## DEVELOPMENT OF NEERA POWDER USING SPRAY DRYING PROCESS

by

ANJALI A V (2017-18-002)



# DEPARTMENT OF PROCESSING AND FOOD ENGINEERING KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY TAVANUR - 679573, MALAPPURAM KERALA, INDIA

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#### **THESIS**

Submitted in partial fulfilment of the requirement for the degree of MASTER OF TECHNOLOGY

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Kerala Agricultural University



## DEPARTMENT OF PROCESSING AND FOOD ENGINEERING KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY TAVANUR - 679573, MALAPPURAM KERALA, INDIA

2019

#### **DECLARATION**

I hereby declare that this thesis entitled "Development of Neera powder using spray drying process" 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.

Tavanur	Anjali A V
Date:	(2017-18-002)

#### **CERTIFICATE**

Certified that this thesis entitled "Development of Neera powder using spray drying process" is a *bonafide* record of research work done independently by Mrs. Anjali A V 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.

Tavanur	
Date:	Dr. Santhi Mary Mathew
Date.	(Chairman, Advisory Board

#### **CERTIFICATE**

We, the undersigned members of the advisory Committee of Mrs. Anjali A V (2017-18-002), a candidate for the degree of Master of Technology in Agricultural Engineering majoring in Agricultural Processing and Food Engineering, agree that the thesis entitled "Development of Neera powder using spray drying process" may be submitted by Mrs. Anjali A V (2017-18-002), in partial fulfillment of the requirement for the degree.

Dr. Santhi Mary Mathew

(Chairman)

Retd. Professor

KCAET, Tavanur.

Dr. Prince M.V

(Member)

Department of Processing and

Food Engineering,

KCAET, Tavanur.

Smt. Sreeja. R

(Member)

**Assistant Professor** 

Department of Processing and Food

Engineering,

KCAET, Tavanur.

Er. Shivaji K. P

(Member)

**Assistant Professor** 

Department of Farm Machinery

and Power Engineering,

KCAET, Tavanur.

(Member)

Dr. M.R. Manikantan

(Member)

Principal scientist and

RE/PI, AICRP on PHET

ICAR-CPCRI, Kasaragod.

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#### SYMBOLS AND ABBREVIATIONS

et al. : and others

% : per cent

& : and / : per

! less than! greater than

 $\pm$ : Plus or minus sign

° degree

°B : Degree brix °C : degree celsius

a\* : Greenness or redness

Al : Aluminium

ALPE: Aluminium laminated

polyethylene

AOAC : Association of analytical chemist

b\* : Blueness or yellowness C.V : Coefficient of variation

Ca : Calcium Cu : Copper

df: Degree of freedom

etc. : etcetera
Fe : Iron
Fig : Figure
g : gram

g/ml : Gram per mililiters

h: Hour

H2SO4 : Sulphuric acid HCL : Hydrochloric acid

HDPE : High density polyethylene

HG: Horse gram extract

K: Potassium

KCAET : Kelappaji College of Agricultural

Engineering and Technology

kg : kilogram

Kg/cm2 : Kilogram per square centimetre

L\* : Lightness or darkness LBD : Loose bulk density

LDPE : Low density polyethylene

MD: Maltodextrin
mg: milli gram
Mg: magnesium
min: minute
ml: milliliter
Mn: Manganese

Na : Sodium Ni : Nickel Number No. : probability p :

pH : percentage of H+ ions

PP :

Poly propylene revolution per minute second rpm :

s : Significant S :

#### **CHAPTER I**

#### INTRODUCTION

The Coconut Palm (*Cocos nucifera*) is one of the most important crops grown in humid tropics, cultivated for its multiple utilities; mainly for the nutritional and medicinal value. It belongs to Arecaceae family, the only living species of the genus Cocos (Debmandal *et al.*, 2011). Since most of the components of coconut palm is getting transformed to useful products, it is referred to as the "Tree of life". It can provide various natural products for the development of medicines and some industrial products. The annual global production of coconut is around 54 billion. India ranks at 3<sup>rd</sup> position with an average production of 13 million coconuts per year. The major conventional regions of coconut farming include the states of Tamil Nadu, Kerala, Puducherry, Karnataka, Odisha and the islands of Andaman and Nicobar and Lakshadweep. 21 Lakh hectares of Indian land is used for coconut plantation and the country produces ten thousand coconuts per hectare. Though Kerala stands first in number of coconut trees, the statistics reveals that Tamil Nadu (TN) is the best and largest producer of coconut in India. Last year TN produced 7 billion coconuts whereas Kerala produced 6 billion (Jegede., 2019).

Coconut is a traditional staple food in India, used in one or another form. It can be consumed in different stages and in different ways. During the flowering and fruiting period of coconut trees, a large amount of nutrients are produced and that are mainly transported through the phloem cells. Tender Coconut Water, the liquid endosperm is an excellent natural soft drink. Similarly the unfermented sap collected from immature inflorescence of coconut tree is named as NEERA which is a non alcoholic natural healthy drink enriched with sugars, minerals and vitamins. Generally this healthy drink can provide important nutrients to our body essential for good health. Since it is in their liquid form, it can be easily digested, making electrolytes and vitamins easily acceptable. Healthy non alcoholic and natural drinks

are always better than unhealthy alcoholic and artificial drinks, because unhealthy drinks are filled with sugar, artificial coloring and flavoring agents, artificial sweeteners, caffeine, and a number of stimulants (Jacob *et al.*, 2013).

The modern people have a keen eye for natural nutritional energy supplements even though market is filled with different brands of artificial energy drinks. Neera, a natural non alcoholic beverage of sweet, oyster white and translucent liquid is fast becoming popular energy drink, owing to its highly nutritive value, delicious taste and acceptable flavor. Coconut palms have an enclosed inflorescence from which the coconuts are developing. These developing fruits are nourished by inflorescence sap or Neera (Rangaswami, 1977). The collection of Neera is mainly by tapping process and around 1.8 liters per inflorescence can be produced in one day. So it fetches much better returns compared to copra (Ghosh et al., 2016). It is a unique source of iron, phosphorous, ascorbic acid and amino acids (Anon., 2019). Hence it can be used for iron and vitamin deficiency. Clinical studies indicate its medicinal applications, as a remedy for asthma, tuberculosis, bronchial suffocation and piles. It can also facilitate clear urination and prevent jaundice. The nutrient rich sap has a low glycemic index value (GI 35), while the sugar that we consume daily has a GI value of 60 (Muralidharan and Nair, 2013). The global demand for low glycemic index sugar (amount of sugar absorbed in to the blood will be very less) is increasing day by day as they can maintain diabetes mellitus and blood cholesterol level.

The major difficulty associated with the Neera production is its natural fermentation. During a long collection period, the sap undergoes fermentation and sugar converts to alcohol (58%), known as toddy. Yeast particularly *Sacharomyces cerevisiae* dominates the fermentation process. Freshly collected sap starts fermenting within 2-3 hours under ambient temperature. During the ambient storage mainly three stages of fermentation will occur. Initially lactic acid fermentation will takes place, followed by alcoholic fermentation and finally acetic acid fermentation due to the

action of Leuconostoc spp., Serratia and Acetobacter (Amoa-Awua et al., 2007). In case natural fermentation volatile acid, total acid, and total phenolic contents increases and vitamin C, amino acids, and total sugar content decreases. Increase in acidity causes reduction in pH. Thus the shelf life maintenance of sap is a major issue, also it is difficult to transport from one place to another. So many studies have been conducted to find a suitable method for the collection and preservation of Neera, but every technique yields a pasteurized sap with change in taste and had not been able to reduce the fermentation causing by microbes (Hebbar et al., 2015).

Since the current technologies are not much effective on the preservation of Neera experiments were conducted for the long term storage by spray drying process. Spray drying is a technique that produces dry powder from a liquid or slurry by rapidly drying with a hot gas (air). The process leads to increase the shelf life by removing the water content without significant alterations in the quality attributes. As a result of removal of water content the microbial activity and enzymatic activity will arrest so that the fermentation will get reduced and product attains maximum shelf life.

The selection of appropriate carrier material is very important as it influence the product yield and physical characteristics of final product (Estevinho *et al.*, 2013). A wide variety of natural and synthetic polymers can be used as the wall materials in the spray drying process. Usually natural food grade compounds are used in order to avoid the toxic effects associated with ingestion. Commonly preferred natural polymers are carbohydrates (e.g., cellulose derivatives, chitosan, cyclodextrins, dextrose, lactose, maltodextrins, sodium alginate and starches) gums (e.g., carrageenans, guar gum and gum arabic), and proteins (e.g., albumin, casein, collagen, egg, gelatine, soy and whey) (Arpagaus *et al.*, 2017). Gum Arabic is a well-known natural gum produced from exudates of Acacia trees has been the standard of excellence as a flavour encapsulating material for many years. It is an excellent emulsifier, bland in flavour and provides good retention properties for the volatiles

during the drying process (Kausadikar et al., 2015). But the use of gum Arabic is restricted in spray drying process due to its high cost, limited supply, and the variation in quality (Kanakdande et al., 2007). Maltodextrin is a suitable option to use as carrier agent as it is a good matrix former and can provide protection against oxidation, permit increased solid content with low viscosity and is economical. The blends of gum arabic with maltodextrin will provide improved properties regarding flavour retention, emulsion stability and protection against oxidation, so in this study the combination of maltodextrin and gum arabic is used for spray drying of Neera. The liquid feed given in to a spray dryer undergoes a series of transformations before becoming powder; they are i) atomization of the feed solution, ii) contact of spray with the hot gas, iii) evaporation of moisture and iv) particle separation. The spray dryers can dry a product very quickly compared to other methods of drying. They also turn a solution or slurry into a dried powder in a single step, which can be the advantage for maximizing the profit and minimizing the process. The physicochemical properties of the final product mainly depend on inlet temperature, air flow rate, feed flow rate, atomizer speed, types of carrier agent and their concentration. Hence, the aim of this study is to describe how these factors affect the properties of finely collected Neera powder from the spray dryer.

In this background the project entitled "Development of Neera powder using spray drying process" was undertaken at Kelappaji College of Agricultural Engineering and Technology (KCAET), Tavanur, Kerala, India with the following objectives

- To optimize the concentration of feed solution for spray drying process.
- 2. To optimize the operating parameters for spray drying process.
- To carryout storage studies of optimally processed spray dried Neera powder.

#### **CHAPTER II**

#### **REVIEW OF LITERATURE**

In the present investigation, an attempt was made to optimise the process parameters for the preparation of spray dried Neera powder. This chapter aims to discuss a detailed literature review about the principle and mechanism of spray drying of liquid products and the storage of spray dried powders, which has direct and indirect bearing on the specific objectives of the investigation. The chapter includes the earlier studies of

- a) Importance and composition of neera
- b) Production techniques for neera powder
- c) Physical and chemical properties of neera powder
- d) Response surface methodology
- e) Packaging and storage of neera powder

Neera is one of the natural healthy drinks, which is mainly consumed by the rural population. The non fermented sap (neera) with zero alcohol content is collected from the unblossomed mature spadix of coconut palm.

#### 2.1 IMPORTANCE AND COMPOSITION OF NEERA

#### 2.1.1 Neera

In India, coconut palm is called as 'tree of heaven', 'tree of life', etc. It is the only palm which produces inflorescence throughout the year and so can be tapped. Coconut sap or neera is one of the important drinks, tapped from coconut spadix. Since it flows through the phloem cells, it is also called phloem sap. This unfermented sap is rich in sugars, protein, minerals, antioxidants, vitamins, etc. Fresh sap is in golden yellow colour, having a pH >7 and has no foul odour. But if we keep neera at room temperature for a couple of hours, it undergoes fermentation and thereby colour changes to whitish. Also the pH decreases below 6 by developing an unpleasant smell. So in order to avoid fermentation the only way is to keep the sap at chilled condition or collecting the sap in ice boxes. In recent years, the Central

Plantation Research Institute has developed a 'coco-sap chiller' with ice cubes inside, which can maintain a temperature near to 2-3 °C up to 10-12 hours. It can keep the sap fresh and unfermented. The sap collected by this method will be non alcoholic, free from contaminants and can be sold as ready to serve health drink. Each spadix, will yield 1.5 litre/day or 60-67.5 liters of neera in a duration of about 45 days (Hebbar *et al.*, 2015a).

Hebbar *et al.* (2015a; 2015b), reported that neera is popular and widely consumed in countries like india, Sri Lanka, Africa, Malaysia, Indonesia, Thailand, and Myanmar. It is rich in carbohydrates, minerals, vitamins and proteins. The honey coloured sweet sap has a neutral pH and is translucent in nature.

Based on the palm variety and growing conditions the neera yield obtained will be different. Studies says that the water content of leaves have a correlation with the flow of sap. If the relative water content of leaves decreases then the sap flow also decreases. Growing conditions of palm like water stress or less rain fall, high temperature and higher transpiration rate, can also affect the neera yield (Ghosh *et al.*, 2018).

Chinnamma *et al.* (2019), explained about the Anti Fermentation Solutions (AFS) used to prevent the natural fermentation of neera. They can prevent the growth of microorganisms in the fresh sap. It is a combination of citric acid and potassium metabisulphate. Traditional method used to prevent the fermentation process was applying lime (calcium hydroxide) on the innerside of mud pot.

New technologies are applied to improve neera production and collection inorder to prove that neera can earn better returns than nuts. It provides employment opportunities for the rural youth. Also, it gives awareness to the consumers about the positive effects of neera.

#### 2.1.2 Harvesting and collection of Neera

In tropical regions, Neera tapped from coconut is considered as a strong sugar solution and is claimed to be an essential product (Van Die, 1974).

Rangaswami in 1977 reported that during the time of flowering and fruiting in coconut trees, a large quantity of nutrients are generated by the tree and they reaches the pinnacle through the phloem. The nutrient rich liquid is known as coconut neera and it can be used for nourishing the coconuts during its development. To take out neera generally coconut palms are tapped two times a day that is in the morning and evening. Neera is tapped gently from the matured unopened inflorescence of coconut palm in a uniform manner by traditional tools like bones, wooden sticks, etc. Before that the spathe should be tied with a coir rope in order to arrest the bursting of inflorescence. After tying spathe at the head portion nearly 7-10cm from the front portion is removed by sharp knife. The oozing out sap is collected by the collection boxes.

Muralidharan and Nair in (2013), narrated that the immature inflorescence which is about to burst is the right stage at which neera tapping is done. It takes 12 to 15 days for the sap to exude from the inflorescence tip.

Hebbar *et al.* (2015), also reported that coconut trees can be tapped at an early age as soon as it attains yield stability. Healthy trees which bear more fruits will give more sap. During the development of female flowers inside the spathe will cause a swelling at the base, which indicates the best time for tapping. Generally taping is done during a period of six months. Based on the health of palm it can give around 4.5 liters of sap per day.

Ghosh *et al.* (2018), described about a new technology for collecting neera developed by CPCRI Kasaragod. They developed a 'coco-sap' chiller box which is farmer friendly and can protect neera from extraneous matters. Thus the sap collected by this method is chemical free, non alcoholic and hygienic. The chiller box is lighter in weight, water proof, easy to connect with spadix, and requires less ice. Also it can maintain long term storage of neera without the fermentation process which leads to an easy transportation of neera from one place to another. The sap which came into contact with air will start fermentation process and technologies are developing by

institutions like Kerala Agricultural University, Central Food Technological Research Institute and Defense Food Research Laboratory for arresting the fermentation process.

The tapping and harvesting process involves several steps. They are explained by Chinnamma et al., (2019). First step is the selection of healthy palm. After that surface sterilization of palm crown will be done. For sterilization process, mild disinfectant like sodium hypochlorate (0.05%) will be sprayed on the crown, which creates aseptic environment for the smooth collection of neera. The third step is the cleaning of spadix. It is the outer covering enclosing the female flowers of coconut palm. Cleaning is done by spreading distilled water from bottom to top and after that it is wiped by tissue paper for drying the surface. Next procedure is beating the spadix and this will be done for 3-4 days from base to top with the help of skilled labours. After the cleaning process the tip of the spadix will be chopped by a sterile knife. Beeting will be continued up to 10-15 days. For preventing the entry of insects and other organisms, the chopped end will be covered with sterilized plastic cover every day. The fifth step is the application of sterilized clay on the spadix. This is to prevent the exudation of sugary neera from the top to base. The clay sterilization is mainly done by autoclaving method. Traditionally neera collection is done by clay pots. But the repeated uses will negatively affect the hygienic conditions. Hence new methods have been created for harvesting neera. Aseptic (sterilized) plastic vessels of five litre capacity are used nowadays. The cleaned plastic bottles are inserted in to the tip of spadix after 15 -20 days of beating. The exudation of neera will be there after 20<sup>th</sup> day of beating. The exudation rate is usually about 100-200 ml. The quantity of neera obtained will differ from palm to palm on its age, health, height and duration of collection.

Refrigerated neera is safe and is one of the best thirst quenching natural healthy drink which gives a cooling effect to body. Addition of preservatives and application of new technologies can increase the shelf life of neera up to 45 days;

wich makes a better transportation to urban areas. So its reachability can be increased just like the artificial alcoholic beverages. Proper commercialization of neera can make it as an alternative to carbonated drinks or mineral water. It has been medically proved that neera is much better than mineral water with low calorific value and of delicious taste.

#### 2.1.3 Composition of Neera

Muralidharan and Nair (2013) also reported that 100ml neera contains 15.2-19.7g total solids, 14.40g total sugars, 9.85g total reducing sugars, 5.58g original reducing sugars, 0.11-0.41g total ash, 0.50g citric acid, 0.23-0.32g total protein, 0.15g iron, 7.59mg phosphorus, 16-30mg ascorbic acid, with pH ranges from 3.9 - 4.7.

According to Akkarakaran (2015), the main constituent of the coconut sap juice is sugar (14-18%). He reported that the freshly collected sap is naturally rich in potassium, magnesium, zinc and iron and is a unique source of 12 of the essential vitamin B complex and ascorbic acid. The health benefits of neera explained by him are given in the table 2.1.

According to Hebbar *et al.* (2015), 100ml of Neera contains 15.18g total sugar, 0.554g reducing sugar, 0.245g amino acid, 0.321 antioxidant activity (mMTE), 90.6mg sodium, 168.4mg potassium, 3.9mg phosphorus, 0.012mg manganese, 0.020mg zinc, and 0.053mg iron, with pH 7.18.

Misra in 2016 reported about the low glycemic index value of neera and he also said that coconut crystal can be made out of this pure, low glycemic natural sap. Sap crystals contain only 0.5% glucose, 1.5% fructose, 16% sucrose and 82% inulinary prebiotic that promotes digestive health hence it can be used as a good sweetener.

Ghosh *et al.* (2018), studied about the sugar content of neera. He reveals that the average sugar content of neera varied between 12 to 14.4%. The maximum sugar content of neera was reported as 17% which shows that there is great variation in sugar content between cultivars. They also reported that "Keraamritham" or neera in 100 ml contains 15 -18% Total sugars, 90.5ppm of Potassium, 1.3mg of Vitamin C,

Calcium 60.ppm, Phosphorous 15ppm, and Acidity 10m eq. /l, 45ppm, Phenols 8mg/100ml, Sodium 9.5ppm with pH 6.8.

#### 2.1.4 Health benefits and value added products of neera

The most significant characteristics of neera is its low glycemic index value. Akkarakaran (2015) reported that the Glycemic Index (GI) is a measure of how quickly and how much a particular carbohydrate present in our food substance can raise the blood sugar level by releasing glucose in to the blood stream. Studies conducted by Food and Nutrition Research Institute (FNRI) in 2007, revealed that coconut sugar developed from neera has naturally lower Glycemic Index rating (GI) of 35 compared to that of most popular commercial sugar such as table sugar's GI index of 70, honey's GI of 55 and cane sugar's GI of 68. He also narrated that it can be used as a remedy for so many health problems, especially for diabetic patients.

Foods which have a GI value less than 55 is considered as low glycemic index foods. The global demand for low GI sugar is increasing day by day, so neera and neera based sugar can be used instead of sucrose which we are using daily (Ghosh *et al.*, 2018).

Table: 2.1. Health benefits of Neera.

Nutrients in Neera	Health benefits
Carbohydrate (g/100g)	Source of energy in our body
Proteins (g/100g)	Build, maintain and repair body
Vitamin C (Ascorbic Acid mg/ 100ml)	Antioxidant, prevent cancer, increase absorption of iron, healing effect
Vitamin B1 (Thiamin) mg/ dI	Release of energy from fuel molecule
Vitamin B8 (Inositol) mg/dI	Helps to reduce high cholesterol, good for heart, formation of healthy cells in the body
Glutamic acid (g/ 100g)	Major role in synthesize of protein , healing of illness,

Potassium (mg/100g)	Reduce hypertension and blood sugar,
	controls cholesterol level and weight
Magnesium (mg/100g)	Essential for metabolism, nerves and
	stimulates brain ( memory)
Nitrogen (mg/100g)	Help treat cardiovascular disease
Phosphorous (mg/100g)	Enhance bone and cell growth, and
	kidney function
Sodium (mg/100g)	Key role in functioning of nerves and
	muscles
Chlorine(mg/100g)	Maintain body fluid volume and acid
	base balance
Zinc(mg/100g)	Necessary for mental development
	(intelligence)
Iron(mg/100g)	Vital for quality of blood and mental
	development and immune system
Copper(mg/100g)	Helps to release energy, helps melanin
	production in the skin and production of
	red blood cell

(Source: Akkarakaran, 2015)

Since neera is highly susceptible to natural fermentation various technologies are developed by different institutes including the addition of available preservatives. Apart from chemical methods, heat preservation techniques are also used for extending the shelf life of neera. According to Swami (2013) heating of neera results in to the development of brown colour after attaining a particular temperature. Further increasing the temperature neera can be converted to so many value added products such as palm syrup, coconut neera nectar, jam, ice-cream, tonic, wine, cookies, chocolate and brown sugar, etc.

