 for HT_4 sample that due to colour and appearance and scores are 0.207, 0.171, 0.175, 0.176 and 0.179 for colour, taste, flavour, astringency and overall acceptability respectively, whereas the corresponding weightage average of these quality attributes are 0.21, 0.18, 0.21, 0.2 and 0.20. So it can be clearly observed that by increasing the quantity of powder it is improving the colour and appearance but all other scores were decreased. This might be due to higher concentration of tea powder increasing the astringency that might be the reason for less score for astringency and taste. In case of cold water reconstituted tea CT_4 got the highest score due it its colour and appearance but all the values are less than the hot water reconstituted tea. So it is clear from the Table 4.7, HT_3 sample can be acceptable by the consumer i.e., concentration of 1.25 g of instant green tea powder in 100 ml of hot water will give the better sensory quality. Then for further studies 1.25 gm of powder is dissolved in 100 ml of water.

Table 4.7 Quality ranking of reconstituted instant flavoured instant green tea sample

Sensory attributes	Weightage	**Sample T_0**	**Sample HT_1**	**Sample HT_2**	**Sample HT_3**	**Sample HT_4**	**Sample CT_1**	**Sample CT_2**	**Sample CT_3**	**Sample CT_4**
Colour and Appearance	0.21	0.210	0.195	0.195	0.208	0.206	0.180	0.187	0.193	0.203
Taste	0.18	0.180	0.180	0.180	0.180	0.171	0.174	0.173	0.174	0.202
Flavour	0.21	0.189	0.184	0.187	0.202	0.174	0.174	0.168	0.176	0.180
Astringency	0.2	0.184	0.173	0.180	0.192	0.176	0.160	0.168	0.180	0.173
Overall acceptability	0.2	0.205	0.187	0.189	0.199	0.178	0.182	0.181	0.181	0.181
		0.210	0.195	0.195	0.208	0.206	0.182	0.187	0.193	0.203
Quality ranking		**I**/Colour and Appearance	**V**/Colour and Appearance	**V**/Colour and Appearance	**II**/Colour and Appearance	**III**/Colour and Appearance	**VIII**/Overall acceptability	**VII**/Colour and Appearance	**VI**/Colour and Appearance	**IV**/Colour and Appearance

Experiment III

4.4 OPTIMISATION OF FLAVOURING COMPOUNDS IN INSTANT FLAVOURED GREEN TEA POWDER

The methodology followed for optimisation of flavouring compounds is given in section 3.5. As per the described procedure green tea extract was prepared at optimised condition with three flavours of different treatment and spray dried at optimised process parameters. Biochemical analysis of these flavoured instant green tea powder was analysed in terms of total polyphenols, total flavonoids and caffeine. The result of biochemical properties and sensory evaluation of instant flavoured green tea is presented below.

4.4.1 Ginger Flavoured Instant Green Tea

Ginger flavoured instant green tea powder was prepared with ginger juice and ginger powder and which is given in table 3.6. Effect of ginger on total polyphenols, total flavonoids and caffeine in instant green tea is presented below.

4.4.1.1 Biochemical Properties of Ginger Flavoured Instant Green Tea

Results of the total flavonoids and total polyphenols in ginger flavoured green tea obtained by adding ginger juice and powder are depicted in Appendix C1. Total flavonoids content of gingered green tea varied from 27.07 to 29.07 $mg.g^{-1}$ and total polyphenols content varied from 44.02 to 45.98 $mg.g^{-1}$ among the eight treatments. The highest total flavonoid and total polyphenols was recorded in G8 treatment whereas, lowest value was recorded in G1 treatment. It was inferred from the data that, as the concentration of ginger juice and ginger powder increases, slight increase in total flavonoid and total polyphenols were observed compared to control sample.

It is evident from Fig.4.23, maximum flavonoid variation in ginger powder and ginger juice was 8.99% and 5.36%, respectively in ginger flavoured instant green tea. Maximum polyphenol variation in ginger powder and ginger juice was 6.58% and 3.06%, respectively in ginger flavoured instant green tea (Fig.4.24). Green tea

flavoured with ginger juice was observed less flavonoids and polyphenols than the green tea flavoured with ginger powder. Since the ginger juice was prepared with cold extraction there might be incomplete extraction of polyphenols and flavonoids, which resulted less concentration of these phytochemicals, where as ginger powder was extracted with hot water which increases these values. As per the report of Ghasemzadeh *et al*. (2010) and Mukherjee *et al*. (2014) ginger have 3.66 $mg.g^{-1}$ total flavonoid and 3.5 to 11.2 $mg.g^{-1}$ of total polyphenols. This increase in concentration of total flavonoid and total polyphenols in flavoured green tea was due to addition of ginger.

It is clear from ANOVA (Appendix C2) addition of ginger juice did not have significant effect on total flavonoid and total polyphenols but addition of ginger powder resulted in slight increase in total polyphenols. Obtained trend was in agreement with the conclusions of Makanjuola *et al*., 2015 for black tea-ginger extract and it was also reported that the optimum value of ginger addition resulted in good quality of extract.

Caffeine

Caffeine content in instant green tea was 15.17 $mg.g^{-1}$ but after addition of ginger juice and ginger powder slight decrease was observed. The instant green tea flavoured with ginger juice has 15.02 $mg.g^{-1}$ whereas, the sample with ginger powder has 14.64 $mg.g^{-1}$ (Fig.4.25). Statistical result (Appendix C4) indicated that, variation of caffeine content among the treatment was insignificant ($p>0.05$).

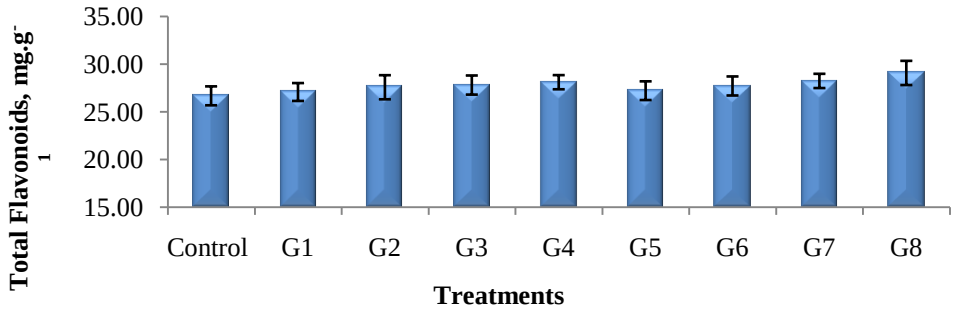


Fig 4.23 Effect of ginger addition on total flavonoid content of instant green tea

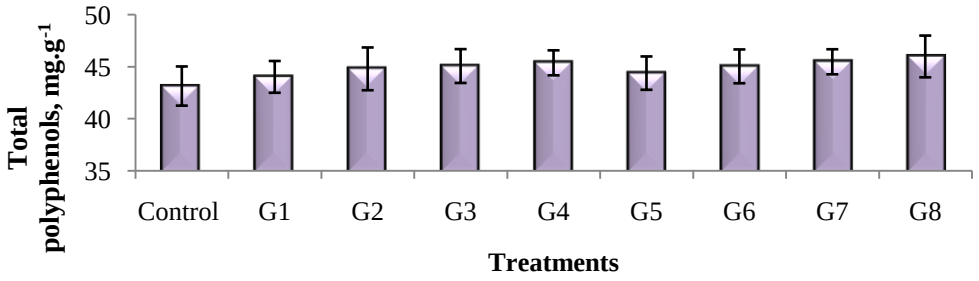


Fig 4.24 Effect of ginger addition on total polyphenol content of instant green tea

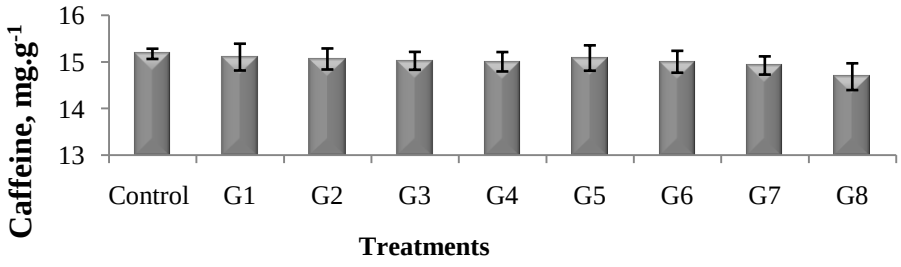


Fig. 4.25 Effect of ginger addition on caffeine content of instant green tea

G1- 2% ginger juice; G2- 4% ginger juice; G3- 6% ginger juice; G4- 4% ginger juice; G5- 1:10 Ginger powder:Dried tea leaves; G6-2:10 Ginger powder : Dried tea leaves; G7- 3:10 Ginger powder : Dried tea leaves; G8- 4:10 Ginger powder : Dried tea leaves

4.4.1.2 Sensory evaluation

Due to the intense pungent flavour three samples are screened out before sensory evaluation. The five samples selected for sensory evaluation were green tea with 2% ginger juice (G1), 4% ginger juice (G2), 6% ginger juice (G3), 2:10 ginger powder to green tea leaf ratio (G6) and 3:10 ginger powder to green tea leaf ratio (G7). The selected treatments of ginger flavoured green tea was reconstituted with optimum quantity of water and fresh green tea which is considered as control was kept for sensory evaluation. Sensory analysis was done using nine point hedonic scale and the sensory attributes were ranked using comprehensive fuzzy logic model.

Scale factors, quality factors, FMF, NFMF and JMF were then calculated and presented in the Appendix C5 and C6. The fuzzy comprehensive model ranking of ginger flavoured green tea was G2>G0=G6>G1>G3>G7. It was observed from the Table 4.8, that the sample G2 (4% ginger juice) ranked first with highest score in flavour attribute. Sample G0 (Control) and G7 (4% ginger juice) ranked second with highest score in colour and appearance. Referring to Table 4.8 it was observed that the reconstituted G2 sample scored 0.191, 0.180, 0.210, 0.197 and 0.199 for colour, taste, flavour, astringency and overall acceptability respectively. The G2 sample satisfied the quality criteria of flavour and taste attributes but not for colour, astringency and overall acceptability. This low score for colour might be due to addition of ginger juice that resulted in deviation from control sample. Conversely, G2 scored higher ranking than control in overall acceptability. Another noteworthy observation was that, ginger flavour could be correlated negatively at higher concentration which might be the reason for lowest ranking in G3 and G7.

Table 4.8 Quality ranking of ginger flavoured instant green tea sample

	Weightage	**Sample G0**	**Sample G1**	**Sample G2**	**Sample G3**	**Sample G7**	**Sample G8**
Colour and Appearance	0.21	0.20	0.195	0.191	0.195	0.20	0.189
Taste	0.18	0.179	0.18	0.18	0.18	0.180	0.180
Flavour	0.21	0.178	0.193	0.21	0.186	0.191	0.191
Astringency	0.20	0.182	0.197	0.197	0.184	0.186	0.193
Overall acceptability	0.20	0.174	0.199	0.199	0.182	0.189	0.189
		0.2/Colour and appearance	0.199/Colour and appearance	0.21/Flavour	0.195/Colour and appearance	0.2/Colour and appearance	0.193/Astringency
Quality ranking		II	III	I	IV	II	V

4.4.2 Cardamom Flavoured Instant Green Tea

The crushed cardamom with pod was added to hot green tea extract and spray dried. The various treatment adopted is given in Table 3.7. The obtained cardamom flavoured instant green tea was tested for total polyphenols, total flavonoids and caffeine. Sensory evaluation was done for selected five treatments and fresh green tea was taken as control. Biochemical properties of cardamom flavoured green tea is given in Appendix C7.

4.4.2.1 Biochemical Properties of Cardamom Flavoured Instant Green Tea

The flavonoid content of cardamom flavoured instant green tea ranged from 26.82 to 27.02 $mg.g^{-1}$ and total polyphenols from 44.04 to 46.82 $mg.g^{-1}$. From Fig. 4.26 and 4.27 it was observed that as the quantity of cardamom and time of extraction increases there was slight increase in total flavonoid content and total polyphenols. This variation was insignificant when compared to instant green tea ($p>0.05$). According to the report of Padmakumari Amma *et al*., 2010 and Deepa *et al*., 2013 cardamom seed contain 0.14 $mg.g^{-1}$ and 1.14 $mg.g^{-1}$ of total flavonoids, and 6.4 $mg.g^{-1}$ and 1.24 $mg.g^{-1}$ of total polyphenols. This might be the reason for negligible increase in total flavonoid and total polyphenol content in cardamom flavoured instant green tea. The effect of cardamom on total flavonoid and total polyphenol of cardamom flavoured green were statistically analysed and presented in Appendix C8 and C9.

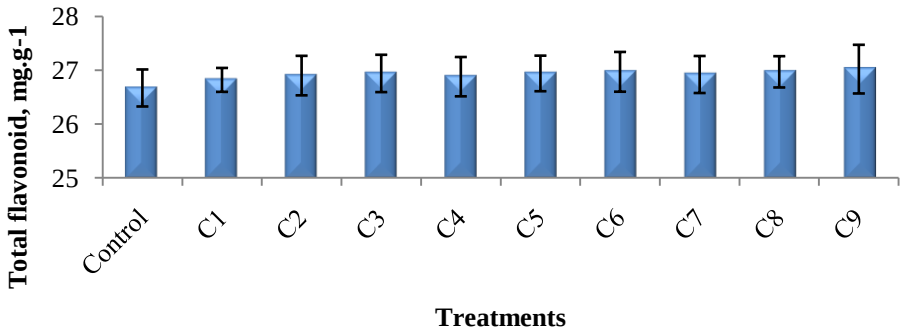


Fig. 4.26 Effect of cardamom addition on total flavonoid content of instant green tea

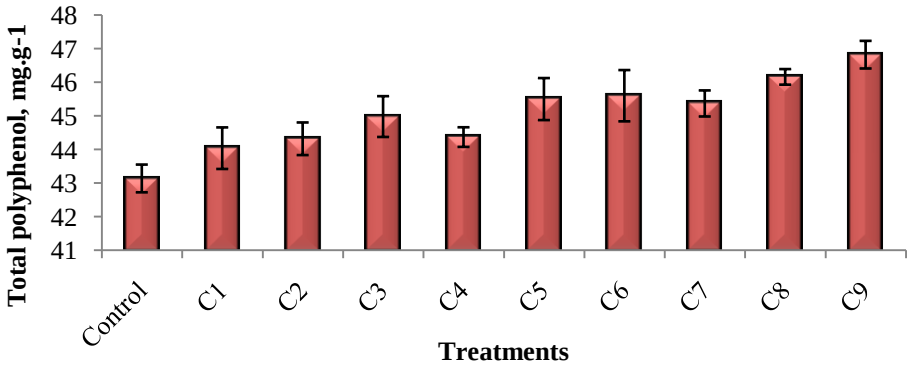


Fig. 4.27 Effect of cardamom addition on total polyphenol content of instant green tea

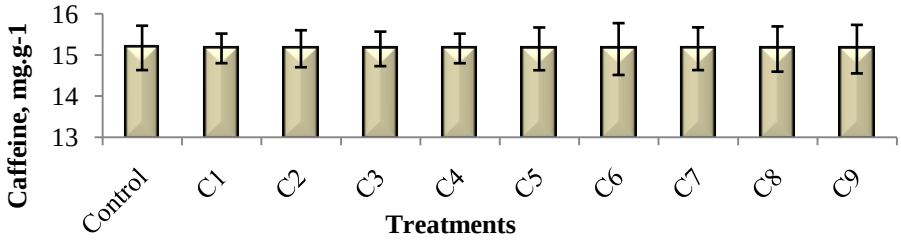


Fig. 4.28 Effect of cardamom addition on caffeine content of instant green tea

C1-1 g cardamom with extraction time of 20 min; C2-2 g cardamom with extraction time of 20 min; C3-3 g cardamom with extraction time of 20 min; C4-1 g cardamom with extraction time of 30 min; C5-2 g cardamom with extraction time of 30 min; C6-3 g cardamom with extraction time of 30 min; C7-1 g cardamom with extraction time of 40 min; C8-2 g cardamom with extraction time of 40 min; C9-3 g cardamom with extraction time of 40 min;

Caffeine

Results of caffeine content in cardamom flavoured green tea are given in Appendix C7 and C10. Caffeine content of instant green tea was 15.17 $mg.g^{-1}$ whereas in case of cardamom flavoured instant green tea it was from 15.158 to 15.140 $mg.g^{-1}$. It was clear from the Fig.4.28 that, addition of cardamom had insignificant effect on caffeine content. Since the cardamom is poor source of caffeine it has not added any caffeine content to instant green tea powder.

4.4.2.2 Sensory Evaluation

The selected samples of cardamom flavoued instant green tea were C3, C5, C6, C8, C9 and these samples was reconstituted with optimum quantity of water and fresh green tea was kept as control for sensory evaluation. Sensory analysis was done using nine point hedonic scale and the sensory attributes were ranked using comprehensive fuzzy logic model.

Scale factors, quality factors, FMF, NFMF and JMF were then calculated and presented in the Appendix C11 and C12. The fuzzy comprehensive model ranking of cardamm flavoured green tea was C6>C5=C0>C7=C3=C7. It was observed from the Table 4.9, that the sample C6 (3 g cardamom with extraction time of 30 min) ranked first with highest score in flavour attribute. Sample C0 (Control) and C5 (2 g cardamom with extraction time of 30 min) ranked second with highest score in colour and appearance. Referring to Table 4.9 it was observed that the reconstituted C6 sample scored 0.195, 0.180, 0.210, 0.199 and 0.199 for colour, taste, flavour, astringency and overall acceptability respectively. The C6 sample satisfied the quality criteria of flavour and taste attributes but not for colour, astringency and overall acceptability. Even though C6 sample did not reach the weightage given to quality attributes it had higher score for flavour and taste which indicate that cardamom flavoured green tea is more acceptable than the green tea without flavour.

Table 4.9 Quality ranking of cardamom flavoured instant green tea sample

Sensory attributes	**Weightage**	**Sample C0**	**Sample C7**	**Sample C6**	**Sample C8**	**Sample C3**	**Sample C5**
Colour and Appearance	0.21	0.199	0.195	0.195	0.192	0.192	0.199
Taste	0.18	0.18	0.18	0.18	0.18	0.18	0.18
Flavour	0.21	0.189	0.192	0.21	0.192	0.192	189
Astringency	0.2	0.191	0.197	0.199	0.197	0.195	0.194
Overall acceptability	0.2	0.194	0.197	0.199	0.195	0.197	0.189
		0.199/colour and appearance	0.197/overall acceptability	0.21/flavour	0.197/astringency	0.197/overall acceptability	0.199/colour and appearance
		II	**III**	**I**	**III**	**III**	**II**

4.4.3 Tulsi Flavoured Instant Green Tea

The dried tulsi and green tea leaves were extracted together to produce tulsi flavoured instant green tea. Production method is explained in section 3.5.3 and treatment combination adopted is given Table 3.8. Tulsi flavoured Instant green tea obtained from different treatment was tested for total polyphenols, flavonoids, caffeine and results are presented in Appendix C13.

4.4.3.1 Biochemical Properties of Tulsi Flavoured Instant Green Tea

The total flavonoid content of tulsi flavoured instant green tea ranged from 27.11 to 29.05 $mg.g^{-1}$ and it found insignificant. However slight increase in total flavonoids and polyphenols were observed and it revealed from Fig. 4.29 as the mass of tulsi increases total flavonoid increased from 27.11 to 28.75. The total polyphenols of tulsi flavoured instant green tea ranged from 44.01 to 48.56 $mg.g^{-1}$. The highest total polyphenols was recorded in H8 treatment and lowest total polyphenols content was recorded in H1. It was clear from the Fig. 4.30, increasing the extraction time and quantity of tulsi enhanced the total polyphenols content of tulsi flavoured instant green tea. By extending the time of extraction slight increase in total flavoinoid concentration and total polyphenols concentration was observed. Same trends was also reported by Upadhyay *et al*. (2015). As per the findings of Pedro *et al*. (2016) the total polyphenol content of tulsi varied from 2.72 to 5.08 $mg.g^{-1}$ and total flavonoids from 0.40 to 1.06 $mg.g^{-1}$ with various extraction condition, that increased phytochemicals in instant tulsi flavoured green tea. Statistical analysis also confirmed insignificant variation and ANOVA is tabulated in Appendix C14 and Appendix C15).

Caffeine

Results of the caffeine content in tulsi flavoured green tea obtained from various treatment are depicted in Appendix C13 and C16. The caffeine content of tulsi flavoured green tea varied from 15.15 to 15.00 $mg.g^{-1}$ among the eight treatment combinations. The highest caffeine content was recorded in H1 treatment and least in

H8. It was clear from the Fig.4.31, increasing the extraction time and concentration of tulsi had no significant variation in caffeine content.

4.4.3.2 Sensory Evaluation

The two samples which have highest phytochemicals (H7 and H8) had more bitterness and H1 which had no flavour was screened out before sensory evaluation. The rest five samples selected for sensory evaluation were, green tea with 2:10 tulsi to green tea leaves extracted for 30 min (H2), green tea with 3:10 tulsi to green tea leaves extracted for 30 min (H3), green tea with 4:10 tulsi to green tea leaves extracted for 30 min (H4), green tea with 1:10 tulsi to green tea leaves extracted for 40 min (H5), green tea with 2:10 tulsi to green tea leaves extracted for 40 min (H6). The selected treatments of tulsi flavoured green tea was reconstituted with optimum quantity of water and fresh green tea as control was kept for sensory evaluation. Sensory analysis was done using 9 point hedonic scale and the sensory attributes were ranked using comprehensive fuzzy logic model. The scale factors, quality factors, FMF, NFMF and judgment membership functions calculated as explained in 3.6.5 and presented in Appendix C17 and C18. The fuzzy comprehensive model ranking for tulsi flavoured green tea was H3>H0>H2=H5>H4>H6.

Based on the quality ranking, H3 samples got first rank (0.207) based on the score obtained for the attribute quality flavour followed by H0 (0.203) sample on its colour and appearance. Referring to Table 4.10 it was observed that the reconstituted H3 sample scored 0.2, 0.180, 0.207, 0.194 and 0.2 for colour, taste, flavour, astringency and overall acceptability respectively, whereas the corresponding weightage average of these quality attributes are 0.21, 0.18, 0.21, 0.2 and 0.20. The H3 sample, satisfied the quality criteria set for taste and overall acceptability but not the colour, astringency and flavour. Even though H3 sample did not reach the weightage given to quality attributes it had higher score than the control so this can be acceptable. It clear from the quality scores of H4, if the concentration of tulsi increases there was noticeable reduction in scores for flavoure, astringency and

overall acceptability. It also clear from the scores of H5 and H6 if the extraction time of tulsi increases, it reduces the flavour, overall acceptability and increases the astringency. That might be the reason for lowest scores of H5 and H6.

4.4.4 Overview on Biochemical Properties

The biochemical variation after each processing steps of instant green tea is given in Table 4.11 The major constituents of green tea *i.e*., total flavonoids, total polyphenols and caffeine had no much variation during the processing. This indicates, the processing steps adopted for production of instant flavour green tea were acceptable.

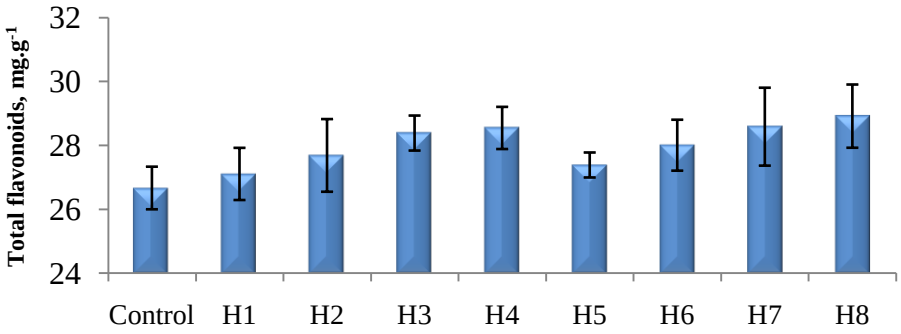


Fig. 4.29 Effect of tulsi addition on total flavonoid content of instant green tea

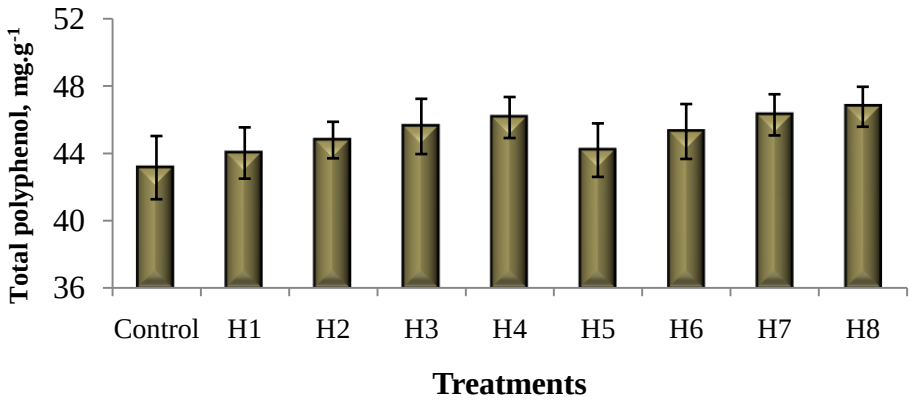


Fig. 4.30 Effect of tulsi addition on total polyphenol content of instant green tea

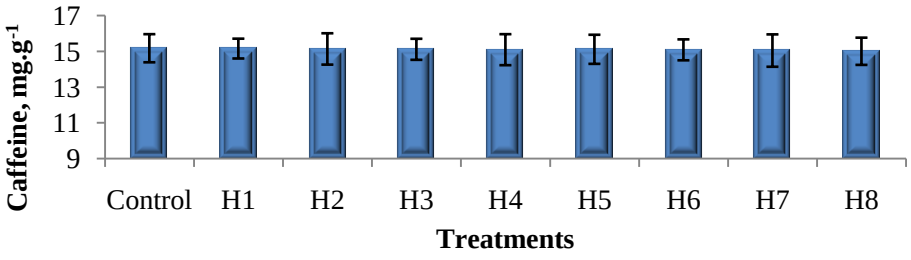


Fig. 4.31 Effect of tulsi addition on total polyphenol content of instant green tea

H1- Green tea with 1:10 tulsi to green tea leaves extracted for 30 min; H2- 2:10 tulsi to green tea leaves extracted for 30 min; H3- green tea with 3:10 tulsi to green tea leaves extracted for 30 min; H4- green tea with 4:10 tulsi to green tea leaves extracted for 30 min; H5- green tea with 1:10 tulsi to green tea leaves extracted for 40 min; H6- green tea with 2:10 tulsi to green tea leaves extracted for 40 min; H7- green tea with 3:10 tulsi to green tea leaves extracted for 40 min; H8- - green tea with 4:10 tulsi to green tea leaves extracted for 40 min.

Table 4.10 Quality ranking of tulsi flavoured instant green tea sample

	Weightage	**Sample H0**	**Sample H6**	**Sample H4**	**Sample H2**	**Sample H3**	**Sample H5**
Colour and Appearance	0.21	0.203	0.186	0.196	0.194	0.2	0.198
Taste	0.18	0.179	0.18	0.18	0.18	0.18	0.18
Flavour	0.21	0.193	0.171	0.188	0.192	0.207	0.19
Astringency	0.2	0.192	0.186	0.19	0.196	0.194	0.186
Overall acceptability	0.2	0.192	0.192	0.196	0.198	0.2	0.198
		0.203/Colour and appearance	0.192/Overall acceptability	0.196/Overall acceptability	0.198/Overall acceptability	0.207/Flavour	0.198/Overall acceptability
		II	V	IV	III	I	III

Table. 4.11 Variation of biochemical properties at each processing steps

Sl. No.	Quality parameters	Processing steps				
		Extraction	Spray dried IGT	Ginger Flavoured IGT	Cardamom Flavoured IGT	Tulsi Flavoured IGT
1	Total polyphenol, $mg.g^{-1}$	50.26	43.18	44.79	45.60	45.59
2	Total flavonoid, $mg.g^{-1}$	26.87	26.67	27.57	26.97	28.39
3	Caffeine, $mg.g^{-1}$	19.22	15.17	15.06	15.14	15.11

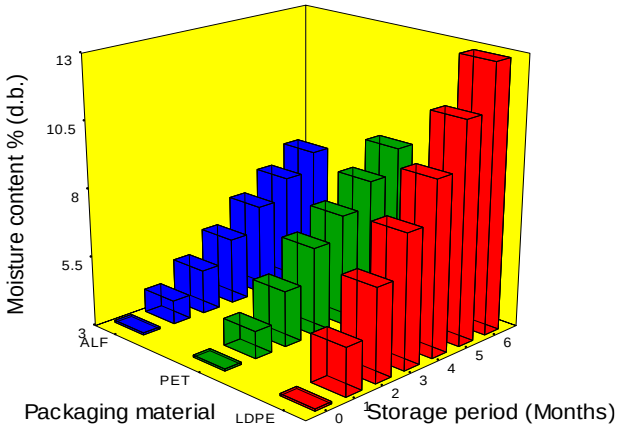
Experiment IV

4.5 STORAGE STUDIES OF INSTANT GREEN TEA POWDER IN DIFFERENT PACKAGING MATERIAL

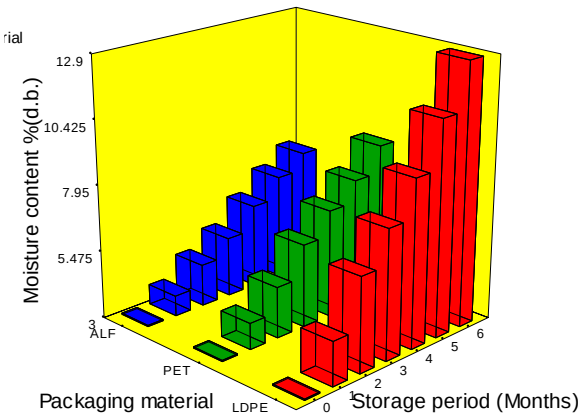
The best green tea sample from each flavour (G2, C6 and H3) was selected and packed in three different packaging materials (ALF, PET and LDPE). Shelf-life studies for three flavoured of instant green tea powder was done for six months. The quality parameters *i.e.,* moisture content, water activity, colour change, bulk density, solubility, wettability, total flavonoids, total polyphenols and caffeine were analysed for an interval of one month and the results are tabulated and discussed.

4.5.1 Moisture Content

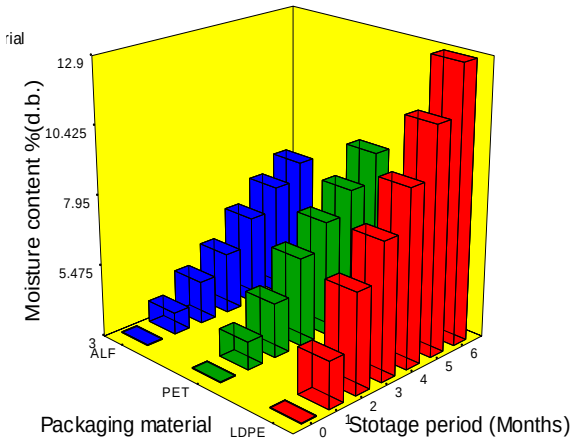
The effect of storage on moisture content of instant flavoured green tea illustrated by plotting three dimensional graph (Fig. 4.32). During the storage period moisture content of all the samples showed increasing trend, irrespective of flavours. The high moisture gain was observed in LDPE followed by PET and least in case of ALF. The moisture uptake of powder depends on water vapour permeability (WVP) of the packaging film and hence ALF shows less uptake of water. ANOVA for moisture indicated that the type of packaging material and storage time were significantly ($p<0.0001$) affected the moisture gain of the powder during storage (Appendix D2). Similar trend of increase in moisture content were reported by many authors such as, Wong and Lim (2016) for spray-dried papaya powder, Jena and Das (2012) for coconut milk powder in ALF.