#### 2.1.4.1 Palm syrup

Muralidharan and Nair (2013), reported that boiling the sap under moderate to low heat yields a golden brown sticky liquid with high mineral content which is called palm syrup. Obtained syrup is free from total fats and cholesterol with a sucrose content of 50% and a Glycemic index of 35 GI. In many countries palm syrup is used as a health and wellness drink and prevalently used in Ayurveda and other system of medicine (Jose *et al.*, 2018).

#### 2.1.4.2 Coconut nectar

Coconut nectar is produced by concentrating the fresh neera to thick slurry by thermal process. When the brix value reached to 78° Brix it is considered to be the end point of the process. Since it is a very healthy source of important nutrients and the mineral iron, it can be used as a chief component in pharmaceuticals as well as can be included in the formulations for the preparation of anemic medicines (Ghazali and Sin, 1986).

#### 2.1.4.3 Coconut jaggery

Coconut jaggery is a converted product from fresh neera having solid or semi solid crystalline structure. It can be consumed directly. The preparation of coconut jaggery is explained by Akkarakaran (2015), the fresh sap is heated at 40°C in a pan and this juice is delimed to neutrality. For that, phosphoric acid or triple super phosphate solution will be added. Continuous stirring is done along with boiling process to enhance the evaporation and mixing. The foam formed due to the mixing and stirring process is removed by a perforated ladle. The thick mass is transferred to molds and allows it to set for some time. The nature of jaggery is always hard, crystalline and golden coloured (Misra, 2016).

#### 2.1.4.4 Coconut molasses

Coconut molasses is a sweet syrupy material obtained as a by-product from palm sugar. Golden syrup and cattle feed are the other products made from Molasses.

By fermentation process, bio-chemical products like ethyl alcohol, acetic acid, citric acid etc., can be prepared out of molasses (Misra, 2016).

#### 2.1.4.5. Coconut Vinegar

Coconut vinegar is produced from the inflorescence sap. Fresh sap is stored in large containers for 10 days. The opening of the container should be coverd with net for providing aeration as well as preventing the entry of foreign matters and should be cleaned. After the fermentation process, it can be harvested as vinegar. It is having a wide range of usage as preservative in pickle industry and as flavoring agent in food processing sector. The palm vinegar has good export potential as compared to the synthetic vinegar (Misra, 2016).

#### 2.1.4.6 Palm wine

Palm wine is an alcoholic beverage created by immediate fermentation of palm sap after collection with the help of natural yeasts present in the pores of pot and air (Chandrasekhar *et al.*, 2012). Fermentation results in to the formation of wine with 4% alcohol. As the day progresses, the wine yields as a sourer, stronger acidic product.

#### 2.1.4.7 Palm Sugar

Coconut sap sugar is obtained by heating the fresh neera at 115°C. Since the sugar is free from chemicals, it is considered as one of the best natural sweetener. It is widely used in South East Asian regions. It is a perfect and healthier substitute for artificial sweetners. In comparison with brown sugar obtained from cane sugar, palm sugar has more minerals, vitamins and has twice the iron, four times the magnesium and over ten times the zinc. Like palm jaggery processing here also the juice is delimed and filtered before boiling. It can be used in the preparation of chocolates, toffees and confectionery items (Jose *et al.*, 2018).

#### 2.1.4.8 Palm Candy

Like palm jaggery, palm candy is also an important by product of neera. It is being produced and used since procuring sweet neera from Palmyra. It has got various uses in ayurvedic medicinal preparations (Misra, 2016).

#### 2.1.4.9 Neera soft drink

Chilled neera soft drink is a healthy nutritious and thirst-quenching drink which can give a cool feeling to the body. It includes a number of minerals, vitamins, ascorbic acid, nicotinic acid, riboflavin and protein. It has been medically proven that neera is better than mineral water with less calorific value. Hence it can be used as a good alternative for carbonated beverages. By providing proper filtration and preservatives immediately after harvesting of neera and before canning or bottling process, the shelf life can be increased up to 45 days. This can make a way for urban transportation and marketing (Swami, 2013).

#### 2.1.4.10 Coconut neera cookies

The main ingredients of neera cookies are coconut neera, flour, grated coconut, and neera jaggery. Misra (2016) reported that it is a good choice for diabetic patients since the glycemic index is very low. So that they can consume it as a healthy snacks in between their main meals. It is possible to add different ingredients along with neera to produce different combinations like oats, multigrain, arrowroot and corn etc.

#### 2.1.4.11 Coconut neera chocolate

Mangalore-based Central Arecanut and Cocoa Marketing and Processing Cooperative (Campco) Ltd in 2016 produced a chocolate that containing sugar made from 'neera' or sap of coconut palm, which will be safe for consumption even for diabetic patients (Misra, 2016).

#### 2.1.5 Processing of neera

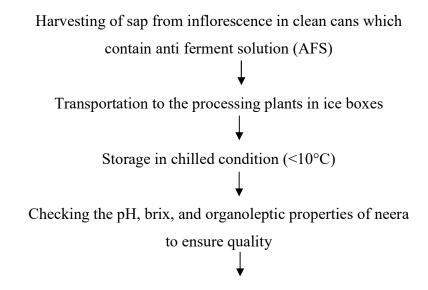
Processing of neera is usually done for increasing the storage life. For that the collected neera is added with preservatives and subjected to high speed

centrifugation, microfiltration and pasteurization. All these processes will help to keep neera under ambient conditions for minimum of six months. It has been proven that the final product is a nutritious beverage which retains vitamins, sugar, and other minerals beneficial for human health.

Baliga and Ivy (1961), collected neera from the coconut palm and is pasteurized and bottled at 170 to 175°F. If the temperature goes above this range it can bring a cooked flavor and holding neera at this pasteurization temperature will kill the yeasts present in the sap. Pasteurization process will be effective when sodium benzoate is added. Addition of malic or citric acid can improve the effectiveness of sodium benzoate. But the addition of malic acid can bring apple juice flavor.

To improve the storage period of neera, efforts were made by Hebbar *et al.* (2015a), they did some common techniques like sanitation, refrigeration, filtration, centrifugation, de-aeration, and pasteurization.

Aneeta joy (2016), processed the coconut neera through a series of steps before bottling and packaging process. The general processing steps of neera are as follows;



Filtration using normal sieve to remove large particles

Removal of suspended particles through double centrifugation

(8000-10000 rpm)

Pasteurization of neera at a specific temperature for a specific time (80°C, 15 minutes)

Aseptic packaging of neera in HDPE/PP/Glass bottles

Figure 2.1 Processing of neera

Ramaswamy and Ramaswamy in 2017 processed the neera in a different method. The addition of strawberry extract (3%) and aloe vera cubes (10%) had given a pleasant smell to the processed neera. The pasteurization temperature selected by them was 80°C for 20 minutes. After the pasteurization process they cooled the neera (40-50°C) and packed in PET bottles.

Chinnamma *et al.* in 2019 also explained the common processing steps of neera done in processing industries. They are microfiltration, pasteurization, and bottling and storage. Microfiltration of collected neera has been done by passing the neera through the micro filters having pore size of 100 microns. Pasteurization was done at 75-85°C for 10-15 minutes. With the help of an automatic bottling machine pasteurized neera was bottled in polypropylene bottles (200 ml), and glass bottles (300 ml). Capping and sealing were also done by automatic machines.

#### 2.2 PRODUCTION TECHNIQUES FOR NEERA POWDER

Different techniques and studies were done by researchers to produce powders mainly from fruits and vegetable juices. The main techniques done by them were spray drying, freeze drying, and vacuum drying. Sometimes the combinations are also used. Since freeze drying and vacuum drying are costly, the main process technique selected by researchers was spray drying process.

Fogler and Kleinschmidt (1938), suggested that, there should be an optimized condition inorder to get good output even though suitable wall materials are used to prepare the feed material. Feed temperature, air inlet temperature, and air outlet temperature were the main factors that have to be optimized in a spray drying process. The selected inlet temperature should not affect the volatile materials present in the feed and also it should not damage the feed material.

If the inlet air temperature is low it will cause the formation of microcapsules with high density membranes, high water content, poor fluidity, and easiness of agglomeration. Increased inlet air temperature leads to a larger evaporation rate and which results in to cracking of the surface of material and loss of volatile materials from the core substances (Zakarian and King, 1982).

Discussions had been done on the basis of spray dryer design by Gauvin and Katta in 1976. According to them atomization is the most important process in spray drying, since it can affect the product size and the initial drop size distribution of the spray generated by the atomizer forms the basis of the chamber design. The different atomizers used generally are centrifugal disk atomizers, pressure nozzles, and pneumatic nozzles. Pneumatic nozzles are used for small capacity works, where as pressure nozzles are preferred for highly viscous feeds and centrifugal disk atomizers for high capacity work.

Kramer *et al.* (1988), studied the possibility of producing fruit juice powder by freeze drying method. The obtained powder was highly hygroscopic and thus showed a tendancy to form agglomerates, which turn in to large solid lumps. This was due to the presence of high sugar content. But the technique can retain the nutritional value of juice.

Masters (1991) narrated that spray drying is the best method for the production of powder colorants with high storage stability, easier to handle, and it reduces the weight for transportation, compared to liquid concentrate.

According to Re (1998), spray drying is a widely used technology for drying of heat sensitive foods, pharmaceuticals and other substances, because of the rapid evaporation process. It can also be used for encapsulating a particular compound generally volatile material inside another material. The main part of spray dryer is the atomizer; the selection of atomozer is very important in the design of spray dryer which can significantly affect the size distribution of final dried product. Uniformity of drop-size, its distribution, and homogeneity of the spray are the most important characteristics of the atomiser from the standpoint of product quality.

Spray drying can be used to preserve food or simply as a quick drying method. The range of product applications continues to expand so that spray drying has connections with many things we use daily (Nath *et al.*, 1998).

Dolinsky *et al.* (2000), conducted studies on drying of fruits and vegetables by spray drying process. They found that nearly 30-55% maltodextrin is needed to get an acceptable amount of fruit powder. The increases in inlet air temperature can reduce the product yield, due to the melting of powder. Also Adhikari *et al.* (2003), reported that the addition of maltodextrin significantly reduced the stickiness of fructose solutions, showing its use as an effective drying aid.

Drying of fruit juices is a difficult operation, mainly because of the undesirable changes in the quality of the dried product. The high temperatures and long drying times required to remove the water from the sugar containing fruit material in conventional air drying may cause serious damage to the flavour, colour, and nutrients of the product. Spray drying reduces these undesirable changes (Saravacos *et al.*, 2002).

Chauca et al. (2005), studied the effect of the carriers on the microstructure of mango powder obtained by spray drying process. Carrier materials used for the study

were maltodextrin, Gum arabic and starch wax in the concentration of 12%. Addition of maltodextrin increased the solubility of powder. Addition of Gum arabic provided good emulsification property.

Chegini and Ghobadian (2007) conducted a study on spray drying of orange juice at 65% concentration. Parameters opted for the study was wall material, inlet and outlet temperatures, feed flow rate and sticky point temperature. Results showed that inlet air temperature and feed flow rate have significant effect on the powder yield and wall deposit of spray dryer chamber. The feed flow rate of 15 ml/min, inlet air temperature of 130°C and outlet air temperature of 85°C and sticky point temperature of 44°C were considered as the optimum condition for getting good quality powder of moisture content 2%.

Quek *et al.* (2007), observed the physicochemical properties of spray-dried watermelon powders. They used two different combination of maltodextrin (3 and 5%) as carrier agent and four different temperatures of 145, 155, 165 and 175°C. Results demonstrated that increasing the temperature decreases the moisture content and also there was no significant effect of inlet air temperature on water activity of powders.

A study was conducted by Tonon *et al.* (2008), to determine the influence of process conditions on the physico chemical properties of Acai (*Euterpe oleraceae*) powder produced by spray drying. The independent variables they considerd were, inlet air temperature (138-202°C), feed flow rate (5-25g min<sup>-1</sup>), and maltodextrin concentration (10-30%). They concluded that the increasing temperature led to higher process yield and powder hygroscopicity and to lower moisture content. Whereas the feed flow rate negatively influenced process yield and hygroscopicity, and positively influenced moisture content. They also found that maltodextrin concentration had negative effect on powder hygroscopicity, confirming its efficiency as a carrier agent.

According to Adameic (2009), spray drying can be used as a suitable method for solvent evaporation to obtain dry stable microencapsulated powder. In the study

he mentioned the limitations of spray drying process, that are, (1) the core material should be heat resistant even though the temperature applying is in short exposure time, (2) the carrier material using should be film forming and also should have acceptable solubility.

Athanasia *et al.* (2010), conducted a study on spray drying of concentrated orange juice using dehumidified air as drying medium and maltodextrin as drying agent. Maltodextrin used for the study had different dextrose equivalent. The feed mixed with maltodextrin is sprayed through the atomiser at different temperatures such as 110, 120, 130, and 140°C. Results revealed that the addition of maltodextrin and use of dehumidified air was found to be the effective way of reducing residue formation.

Tuyen et al. (2010), determined the effect of spray drying conditions such as inlet drying air temperature (120, 140, 160, 180 and 200°C) and maltodextrin addition (10, 20 and 30%) on the physicochemical and antioxidant properties of Gac (Monocardia cochinchinensis) fruit aril powder. Moisture content, bulk density, colour characteristics, total carotenoid content, encapsulation efficiency and total antioxidant activity were significantly affected by maltodextrin concentration and by the inlet air temperatures. They got good quality Gac powder mainly in terms of colour, antioxidant content produced by spray drying at inlet temperature of 120°C and a maltodextrin concentration of 10%.

Effect of carrier type and spray drying on the physicochemical properties of powdered and reconstituted pomegranate juice was observed by Yousefi *et al.*, (2010). They diluted the pomegranate juice to 12°Brix, and a combination of maltodextrin, Gum arabic, and waxy starch were added in to it. They found that Gum arabic could produce more powder yield comparing to other carrier agents. Increasing the carrier concentration resulted into a decreased bulk density of the powder product and the addition of cellulose decreased the solubility of final product. Powder produced with gumarabic caused a greater colour change. They also found that

adding the carrier agents will increase the glass transition temperature and storage stability. Variation in the anthocyanin content depends up on the type and amount of carrier material used in the study.

Solval *et al.* (2012), prepared cantaloupe juice powders using spray drying technology. The feed for the spray drying process was prepared by adding cantaloupe fruit juice with 10% maltodextrin and the selected inlet temperatures were 170, 180 and 190°C. The juice powder produced at 170°C had higher moisture content, and higher vitamin C and  $\beta$  carotene than those produced at 180 and 190°C. This indicated that the inlet air temperature can affect vitamin C and  $\beta$ -carotene contents of cantaloupe juice powders.

Fazaeli *et al.* (2012), explained about the effect of spray drying conditions and feed composition on the physical properties of black mulberry juice powder. The processing parameters taken by them for the study were inlet air temperature (110, 130, and 150°C), compressed air flow rate (400, 600, and 800 L/h), concentration of drying agents (8, 12, and 16%). The best result was obtained with the addition of maltodextrin having dextrose equivalent 6. The drying yield ranges from 45 to 82%. The result showed that the lowest moisture content (1.5%) was obtained, at a compressed air flow rate of 800 L/h. They also found that inlet air temperature negatively influenced the bulk density due to the increase of powder's porosity. The lower the bulk density, the higher is the solubility of powder.

According to Phisut (2012) spray drying is one of the common and widely using processes for the production of fruit juice powders. As the output product is in powder form, the quality will be high and it will be easy to transport from one place to another. Low water activity is another advantage which can increase the shelf life and storage stability of powder product. He also described that processing or operating parameters can also affect the quality of the final product. The main parameters affecting the output is inlet temperature, air flow rate, atomizer speed, feed flow rate, types of carrier agent and their concentration.

Simha *et al.* (2012), conducted a comparison study of spray drying and freeze drying of pomegranate juice fermented with *L. Acidophilus*. For the spray drying process they have chosen the inlet air temperature of 130, 140, 150°C and freeze drying was carried out at -40°C. The observed changes of final product were moisture content, bulk density, water activity, pH, acidity and solubility of the final product.

Fernandes *et al.* (2013), conducted experiments to find the influence of spray drying operating conditions on microencapsulated rosemary essential oil properties. The results indicated that moderate wall material concentration (24%), low inlet air temperature (135°C), and moderate feed flow rate (0.7 Lh<sup>-1</sup>) are the best spray drying conditions.

Ratti (2013) narrated that freeze drying can also be used for drying of wet material inorder to get good quality food powder. She says that freeze drying is an expensive process and it consumes more energy. Freeze drying techniques and technological challenges in food preservation field is also mentioned by them.

The physicochemical and storage properties of spray dried orange juice powder were studied by Sabhadinde (2014). The study was conducted by a piolet plant spray dryer with concurrent air flow. The maltodextrin combinations they have taken were (10, 12.5, 15, 17.5 and 20%) along with inlet/outlet air temperatures of 200°C and 120°C. The characteristics of feed, air, type of atomiser and type of carrier materials were affected the physicochemical properties of powder.

Patil *et al.* (2014), done experiments on spray drying of guava to produce guava powder by using three different combinations of maltodextrin (7, 9.5 and 12) and three temperature ranges (170C, 180 and 185°C). The powder was analysed for moisture content and dissolution. It was observed that as the inlet temperature increased, the moisture content and dissolution decreased. But there was no significant effect of inlet air temperature on water activity of powders. As the porosity increased while increasing the temperature, it caused a negative impact on

bulk density. Higher solubility was noticed for those having a lower bulk density. The best powder was obtained for the temperature of 185°C.

Avila *et al.* (2015), evaluated the effect of maltodextrin and spray dryer operating conditions on sugar cane juice powder. For that they selected four factors like maltodextrin concentration (10-20 %), inlet air temperature (130-150°C), outlet air temperature (75-85°C) and atomization speed (22,000-26,000rpm). The optimized factors for the better drying of sugar cane juice was 20% of maltodextrin combination with feed, 130°C inlet air temperature, 75°C outlet air temperature and atomization speed of 22,000 rpm.

Santhalakshmi *et al.* (2015), worked on the effect of inlet air temperature on physicochemical properties of spray dried Jamun fruit. The result of their study showed that higher inlet air temperature increased the moisture content of powder, and led to the formation of larger particles, also the inlet temperature of 150°C gave higher product yield. Best processing condition obtained for powder production and colour characteristics was used as an inlet temperature of 140-150°C.

Wisniewski (2015) narrated that spray drying is a process which can turn liquid product in to solid product in one step. This technology has a lot of applications in the food, pharmaceutical, chemical and nanotechnology industries. The main principle employed in the process is spraying a liquid in to a hot gas stream, and after evaporation the fine particle are separated out and collected in glass bottles. It is a fast process and the hot air contact time with the feed product is too short. He also mentioned that the temperature of gas decreases rapidly due to the intensive evaporative cooling and the droplet temperature remains low, thus thermally sensitive products can also be subjected successfully in spray drying process.

Shishir *et al.* (2017), conducted a study on the effects of spray drying conditions on fruits and vegetables. He finds that the most significant factors in spray drying process were inlet temperature and carrier agent. Spray drying can preserve bio active compounds with the help of carrier agents or with their combinations. He

suggested that response surface methodology is the most suitable tool for optimising spray drying conditions. Some of the modified spray drying techniques can improve the drying of fruits and vegetables such as ultrasound technique during atomization, vacuum drying chamber with controlled atmosphere, and supply of dehumidified air to drying chamber of basic spray dryer.

Lee *et al.* (2017), studied the effectiveness of additives in spray drying process. They found that different food powder has shown different characteristics under the influence of different additives. Additives can improve product yield, through the manipulation of transition temperature. Usage of combination of additives can give positive results such as better yield, solubility and bulk density. The major additives explained by them are carbohydrates, protein, and gums.

Anon, 2019 has explained the differences between spray drying and freeze drying of liquid products. The concluded that spray drying is cheaper than freeze drying, and hence spray dried products are cheaper than freeze dried products. Also spray dryer have a larger capacity than freeze dryer. Comparing to freeze drying process, spray drying process is short and simple, and spray dried product will show higher solubility.

#### 2.2.1 Carrier materials for spray drying

Carrier materials that are used in spray drying process can protect sensitive food components against unfavorable conditions. They can mask or preserve the active compound present in the food material by reducing the volatility and reactivity. Some of the additives can give attractive appearance to the final product (Physut, 2012). Stickiness is the major problem associated with powder materials. It is mainly due to low glass transition temperature and low molecular weight of sugar material present in the respective food product, especially sucrose, fructose and glucose. Glass transition temperature is the temperature at which the amorphous phase of the polymer is converted between rubbery and glassy state. The presence of low molecular weight sugar and acids can cause some problems in fruit juice powder

properties such as, stickiness, solubility and hygroscopicity. Because of low glass transition temperature, the powder will stick on the spray drying chamber and this leads to lesser product yield. This situation can be solved by adding carrier agents like polymers, gums. The commonly used carrier agents in spray drying technology are carbohydrates, gums, proteins and lipids. Among these, the polymers of high molecular weight and high glass transition temperature (Tg) *viz.*, maltodextrin, arabic gum and modified starch are generally used (Cano-Chauca *et al.*, 2011).