(a)
Ginger flavoured instant green tea



(b)
Tulsi flavoured instant green tea

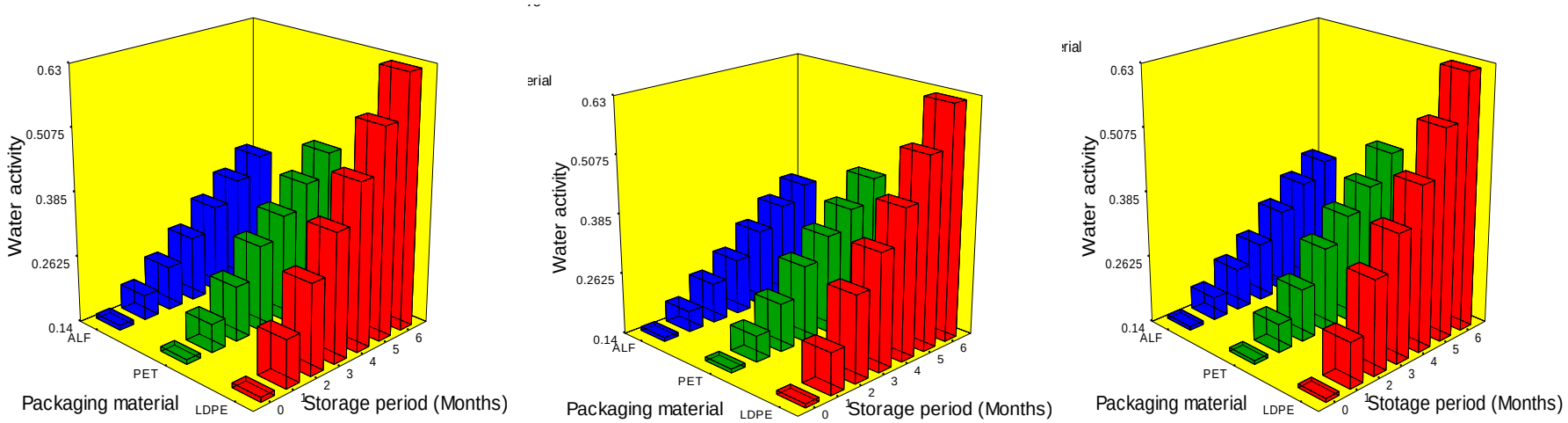


(c)
Cardamom flavoured instant green

Fig. 4. 32 Effect of storage period on moisture content of flavoured instant green tea in different packaging materials

4.5.2 Water Activity

Water activity of instant flavoured green tea increased significantly with storage and packaging material. The Fig. 4.33 (a-c) represents the change in water activity instant flavoured green tea with respect to storage and packaging. The initial water activity of ginger, tulsi and cardamom flavoured green tea were 0.149, 0.148 and 0.147. During the storage period, the water activity of ginger flavoured green tea was found to be increased to 0.374 in ALF, 0.433 in PET and 0.625 in LDPE after six months of storage. In case of tulsi flavoured green tea, water activity increased to 0.372 in ALF, 0.432 in PET and 0.615 in LDPE after six months of storage. In cardamom flavoured green tea, water activity was increase to 0.369 in ALF, 0.432 in PET and 0.610 in LDPE after six months of storage. Water activity is directly proportional to moisture content, that is the reason behind gradual increases in a_w of all the samples. Therefore ALF is a more effective packaging material in preventing the increase of water activity. The results were analogous to the finding of Pua *et al*. (2008) for Jackfruit (*Artocarpus heterophyllus*) powder packaged in aluminium laminated polyethylene and metallized co-extruded biaxially oriented polypropylene, Ramachandra and Rao (2013) for Aloe vera gel power in ALF, PP and BOPP and Kumar and Mishra (2004) for yoghurt powder stored in HDPE and ALF. Analysis of variance (ANOVA) for water activity of ginger, tulsi and cardamom flavoured green tea stored in three different packaging materials is given in Appendix D4.



(a)
Ginger flavoured instant green tea

(b)
Tulsi flavoured instant green tea

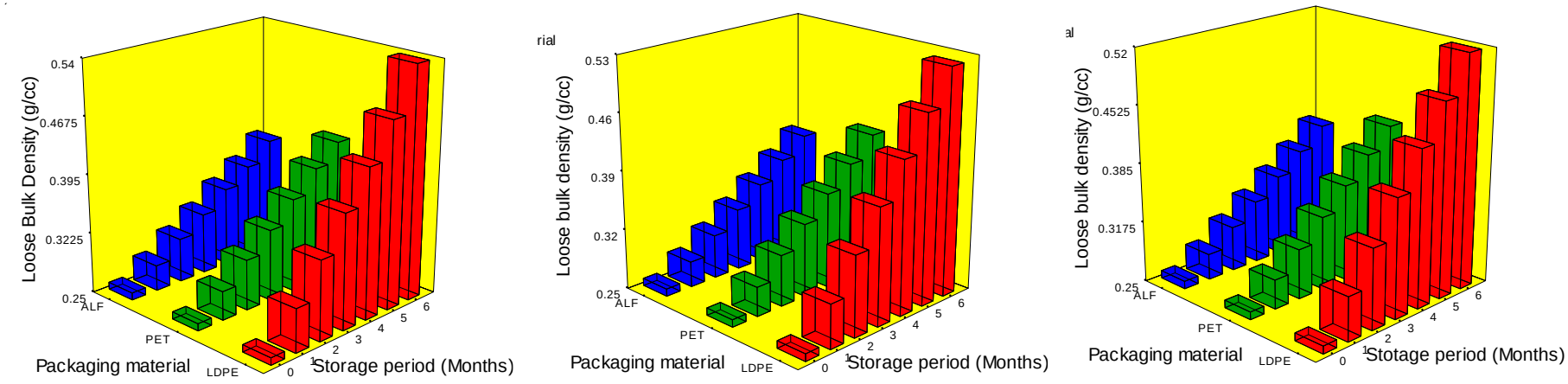
(c)
Cardamom flavoured instant green tea

Fig. 4. 33 Effect of storage period on water activity of flavoured instant green tea in different packaging materials

4.5.3 Loose Bulk Density and Tapped Bulk Density

The effect of storage on loose bulk density and tapped density of instant flavoured green tea powder in three different packaging materials under atmospheric condition was analysed and presented in Appendix D5 and D7. The both bulk density showed significant variation ($p<0.0001$) during storage and it is represented in Fig. 4.34 and 4.35. The initial loose bulk density and tapped bulk density of instant flavoured flavoured green tea were 0.259 and 0.337 $g.cc^{-1}$ respectively. The increase bulk density at the end of 6^{th} month was observed in all the three packaging material, irrespective of flavours. The highest loose bulk density of ginger flavoured green tea was 0.540 $g.cc^{-1}$, in tulsi flavoured green tea 0.523 $g.cc^{-1}$, and in cardamom flavoured green tea 0.520 $g.cc^{-1}$. The highest tapped bulk density of ginger flavoured green tea was 0.618 $g.cc^{-1}$, in case of tulsi flavoured green tea 0.600 $g.cc^{-1}$ and in cardamom flavoured green tea 0.598 $g.cc^{-1}$.

There was a gradual increase in density of the all three samples in three packaging materials. LDPE packed sample showed greater degree of increase in bulk density followed by PET and lowest in case of ALF packed samples. The variation in bulk density is comparable with the extent of moisture gain of instant flavoured green tea powder packed in different packaging materials. The reason behind the increase in bulk density may also be attributed to increased cohesiveness between powder particles caused by absorption of moisture during storage (Purushotham, 2016). This tendency is also reported by Muzaffar and Kumar (2016) for spray dried tamarind pulp powder which was packed in LDPE, ALP and glass, Chauhan and Patil (2013) in mango milk powder. ANOVA for loose bulk density and tapped bulk density indicated that the type of packaging material ($p<0.0001$), storage time ($p<0.0001$) and interaction ($p<0.005$) of these parameters have significantly affected density of instant flavoured green tea powder and same is tabulated in Appendix D6 and D8.

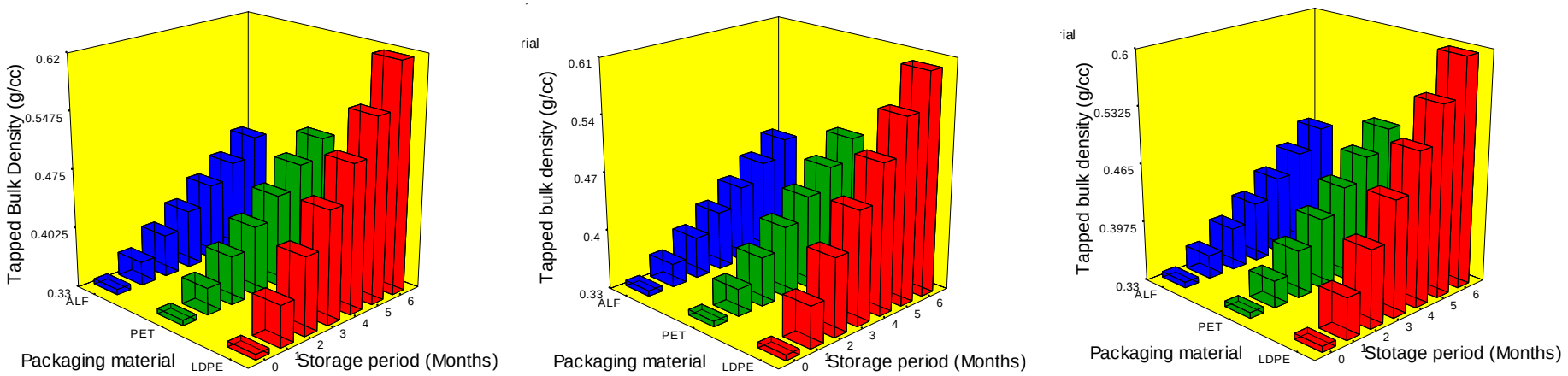


(a)
Ginger flavoured instant green tea

(b)
Tulsi flavoured instant green tea

(c)
Cardamom flavoured instant green tea

Fig. 4. 34 Effect of storage period on loose bulk density of flavoured instant green tea in different packaging materials



(a) Ginger flavoured instant green tea

(b) Tulsi flavoured instant green tea

(c) Cardamom flavoured instant green tea

Fig. 4.35 Effect of storage period on tapped bulk density of flavoured instant green tea in different packaging materials

4.5.4 Colour change

Colour change (ΔE) was calculated as the change in L*, a* and b* values between fresh and stored instant flavoured green tea powder and the variation during storage is given in Appendix D9. It can be noticed from Fig 4.36, colour changes were found to be more pronounced in LDPE and PET than ALF. During storage period, L* value decreases and a* value increases that lead to more darker colour of the instant flavoured green tea powder that bring about with higher ΔE value and also non-enzymatic browning might have influenced the variation in the colour of instant green tea powder (Pua *et al*., 2008). Factors such as temperature, moisture, carbonyl compounds, organic acids, a_w, O_2 and sugars are responsible for causing non-enzymatic browning in stored foods (Muralikrishna *et al*., 1969), therefore higher moisture containing sample resulted with grater colour difference in the present study. ANOVA (Appendix D10) for overall coloure change indicated that the type of packaging material, storage time and there interaction significantly ($p<0.0001$) affected the overall coloure change of instant flavoured green tea powder during storage. Results are in agreement with the findings of Kumar and Mishra (2004) with greater colour deviation in HDPE than ALF for fortified yoghurt powder, Yu *et al*., 2015 in for spray-dried bovine colostrums, Singh and Hathan, 2017 for beetroot powder packed in laminated aluminium film package and HDPE and Swain *et al*., 2013 for dried sweet pepper.

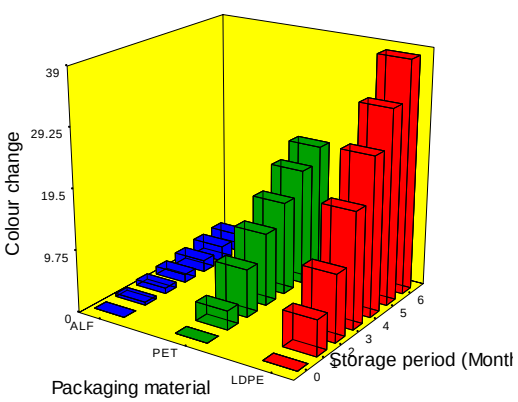
4.5.5 Solubility

The effect of packaging material on solubility of instant flavoured green tea powder during storage was analysed and presented in Appendix D11. In the beginning of storage solubility of ginger, tulsi and cardamom flavoured green tea was 84.34%, 86.24% and 86.64%, respectively. It can be understandable from Fig 4.37, irrespective of packaging material solubility of the instant flavoured green tea was decreased during the storage period in the entire sample. The maximum reduction in solubility was observed in LDPE packed samples i.e., 4.99%, 5.11% and 5.25% in

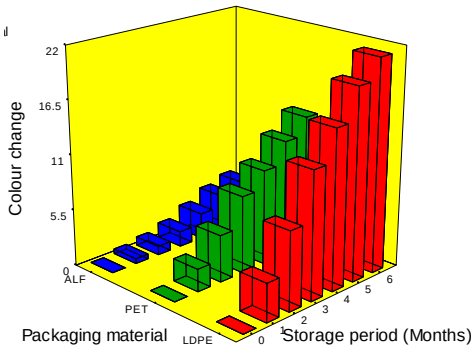
case of ginger, cardamom and tulsi flavoured green tea and very slight reduction in case of ALF packed samples. But the variation is not prominent in all the samples and it is also confirmed by ANOVA (Appendix D12), which indicate that packaging material and storage time don't have significant ($p>0.05$) effect on solubility of instant flavoured green tea powder. The decrease of powder solubility was related to the residual moisture in the powder which lead to being less soluble when moisture content was high (Goula and Adamopoulos 2010; Muzaffar, and Kumar 2016). Therefore LDPE packed sample showed higher reduction in solubility followed by PET and ALF packed samples. Similar trend was observed by Wong and Lim (2016) for spray-dried papaya powder packaged in aluminium laminated polyethylene and polyethylene terephthalate, Chauhan and Patil, 2013 for mango milk powder stored in flexible packaging material.

4.5.6 Wettability

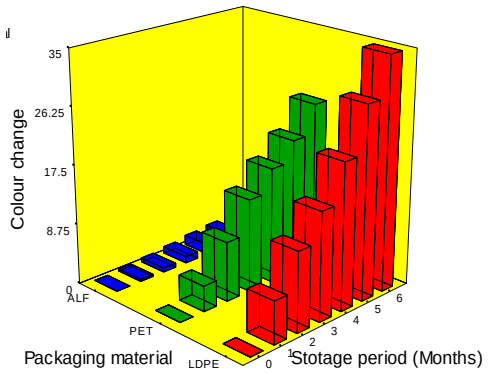
The effect of packaging materials on wettability of instant flavoured green tea powder during storage period was analysed and illustrate in Appendix D13. At initial stage wettability of ginger, tulsi and cardamom flavoured green tea was 152, 150 and 149 s, respectively. It can point out from Fig. 4.38 wettability of all the samples decreased during the storage, but the highest reduction can be observed in case of LDPE packed sample i.e., 62 s in ginger flavoured green tea, 60 s in case of tulsi flavoured green tea and 59 s in cardamom flavoured green tea. Reason for the reduction in average wettability time can be correlated with the moisture content. At lower moisture content particle instantanization times is higher (Fernandes *et al*., 2013), that might be the reason for reduction of average wettability time during the storage period. Since the moisture migration is higher in LDPE and PET there was greater reduction in average wettability time in these packaging materials. The statistical analysis also confirms that, type of packaging material and storage time significantly ($p<0.0001$) affected the wettability of instant flavoured green tea (Appendix D14).



(a)
Ginger flavoured instant green tea

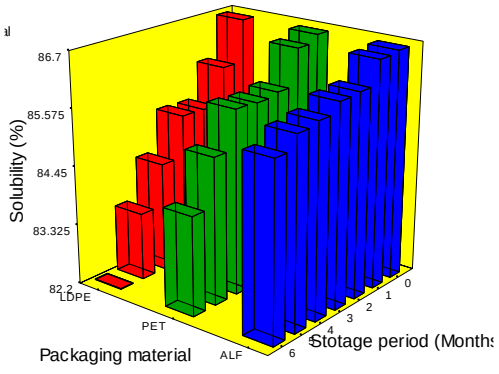


(b)
Tulsi flavoured instant green tea

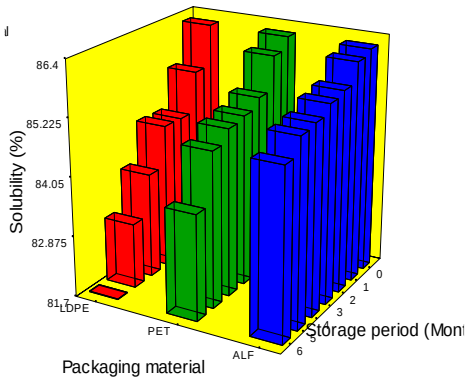


(c)
Cardamom flavoured instant green tea

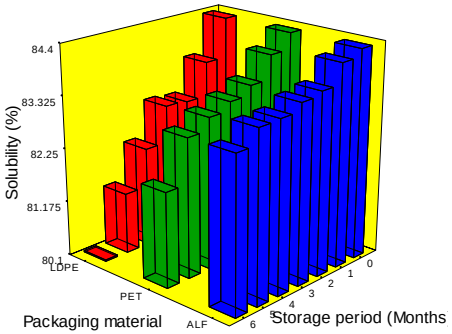
Fig. 4.36 Effect of storage period on colour change of flavoured instant green tea in different packaging materials



(a)
Ginger flavoured instant green



(b)
Tulsi flavoured instant green tea

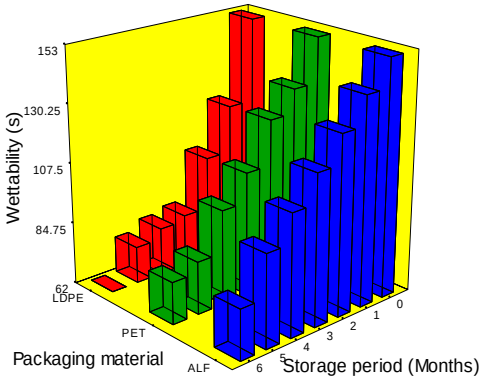


(c)
Cardamom flavoured instant green

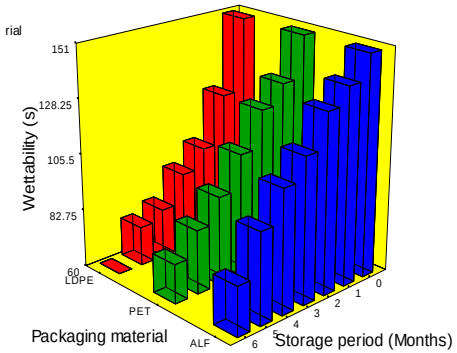
Fig. 4.37 Effect of storage period on solubility of flavoured instant green tea in different packaging materials

4.5.7 Total Polyphenol

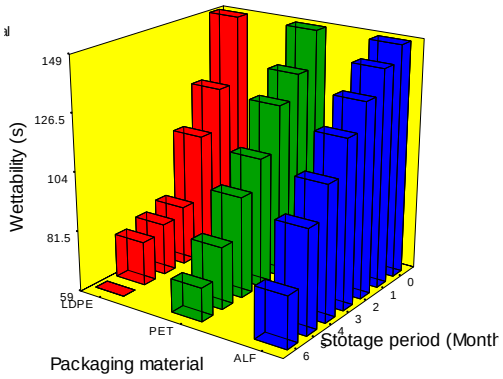
The total polyphenol content of instant flavoured green tea powders showed significant variation during the storage. The Fig. 4.39 represented the variation of total polyphenols during the storage with respect to three packaging materials. Initially total polyphenols of ginger, cardamom and tulsi flavoured green tea was 44.74, 45.60 and 45.56 $mg.g^{-1}$, respectively. However, the decrease in total polyphenols was gradual for the first four months of storage, after which it decreases rapidly. The total polyphenol content of ginger flavoured green tea at the end of 6^{th} month is 37.41 $mg.g^{-1}$ in ALF, 29.91 $mg.g^{-1}$ in PET and 23.91 $mg.g^{-1}$ in LDPE. In case of tulsi flavoured green tea, 41.49 in ALF, 34.38 in PET and 30.38 $mg.g^{-1}$ in LDPE after six months of storage. In cardamom flavoured green tea, 37.16 in ALF, 30.91 in PET and 21.92 $mg.g^{-1}$ in LDPE after six months of storage. Reduction of polyphenols might be due to the degradation of polyphenols during storage period. Since the polyphenols are susceptible to moisture and oxygen there was more degradation can be observed in LDPE and PET than ALF. Similar trend was observed by Zoric *et al*., 2017 for phenolic content of spray dried cherry powder packed in different laminated pouches. Obtained results are also supported by Cheng *et al*., 2017 and Fang and Bhandari, 2011 for spray dried bayberry powder and bayberry polyphenols. ANOVA (Appendix D16) also indicated significant effect of packaging material, storage time and their interaction ($p<0.0001$) on total polyphenols of instant flavoured green tea powder.



(a)
Ginger flavoured instant green

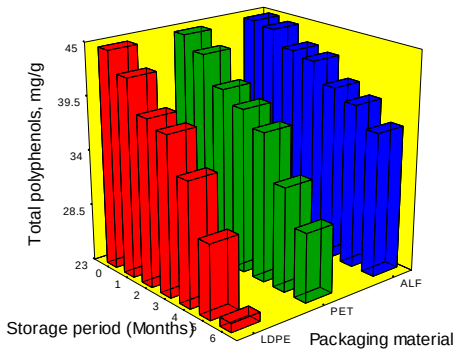


(b)
Tulsi flavoured instant green tea

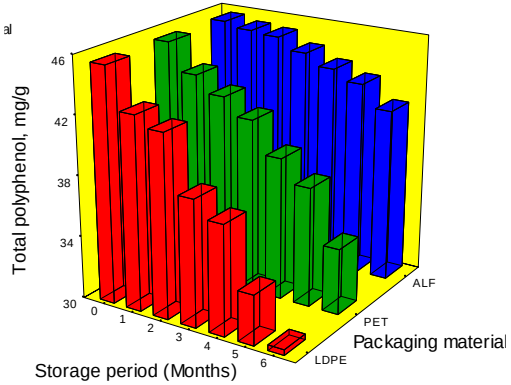


(c)
Cardamom flavoured instant green tea

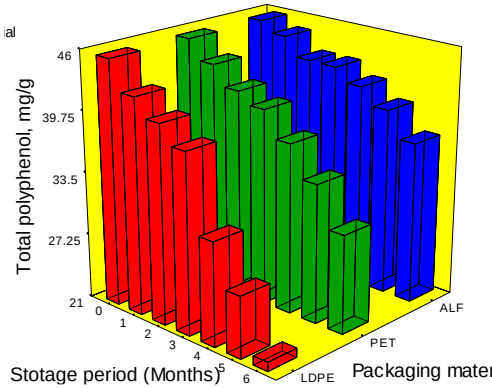
Fig. 4.38 Effect of storage period on wettability of flavoured instant green tea in different packaging materials



(a)
Ginger flavoured instant green



(b)
Tulsi flavoured instant green tea



(c)
Cardamom flavoured instant green tea

Fig. 4.39 Effect of storage period on total polyphenols of flavoured instant green tea in different packaging materials

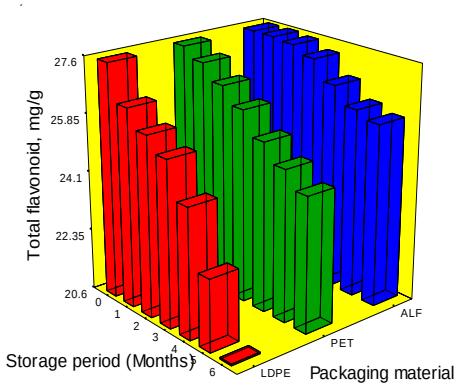
4.5.8 Total Flavonoids

The initial total flavonoid content of ginger, cardamom and tulsi flavoured green tea was 27.57, 26.97 and 28.39 $mg.g^{-1}$, respectively. The total flavonoid content decreases with storage period. The Fig. 4.40 displays the changes in total flavonoid content during storage with three packaging materials. The maximum reduction of total flavonoids was observed in LDPE packed instant flavoured green tea powder followed by PET and ALF after six month of storage period. Reduction of in total flavonoids is due to degradation of total flavonoid during storage period. Obtained results were supported by the report of Kosinska and Andlavera (2014) and they stated that, tea catechins are not stable during long-term storage. According to the findings of Friedman *et al.*, 2009 in six months' storage EGCG decreased by one-third and ECG decreased by half but in present study it is true in case of LDPE packaging but comparatively less in ALF that might be due to better barrier properties of ALF. Ortiz *et al*., 2008 reported that, catechins in dried green tea beverage powders was stable for 3 months and catechin are stable when sample was stored below the onset glass transition temperature (Tg), this might be the reason for less degradation of catechins in ALF packed instant flavoured green tea. Analysis of variance (Appendix D18) for total flavonoids also indicated that the type of packaging material and storage time significantly ($p<0.0001$) affected the total flavonoids of instant flavoured green tea powder.

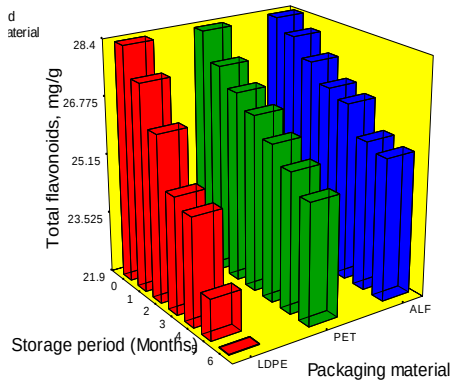
4.5.9 Caffeine

The initial caffeine content of ginger, cardamom and tulsi flavoured green tea was 15.06, 15.142 and 15.11 $mg.g^{-1}$, respectively. Variation of caffeine during storage with three different packaging materials and ANOVA for the same is given in Appendix D19 and Appendix D20. The storage period and packaging materials did not show significant variation in the caffeine content of instant flavoured green tea and is represented in Fig. 4.41. As per the report of Bazinet *et al*., 2010, caffeine is more stable than catechins during the storage. That might be the reason for retention

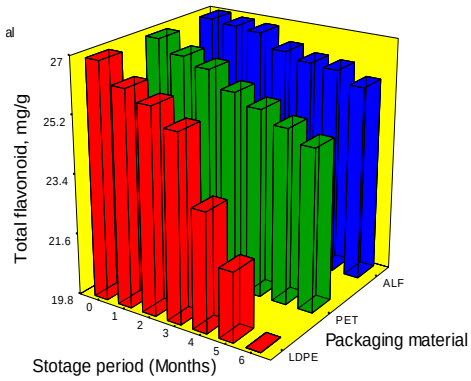
of caffeine during the storage. Similar findings was reported by Sakata (2011) with no reduction of caffeine content after 28 days of storage of freshly prepared and RTD green tea. Present findings are also supported by Haqiqat *et al*., 2004 who as reported that tea caffeine is not significantly affected by packaging material.



(a)
Ginger flavoured instant green

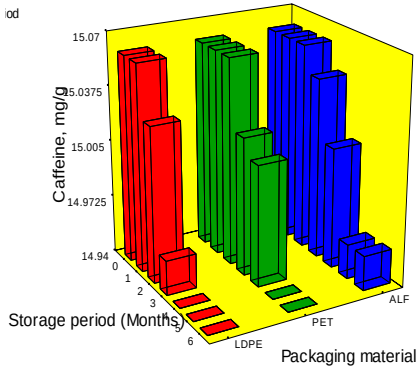


(b)
Tulsi flavoured instant green tea

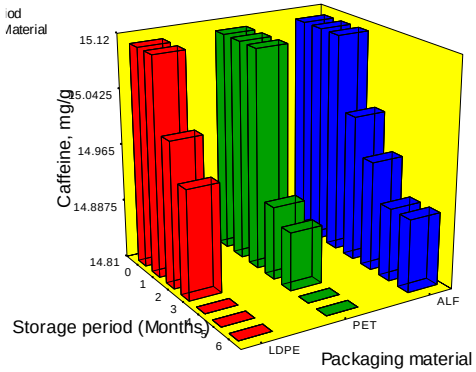


(c)
Cardamom flavoured instant green tea

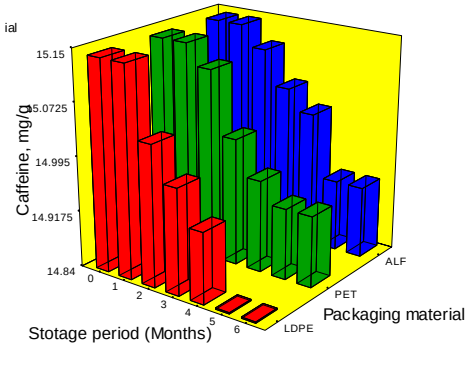
Fig. 4.40 Effect of storage period on total flavonoids of flavoured instant green tea in different packaging materials



(a)
Ginger flavoured instant green tea



(b)
Tulsi flavoured instant green tea



(c)
Cardamom flavoured instant green tea

Fig. 4.41 Effect of storage period on caffeine of flavoured instant green tea in different packaging materials

4.6 SHELF-LIFE PREDICTION OF INSTANT FLAVOURED GREEN TEA POWDER BASED ON MOISTURE GAIN

The effect of packaging materials and storage conditions on the shelf-life of instant flavoured green tea powder was assessed with accelerated environment condition of 38±1°C and 90±1% RH and results are presented below.

The initial moisture content of the instant flavoured green tea was 0.030 kg water.kg^{-1} dry solids. The actual values of water activity with respect to moisture content of three instant flavoured green tea with respect to three packaging material is substituted to GAB equation and resultant predicted moisture content along with actual moisture content with three packaging material LDPE, PET and ALF has been shown in Fig. 4.42-4.50. The maximum moisture gain of ginger flavoured tea was 0.2270, 0.1929 and 0.1748 in LDPE, PET and ALF, respectively for 35th day. The maximum moisture gain of cardaom flavoured tea is 0.2095, 0.1683 and 0.1580 in LDPE, PET and ALF, respectively for 35th day. The maximum moisture gain of tulsi flavoured tea is 0.1940, 0.1642 and 0.1500 in LDPE, PET and ALF, respectively for 35th day.

The moisture content and corresponding water activity of three instant flavoured green tea is given in Appendix E (Table E1-E3). By substituting the area of the pouch containing silica gel and weight of silica gel at different duration (equations 3.21 and 3.22) permeability of packaging material was calculated as 4.08×10^{-12} 3.70×10^{-12} and 2.37×10^{-12} and kg water.m^{-2}.s^{-1}.Pa^{-1} for LDPE, PET and ALF, respectively. The length and breadth considered for the calculation is tabulated in Appendix E4. Shelf life of instant flavoured was evaluated by substituting the area of packaging material and weight of powder in the equation 3.23 gives the time required to attain the critical moisture content of 0.08 kg water.kg^{-1} spray dried powder. MATLAB program for shelf life prediction is given in Appendix F

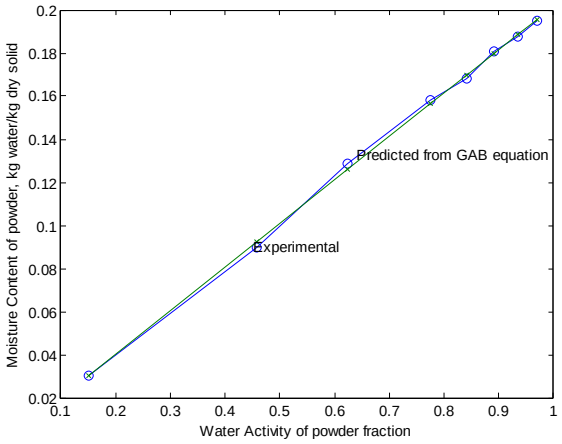


Fig. 4.42 Graphical relationship between water activity and moisture content of ginger flavoured instant green tea powder stored in LDPE

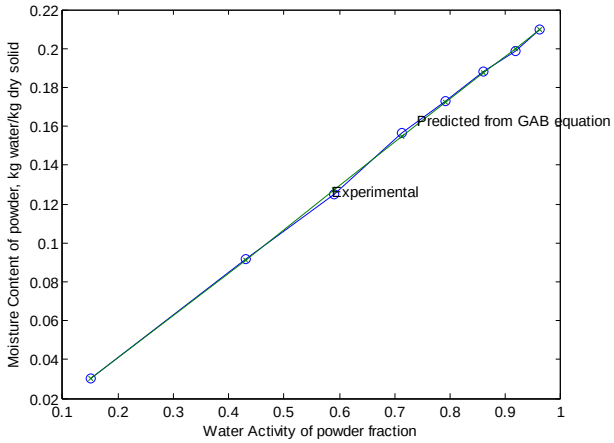


Fig. 4.43 Graphical relationship between water activity and moisture content of cardamom flavoured instant green tea powder stored in LDPE

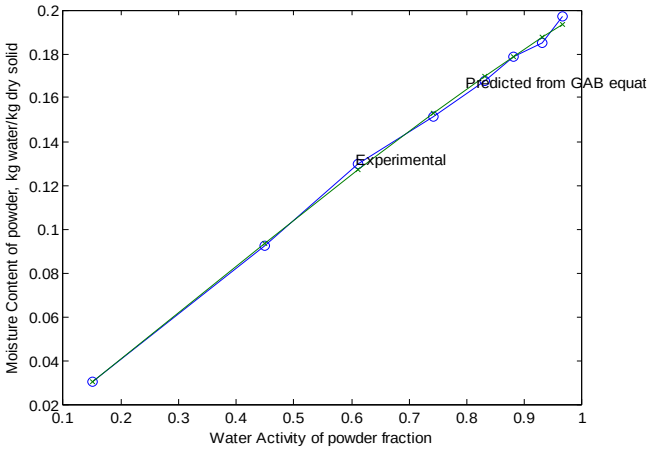


Fig. 4.44 Graphical relationship between water activity and moisture content of tulsi flavoured instant green tea powder stored in LDPE

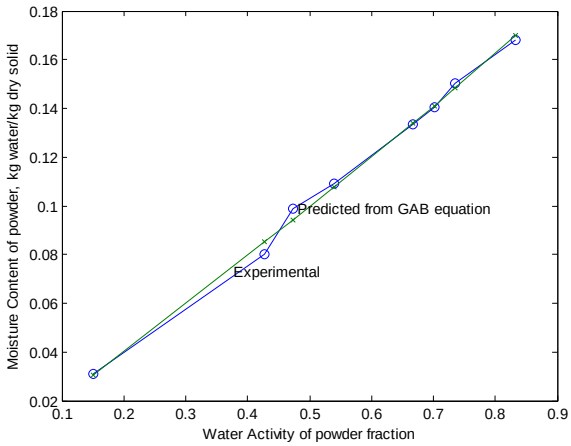


Fig. 4.45 Graphical relationship between water activity and moisture content of ginger flavoured instant green tea powder stored in PET

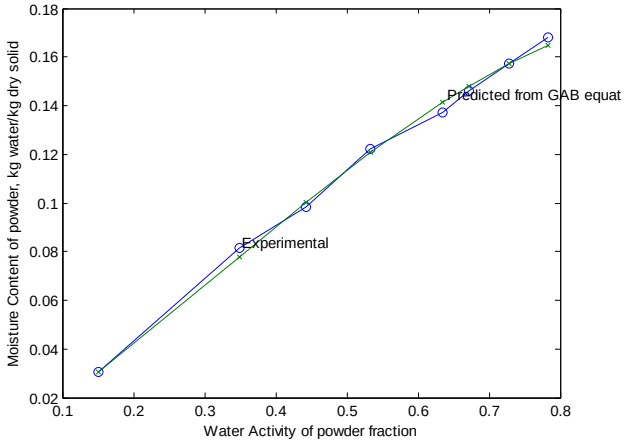


Fig. 4.46 Graphical relationship between water activity and moisture content of cardamom flavoured instant green tea powder stored in PET

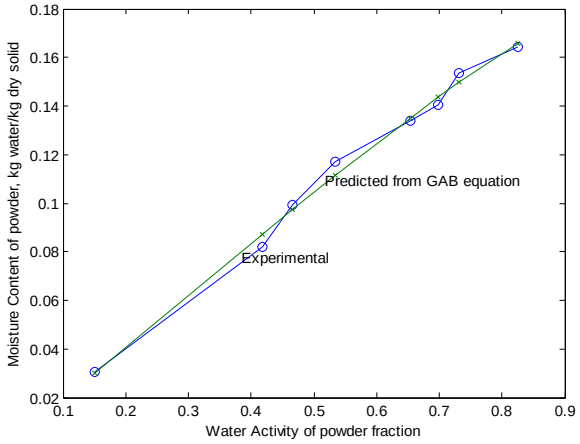


Fig. 4.47 Graphical relationship between water activity and moisture content of tulsi flavoured instant green tea powder stored in PET

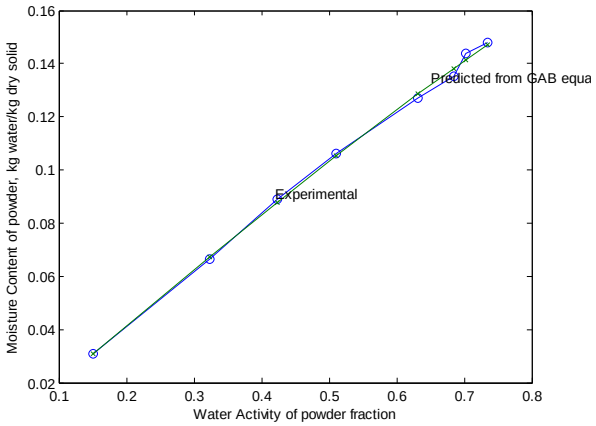


Fig. 4.48 Graphical relationship between water activity and moisture content of ginger flavoured instant green tea powder stored in ALF

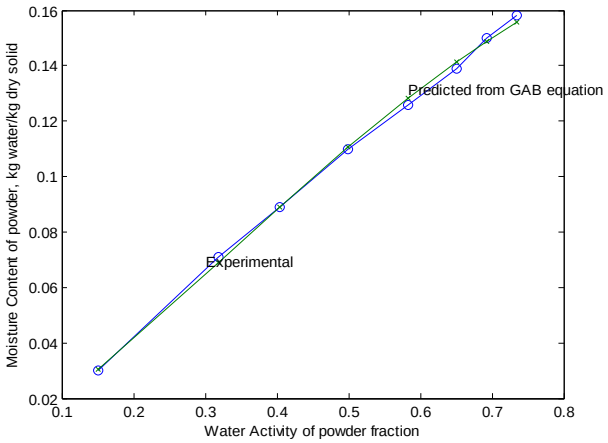


Fig. 4.49 Graphical relationship between water activity and moisture content of cardamom flavoured instant green tea powder stored in ALF

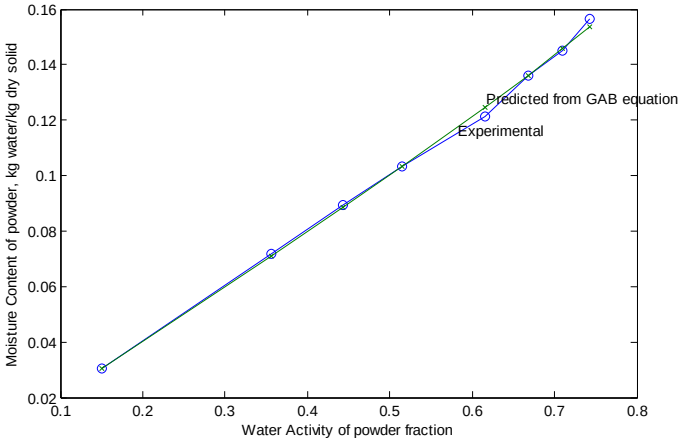


Fig. 4.50 Graphical relationship between water activity and moisture content of cardamom flavoured instant green tea powder stored in ALF

The shelf life of powder predicted is 92 days when it is packed in LDPE pouches, 152 days when it is packed in PET pouches and 210 days when it is packed in ALF pouches for all the three instant flavoured green tea powders. Fig. 4.51-4.53 shows the moisture content of instant green tea powder and time required to attain critical moisture content. The predicated shelf life instant green tea powder which is packed in aluminium laminated pouches was greater than PET pouches and lowest shelf life was predicted for instant green tea powder which is packed in LDPE pouches that might be due to higher water vapour permeability of LDPE pouches than PET and ALF. Kumar and Mishra, 2004 reported the shelf life of mango soy fortified yoghurt powder was less in HDPP than ALP pouches. Singh and Hathan, 2017 reported highest moisture gain in the case of the beetroot powder packed in HDPE and least in the case Alumimium laminated pouches. Devi *et al.* (2016) for honey powder with predicted shelf-life as 222 days packed in ALF. The contradictory result was reported by Jaya and Das (2005) for mango powder with predicted shelf-life of 114 days packed in ALF; Yu *et al*., 2015 predicted the spray-dried bovine colostrums powders packaged in ALPE and PET pouches as 425.5 and 86.5 days under storage conditions of 25°C and 50% RH. The variation might due to the thickness and permeability of package used for the experiment and storage environment.

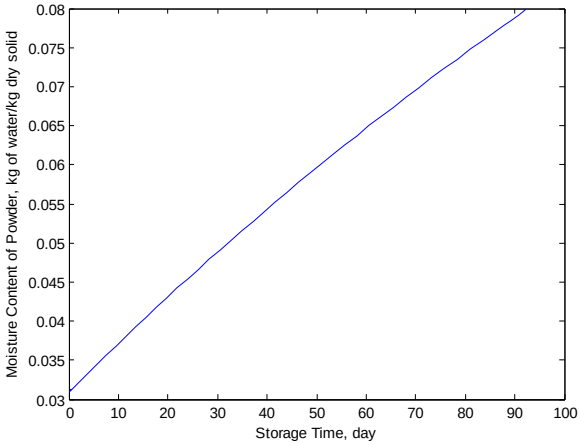


Fig. 4.51 Relationship between storage period and moisture content of instant flavoured green tea packaged in LDPE

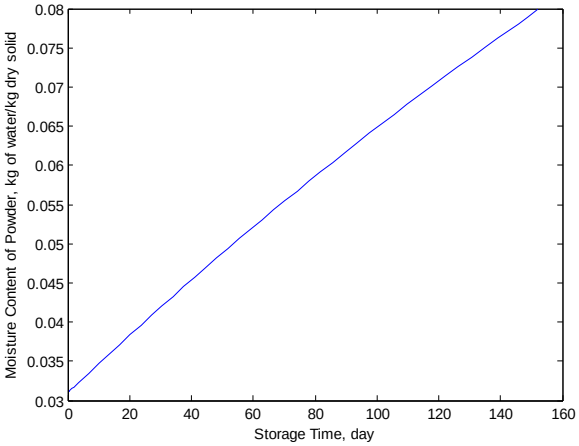


Fig. 4.52 Relationship between storage period and moisture content of instant flavoured green tea packaged in PET

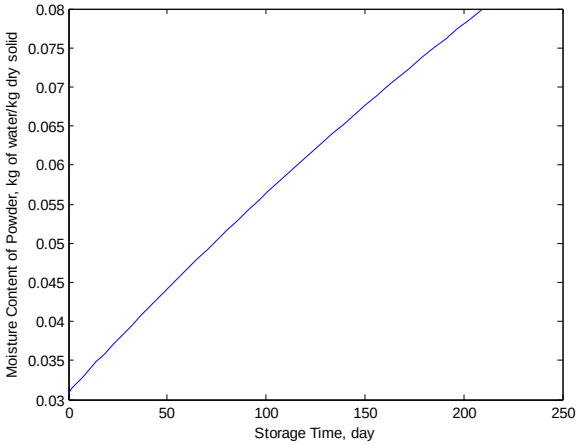


Fig. 4.53 Relationship between storage period and moisture content of instant flavoured green tea packaged in LDPE

4.7 MOISTURE SORPTION BEHAVIOUR OF INSTANT FLAVOURED GREEN TEA POWDERS

The moisture sorption process is of prime importance since many physical properties of macromolecular materials are greatly changed by the presence of sorbed moisture. Experimental evaluation of sorption characteristics and the subsequent use of mathematical models, can aid in the improvement of the processing quality of foods and appraisal of their packaging and storage requirements.

4.7.1 Moisture Sorption Isotherms

Sorption characteristics of three instant flavoured green tea powder prepared at optimized conditions were studied. The experiments were conducted as explained in Section 3.9. Experimental data of equilibrium moisture content and water activity at different experimental conditions are presented in Table 4.12. The moisture adsorption behaviour is manifested in the form of sigmoid shaped curves reflecting a Type II isotherm (Brunaue *et al*, 1938), which is typical to most of the foods. The isotherms demonstrate an increase in equilibrium moisture content with increasing water activity at constant temperature (Table 4.12). Reason for this might be hydrophilic nature of carbohydrates present in sample (Muzaffar and Kumar, 2016).

Experimental result also indicates that equilibrium moisture content of all the samples decreased with increasing temperature, at a constant water activity and it is shown in Fig. 4.54 (a-c). This behavior may be due to the decrease in the total number of active sites for water binding as a result of physical and/or chemical changes induced by temperature. At higher temperatures water molecules get excited and break away from the water binding sites of the food that decreasing the attractive force between them, thus result in decreasing the equilibrium moisture content food materials. (Muzaffar and Kumar, 2016; Sinija and Mishra, 2008). In all isotherms, the moisture uptake was slow up to a water activity of 0.42, it was moderate between 0.42 and 0.70 and the equilibrium moisture content increased sharply beyond 0.7. The isotherms showed that the product adsorbed proportionately more water towards the

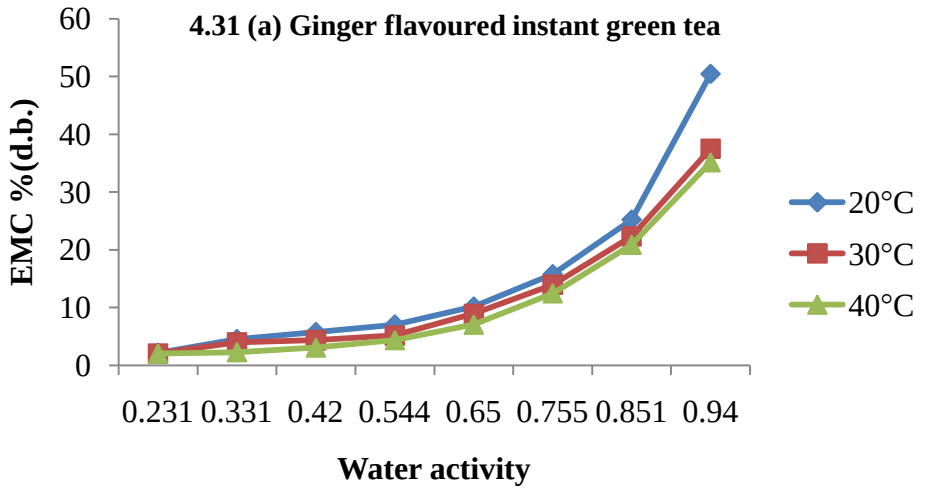
later part of the curve. Similar results have been reported by Shrestha *et al*., 2007 for spray dried lactose hydrolysed skim milk powder, Muzaffar and Kumar, 2016 for spray dried tamarind pulp powder, Bastioglu *et al*., 2016 for yogurt powder, Zuo *et al*., 2015 for vacuum-dried powder and Ramachandra and Rao, 2009 for Aloe vera gel powder.

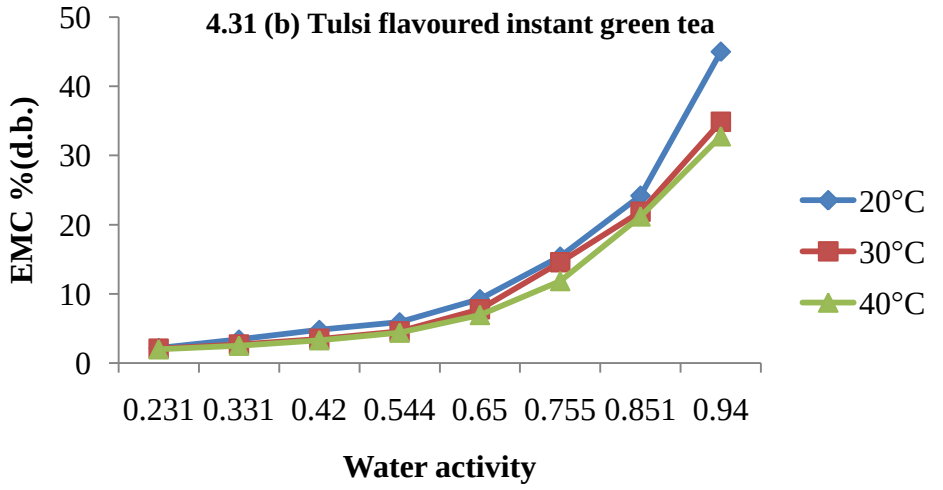
4.7.2 Isotherm Modeling

The experimental data of moisture sorption was fitted to six different models using GAB, Oswin, BET, Smith, Halsey and Henderson models (Table 3.11). Sorption curves (Fig. 4.54) are used to estimate the coefficients of different sorption models. Estimated parameters are R^2, SSE and RMSE for selected models of isotherm in the water activity and temperature ranges studied were presented in Table 4.13. From Table 4.13, GAB model gave best fit to the experimental data of ginger flavoured instant green tea was as follows, the highest R^2 values 0.999, 0.9957 and 0.9935; the lowest SSE values 1.838, 4.494 and 4.447 and lowest RMSE values 0.6063, 0.948 and 0.9431 values at 20, 30 and 40°C, respectively. The goodness of fit for GAB model as follows, the highest R^2 values 0.9989, 0.9933 and 0.9903; the lowest SSE values 1.1655, 1.192 and 1.087 and lowest RMSE values 0.5754, 1.132 and 1.287 values at 20, 30 and 40°C, respectively for tulsi flavoured instant green tea. For cardamom flavoured instant green tea GAB model goodness of fit was as follows, the highest R^2 values 0.998, 0.992 and 0.992; the lowest SSE values 8.8989, 7.013 and 5.913 and lowest RMSE values 0.7614, 1.192 and 1.087 values at 20, 30 and 40°C, respectively.

Table 4.12 Equilibrium moisture content of three different flavours of instant green tea powder at different temperatures and water activities (aw) for adsorption

Sl. No.	Temperature, (°C)	Water activity	Ginger flavoured instant green tea	Cardamom flavoured instant green tea	Tulsi flavoured instant green tea
			EMC %(d.b.)	**EMC %(d.b.)**	**EMC %(d.b.)**
1	20	0.23	2.20	2.18	2.17
2		0.33	4.58	3.50	3.40
3		0.42	5.80	4.60	4.80
4		0.54	7.09	5.60	5.90
5		0.65	10.20	8.45	9.25
6		0.76	15.80	15.10	15.40
7		0.85	25.25	23.50	24.20
8		0.94	50.47	44.00	45.00
9	30	0.22	2.10	2.00	2.10
10		0.32	4.00	2.50	2.70
11		0.43	4.40	3.20	3.50
12		0.51	5.20	4.30	4.60
13		0.67	8.96	7.30	7.80
14		0.75	13.98	14.40	14.60
15		0.84	22.34	21.30	21.90
16		0.93	37.51	34.40	34.90
17	40	0.21	2.05	1.49	1.99
18		0.32	2.30	2.11	2.51
19		0.42	3.10	2.90	3.30
20		0.48	4.39	3.80	4.40
21		0.65	7.07	6.55	6.95
22		0.75	12.50	11.35	11.85
23		0.82	20.91	20.20	21.20
24		0.90	35.20	31.78	32.78





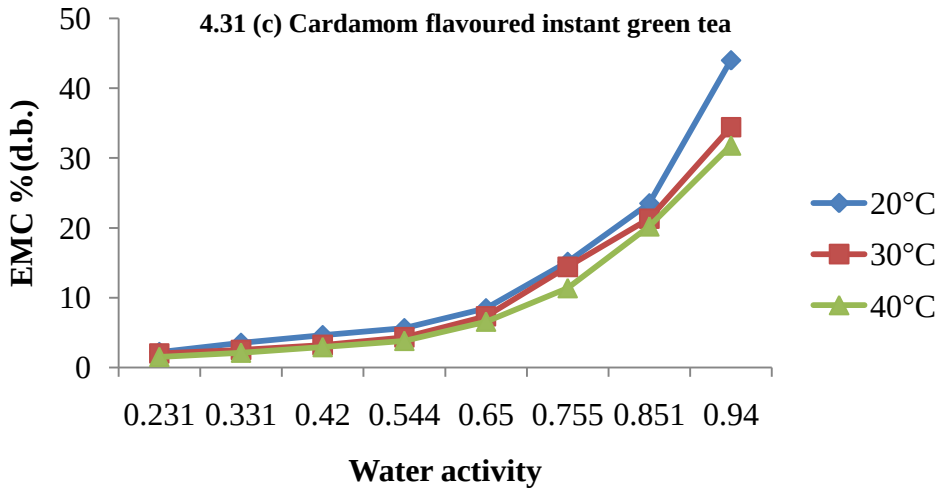


Fig. 4.54 Adsorption isotherms of flavoured instant green tea

From Table 4.13, it is evident that after GAB the corresponding fitting models were Henderson, Oswin, BET, Halsey and Smith models with corresponding decreasing higher R^2 and increasing lower SSE and RMSE values as given the Table 4.13. Constants obtained for three samples at different temperature is given in Table 4.14. From the result isotherm modeling it is clear that GAB was the most suitable model to describe the relationship between equilibrium moisture content and water activity and also it best describes the experimental adsorption data through the entire range of water activity. The actual and predicted EMC by GAB model is shown in Fig. 4.55.

Many researchers observed that GAB model was best to fit experimental adsorption data and a few of them are mentioned here. Muzaffar and Kumar, 2016 reported for spray dried tamarind powder at temperature range of 20-50°C, Bastiogula *et al*., 2016 for yogurt powder, Ramachandra and Rao, 2009 for Aloe vera gel powder at temperature range of 25-36°C, Pedro *et al*., 2010 for passion fruit pulp powder at temperature range of 20-50°C, Prasad and Agrawal (2012) in infant milk food and Bronlund and Tony (2004) for the lactose powders.

Table 4.13 Statistical parameters obtained with different models of instant flavoured green tea

		Ginger flavoured instant green tea				Cardamom flavoured instant green tea				Tulsi flavoured instant green tea			
		SSE	R^2	Adj-R^2	RMSE	SSE	R^2	Adj-R^2	RMSE	SSE	R^2	Adj-R^2	RMSE
20°C	GAB	1.838	0.9990	0.9986	0.6063	2.8989	0.998	0.9972	0.7614	1.655	0.9989	0.9984	0.5754
	Oswin	2.525	0.9986	0.9984	0.6487	9.182	0.9936	0.9925	1.237	8.48	0.9943	0.9934	1.189
	BET	6.958	0.9961	0.9955	1.077	17.17	0.988	0.986	1.689	16.67	0.9888	0.987	1.667
	Smith	88.59	0.9509	0.9428	3.843	54.35	0.9619	0.9556	3.01	50.9	0.9659	0.9602	2.913
	Halsey	8.285	0.9954	0.9946	1.175	18.03	0.9874	0.9853	1.733	18.42	0.9876	0.9856	1.752
	Henderson	14.63	0.9919	0.9905	1.562	5.876	0.9959	0.9952	0.9896	4.964	0.997	0.9965	0.8626
30°C	GAB	4.494	0.9957	0.9939	0.948	7.103	0.9924	0.9894	1.192	6.403	0.9933	0.9906	1.132
	Oswin	11.22	0.9892	0.9874	1.368	24.39	0.974	0.9697	2.016	23.54	0.9754	0.9713	1.981
	BET	17.74	0.9829	0.98	1.719	32.83	0.965	0.9592	2.339	32.13	0.9664	0.9608	2.314
	Smith	35.5	0.9657	0.96	2.43	29.73	0.9683	0.963	2.226	27.85	0.9709	0.966	2.154
	Halsey	18.93	0.9817	0.9787	1.776	35.08	0.9626	0.9564	2.418	34054	0.9639	0.9579	2.399
	Henderson	7.073	0.9932	0.992	1.086	9.936	0.9894	0.9876	1.287	9.189	0.9908	0.9888	1.238
40°C	GAB	4.447	0.9954	0.9935	0.9431	5.913	0.9928	0.9899	1.087	8.287	0.9903	0.9864	1.287
	Oswin	5.947	0.9938	0.9928	0.9956	9.672	0.9881	0.9862	1.27	11.17	0.9869	0.9847	1.364
	BET	6.81	0.9922	0.9918	1.065	11.04	0.9865	0.9842	1.356	12.86	0.9849	0.9824	1.464
	Smith	67.14	0.9303	0.9187	3.345	50.55	0.938	0.9277	2.903	54.59	0.9358	0.9251	3.016
	Halsey	8.884	0.9908	0.989	1.217	14	0.9828	0.98	1.527	14.81	0.9826	0.9797	1.571
	Henderson	7.341	0.9924	0.9911	1.106	7.171	0.9912	0.9897	1.093	10.54	0.9876	0.9855	1.325

Table 4.14 Constants of isotherm models of for three instant flavoured green tea

		20°C			30°C			40°C		
		Ginger	**Cardamom**	**Tulsi**	**Ginger**	**Cardamom**	**Tulsi**	**Ginger**	**Cardamom**	**Tulsi**
GAB	**Cg**	21.69	7.521	8.283	6.691	0.6929	1.13	2.51	0.9353	2.168
	Kg	0.9743	0.9409	0.9393	0.921	0.8651	0.8655	0.965	0.94	0.9451
	Mo	0.445	0.8671	0.8382	0.9649	3.952	2.932	1.495	2.628	1.691
Oswin	**A**	6.907	6.229	6.493	6.234	5.651	5.913	4.742	4.474	4.896
	B	0.7246	0.7159	0.7091	0.698	0.7073	0.6952	0.9189	0.9035	0.8761
BET	**Xm**	53.93	50.63	55.04	52.75	45.71	51.43	8.651	8.821	11.93
	C	0.2236	0.2156	0.2083	0.2069	0.2151	0.2027	0.6623	0.6241	0.5383
Smith	**A**	-5.538	-5.158	-5.207	-3.85	-4.224	-4.00	-5.284	-4.973	-4.672
	B	-18.07	-16.16	-16.55	-14.49	-13.81	-13.96	-15.71	-14.52	-14.81
Halsey	**A1**	5.377	4.855	5.063	4.836	4.37	4.59	3.41	3.221	3.55
	A2	-0.8089	-0.7996	-0.7928	-0.7874	-0.7973	-0.7845	-1.047	-1.032	-1.002
Henderson	**A1**	9.732	8.802	9.188	8.916	8.133	8.497	7.478	7.055	7.645
	A2	1.575	1.55	1.53	1.461	1.483	1.452	1.854	1.813	1.752

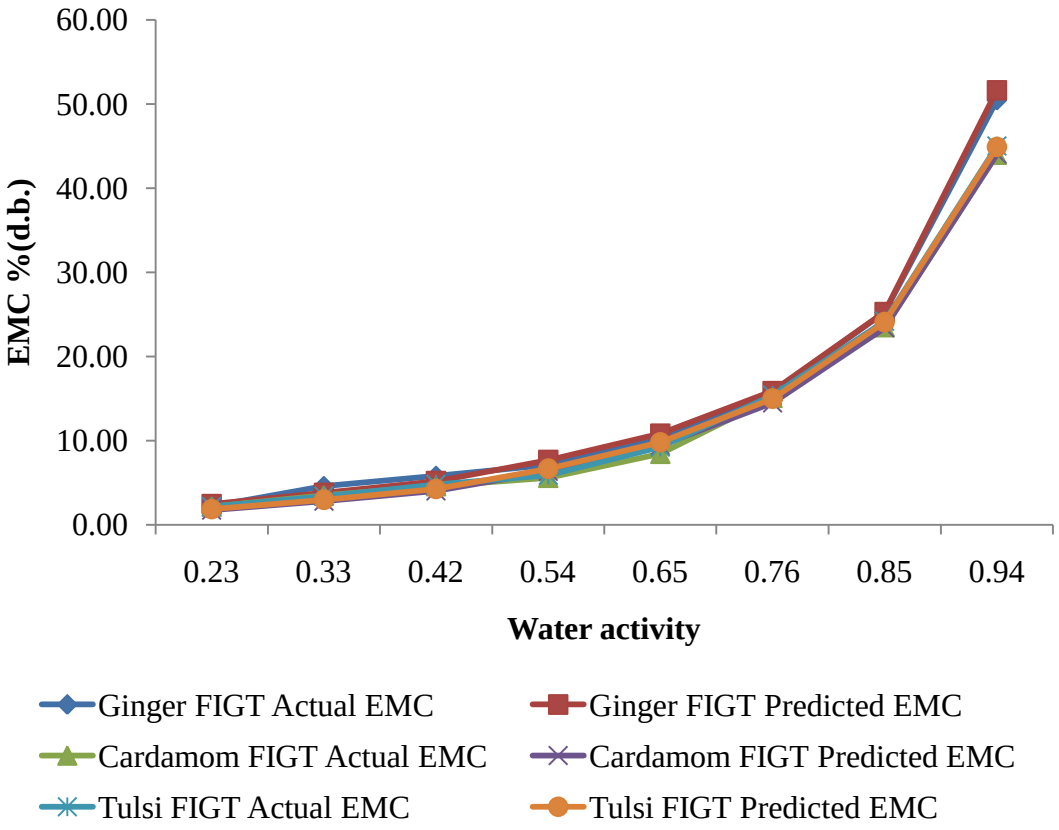


Fig. 4.55 Actual and GAB model predicted adsorption isotherms of flavoured instant green tea

4.8 PARTICLE SIZE ANALYSIS OF FLAVOURED INSTANT GREEN TEA POWDER

The particle size (nm) of optimized ginger flavoured instant green tea powder, tulsi flavoured instant green tea powder, cardamom flavoured instant green tea powder and instant green tea powder without any flavour were carried out using Zetasizer nano. Zetasizer provides the information of particle size and size distribution. The particle size and size distribution were explained through peak number, peak area and peak amplitude, which were produced by the software and were extensively used for the explanation of the particle size and size distribution. The analysis performed with Zetasizer gave the particle size and size distribution. If there is one peak, Zetasizer shows real size average of particles. If there is more than one peak Zetasizer distribution should be considered for the size of particles (Tabrizi *et al*., 2009). From the results it was observed that, particle size ginger flavoured instant green tea powder is 410 nm, , cardamom is 291 nm and tulsi flavoured instant green tea powder is 338 nm. It is evident from Fig. 4.56, particle size of all three instant flavoured green tea were comparable.