#### 2.2.1.1 Maltodextrin

Maltodextrin [( $C_6H_{12}O_5$ ) nH<sub>2</sub>O] is a generally used carbohydrate derivative. It is a white coloured powder, non sweet in taste having neutral smell. Maltodextrin is produced by the partial hydrolysis of the corn starch by acid or by enzyme (Bae and Lee, 2008). The structure of maltodextrin contains *D-glucose* unities linked by  $\alpha$  1-4 chain. The powder is cheaper and available in different dextrose equivalants (DE) which indicates the hydrolysis of starch polymer. They provide low viscocity at higher concentrations. Generally maltodextrin is having a DE less than 20. Those having DE more than 20 are called dried glucose syrup. Higher DE carbohydrates have both advantage and disadvantage that, it can provide low oxygen permeability, but will cause caking during storage (Tontul and Topuz, 2017). Maltodextrin have low emulsifying efficiency and studies reveals that the usage of maltodextrin with some other additives can improve the powder charecterictics. Generally the combibnation materials used with maltodextrin are Gum arabic, modified starch, whey proteins etc.

Desobry *et al.* (1997), explained that the stability of spray dryed powder depends on the composition of the wall material. Maltodextrin is the best choice to use as a wall material having low viscosity at high solids ratio and are available in different average molecular weights (DE values of 4, 10, 15, 20, 25, 30 and 42 with average molecular weight decreasing as DE increases). Addition of maltodextrin can protect the core material from oxidation. More DE maltodextrins provide a matrix

capable of preventing oxygen permeability for a long term. However it affects the glass transition temperature negatively as molecular weight decrease, the product shows higher hygroscopicity and tends to form cake.

Amaranthus betacyanin extracts were spray-dried by Cai and Corke (2000) with the addition of maltodextrin ranging from 10-25 DE and starches. The storage stability of betacyanine extract was enhanced by the addition of maltodextrin and starches and provided a reduced hygroscopicity. Mixed combination of 10 and 25 DE maltodextrin resulted in to best output compared to the addition of 10 and 25 DE maltodextrin separately.

Carneiro *et al.* (2013), describes the limitations of maltodextrin. That is maltodextrin has a low emulsion capacity so that it is desirable to use along with some other wall materials such as Gum arabic or modified starch.

Parikh *et al.* (2014), studied the applications of maltodextrin in pharmaceutical industry. According to them, maltodextrin is a polysaccharide, having a long carbohydrate chains along with 2-3% glucose and 5-7% maltose. Usually the production of maltodextrin is by partial hydrolysis of starch material such as corn, potato, and wheat. It is available in white hygroscopic spray-dried powder which is slightly sweet and almost flavourless. Our body can digest maltodextrin as a simple carbohydrate so that it can be used in sports drinks and instant energy providing powders. It is considered as good encapsulant since it exhibits low viscosity at high solids contents and also good water solubility. It can contribute highest flavour retention and up to 35.5% can disperse completely in water without forming any cloudy or haze appearance.

According to Mahdavi *et al.* (2016), the best product in the microencapsulation of natural anthocyanins was obtained when maltodextrin and Gum arabic was used together as the carrier agent. The combination offered highest process efficiency and good powder quality.

#### 2.2.1.2 Gum arabic

Gum arabic used in spray drying is an excellent emulsifier. But the application of Gum arabic in food processing sector is limited since the gum is costlier than maltodextrin (Shiga *et al.*, 2001).

Turchiuli *et al.* (2005), used Gum arabic and maltodextrin as their carrier material for finding the encapsulation feasibility of vegetable oil. According to them Gum arabic is a hydrocolloid produced by natural exudation of acacia trees. The gum can be successfully used for the encapsulation of lipids products. It is composed of simple sugars, glucoronic acid, and a protein compound. The protein component can play an important role in the functional properties of Gum arabic.

Madene *et al.* (2006), explains that gums are thickners used in encapsulation process. Generally they are tasteless but they can have a pronounced effect on the taste and flavor of the final product. Usually the addition of gums reduces the sweetness and provides viscocity to the output. It can take the role as a drying agent as well as surface active agent. But there is a need of finding alternatives to Gum arabic since it is highly expensive and the availability of Gum arabic is also less. The mixture of Gum arabic and maltodextrin can give a promising effect on the finished product.

Ferrari *et al.*, (2013) studied the stability of blackberry powder produced by maltodextrin and Gum arabic. The results showed that powder produced with maltodextrin as wall material had the longest half-life, lowest degradation rate of anthocyanin at 25°C and greater stability. They concluded that the use of maltodextrin or the combination of both maltodextrin and Gum arabic as carrier agents could promote better maintenance of antioxidant potential of blackberry powders.

Alves *et al.*, (2014) reported that the Gum arabic has a colloid functionality which makes it suitable for encapsulation process. It can be used together with maltodextrin, protein, modified strarch etc. generally mixture of maltodextrin and Gum arabic can provide a balanced efficiency and cost effective.

Gum arabic (acacia gum) is the most commonly used gum additive collected from Acacia senegal and Acacia seyal trees, it is a complex branched hetero polysacharide composed of 1, 3-linked  $\beta$ -D-galactopyranosyl units. It is the only gum used in food industry having high solubility and low viscocity in water. The gum is having high emulsification properties. It is the combination of D-glucuronic acid, L-rhamnose, D-galatose and L-arabinose in the proportion of 4:2:2:1, and pale white in colour (Patel and Goyal, 2015).

#### 2.3 PHYSICAL AND CHEMICAL PROPERTIES OF NEERA POWDER

Abadio *et al.*, (2004) described the effect of maltodextrin concentration and atomization speed on the physical properties of spray dried pineapple juice powder (*Ananas comosus*). The properties studied by them were bulk and tapped densities, colour, moisture content and solubility. The increased atomization speed resulted in a reduction in particle size. Since the addition of carrier agent (maltodextrin) increases the solid content in feed it reduces the amount of free water for evaporation.

Chegini and Ghobadian, (2007) studied the influence of spray drying process parameters on the spray drying orange juice. They found that inlet air temperature and feed flow rate have a significant effect on powder yield and its wall deposition. Their results also revealed that the bulk density of powder decreases with increase in the temperature at a constant flow rate. The more bulker material shows the highest moisture content. Product yield was empty when the concentrated juice was spray dried alone. The wall deposit increased with the use of maltodextrin as carrier agent. Use of liquid glucose resulted in to a less wall deposition. Good quality powder were obtained with 130°C inlet air temperature and 85°C outlet air temperature.

Kha *et al.* (2010), investigated the effect of drying parameters on the physicochemical and antioxidant properties of Gac (*Momordica cochinchinensis*) fruit aril powder. They revealed that the pH, water activity and solubility had no significant effect on the processing conditions. But increase in maltodextrin (10 to 20%) and temperature (120 to 200°C) reduce the moisture content from 5.29 to

3.88%. An increase in maltodextrin concentration from 10 to 20% increased the lightness of powder. Increase in temperature reduced the antioxidant property also.

Patil *et al.* (2014), examined the physical and nutritional properties of spray dried guava powder. They observed that the moisture content of the powder decreased with increase in the temperature, but there were no significant change for water activity due to the elevation of inlet air temperature. As the porosity of powder increases due to increase in temperature, the bulk density showed decreasing pattern. Lower bulk density shows greater solubility. The optimised product was obtained at an inlet temperature of 185°C.

Fernandes *et al.* (2013), described the effect of wall material concentration, inlet air temperature and feed flow rate on the moisture content, hygroscopicity, wettability, solubility, bulk and tapped densities, particle density, flowability, and cohesiveness of the microencapsulated rosemary essential oil. Bulk density was positively affected by carrier concentration and negatively affected by inlet air temperature. Wall material concentration and inlet air temperature negatively affected the true density. The best powder was obtained at 135°C inlet air temperature, 24% maltodextrin and at a feed flow rate of 0.7L.h <sup>-1</sup>.

Mishra *et al.* (2014), conducted experiments to find the effect of wall material concentration and temperature on the physicochemical and antioxidant properties of amla (*Emblica officinalis*) juice powder. They found that temperature and wall material concentration had significant effect on the moisture content and hygroscopicity of powder. But the increase in maltodextrin concentration does not show any significant change in the bulk density and water solubility index of powder. The drying temperature and carrier concentration negatively affected the antioxidant properties of amla powder.

Saranya *et al.* (2016), microencapsulated banana pseudostem juice with the addition of maltodxtrin, horse gram extract, sugar and milk. The moisture content of the product was in the range of 2.9 to 4.60%. They showed that the moisture content

decreased with an increase in maltodextrin concentration and temperature. The water activity of powder varied from 0.295 to 0.430. They also mentioned that the addition of maltodextrin increased the lightness of the powder.

Silva *et al.* (2013), produced spray dryed propolis powder and assessed the physicochemical charecteristics and storage stability. They evaluated the moisture content, hygroscopicity, water activity, antioxidant properties of the produced powder. They concluded that the spray drying process could preserve the antioxidant content of propil powder and it was stable at room temperature. The powder form of this food additive obtained by spray drying process can be used as an antioxidant as well as antimicrobial agent in food.

Sarabandi *et al.* (2014), explained about the effect of spray drying conditions on the physicochemical properties of grape syrup powder. The quality parameters selected by them were moisture content, bulk density, solubility, hygroscopicity, water activity and wettability. The results revealed that moisture content, water activity, bulk density and hygroscopicity were decreased due to the elevation of temperature were as wettability and solubility values increased with increase in inlet temperature. They reported that the concentration of carrier agent can also significantly affect the wettability, bulk density, solubility and hygroscopicity of powder, these factors showed a negative effect.

Seth *et al.* (2016), studied the reconstitution properties of spray-dried sweetened yogurt powder, which was effected by processing parameters. Twenty experiments were done with varying processing parameters with the help of a central composit experimental design. The analysis of experimental data showed that the solubility and dispersibility was affected negatively with increasing temperature and pressure. They also found that the feed rate can significantly affect the solubility. There was an increase in wetting time and density, during the elevation of temperature and the recorded range of bulk density, tapped and particle density were 344.8–475.7, 551.7–782.5, and 1187.5–1666.7 kg/m³, respectively.

#### 2.4 RESPONSE SURFACE METHODOLOGY

According to Bas and Boyaci, (2007) RSM is the collection of statistical and mathematical techniques useful for developing formulations of new product as well as improving the existing product. It defines the influence of independent variables on the process. In RSM it is possible to use higher degrees in a packed data instead of using lower degrees.

Ferreira *et al.* (2007), narrated that the best design for the response surface methodology is Box-Behnken design, because it gives estimation of parameters, building of design, identifies the lack of fit of model, and permits use of block system.

Ahn *et al.* (2008), microencapsulated sunflower oil and optimized the result by RSM. The parameters selected by them were sunflower oil concentration, proportion of milk protein isolates to coating wall, soy lecithin concentration, and homogenizing pressure. The microencapsulation efficiency was investigated with respect to these four parameters. The optimal conditions obtained were 23.6% sunflower oil, 19.0% milk protein isolates, 2.5% soy lecithin and 54.8% dextrin.

Aghbashlo *et al.* (2012), found the relation between the processing factors (inlet drying air temperature, aspirator rate, and peristaltic pump rate) and the responses (energy efficiency and encapsulation efficiency) by using Response Surface Methodology (RSM). They explained the main problem associated with the RSM methodology, is that the relationship between the input variables and output responses is unknown. So the first step is to establish a suitable approximation for the true relationship between output responses and input variables. They also mentioned that it is the best method for building accurate models in optimization design.

## 2.5 PACKAGING AND STORAGE OF SPRAY DRIED POWDER

Yoghurt is a fermented milk product, which is a rich source of digestable proteins, calcium and vitamin B complex. Mango soy fortified powder will help to reduce the blood cholesterol level and can minimize the risk of several cancers. But

the shelf life of yoghurt is very short (1 day at ambient temperature (25-35°C), 4-5 days at 7°C). So yoghurt was dried by freeze, spray, microwave, and conventional is drying methods by Kumar and Misra in (2004). The powder packed in two different packaging materials, high density poly propylene (HDPP) and Aluminium laminated pouches (ALP). They studied the storage stability and they investigated that the quality charecteristics such as free fatty acid, thiobarbituric acid, hydroxymethyl furfural, colour etc. were good in ALP when compared to HDPP.

Jaya and Das (2005), studied the shelf life and colour of mango powder. The powder was stored in aluminium foil, laminated pouches and stored in accelerated storage environment (90% RH and 38°C). The shelf-life of the powder was 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.

Mary *et al.* (2007), conducted experiments on the storage stability of spray dried banana powder, under ambient conditions. They noticed that the powder could be kept at ambient condition for one year. They used laminated aluminium foil pouches filled with nitrogen as the packaging material. The results showed that there was only less change in colour, flavor, organoleptic property etc.

Ramachandra and Rao (2013), produced aloe vera gel through dehumidified air drying of aloe vera gel. The optimized conditions were 64°C temperature, 18% relative humidity, and 0.8 m s<sup>-1</sup> air velocity. They used laminated aluminum foil, biaxially oriented polypropylene and polypropylene for the packaging of aloe vera powder. The storage stability of powder was mainly determined by the colour change and the best packaging material was the laminated aluminium foil.

Sagar and Kumar (2014) studied the influence of drying treatments and storage stability on quality characteristics of bael powder. They used tricalcium phosphate along with maltodextrin to prevent Caking. After the mechanical drying

process the powder was packed in 400 gauge and 200 gauge HDPE, 150 gauge PP and LDPE pouches and kept for storage at low temperature (7°C) and at ambient condition (18–35°C) up to 6 months. They got best results for the material packed in 200 gauges HDPE.

Wong and Lim (2016), studied the storage stability of spray dried papaya (*Carica papaya*) in aluminium laminated polyethylene (ALP) and polyethylene terephthalate (PET). After packing the product in the respective packaging material, the physical and microbial characteristics were examined after one week. The temperature and relative humidity of was  $38 \pm 2^{\circ}$ C, 90% respectively and they found the water activity does not increased more than 0.6 for both packaging materials. The moisture content of the product showed an increase in PET packets while comparing with ALP packets. The flowability was decreased in both packets after seven days of storage.  $\beta$ -carotene was degraded from 1.83  $\mu$ g/g to 0.95  $\mu$ g/g, 0.16  $\mu$ g/g in ALP and PET respectively. So they concluded that, the best material for packaging of papaya powder was ALP.

Singh and Hathan (2017) observed the moisture content and colour change of optimally produced beetroot powder packed in high density poly ethylene (HDPE) and laminate aluminum pouches (LAP). The storage condition was  $40 \pm 1^{\circ}$ C temperature, and 90% relative humidity. They found that laminated aluminum pouches was the best choice for keeping spray dried beetroot powder than HDPE.

#### **CHAPTER III**

#### **MATERIALS AND METHODS**

This chapter discusses in detail the materials and methodology adopted for the development and quality evaluation of spray dried neera powder.

#### 3.1 RAW MATERIALS

#### 3.1.1 Core Material

Neera procured for the study was from Central Plantation Crops Research institute (CPCRI) and Kerala Agricultural University (KAU) and was kept under refrigerated conditions prior to spray drying process. The CPCRI Neera harvested under refrigerated condition and stored in chiller boxes without processing. Whereas Neera collected from KAU was processed (applied for pattent) carbonated and bottled. The CPCRI Neera stored at -25°C and KAU Neera stored at 4°C because the thawing process may alter the quality of Neera.

Parameters	Kalparasa	Keramrutham
Variety	West cost tall	Tall and hydbrid
Age	5 years	20 - 40 years
Season	July - February	June - December
Geographical location	CPCRI Kasaragod	Collage of Agricuture, Padannakad

## 3.1.2 Wall Material (Carrier)

The wall materials used for the study were Gum arabic and maltodextrin. Gum arabic was purchased from M/s. Chemind, Thrissur, Kerala and Maltodextrin procured from M/s. Viveka agencies, Coimbatore, having a dextrose equivalence of 20 (20 DE).

The amount of wall material added with the core material mainly depends up on the %TSS of the core material. Since the CPCRI Neera has 16% TSS the

proportion of maltodextrin and Gum arabic taken was 2.5% MD + 0.25% GA, 5% MD + 0.5% GA and 7.5% MD + 0.75% GA. But KAU Neera contains 11.3% TSS, so in order to get higher product yield the proportion of maltodextrin and Gum arabic taken was 7% MD + 0.7% GA, 8% MD + 0.8% GA and 9% MD + 0.9 % GA.

#### 3.2. PHYSICO-CHEMICAL PROPERTIES OF RAW MATERIALS

## 3.2.1 Physico-chemical Properties of neera

## 3.2.1.1 Specific gravity

Specific gravity is the ratio of the density of a substance to the density (mass of the same unit volume) of a reference substance. Specific gravity of neera was calculated by dividing the weight of 1 ml neera by the weight of 1 ml distilled water (Sariga, 2016)

Specific gravity = 
$$\frac{\text{Density of substance}}{\text{Density of water (Reference)}}$$
 ...3.1

#### 3.2.1.2 pH

The pH of neera was analysed with the help of a digital pH meter (M/s. Systronics; Model MK VI), shown in (Plate 3.1). Before the analysis, the pH meter was standardised with double distilled water of pH 7.0 and standards of pH 4.0, 7.0 and 9.0.

#### 3.2.1.3 Colour

The colour of the liquid neera was determined by a Hunter lab colour flex meter (Hunter Association laboratory, Inc., Reston, Virgina, USA; model: HunterLab's ColourFlex EZ) which is shown in Plate 3.2. The Hunter lab's colour flex spectro calorimeter works on the principle of focussing the light and measuring energy reflected from the sample across the entire visible spectrum and it consists of a measurement (sample) port, opaque cover and display unit. The equipment required preliminary lights for matching a series of colour across the visible spectrum and it describes the colour by the mathematical model which is known as Hunter model. The colour measurements was given in terms of a three dimensional scale (L\*, a\* and b\* values). The L\* denote the lightness coefficient, ranging from 0 (black) to 100

(white), a\* gives greenness and redness (+100 for red and -80 for green) while b\* represents yellowness and blueness (+70 for yellow and -80 for blue) (Reddy *et al.*, 2014). The equipment should be calibrated before the analysis of sample. Calibration was done by placing black and white tile (provided with the instrument) at the measurement port. After the calibration process the sample was filled in the transparent glass cup placed over the port of the instrument and an opaque cover which act as a light trap to exclude the interference of external light was placed over the cup. After keeping the sample in port, the values of L\*, a\* and b\* were recorded.

#### 3.2.1.4 Total soluble solid

Total soluble solid (TSS) was measured by using a hand refractometer (Erma inc, Tokyo) as per AOAC, (1990), and expressed in degree Brix, shown in plate 3.3.

## 3.2.1.5 Total Poly Phenol

Total phenols were determined by Folin and Ciocalteu method. 800 µl of Folin Ciocalteu reagent mixture was added with 200 µl of the sample and to this mixture 2 ml of 7.5% sodium carbonate was added. The total content was diluted to 7 volumes with distilled water. The diluted samples were kept in the dark for 2 hours. The absorbance was messured at 760 nm and the galic acid dilutions were used as the standard solutions. The result was expressed in terms of gallic acid in mg/ml of extract (Prabhavathi *et al.*, 2016).

#### 3.2.1.6 Vitamin C

Vitamin C content of fresh neera was determined by using volumetric method as explained by (Sadasivam and Manickam, 1992). For the preparation of dye solution, 42 mg of sodium bicarbonate and 52 mg of 2, 6, dichloro phenol indophenols (dye) were added in 200 ml of distilled water. Stock solution was made by mixing 100 mg of ascorbic acid with 100 ml 4% oxalic acid. 10 ml of stock solution was diluted with 100 ml of 4% oxalic acid which is known as the working solution. Titration was done against the dye solution which was taken in the burette. At the end point, a pink colour will appear which will be observed for few minutes.

Repeat the titration for the concordant value. The amount of dye consumed (V1) will be equal to the amount of ascorbic acid present in the working standard solution. 10 ml of neera was taken and made up to 100 ml with oxalic acid (4%). Pipette out 5 ml of this solution in to conical flask and titrated against the dye (V2).

Equation for calculating the presence of ascorbic acid is given as

Ascorbic acid (mg/100g) = 
$$\frac{0.5}{V1} \times \frac{V2}{5} \times \frac{100}{Vs} \times 100$$
 ...3.2

## 3.2.1.7Antioxidant activity

The antioxidant activity of neera was determined by DPPH assay method. For that 0.394mg of DPPH was taken and mixed with 100ml of ethanol. From that 0.5ml, 1ml, 1.5ml dilutions were made and 3ml of DPPH solution added in to it. The control for the experiment taken was galic acid. The OD values of the samples taken at 517 nm. The inhibition rate is calculated by using the following formula, (Nazir *et al.*, 2013).

$$I\% = \frac{A_{c} - (A_{i} - A_{j})}{A_{c}}$$
 ...3.3

I = Scavenging rate

 $A_c = Absorbance of blank$ 

 $A_i$  = Absorbance in presence of sample

 $A_i$  = Absorbance of sample without DPPH

# 3.2.2 Physico-chemical Properties of Wall Materials

#### 3.2.2.1 Moisture Content

The moisture content of wall materials was determined as per AOAC (1999). Ten grams of accurately weighed sample was taken in petridishes and kept in electric oven at 70°C for 16 hours. Then the powder samples were taken out and weight of the samples were noted. The procedure was repeated until the difference between the two successive values of weights were not more than 0.5-1 mg. The moisture content of powder was calculated by using the initial and final weight of the powder. The equation used for calculating moisture content was recorded as

Moisture content (%wb) = 
$$\frac{W_i - W_d}{W_i} \times 100$$
 ...3.4

Where,

M = Moisture content, % (wet basis)

W<sub>i</sub> = Initial weight of the powder,(g)

 $W_d$  = Dry weight of the powder, (g).

## 3.2.2.2 Water Activity

The water activity of wall materials were analysed with the help of Aqua lab water activity meter (model: Aqua lab, Decagon Devices Inc., Pullman (Wa), USA), as shown in plate 3.4. Along with the equipment, some disposable cups were provided in which the powder samples were filled for the analysis purpose. It is important to note that the overfilling of cups may contaminate the sensors. After filling the cups the drawer knob of water activity meter will be turned to OPEN or LOAD position. After keeping the wall material sample in the drawer it was closed and then the knob turned to READ position. Water activity of powder sample was noted from the display unit of water activity meter (Kha *et al.*, 2010).

#### 3.2.2.3 Bulk Density

The bulk density of material was calculated by the method described by Santhalakshmi *et al.* (2015). In this method a known quantity of wall material sample is taken and loaded in to 10ml graduated cylinder and the volume occupied was noted and then bulkdensity was calculated and expressed in g/cm<sup>3</sup>.

Bulk density(g/cm<sup>3</sup>) = 
$$\frac{\text{Weight of the wall material}(g)}{\text{Volume of wall material}(cm^3)}$$
 ...3.5

#### 3.2.2.4 Colour

Colour values were measured by Hunter lab colour flex meter (Hunter Association laboratory, Inc., Reston, Virgina, USA) as described in the previous section 3.2.1.3. Wall material samples were filled in the transparent cups and placed over the port of colourimeter. The cup was covered with an opaque cover and the colour was recorded in terms of L\*, a\*, b\* values from the display unit.

## 3.2.2.5 Wettability

Wettability of wall material samples were analysed by the time taken for the complete wetting of the powder. Wall material sample (1.5g) was taken and then gently placed on the surface of 100 ml water at 30°C. With the help of a stop watch time taken for the complete wetting of powder was recorded (A/S Niro Atomizer *et al.*, 1978).

## 3.2.2.6 Cold Water Solubility

The cold water solubility of wall material samples were determined by the procedure described by Loksuwan (2007). One gram of sample was mixed with 100 ml of distilled water at room temperature for 30 minutes. Continuous stirring was provided by the magnetic stirrer. From this 10 ml aliquot was taken and transferred to 15 ml centrifuge tube and then centrifuged for 15 minutes. Supernatant was taken in pre weighed petridishes and then evaporated in oven at 110°C. Cold water solubility was measured by the formula,

Cold water solubility (%) = 
$$\frac{10 \times \text{Grams of solid in supernatant}}{\text{Grams of sample}} \times 100$$
 ...3.6

## 3.3 Physicochemical properties of feed material

The physico chemical properties of feed material such as specific gravity, pH, colour and TSS were determined as per the procedure explained in the previous section 3.2.1.1 to 3.2.1.4

## 3.3.1 Viscosity

The viscosity of feed material was measured by using a Brookfield DV-E Viscometer shown in Plate 3.5. The viscometer was provided with a spindle that can rotate inside the feed sample. While rotating the spindle in the feed matrix it offers a resistant force, and the torque required to overcome this force is measured by the instrument. Before the analysis, the equipment should be calibrated without the spindle from 0 to 100 rpm. Five hundred ml of feed sample was taken in a beaker and

the spindle (suitable for the feed) was immersed in to the matrix without touching the bottom and walls of beaker. After that the speed rpm was chosen for obtaining the torque. The viscosity values attained a steady state after 30s and the values were recorded.



Plate 3.1 pH meter



Plate 3.3 Refractometer



Plate 3.2 Colorimeter



Plate 3.4 Water Activity meter



Plate 3.5 Viscometer

3.4 EXPERIMENTAL DESIGN FOR THE OPTIMIZATION OF THE WALL MATERIAL, CORE CONCENTRATION, SPRAY DRYING TEMPERATURE AND FEED RATE FOR THE SPRAY DRYING OF NEERA

After the thorough studies of literature reviews and preliminary studies the process parameters (independent variables) for the spray drying process were chosen,

that can affect the product quality characteristics, utilization potential and storage stability.

In the present work, Response Surface Methodology (RSM) was used for the experimental design. RSM is the collection of statistical and mathematical techniques useful for developing formulations of new product as well as improving the existing product. It can clearly give the influence of independent parameters on the process. The design can optimize an output variable which is affected by a number of input variables. It includes a set of experiments (tests) known as runs. Experimental design was based on Box–Behnken design, which includes three levels including -1, 0, +1, where -1 indicates the lower limit value +1 is the upper limit value of the independent parameters opted for the spray drying process. The independent parameters selected for the drying process were concentration of feed solution (g) (core material + wall materials), inlet air temperature ( $^{\circ}$ C) and feed rate (ml/min). Number of runs were calculated by the formula N =2 k (k - 1) + Co, (K= number of factors and Co= number of central points). According to this study total numbers of runs were 17 and the statistical analysis was done by Design-Expert software (version 7.0.0).

While doing the statistical analysis all the independent parameters should be considered within range and the response should be maximum or minimum. Optimization process can be mainly done by numerical optimization, graphical method and by point prediction. All these were done based on the quality parameters. Acceptability of the model was decided by model analysis; Lack- of fit test and coefficient of determination (R<sup>2</sup>). The response model will have good fitness only when the R<sup>2</sup> value is greater than 0.80.

# 3.4.1 Development of Spray Dried Neera Powder

# 3.4.1.1 Independent Variables:

(MD: Maltodextrin, GA: Gum arabic)

a) C1: 2.5% MD and 0.25 % GA

(a) T1: 165

(a) F1: 4

(b) C2: 5% MD and 0.5% GA

(b) T2: 170

(b) F2: 5

(c) C3: 7.5% MD and 0.75% GA

(c) T3: 175

(c) F3: 6

(a) C1: 7% MD and 0.7 % GA

(b) C2: 8% MD and 0.8% GA

(c) C3: 9% MD and 0.9% GA

## 3.4.1.2 Dependent variables:

# (1) Characteristics of Neera (2) Characteristics of feed (3) Characteristics of powder

Specific gravity

pH

• Colour

TSS

• Phenolic content

Vitamin C

Anti-oxidant content

• Specific gravity

• pH

• Colour

TSS

Viscosity

Wettability

Bulk density

• Cold water solubility

Colour

Moisture content

Water activity

3.4.2 Physico-Chemical Properties of Optimised Neera Powder

(a) Dispersibility

(g) pH

(b) Vitamin C

(h) Flowability

(c) Phenolic content

(i) Titrable acidity

(d) Anti oxidant activity

(j) TSS

(e) Powder yield

(k) Total sugar

(f) Water activity

(l) Reducing sugar

(m) Colour

(p) Moisture content

(n) Cold water solubility

(q) Bulk density

(o) Wettability

# 3.4.3 Shelf Life Studies of the optimally produced Neera powder

- (a) pH
- (b) TSS
- (c) Phenolic content
- (d) Anti oxidant activity
- (e) Moisture content
- (f) Microbial load

#### 3.5 DEVELOPMENT OF NEERA POWDER BY SPRAY DRYING PROCESS

For obtaining the neera powder the feed is prepared with suitable conditions (Table 3.1 and 3.2) and subjected to spray drying process (Table 3.3 and 3.4). Spray drying is a rapid, one- step continuous process in which the liquid or slurry feed material is converted to powder form.

## 3.5.1 Preparation of Feed Solution

The core material is thoroughly mixed with the carrier agents and filtered with the aid of a muslin cloth, before subjecting to the atomization process to prevent the atomization difficulties. The study was conducted with two types of neera. The carrier materials were directly added in to the core material, 500 ml of feed solution was prepared based on the combinations and kept under refrigerated condition throughout the process since the chance of fermentation is more in atmospheric condition. After the atomization process, the water content in the feed droplets was evaporated and the dried powder was collected at the bottom of the drying chamber and at the cyclone.

The experiments were done using a lab scale spray dryer (vertical co-current SMST tall type spray dryer) as shown in plate 3.6. The evaporation rate of the spray dryer used for the present study was about 1000 ml/hr and it consists of hot air supply system, feed supply system, atomizer, drying chamber, powder recovery system and a control panel. Process flow chart for the development of neera powder is given in Fig.3.1.

## 3.5.1.1 Hot air supply system

Hot air supply system includes a compressor, air filter and an air heater. The air is compressed and subjected into the twin fluid pressure nozzle atomizer. Before that the air is passed through a filter and gets heated by the heater. The filter can prevent the entry of microorganism and it is an essential part in the air supply system. Air can be heated with electrical heating coils and the maximum temperature that can be achieved is 350°C. The compressed air splits the feed emulsion into fine mist which makes the drying process easier.

## 3.5.1.2 Feed supply system

The feed solution was supplied to the atomizer with the aid of a peristaltic pumb. The feed solution is filled in a 500 ml beaker, known as feed source was directly given to the peristaltic pump (DC operated). The pump is having five rollers through which a hypalon natural rubber tube with 6 mm diameter passes. The rollers can squeze the peristaltic tube along with the feed matrix, which helps in the forward movement of the feed and create vacuum behind each rollers. This vacuum can suck the feed solution again and the forward motion will be continued. The speed (rpm) of the pump can be controlled with a rotary knob.

#### 3.5.1.3 Atomizer

The feed solution was given into the drying chamber in the form of fine spray by means of a twin fluid pressure nozzle, from the ceiling of the drying chamber in downward direction. The feed and compressed air were given in to the nozzle at the same time. A cone shaped spray pattern is produced inside the drying chamber by this nozzle. The atomizer has an ability to produce wide range of flow rate and droplet size. The present study was conducted by choosing the air compressor pressure of 2 kg/cm<sup>2</sup>.

## 3.5.1.4 Drying chamber

The drying chamber of the spray drier is cylindrical in shape and taperd towards the bottom for the easy movement of dried powder. It is made up of SS 304 stainless steel. For avoiding the surface deposition of powder the inner surface of the chamber polished to 180-200 grit fineness. The atomizer is provided at the top of the chamber and the drying of the fine mist particles will take place in drying chamber. There are two inspection window glasses with light on two sides of the wall to see the operation takes place inside the drying chamber. A glass bottle of 1000 ml is flanged at the bottom through teflon gaskets at the conical portion of the drying chamber for collecting the dried powder.

## 3.5.1.5 Powder recovery system

Powder particles and hot air enters in to the cyclone jointly. The entry to the cyclone is through a duct of 65 mm diameter. In the cyclone, separation of fine powder particles are from the exhaust air is taking place. The swirling movement of air in the cyclone causes a pressure difference so that the less dense air leaves the cyclone and the powder particles collected in the glass bottles attached at the bottom of the cyclone through threaded flange with a teflon gasket.

## 3.5.1.6 Control panel

The operating parameters of the spray drying process were controlled through an electrical control panel with appropriate regulators, ON/OFF push buttons and indicators. Along with these an automatic and manual de blocking button, are also provided in the control panel in order to avoid the clogging of the atomizer.



Plate 3.6 Spray dryer

Distilled water was pumped in to the drying chamber in order to stabilize the outlet air temperature and after attaining the suitable stable temperature the feed was supplied to the spray dryer. The collection of the powder was done in laminated aluminum pouches, polyethylene pouches and in retort pouches for studying the shelf life characteristics in both ambient and refrigerated conditions. After collecting the powder in packets it was sealed by band sealer in which we can set the sealing temperature and speed. For laminated aluminum pouches and polyethylene pouches the suitable temperature was about 150°C where as for the retort pouches, 200°C

## 3.5.2 Standardisation of Spray Dryer Parameters

In the present study, spray drying was done with different process parameters such as feed concentration, inlet air temperature, and feed rate. Based on the quality, yield, stability, and appearance of the powder the process parameters were optimized. As per the previous literature reviews spray drying parameters like main blower rpm and outlet air temperature were kept constant. Blower rpm and outlet air temperature were selected as 1200 rpm and 75°C respectively. Atomization pressure for the study was kept as 2 kg/cm<sup>2</sup>.

For a spray drying process, there involves two main steps *viz*; preparation of stable feed emulsion and atomization of prepared feed emulsion in to the drying chamber followed by evaporation of moisture. Before giving the feed the spray dryer conditions should be achieved with distilled water as feed for 30 minutes. After

attaining suitable spray drying conditions the feed solution is given to the pump and finally to the spray dryer. The dried powder collected from preliminary and secondary outlets and was packed, sealed and stored in ambient as well as refrigerated conditions. Further analysis of the powder was done based on the technical programme.

Table 3.1 Coded and un-coded values of independent variables in Box-Behnken design for Spray drying of neera, from CPCRI (Kalparasa)

Independent variables	Code variables	Levels in coded form		
		-1	0	+1
Concentration of wall materials (%)	A	2.5% MD +0.25%GA	5%MD+ 0.5%GA	7.5%MD +0.75%GA
Spray drying inlet air temperature (°C)	В	165	170	175
Feed rate (rpm)	C	4	5	6

(MD – Maltodextrin, GA - Gumarabic)

Table 3.2 Coded and un-coded values of independent variables in Box-Behnken design for Spray drying of neera, from KAU (Keramrutham)

Independent variables	Code variables	Levels in coded form		
		-1	0	+1
Concentration of wall	A	7% MD	8%MD+	9%MD
materials (%)	71	+0.7%GA	0.8%GA	+0.9%GA
Spray drying inlet air temperature (°C)	В	165	170	175
Feed rate (rpm)	С	4	5	6

(MD – Maltodextrin, GA - Gumarabic)

Table 3.3 Box-Behnken experimental design for the production of spray dried neera powder (Kalparasa)

	Coded variables			Un-coded variables		
Run	Conc.of wall material (%)	Temper ature (°C)	Feed rate (rpm)	Conc.of wall material (%)	Tempe rature (°C)	Feed rate (rpm)
1	0	0	0	5	170	5
2	-1	-1	0	2.5	165	5
3	0	0	0	5	170	5
4	0	+1	+1	5	175	6
5	0	0	0	5	170	5
6	0	0	0	5	170	5
7	+1	0	+1	7.5	170	6
8	+1	0	-1	7.5	170	4
9	+1	+1	0	7.5	175	5
10	-1	0	+1	2.5	170	6
11	0	-1	-1	5	165	4
12	0	0	0	5	170	5
13	+1	-1	0	7.5	165	5
14	-1	+1	0	2.5	175	5
15	-1	0	-1	2.5	170	4
16	0	-1	+1	5	165	6
17	0	+1	-1	5	175	4

Table 3.4 Box-Behnken experimental design for the production of spray dried neera powder (Keramrutham)

	Coded variables			Un-coded variables		
Run	Conc.of wall material (%)	Temper ature (°C)	Feed rate (rpm)	Conc.of wall material (%)	Tempe rature (°C)	Feed rate (rpm)
1	0	+1	-1	8	175	4
2	0	0	0	8	170	5
3	0	-1	-1	8	165	4
4	-1	+1	0	7	175	5
5	+1	0	+1	9	170	6
6	0	0	0	8	170	5
7	0	0	0	8	170	5
8	-1	-1	0	7	165	5
9	0	0	0	8	170	5
10	0	-1	+1	8	165	6
11	-1	0	-1	7	170	4
12	-1	0	+1	7	170	6
13	0	+1	+1	8	175	6
14	+1	-1	0	9	165	5
15	+1	0	-1	9	170	4
16	+1	+1	0	9	175	5
17	0	0	0	8	170	5

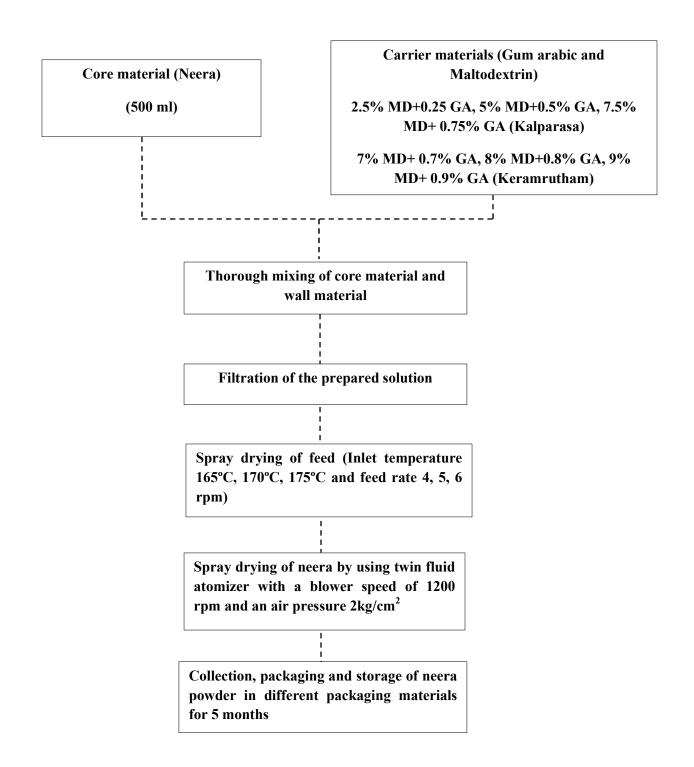


Fig. 3.1 Process flow chart for the production of spray dried neera powder

#### 3.6 QUALITY ANALYSIS OF SPRAY DRIED NEERA POWDER

#### 3.6.1 Moisture Content

The moisture content of the samples were calculated by hot air oven method as explained in section 3.2.2.1

## 3.6.2 Water activity

The water activity of neera powder was carried out using Aqua lab water activity meter (M/s. Aqua Lab, U.S.A; model: Series 3TE) as explained in the section 3.2.2.2.

#### 3.6.3 Colour characteristics

The colour of the neera powder was measured using Hunter lab colour flex meter (Hunter Association laboratory, Inc., Reston, Virgina, USA). Detailed procedure for colour determination was explained in the section 3.2.1.3. The spray dried neera powder was kept over the colour measuring port of the flex meter and 'L\*', 'a\*', 'b\*' values were recorded.

## 3.6.4 Bulk density

As per the procedure described in section 3.2.2.3 bulk density of the spray dried powder samples were calculated.

## 3.6.5 Cold Water Solubility

The cold water solubility of the spray dried neera powder samples were determined by the procedure described by Loksuwan (2007), as explained in the section 3.2.2.6.

## 3.6.6 Wettability

It was calculated by the procedure explained by A/S Niro Atomizer *et al.*, 1978, as narrated in the previous section 3.2.2.5.

# 3.7 QUALITY ANALYSIS OF OPTIMALLY PRODUCED SPRAY DRIED NEERA POWDER

#### 3.7.1 Dispersibility

Dispersibility was detrmined by the procedure explained by Jinapong *et al.*, (2008). One gram of powder was added to the 10 ml of distilled water and stirred

vigorously for 15s. After sieving, it was kept in hot air oven at 105°C and dried for 4h. The dispersibility of the powder was calculated as follows:

Dispersibility (%) = 
$$\frac{(10+a) \times \%TS}{a \times (100-b)/100}$$
 ...3.7

Where,

a = amount of powder (g)

b = moisture content in the powder and

%TS = dry matter in reconstituted powder.

# 3.7.2 Powder yield

Yield obtained after spray drying process was determined by calculating the ratio of weight of sample collected to the juice fed to the drier (Fazaeli *et al.*, 2012) and it is given by:

Powder yield (%) = 
$$\frac{\text{Solid content in the product}}{\text{Solid content in the feed}} \times 100$$
 ...3.8

## 3.7.3 Flowability and Cohesiveness

## 3.7.3.1 *Carr's index (CI)*

The carr's index or compressibility index was calculated from the loose bulk density and tapped bulk density (Jinapong *et al.*, 2008).

Carr's index (%) = 
$$\frac{\text{Tapped bulk density (g/ml) - Loose bulk density (g/ml)}}{\text{Tapped bulk density (g/ml)}} \times 100 \quad ...3.9$$

## 3.7.3.2 Hausner ratio (HR)

The cohesiveness was evaluated in terms of Hausner ratio (Shishir *et al.*, 2015). It was calculated from loose bulk density and tapped bulk density. The equation is given below:

Hausner ratio (HR) = 
$$\frac{\text{Tapped bulk density (g/ml)}}{\text{Loose bulk density (g/ml)}}$$
...3.10

# 3.7.4 Titrable acidity

Acidity of optimally produced powder was calculated by the titration method (No. 947.05 AOAC 2005). One gram powder was diluted to 10 ml and two drops of phenolphthalein indicator was added to it. This was taken in a conical flask and titrated against 0.1N NaOH taken in the burette until light pink end point appeared for few seconds. Volume of NaOH used was recorded. Experiment was repeated three times and the average acidity was taken that as titrable acidity of the product. The formula is given below:

Percentage acidity of powder = 
$$\frac{9 \times \text{ml of } 0.1 \text{ N NaOH} \times 0.1}{\text{Weight of powder (g)}}$$
...3.11

## 3.7.5 Water activity

It was calculated with the help of Aqua lab water activity meter as explained in the previous section 3.2.2.2.