Similar results were found by Sathyashree, 2015 for spray dried sweet orange juice powder, Fazaeli *et al.* (2012) for spray dried black mulberry juice powders and Krishnaiah *et al.* (2012) for spray dried sole *Beta vulgaris* L, spray-dried sole *Morindacitrifolia.*

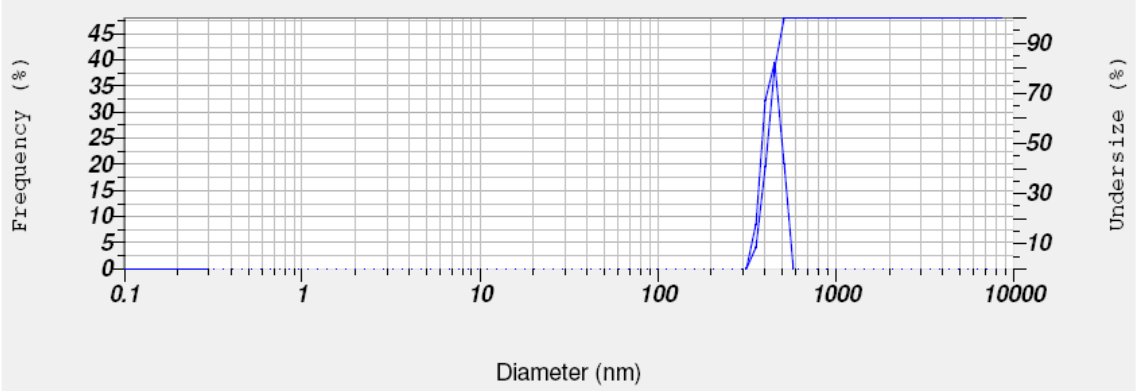


Fig. 4.56a Particle size ginger flavoured instant green tea

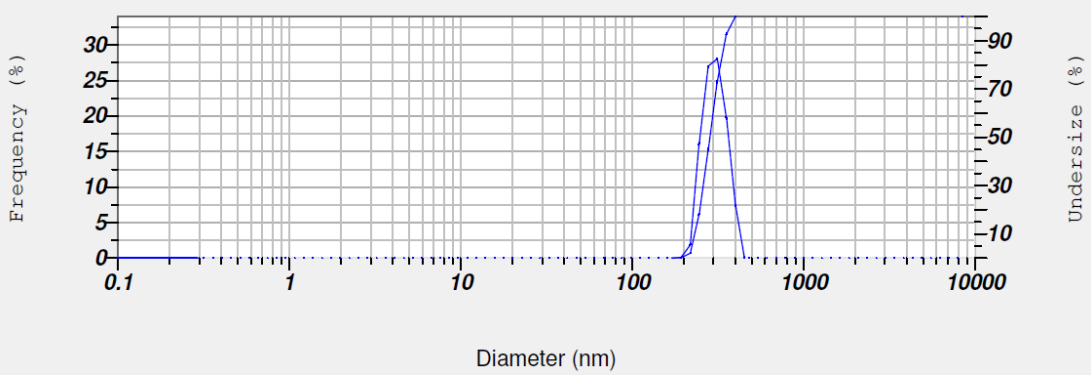


Fig. 4.56b Particle size cardamom flavoured instant green tea

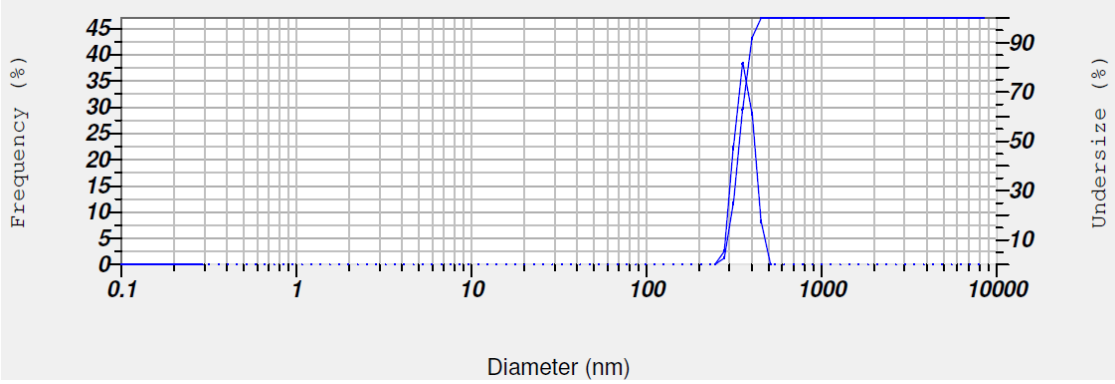


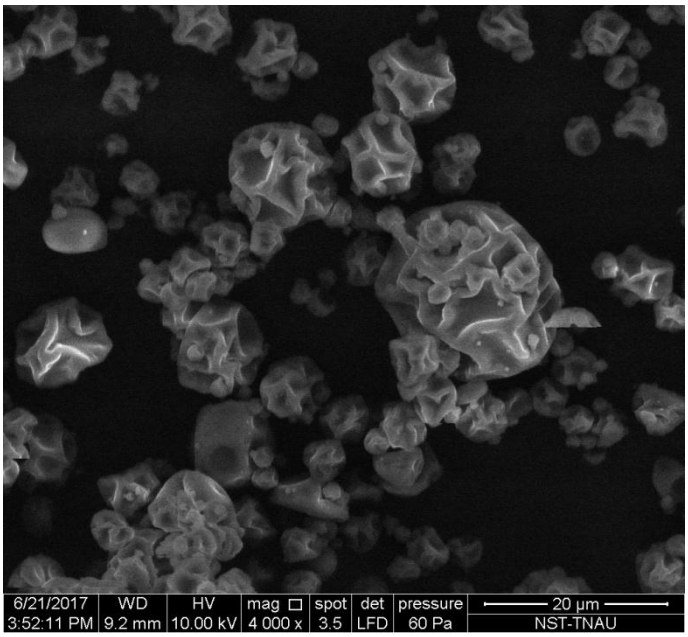
Fig. 4.56cd Particle size tulsi flavoured instant green tea

4.9 SCANNING ELECTRON MICROSCOPE (SEM) ANALYSIS OF FLAVOURED INSTANT GREEN TEA

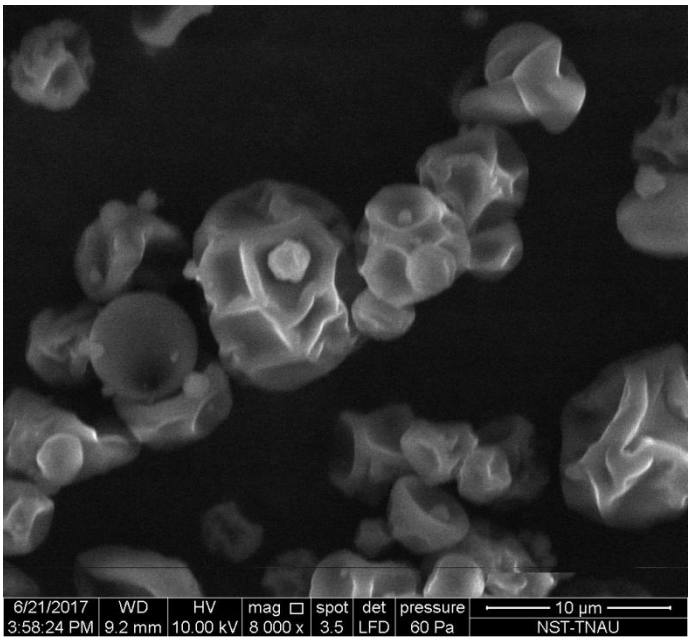
Particle morphology of three flavoured instant green tea was analysed using scanning electron microscope (SEM). Selected images from the SEM microstructure analysis of the three flavoured instant green tea powders are illustrated in Fig. 4.57 to 4.59. SEM result showed that, ginger, tulsi, cardamom flavoured instant green tea having similar morphology since it has processed under same spray drying parameters. That shows addition of flavours do not have significant effect on the morphology of the spray dried instant green tea. Spray dried flavoured instant green tea powder particles having irregular spherical shape with many shrinkages and dents on the surface. It has been reported that both the shrinkages on the surface of the particles and the expansion in the size of the particles occur due addition of the carrier materials which decrease the evaporation rate of water from the system due to their water holding capacity (Loksuwan, 2007). Similar structure were reported by Nadeem *et al*., 2011 for mountain tea green tea powder, Jafari *et al*., 2017 for spray dried pomegranate juice powder and Wilkowska *et al*., 2017 for spray dried wine powder.

4.10 X-RAY DIFFRACTION (XRD) CHARACTERISTICS OF FLAVOURED INSTANT GREEN TEA

X-ray diffraction technique is used to confirm the crystalline–amorphous state of dried products in a powder form. In general, crystalline material shows a series of sharp peaks, while amorphous product produces a broad background pattern. In present study all the three tested samples shows broad background without any crystalline peak formation. Therefore it is clear from the results, all the samples are amorphous in nature. For all the samples the amorphous peak with strong intensity is formed at about 20° (2θ). Peak formation indicates that flavour addition don't have significant effect on the amorphous nature. Fig. 4.60-4.62 shows the XRD profiles of flavoured instant green tea. The presence of amorphous material could be due to the fact that during the drying process, the material did not crystallize because the high molecular weight and high viscosity that increased the glass transition temperature, turning the surface amorphous (Cano-Chauca *et al*. 2005).

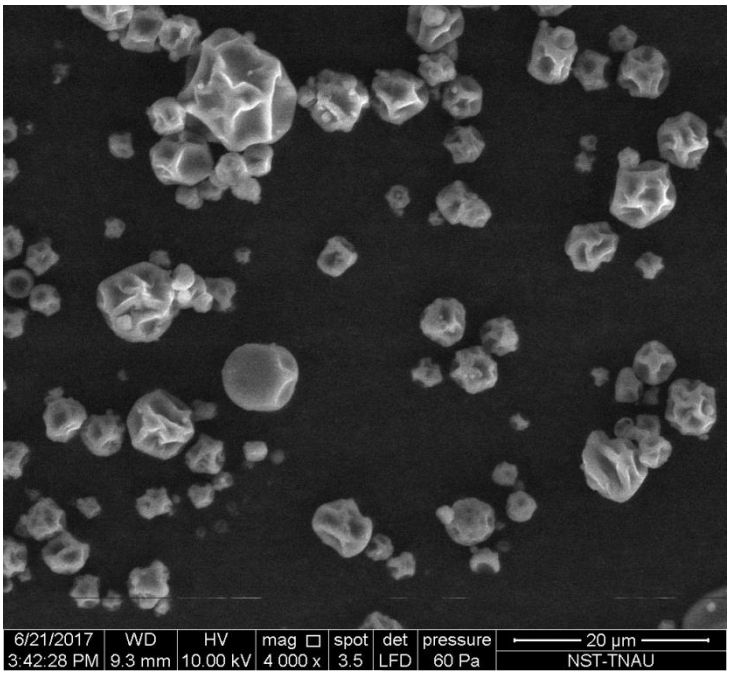


a

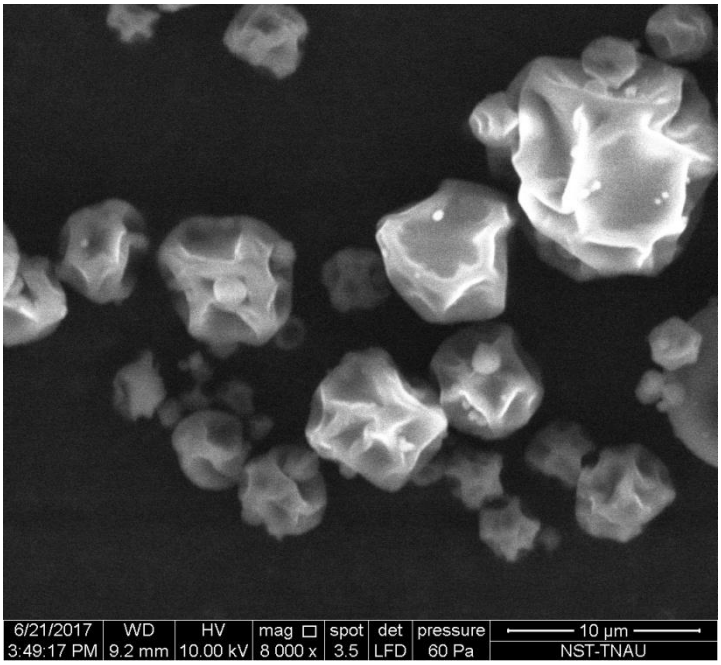


b

Fig. 4.57 SEM micrograph (a.4000×, b.8000×) of ginger flavoured instant green tea powder

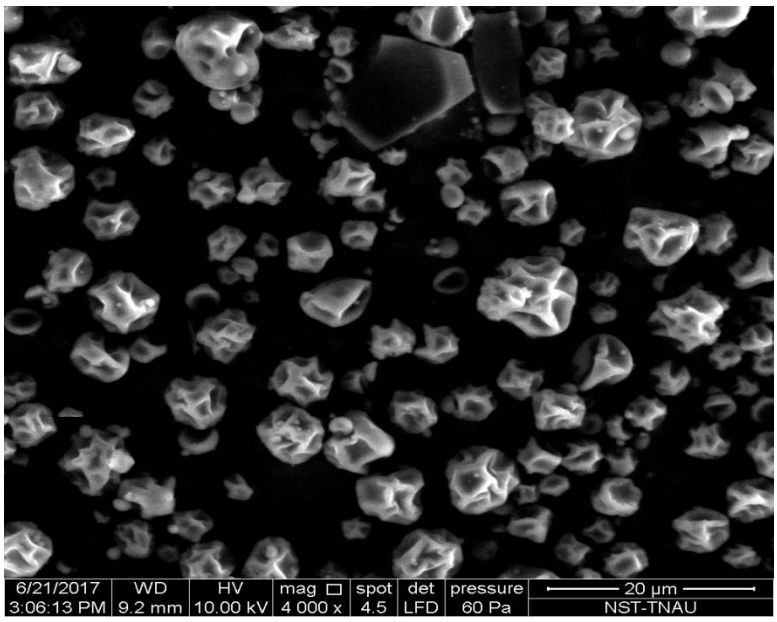


a



b

Fig. 4.58 SEM micrograph (a.4000×, b.8000×) of cardamom flavoured instant green tea powder



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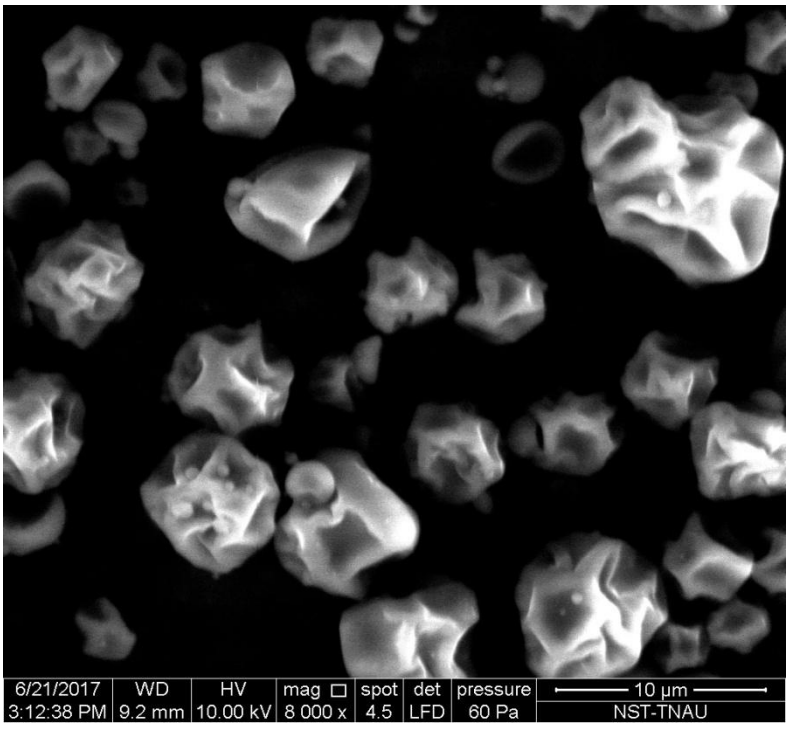

a

Fig. 4.59 SEM micrograph (a.4000×, b.8000×) of tulsi flavoured instant green tea powder

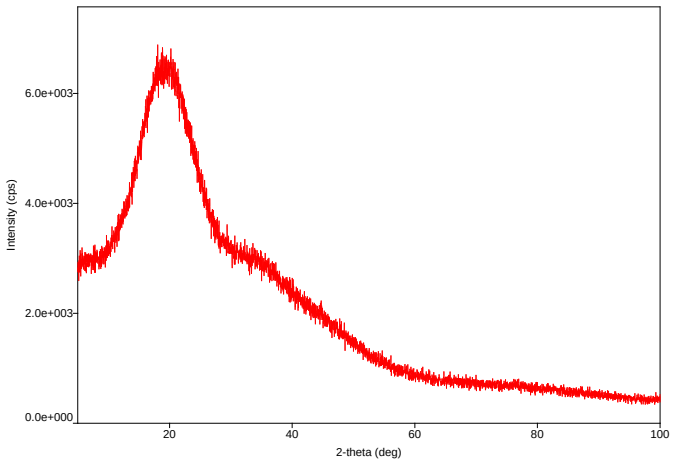

Fig. 4.60 X-ray diffraction patterns of ginger flavoured instant green tea

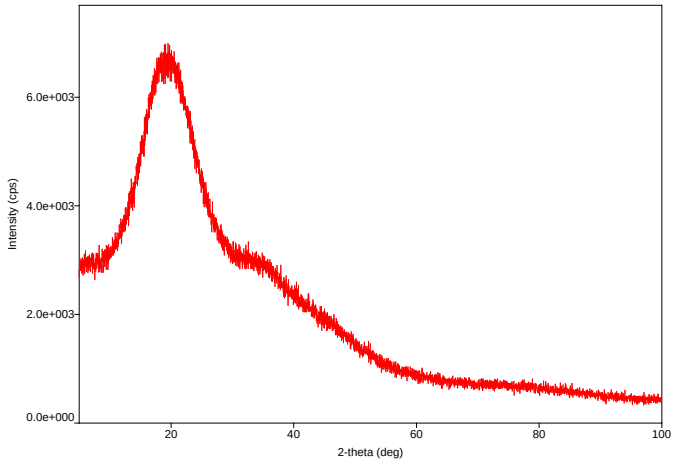

Fig. 4.61 X-ray diffraction patterns of cardamom flavoured instant green tea

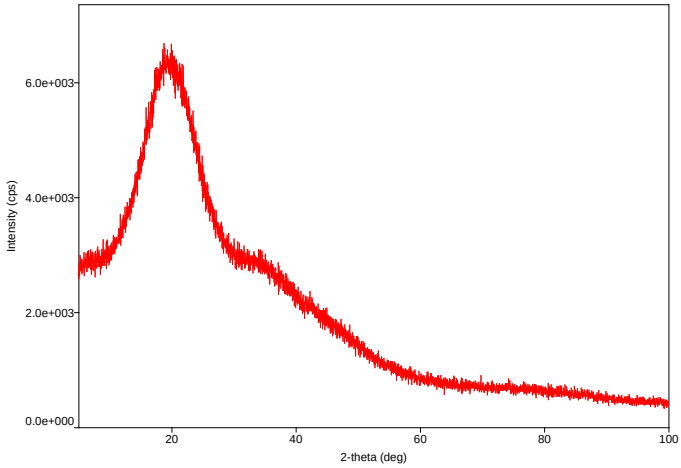

Fig. 4.62 X-ray diffraction patterns of tulsi flavoured instant green tea

Diffractogram obtained in present study is similar to the report of Caparino *et al*., 2012 for the spray dried mango powder.

4.11 IDENTIFICATION AND QUANTIFICATION OF CATECHIN

Green tea catechins of four best samples are estimated by 1SO standard procedure. Catechin fractions mainly include epigallocatechin (EGC), catechin (C), epicatechin (EC), epigallocatechin gallate (EGCG), epicatechin gallate (ECG). Catechin fraction of ginger flavoured instant green tea, tulsi instant flavoured green tea, cardamom instant flavoured green tea and optimized instant green tea without flavour is given in Table 4.15. It is clear from the Table 4.15 that all the catechines fraction except EGCG and ECG are not affected by addition of flavours. As according to the report of Wang and Zhou (2004) EGCG and ECG are susceptible to degradation than when it is used with other food material that might be the reason for negligible reduction in those components. In all the four tested samples EGCG is reported as the dominating fraction with 3.07 to 2.56% among the instant green tea. Similar values were reported by El-Shahawi *et al*., 2012 for many commercial green tea samples in a Saudi Arabian, Wang *et al*., 2003 for green tea catechin and Zhang *et al*., 2016 for green tea infusion. Obtained results contradicts the observation of Wu *et al*., 2012 and Perva-Uzunalic *et al*., 2006 for Chinese tea varieties and commercial green tea with higher catechin fraction than the present investigation, these variation might be due to variation of cultivars and processing steps. Chromatograms of four tested samples are shown in Appendix G.

Table 4.15 Catechin fraction in instant flavoured green tea

Sl. No.	Catechin fraction	Instant green tea	Ginger flavoured instant green tea	Tulsi flavoured instant green tea	Cardamom flavoured instant green tea
1	EGC (% d.b.)	2.78	2.53	2.45	2.64
2	C (% d.b.)	0.58	0.56	0.56	0.56
3	EC (% d.b.)	0.84	0.82	0.80	0.84
4	EGCG (% d.b.)	3.07	2.56	2.62	2.66
5	ECG (% d.b.)	1.04	0.93	0.87	0.96

4.12 HEAT UTILIZATION EFFICIENCY OF SPRAY DRYER FOR DRYING OF GREEN TEA EXTRACT

The heat utilization efficiency of spray dryer was evaluated in MATLAB® R2013a platform. From the results, heat utilization efficiency during the evaporation of water droplets under adiabatic condition is 89.4% and heat utilization efficiency under actual drying condition is 21.67%. The heat utilization efficiency of spray dryer under non adiabatic condition was found to be 18.18% under optimised spray drying condition. Similar values was reported by Reddy, 2013 for spray dried goat milk powder with inter temperature of 160°C. Bahnasawy *et al*. (2010) reported that the energy consumption decreased with increase in both inlet air temperature and atomization speed and dryer efficiency increased with increase in drying inlet air temperature and atomization speed. In present study low drying temperature and feed rate might be the reason for lower efficiency. MATLAB program and detail calculation for the heat utilization efficiency is given in Appendix H.

4.13 ECONOMICS OF DEVELOPED PROCESS TECHNOLOGY FOR INSTANT FLAVOURED GREEN TEA

The cost estimation for the production of instant green tea and instant flavoured green tea powder was carried out as explained in Chapter III. The equipment used for production of instant green tea powder is mainly spray drier. The production cost for kilogram of instant green tea, ginger flavoured instant green tea, cardamom flavoured instant green tea and tulsi flavoured instant green tea was Rs. 4,089, Rs 4,179, Rs 4,521 and Rs 4,127, respectively. B:C ratio of instant green tea, ginger flavoured instant green tea, cardamom flavoured instant green tea and tulsi flavoured instant green tea is 1.35, 1.43, 1.33 and 1.45 shows that the developed process technology for instant flavoured green tea is economically feasible. The detailed calculation is presented in Appendix I.

CHAPTER V
SUMMARY AND CONCLUSION

Tea (*Camellia sinensis*) is a shrub of eastern Asia having fragrant white flowers and evergreen leaves. Tea is cultivated in more than 30 countries which mainly include China, India and Japan. In India Tea is grown in 16 states, of which Assam, West Bengal, Tamil Nadu and Kerala accounted for about 96% of total tea production. Based on the processing condition there are mainly three types of tea *viz.,* black tea, green tea and oolang tea. In the produced tea 88% is converted into black tea, 10% to orthodox tea and only 2% to green tea. Due to the increased awareness of green tea health benefits, the production of green tea is growing 11% annually, while that of black tea only 3.9%.

Green tea consumption helps in preventing cardiovascular diseases, cancer, diabetes and obesity. The major chemo-preventive constituents in green tea responsible for these pharmacological effects are catechins and its constituents. During black tea processing catechins were converted into other compounds which have less effect than green tea. Recently, demand is increasing for instant tea, decaffeinated tea and flavoured tea. The use of instant tea powder will reduce the preparation time to a large extent and save energy. Commonly herbs and spices are used for flavouring food and they also enhances medicinal value. Due to the reason, efforts were undertaken for optimisation of process parameters for preparation of flavoured instant green tea. Production of flavoured instant green tea mainly include three steps, extraction, flavour addition and drying.

Extraction was performed with hot water at three different temperatures, time and leaf-water ratio. The leaf-water ratio were 1:10, 1:30 and 1:50 for extraction time of 15, 30 and 45 min at temperature of 40, 50 and 60°C.

Extract obtained with optimised condition was concentrated with three levels of maltodextrins *i.e.*, 2%, 3% and 4% then subjected to spray drying at inlet temperature of 160, 170 and 180°C with three levels of feed flow rate *i.e.,* 420, 672 and 924 $ml.h^{-1}$. Spray drying process parameters were optimised with RSM

Box- Behnken design based on quality parameters of spray dried powder which includes moisture content, yield, colour, water activity, loose bulk density, tapped bulk density, Hausner Ratio (HR), Carr's Index (CI), solubility, wettability, total polyphenols, total flavonoids and caffeine. The heat utilization efficiency of spray dryer was also evaluated using MATLAB® R2013a software for optimized spray drying condition.

Three flavours namely ginger, cardamom and tulsi were incorporated in to instant green tea flavour at different levels. Then flavour concentration was optimised based on sensory evaluation and quality of spray dried instant green tea powder in terms of total flavonoids, total polyphenols and caffeine. The sensory analysis done subjectively was ranked based on its attribute preference mathematically using fuzzy comprehensive model.

The best one sample from each of ginger, cardamom and tulsi flavoured instant green tea were packed in PET, ALF and LDPE and kept for storage studies at room temperature. The quality *viz.*, moisture content, water activity, loose bulk density, tapped bulk density, colour change, solubility, wettability, total polyphenols, total flavonoids and caffeine was tested up to six months. Prediction of shelf-life of powder was made using moisture gain and water activity.

Moisture sorption isotherms study was carried out at three temperatures and eight levels of humidity to know the stability of product during handling, processing, storage and packaging. Different sorption models were fitted to the data of sorption isotherms and GAB model was selected based on R^2, SSE and RMSE value.

Surface morphology, particle size, x-ray diffraction and catechin fraction of instant flavoured green tea powder were analysed using SEM, particle size analyser, x-ray diffractometer and HPLC, respectively.

Conclusions

- The optimum green tea extraction process conditions were 1:47 leaf-water ratio, 30 min extraction time and 52°C extraction temperature with maximum total polyphenols, total

flavonoids, caffeine, antioxidant activity of 50.26 $mg.g^{-1}$, 26.87 $mg.g^{-1}$, 19.22 $mg.g^{-1}$, 2.30 $mM.g^{-1}$ and minimum tannins content of 2.89%.

- Among the three drying methods (spray drying, freeze drying and vacuum drying) spray drying was selected for drying of green tea extract due to better colour and good sensory properties.
- The optimum process conditions for spray drying of green tea extract was 174°C inlet temperature, 2.7% of maltodextrin concentration and 671 $ml.h^{-1}$ feed rate with desirability of 0.723. At optimized condition, the maximum yield of 32.59%, solubility of 87.97, a* value of 2.581, b* value of 20.892, hue angle of 81.9°, total flavonoids of 26.67 $mg.g^{-1}$, total polyphenols of 43.18 $mg.g^{-1}$, caffeine content of 15.17 $mg.g^{-1}$ and minimum loose bulk density of 0.259 $g.cc^{-1}$, tapped bulk density of 0.338 $g.cc^{-1}$, Carr's index of 23.345, Hausner ratio of 1.305, moisture content of 3.08% (w.b.), water activity of 0.150 and wettability of 160 s was recorded.
- The heat utilization efficiency of spray dryer for spray drying of green tea extract under non adiabatic condition was 18.18% and under adiabatic condition was 89.4%.
- Fuzzy logic sensory analysis of reconstituted instant green tea powder indicated good consumer satisfaction with 1.25 g of instant green tea powder dissolved in 100 ml of hot water.
- In ginger flavoured instant green tea, G2 treatment (4% ginger extract) was selected as best combination based on sensory evaluation. The total polyphenols, total flavonoid content and caffeine content in G2 treatment was 44.79 $mg.g^{-1}$, 27.57 $mg.g^{-1}$ and 15.06 $mg.g^{-1}$, respectively.
- In cardamom flavoured instant green tea, C6 treatment (3 g of cardamom with 30 min of soaking) was selected as best combination. The total polyphenols, total flavonoid content and caffeine content of best treatment was 45.6 $mg.g^{-1}$, 26.97 $mg.g^{-1}$ and 15.14 $mg.g^{-1}$, respectively.
- Green tea extracted at 3:10 tulsi to green tea ratio for 30 min and spray dried at optimized spray drying condition was selected as best tulsi flavoured instant green tea based on

sensory evaluation and quality parameters. The total polyphenols, total flavonoid content and caffeine content of best tulsi flavoured instant green tea powder was 45.59 $mg.g^{-1}$, 28.39 $mg.g^{-1}$ and 15.11 $mg.g^{-1}$, respectively.