## 3.7.6 pH

pH was measured with the aid of a pH meter as described in the section 3.2.1.2

#### **3.7.7** Colour

Colour measurment of the optimally produced powder was determined by the Huntel lab colour flex meter, as narrated in section 3.2.1.3.

#### 3.7.8 TSS

The total soluble solids were calculated after reconstituting the neera powder as mentioned in 3.2.1.4.

#### 3.7.9 Bulk density

It was calculated by the procedure was explained in previous section 3.2.2.3.

# 3.7.10 Wettability

It was calculated by taking the time required for the complete wetting of powder sample with the aid of a stop watch, described in section 3.2.2.5.

#### 3.7.11 Moisture content

Moisture content of powder sample was calculate by the equation 3.3 explained in section 3.2.2.1

#### **3.7.12 Vitamin C**

The method for calculating vitamin C content of the optimally produced neera powder was explained in section 3.2.1.6.

### 3.7.13 Phenolic content

According to the procedure explained by Jayaramu *et al.* (2016), phenolic content of the optimally produced neera powder was calculated.

# 3.7.14 Antioxidant activity

According to the procedure explained by Nazir *et al.*, 2013, as sexplained in previous section 3.2.1.7.

### 3.7.15 Reducing sugar

Twenty five gram of sample was ground with 100 ml of distilled water and ransfered to conical flask, was neutralized with 1N sodium hydroxide in the presence of phenolphthalein. Two ml of lead acetate was added to it for clarification of the mixture, followed by the addition of 2 ml of potassium oxalate. Allow the mixture to settle for 10 minutes and after that filter the mixture by using Whatman's filter paper. Aliquote of the mixture was titrated against a boiling mixture of Fehlings solution A and B using methelene blue as indicator. At the end point the appearance of brick red colour will occur and repeat the titration for the concordant value. It is calculated by,

Reducing sugar (%) = 
$$\frac{\text{Fehling's factor} \times \text{dilution} \times 100}{\text{Titre value} \times \text{weight of the sample}}$$
 ...3.12

# 3.7.16 Total sugar

The total sugar was determined using the method given by Ranganna, (1986). From the clarified solution used for the estimation of reducing ssugar, 50 ml was taken and boild gently after adding citric acid and water. It was then neutralized with sodium hydroxide and the volume made up to 250 ml. It was titrated against fehling solution A and B. The equation for calculating total sugar was given below,

Total sugar (%) = 
$$\frac{\text{Fehlins factor} \times 250 \times \text{dilution} \times 100}{\text{Titre value} \times 50 \times \text{weight of the sample}} \qquad \dots 3.13$$

# 3.8 SHELF LIFE STUDY OF THE OPTIMALLY PRODUCED SPRAY DRIED NEERA POWDER

Optimally produced neera powders was packed in aluminum laminated pouches, poly ethylene pouches and in 3 layer transp-arent retort pouches comprising 12 Mic Alox pet, 15 Mic Oriented Nylon, 70 Mic cast Propylene and sealed using a band sealer machine and kept for storage studies. Sealed pouches were stored at both ambient (25-30°C) and refrigerated conditions (4°C) for a period of 5 months. The effect of physico-chemical and microbiological characteristics during storage was studied. Analysis of sample at every 30 days interval was also done.

# 3.8.1Quality Attributes for the Storage Study

Qualaity parameters like moisture content, pH, TSS, anti oxidant content, and phenolic content were determined by the procedure as explained in 3.2 and 3.2.2

### 3.8.1.1 Determination of microbial load

Microbiological analyses of prepared samples were calculated by the total plate count method. The media prepared for the analysis was nutrient agar. Before the analysis, the prepared media and glass wares were sterilized in an autoclave at 121°C for 15 minutes. The total plate count was obtained by serial dilution process. For that one gram of sample was diluted in 10 ml of sterile water blank. The solution was mixed well for 10 minutes for getting a homogenized suspension; this solution was known as 10<sup>-1</sup> dilution. From this solution, 1 ml of aliquot was transferred to another

9 ml of sterile distilled water test tube and mentioned as 10<sup>-2</sup> dilution. Similar process was repeated up to 10<sup>-4</sup> dilution. After that the sterile petriplates were filled with sterile media. Pour the media into the pertiplates carefully and within the presence of a spirit lamb. After that one ml of prepared dilution samples were added to the petriplates that contains media. The entire processes were carried out in the laminar air flow chamber to prevent the contamination. The filled petriplates were rotated clockwise and in anticlockwise direction and kept undisturbed. After solidification of media, plates were inverted and incubated at room temperature for 2-5 days. Colonies were counted after the incubation period and calculated as colony forming units (CFU's) per gram of the sample. The equation used as follows

$$CFU = \frac{\text{Mean number of CFU's} \times \text{dilution factor}}{\text{weight of sample}} \qquad ...3.14$$

### 3.9 SENSORY EVALUATION

The neera prepared by reconstituting the neera powder was assessed for the sensory attributes like appearance, taste, flavor, and overall acceptability, by a 9 point hedonic scale test (Appendix C). The reconstitution was done at the ratio of 1:10. The fresh neera was kept as control for the comparison.

# 3.10 COST ANALYSIS FOR THE PRODUCTION OF SPRAY DRIED NEERA POWDER

The cost analysis for the production of neera powders by the spray drying technique was estimated by considering the fixed, variable and other related costs which include the following costs viz., cost of building, spray dryer, raw materials, processing, labour, electricity and other related costs...

### **CHAPTER IV**

### **RESULTS AND DISCUSSION**

This chapter deals with the results and discussion of the experiments conducted on neera powder obtained by spray drying process and its quality analysis. The chapter also discusses the quality changes occurred during the storage period of optimised sample.

# 4.1 PHYSICO-CHEMICAL PROPERTIES

# 4.1.1 Physico-chemical Properties of raw materials

# 4.1.1.1 Physico-chemical Properties of Neera

The physico-chemical properties of neera collected from CPCRI- Kasaragod (Kalparasa) and from Kerala Agricultural University (Keramrutham), were determined by standard procedures. Results thus obtained were statistically analysed and tabulated in table 4.1.

Table 4.1 physicochemical properties of Neera

Parameters	Parameters		Keramrutham
рН		$6.370 \pm 0.410$	$5.830 \pm 0.210$
Specific gravit	y (at 25°C)	$1.062 \pm 0.106$	$0.805 \pm 0.131$
TSS (°Brix)	TSS (°Brix)		$11.30 \pm 1.224$
Colour	L*	$8.525 \pm 4.637$	$0.99 \pm 3.231$
	a*	$-1.006 \pm 1.445$	$0.03 \pm 0.445$
	b*	$-1.896 \pm 3.052$	$-0.44 \pm 2.154$
Vitamin C (mg/100g)		$9.0275 \pm 2.046$	4.51± 1.034
Phenolic content (mg GAE) <sup>a</sup>		4.801 ± 1.3828	2.03± 1.261
Antioxidant ac	tivity (%)	8.10 ± 0.5	$6.3 \pm 0.3$

<sup>(&</sup>lt;sup>a</sup> GAE gallic acid equivalent)

The difference in major quality parameters between kalparasa and keramrutham might be due to the processing stages in keramrutham before carbonation process.

# 4.1.1.2 Physico-chemical Properties of wall materials

The physico chemical properties of carrier agents such as maltodextrin and gum arabic were analysed and tabulated in table 4.2. It was noted that the hygroscopic nature of gum arabic is more than that of maltodextrin. The moisture content of gum arabic is observed as  $4.010\pm0.011$  where as the moisture content of maltodextrin was only  $3.641\pm0.061$ . The bulk density values of gum arabic and maltodextrin were recorded as 0.615 g/cm<sup>3</sup> and 0.322g/cm<sup>3</sup> respectively. It was found that the water activity of gum arabic is slightly greater than that of maltodexxtrin. The recorded water activity values for gumarabic and maltodextrin were 0.364 and 0.340. Colour characteristics such as L\*, a\*, b\* values of gum arabic and maltodextrin were noted as 82.36, 2.37, 11.21 and 94.65, -0.54, 2.57 respectively. Both carrier agents showed higher solubility and wettability. All these properties can affect the final product quality directly or indirectly.

Table 4.2 Physico-chemical properties of wall materials

Physico-chemical pr	operties	Gum arabic	Maltodextrin
Moisture content (% v	Moisture content (% wb)		3.641±0.061
Water activity	Water activity		0.340±0.006
Bulk density (g/cm <sup>3</sup> )		0.615±0.036	0.322±0.011
Colour	L*	82.36±0.548	94.65±0.807
	a*	2.37±0.007	-0.54±0.014
b*		11.21±0.034	2.57±0.010
Wettability (s)		112.94±3.04	130.5±4.10
Solubility (%)		97.05±0.66	94.96±1.54

# 4.1.2 Physico-chemical Properties of feed

The physicochemical properties of the two feed solutions (neera and wall materials) prepared with kalparasa and keramrutham were checked for different combinations and analysed based on the standard procedures were given in table 4.3 and 4.4

Table 4.3 Physico-chemical characteristics of feed (Kalparasa) solution

Parameters		Feed combination			
		2.5% MD+0.25%GA	5%MD+0.5%GA	7.5%MD+0.75%GA	
TSS (°Brix)		$18.96 \pm 2.040$	$20.15 \pm 0.070$	21.95 ±0.919	
рН		6.505 ±0.275	$6.630 \pm 0.115$	6.055 ±0.360	
Specific gravity		0.941 ±0.052	$1.035 \pm 0.053$	1.115±0.021	
	L*	7.69 ±1.42	$8.30 \pm 1.03$	14.7 ±1.54	
Colour	a*	-0.67±0.23	-0.66±0.34	-0.48±0.47	
	b*	-2.75±0.42	-2.30 ±0.32	-0.55 ±0.25	
Viscosity (cp)		4215±14.14	4236±12.24	4312 ±11.23	

(MD- Maltodextrin, GA- Gum arabic)

Table 4.4 Physico-chemical characteristics of feed (Keramrutham) solution

Parameters		Feed combination			
1 01 011		7%MD+0.7%GA	8%MD+0.8%GA	9%MD+0.9%GA	
TSS (°Brix)		$18.31 \pm 0.020$	21.56 ±0.213	23.31 ±0.216	
рН		5.931 ±0.254	6.01±0.247	$6.06 \pm 0.250$	
Specific gravity		0.91 ±0.043	0.93 ±0.046	0.96±0.212	
Colour L*		3.42 ±1.22	4.1 ±1.06	5.32±1.37	
	a*	0.12±0.14	0.08±0.25	0.01±0.29	

1	b*	-2.28±0.32	-1.84 ±0.42	-1.65 ±0.34
Viscosity (cp)		4185±12.10	4246±12.17	4286 ±9.27

(MD- Maltodextrin, GA- Gum arabic)

From the table 4.3 and 4.4 it is clear that the addition of carrier materials can affect the physico-chemical properties of raw neera. Adiition of maltodextrin and gum arabic increased the total soluble solid content in both neera. Not anly the TSS, the pH, Specific gravity, viscocity were also increased with the addition of wall materials. These parameters also change (increase) along with combination ratio of maltodextrin and gum arabic. If more concentration of maltodextrin and gum arabic were used in the preparation of feed solution it can increase the pH, TSS, Specific gravity, viscocity etc. In the case of colour characteristics; the lightness coefficient (L\*) showed an increasing behaviour where as a\* and b\* values were declining with the increase in carrier material concentration.

# 4.2 EXPERIMENTAL DESIGN FOR THE OPTIMISATION OF THE WALL MATERIAL, CORE CONCENTRATION SPRAY DRYING TEMPERATURE AND FEED RATE FOR THE SPRAYDRYING OF NEERA

The best combination of process parameters were determined by Box-Behnkon experimental design explained in section 3.4. The spray drying study was conducted with the aid of a lab model vertical co current SMST tall type spray dryer. Different combination of maltodextrin and Gum arabic were used for the spray drying study at different operating parameters such as inlet air temperature and feed rate explained in section 3.5. The outlet air temperature (75°C), blower speed rpm (1200 rpm) and atomization pressure (2kg/cm²) were kept constant during the entire study. Based on the quality of the spray dried powders the process parameters were optimized. The quality of spray dried neera powder was evaluated based on moisture content, water activity, bulk density, cold water solubility and colour. The analysis of variance (ANOVA) was done to determine the significant effects of process

parameters on each response. The significance of each response was checked by using P- values. When the P-value is less than 0.05then the model terms will be significant. If the P-value is smaller, then the model becomes more significant. The acceptability of model was checked by R<sup>2</sup>, adjusted R<sup>2</sup>, adequate precision and F test (Montgomery, 2001).

The variation around the mean was denoted by adjusted R<sup>2</sup> and it is adjusted for the number of terms in the model. When there is an increase in number of terms the adjusted R<sup>2</sup> decreases. Adequate precision compares the range of predicted values at design points to the average prediction error. Optimization of process variables was done by differentiating the model with respect to each parameter and was equated to zero. The statistical calculation was done by regression coefficients. That can develop three dimensional plots for the regression models. The plotted three dimensional response surfaces can reveal the relationship between the process variables and response variables.

# 4.2.1 Physico-chemical properties of spray dried neera powder

# 4.2.1.1 Effect of spray drying parameters on moisture content

It is the total amount of water present in the food which determines the stability and caking property of food products during their storage. According to Barroso *et al.*, 2014 food products having moisture content in between 3-10%will show good storage stability. Here in the present work the effect of feed concentration, inlet air temperature and feed rate rpm on the moisture contents of the spray dried products was calculated and tabulated in appendix A1 and appendix B1

The moisture content of powder produced from Kalparasa showed a range of 2.41 -3.89 % wb, whereas for the keramrutham moisture content was ranging from 2.32 -3.78% wb. In both cases the moisture content was higher for the combination of 2.5% MD + 0.25% GA. The treated temperature for this combination was 165°C. The minimum value for the moisture content was observed for the combination 7.5% MD + .75% GA. And the temperature for the treatment was 175 °C.

Fig.4.1 and fig.4.2 shows the 3-D graphs representing the effect of feed combination, inlet air temperature and feed rate on the moisture content of the spray dried neera powders (Equation 4.1 and 4.2).

From the graph it is clear that the inlet air temperature have a great influence on the moisture content of spray dried powders. The moisture content of the powders showed a decreasing tendency when the inlet air temperature was increased from 165-175°C. The difference between the inlet and outlet air decreases the relative humidity of the dryer air so the moisture content of the product decreases. Similar results were reported by Goula *et al.*, (2004), Chegini and Ghobadian (2005). They studied the effect of temperature on the moisture content of tomato powder and orange juice powder and observed that there is a decrease in moisture content of both powders while increasing the temperature.

The increase in maltodextrin and gum arabic ratio negatively affects the moisture content. When the carrier concentration increases from (2.5% MD + 0.25%GA) to (7.5% MD + .75% GA) the moisture content reduced from 3.78 to 2.32. Similar results were described by Kha *et al.*, (2010). They studied the effect of spray drying conditions on the moisture content of spray dried Gac (*Momordics cochinchinensis*) fruit aril powder. They observed that the increase in maltodextrin concentration from 10% to 20% reduced the moisture content of samples from 4.87% to 4.06%. Similarly Abadio *et al.*, (2004) found that an increased concentration of maltodextrin (10 DE) (10-15%) reduced the moisture content of pineapple juice powders, it could be due to an increase in solids in the feed and decreased amount of free water for evaporation. Addition of gum arabic made a slight increase in the moisture content may be due to its higher hygroscopicity (Tonon *et al.*, 2012).

There seems a slight increase in the moisture content of neera powder while increasing the feed rate. Maximum moisture content observed for the feed rate 6 was 3.89 and it gradually decreases with decrease in feed rpm. The increase in feed rate increases the flow of feed emulsion in to the atomizer which resulted in to the

formation of larger droplets. This can increase the space between the inner part and outside surface of droplets so that there will not be effective transformation of moisture content from the inside to outside surface leading to an increased moisture content of powders, (Tan *et al.*, 2011).

A second order non linear regression equation was fitted between dependent and independent variables using the experimental values. Regression models are given below (4.1 and 4.2). By using the models the moisture content of neera powder produced by the spray drying process can be predicted.

Moisture content = 
$$3.78 + 3.750E - 003A - 0.54B + 0.029 C - 0.38AB + 0.33AC$$
  
 $0.070BC - 0.27A^2 - 0.48B^2 + 0.11C^2$  ...4.1  
Moisture content =  $3.25 - 0.071A - 0.52B + 0.17C - 0.025 AB + 0.048 AC$   
 $+0.057BC - 0.031A^2 - 0.14B^2 - 0.054C^2$  ...4.2

Where A: Feed concentration (maltodextrin and gum arabic)

B: Spray dryer inlet temperature (°C) and

C: Feed rate (rpm).

The above equations showed that there will be significant effect for the processing variables (A, B, C) on the moisture content of neera powder. ANOVA tables for moisture content of Kalparasa and Keramrutham were shown in Appendix A and Appendix B (Table A2 and table B2). The p values for both models were recorded as 0.0008 (For kalparasa) and 0.0052 for (keramrutham). From the table it has been noted that the model fitness was significant since the R squared value obtained were 0.9516 (kalparasa) and 0.9153 (Keramrutham) which means that 95.16 and 91.53 per cent of the variability of the response could be explained by the model.

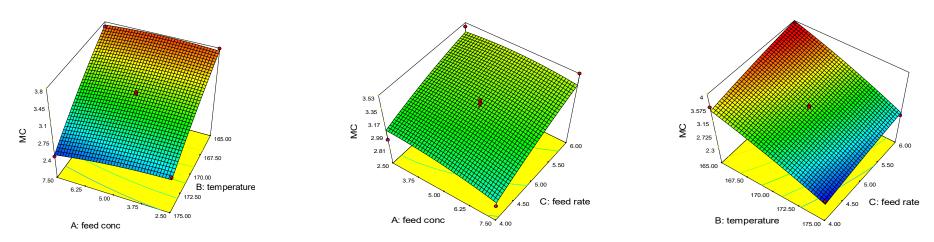


Fig. 4.1 Effect of process variables on moisture content of Spray dried Kalparasa powder

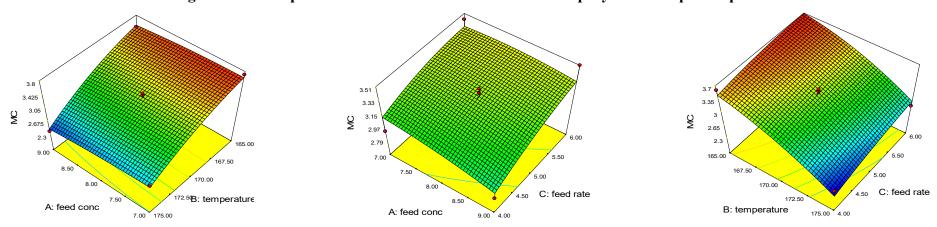


Fig. 4.2 Effect of process variables on moisture content of Spray dried Keramrutham powder

# 4.2.1.2 Effect of spray drying parameters on Water Activity

It is the amount of water present in the food material in which the micro organism can grow. Water activity is a very important response since it can affect the shelf life of the spray dried powder, due to the growth of microorganism. If the water activity of a commodity is higher in value, it reduces the storage life of that particular commodity. The biochemical and microbiological activity within the product will be prevented if the water activity was lower than 0.6, (Kha *et al.*, 2010).

The water activity for both spray dried powders (Kalparasa and keramrutham) was tabulated in Appendix A (Table A3) and Appendix B (Table B3) respectively. The water activity range obtained for kalparasa powder was 0.165 to 0.351 where as for keramrutham powder it was recorded as 0.163 to 0.321.

From the table A3 and B3 it is observed that the feed combination 5% MD+ 0.5%GA, processed at 165°C, with 6 rpm feed rate (Kalparasa) and 7%MD+0.7% GA processed at 165°C, with 5 rpm feed rate (Keramrutham) have highest water activity, while lowest value was obtained for those produced with 5% MD + 0.5% GA, processed at 175°C, with 6rpm feed rate (kalparasa)and 9% MD+0.9%GA processed at 175°C, with 5 rpm feed rate (Keramrutham).

The obtained water activity values were presented in three- D graphs fig. 4.3 and 4.4 respectively. Graph represents the effect of different process parameters on the water activity of spray dried powders. It has been noted that water activity showed a negative effect for an increase in temperature similar to the moisture content effect. Similar results were reported by Demertzis *et al.*, 1989. The kinetic energy of water molecules will increase as the temperature increases. So the water absorbance at a given water activity will be low. Koc *et al.*, 2010 also explained the same effect.