- After storage studies among the three packaging material, ALF maintained the quality of instant flavoured green tea. The moisture content, water activity, tapped bulk density, loose bulk density, colour change, solubility, wettability, total flavonoid, total polyphenol and caffeine content of ginger flavoured instant green tea was 7.87%, 0.374, 0.392 $g.cc^{-1}$, 0.470 $g.cc^{-1}$, 2.913, 83.210, 81 s, 37.14 $mg.100ml^{-1}$, 26.00 $mg.g^{-1}$ and 14.96 $mg.g^{-1}$, respectively after six months of storage.
- In cardamom flavoured green tea, moisture content, water activity, tapped bulk density, loose bulk density, colour change, solubility, wettability, total flavonoid, total polyphenol and caffeine was 7.68%, 0.373, 0.389 $g.cc^{-1}$, 0.467 $g.cc^{-1}$, 2.159, 85.60, 79 s, 37.16 $mg.g^{-1}$, 25.75 $mg.g^{-1}$ and 14.942 $mg.g^{-1}$, respectively after six months of storage.
- In tulsi flavoured green tea, moisture content, water activity, tapped bulk density, loose bulk density, colour change, solubility, wettability, total flavonoid, total polyphenol and caffeine content was 7.62%, 0.370, 0.391 $g.cc^{-1}$, 0.469 $g.cc^{-1}$, 2.70, 85.10, 80 s, 41.49 $mg.ml^{-1}$, 25.82 $mg.g^{-1}$ and 14.91 $mg.g^{-1}$, respectively after six months of storage.
- Predicted shelf-life period of instant flavoured green tea powder packaged in ALF, PET and LDPE based on moisture gain was found to be 210, 152 and 92 days, respectively.
- Among the six tested sorption models namely, GAB, Oswin, BET, Smith, Halsey and Henderson, the GAB model described the best fit to the experimental data with higher R^2 value and lowest SSE and RMSE value for three different temperatures.
- Particle size of instant green tea was 247 nm, ginger flavoured instant green tea powder was 410 nm, cardamom flavoured instant green tea was 291 nm and tulsi flavoured instant green tea powder was 338 nm.
- Scanning electron microscope (SEM) analysis of spray dried instant flavoured green tea powders showed that, particles had irregular spherical shape with many shrinkages and dents on the surface.

- X-ray diffraction result of spray dried instant flavoured green tea powders showed that, all the samples were amorphous in nature.
- The HPLC analysis of catechin in instant green tea, ginger, cardamom and tulsi flavoured instant green tea was 8.31%, 7.40%, 7.3% and 7.66%, respectively.
- The total cost of production for 1 kg of instant green tea was Rs. 4,089/-, ginger flavoured instant green tea was Rs. 4,179/-, cardamom flavoured instant green tea was 4,521/- and tulsi flavoured instant green tea was Rs. 4,127/- and benefit cost ratio was 1.35:1, 1.43:1, 1.33:1 and 1.45:1, respectively.

Further investigations are to be directed towards

- Granulation of powder to increase the flow characteristics
- Incorporation of powder and green tea extract in food as a preservative and functional additive
- Other technique of concentration of green tea extract before spray drying

CHAPTER VI

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APPENDIX A

Table A1 Box-Behnkan design with independent and response variables for green tea extraction

Run	water	Time	Temperature	Flavonoids	Polyphenols	Caffeine	Antioxidant	Tannins

		(min)	(°C)	(mg.g^{-1})	(mg.g^{-1})	(mg.g^{-1})	activity (mM.g^{-1})	(%)
1	10	15	50	**12.500**	28.48	6.750	1.079	**1.181**
2	50	15	50	17.000	43.93	13.500	1.968	2.300
3	10	45	50	15.000	34.2	7.150	1.180	1.729
4	50	45	50	25.000	44.93	16.950	**2.499**	3.625
5	10	30	40	14.000	32.5	4.950	**1.025**	1.390
6	50	30	40	20.500	44.5	12.350	2.267	2.778
7	10	30	60	18.000	34.35	15.870	1.395	1.792
8	50	30	60	**26.500**	50.4	**23.750**	2.464	**3.964**
9	30	15	40	13.000	34.3	**5.500**	1.343	1.635
10	30	45	40	15.500	37.83	7.950	1.535	2.147
11	30	15	60	17.000	40.98	15.250	1.698	2.475
12	30	45	60	21.500	42.45	17.750	1.870	3.798
13	30	30	50	23.000	41.8	12.000	1.615	2.042
14	30	30	50	23.400	45.86	16.980	1.734	2.192
15	30	30	50	23.900	48.79	16.950	1.751	2.213
16	30	30	50	23.300	45.86	16.920	1.632	2.063
17	30	30	50	23.800	47.96	16.970	1.768	2.235

Table A2. Analysis of variance (ANOVA) for Total polyphenols in green tea extract

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	626.21	9	69.58	15.38	0.0008	46.05	1	0.95	43.80	48.30
A-Temperature	367.61	1	367.61	81.25	< 0.0001	6.78	1	0.75	5.00	8.56
B-MD	17.17	1	17.17	3.79	0.0924	1.47	1	0.75	-0.31	3.24
C-Feed rate	45.36	1	45.36	10.03	0.0158	2.38	1	0.75	0.60	4.16
AB	5.57	1	5.57	1.23	0.3039	-1.18	1	1.06	-3.69	1.33
AC	4.10	1	4.10	0.91	0.3728	1.01	1	1.06	-1.50	3.53
BC	1.06	1	1.06	0.23	0.6430	-0.52	1	1.06	-3.03	2.00
A^2	46.15	1	46.15	10.20	0.0152	-3.31	1	1.04	-5.76	-0.86
B^2	99.38	1	99.38	21.96	0.0022	-4.86	1	1.04	-7.31	-2.41
C^2	22.39	1	22.39	4.95	0.0615	-2.31	1	1.04	-4.76	0.15
Residual	31.67	7	4.52							
Lack of Fit	2.38	3	0.79	0.11	0.9508					
Pure Error	29.29	4	7.32							
Cor Total	657.89	16								

Std. Dev.	2.1271	R-Squared	0.9519	df= degrees of freedom; SE= Standard Error
Mean	41.1247	Adj R-Squared	0.8900	Coeff. Est = Coefficient of estimate
C.V. %	5.1724	Pred R-Squared	0.8725	CI = Confidence of Interval
PRESS	83.8817	Adeq Precision	13.5763	ns= non significance

Table A3. Analysis of variance (ANOVA) for Total flavonoids in green tea extract

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	334.27	9	37.14	123.19	< 0.0001	23.48	1	0.25	22.90	24.06
A-Temperature	108.78	1	108.78	360.80	< 0.0001	3.69	1	0.19	3.23	4.15
B-MD	38.28	1	38.28	126.97	< 0.0001	2.19	1	0.19	1.73	2.65
C-Feed rate	50.00	1	50.00	165.84	< 0.0001	2.50	1	0.19	2.04	2.96
AB	7.56	1	7.56	25.08	0.0016	1.38	1	0.27	0.73	2.02
AC	1.00	1	1.00	3.32	0.1114	0.50	1	0.27	-0.15	1.15
BC	1.00	1	1.00	3.32	0.1114	0.50	1	0.27	-0.15	1.15
A^2	10.15	1	10.15	33.66	0.0007	-1.55	1	0.27	-2.19	-0.92
B^2	87.26	1	87.26	289.43	< 0.0001	-4.55	1	0.27	-5.19	-3.92
C^2	19.96	1	19.96	66.22	< 0.0001	-2.18	1	0.27	-2.81	-1.54
Residual	2.11	7	0.30							
Lack of Fit	1.56	3	0.52	3.80	0.1149					
Pure Error	0.55	4	0.14							
Cor Total	336.38	16								

Std. Dev.	0.5491	R-Squared	0.9937	df= degrees of freedom; SE= Standard Error
Mean	19.5824	Adj R-Squared	0.9857	Coeff. Est = Coefficient of estimate
C.V. %	2.8040	Pred R-Squared	0.9231	CI = Confidence of Interval
PRESS	25.8563	Adeq Precision	32.9468	ns= non significance

Table A4. Analysis of variance (ANOVA) for Caffiene in green tea extract

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	427.64	9	47.52	15.87	0.0007	15.96	1	0.77	14.13	17.79
A-Temperature	126.64	1	126.64	42.30	0.0003	3.98	1	0.61	2.53	5.43
B-MD	9.68	1	9.68	3.23	0.1152	1.10	1	0.61	-0.35	2.55
C-Feed rate	219.14	1	219.14	73.20	< 0.0001	5.23	1	0.61	3.79	6.68
AB	2.33	1	2.33	0.78	0.4074	0.76	1	0.87	-1.28	2.81
AC	0.06	1	0.06	0.02	0.8936	0.12	1	0.87	-1.93	2.17
BC	0.00	1	0.00	0.00	0.9889	0.01	1	0.87	-2.03	2.06
A^2	5.37	1	5.37	1.79	0.2223	-1.13	1	0.84	-3.12	0.86
B^2	59.12	1	59.12	19.75	0.0030	-3.75	1	0.84	-5.74	-1.75
C^2	1.54	1	1.54	0.51	0.4966	-0.60	1	0.84	-2.60	1.39
Residual	20.96	7	2.99							
Lack of Fit	1.31	3	0.44	0.09	0.9623					
Pure Error	19.64	4	4.91							
Cor Total	448.59	16								

Std. Dev.	1.7302	R-Squared	0.9533	df= degrees of freedom; SE= Standard Error
Mean	13.3847	Adj R-Squared	0.8932	Coeff. Est = Coefficient of estimate
C.V. %	12.9269	Pred R-Squared	0.8848	CI = Confidence of Interval
PRESS	51.6849	Adeq Precision	13.8845	ns= non significance

Table A5. Analysis of variance (ANOVA) for Antioxidant activity in green tea extract

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	2.99	9	0.33	73.43	< 0.0001	1.70	1	0.03	1.63	1.77
A-Temperature	2.55	1	2.55	563.80	< 0.0001	0.56	1	0.02	0.51	0.62
B-MD	0.12	1	0.12	25.50	0.0015	0.12	1	0.02	0.06	0.18
C-Feed rate	0.21	1	0.21	46.11	0.0003	0.16	1	0.02	0.11	0.22
AB	0.04	1	0.04	8.59	0.0220	0.10	1	0.03	0.02	0.18
AC	0.00	1	0.00	1.06	0.3373	-0.03	1	0.03	-0.11	0.04
BC	0.00	1	0.00	0.02	0.8821	-0.01	1	0.03	-0.08	0.07
A^2	0.03	1	0.03	5.82	0.0466	0.08	1	0.03	0.00	0.16
B^2	0.05	1	0.05	10.49	0.0143	-0.11	1	0.03	-0.18	-0.03
C^2	0.00	1	0.00	0.29	0.6066	0.02	1	0.03	-0.06	0.10
Residual	0.03	7	0.00							
Lack of Fit	0.01	3	0.00	0.76	0.5741					
Pure Error	0.02	4	0.01							
Cor Total	3.02	16								

Std. Dev.	0.0673	R-Squared	0.9895	df= degrees of freedom; SE= Standard Error
Mean	1.6955	Adj R-Squared	0.9760	Coeff. Est = Coefficient of estimate
C.V. %	3.9687	Pred R-Squared	0.9289	CI = Confidence of Interval
PRESS	0.2151	Adeq Precision	28.1515	ns= non significance

Table A6. Analysis of variance (ANOVA) for Tannins in green tea extract

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	10.12	9	1.12	45.92	< 0.0001	2.15	1	0.07	1.98	2.31
A-Temperature	5.40	1	5.40	220.67	< 0.0001	0.82	1	0.06	0.69	0.95
B-MD	1.72	1	1.72	70.18	< 0.0001	0.46	1	0.06	0.33	0.59
C-Feed rate	2.08	1	2.08	84.93	< 0.0001	0.51	1	0.06	0.38	0.64
AB	0.15	1	0.15	6.16	0.0420	0.19	1	0.08	0.01	0.38
AC	0.15	1	0.15	6.27	0.0407	0.20	1	0.08	0.01	0.38
BC	0.16	1	0.16	6.71	0.0359	0.20	1	0.08	0.02	0.39
A^2	0.00	1	0.00	0.03	0.8645	0.01	1	0.08	-0.17	0.19
B^2	0.01	1	0.01	0.37	0.5634	0.05	1	0.08	-0.13	0.23
C^2	0.43	1	0.43	17.44	0.0042	0.32	1	0.08	0.14	0.50
Residual	0.17	7	0.02							
Lack of Fit	0.14	3	0.05	5.77	0.0618					
Pure Error	0.03	4	0.01							
Cor Total	10.29	16								

Std. Dev.	0.1565	R-Squared	0.9833	df= degrees of freedom; SE= Standard Error
Mean	2.3270	Adj R-Squared	0.9619	Coeff. Est = Coefficient of estimate
C.V. %	6.7249	Pred R-Squared	0.7786	CI = Confidence of Interval
PRESS	2.2780	Adeq Precision	24.0884	ns= non significance

Table A7 Response optimization constraints of green tea extraction

Sl. No.	Parameters	Goal	Lower limit	Upper limit	Importance
1	Water	In range	10	50	3
2	Time of extraction	In range	15	45	3
3	Temperature	In range	40	60	3
4	Total polyphenols	Maximize	28.48	50.4	3
5	Total flavonoids	Maximize	12.5	26.5	3
6	Caffiene	Maximize	4.95	23.75	3
7	Antiaxidant activity	Maximize	1.0254	2.4987	3
8	Tannins	Minimize	1.181	3.964	3

APPENDIX B

Table B1 Effect of spray drying processing conditions on yield of instant green tea powder

Run	Temperature (°C)	MD conc. (%)	Feed flow rate ($ml.h^{-1}$)	Yield (%)
1	160	2	672	27.333
2	180	2	672	28.917
3	160	4	672	31.591
4	180	4	672	34.27
5	160	3	420	27.647
6	180	3	420	30.706
7	160	3	924	27.235
8	180	3	924	27.35
9	170	2	420	30
10	170	4	420	35.727
11	170	2	924	29
12	170	4	924	33.636
13	170	3	672	35.529
14	170	3	672	35.09
15	170	3	672	34.588
16	170	3	672	33.353
17	170	3	672	35.294

Table B2 Analysis of variance (ANOVA) for Yield of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	756.74	9	84.08	17.40	0.0005	40.52	1	0.98	38.20	42.85
A-Temperature	12.87	1	12.87	2.66	0.1467	1.27	1	0.78	-0.57	3.11
B-MD	13.90	1	13.90	2.88	0.1337	1.32	1	0.78	-0.52	3.16
C-Feed rate	179.13	1	179.13	37.07	0.0005	-4.73	1	0.78	-6.57	-2.89
AB	3.42	1	3.42	0.71	0.4281	0.92	1	1.10	-1.67	3.52
AC	3.88	1	3.88	0.80	0.3998	-0.99	1	1.10	-3.58	1.61
BC	42.84	1	42.84	8.87	0.0206	-3.27	1	1.10	-5.87	-0.67
A^2	57.98	1	57.98	12.00	0.0105	-3.71	1	1.07	-6.24	-1.18
B^2	7.73	1	7.73	1.60	0.2463	-1.36	1	1.07	-3.89	1.18
C^2	406.41	1	406.41	84.09	< 0.0001	-9.82	1	1.07	-12.36	-7.29
Residual	33.83	7	4.83							
Lack of Fit	24.00	3	8.00	3.25	0.1421					
Pure Error	9.83	4	2.46							
Cor Total	790.57	16								

Std. Dev.	2.1983	R-Squared	0.9572	df= degrees of freedom; SE= Standard Error
Mean	33.5132	Adj R-Squared	0.9022	Coeff. Est = Coefficient of estimate
C.V. %	6.5596	Pred R-Squared	0.4949	CI = Confidence of Interval
PRESS	399.2891	Adeq Precision	11.0023	ns= non significance

Table B3 Effect of spray drying processing conditions on biochemical properties of instant green tea powder

Run	Temperature (°C)	MD conc. (%)	Feed flow rate ($ml.h^{-1}$)	Flavonoids ($mg.g^{-1}$)	Polyphenols ($mg.g^{-1}$)	Caffeine ($mg.g^{-1}$)
1	160	2	672	25.00	52.00	18.32
2	180	2	672	27.60	40.00	15.69
3	160	4	672	21.90	37.00	17.46
4	180	4	672	23.78	27.00	14.62
5	160	3	420	19.80	38.00	20.92
6	180	3	420	22.34	28.00	15.22
7	160	3	924	26.00	54.00	17.28
8	180	3	924	27.86	42.00	14.06
9	170	2	420	23.00	40.00	16.92
10	170	4	420	18.78	28.00	15.8
11	170	2	924	28.34	59.00	15.18
12	170	4	924	23.64	38.00	14.44
13	170	3	672	24.76	42.00	15.04
14	170	3	672	24.42	48.00	15.29
15	170	3	672	25.43	46.00	16.44
16	170	3	672	23.34	40.00	15.25
17	170	3	672	24.34	44.00	16.4

Table B4 Analysis of variance (ANOVA) for Total flavonoids content of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High	VIF
Model	106.78	9	11.86	29.15	< 0.0001	24.46	1	0.29	23.78	25.13	
ATemperature	8.36	1	8.36	20.55	0.0027	1.02	1	0.23	0.49	1.56	1.00
B-MD	34.20	1	34.20	84.01	< 0.0001	-2.07	1	0.23	-2.60	-1.53	1.00
C-Feed rate	60.06	1	60.06	147.55	< 0.0001	2.74	1	0.23	2.21	3.27	1.00
AB	0.00	1	0.00	0.00	0.9879	-0.01	1	0.32	-0.76	0.75	1.00
AC	0.12	1	0.12	0.28	0.6106	-0.17	1	0.32	-0.92	0.58	1.00
BC	0.06	1	0.06	0.14	0.7179	-0.12	1	0.32	-0.87	0.63	1.00
A^2	0.76	1	0.76	1.86	0.2154	0.42	1	0.31	-0.31	1.16	1.01
B^2	0.08	1	0.08	0.19	0.6739	-0.14	1	0.31	-0.87	0.60	1.01
C^2	3.27	1	3.27	8.04	0.0252	-0.88	1	0.31	-1.62	-0.15	1.01
Residual	2.85	7	0.41								
Lack of Fit	0.55	3	0.18	0.32	0.8134						
Pure Error	2.30	4	0.58								
Cor Total	109.63	16									

Std. Dev.	0.638	R-Squared	0.9740	df= degrees of freedom; SE= Standard Error
Mean	24.178	Adj R-Squared	0.9406	Coeff. Est = Coefficient of estimate
C.V. %	2.639	Pred R-Squared	0.8872	CI = Confidence of Interval
PRESS	12.365	Adeq Precision	19.6493	ns= non significance

Table B5 Analysis of variance (ANOVA) for Total polyphenols content of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	1224.63	9	136.07	22.02	0.0002	44.00	1	1.11	41.37	46.63
A-Temperature	242.00	1	242.00	39.17	0.0004	-5.50	1	0.88	-7.58	-3.42
B-MD	465.13	1	465.13	75.28	< 0.0001	-7.63	1	0.88	-9.70	-5.55
C-Feed rate	435.13	1	435.13	70.42	< 0.0001	7.38	1	0.88	5.30	9.45
AB	1.00	1	1.00	0.16	0.6995	0.50	1	1.24	-2.44	3.44
AC	1.00	1	1.00	0.16	0.6995	-0.50	1	1.24	-3.44	2.44
BC	20.25	1	20.25	3.28	0.1132	-2.25	1	1.24	-5.19	0.69
A^2	34.80	1	34.80	5.63	0.0494	-2.88	1	1.21	-5.74	-0.01
B^2	19.01	1	19.01	3.08	0.1228	-2.13	1	1.21	-4.99	0.74
C^2	1.64	1	1.64	0.27	0.6218	-0.63	1	1.21	-3.49	2.24
Residual	43.25	7	6.18							
Lack of Fit	3.25	3	1.08	0.11	0.9508					
Pure Error	40.00	4	10.00							
Cor Total	1267.88	16								

Std. Dev.	2.4857	R-Squared	0.9659	df= degrees of freedom; SE= Standard Error
Mean	41.3529	Adj R-Squared	0.9220	Coeff. Est = Coefficient of estimate
C.V. %	6.0109	Pred R-Squared	0.9097	CI = Confidence of Interval
PRESS	114.500	Adeq Precision	16.8509	ns= non significance

Table B6. Analysis of variance (ANOVA) for total caffeine content of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	42.05	9	4.67	8.86	0.0044	15.68	1	0.32	14.92	16.45
A-Temperature	25.88	1	25.88	49.07	0.0002	-1.80	1	0.26	-2.41	-1.19
B-MD	1.80	1	1.80	3.40	0.1076	-0.47	1	0.26	-1.08	0.13
C-Feed rate	7.80	1	7.80	14.79	0.0063	-0.99	1	0.26	-1.59	-0.38
AB	0.01	1	0.01	0.02	0.8891	-0.05	1	0.36	-0.91	0.81
AC	1.54	1	1.54	2.92	0.1315	0.62	1	0.36	-0.24	1.48
BC	0.04	1	0.04	0.07	0.8012	0.10	1	0.36	-0.76	0.95
A^2	4.75	1	4.75	9.00	0.0199	1.06	1	0.35	0.22	1.90
B^2	0.21	1	0.21	0.40	0.5482	-0.22	1	0.35	-1.06	0.61
C^2	0.07	1	0.07	0.12	0.7359	0.12	1	0.35	-0.71	0.96
Residual	3.69	7	0.53							
Lack of Fit	1.85	3	0.62	1.34	0.3800					
Pure Error	1.84	4	0.46							
Cor Total	45.75	16								

Std. Dev.	0.7263	R-Squared	0.9193	df= degrees of freedom; SE= Standard Error
Mean	16.1371	Adj R-Squared	0.8155	Coeff. Est = Coefficient of estimate
C.V. %	4.5006	Pred R-Squared	0.2901	CI = Confidence of Interval
PRESS	32.4737	Adeq Precision	10.9130	ns= non significance

Table B7 Effect of spray drying processing conditions on colour value of instant green tea powder

Run	Temperature (°C)	MD conc. (%)	Feed flow rate ($ml.h^{-1}$)	L*	a*	b*	Hue (°)
1	160	2	672	84.94	2.63	18.99	82.13
2	180	2	672	81.71	2.83	22.07	82.71
3	160	4	672	88.15	2.17	15.87	82.23
4	180	4	672	84.37	2.47	20.7	83.21
5	160	3	420	86.67	2.54	18.19	82.06
6	180	3	420	82.97	2.80	21.86	82.71
7	160	3	924	87.08	2.18	16.34	82.41
8	180	3	924	84.25	2.44	18.29	82.41
9	170	2	420	82.87	2.82	22.56	82.89
10	170	4	420	85.26	2.53	18.33	82.16
11	170	2	924	84.83	2.67	19.25	82.12
12	170	4	924	87.17	2.10	15.25	82.18
13	170	3	672	84.79	2.41	18.9	82.75
14	170	3	672	83.9	2.42	18.93	82.73
15	170	3	672	84.47	2.43	19.69	82.98
16	170	3	672	83.5	2.34	19.36	83.12
17	170	3	672	82.9	2.34	19.28	82.09

Table B8 Analysis of variance (ANOVA) for L* value of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	45.85	9	5.09	11.65	0.0019	83.91	1	0.30	83.21	84.61
A-Temperature	22.92	1	22.92	52.40	0.0002	-1.69	1	0.23	-2.25	-1.14
B-MD	14.05	1	14.05	32.11	0.0008	1.33	1	0.23	0.77	1.88
C-Feed rate	3.86	1	3.86	8.84	0.0207	0.70	1	0.23	0.14	1.25
AB	0.08	1	0.08	0.17	0.6900	-0.14	1	0.33	-0.92	0.64
AC	0.19	1	0.19	0.43	0.5317	0.22	1	0.33	-0.56	1.00
BC	0.00	1	0.00	0.00	0.9709	-0.01	1	0.33	-0.79	0.77
A^2	1.25	1	1.25	2.86	0.1345	0.55	1	0.32	-0.22	1.31
B^2	0.47	1	0.47	1.08	0.3328	0.34	1	0.32	-0.43	1.10
C^2	2.60	1	2.60	5.94	0.0450	0.79	1	0.32	0.02	1.55
Residual	3.06	7	0.44							
Lack of Fit	0.79	3	0.26	0.46	0.7252					
Pure Error	2.28	4	0.57							
Cor Total	48.91	16								

Std. Dev.	0.6613	R-Squared	0.9374	df= degrees of freedom; SE= Standard Error
Mean	84.6959	Adj R-Squared	0.8569	Coeff. Est = Coefficient of estimate
C.V. %	0.7808	Pred R-Squared	0.6704	CI = Confidence of Interval
PRESS	16.1215	Adeq Precision	11.8981	ns= non significance

Table B9 Analysis of variance (ANOVA) for a* value of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	0.78	9.00	0.09	57.11	< 0.0001	2.39	1.00	0.02	2.35	2.43
A-Temperature	0.13	1.00	0.13	86.04	< 0.0001	0.13	1.00	0.01	0.09	0.16
B-MD	0.35	1.00	0.35	233.42	< 0.0001	-0.21	1.00	0.01	-0.24	-0.18
C-Feed rate	0.21	1.00	0.21	139.77	< 0.0001	-0.16	1.00	0.01	-0.20	-0.13
AB	0.00	1.00	0.00	1.65	0.2393	0.03	1.00	0.02	-0.02	0.07
AC	0.00	1.00	0.00	0.00	1.0000	0.00	1.00	0.02	-0.05	0.05
BC	0.02	1.00	0.02	12.97	0.0087	-0.07	1.00	0.02	-0.12	-0.02
A^2	0.01	1.00	0.01	6.55	0.0376	0.05	1.00	0.02	0.00	0.09
B^2	0.03	1.00	0.03	21.82	0.0023	0.09	1.00	0.02	0.04	0.13
C^2	0.01	1.00	0.01	7.97	0.0256	0.05	1.00	0.02	0.01	0.10
Residual	0.01	7.00	0.00							
Lack of Fit	0.00	3.00	0.00	0.46	0.7271					
Pure Error	0.01	4.00	0.00							
Cor Total	0.7399	16								

Std. Dev.	0.0389	R-Squared	0.9866	df= degrees of freedom; SE= Standard Error
Mean	2.4776	Adj R-Squared	0.9693	Coeff. Est = Coefficient of estimate
C.V. %	1.5691	Pred R-Squared	0.9295	CI = Confidence of Interval
PRESS	0.0555	Adeq Precision	25.1531	ns= non significance

Table B10 Analysis of variance (ANOVA) for b* value of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	63.48	9	7.05	16.73	0.0006	19.23	1	0.29	18.55	19.92
A-Temperature	22.88	1	22.88	54.27	0.0002	1.69	1	0.23	1.15	2.23
B-MD	20.22	1	20.22	47.97	0.0002	-1.59	1	0.23	-2.13	-1.05
C-Feed rate	17.43	1	17.43	41.35	0.0004	-1.48	1	0.23	-2.02	-0.93
AB	0.77	1	0.77	1.82	0.2198	0.44	1	0.32	-0.33	1.21
AC	0.74	1	0.74	1.75	0.2270	-0.43	1	0.32	-1.20	0.34
BC	0.01	1	0.01	0.03	0.8644	0.06	1	0.32	-0.71	0.83
A^2	0.00	1	0.00	0.00	0.9976	0.00	1	0.32	-0.75	0.75
B^2	0.13	1	0.13	0.31	0.5944	0.18	1	0.32	-0.57	0.92
C^2	1.33	1	1.33	3.14	0.1195	-0.56	1	0.32	-1.31	0.19
Residual	2.95	7	0.42							
Lack of Fit	2.52	3	0.84	7.82	0.0378					
Pure Error	0.43	4	0.11							
Cor Total	66.43	16								

Std. Dev.	0.6493	R-Squared	0.9556	df= degrees of freedom; SE= Standard Error
Mean	19.0506	Adj R-Squared	0.8984	Coeff. Est = Coefficient of estimate
C.V. %	3.4085	Pred R-Squared	0.3826	CI = Confidence of Interval
PRESS	41.0169	Adeq Precision	13.2099	ns= non significance

Table B11 Analysis of variance (ANOVA) for Hue value of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	1.97	9	0.22	2.90	0.0871	82.94	1	0.12	82.65	83.23
A-Temperature	0.61	1	0.61	8.09	0.0249	0.28	1	0.10	0.05	0.51
B-MD	0.00	1	0.00	0.01	0.9246	-0.01	1	0.10	-0.24	0.22
C-Feed rate	0.06	1	0.06	0.82	0.3956	-0.09	1	0.10	-0.32	0.14
AB	0.04	1	0.04	0.54	0.4865	0.10	1	0.14	-0.22	0.43
AC	0.11	1	0.11	1.40	0.2758	-0.16	1	0.14	-0.49	0.16
BC	0.16	1	0.16	2.06	0.1939	0.20	1	0.14	-0.13	0.52
A^2	0.09	1	0.09	1.23	0.3037	-0.15	1	0.13	-0.47	0.17
B^2	0.20	1	0.20	2.63	0.1488	-0.22	1	0.13	-0.53	0.10
C^2	0.62	1	0.62	8.19	0.0243	-0.38	1	0.13	-0.70	-0.07
Residual	0.53	7	0.08							
Lack of Fit	0.39	3	0.13	3.70	0.1192					
Pure Error	0.14	4	0.03							
Cor Total	2.50	16								

Std. Dev.	0.2748	R-Squared	0.7886	df= degrees of freedom; SE= Standard Error
Mean	82.5833	Adj R-Squared	0.5168	Coeff. Est = Coefficient of estimate
C.V. %	0.3328	Pred R-Squared	-1.5741	CI = Confidence of Interval
PRESS	6.4372	Adeq Precision	4.1963	ns= non significance

Table B12 Effect of spray drying processing conditions on functional properties of instant green tea powder

Run	Temp. (°C)	MD conc. (%)	Feed flow rate ($ml.h^{-1}$)	Moisture (% w.b.)	Water activity	Tapped bulk density	Loose bulk density
1	160	2	672	8.320	0.355	0.412	0.265
2	180	2	672	5.049	0.217	0.324	0.243
3	160	4	672	6.196	0.263	0.43	0.283
4	180	4	672	3.243	0.132	0.336	0.261
5	160	3	420	6.440	0.282	0.41	0.268
6	180	3	420	3.724	0.158	0.324	0.246
7	160	3	924	8.160	0.355	0.426	0.288
8	180	3	924	5.480	0.233	0.336	0.264
9	170	2	420	5.190	0.214	0.331	0.244
10	170	4	420	3.480	0.145	0.342	0.263
11	170	2	924	7.224	0.312	0.355	0.264
12	170	4	924	5.631	0.232	0.362	0.278
13	170	3	672	3.272	0.139	0.35	0.265
14	170	3	672	3.323	0.141	0.345	0.258
15	170	3	672	3.475	0.151	0.343	0.259
16	170	3	672	3.290	0.141	0.34	0.255
17	170	3	672	3.480	0.147	0.341	0.257

Table B13 Analysis of variance (ANOVA) for Moisture content of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	50.27	9	5.59	163.60	< 0.0001	3.37	1	0.08	3.17	3.56
A-Temperature	16.88	1	16.88	494.35	< 0.0001	-1.45	1	0.07	-1.61	-1.30
B-MD	6.54	1	6.54	191.54	< 0.0001	-0.90	1	0.07	-1.06	-0.75
C-Feed rate	7.34	1	7.34	214.88	< 0.0001	0.96	1	0.07	0.80	1.11
AB	0.03	1	0.03	0.74	0.4180	0.08	1	0.09	-0.14	0.30
AC	0.00	1	0.00	0.01	0.9251	0.01	1	0.09	-0.21	0.23
BC	0.00	1	0.00	0.10	0.7608	0.03	1	0.09	-0.19	0.25
A^2	8.88	1	8.88	259.96	< 0.0001	1.45	1	0.09	1.24	1.66
B^2	3.28	1	3.28	95.96	< 0.0001	0.88	1	0.09	0.67	1.10
C^2	5.39	1	5.39	157.79	< 0.0001	1.13	1	0.09	0.92	1.34
Residual	0.24	7	0.03							
Lack of Fit	0.20	3	0.07	6.38	0.0527					
Pure Error	0.04	4	0.01							
Cor Total	50.51	16								