From the figure it was clear that by increasing maltodextrin, gum arabic combination, inlet air temperature, and decreasing feed flow rate led to a decreased water activity condition of spray dried powders. Similar trends was narrated by Fang

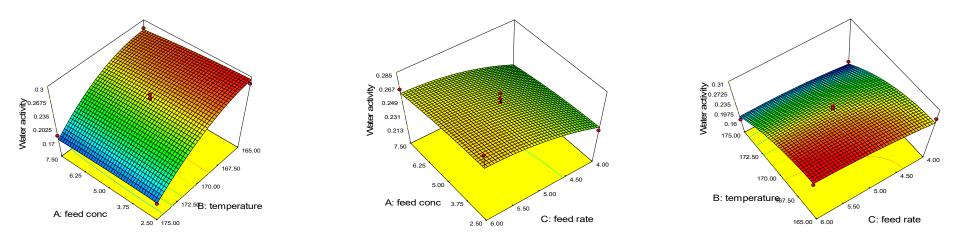


Fig. 4.3 Effect of process variables on water activity of spray dried kalparasa powder

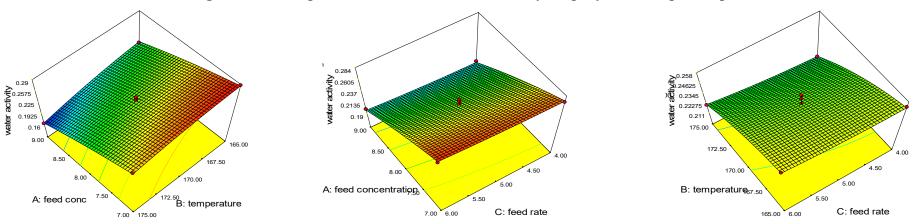


Fig. 4.4 Effect of process variables on water activity of Spray dried keramrutham powder

and Bhandari (2011) in black bay berry juice powder and fazaeli *et al.*, 2012 in black mulberry juice powder. While comparing the obtained water activity values of spray dried kalpasra and keramrutham powder it has been observed that water activity value of keramrutham powder was less than kalparasa powder. The highest  $a_w$  value obtained for keramrutham powder was recorded as 0.288 and as 0.3 for kalparasa powder.

The regression model (4.3 and 4.4) for the water activity of spray dried powders was given below:

Water activity =0.26 +4.125E-003A -0.057 B +0.018 C+2.500E-004A B -6.500E 003AC -6.750E-003BC -5.000E-005A<sup>2</sup> -0.027 B<sup>2</sup> -4.550E-003 C<sup>2</sup>....4.3

Water activity = 0.24- 0.042A -0.019B + 4.125E-003C -5.250E -003AB -2.500E-004AC +1.000E-003BC-3.300E-003A<sup>2</sup>-4.550E-003B<sup>2</sup>+1.450E 003C<sup>2</sup>...4.4

Table A4 and B4 (Appendix A and B) gives the analysis of variance done to evaluate the coefficient of quadratic polynomial models. The p value obtained from table A4 was 0.0009 indicates that the model is significant and the significant model terms obtained in ANOVA table was B, C, and B<sup>2</sup>. The R<sup>2</sup> value was found to be 0.9507.

ANOVA for spray dried Keramrutham powder is shown in table B4. From the table the p value obtained was less than 0.0001 and the significant model terms explained here were A and B. The recorded R<sup>2</sup> value was 0.9803.

# 4.2.1.3 Effect of spray drying parameters on Bulk Density

Bulkdensity (apparent/volumetric density) is not an intrinsic property of a material. Based on the way of material handling the value of bulk density could be changed. The property is important in packaging and transportation, since it can significantly reduce the costs. If the powder particle is with higher bulk density, then more amount of powder material can be stored in smaller containers when compared to low bulk density powders.

The change in bulk density of spray dried powders with different feed combinations, inlet air temperature and feed rate where analysed and given in Table A7 (Appendix A) and Table B7 (Appendix B) respectively.

The bulk density of spray dried kalparasa powder was varied between 0.294-0.438 gm/cm<sup>3</sup>. The highest bulk density value obtained with 2.5%MD+0.25%GA feed combination, processed at 165°C for 5 rpm feed rate. Whereas the lowest value obtained for the combination 7.5%MD+0.75%GA spray dried at 175°C, for 5 rpm feed rate.

For spray dried keramrutham powder the range obtained for bulk density was 0.296-0.44gm/cm<sup>3</sup>. Feed mixed with 7%MD+0.7%GA produced higher bulk density powder processed at 165°C for 5 rpm feed rate. Whereas lowest bulk density was noted for the combination prepared by mixing 9%MD+0.9%GA, was dried at 175°C for 5 rpm feed rate.

The effects of spray drying parameters on the bulk density of spray dried powders were shown in 3D graphs (Fig.4.5 and 4.6 respectively) which represents response surface generated by the regression model (equation 4.5 and 4.6)

It was observed that among the 3 spray drying process parameters inlet air temperature showed a greater influence on the bulk density of spray dried powder. Here in this process study the temperature negatively affects the bulk density of both spray dried powders. The reduction in bulk density may be because of the rapid formation of dried layer at the droplet surface. Thus the case hardening of droplets takes place and this forms a water impermeable layer. The condition resulted in the formation of higher moisture content product. So the powder with higher moisture content showed a higher bulk density value. These results are consistent with the findings of various studies (Chegini and Ghobadian, 2005, Cai and Corke, 2000)

The feed combination and feed rate does not show a wide range difference in the bulk density value. But there is a small decrease in the bulk density value while increasing the maltodextrin, Gum arabic combination and feed rate. This may be due

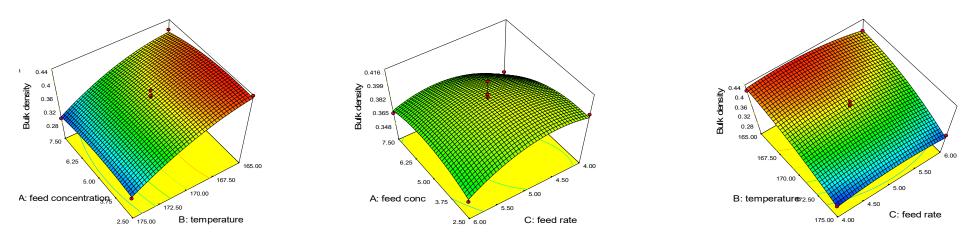


Fig. 4.5 Effect of process variables on bulk density of spray dried kalparasa powder

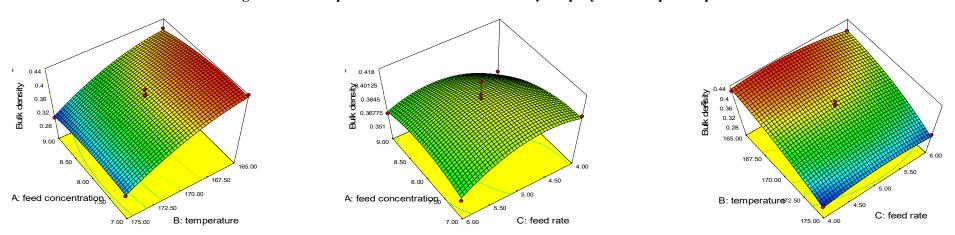


Fig. 4.6 Effect of process variables on bulk density of spray dried keramrutham powder

to the fact that increased maltodextrin proportion might have increased the volume of air that get trapped in the particles, cause decrease in apparent density of powder particles, (Santha Lakshmi *et al.*, 2015)

The regression model for predicting the bulk density of spray dried powder samples were given in equation 4.5 and 4.6 respectively.

Bulk density = 
$$0.40 - 6.625E - 003A - 0.057B - 4.125E - 003C + 3.000E - 003AB$$
  
+ $0.012AC + 7.500E - 003BC - 0.014A^2 - 0.022B^2 - 0.016C^2 - 0.4.5$ 

Bulk density = 
$$0.40 - 8.625E - 003A - 0.057B - 3.500E - 003C + 2.750E - 003AB$$
  
+ $0.011AC + 9.000E - 003BC - 0.011A^2 - 0.020B^2 - 0.018C^2 \dots 4.6$ 

Analysis of variance was done to get the coefficients of the quadratic polynomial model, given in Appendix A (Table A8) and Appendix B(Table B8) for both kalparasa and keramrutham powders.

For both spray dried keramrutham and kalparasa powders the p value obtained were 0.004, indicates that the model terms were significant (B,  $B^2$  and  $C^2$ ) and was concluded with obtained  $R^2$  value , 0.9620 (96.20% for keramrutham powder) and 0.9616 (96.16% for kalparasa powder).

### 4.2.1.4 Effect of spray drying parameters on wettability

It is the tendency of fluid to spread on to a solid surface (Anderson, 1986). Wettability is one of the reconstitution properties which determine the extent of solubility of spray dried powders expressed in seconds, mainly takes place with the help of capillary force. The powder or solid particles will immerse in water and they get dissolved in the immersing medium (if soluble). In the present study the wettability was calculated by noting the time taken to completely dissolve the spray dried powder in distilled water and it is tabulated in Appendix A (Table A5) and Appendix B (Table B5).

For spray dried kalparasa powder the wettability values varied from 55 seconds to 110 seconds. Maximum value was noted with the feed combination ratio

of 7.5% MD+0.75% GA, 175°C inlet air temperature and for 5 rpm feed rate. The minimum value obtained for the feed combination 2.5%MD+ 0.25% GA, spray dried at 165°C, for 5 rpm feed rate. While checking the wettabilty of spray dried keramrutham powder, it was found that the maximum and the minimum values ranges from 63-108 seconds. Here the minimum value was recorded for the combination 8%MD+ 0.8% GA processed at 165°C and for 4rpm feed rate. 9%MD+ 0.9% GA feed combination produced the highest wettability powder which required more time to wet and was processed at 175°C for 5 rpm feed rate. The effect of spray drying parameters on wettability of kalparasa and keramrutham powder shown in 3D graphs, (Fig 4.7 and 4.8) which represents the response surface generated by the regression model (equation 4.5 and 4.6).

The graph gives a perfect relationship between the wettability and process parameters. The wettability shows a positive relation to the increase in temperature, may be because increasing the temperature causes structural damages and more dried powder. So that the dried powders will take more time to wet.(Reddy *et al.*, 2014) reported similar results in their work and narrated that wetting time is lower for reduced moisture content goat milk powder at high inlet air temperature

Increasing the maltodextrin and gum arabic combination along with the core material also increases the wetting time. According to Vega and Roose 2006 the wettability increases with increase in carrier agents and it depends on powder particle size, density, porosity and surface area.

Increase in feed rate increases the moisture content of powder particles, so that the wetting time required was observed as less (Fernandes *et al.*, 2013)

The regression mode for predicting the wettability of spray dried powder samble is given in equation 4.7 and 4.8 respectively.

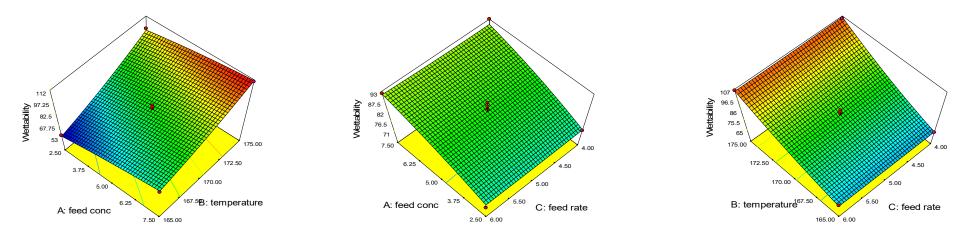


Fig. 4.7 Effect of process variables on wettability of spray dried kalparasa powder

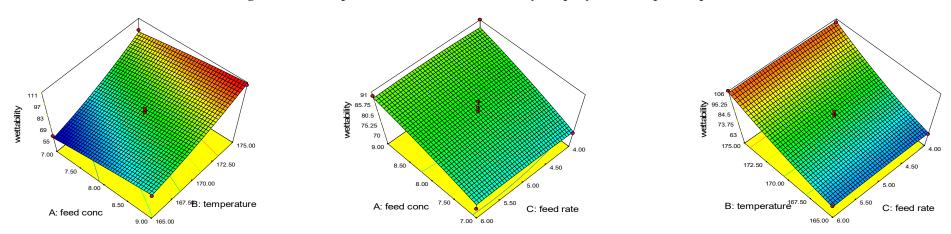


Fig. 4.8 Effect of process variables on wettability of spray dried keramrutham powder

to the fact that increased maltodextrin proportion might have increased the volume of air that get trapped in the particles, cause decrease in apparent density of powder particles, (Santha Lakshmi *et al.*, 2015)

The regression model for predicting the bulk density of spray dried powder samples were given in equation 4.5 and 4.6 respectively.

Bulk density = 
$$0.40 - 6.625E - 003A - 0.057B - 4.125E - 003C + 3.000E - 003AB$$
  
+ $0.012AC + 7.500E - 003BC - 0.014A^2 - 0.022B^2 - 0.016C^2 ... 4.5$ 

Bulk density = 
$$0.40 - 8.625E - 003A - 0.057B - 3.500E - 003C + 2.750E - 003AB$$
  
+ $0.011AC + 9.000E - 003BC - 0.011A^2 - 0.020B^2 - 0.018C^2 \dots 4.6$ 

Analysis of variance was done to get the coefficients of the quadratic polynomial model, given in Appendix A (Table A8) and Appendix B(Table B8) for both kalparasa and keramrutham powders.

For both spray dried keramrutham and kalparasa powders the p value obtained were 0.004, indicates that the model terms were significant (B,  $B^2$  and  $C^2$ ) and was concluded with obtained  $R^2$  value , 0.9620 (96.20% for keramrutham powder) and 0.9616 (96.16% for kalparasa powder).

### 4.2.1.4 Effect of spray drying parameters on wettability

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For spray dried kalparasa powder the wettability values varied from 55 seconds to 110 seconds. Maximum value was noted with the feed combination ratio

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The graph gives a perfect relationship between the wettability and process parameters. The wettability shows a positive relation to the increase in temperature, may be because increasing the temperature causes structural damages and more dried powder. So that the dried powders will take more time to wet.(Reddy *et al.*, 2014) reported similar results in their work and narrated that wetting time is lower for reduced moisture content goat milk powder at high inlet air temperature

Increasing the maltodextrin and gum arabic combination along with the core material also increases the wetting time. According to Vega and Roose 2006 the wettability increases with increase in carrier agents and it depends on powder particle size, density, porosity and surface area.

Increase in feed rate increases the moisture content of powder particles, so that the wetting time required was observed as less (Fernandes *et al.*, 2013)

The regression mode for predicting the wettability of spray dried powder samble is given in equation 4.7 and 4.8 respectively.

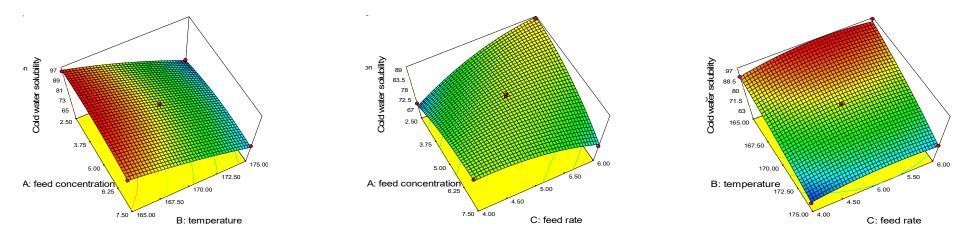


Fig. 4.9 Effect of process variables on cold water solubility of spray dried kalparasa powder

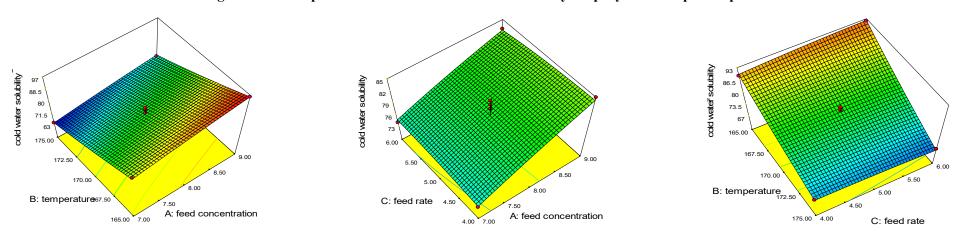


Fig. 4.10 Effect of process variables on cold water solubility of spray dried keramrutham powder

water solubility where as B, C, AB, AC, BC and A<sup>2</sup> negatively affects the cold water solubility.

For keramrutham powder p value obtained is less than 0.0001. The noted  $R^2$  value is 0.9775 means that the model could account 97.75% variability. Here the model terms A and B are significant.

# 4.2.1.6 Effect of spray drying parameters on colour

The acceptability of powder particles mainly depends on the colour of the powder material.

#### L\* value

The L\* value of spray dried Kalparasa powder varied from 83.48 to 90.69. The highest value of L\* value observed for the feed combination 7.5% MD+0.75% GA processed at 170°C for 6 rpm feed flow rate, where as lower value noted at 175°C for the feed combination 5%MD+ 0.5% GA for the feed flow rate 4 rpm.

For keramrutham powder the lowest value (84.62) of L\* was recorded for the combination 8% MD + 0.8% GA dried at  $175^{\circ}$ C for 4 rpm feed rate, whereas highest value (91.34) was observed for the feed combination 9% MD + 0.9% GA, processed at  $170^{\circ}$ C for the feed flow rate 6 rpm.

Fig.4.11 and fig.4.12 represents the 3-D graphs of spray dried kalparasa and keramrutham powder respectively. When the temperature increases from 165 to 175°C the colour (lightness, L\* value) decreases gradually. This effect might be due to the action of higher temperature on the maltodextrim compounds resulting in to the formation of non enzymatic browning (Quek *et al.*, 2007). Lee *et al.*, 2017 reported that there might be caramelization and millard reactions for sugar compounds at higher temperatures and thus the powder samples turns in to yellow colour slightly. From both figures it was clear that increasing maltodextrin, Gum arabic concentration and feed flow rate showed positive effect on the L\* value. This is because of the shorter contact period between powder particles and hot air at drying chamber. Similar observations were done by Kha *et al.*, 2010 for Gac aril powder

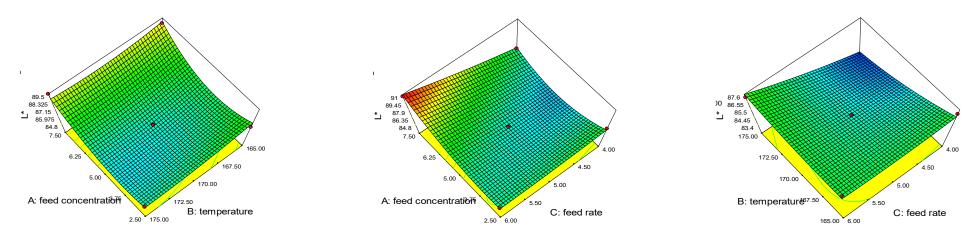


Fig. 4.11 Effect of process variables on colour (lightness value) of spray dried kalparasa powder

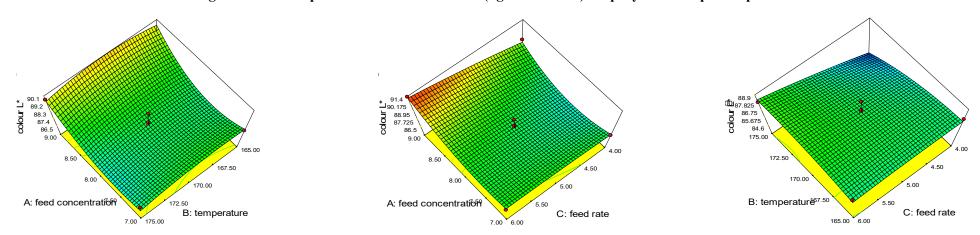


Fig. 4.12 Effect of process variables on colour (Lightness) of spray dried keramrutham powder

The effect of process parameters on the L\* value of spray dried kalparasa and keramrutham powder was shown in Appendix A (Table A11) and Appendix B (Table B11) respectively.

Regression model for predicting the L\* value of both spray dried powders given in equation 4.11 and 4.12.

$$L^* = 85.66 + 1.16A - 0.45B + 0.92 C + 0.31AB + 1.31AC + 0.89BC + 1.75A^2 + 0.38 B^2 + 0.20C^2 \qquad ...4.11$$

$$L^* = 87.55 + 1.08A - 0.23B + 0.76C + 0.17 AB + 0.47 AC + 0.82BC + 1.44 A^2 - 0.50B^2 - 0.13C^2 \qquad ...4.12$$

The p value for kalparasa powder obtained was 0.0016, means that the model fitness was significant. A, AC, C, BC,  $A^2$  were significant model terms and the  $R^2$  value was found to be 0.9403. For keramrutham powder the model showed a p value of 0.0357 which showed that the model terms is significant. F- Value of 4.21 also implies that the model is significant and the significant model terms were A, AC,  $A^2$ . The observed  $R^2$  value was 0.8440.

# a\* value

Appendix A (Table A13) and Appendix B (Table B13) shows the a\* value of spray dried kalparasa and keramrutham powder respectively. For kalparasa powder the highest value (1.38) found for the feed combination 2.5% MD+ 0.25% GA processed at 175°C for 5 rpm feed rate and lower value (0.36) observed for the feed combination 5% MD + 0.5% GA, dried at 170°C, for a feed flow rate 5rpm. In the case of keramrutham powder the highest value (1.34) showed by 7% MD+ 0.7% GA at 175°C, for 5 rpm feed flow rate, where as lower value (0.34) showed by 8% MD+ 0.8% GA feed combination, 170°C, and 5rpm feed rate.

The 3-D graphs of the relationship between the processing parameters and a\* value of spray dried powders shown in fig. 4.13 and 4.14.

From the graph, in both cases temperature showed a positive effect on the a\* value whereas the feed combination and feed rate showed negative effect on the

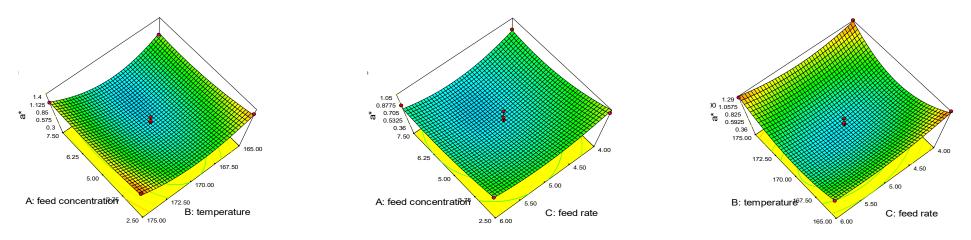


Fig. 4.13 Effect of process variables on a\* value of spray dried kalparasa powder

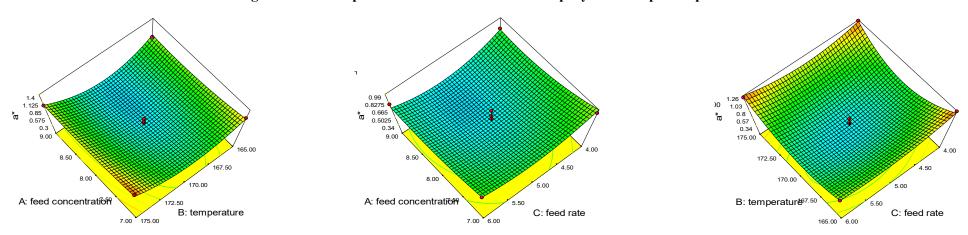


Fig. 4.14 Effect of process variables on a\* value of spray dried keramrutham powder

redness value. The increase in redness value at higher temperature might be due to the non enzymatic browning of sugar or starchy materials (Quek *et al.*, 2007). For an increase in the maltodextrin along with the core material reduces a\* value (redness). Regression model for predicting a\* value of spraydried kalparasa and keramrutham powder (Equation 4.13 and 4.14) given as follows.

$$a^* = 0.61-0.100A +0.069B -0.049C +5.000E-003AB +0.000AC +0.058BC+0.12A^2 +0.43B^2 +0.17C^2$$
 ...4.13 
$$a^* = +0.59 -0.090A +0.071B -0.044C -0.025AB +0.000AC +0.058BC +0.094A^2 +0.41B^2 +0.17C^2$$
 ...4.14

For kalparasa powder p value obtained was 0.0174 and F value noted was 5.52. Obtained  $R^2$  value for model was 0.8764. According to spray dried keramrutham powder p value obtained for the model was 0.0219 and  $R^2$  value obtained was 0.8670. In both cases  $B^2$  was the significant model term.

# b\* value

b\* value obtained for both kalparasa and keramrutham powder showed in Appendix A (Table A15) and Appendix B (Table B15). It was noted that in the case of kalparasa powder the highest b\* value (11.36) observed for the feed combination 2.5% MD+ 0.25%GA processed at 175°C for 5 rpm feed flow rate and lowest value (5.92) recorded for the feed combination 7.5% MD+ 0.75%GA dried at 165°C at 5rpm feed flow rate. For keramrutham powder the b\* value ranges from 80 (8% MD+ 0.8% GA, 165°C and 4 rpm feed rate) to 87.79 (8% MD+ 0.8% GA, 170°C and 5 rpm feed rate).

Effect of feed combination, inlet air temperature and feed flow rate on the yellowness of the kalparasa and keramrutham powder is given in fig. 4.15 amd 4.16. The yellowness mainly influenced by inler air temperature. As the temperature increases from 165°C to 175°C, the yellowness also increases. But in both case the fed combination and feed flow rate showed a negative effect on the yellowness of

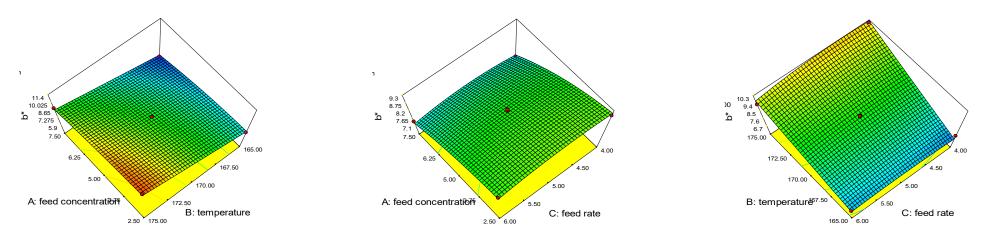


Fig. 4.15 Effect of process variables on b\* value of spray dried kalparasa powder

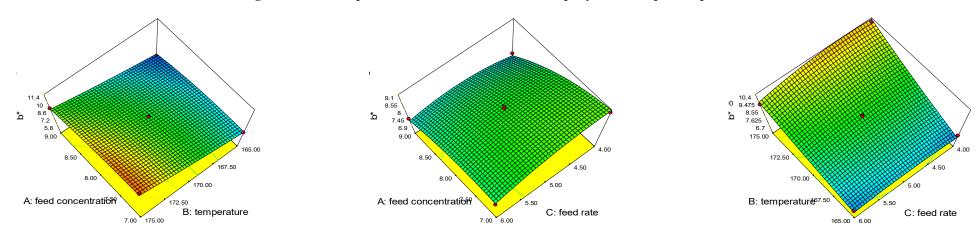


Fig. 4.16 Effect of process variables on b\* value of spray dried keramrutham powder

spray dried powders. Similar trend was reported by Quek et al., in 2007 for spray dried watermelon powder.

The equation for predicting the b\* value of both spray dried powders are given below

The p value obtained in the case of kalparasa powder was 0.0008 which means the model terms are significant and are recorded as A and B. Recorded  $R^2$  value was 0.9527. In the case of keramrutham powder here also significant model terms are A and B, obtained p and  $R^2$  values noted as 0.0011 and 0.9473 respectively.

## Hue angle

The hue angle calculated for the spray dried powders tabulated in Appendix A (Table A17) and Appendix B (Table B17). From the hue angle values it was observed that the colour of powder was near to yellow. It was significantly affected by temperature, feed flow rate and feed combination.

For both spray dried powders hue angle was greatly influenced by temperature. As the temperature increases the appearance of yellow colour in the powder also increases. For kalparasa powder the feed combination and feed rate negatively affects the hue angle. Similar effects were observed in keramrutham powder also. Fig. 4.17 and 4.18 represents the 3-D graphs of effect of process parameters on the hue angle of spray dried neera powders. Obtained results are matching with the findings of Yousefi *et al.*, 2010.

The regression models for predicting the hue angle of spray dried powders were given in equation 4.17 and 4.18.

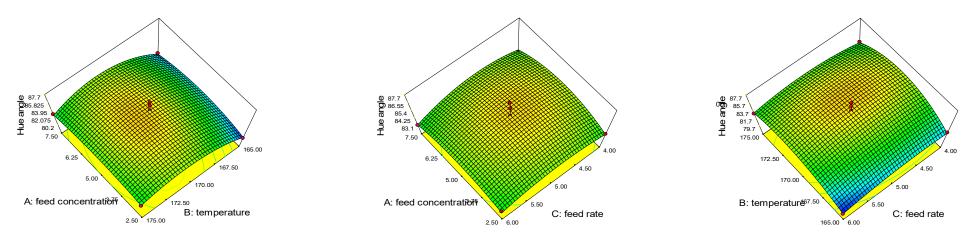


Fig. 4.17 Effect of process variables on hue angle of spray dried kalparasa powder

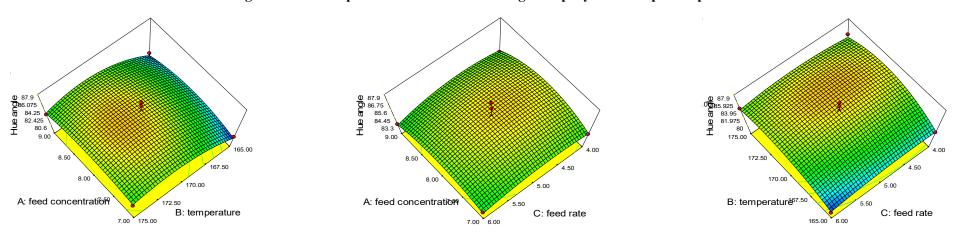


Fig. 4.18 Effect of process variables on hue angle of spray dried keramrutham powder

Hue angle =  $85.91 - 0.032 \text{ A} + 1.18 \text{ B} - 0.34 \text{ C} - 0.19 \text{ AB} - 0.070 \text{ AC} + 0.51 \text{ BC} - 1.19 \text{ A}^2 - 2.89 \text{ B}^2 - 1.08 \text{ C}^2$  ...4.17 Hue angle =  $86.01 - 0.035 \text{ A} + 1.49 \text{ B} - 0.41 \text{ C} - 0.13 \text{ AB} + 0.023 \text{ AC} + 0.40 \text{ BC} - 1.44 \text{ A}^2 - 2.32 \text{ B}^2 - 0.83 \text{ C}^2$  ...4.18

# 4.3 OPTIMIZATION OF PROCESS PARAMETERS FOR SPRAY DRIED KALPARASA AND SPRAY DRIED KERAMRUTHAM POWDERS

The effect of spray drying parameters on the quality characteristics of spray dryied kalparasa and keramrutham powder were experimented in this present study. The response surface methodology (Design Expert Software 7.7.0) was used for the optimization of three independent variables selected for spray drying process such as feed combination (2.5% MD + 0.25% GA, 5% MD + 0.5% GA, 7.5% MD + 0.75% GA for kalparasa and 7% MD + 0.7% GA, 8% MD + 0.8% GA, 9% MD+0.9% GA for keramrutham powder), spray dryer inlet temperature (165, 170 and 175°C) and feed flow rate of (4, 5, 6rpm). After completing optimization the desirability of the product also analysed with the same software. Generally desirability ranges from 0-1. When it gives the value one it should be called ideal case (Maran *et al.*, 2013). Here for the optimization process maximum and minimum ranges were chosen for the dependant variables and the independent variables kept within range. The optimization constrains for spraydrying of kalparasa and keramrutham powders respectively are given in Table 4.6 and 4.7.

For kalparasa powder the optimum condition obtained as per the Box-Behnken design is 3.53% MD + 0.353% GA as feed combination, inlet air temperature of 168°C and 4.25 rpm feed flow rate. Whereas for the keramrutham powder the obtained optimum condition is 7.58% MD + 0.758%GA feed combination, and 171.89°C inlet air temperature and 4.82 rpm feed rate. The desirability of spray dried kalparasa powder is noted as 0.719 and the recorded

desirability of spray dried keramrutham powder is 0.734. Both the desirability values are nearer to 1 so the optimized values could be considered as ideal.



Fig. 4.19 Optimally produced Kalparasa



Fig. 4. 20 Optimally produced Keramrutham



Fig. 4.21 Reconstituted optimally produced Kalparasa powder



Fig. 4. 22 Reconstituted optimally produced Keramrutham powder

Table 4.5 Response optimization constrains of spray dried kalparasa powder from the desirability analysis

Sl. No.	Response	Desirability	<b>Optimal level</b>	Lower limit	Upper limit
1	Feed combination (%)	Is in range	3.53	2.5	7.5
2	Inlet air temperature (°C)	Is in range	168.58	165	175
3	Feed flow rate (ml/min)	Is in range	4.52	4	6
4	Moisture content (%)	Minimise	3.26	2.41	3.89
5	Bulk density (g/cm <sup>3</sup> )	Maximise	0.410	0.294	0.438
6	Wettability (s)	Minimise	67.261	55	110
7	Cold water solubility (%)	Maximise	76.070	63	96
8	Water activity	Minimise	0.246	0.165	0.3
9	L*	Maximise	87.08	83.48	90.69
10	a*	Minimise	0.991	0.36	1.38
11	b*	Minimise	8.643	5.92	11.36
12	Hue value (°)	Minimise	83.84	79.8	87.67

Table 4.6 Response optimization constrains of spray dried keramrutham powder the desirability analysis

Sl. No.	Response	Desirability	<b>Optimal level</b>	Lower limit	<b>Upper Limit</b>
1	Feed combination (%)	Is in range	7.58	7	9
2	Inlet air temperature (°C)	Is in range	170.32	165	175
3	Feed flow rate (ml/min)	Is in range	4.5	4	6
4	Moisture content (%)	Minimise	3.044	2.39	3.72
5	Bulk density (g/cm <sup>3</sup> )	Maximise	0.377	0.296	0.44
6	Wettability (s)	Minimise	86.95	58	108
7	Cold water solubility (%)	Maximise	81.621	64	96

8	Water activity	Minimise	0.208	0.163	0.288
9	L*	Maximise	88.133	84.62	91.34
10	a*	Minimise	0.638	0.34	1.34
11	b*	Minimise	8.00	5.89	11.32
12	Hue value (°)	Minimise	85.521	80	87.79

# 4.4 QUALITY ANALYSIS OF OPTIMALLY PRODUCED SPRAY DRIED KALPARASA AND KERAMRUTHAM POWDERS

Table 4.7 Quality characteristics of optimally produced Spray dried powder samples

Sl. No.	Characteristics	Kalparasa powder	Keramrutham powder
1	Moisture content (%)	3.10±0.012	2.98±0.014
2	Bulk density (g/ml)	0.408±0.011	0.352±0.036
3	Wettability (s)	65±1.24	85±1.28
4	Cold water solubility (%)	74±2.41	79±1.54
5	Water activity	0.235±0.014	0.2±0.063
6	L*	85.05±0.087	85.18±0.054
7	a*	0.85±0.016	0.589±0.02
8	b*	7.01±0.035	6.3±0.04
9	Hue value (°)	83.08±1.28	85.31±1.44
10	Dispersibility (%)	95.70±1.02	98.43±1.47

11	Powder yield (%)	5.89±2.45	5.63±2.68
12	Carr's index (CI)	5.71±0.16	9.5±0.12
13	Hausner ratio (HR)	1.05±0.02	1.10±0.06
14	Titrable acidity (%)	0.215±0.043	0.219±0.036
15	рН	5.93±0.23	5.51±0.45
16	TSS (°Brix)	15.84±0.14	11.1±0.13
13	Vitamin C(mg/100g)	7.06±1.25	1.95±1.13
14	Total PolyPhenol (mg GAE)	0.033±0.004	0.028±0.012
15	Antioxidant activity (%)	4.3 ±0.03	3.8 ±0.01
16	Total sugar (%)	13±0.32	9±0.14
17	Reducing sugar (%)	0.12±0.20	0.02±0.10

As per the above tabulated values it is clear that the amount of feed combination significantly affects the quality characteristics of spray dried powders. Here the moisture content of Keramruthum powder is less than that of Kalparasa powder since it was added with more maltodextrin and gum arabic compounds compared to Kalparasa. This is because of the less TSS of Keramruthum which causes stickiness in the drying chamber during the spray drying process.

But the bulk density of Kalparasa powder showed slightly greater value than Keramruthum which indicates that the powder obtained after spray drying process was of good quality.

Coming to the wettability, there present a clear difference in the values. This is also due to the increment in the maltodextrin and gum arabic concentration in

Keramruthum powder which increases the wetting time of the powder. The powder with lesser wettability will show good quality. So Kalparasa powder will dissolve in water more easily than Keramruthum powder.

Comparing the solubility of the powder spray dried Keramruthum powder seemed to be more soluble than Kalparasa powder due to the higher concentration of carrier agents which can increase the solubility (Nadeem *et al.*,). Addition of more carrier agents can also reduce available moisture content for the growth of micro organism. So the water activity recorded for the Keramruthum powder was less than Kalparasa powder. But both powders are microbiologically safe because the water activity value in both case is below 0.6 (Kha *et al.*, 2010).

From the L\*, a\*, b\* values the powder with good colour and appearance was noted as Keramruthum powder since the white colour will catch more attention than yellow shade. This is because the colour of both raw neera (Kalparasa and Keramruthum) was entirely different. The colour of Kalparasa before processing was golden brown and for Keramruthum it was Oyster white. So the powder products also showed the same colour difference.

Coming to the flowability and cohesiveness of the powder (checked by carr's index(CI), Hausner ratio(HR)) it was observed that the obtained CI value was less than 15 for both powders which showed good flowablity and according to cohesiveness the obtained values are less than 1.2 which showed low cohesiveness (Santha Lakshmi *et al.*, 2015). The reference table was given below,

Table 4.8 Clasification of powder flowability based on carr's index (CI)

CI %	Flowability
<15	Very Good
15 – 20	Good
20 – 35	Fair

35 – 45	Bad
>45	Very Bad

Table 4.9 Classification of powder based on Hausner ratio (HR)

HR	Cohesiveness
<1.2	Low
-1-2	
1.2 – 1.4	Intermediate
>1.4	High

Here Keramruthum powder showed higher Carr's Index value indicates poor flowability where as lowest value of cohesiveness noticed for Kalparasa powder. Cohesiveness of the powder determines its consistency and flow properties. Lower the cohesiveness, better the flowability of powders (Santha Lakshmi *et al.*, 2015).

The obtained pH values and TSS values showed greater difference, this is mainly because the carbonation processe which can reduce the pH and TSS and can also increase the acidity of the product (Jori *et al.*, 2013).

The Vitamin C, anti oxidant activity and phenolic content where widely influenced and affected by inlet air temperature. When the neera processed at higher temperature (165,170, 175 °C), there observed a loss in heat sensitive compounds. Comparing the Vitamin C, antioxidant content and phenolic content of spray dried powder with raw neera (Keramruthum and Kalparasa), it is noted that the addition of carrier agents could protect the sensitive components only up to an extend and the increase in the processing temperature significantly reduces the vitamin C, anti oxidant contents and phenolic contents. After the powder analysis it is observed that the heat sensitive compounds were well preserved in Kalparasa powder where as in Keramruthum powder it was less. This might be due to the fact that the bottled

Keramruthum neera was prepared by several series of process including centrifugation, pasteurisation and carbonation etc. These treatments may cause changes in their natural properties as well as qualities. So the heat sensitive compounds such as vitamin C, anti oxidant contents and phenolic contents are lesser in spray dried Keramruthum powder. The observed result well supports the above scientific facts. Similar results were observed by Sanganamoni (2017) and Purohit (2017) on thermal treatment of tender coconut water.

The overall nature of the raw neera such as pH, TSS, colour etc..for kalparasa and keramrutham is entirely different so the powder produced from both are not meant to be comparable. The quality differences may be due to the difference in harvesting methods and processing stages. The kalparasa was un processed and keramrutham was processed and bottled. Studies are going to protect the antioxidant activity and vitamin C content, in keramrutham by KAU adopting advanced technologies.

## 4.5 SENSORY ANALYSIS OF OPTIMALLY PRODUCED SPRAY DRIED SAMPLES

The optimized treatments of neera powder were reconstituted and kept for sensory evaluation. The products were analysed by 15- semi trained sensory panelists including scientists, teachers, and pg students from both gender. Powders were reconstituted with normal water in 1:10 ratio. Fresh neera samples were kept as control. The results of sensory evaluation are shown in Fig. 4.23.

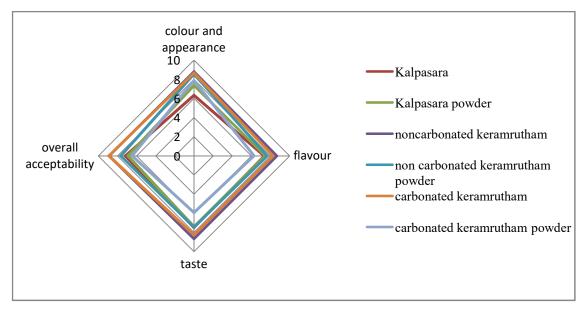


Fig. 4.23 Sensory score obtained for Spray dried powders

Based on the sensory analysis, Kalparasa powder scored an overall acceptability of 7.36, whereas, non carbonated keramrutham powder scored 7.66. Analysis showed that, both powders have an appreciable acceptability. The overall acceptance of carbonated neera powder noted as 6.46. This is may be due to the reduction in pH, TSS and Total sugar during the processing and carbonation (Jori *et al.*, 2013). Carbonation of drinks will prevent the microbial growth and enzymatic activities which can give more shelf life to the product. According to Verma (2018), carbonated products are safer than non carbonated products and the prolonged storage of these products may show the differences.

# 4.6 PACKAGING AND STORAGE STUDIES OF OPTIMALLY PRODUCED SPRAYDRIED SAMPLES

As per the approved work plan the optimally produced spray dried powder samples were packed in poly ethelene packets, laminated aluminium packets, and retort packets and stored at room temperature (25-30°C) as well as at refrigerated condition (4°C). It was observed that the powder samples packed in laminated aluminium pouches and poly ethylene pouches started caking and clogging after one

week. Since the product got decayed before completing one month it was decided to pack the samples for storage studies in retort pouches.



Fig.4. 24 Neera packed in Laminated Aluminum Pouches



Fig. 4.25 Neera packed in LDPE Pouches

The moisture content of neera powder stored in refrigerated condition and ambient condition showed a slight increase with increase in the storage period. The addition of maltodextrin and absorption of moisture from the outside environment might be the reason for moisture content variation. More variation of moisture content was observed with the packet that was stored at room temperature. So the quality of powder will be good when the product is stored at refrigerated conditions.