Std. Dev.	0.1848	R-Squared	0.9953	df= degrees of freedom; SE= Standard Error
Mean	4.9986	Adj R-Squared	0.9892	Coeff. Est = Coefficient of estimate
C.V. %	3.6965	Pred R-Squared	0.9361	CI = Confidence of Interval
PRESS	3.2273	Adeq Precision	35.1698	ns= non significance

Table B14 Analysis of variance (ANOVA) for Water activity of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	0.09596	9	0.010662	170.2465	< 0.0001	0.143582	1	0.003539	0.135213	0.151951
A-Temperature	0.033089	1	0.033089	528.3484	< 0.0001	-0.06431	1	0.002798	-0.07093	-0.0577
B-MD	0.013244	1	0.013244	211.4677	< 0.0001	-0.04069	1	0.002798	-0.0473	-0.03407
C-Feed rate	0.013872	1	0.013872	221.4987	< 0.0001	0.041641	1	0.002798	0.035025	0.048257
AB	1.06E-05	1	1.06E-05	0.169357	0.6930	0.001628	1	0.003957	-0.00773	0.010985
AC	1.2E-06	1	1.2E-06	0.019131	0.8939	0.000547	1	0.003957	-0.00881	0.009904
BC	2.53E-05	1	2.53E-05	0.404596	0.5450	-0.00252	1	0.003957	-0.01187	0.00684
A^2	0.017633	1	0.017633	281.547	< 0.0001	0.064713	1	0.003857	0.055593	0.073833
B^2	0.004727	1	0.004727	75.47059	< 0.0001	0.033505	1	0.003857	0.024385	0.042624
C^2	0.009906	1	0.009906	158.1738	< 0.0001	0.048505	1	0.003857	0.039385	0.057624
Residual	0.000438	7	6.26E-05							
Lack of Fit	0.000342	3	0.000114	4.761256	0.0829					
Pure Error	9.59E-05	4	2.4E-05							
Cor Total	0.096398	16								

Std. Dev.	0.0079	R-Squared	0.9955	df= degrees of freedom; SE= Standard Error	
Mean	0.2126	Adj R-Squared	0.9896	Coeff. Est = Coefficient of estimate	
C.V. %	3.7219	Pred R-Squared	0.9416	CI = Confidence of Interval	
PRESS	0.0056	Adeq Precision	36.8689	ns= non significance	

Table B15 Analysis of variance (ANOVA) for Tapped bulk density of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	0.020646	9	0.002294	136.8952	< 0.0001	0.3438	1	0.001831	0.339471	0.348129
A-Temperature	0.016021	1	0.016021	956.0401	< 0.0001	-0.0448	1	0.001447	-0.04817	-0.04133
B-MD	0.000288	1	0.000288	17.1867	0.0043	0.006	1	0.001447	0.002578	0.009422
C-Feed rate	0.000648	1	0.000648	38.67008	0.0004	0.009	1	0.001447	0.005578	0.012422
AB	9E-06	1	9E-06	0.537084	0.4875	-0.0015	1	0.002047	-0.00634	0.00334
AC	4E-06	1	4E-06	0.238704	0.6401	-0.001	1	0.002047	-0.00584	0.00384
BC	4E-06	1	4E-06	0.238704	0.6401	-0.001	1	0.002047	-0.00584	0.00384
A^2	0.003566	1	0.003566	212.7759	< 0.0001	0.0291	1	0.001995	0.024383	0.033817
B^2	2.85E-05	1	2.85E-05	1.698569	0.2337	0.0026	1	0.001995	-0.00212	0.007317
C^2	5.09E-06	1	5.09E-06	0.304034	0.5985	0.0011	1	0.001995	-0.00362	0.005817
Residual	0.000117	7	1.68E-05							
Lack of Fit	5.45E-05	3	1.82E-05	1.157113	0.4290					
Pure Error	6.28E-05	4	1.57E-05							
Cor Total	0.020763	16								

Std. Dev.	0.0041	R-Squared	0.9944	df= degrees of freedom; SE= Standard Error
Mean	0.3592	Adj R-Squared	0.9871	Coeff. Est = Coefficient of estimate
C.V. %	1.1395	Pred R-Squared	0.9533	CI = Confidence of Interval
PRESS	0.0010	Adeq Precision	34.2399	ns= non significance

Table B16 Analysis of variance (ANOVA) for Loose bulk density of spray dried instant green tea

Source	Sum of Squares	df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	0.002446	9	0.000272	83.61	< 0.0001	0.258	1	0.000806	0.256094	0.259906
A-Temperature	0.001013	1	0.001013	311.54	< 0.0001	-0.01125	1	0.000637	-0.01276	-0.00974
B-MD	0.000595	1	0.000595	183.12	< 0.0001	0.008625	1	0.000637	0.007118	0.010132
C-Feed rate	0.000666	1	0.000666	204.96	< 0.0001	0.009125	1	0.000637	0.007618	0.010632
AB	0	1	0	0.00	1.0000	0	1	0.000901	-0.00213	0.002131
AC	1E-06	1	1E-06	0.31	0.5964	-0.0005	1	0.000901	-0.00263	0.001631
BC	6.25E-06	1	6.25E-06	1.92	0.2081	-0.00125	1	0.000901	-0.00338	0.000881
A^2	9.01E-05	1	9.01E-05	27.71	0.0012	0.004625	1	0.000879	0.002548	0.006702
B^2	5.92E-07	1	5.92E-07	0.18	0.6823	0.000375	1	0.000879	-0.0017	0.002452
C^2	6.32E-05	1	6.32E-05	19.45	0.0031	0.003875	1	0.000879	0.001798	0.005952
Residual	2.28E-05	7	3.25E-06							
Lack of Fit	2.75E-06	3	9.17E-07	0.18	0.9026					
Pure Error	0.00002	4	5E-06							
Cor Total	0.002468	16								

Std. Dev.	0.0018	R-Squared	0.9908	df= degrees of freedom; SE= Standard Error
Mean	0.2622	Adj R-Squared	0.9789	Coeff. Est = Coefficient of estimate
C.V. %	0.6876	Pred R-Squared	0.9695	CI = Confidence of Interval
PRESS	0.0001	Adeq Precision	32.0034	ns= non significance

Table B17 Effect of spray drying processing conditions on reconstitution properties of instant green tea powder

Run	Temp. (°C)	MD conc. (%)	Feed flow rate ($ml.h^{-1}$)	Solubility (%)	Wettability (s)
1	160	2	672	120	80.800
2	180	2	672	202	80.400
3	160	4	672	180	84.600
4	180	4	672	221	83.800
5	160	3	420	180	83.000
6	180	3	420	220	83.400
7	160	3	924	42	81.000
8	180	3	924	83	81.800
9	170	2	420	169	85.400
10	170	4	420	224	89.800
11	170	2	924	50	81.400
12	170	4	924	91	85.900
13	170	3	672	172	85.700
14	170	3	672	150	86.000
15	170	3	672	149	86.400
16	170	3	672	166	85.900
17	170	3	672	169	85.700

Table B18 Analysis of variance (ANOVA) for Wettability of spray dried instant green tea

Source	Sum of Squares	Df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	51035.51	9	5670.61	49.77	< 0.0001	161.20	1	4.77	149.91	172.49
A-Temperature	5202	1	5202.00	45.66	0.0003	25.50	1	3.77	16.58	34.42
B-MD	3828.125	1	3828.13	33.60	0.0007	21.88	1	3.77	12.95	30.80
C-Feed rate	34716.13	1	34716.13	304.70	< 0.0001	-65.88	1	3.77	-74.80	-56.95
AB	420.25	1	420.25	3.69	0.0963	-10.25	1	5.34	-22.87	2.37
AC	0.25	1	0.25	0.00	0.9639	0.25	1	5.34	-12.37	12.87
BC	49	1	49.00	0.43	0.5329	-3.50	1	5.34	-16.12	9.12
A^2	315.0421	1	315.04	2.77	0.1403	8.65	1	5.20	-3.65	20.95
B^2	500.2526	1	500.25	4.39	0.0744	10.90	1	5.20	-1.40	23.20
C^2	6273.516	1	6273.52	55.06	0.0001	-38.60	1	5.20	-50.90	-26.30
Residual	797.55	7	113.94							
Lack of Fit	322.75	3	107.58	0.91	0.5127					
Pure Error	474.8	4	118.70							
Cor Total	51833.06	16								

Std. Dev.	10.6741	R-Squared	0.9846	df= degrees of freedom; SE= Standard Error
Mean	152.2353	Adj R-Squared	0.9648	Coeff. Est = Coefficient of estimate
C.V. %	7.0116	Pred R-Squared	0.8861	CI = Confidence of Interval
PRESS	5905.8750	Adeq Precision	22.6131	ns= non significance

Table B19 Analysis of variance (ANOVA) for Solubility of spray dried instant green tea

Source	Sum of Squares	Df	Mean Sums of Square	F Value	p-value Prob > F	Coeff. Est.	df	SE	95% CI Low	95% CI High
Model	99.67	9	11.07	20.81	0.0003	85.94	1	0.33	85.17	86.71
A-Temperature	0.00	1	0.00	0.00	1.0000	0.00	1	0.26	-0.61	0.61
B-MD	32.40	1	32.40	60.90	0.0001	2.01	1	0.26	1.40	2.62
C-Feed rate	16.53	1	16.53	31.07	0.0008	-1.44	1	0.26	-2.05	-0.83
AB	0.04	1	0.04	0.08	0.7919	-0.10	1	0.36	-0.96	0.76
AC	0.04	1	0.04	0.08	0.7919	0.10	1	0.36	-0.76	0.96
BC	0.00	1	0.00	0.00	0.9473	0.03	1	0.36	-0.84	0.89
A^2	49.61	1	49.61	93.24	< 0.0001	-3.43	1	0.36	-4.27	-2.59
B^2	0.05	1	0.05	0.09	0.7711	-0.11	1	0.36	-0.95	0.73
C^2	0.18	1	0.18	0.34	0.5777	-0.21	1	0.36	-1.05	0.63
Residual	3.72	7	0.53							
Lack of Fit	3.39	3	1.13	13.62	0.0144					
Pure Error	0.33	4	0.08							
Cor Total	103.39	16								

Std. Dev.	0.7294	R-Squared	0.9640	df= degrees of freedom; SE= Standard Error
Mean	84.1765	Adj R-Squared	0.9177	Coeff. Est = Coefficient of estimate
C.V. %	0.8666	Pred R-Squared	0.4700	CI = Confidence of Interval
PRESS	54.7988	Adeq Precision	15.6627	ns= non significance

Table B20 Effect of spray drying processing conditions on flow properties of instant green tea powder

Run	Temp. (°C)	MD conc. (%)	Feed flow rate ($ml.h^{-1}$)	Carr index	Hausner ratio
1	160	2	672	1.555	35.6796
2	180	2	672	1.333	25.0000
3	160	4	672	1.519	34.1860
4	180	4	672	1.287	22.3214
5	160	3	420	1.530	34.6341
6	180	3	420	1.317	24.0741
7	160	3	924	1.479	32.3944
8	180	3	924	1.273	21.4286
9	170	2	420	1.357	26.2840
10	170	4	420	1.300	23.0994
11	170	2	924	1.345	25.6338
12	170	4	924	1.302	23.2044
13	170	3	672	1.321	24.2857
14	170	3	672	1.337	25.2174
15	170	3	672	1.324	24.4898
16	170	3	672	1.333	25.0000
17	170	3	672	1.327	24.6334

Table B21 Response optimization constraints of spray drying of green tea extraction

Sl. No.	Parameters	Goal	Lower limit	Upper limit	Importance
1	Temperature	Is in range	160.00	180.00	3
2	MD	Is in range	2.00	4.00	3
3	Feed rate	Is in range	420.00	924.00	3
4	Yield	Maximize	27.24	35.73	3
5	Loose bulk density	Minimize	0.24	0.29	3
6	Tapped bulk density	Minimize	0.32	0.43	3
7	Carr Index	Minimize	21.43	35.68	3
8	Hausner Ratio	Minimize	1.27	1.55	3
9	Moisture content	Minimize	3.24	8.32	3
10	Solubility	Maximize	80.40	89.80	3
11	Water activity	Minimize	0.13	0.35	3
12	Wettability	Minimize	42.00	224.00	3
13	L*	Minimize	81.71	88.15	3
14	a*	Maximize	2.10	2.83	3
15	b*	Maximize	15.25	22.56	3
16	Total flavonoids	Maximize	18.78	28.34	3
17	Caffein	Minimize	14.06	20.92	3
19	Total polyphenols	Maximize	27.00	59.00	3
20	Hue value	Maximize	80.60	83.65	3

Table B22: Scale factor, fuzzy membership and normalized membership functions for quality attributes of reconstituted instant green tea powder

			Sample T_0			Sample HT_1			Sample HT_2			Sample HT_3			Sample HT_4			Sample CT_5			Sample CT_6			Sample CT_7			Sample CT_8		
SA	**SF**	**SF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**
Colour and Appearance	**EX**	1.0	9	0.60	0.60	4	0.27	0.27	4	0.27	0.27	5	0.33	0.33	10	0.67	0.67	1	0.07	0.07	1	0.07	0.07	3	0.20	0.20	9	0.60	0.60
	GD	0.9	6	0.40	0.36	9	0.60	0.54	9	0.60	0.54	9	0.60	0.54	4	0.27	0.24	10	0.67	0.60	9	0.60	0.54	8	0.53	0.48	4	0.27	0.24
	MD	0.7	0	0.00	0.00	1	0.07	0.05	1	0.07	0.05	1	0.07	0.05	0	0.00	0.00	2	0.13	0.09	5	0.33	0.23	4	0.27	0.19	1	0.07	0.05
	FR	0.4	0	0.00	0.00	1	0.07	0.03	1	0.07	0.03	0	0.00	0.00	1	0.07	0.03	2	0.13	0.05	0	0.00	0.00	0	0.00	0.00	1	0.07	0.03
	NS	0.1	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**15**	**1.00**	**0.96**	**15**	**1.00**	**0.88**	**15**	**1.00**	**0.88**	**15**	**1.00**	**0.92**	**15**	**1.00**	**0.93**	**15**	**1.00**	**0.81**	**15**	**1.00**	**0.84**	**15**	**1.00**	**0.87**	**15**	**1.00**	**0.91**
Taste	**EX**	1.0	8.0	0.53	0.53	2	0.13	0.13	2	0.13	0.13	7	0.47	0.47	4	0.27	0.27	1	0.07	0.07	3	0.20	0.20	2	0.13	0.13	4	0.27	0.27
	GD	0.9	6.0	0.40	0.36	10	0.67	0.60	8	0.53	0.48	6	0.40	0.36	7	0.47	0.42	8	0.53	0.48	6	0.40	0.36	8	0.53	0.48	6	0.40	0.36
	MD	0.7	0.0	0.00	0.00	1	0.07	0.05	4	0.27	0.19	1	0.07	0.05	1	0.07	0.05	4	0.27	0.19	4	0.27	0.19	3	0.20	0.14	3	0.20	0.14
	FR	0.4	1.0	0.07	0.03	2	0.13	0.05	1	0.07	0.03	1	0.07	0.03	1	0.07	0.03	2	0.13	0.05	1	0.07	0.03	1	0.07	0.03	1	0.07	0.03
	NS	0.1	0.0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	2	0.13	0.01		0.00	0.00	1	0.07	0.01	1	0.07	0.01	1	0.07	0.01
Total			**15.0**	**1.00**	**0.92**	**15**	**1.00**	**0.83**	**15**	**1.00**	**0.83**	**15**	**1.00**	**0.90**	**15**	**1.00**	**0.77**	**15**	**1.00**	**0.79**	**15**	**1.00**	**0.78**	**15**	**1.00**	**0.79**	**15**	**1.00**	**0.80**
Flavour	**EX**	1.0	5.0	0.33	0.33	2	0.13	0.13	2	0.13	0.13	7	0.47	0.47	3	0.20	0.20	3	0.20	0.20	2	0.13	0.13	5	0.33	0.33	4	0.27	0.27
	GD	0.9	7.0	0.47	0.42	10	0.67	0.60	8	0.53	0.48	7	0.47	0.42	8	0.53	0.48	7	0.47	0.42	6	0.40	0.36	4	0.27	0.24	7	0.47	0.42
	MD	0.7	1.0	0.07	0.05	1	0.07	0.05	5	0.33	0.23	0	0.00	0.00	1	0.07	0.05	3	0.20	0.14	5	0.33	0.23	4	0.27	0.19	2	0.13	0.09
	FR	0.4	2.0	0.13	0.05	2	0.13	0.05	0	0.00	0.00	1	0.07	0.03	2	0.13	0.05	2	0.13	0.05	1	0.07	0.03	1	0.07	0.03	1	0.07	0.03
	NS	0.1	0.0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	1	0.07	0.01	0	0.00	0.00	1	0.07	0.01	1	0.07	0.01	1	0.07	0.01
Total			**15.0**	**1.00**	**0.85**	**15**	**1.00**	**0.83**	**15**	**1.00**	**0.85**	**15**	**1.00**	**0.91**	**15**	**1.00**	**0.79**	**15**	**1.00**	**0.81**	**15**	**1.00**	**0.76**	**15**	**1.00**	**0.79**	**15**	**1.00**	**0.81**
Astringency	**EX**	1.0	6.0	0.40	0.40	2	0.13	0.13	2	0.13	0.13	4	0.27	0.27	3	0.20	0.20	1	0.07	0.07	2	0.13	0.13	3	0.20	0.20	4	0.27	0.27
	GD	0.9	4.0	0.27	0.24	9	0.60	0.54	7	0.47	0.42	8	0.53	0.48	7	0.47	0.42	8	0.53	0.48	6	0.40	0.36	7	0.47	0.42	6	0.40	0.36
	MD	0.7	3.0	0.20	0.14	0	0.00	0.00	5	0.33	0.23	2	0.13	0.09	3	0.20	0.14	3	0.20	0.14	5	0.33	0.23	3	0.20	0.14	2	0.13	0.09
	FR	0.4	2.0	0.13	0.05	4	0.27	0.11	1	0.07	0.03	1	0.07	0.03	1	0.07	0.03	3	0.20	0.08	1	0.07	0.03	2	0.13	0.05	2	0.13	0.05
	NS	0.1	0.0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	1	0.07	0.01	0	0.00	0.00	1	0.07	0.01	0	0.00	0.00	1	0.07	0.01
Total			**15.0**	**1.00**	**0.83**	**15**	**1.00**	**0.78**	**15**	**1.00**	**0.81**	**15**	**1.00**	**0.87**	**15**	**1.00**	**0.79**	**15**	**1.00**	**0.77**	**15**	**1.00**	**0.76**	**15**	**1.00**	**0.81**	**15**	**1.00**	**0.78**
Overall acceptability	**EX**	1.0	6.0	0.40	0.40	1	0.07	0.07	1	0.07	0.07	7	0.47	0.47	3	0.20	0.20	3	0.20	0.20	2	0.13	0.13	5	0.33	0.33	4	0.27	0.27
	GD	0.9	8.0	0.53	0.48	11	0.73	0.66	10	0.67	0.60	6	0.40	0.36	8	0.53	0.48	6	0.40	0.36	7	0.47	0.42	4	0.27	0.24	6	0.40	0.36
	MD	0.7	1.0	0.07	0.05	2	0.13	0.09	4	0.27	0.19	1	0.07	0.05	1	0.07	0.05	5	0.33	0.23	5	0.33	0.23	4	0.27	0.19	3	0.20	0.14
	FR	0.4	0.0	0.00	0.00	1	0.07	0.03	0	0.00	0.00	1	0.07	0.03	3	0.20	0.08	1	0.07	0.03	1	0.07	0.03	2	0.13	0.05	2	0.13	0.05
	NS	0.1	0.0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**15.0**	**1.00**	**0.93**	**15**	**1.00**	**0.85**	**15**	**1.00**	**0.85**	**15**	**1.00**	**0.90**	**15**	**1.00**	**0.81**	**15**	**1.00**	**0.82**	**15**	**1.00**	**0.81**	**15**	**1.00**	**0.81**	**15**	**1.00**	**0.82**

SA: sensory attribute; SF: Scale factor; FMF: fuzzy membership functions; NFMF: normalized fuzzy membership functions

Table B23 Judgment membership functions of reconstituted instant green tea sample

	Sample T_0	**Sample HT_1**	**Sample HT_2**	**Sample HT_3**	**Sample HT_4**	**Sample CT_5**	**Sample CT_6**	**Sample CT_7**	**Sample CT_8**
Colour and Appearance	0.213	0.196	0.196	0.204	0.207	0.181	0.187	0.193	0.203
Taste	0.204	0.185	0.184	0.200	0.172	0.175	0.173	0.175	0.203
Flavour	0.190	0.185	0.188	0.203	0.175	0.175	0.169	0.176	0.181
Astringency	0.185	0.173	0.181	0.193	0.176	0.170	0.169	0.181	0.173
Overall acceptability	0.206	0.188	0.190	0.200	0.179	0.182	0.181	0.181	0.182

Appendix C

Table C1 Biochemical properties of ginger flavoured instant green tea

Sl. No.	Treatment	Total flavonoids, $mg.g^{-1}$	Total polyphenols, $mg.g^{-1}$	Caffeine, $mg.g^{-1}$
1	Control	26.67	43.14	15.17
2	G1	27.07	44.02	15.10
3	G2	27.57	44.79	15.06
4	G3	27.80	45.06	15.02
5	G4	28.10	45.37	15.00
6	G5	27.21	44.38	15.08
7	G6	27.70	45.03	15.00
8	G7	28.23	45.47	14.92
9	G8	29.07	45.98	14.68

Table C2 Analysis of variance (ANOVA) for total flavonoid content of ginger flavoured instant green tea

Source	D.F.	SS	MSS	Cal. F	TAB. F(5%)	TAB. F(1%)
Treatment	8	12.133	1.517	1.436	**NS**	**NS**
Replication	2	1.063	0.532	0.503	**NS**	**NS**
Error	18	19.017	1.056			
TOTAL	26					
S.Em=	0.593	**CD(5%)=**	1.763	**TAB F (5%)=**	2.510	
CV=	3.709	**CD(1%)=**	2.416	**TAB F (1%)=**	3.705	

Table C3 Analysis of variance (ANOVA) for total polyphenol content of ginger flavoured instant green tea

Source	D.F.	SS	MSS	Cal. F	TAB. F(5%)	TAB. F(1%)
Treatment	8	17.481	2.185	0.793	**NS**	**NS**
Replication	2	2.779	1.390	0.504	**NS**	**NS**
Error	18	49.631	2.757			
TOTAL	26					
S.Em=	0.959	**CD(5%)=**	2.848	**TAB F (5%)=**	2.510	
CV=	3.706	**CD(1%)=**	3.903	**TAB F (1%)=**	3.705	

Table C4 Analysis of variance (ANOVA) for caffeine content of ginger flavoured instant green tea

Source	D.F.	SS	MSS	Cal. F	TAB. F(5%)	TAB. F(1%)
Treatment	8	0.476	0.060	1.129	**NS**	**NS**
Replication	2	0.079	0.039	0.749	**NS**	**NS**
Error	18	0.949	0.053			
Total	26					
S.Em=	0.133	**CD(5%)=**	0.394	**TAB F (5%)=**	2.510	
CV=	1.531	**CD(1%)=**	0.540	**TAB F (1%)=**	3.705	

Table C5: Scale factor, fuzzy membership and normalized membership functions for quality attributes of ginger flavoured instant green tea sample

			Sample G_0			Sample G1			Sample G2			Sample G3			Sample G7			Sample G8		
Sensory attributes	**SF**	**SF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**
Colour and appearance	**EX**	1	8	0.8	0.8	3	0.3	0.3	1	0.1	0.1	3	0.3	0.3	7	0.7	0.7	0	0	0
	GD	0.9	2	0.2	0.18	7	0.7	0.63	9	0.9	0.81	7	0.7	0.63	3	0.3	0.27	10	1	0.9
	MD	0.7	0	0.0	0.00	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	FR	0.4	0	0.0	0.00	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	NS	0.1	0	0.0	0.00	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Total			**10**	**1**	**0.98**	**10**	**1**	**0.93**	**10**	**1**	**0.91**	**10**	**1**	**0.93**	**10**	**1**	**0.97**	**10**	**1**	**0.9**
Taste	**EX**	1	0	0	0	4	0.4	0.4	6	0.6	0.6	1	0.1	0.1	4	0.4	0.4	3	0.3	0.3
	GD	0.9	9	0.9	0.81	6	0.6	0.54	4	0.4	0.36	8	0.8	0.72	5	0.5	0.45	6	0.6	0.54
	MD	0.7	0	0	0	0	0	0	0	0	0	1	0.1	0.07	1	0.1	0.07	1	0.1	0.07
	FR	0.4	1	0.1	0.04	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	NS	0.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Total			**10**	**1**	**0.85**	**10**	**1**	**0.94**	**10**	**1**	**0.96**	**10**	**1**	**0.89**	**10**	**1**	**0.92**	**10**	**1**	**0.91**
Flavour	**EX**	1	1	0.1	0.1	4	0.4	0.4	10	1	1	1	0.1	0.1	3	0.3	0.3	3	0.3	0.3
	GD	0.9	6	0.6	0.54	5	0.5	0.45	0	0	0	8	0.8	0.72	6	0.6	0.54	6	0.6	0.54
	MD	0.7	3	0.3	0.21	1	0.1	0.07	0	0	0	0	0	0	1	0.1	0.07	1	0.1	0.07
	FR	0.4	0	0	0	0	0	0	0	0	0	1	0.1	0.04	0	0	0	0	0	0
	NS	0.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Total			**10**	**1**	**0.85**	**10**	**1**	**0.92**	**10**	**1**	**1**	**10**	**1**	**0.86**	**10**	**1**	**0.91**	**10**	**1**	**0.91**
Astringency	**EX**	1	4	0.4	0.4	6	0.6	0.6	6	0.6	0.6	3	0.3	0.3	3	0.3	0.3	4	0.4	0.4
	GD	0.9	4	0.4	0.36	3	0.3	0.27	3	0.3	0.27	6	0.6	0.54	5	0.5	0.45	5	0.5	0.45
	MD	0.7	1	0.1	0.07	1	0.1	0.07	1	0.1	0.07	0	0	0	2	0.2	0.14	1	0.1	0.07
	FR	0.4	1	0.1	0.04	0	0	0	0	0	0	1	0.1	0.04	0	0	0	0	0	0
	NS	0.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Total			**10**	**1**	**0.87**	**10**	**1**	**0.94**	**10**	**1**	**0.94**	**10**	**1**	**0.88**	**10**	**1**	**0.89**	**10**	**1**	**0.92**
Overall acceptability	**EX**	1	2	0.2	0.2	5	0.5	0.5	7	0.7	0.7	2	0.2	0.2	4	0.4	0.4	2	0.2	0.2
	GD	0.9	5	0.5	0.45	5	0.5	0.45	2	0.2	0.18	7	0.7	0.63	5	0.5	0.45	7	0.7	0.63
	MD	0.7	2	0.2	0.14	0	0	0	1	0.1	0.07	0	0	0	0	0	0	1	0.1	0.07
	FR	0.4	1	0.1	0.04	0	0	0	0	0	0	1	0.1	0.04	1	0.1	0.04	0	0	0
	NS	0.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Total			**10**	1	**0.83**	**10**	**1**	**0.95**	**10**	**1**	**0.95**	**10**	**1**	**0.87**	**10**	**1**	**0.89**	**10**	**1**	**0.9**

SF: Scale factor; FMF: Fuzzy membership functions; NFMF: Normalized fuzzy membership functions; EX: Excellent; GD: Good: MD: Medium; FR: Fair; NS: Not satisfactory

Table C6 Judgment membership functions of ginger flavoured instant green tea sample

	Sample G0	**Sample G1**	**Sample G2**	**Sample G3**	**Sample G7**	**Sample G8**
Colour and Appearance	0.206	0.195	0.191	0.195	0.204	0.189
Taste	0.179	0.197	0.202	0.187	0.193	0.191
Flavour	0.179	0.193	0.210	0.187	0.191	0.191
Astringency	0.183	0.197	0.197	0.185	0.187	0.193
Overall acceptability	0.174	0.200	0.200	0.183	0.187	0.189

Table C7 Biochemical properties of cardamom flavoured instant green tea

Sl. No.	Treatment	Total flavonoids ($mg.g^{-1}$)	Total polyphenols ($mg.g^{-1}$)	Caffeine ($mg.g^{-1}$)
1	Control	26.670	43.140	15.170
2	C1	26.820	44.040	15.158
3	C2	26.900	44.320	15.150
4	C3	26.940	44.980	15.146
5	C4	26.880	44.370	15.157
6	C5	26.940	45.50	15.147
7	C6	26.97	45.600	15.142
8	C7	26.92	45.370	15.150
9	C8	26.97	46.160	15.143
10	C9	27.02	46.820	15.140

Table C8 Analysis of variance (ANOVA) for total flavonoid content of cardamom flavoured instant green tea

Source	D.F.	SS	MSS	Cal. F	TAB. F(5%)	TAB. F(1%)
Treatment	9	0.262	0.029	0.042	**NS**	**NS**
Replication	2	0.631	0.316	0.459	**NS**	**NS**
Error	20	13.759	0.688			
TOTAL	29					
S.Em=	0.479	**CD(5%)=**	1.413	**TAB F (5%)=**	2.393	
CV=	3.083	**CD(1%)=**	1.927	**TAB F (1%)=**	3.457	

Table C9 Analysis of variance (ANOVA) for total polyphenol content of cardamom flavoured instant green tea

Source	D.F.	SS	MSS	Cal. F	TAB. F(5%)	TAB. F(1%)
Treatment	9	33.135	3.682	1.244	**NS**	**NS**
Replication	2	11.968	5.984	2.022	**NS**	**NS**
Error	20	59.192	2.960			
TOTAL	29					
S.Em=	0.993	**CD(5%)=**	2.930	**TAB F (5%)=**	2.393	
CV=	3.818	**CD(1%)=**	3.997	**TAB F (1%)=**	3.457	

Table C10 Analysis of variance (ANOVA) for caffeine content of cardamom flavoured instant green tea

Source	D.F.	SS	MSS	Cal. F	TAB. F(5%)	TAB. F(1%)
Treatment	9	0.002	0.000	0.000	**NS**	**NS**
Replication	2	5.294	2.647	5.224	**S**	**NS**
Error	20	10.135	0.507			
TOTAL	29					
S.Em=	0.411	**CD(5%)=**	1.212	**TAB F (5%)=**	2.393	
CV=	4.699	**CD(1%)=**	1.654	**TAB F (1%)=**	3.457	

Table C11 Scale factor, fuzzy membership and normalized membership functions for quality attributes of cardamom flavoured instant green tea sample

			Sample C0			Sample C7			Sample C6			Sample C8			Sample C3			Sample C5		
Sensory attributes	**SF**	**SF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**
Colour and Appearance	**EX**	1	6	0.46	0.46	4	0.31	0.31	4	0.31	0.31	2	0.15	0.15	2	0.15	0.15	3	0.23	0.23
	GD	0.9	7	0.54	0.48	9	0.69	0.62	9	0.69	0.62	11	0.85	0.76	11	0.85	0.76	10	0.77	0.69
	MD	0.7	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	FR	0.4	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	NS	0.1	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**13**	**1.00**	**0.95**	**13**	**1.00**	**0.93**	**13**	**1.00**	**0.93**	**13**	**1.00**	**0.92**	**13**	**1.00**	**0.92**	**13**	**1.00**	**0.92**
Taste	**EX**	1	3	0.23	0.23	2	0.15	0.15	4	0.31	0.31	2	0.15	0.15	5	0.38	0.38	2	0.15	0.15
	GD	0.9	8	0.62	0.55	10	0.77	0.69	9	0.69	0.62	11	0.85	0.76	7	0.54	0.48	9	0.69	0.62
	MD	0.7	2	0.15	0.11	1	0.08	0.05	0	0.00	0.00	0	0.00	0.00	1	0.08	0.05	2	0.15	0.11
	FR	0.4	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	NS	0.1	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**13**	**1.00**	**0.89**	**13**	**1.00**	**0.90**	**13**	**1.00**	**0.93**	**13**	**1.00**	**0.92**	**13**	**1.00**	**0.92**	**13**	**1.00**	**0.88**
Flavour	**EX**	1	2	0.15	0.15	4	0.31	0.31	13	1.00	1.00	6	0.46	0.46	4	0.31	0.31	2	0.15	0.15
	GD	0.9	10	0.77	0.69	8	0.62	0.55	0	0.00	0.00	6	0.46	0.42	8	0.62	0.55	10	0.77	0.69
	MD	0.7	1	0.08	0.05	1	0.08	0.05	0	0.00	0.00	1	0.08	0.05	1	0.08	0.05	1	0.08	0.05
	FR	0.4	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	NS	0.1	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**13**	**1.00**	**0.90**	**13**	**1.00**	**0.92**	**13**	**1.00**	**1.00**	**13**	**1.00**	**0.93**	**13**	**1.00**	**0.92**	**13**	**1.00**	**0.90**
Astringency	**EX**	1	3	0.23	0.23	5	0.38	0.38	6	0.46	0.46	5	0.38	0.38	6	0.46	0.46	3	0.23	0.23
	GD	0.9	9	0.69	0.62	8	0.62	0.55	7	0.54	0.48	8	0.62	0.55	6	0.46	0.42	10	0.77	0.69
	MD	0.7	1	0.08	0.05	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	1	0.08	0.05		0.00	0.00
	FR	0.4	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	NS	0.1	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**13**	**1.00**	**0.91**	**13**	**1.00**	**0.94**	**13**	**1.00**	**0.95**	**13**	**1.00**	**0.94**	**13**	**1.00**	**0.93**	**13**	**1.00**	**0.92**
Overall acceptability	**EX**	1	5	0.38	0.38	5	0.38	0.38	6	0.46	0.46	4	0.31	0.31	5	0.38	0.38	2	0.15	0.15
	GD	0.9	7	0.54	0.48	8	0.62	0.55	7	0.54	0.48	9	0.69	0.62	8	0.62	0.55	10	0.77	0.69
	MD	0.7	1	0.08	0.05	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	1	0.08	0.05
	FR	0.4	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	NS	0.1	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**13**	1.00	**0.92**	**13**	**1.00**	**0.94**	**13**	**1.00**	**0.95**	**13**	**1.00**	**0.93**	**13**	**1.00**	**0.94**	**13**	**1.00**	**0.90**

SF: Scale factor; FMF: Fuzzy membership functions; NFMF: Normalized fuzzy membership functions; EX: Excellent; GD: Good: MD: Medium; FR: Fair; NS: Not satisfactory

Table 4.12 Judgment membership functions of cardamom flavoured instant green tea sample

	Sample C0	**Sample C7**	**Sample C6**	**Sample C8**	**Sample C3**	**Sample C5**
Colour and Appearance	0.199	0.196	0.196	0.193	0.193	0.200
Taste	0.188	0.189	0.196	0.193	0.194	0.186
Flavour	0.189	0.193	0.210	0.193	0.193	0.189
Astringency	0.191	0.197	0.199	0.197	0.196	0.194
Overall acceptability	0.194	0.197	0.199	0.196	0.197	0.189

Table C13 Biochemical properties of tulsi flavoured instant green tea

Sl. No.	Treatment	Total polyphenols ($mg.g^{-1}$)	Total flavonoids ($mg.g^{-1}$)	Caffeine ($mg.g^{-1}$)
1	Control	43.14	26.67	15.17
2	H1	44.01	27.11	15.15
3	H2	44.78	27.69	15.13
4	H3	45.59	28.39	15.11
5	H4	46.12	28.75	15.09
6	H5	44.18	27.59	15.11
7	H6	45.29	28.15	15.08
8	H7	46.28	28.72	15.04
9	H8	46.76	29.05	15.00

Table C14 Analysis of variance (ANOVA) for total flavonoid content of tulsi flavoured instant green tea

Source	D.F.	SS	MSS	Cal. F	TAB. F(5%)	TAB. F(1%)
Treatment	8	14.189	1.774	2.493	**NS**	**NS**
Replication	2	1.410	0.705	0.991	**NS**	**NS**
Error	18	12.808	0.712			
TOTAL	26					
S.Em=	0.487	**CD(5%)=**	1.447	**TAB F (5%)=**	2.510	
CV=	3.021	**CD(1%)=**	1.983	**TAB F (1%)=**	3.705	

Table C15 Analysis of variance (ANOVA) for total polyphenol content of tulsi flavoured instant green tea

Source	D.F.	SS	MSS	Cal. F	TAB. F(5%)	TAB. F(1%)
Treatment	8	34.308	4.289	1.995	**NS**	**NS**
Replication	2	1.806	0.903	0.420	**NS**	**NS**
Error	18	38.698	2.150			
TOTAL	26					
S.Em=	0.847	**CD(5%)=**	2.515	**TAB F (5%)=**	2.510	
CV=	3.249	**CD(1%)=**	3.446	**TAB F (1%)=**	3.705	

Table C16 Analysis of variance (ANOVA) for caffeine content of tulsi flavoured instant green tea

Source	D.F.	SS	MSS	Cal. F	TAB. F(5%)	TAB. F(1%)
Treatment	8	0.068	0.008	0.015	**NS**	**NS**
Replication	2	6.204	3.102	5.361	**S**	**NS**
Error	18	10.415	0.579			
TOTAL	26					
S.Em=	0.439	**CD(5%)=**	1.305	**TAB F (5%)=**	2.510	
CV=	5.038	**CD(1%)=**	1.788	**TAB F (1%)=**	3.705	

Table C17 Scale factor, fuzzy membership and normalized membership functions for quality attributes of tulsi flavoured instant green tea sample

			Sample H0			Sample H6			Sample H4			Sample H2			Sample H3			Sample H5		
Sensory attributes	**SF**	**SF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**		**FMF**	**NFMF**
Colour and Appearance	**EX**	1	8	0.73	0.73	2	0.18	0.18	4	0.36	0.36	3	0.27	0.27	6	0.55	0.55	5	0.45	0.45
	GD	0.9	3	0.27	0.25	7	0.64	0.57	7	0.64	0.57	8	0.73	0.65	5	0.45	0.41	6	0.55	0.49
	MD	0.7	0	0.00	0.00	2	0.18	0.13	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	FR	0.4	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	NS	0.1	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**11**	**1.00**	**0.97**	**11**	**1.00**	**0.88**	**11**	**1.00**	**0.94**	**11**	**1.00**	**0.93**	**11**	**1.00**	**0.95**	**11**	**1.00**	**0.95**
Taste	**EX**	1	2	0.18	0.18	4	0.36	0.36	3	0.27	0.27	6	0.55	0.55	3	0.27	0.27	3	0.27	0.27
	GD	0.9	7	0.64	0.57	6	0.55	0.49	8	0.73	0.65	4	0.36	0.33	8	0.73	0.65	7	0.64	0.57
	MD	0.7	1	0.09	0.06	1	0.09	0.06	0	0.00	0.00	1	0.09	0.06	0	0.00	0.00	1	0.09	0.06
	FR	0.4	1	0.09	0.04	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	NS	0.1	0	0.00	0.00		0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**11**	**1.00**	**0.85**	**11**	**1.00**	**0.92**	**11**	**1.00**	**0.93**	**11**	**1.00**	**0.94**	**11**	**1.00**	**0.93**	**11**	**1.00**	**0.91**
Flavour	**EX**	1	4	0.36	0.36	0	0.00	0.00	4	0.36	0.36	4	0.36	0.36	10	0.91	0.91	5	0.45	0.45
	GD	0.9	6	0.55	0.49	8	0.73	0.65	5	0.45	0.41	6	0.55	0.49	1	0.09	0.08	6	0.55	0.49
	MD	0.7	1	0.09	0.06	2	0.18	0.13	2	0.18	0.13	1	0.09	0.06	0	0.00	0.00	0	0.00	0.00
	FR	0.4	0	0.00	0.00	1	0.09	0.04	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	NS	0.1	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**11**	**1.00**	**0.92**	**11**	**1.00**	**0.82**	**11**	**1.00**	**0.90**	**11**	**1.00**	**0.92**	**11**	**1.00**	**0.99**	**11**	**1.00**	**0.95**
Astringency	**EX**	1	4	0.36	0.36	3	0.27	0.27	3	0.27	0.27	4	0.36	0.36	3	0.27	0.27	4	0.36	0.36
	GD	0.9	6	0.55	0.49	6	0.55	0.49	7	0.64	0.57	7	0.64	0.57	8	0.73	0.65	6	0.55	0.49
	MD	0.7	1	0.09	0.06	2	0.18	0.13	1	0.09	0.06	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	FR	0.4	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	1	0.09	0.04
	NS	0.1	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**11**	**1.00**	**0.92**	**11**	**1.00**	**0.89**	**11**	**1.00**	**0.91**	**11**	**1.00**	**0.94**	**11**	**1.00**	**0.93**	**11**	**1.00**	**0.89**
Overall acceptability	**EX**	1	2	0.18	0.18	2	0.18	0.18	4	0.36	0.36	5	0.45	0.45	8	0.73	0.73	5	0.45	0.45
	GD	0.9	9	0.82	0.74	9	0.82	0.74	7	0.64	0.57	6	0.55	0.49	3	0.27	0.25	6	0.55	0.49
	MD	0.7	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	FR	0.4	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
	NS	0.1	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00	0	0.00	0.00
Total			**11**	1.00	**0.92**	**11**	**1.00**	**0.92**	**11**	**1.00**	**0.94**	**11**	**1.00**	**0.95**	**11**	**1.00**	**0.97**	**11**	**1.00**	**0.95**

SF: Scale factor; FMF: Fuzzy membership functions; NFMF: Normalized fuzzy membership functions; EX: Excellent; GD: Good: MD: Medium; FR: Fair; NS: Not satisfactory

Table C18 Judgment membership functions of tulsi flavoured instant green tea sample

	Sample H0	**Sample H2**	**Sample H3**	**Sample H4**	**Sample H5**	**Sample H6**
Colour and Appearance	0.204	0.194	0.200	0.196	0.198	0.185
Taste	0.179	0.196	0.194	0.194	0.190	0.192
Flavour	0.192	0.192	0.208	0.189	0.190	0.171
Astringency	0.192	0.196	0.194	0.190	0.187	0.187
Overall acceptability	0.192	0.198	0.204	0.196	0.198	0.192

APPENDIX D

Table D1 Effect of storage on moisture content of instant flavoured green tea powder in different packaging materials

Sl. No.	Months	Ginger flavoured instant green tea			Cardamom flavoured instant green tea			Tulsi flavoured instant green tea		
1		ALF	PET	LDPE	ALF	PET	LDPE	ALF	PET	LDPE
2	0	3.08	3.08	3.08	3.03	3.03	3.03	3.05	3.05	3.05
3	1	3.88	4.01	4.78	3.78	4.00	4.66	3.78	4.00	4.66
4	2	4.64	5.06	6.46	4.53	4.94	6.57	4.60	4.94	6.57
5	3	5.46	6.28	7.97	5.18	6.16	7.90	5.28	6.16	7.90
6	4	6.38	7.11	9.48	6.17	7.09	9.33	6.21	7.09	9.33
7	5	7.16	8.05	11.22	7.00	7.91	11.12	7.03	7.91	11.12
8	6	7.70	8.96	12.93	7.62	8.90	12.87	7.68	8.91	12.89

Table D2. Analysis of variance (ANOVA) for moisture content of instant flavoured green tea during storage

Ginger flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	430.40	20	21.52	314.30	< 0.0001	0.26	4.01
A-Storage period	328.15	6	54.69	798.76	< 0.0001		
B-Packaging	71.37	2	35.68	521.16	< 0.0001		
AB	30.89	12	2.57	37.60	< 0.0001		
Pure error	2.88	42	0.07				
Cor total	433.28	62					

Tulsi flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	427.18	20	21.36	299.31	< 0.0001	0.27	4.15
A-Storage period	320.70	6	53.45	749.02	< 0.0001	6.44	
B-Packaging	74.46	2	37.23	521.71	< 0.0001		
AB	32.02	12	2.67	37.39	< 0.0001		
Pure error	3.00	42	0.07				
Cor total	430.18	62					

Cardamom flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	429.46	20	21.47	330.80	< 0.0001	0.25	3.97
A-Storage period	320.52	6	53.42	822.95	< 0.0001		
B-Packaging	76.42	2	38.21	588.63	< 0.0001		
AB	32.53	12	2.71	41.76	< 0.0001		
Pure error	2.73	42	0.06				
Cor total	432.19	62					

Table D3 Effect of storage on water activity of instant flavoured green tea powder in different packaging materials

Sl. No.	Months	Ginger flavoured instant green tea			Cardamom flavoured instant green tea			Tulsi flavoured instant green tea		
1		ALF	PET	LDPE	ALF	PET	LDPE	ALF	PET	LDPE
2	0	0.149	0.149	0.149	0.147	0.147	0.147	0.148	0.148	0.148
3	1	0.188	0.194	0.231	0.183	0.194	0.226	0.183	0.194	0.226
4	2	0.224	0.245	0.312	0.220	0.240	0.319	0.223	0.240	0.319
5	3	0.264	0.304	0.385	0.251	0.299	0.383	0.256	0.299	0.383
6	4	0.309	0.344	0.459	0.299	0.344	0.453	0.301	0.344	0.453
7	5	0.346	0.389	0.543	0.340	0.384	0.540	0.341	0.384	0.540
8	6	0.374	0.433	0.625	0.370	0.432	0.610	0.373	0.432	0.616

Table D4. Analysis of variance (ANOVA) for water activity of instant flavoured green tea during storage

Ginger flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	1.01	20	0.05	138.43	< 0.0001	0.019	6.047
A-Storage period	0.77	6	0.13	351.80	< 0.0001		
B-Packaging	0.17	2	0.08	229.54	< 0.0001		
AB	0.07	12	0.01	16.56	< 0.0001		
Pure error	0.02	42	0.00				
Cor total	1.02	62					
Tulsi flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	1.01	20	0.050	140.694	< 0.0001	0.02	6.05
A-Storage period	0.76	6	0.126	352.085	< 0.0001		
B-Packaging	0.18	2	0.088	245.237	< 0.0001		
AB	0.08	12	0.006	17.575	< 0.0001		
Pure error	0.02	42	0.000				
Cor total	1.02	62					
Cardamom flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	1.01	20	0.05	142.08	< 0.0001	0.02	6.05
A-Storage period	0.75	6	0.13	353.46	< 0.0001	0.31	
B-Packaging	0.18	2	0.09	252.82	< 0.0001		
AB	0.08	12	0.01	17.93	< 0.0001		
Pure error	0.01	42	0.00				
Cor total	1.03	62					

Table D5 Effect of storage on Loose bulk density of instant flavoured green tea powder in different packaging materials

Sl. No.	Months	Ginger flavoured instant green tea			Cardamom flavoured instant green tea			Tulsi flavoured instant green tea		
1		ALF	PET	LDPE	ALF	PET	LDPE	ALF	PET	LDPE
2	0	0.259	0.259	0.259	0.259	0.259	0.259	0.259	0.259	0.259
3	1	0.282	0.286	0.304	0.280	0.284	0.301	0.281	0.2856	0.303
4	2	0.305	0.313	0.349	0.302	0.308	0.343	0.303	0.3122	0.347
5	3	0.326	0.339	0.393	0.323	0.335	0.388	0.325	0.3388	0.391
6	4	0.349	0.3669	0.437	0.344	0.362	0.432	0.347	0.3654	0.435
7	5	0.369	0.396	0.483	0.366	0.389	0.475	0.369	0.392	0.479
8	6	0.392	0.42	0.54	0.389	0.414	0.520	0.391	0.4186	0.523

Table D6 Analysis of variance (ANOVA) for Loose bulk density of instant flavoured green tea during storage

Ginger flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	0.34	20	0.02	40.65	< 0.0001	0.02	5.75
A-Storage period	0.25	6	0.04	102.36	< 0.0001		
B-Packaging	0.06	2	0.03	67.03	< 0.0001		
AB	0.03	12	0.00	5.39	< 0.0001		
Pure error	0.02	42	0.00				
Cor total	0.35	62					
Tulsi flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	0.31	20	0.02	38.39	< 0.0001	0.02	5.75
A-Storage period	0.24	6	0.04	97.98	< 0.0001		
B-Packaging	0.05	2	0.03	62.31	< 0.0001		
AB	0.02	12	0.00	4.62	0.0001		
Pure error	0.02	42	0.00				
Cor total	0.33	62					
Cardamom flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	0.31	20	0.02	37.93	< 0.0001	0.02	5.75
A-Storage period	0.23	6	0.04	96.36	< 0.0001		
B-Packaging	0.05	2	0.02	61.99	< 0.0001		
AB	0.02	12	0.00	4.71	< 0.0001		
Pure error	0.02	42	0.00				
Cor total	0.32	62					

Table D7 Effect of storage on tapped bulk density of instant flavoured green tea powder in different packaging materials

Sl. No.	Months	Ginger flavoured instant green tea			Cardamom flavoured instant green tea			Tulsi flavoured instant green tea		
1		ALF	PET	LDPE	ALF	PET	LDPE	ALF	PET	LDPE
2	0	0.337	0.337	0.337	0.337	0.337	0.337	0.337	0.337	0.337
3	1	0.360	0.364	0.382	0.358	0.362	0.379	0.359	0.363	0.381
4	2	0.383	0.391	0.427	0.380	0.386	0.421	0.381	0.390	0.425
5	3	0.403	0.416	0.470	0.400	0.412	0.465	0.402	0.416	0.468
6	4	0.427	0.445	0.515	0.422	0.440	0.510	0.425	0.443	0.513
7	5	0.447	0.474	0.561	0.444	0.467	0.553	0.447	0.470	0.557
8	6	0.470	0.498	0.618	0.467	0.492	0.598	0.469	0.496	0.601

Table D8 Analysis of variance (ANOVA) for tapped bulk density of instant flavoured green tea during storage

Ginger flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	0.34	20	0.02	27.70	< 0.0001	0.02	5.72
A-Storage period	0.26	6	0.04	69.79	< 0.0001		
B-Packaging	0.06	2	0.03	45.60	< 0.0001		
AB	0.03	12	0.00	3.67	0.0008		
Pure error	0.03	42	0.00				
Cor total	0.36	62					
Tulsi flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	0.31	20	0.02	26.09	< 0.0001	0.02	5.72
A-Storage period	0.24	6	0.04	66.62	< 0.0001		
B-Packaging	0.05	2	0.03	42.27	< 0.0001		
AB	0.02	12	0.00	3.13	0.0030		
Pure error	0.03	42	0.00				
Cor total	0.34	62					
Cardamom flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	0.31	20	0.02	25.71	< 0.0001	0.02	5.71
A-Storage period	0.23	6	0.04	65.35	< 0.0001		
B-Packaging	0.05	2	0.02	41.94	< 0.0001		
AB	0.02	12	0.00	3.18	0.0026		
Pure error	0.02	42	0.00				
Cor total	0.33	62					

Table D9 Effect of storage on colour change of instant flavoured green tea powder in different packaging materials

Sl. No.	Months	Ginger flavoured instant green tea			Cardamom flavoured instant green tea			Tulsi flavoured instant green tea		
1		ALF	PET	LDPE	ALF	PET	LDPE	ALF	PET	LDPE
2	0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3	1	0.516	3.123	5.876	0.341	3.862	6.384	0.513	2.293	3.863
4	2	0.932	7.864	10.985	0.642	8.932	11.942	0.912	5.756	7.925
5	3	1.346	11.865	18.984	1.047	13.952	16.210	1.562	9.826	12.847
6	4	1.903	15.238	25.962	1.517	17.346	21.952	2.640	13.625	19.017
7	5	2.543	18.974	31.863	1.848	20.394	28.931	3.756	16.842	25.260
8	6	2.913	21.473	38.180	2.159	24.793	34.875	4.469	21.620	33.348

Table D10 Analysis of variance (ANOVA) for colour change of instant flavoured green tea during storage

Ginger flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	7901.00	20	395.05	553.90	< 0.0001	0.84	8.04
A-Storage period	3191.88	6	531.98	745.89	< 0.0001		
B-Packaging	3189.80	2	1594.90	2236.22	< 0.0001		
AB	1519.32	12	126.61	177.52	< 0.0001		
Pure error	29.95	42	0.71				
Cor total	7930.95	62					

Tulsi flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	2627.64	20	131.38	484.33	< 0.0001	0.52	7.54
A-Storage period	1287.25	6	214.54	790.88	< 0.0001		
B-Packaging	975.33	2	487.66	1797.72	< 0.0001		
AB	365.06	12	30.42	112.15	< 0.0001		
Pure error	11.39	42	0.27				
Cor total	2639.03	62					

Cardamom flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	7107.56	20	355.38	539.82	< 0.0001	0.81	7.85
A-Storage period	2921.26	6	486.88	739.57	< 0.0001		
B-Packaging	2907.34	2	1453.67	2208.14	< 0.0001		
AB	1278.97	12	106.58	161.90	< 0.0001		
Pure error	27.65	42	0.66				
Cor total	7135.21	62					

Table D11 Effect of storage on solubility of instant flavoured green tea powder in different packaging materials

Sl. No.	Months	Ginger flavoured instant green tea			Cardamom flavoured instant green tea			Tulsi flavoured instant green tea		
1		ALF	PET	LDPE	ALF	PET	LDPE	ALF	PET	LDPE
2	0	84.340	84.340	84.340	86.640	86.640	86.640	86.240	86.240	86.240
3	1	84.190	84.012	83.530	86.600	86.500	85.810	86.140	86.000	85.410
4	2	83.780	83.530	82.830	86.150	85.800	85.110	85.750	85.330	84.610
5	3	83.700	83.340	82.870	86.120	85.740	85.127	85.670	85.140	84.627
6	4	83.540	83.170	82.120	85.930	85.770	84.320	85.490	85.070	83.820
7	5	83.520	82.920	81.340	85.860	85.040	83.510	85.432	84.830	83.010
8	6	83.210	82.020	80.130	85.600	84.120	82.213	85.100	83.820	81.713

Table D12 Analysis of variance (ANOVA) for solubility of instant flavoured green tea during storage

Ginger flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	67.02	20	3.35	0.15	1.0000	4.71	5.66
A-Storage period	38.49	6	6.41	0.29	0.9387		
B-Packaging	18.57	2	9.28	0.42	0.6605		
AB	9.97	12	0.83	0.04	1.0000		
Pure error	930.73	42	22.16				
Cor total	997.75	62					

Tulsi flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	79.53	20	3.98	0.17	1.0000	4.81	5.66
A-Storage period	43.38	6	7.23	0.31	0.9271		
B-Packaging	24.07	2	12.04	0.52	0.5985		
AB	12.07	12	1.01	0.04	1.0000		
Pure error	972.76	42	23.16				
Cor total	1052.3	62					

Cardamom flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	78.35	20	3.92	0.17	1.0000	4.84	5.66
A-Storage period	43.50	6	7.25	0.31	0.9284		
B-Packaging	23.08	2	11.54	0.49	0.6142		
AB	11.77	12	0.98	0.04	1.0000		
Pure error	983.17	42	23.41				
Cor total	1061.6	62					

Table D13 Effect of storage on wettability of instant flavoured green tea powder in different packaging materials

Sl. No.	Months	Ginger flavoured instant green tea			Cardamom flavoured instant green tea			Tulsi flavoured instant green tea		
1		ALF	PET	LDPE	ALF	PET	LDPE	ALF	PET	LDPE
2	0	152	152	152	149	149	149	150	150	150
3	1	141	135	120	143	135	123	140	133	120
4	2	130	126	102	134	126	107	133	125	100
5	3	119	109	82	124	109	82	119	110	92
6	4	108	98	80	111	98	79	110	96	80
7	5	97	82	76	99	83	76	97	86	76
8	6	81	78	62	79	72	59	80	76	60

Table D14. Analysis of variance (ANOVA) for wettability of instant flavoured green tea during storage

Ginger flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	47558	20	2377.90	58.65	< 0.0001	6.37	5.86
A-Storage period	40746	6	6791.00	167.50	< 0.0001		
B-Packaging	5322.2857	2	2661.14	65.64	< 0.0001		
AB	1489.7143	12	124.14	3.06	0.0036		
Pure error	1702.8632	42	40.54				
Cor total	49260.863	62					

Tulsi flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	45318.86	20	2265.94	55.97	< 0.0001	6.36	5.85
A-Storage period	39102.86	6	6517.14	160.96	< 0.0001		
B-Packaging	5030.57	2	2515.29	62.12	< 0.0001		
AB	1185.43	12	98.79	2.44	0.0164		
Pure error	1700.52	42	40.49				
Cor total	47019.37	62					

Cardamom flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	49075.71	20	2453.79	60.07	< 0.0001	6.39	5.87
A-Storage period	41589.71	6	6931.62	169.70	< 0.0001		
B-Packaging	5827.71	2	2913.86	71.34	< 0.0001		
AB	1658.29	12	138.19	3.38	0.0016		
Pure error	1715.58	42	40.85				
Cor total	50791.29	62					

Table D15 Effect of storage on total polyphenols of instant flavoured green tea powder in different packaging materials

Sl. No.	Months	Ginger flavoured instant green tea			Cardamom flavoured instant green tea			Tulsi flavoured instant green tea		
1		ALF	PET	LDPE	ALF	PET	LDPE	ALF	PET	LDPE
2	0	44.74	44.74	44.74	45.6	45.60	45.60	45.59	45.59	45.59
3	1	44.41	43.41	42.76	44.57	43.67	42.66	45.26	43.76	42.86
4	2	42.86	40.64	39.64	42.72	41.73	40.89	45.11	42.69	42.16
5	3	42.41	39.41	38.91	42.68	40.64	39.05	44.34	41.54	38.42
6	4	40.41	37.91	35.41	41.38	38.07	31.40	43.6	39.43	37.30
7	5	39.41	33.41	30.41	39.73	34.95	27.18	42.93	37.93	33.32
8	6	37.41	29.91	23.91	37.16	30.91	21.92	41.49	34.38	30.38

Table D16 Analysis of variance (ANOVA) for total polyphenol of instant flavoured green tea during storage

Ginger flavoured instant green tea

Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	1876.17	20	93.81	38.85	< 0.0001	1.55	3.99
A-Storage period	1396.17	6	232.70	96.37	< 0.0001		
B-Packaging	281.04	2	140.52	58.20	< 0.0001		
AB	198.96	12	16.58	6.87	< 0.0001		
Pure error	101.41	42	2.41				
Cor total	1977.58	62					

Tulsi flavoured instant green tea

Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	1150.10	20	57.50	22.7	< 0.0001	1.60	3.90
A-Storage period	683.32	6	113.89	45.0	< 0.0001		
B-Packaging	318.60	2	159.30	63.0	< 0.0001		
AB	148.17	12	12.35	4.9	< 0.0001		
Pure error	106.25	42	2.53				
Cor total	1256.34	62					

Cardamom flavoured instant green tea

Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	2458.97	20	122.949	63.01	< 0.0001	1.39	3.58
A-Storage period	1680.45	6	280.074	143.53	< 0.0001		
B-Packaging	442.11	2	221.055	113.28	< 0.0001		
AB	336.42	12	28.0349	14.37	< 0.0001		
Pure error	81.96	42	1.95135				
Cor total	2540.93	62					

Table D17 Effect of storage on total flavonoid of instant flavoured green tea powder in different packaging materials

Sl. No.	Months	Ginger flavoured instant green tea			Cardamom flavoured instant green tea			Tulsi flavoured instant green tea		
1		ALF	PET	LDPE	ALF	PET	LDPE	ALF	PET	LDPE
2	0	27.57	21.57	21.57	26.97	26.97	26.97	28.39	28.39	28.39
3	1	27.57	27.27	26.50	26.88	26.59	26.34	28.04	27.63	27.61
4	2	27.51	26.81	25.96	26.81	26.36	26.02	27.53	27.13	26.50
5	3	27.28	26.33	25.53	26.38	25.84	25.48	27.02	26.75	25.12
6	4	26.70	25.64	24.46	26.18	25.51	23.41	26.81	26.23	24.88
7	5	26.20	25.11	22.74	26.11	25.14	21.91	26.02	25.76	23.03
8	6	26.00	24.61	20.65	25.75	24.76	19.81	25.82	25.24	21.92

Table D18 Analysis of variance (ANOVA) for total flavonoid of instant flavoured green tea during storage

Ginger flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	185.06	20	9.25	9.39	< 0.0001	0.99	3.82
A-Storage period	102.36	6	17.06	17.31	< 0.0001		
B-Packaging	52.45	2	26.22	26.62	< 0.0001		
AB	30.25	12	2.52	2.56	0.0122		
Pure error	41.38	42	0.99				
Cor total	226.44	62					

Tulsi flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	172.995	20	8.65	8.09	< 0.0001	1.03	3.91
A-Storage period	115.838	6	19.31	18.06	< 0.0001		
B-Packaging	35.442	2	17.72	16.58	< 0.0001		
AB	21.7152	12	1.81	1.69	0.1032		
Pure error	44.8893	42	1.07				
Cor total	217.884	62					

Cardamom flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	195.536	20	9.78	10.00	< 0.0001	0.98	3.87
A-Storage period	90.4778	6	15.08	15.42	< 0.0001		
B-Packaging	53.0275	2	26.51	27.11	< 0.0001		
AB	52.0308	12	4.34	4.43	0.0001		
Pure error	41.0813	42	0.98				
Cor total	236.617	62					

Table D19 Effect of storage on caffeine content of instant flavoured green tea powder in different packaging materials

Sl. No.	Months	Ginger flavoured instant green tea			Cardamom flavoured instant green tea			Tulsi flavoured instant green tea		
1		ALF	PET	LDPE	ALF	PET	LDPE	ALF	PET	LDPE
2	0	15.06	15.06	15.06	15.142	15.142	15.142	15.11	15.11	15.11
3	1	15.06	15.06	15.06	15.142	15.142	15.142	15.11	15.11	15.11
4	2	15.06	15.06	15.03	15.112	15.112	15.042	15.11	15.11	15.01
5	3	15.05	15.02	14.96	15.062	15.022	14.992	15.01	14.91	14.96
6	4	15.01	15.01	14.94	15.032	14.972	14.942	14.96	14.89	14.81
7	5	14.96	14.94	14.94	14.942	14.942	14.842	14.91	14.81	14.81
8	6	14.96	14.94	14.94	14.942	14.942	14.842	14.91	14.81	14.81

Table D20 Analysis of variance (ANOVA) for caffeine content of instant flavoured green tea during storage

Ginger flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	0.161	20	0.008	0.024	1.000	0.57	3.83
A-Storage period	0.136	6	0.023	0.069	0.999		
B-Packaging	0.011	2	0.006	0.017	0.983		
AB	0.013	12	0.001	0.003	1.000		
Pure error	13.93	42	0.332				
Cor total	14.09	62					
Tulsi flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	0.91	20	0.046	0.153	1	0.54	3.64
A-Storage period	0.81	6	0.134	0.451	0.8404		
B-Packaging	0.06	2	0.029	0.097	0.9079		
AB	0.05	12	0.004	0.014	1		
Pure error	12.51	42	0.298				
Cor total	13.42	62					
Cardamom flavoured instant green tea							
Source	Sum of squares	df	Mean square	F value	p- value Prob > F	Std. Dev.	C.V. (%)
Model	0.61	20	0.031	0.10	1	0.56	3.77
A-Storage period	0.54	6	0.090	0.28	0.9426		
B-Packaging	0.04	2	0.022	0.07	0.9348		
AB	0.03	12	0.002	0.01	1		
Pure error	13.50	42	0.321				
Cor total	14.11	62					

Appendix E

Table E1. Water activity and moisture content of ginger flavoured instant green tea in three packaging material at accelerated storage condition

	ALF		PET		LDPE	
Time of Storage (days)	Water activity	Moisture content (kg water.kg dry solids^{-1})	Water activity	Moisture content (kg water.kg dry solids^{-1})	Water activity	Moisture content (kg water.kg dry solids^{-1})
0	0.15	0.0308	0.150	0.0309	0.15	0.0303
5	0.323	0.0665	0.420	0.0929	0.51	0.1110
10	0.423	0.0888	0.473	0.1219	0.670	0.1450
15	0.510	0.1063	0.540	0.1419	0.750	0.1700
20	0.632	0.1271	0.667	0.1629	0.85	0.1930
25	0.684	0.1353	0.702	0.1729	0.91	0.2060
30	0.703	0.1438	0.735	0.1849	0.940	0.2220
35	0.735	0.1480	0.832	0.1929	0.978	0.2270

Table E2. Water activity and moisture content of cardamom flavoured instant green tea in three packaging material at accelerated storage condition

	ALF		PET		LDPE	
Time of Storage (days)	Water activity	Moisture content (kg water.kg dry solids^{-1})	Water activity	Moisture content (kg water.kg dry solids^{-1})	Water activity	Moisture content (kg water.kg dry solids^{-1})
0	0.15	0.0300	0.15	0.0303	0.15	0.0305
5	0.319	0.0710	0.350	0.0813	0.430	0.0905
10	0.404	0.0890	0.443	0.0983	0.570	0.1245
15	0.499	0.1100	0.533	0.1223	0.713	0.1535
20	0.583	0.1260	0.635	0.1373	0.792	0.1695
25	0.650	0.1390	0.671	0.1463	0.850	0.1855
30	0.692	0.1500	0.729	0.1573	0.918	0.1975
35	0.735	0.1580	0.783	0.1683	0.957	0.2095

Table E3. Water activity and moisture content of cardamom flavoured instant green tea in three packaging material at accelerated storage condition

	ALF		PET		LDPE	
Time of Storage (days)	Water activity	Moisture content (kg water.kg dry solids^{-1})	Water activity	Moisture content (kg water.kg dry solids^{-1})	Water activity	Moisture content (kg water.kg dry solids^{-1})
0	0.15	0.0303	0.15	0.0303	0.15	0.0303
5	0.356	0.0719	0.418	0.0821	0.457	0.0900
10	0.443	0.0894	0.466	0.0993	0.624	0.1288
15	0.515	0.1034	0.534	0.117	0.774	0.1583
20	0.615	0.1214	0.653	0.134	0.841	0.1680
25	0.668	0.1359	0.699	0.1403	0.891	0.1809
30	0.710	0.1449	0.732	0.1536	0.934	0.1876
35	0.743	0.1567	0.825	0.1642	0.970	0.1950

Table E4. Dimension of packaging material used for shelf life prediction

Packaging material	Length	Breadth
ALF	0.08	0.06
PET	0.07	0.06
LDPE	0.13	0.08

Appendix F

MATLAB program shelf life prediction of ginger flavoured instant green tea packed in laminated aluminium foil pouches

```
format short e
global C K M Wp bp lp kg Pp Rhp
aw=[0.15, 0.373, 0.498, 0.580, 0.680, 0.744, 0.750, 0.803];
X=[0.0308, 0.0838, 0.1088, 0.1278, 0.1498, 0.1598, 0.1668, 0.1748];
y=aw./X;
b=polyfit(aw,y,2);
A=(b(2)^2-4*b(3)*b(1))^0.5;
K=(-b(2)+A)/(2*b(3))
M=(b(2)+2*b(3)*K)^-1
C=(b(3)*M*K)^-1
Xc=(M*C*K*aw)./((1-K*aw).*(1-K*aw+C*K*aw));
plot(aw,X,'o-',aw,Xc,'x-')
xlabel('Water Activity of powder fraction');
ylabel('Moisture Content of powder, kg water/kg dry solid');
gtext('Experimental')
gtext('Predicted from GAB equation')
[m,m1]=size(aw);
rd=0;
for i=1:m
rd1(i)=abs(X-Xc)/X;
rd=rd+rd1(i);
end
Rel_dev_percent_for_MC=100*rd/m
Xs=[0.0 0.010 0.020 0.030 0.040 0.050 0.060 0.070];
Ts=[68 64 60 55 51 49 48 46];
plot(Ts,Xs,'o-')
xlabel('Storage Temperature,oC');
ylabel('Sticky Point MC,kg water/kg dry solid');
aa=polyfit(Ts,Xs,1);
Tp=40;
XPc=polyval(aa,Tp);
sticklypoint_moisture_content=XPc
lg=0.08;
```

```
bg=0.125;
Tg=38;
Rhg=0.9;
Pg=exp(23.0603-3723.67/(222.857+Tg));
theTag1=[0 0.96 2 2.46 3 3.48 4.63];
theTag=3600*24*theTag1;
wg1=[5.032 5.052 5.072 5.081 5.107 5.120 5.138];
wg=1e-3*wg1;
plot(theTag,wg,'x-')
xlabel('Time of Moisture Permeation,s');
ylabel('Weight of Silica gel kept inside package,kg')
S=polyfit(theTag,wg,1);
kg=S(1)*(2*bg*lg*Pg*Rhg)^-1;
permiability_kgwater_per_sqm_S_Pa=kg
bp=0.06;
lp=0.08;
wp=0.04;
xi=0.03;
Xi=xi/(1-xi);
Wp=wp/(1+xi);
Rhp=0.90;
Pp=exp(23.0603-3723.67/(222.857+Tp));
Xspan=[Xi XPc];
thetai=0;
[Xp,theta]=ode45('sf',Xspan,thetai);
plot(theta/(24*3600),Xp,'-')
xlabel('Storage Time, day');
ylabel('Moisture Content of Powder, kg of water/kg dry solid');
sf=interp1(Xp,theta,XPc,'cubic');
Shelflife_day=sf/(24*3600);
```

Appendix G

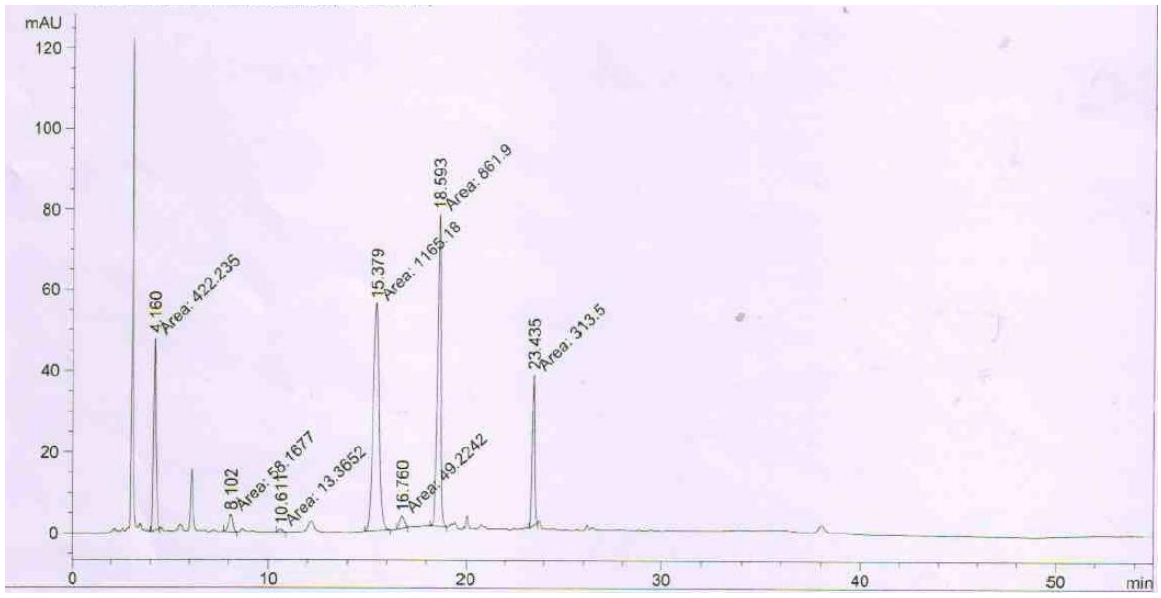

Fig. G1 Chromatogram for instant green tea

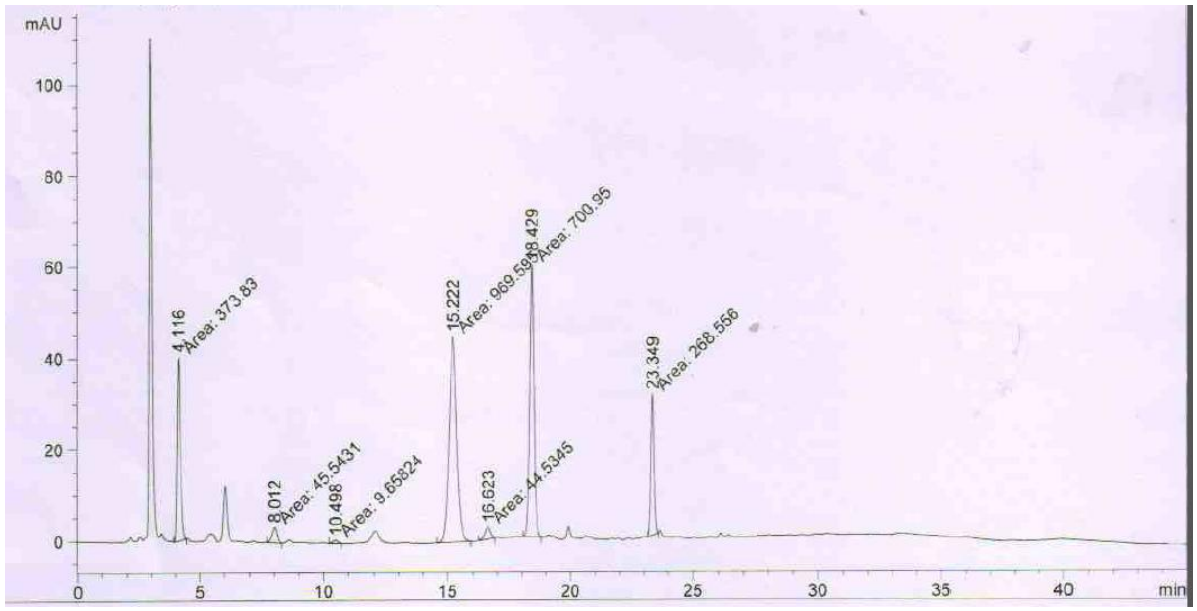

Fig. G2 Chromatogram for ginger flavoured instant green tea

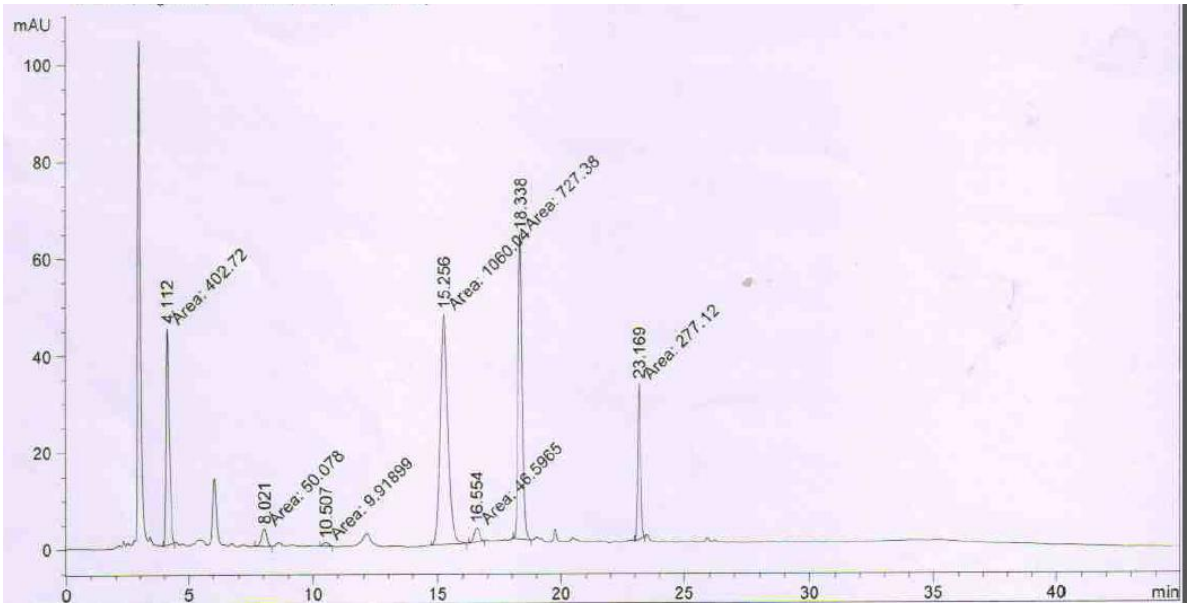

Fig. G3 Chromatogram for cardamom flavoured instant green tea

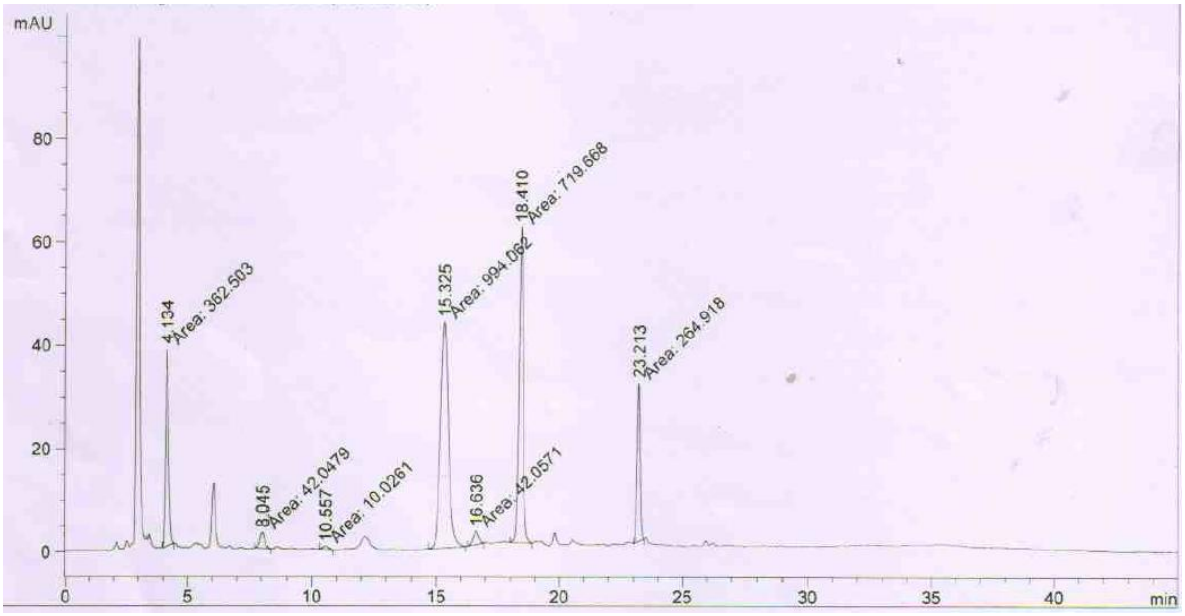

Fig. G4 Chromatogram for tulsi flavoured instant green tea

Appendix H

Computation of heat utilization efficiency of spray dryer during drying of green tea extract droplets using MATLAB

Program in MATLAB	**Legend and program output**
Dd=0.367;	Diameter of drying chamber, Dd (m)
Ld=0.91;	Length of drying chamber, Ld(m)
Db=0.056;	Diameter of pipe at blower outlet, Db (m)
d*nozzle* = 8.6e-04	Diameter of nozzle, d*nozzle* (m)
Van = 22.138	Velocity of air at nozzle outlet, Van (m/s)
Qa=(pi/4)*(d*nozzle*)^2*Van	Volumetric flow rate of air from the nozzle, Qa (m3/s), Ans = 1.28e-005
Ta=34;	Dry bulb temperature of atmospheric air, Ta (°C)
Taw=30.02;	Wet bulb temperature of atmospheric air, Taw (°C)
Th=174;	Inlet air temperature, Th (°C)
vb = pi*Db*2100/60	Velocity of air at blower outlet, Tb (Blower rpm- 2100), 5.86 m/s
Tb=57.8;	Temperature of air at blower outlet, Tb (°C)
Xm=0.07;	Concentration of solids in green tea extract, Xm,
Mw=0.194e-3;	Maximum possible water flow rate into drying chamber, kg/s
Mm=0.175e-3;	Maximum possible green tea extract

	flow rate into drying chamber, kg/s
Tf=65;	Temperature of air at the bottom of drying chamber during water evaporation,°C
Tfm = 74;	Temperature of air at the bottom of drying chamber during green tea extract drying, °C
Pa=101325;	Atmospheric pressure of air, Pa
ps=exp(23.0603-3723.67/(222.857+Taw));	Saturation vapour pressure of water at wet bulb temperature of atmospheric air, Ans: 4.167e+003
Yaw=(18/28.92)*ps/(Pa-ps);	Saturation absolute humidity of atmospheric air, Ans : 0.0266
lamda=2501000+1880*Taw-4186*Taw;	Latent heat of vapourization of water wet bulb temperature of atmospheric air; Ans: 2.4317e+006
A=Yaw*lamda-(Ta-Taw)*1005;	A = 6.0683e+004
B=1880*(Ta-Taw)+lamda;	B = 2.43918e+006
Ya=A/B	Absolute humidity of atmospheric air,Ya; Ans = 0.0.250
Va=(0.00283+0.00456*Ya)*(Ta+273)	Humid volume of atmospheric air,Va; Ans = 0.9038
Ma=Qa/Va;	Mass flow rate of atomizing air, Ma; Ans = 1.4162e-005
Mass rate atomising air=Ma	
A=(pi/4)*(Db^2)*vb;	Ans; 0.00596
B=Mw*(Tb+273);	Ans; 0.06411
Yb=(0.00283*B+A*Ya)/(A-0.00456*B)	Absolute humidity of air leaving the dryer

	= 0.0383 kg of water. kg dry air^{-1}
Vb=(0.00283+0.00456*Yb)*(Tb+273);	Net mass flow of air that leaves the dryer = 0.045 kg.s^{-1}
Mb=A/Vb;	
Net mass rate of air=Mb	
Ha=(1005+1880*Ya)*Ta+2501000*Ya;	Enthalpy of ambient air; Ans = 9.8293e+004
Hh=(1005+1880*Ya)*Th+2501000*Ya;	Enthalpy of heated air: Ans = 2.45573e+005
A=Ma*Ha+(Mb-Ma)*Hh;	Ans = 1.1048e+004
B=A/Mb-2501000*Ya;	Ans = 1.82986e+005
Tx=B/(1005+1880*Ya);	Temperature of mixed air at atomizer outlet, T_x =173.863°C
Mixed air temperature=Tx	
pa=28.9*Ya*Pa/(28.9*Ya+18);	Partial pressure of water vapour in atmospheric air: pa = 3.910e+003
ps=exp(23.0603-3723.67/(222.857+Tx));	Saturation vapour pressure of water at temperature Tx of mixed air; ps = 8.68183e+005
Rhx=pa/ps;	Relative humidity of mixed air at atomizer outlet, Rhx= 0.0045
Air relative humidity before drying= Rhx	
global Y T	Wet bulb temperature of mixed air at dry bulb temperature, Tx and absolute humidity; Txw= 48.8218 °C
T=Tx;	
Y=Ya;	
Txw=48.8218:	
Wetbulb temperature of mixed air=Txw	
pf=28.9*Yb*Pa/(28.9*Yb+18);	Partial pressure of water vapour in air

	at bottom of drying chamber; pf = 5.8698e+003
pfs=exp(23.0603-3723.67/(222.857+Tf));	Saturaton vapour pressure of water at temperature Tf of air, pfs ; 2.4945e+004
Rhf=pf/pfs;	Relative humidity of air at the bottom of drying chamber during evaporation of water droplets, Rhf = 0.235
Relative humidity after drying=Rhf	
ps=exp(23.0603-3723.67/(222.857+Txw));	Saturation vapour pressure of water at wet bulb temperature, Txw of mixed air; ps= 11.546e+003
Yxw=(18/28.92)*ps/(Pa-ps);	Saturation absolute humidity of air at tempeature, Txw.; Yxw = 0.0800
Rw=1/(Yxw-Ya);	Ratio of actual dry air flow rate and maximum possible water evaporation rate under adiabatic condition; Rw = 18.157
Mass_ratio_adiabatic=Rw	
Rwn=Mb/Mw	Ratio of dry air low rate and water evaporation rate under non-adiabatic condition ; Rwn = 74.9171
Mass ratio nonadiabatic=Rwn	
lamda=2501000+1880*Txw-4186*Txw;	Latent heat of vaporization of water at wet bulb temperature Txw of mixed air = 2.388416 e+006
Hx=(1005+1880*Ya)*Tx+2501000*Ya;	Enthalpy of mixed air temperature Tx; Hx = 2.45428e+005
Ha=(1005+1880*Ya)*Ta+2501000*Ya;	Enthalpy of atmospheric air at temperature Ta; Ha = 9.8293e+004
eta=(Yxw-Ya)*lamda/(Hx-Ha);	Heat utilization efficiency during evaporation of water droplets under adiabatic condition $\eta_w = 0.894$
Adiabatic efficiency=eta	
etan=Mw*lamda/(Mb*(Hx-Ha));	Heat utilization efficiency under

Nonadiabatic efficiency=etan	drying condition, η_{wn} = 0.2167
Drying of green tea extract	
Mwm=Mm*(1-Xm);	Rate of water evaporation from green tea extract droplets, Mwm = 1.627e-004
Water evaporationrate=Mwm	
Ybm=Mwm/Mb+Ya	Absolute humidity of air leaving the dryer; Ybm = 0.0286
pm=28.9*Ybm*Pa/(28.9*Ybm+18);	Partial pressure of water vapour in the bottom of drying chamber; pm= 4.4484e+003
ps=exp(23.0603-3723.67/(222.857+Tfm));	Saturated vapour pressure of water at temperature Tfm of air measured at the bottom of drying chamber; ps = 3.6925e+004
Rhm=pm/ps;	Relative humidity of air at the lower end of drying chamber,; Rhm = 0.2354
Relative humidity after drying=Rhf	
Rm=Mb/Mwm;	Ratio of dry air flow rate and water evaporation rate from green tea extract under non-adiabatic concition; Rm= 89.302
Mass_ratio_nonadiabatic=Rm	
η_m = Mw *lamda/(Mb*(Hx - Ha)	Heat utalization efficiency of dryer when green tea exctract is dried; η_m = 0.1818 (i.e., 18.18%)

Appendix I

Cost economics of instant flavoured green tea

Cost of machineries		
Cost of spray dryer	=	Rs.12,00,000/-
Cost of induction stove	=	Rs.3,500/-
Cost of sealing machine	=	Rs.4,000/-
Floor space 5 m^2		1,00,000
Miscellaneous item	=	Rs.10,000/-
Total cost	=	Rs.13,17,500/-

Assumptions

Life span (L)	=	10 years
Annual working hours (H)	=	275days (per day 24 hrs) = 6600 hours
Salvage value (S)	=	10% of initial cost
Interest on initial cost (i)	=	15% annually
Repair and maintenance	=	10% of initial cost
Insurance and taxes	=	2% of initial cost
Electricity charge	=	Rs.7/unit
Labour wages/person	=	Rs 400/day

1. Total fixed cost per year		
i. Depreciation	=	$\frac{C-S}{L\times H}=\frac{13,17,500-13,750}{10\times 6600}=$ Rs.19.75/h
ii. Interest	=	$\frac{C+S}{2}\times\frac{i}{H}$ $\frac{13,17,500+13,175}{2}\times\frac{15}{100\times 6600}$ = Rs.15.12/h
iii. Insurance & taxes	=	2% of initial cost $\frac{2}{100\times 6600}\times 13,17,500$ = Rs.3.99/h
Total fixed cost	=	i + ii + iii = 38.86/h =Rs. 2,56,476/-

2. Total variable cost per year		
i. Repair & maintenance		10% of initial cost $=\frac{10}{100\times 2200}\times 13,17,500$ = Rs 19.96/h =1,31,750/-
ii. Electricity cost		
a) Energy consumed by the spray dryer	=	5.8 kwh
b) Energy consumed by induction, sealing machine and weighing balance	=	0.2 kwh
Total energy consumption	=	6.0 kwh
Cost of energy consumption/h	=	Power × duration × cost of 1 unit 6×6600×7= Rs. 2,77,200

iii. Labour cost (1 persons)	= =	Rs.350 Rs. 2,88,750
iv. Packaging cost	= =	Rs 200/day Rs. 55,000
v. Cost of raw materials for instant flavoured green tea		
Quantity of green tea leaves required Cost of green tea leaves Quantity of maltodextrin required Cost of maltodextrin Cost major raw materials	= = = = = = =	375 g/day Rs. 1000/ kg Rs. 1,03,125 450 g/day Rs. 100/kg Rs. 12,375 Rs. 1,15,500/-
vi. Cost of Flavouring materials A) Quantity of ginger required Cost of ginger B) Quantity of Cardamom required Cost of cardamom C) Quantity of tulsi required Cost of tulsi	 = = = = = = = =	 900 g/day Rs. 100/ kg Rs. 24,750/- 360 g/day Rs.1200/kg Rs. 1,18,800/- 75 g/day Rs.500/kg Rs. 10,314/-

Total variable cost for instant green tea	=	i + ii + iii + iv + v Rs. 8,68,200/-
Total cost for production of one kg of instant green tea powder	=	Fixed cost + Variable cost = 2,56,476+8,68,200 = 11,24,676/-

		=Rs. 4,089/kg
Total variable cost for ginger flavoured instant green tea	=	i + ii + iii + iv + v+vi(A) = Rs. 8,92,950/-
Total cost for production of one kg of ginger flavoured instant green tea powder	=	Fixed cost + Variable cost = 2,56,476+8,92,950 = 11,49,426/- =Rs. 4,179/g
Total variable cost for cardamom flavoured instant green tea	=	i + ii + iii + iv + v+vi(B) Rs.9,87,000 /-
Total cost for production of one kg of cardamom flavoured instant green tea powder	=	Fixed cost + Variable cost = 2,56,476+9,87,000 = 12,43,476/- =Rs. 4,521 /g
Total variable cost for tulsi flavoured instant green tea	=	i + ii + iii + iv + v+vi(A) = Rs. 8,78,514/-
Total cost for production of one kg of tulsi flavoured instant green tea powder	=	Fixed cost + Variable cost = 2,56,476+8,78,514 = 11,34,900/- =Rs. 4,127/kg

- The market selling price of one kilogram of instant green tea is Rs. 5,500/-

$$\text{Benefit -cost ratio} = \frac{5,500}{4,089} = 1.35$$

- The market selling price of one kilogram of ginger flavoured instant green tea is Rs. 6,000/-

$$\text{Benefit -cost ratio} = \frac{6000}{4,179} = 1.43$$

- The market selling price of one kiligram of cardamom flavoured instant green tea is Rs. 6,000/-

$$\text{Benefit -cost ratio} = \frac{6000}{4,521} = 1.33$$

- The market selling price of one kiligram of tulsi flavoured instant green tea = Rs. 6,000/g

$$\text{Benefit -cost ratio} = \frac{6000}{4{,}127} = 1.45$$

OPTIMISATION OF PROCESS PARAMETERS FOR PREPARATION OF FLAVOURED INSTANT GREEN TEA

by

SANKALPA K. B.
(2014-28-104)

ABSTRACT OF THE THESIS

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IN

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(Agricultural Processing and Food Engineering)

Faculty of Agricultural Engineering & Technology

Kerala Agricultural University

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2017

Abstract

Tea is the second most popular drink in the world after water. Tea can be classified into three major categories: unfermented green tea, partially fermented oolong tea, and fermented black tea. Green tea contains more catechins, than black tea and oolong tea. So it is included in the group of beverages with functional properties. Green tea consumption helps in preventing cardiovascular diseases, cancer, diabetes and obesity. Nowadays, demand is increasing for instant tea, decaffeinated tea and flavoured tea. The use of instant tea powder will reduce the preparation time to a large extent and saves energy as well. Due to these reasons efforts were under taken for optimisation of process parameters for preparation of flavoured instant green tea. Production of flavoured instant green tea mainly includes three steps, extraction, flavour addition and drying. Extraction was performed and optimised conditions were 1:47 leaf-water ratio, 30 min extraction time and 52°C extraction temperature. Extract obtained with optimised condition was subjected to spray drying and optimised condition were 174°C inlet temperature, 2.7% MD and 671 $ml.h^{-1}$ feed rate to produce instant green tea. Further flavouring was carried out with two spices (ginger and cardamom) and one herb (tulsi) of different concentration and spray dried at optimised spray drying condition. The best combination selected after sensory evaluation are G2 (4% ginger extract), C6 (3 g of cardamom with 30 min of soaking) and H3 (3:10 tulsi to green tea ratio, 30 min extraction time). The best samples were packed in PET, ALF and LDPE and kept for storage studies at room temperature. Predicted shelf-life period of instant flavoured green tea powder packaged in ALF, PET and LDPE based on moisture gain was found to be 210, 152 and 92 days, respectively. Among the six tested sorption models, the GAB model described the best fit to the experimental data with higher R^2 value and lowest SSE and RMSE. The HPLC analysis for catechin fraction of instant flavoured green tea indicates that, flavour addition will not affect the green tea catechins. The heat utilization efficiency of spray dryer for spray drying of green tea extract under non adiabatic condition was 18.18% and under adiabatic condition was 89.4%. The total cost of production for

1 kg of instant green tea was Rs. 3590/-, ginger flavoured instant green tea was Rs. 3683/-, cardamom flavoured instant green tea was 4025/- and tulsi flavoured instant green tea was Rs. 3631/- and benefit cost ratio was 1.40:1, 1.63:1, 1.49:1 and 1.65:1, respectively.