Table 4.10 Storage studies of optimally produced Kalparasa powder (Refrigerated sample)

SI. No	Quality characteristics	Spray dried Kalparasa powder (Refrigerated sample 4°C)					
			S	torage per	riod (mont	th)	
		Fresh	1 <sup>st</sup>	2 <sup>nd</sup>	3 <sup>rd</sup>	4 <sup>th</sup>	5 <sup>th</sup>
1.	Moisture content,% (w.b)	3.10	3.10	3.15	3.175	3.181	3.186
2.	рН	5.93	5.93	5.91	5.88	5.86	5.86
3.	TSS	15.84	15.84	15.84	15.85	15.85	15.87
4.	Antioxidant content, %	8.10	8.10	7.98	7.94	7.92	7.89
5.	Phenolic content, mg GAE	0.033	0.033	0.03	0.028	0.022	0.02
6.	Microbial load, cfu/g	1×10 <sup>3</sup>	1×10 <sup>3</sup>	1×10 <sup>3</sup>	1×10 <sup>3</sup>	1×10 <sup>3</sup>	1×10 <sup>3</sup>

Table 4.11 Storage studies of optimally produced Kalparasa powder (Roomtemperature)

SI.	Quality characteristics	Spray dried Kalparasa powder (Room temperature)					erature)
No			Storage period (month)				
		Fresh	1 <sup>st</sup>	2 <sup>nd</sup>	3 <sup>rd</sup>	4 <sup>th</sup>	5 <sup>th</sup>
1.	Moisture content,% (w.b)	3.10	3.10	3.14	3.16	3.18	3.19
2.	рН	5.93	5.93	5.89	5.87	5.874	5.851
3.	TSS	15.84	15.84	15.85	15.87	15.87	15.89
4.	Antioxidant content, %	8.1	8.1	8.0	7.89	7.82	7.76
5.	Phenolic content, mg GAE	0.033	0.030	0.03	0.029	0.026	0.024

6.	Microbial load, cfu/g	1×10 <sup>3</sup>						
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Table 4.12 Storage studies of optimally produced Keramrutham powder (Refrigerated sample)

SI. No	Quality characteristics	Spray dried Keramrutham powder (Refrigerated sample 4°C)					
			Storage period (month)				
		Fresh	1 <sup>st</sup>	2 <sup>nd</sup>	3 <sup>rd</sup>	4 <sup>th</sup>	5 <sup>th</sup>
1.	Moisture content,% (w.b)	2.98	2.98	3.14	3.18	3.21	3.26
2.	рН	5.51	5.51	5.482	5.49	5.496	5.48
3.	TSS	11.1	11.1	11.13	11.15	11.15	11.16
4.	Antioxidant content, %	6.3	6.3	5.8	5.8	5.73	5.69
5.	Phenolic content, mg GAE	0.002	0.002	0.002	0.001	0.001	0.001
6.	Microbial load, cfu/g	1×10 <sup>3</sup>	1×10 <sup>3</sup>	1×10 <sup>3</sup>	1×10 <sup>3</sup>	1×10 <sup>3</sup>	1×10 <sup>3</sup>

Table 4.13 Storage studies of optimally produced Keramrutham powder (room temperature)

SI.	Quality characteristics	Spray	dried	Keramrı	ıtham	powder	(Room
No		tempera	ature)				
			St	orage per	riod (mor	nth)	
		Fresh	1 <sup>st</sup>	2 <sup>nd</sup>	3 <sup>rd</sup>	4 <sup>th</sup>	5 <sup>th</sup>
1.	Moisture content,% (w.b)	2.98	2.98	3.16	3.19	3.26	3.28
2.	рН	5.51	5.51	5.49	5.48	5.46	5.46
3.	TSS	11.1	11.1	11.13	11.16	11.16	11.17
4.	Antioxidant content, %	6.1	6.1	5.6	5.54	5.41	5.38
5.	Phenolic content, mg GAE	0.002	0.001	0.001	-	-	-
6.	Microbial load, cfu/g	1×10 <sup>3</sup>					

There will not be significant difference in the pH of samples stored at both conditions during the storage. pH is slightly changes from the fourth month of storage and similar trend was observed in TSS also. But the anti oxidant activity and phenolic content is gradually decreases with the storage period. It may be noted that the microbial load in cfu/g of the powder remained as safe during the entire storage periods indicating the microbial stability of the product. There is no growth obtained in 10<sup>-3</sup> dilution. Even though the powder is microbiologically safe during the entire storage period it doesn't mean that the powder is keeping its quality. The quality characteristics such as pH, TSS, vitamin C, antioxidant activity were reduced during the storage period this may reflect in the sensory evaluation. The present work did not carried out the sensory evaluation in all the five months of storage so this can be taken as a suggestion for future work.

#### 4.7 COST ANALYSIS

The computation of cost of production of one kilogram of spray dried neera powder shown in Appendix D. The computed cost of one kilogram of spray dried neera powder using twin fluid nozzle was found to be Rs. 2168.6/kg

#### **CHAPTER V**

#### **SUMMARY AND CONCLUSION**

Neera is one of the natural, non alcoholic healthy drinks collected from the unopened inflorescence of coconut palm (*Cocus nusifera*). It is a unique source of iron, phosphorous, ascorbic acid and amino acids. The studies indicate that the sap is having a lower glycemic index value so that the absorption of sugar content to the blood will be very less. So the consumption of neera and neera based products are healthy and diabetic friendly. The problem associated with the neera production is its natural fermentation. The freshly collected sap starts fermenting within 2-3 hr under ambient temperature. The present study tried to find a technique for increasing the shelf life of neera by spray drying process and to make possible for the longer transportation of neera which leads to the wide range availability of neera at local markets as an alternative to soft drinks.

Spray drying is a technique that produces dry powder from a liquid or slurry by rapidly drying with a hot gas (air). The process leads to increase the shelf life by removing the water content without significant alterations in the quality attributes. As a result of removal of water content, the microbial activity and enzymatic activity will arrest, so that the fermentation will get reduced and product attains maximum shelf life. Preperation of liquid feed for the spray drying process is mainly by the addition of carrier materials along with the core material. The wall materials used in this study were maltodextrin and gum Arabic. The intention of the study was to optimize the process parameters for the spray drying of neera (kalparasa and keramrutham).

Neera collected from CPCRI (Kalparasa) and KAU (Keramrutham) was stored at refrigerated condition and the spray drying was done by preparing feed material. Kalparasa was directly harvested and stored in chiller boxes where as keramrutham was processed, carbonated and bottled neera. The physicochemical characteristics of both neera was analysed before spray drying process and the difference in quality parameters such as TSS, pH, vitamin C and antioxidant content

in keramrutham might be due to the processing methods done before the carbonation. Spray drying was done by adding carrier materials along with the core material inorder to increase the total soluble solids and to reduce the stickiness of powder at drying chamber. The feed combination selected for kalparasa was 2.5% MD + 0.25% GA, 5% MD + 0.5% GA and 7.5% MD + 0.75% GA. whereas for keramrutham powder the selected combination was 7% MD + 0.7% GA, 8% MD + 0.8% GA and 9% MD + 0.9% GA. The feed combination is different in both cases because the TSS of both neera is different. The drying temperature and feed flow rate was taken as same for both neera, that is 165, 170, and 175°C and 4, 5, 6 rpm respectively. The powderd particles after processing were collected and stored in retort pouches. The optimization was done with Box-behnken design in response surface methodology.

The moisture content of powder produced from Kalparasa showed a range of 2.41 -3.89 % wb, whereas for the keramrutham moisture content was ranging from 2.32 -3.78% wb. In both cases the moisture content was higher for the combination of 2.5% MD + 0.25% GA. The treated temperature for this combination was 165°C. The minimum value for the moisture content was observed for the combination 7.5% MD + .75% GA. And the temperature for the treatment was 175 °C. The moisture content of the powders showed a decreasing tendency when the inlet air temperature was increased from 165-175°C. The increase in maltodextrin and gum Arabic ratio negatively affects the moisture content. When the carrier concentration increases from (2.5% MD + 0.25%GA) to (7.5% MD + .75% GA) the moisture content reduced from 3.78 to 2.32. The increase in the feed flow rate also increases the moisture content of the powder samples.

The water activity range obtained for kalparasa powder was 0.165 to 0.351 where as for keramrutham powder it was recorded as 0.163 to 0.321. The feed combination 5% MD+ 0.5%GA, processed at 165°C, with 6 rpm feed rate (Kalparasa) and 7%MD+0.7% GA processed at 165°C, with 5 rpm feed rate (Keramrutham) have highest water activity, while lowest value was obtained for

those produced with 5% MD + 0.5% GA, processed at 175°C, with 6 rpm feed rate (kalparasa)and 9% MD+0.9%GA processed at 175°C, with 5 rpm feed rate (Keramrutham). The increasing maltodextrin, gum Arabic combination, inlet air temperature, and decreasing feed flow rate led to a decreased water activity condition of spray dried powders.

The bulk density of spray dried kalparasa powder was varied between 0.294-0.438 gm/cm<sup>3</sup>. The highest bulk density value obtained with 2.5%MD+0.25%GA feed combination, processed at 165°C for 5 rpm feed rate. Whereas the lowest value obtained for the combination 7.5%MD+0.75%GA spray dried at 175°C, for 5 rpm feed rate. For spray dried keramrutham powder the range obtained for bulk density was

0.296-0.44gm/cm<sup>3</sup>. Feed mixed with 7%MD+0.7%GA produced higher bulk density powder processed at 165°C for 5 rpm feed rate. Whereas lowest bulk density was noted for the combination prepared by mixing 9%MD+0.9%GA, was dried at 175°C for 5 rpm feed rate. Here in this study the temperature negatively affects the bulk density of both spray dried powders and the powder showed higher bulk density was of higher moisture content. The feed combination and feed rate does not show a wide range difference in the bulk density value.

For spray dried kalparasa powder the wettability values varied from 55 seconds to 110 seconds. Maximum value was noted with the feed combination ratio of 7.5% MD+0.75% GA, 175°C inlet air temperature and for 5 rpm feed rate. The minimum value obtained for the feed combination 2.5%MD+ 0.25% GA, spray dried at 165°C, for 5 rpm feed rate. The wettabilty of spray dried keramrutham powder, it was found that the maximum and the minimum values ranges from 63-108 seconds. Here the minimum value was recorded for the combination 8%MD+ 0.8% GA processed at 165°C and for 4rpm feed rate. 9%MD+ 0.9% GA feed combination produced the highest wettability powder which required more time to wet and was processed at 175°C for 5 rpm feed rate. The wettability showed a positive relation to

the increase in temperature, might be because increasing the temperature causes structural damages and more dried powder. Increasing the maltodextrin, gum Arabic combination along with the core material and feed rate also increases the wetting time, so that the wetting time required was observed as less.

The cold water solubility of spray dried Kalparasa powder varied from 64 to 96%. Maximum solubility was recorded for 165°C, 5% MD + 0.5% GA and 6 rpm feed rate, minimum solubility found at inlet air temperature 175°C, 5% MD + 0.5%GA and 4 rpm feed rate. In the case of spray dried Keramruthum powder solubility varied between 64 to 96%. Minimum value recorded for 7% MD+0.7% GA, processed at 175°C for 5rpm feed rate where as maximum value noted for 9% MD + 0.9% GA dried at 165°C for 5 rpm feed rate. In both spraydrying process the incressed concentration of Maltodextrin, ncreases the cold water solubility of spray dried particles whereas the effect of inlet air temperature on the cold water solubility showed a decreasing pattern. The effect of feed flow rate on the cold water solubility of Kalparasa powder was negligible where as for the keramrutham powder the cold water solubility showed a negative effect on increasing the feed rate. This might be due to the formation of large sized particle at higher feed rate.

The acceptability of powder particles mainly depends on the colour of the powder material. The L\* value of spray dried Kalparasa powder varied from 83.48 to 90.69. The highest value of L\* value observed for the feed combination 7.5% MD+0.75% GA processed at 170°C for 6 rpm feed flow rate, where as lower value noted at 175°C for the feed combination 5%MD+ 0.5% GA for the feed flow rate 4 rpm. For keramrutham powder the lowest value (84.62) of L\* was recorded for the combination 8% MD + 0.8% GA dried at 175°C for 4 rpm feed rate, whereas highest value (91.34) was observed for the feed combination 9% MD + 0.9% GA, processed at 170°C for the feed flow rate 6 rpm. When the temperature increases from 165 to 175°C the colour (lightness, L\* value) decreases gradually. This effect might be due

to the action of higher temperature on the maltodextrim compounds resulting in to the formation of non enzymatic browning

For kalparasa powder the highest a\* value (1.38) found for the feed combination 2.5% MD+ 0.25% GA processed at 175°C for 5 rpm feed rate and lower value (0.36) observed for the feed combination 5% MD + 0.5% GA, dried at 170°C, for a feed flow rate 5rpm. In the case of keramrutham powder the highest value (1.34) showed by 7% MD+ 0.7% GA at 175°C, for 5 rpm feed flow rate, where as lower value (0.34) showed by 8% MD+ 0.8% GA feed combination, 170°C, and 5 rpm feed rate. In both cases temperature showed a positive effect on a\* value whereas the feed combination and feed rate showed negative effect on the redness value. The increase in redness value at higher temperature might be due to the non enzymatic browning of sugar or starchy materials.

It was noted that in the case of kalparasa powder the highest b\* value (11.36) observed for the feed combination 2.5% MD+ 0.25%GA processed at 175°C for 5 rpm feed flow rate and lowest value (5.92) recorded for the feed combination 7.5% MD+ 0.75%GA dried at 165°C at 5 rpm feed flow rate. For keramrutham powder the b\* value ranges from 80 (8% MD+ 0.8% GA, 165°C and 4 rpm feed rate) to 87.79 (8% MD+ 0.8% GA, 170°C and 5 rpm feed rate). The yellowness mainly influenced by inler air temperature. As the temperature increases from 165°C to 175°C, the yellowness also increases. But in both case the fed combination and feed flow rate showed a negative effect on the yellowness of spray dried powders.

For both spray dried powders hue angle was greatly influenced by temperature. As the temperature increases the appearance of yellow colour in the powder also increases. For kalparasa powder the feed combination and feed rate negatively affects the hue angle. For kalparasa powder the optimum condition obtained as per the Box-Behnken design is 3.53% MD + 0.353% GA as feed combination, inlet air temperature of 168°C and 4.25 rpm feed flow rate. Whereas for the keramrutham powder the obtained optimum condition is 7.58% MD + 0.758%

GA feed combination, and 171.89°C inlet air temperature and 4.82 rpm feed rate. The quality characteristics of optimized powders were analysed. The moisture content of spray dried keramrutham powder was lesser than kalparasa powder, but both powders were safe because the obtained moisture content value was less than 0.6%. Similarly slight difference in the bulk density values was also observed. It was more for kalparasa powder it showed that the quality of the powder was a little bit more for kalparasa powder than keramrutham powder. The wetting time taken by the keramrutham powder was higher than that of kalparasa powder but keramrutham powder showed good solubility than kalparasa powder. Both powders showed very less water activity value, which indicates that, their storage will be microbiologically safe. According to the colour characteristics, the colour of the keramrutham powder was lighter than the kalparasa powder (yellow shade) since the raw sample (kalparasa) itself has a golden honey shade. After analyzing the flowability and cohesiveness of the powder it was observed that the obtained Carr's Index value was less than 15 for both powders which showed good flowablity. According to cohesive nature, the obtained values were less than 1.2 which showed that the powder samples were less cohesive in nature.

Eventhough the antioxidant activity and vitamin C is lesser in Keramrutham due to processing the sensory evaluation showed good acceptability. This may be due to the colour of the reconstituted neera powder of keramrutham. Further studies are going to protect antioxidant activity and vitamine C in keramrutham by adopting advanced technologies. Even though there is no microbial attack till the fifth month of storage, it is be better to do sensory analysis on each month to check the quality characteristics of neera powder during the storage period. Because there present slight changes in the pH, TSS, antioxidant activity and moisture content which may affect the sensory characteristics and quality of the product. The present study didn't conducted sensory evaluation in each month so this can be taken as a suggestion for future work.

#### **CHAPTER VI**

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### APPENDIX A

Table A1 Effect of process parameters on moisture content of Spray dried kalparasa powder

Run	Feed concentration	Inlet air temperature	Feed rate	Moisture content % wb
	(%)	(°C)	(rpm)	
1	5.00	170.00	5.00	3.33
2	2.50	165.00	5.00	3.89
3	5.00	170.00	5.00	3.28
4	5.00	175.00	6.00	2.68
5	5.00	170.00	5.00	3.31
6	5.00	170.00	5.00	3.24
7	7.50	170.00	6.00	3.53
8	7.50	170.00	4.00	2.81
9	7.50	175.00	5.00	2.41
10	2.50	170.00	6.00	3.48
11	5.00	165.00	4.00	3.61
12	5.00	170.00	5.00	3.13
13	7.50	165.00	5.00	3.7
14	2.50	175.00	5.00	2.72
15	2.50	170.00	4.00	2.93
16	5.00	165.00	6.00	3.79
17	5.00	175.00	4.00	2.49

Table A2 Analysis of variance (ANOVA) for moisture content of spray dried neera(Kalparasa) powder

			Mean	<del></del>	p-value	
Source	Sum of Squares	df	Square	F Value	Prob > F	7
Model	4.76	9	0.53	15.28	0.0008	significant
A-concentration	0.15	1	0.15	4.21	0.0792	
B-temperature	3.45	1	3.45	99.55	< 0.0001	
C-feed rate	6.613E-003	1	6.613E-003	0.19	0.6752	
AB	0.038	1	0.038	1.10	0.3294	
AC	0.44	1	0.44	12.78	0.0090	
BC	0.020	1	0.020	0.57	0.4763	
A^2	0.093	1	0.093	2.70	0.1443	
B^2	0.54	1	0.54	15.46	0.0057	
C^2	5.568E-004	1	5.568E-004	0.016	0.9026	
Residual	0.24	7	0.035			
Lack of Fit	0.14	3	0.048	1.96	0.2621	not significant
Pure Error	0.098	4	0.025			
Cor Total	5.00	16				
Std. Dev	0.19		R-S	Squared	0.9516	
Mean	3.53		Adj	R-Squared	0.8893	
C.V. %	5.26		Pred	l R-Squared	0.5080	
PRESS	2.46		Ade	q Precision	12.484	

Table A3 Effect of process parameters on water activity of Spray dried kalparasa powder

Run	Feed	Inlet air	Feed	Water activity
	concentration	temperature	rate	$(\mathbf{a}_{\mathbf{w}})$
1	(%) 5.00	(°C) 170.00	( <b>rpm</b> ) 5.00	0.248
1	3.00	170.00	3.00	0.240
2	2.50	165.00	5.00	0.282
3	5.00	170.00	5.00	0.251
4	5.00	175.00	6.00	0.165
5	5.00	170.00	5.00	0.259
6	5.00	170.00	5.00	0.264
7	7.50	170.00	6.00	0.282
8	7.50	170.00	4.00	0.235
9	7.50	175.00	5.00	0.182
10	2.50	170.00	6.00	0.286
11	5.00	165.00	4.00	0.276
12	5.00	170.00	5.00	0.271
13	7.50	165.00	5.00	0.289
14	2.50	175.00	5.00	0.174
15	2.50	170.00	4.00	0.213
16	5.00	165.00	6.00	0.3
17	5.00	175.00	4.00	0.168

Table A4 Analysis of variance (ANOVA) for water activity of spray dried neera (Kalparasa) powder

Source	Sum of Squares	df	Square	F Value	p-value Prob > F	
Model	0.026	9	2.848E-003	115.26	< 0.0001	significant
A-concentration	1.513E-005	1	1.513E-005	0.61	0.4596	
B-temperature	0.016	1	0.016	655.68	< 0.0001	
C-feed rate	4.278E-003	1	4.278E-003	173.15	< 0.0001	
AB	2.250E-006	1	2.250E-006	0.091	0.7716	
AC	4.000E-006	1	4.000E-006	0.16	0.6994	
BC	9.303E-004	1	9.303E-004	37.65	0.0005	
$A^2$	1.541E-004	1	1.541E-004	6.24	0.0411	
$\mathbf{B}^2$	3.866E-003	1	3.866E-003	156.46	< 0.0001	
$\mathbb{C}^2$	2.738E-005	1	2.738E-005	1.11	0.3275	
Residual	1.729E-004	7	2.471E-005			
Lack of Fit	1.397E-004	3	4.658E-005	5.61	0.0645	not significant
Pure Error	3.320E-005	4	8.300E-006			
Cor Total	0.026	16				
Std. Dev.	4.971E-003		R-Squared		0.9933	
Mean	0.26	I	Adj R-Squared		0.9847	
C.V. %	1.93	P	red R-Squared		0.9113	
PRESS	2.288E-003	A	Adeq Precision		35.740	

Table A5 Effect of process parameters on wettability of Spray dried kalparasa powder

Run	Feed concentration (%)	Inlet air temperature (°C)	Feed rate (rpm)	Wettability (s)
1	5.00	170.00	5.00	78
2	2.50	165.00	5.00	55
3	5.00	170.00	5.00	80
4	5.00	175.00	6.00	106
5	5.00	170.00	5.00	84
6	5.00	170.00	5.00	86
7	7.50	170.00	6.00	92
8	7.50	170.00	4.00	93
9	7.50	175.00	5.00	110
10	2.50	170.00	6.00	71
11	5.00	165.00	4.00	65
12	5.00	170.00	5.00	88
13	7.50	165.00	5.00	74
14	2.50	175.00	5.00	99
15	2.50	170.00	4.00	73
16	5.00	165.00	6.00	68
17	5.00	175.00	4.00	105

Table A6 Analysis of variance (ANOVA) for wettability of spray dried neera (Kalparasa) powder

Table Ao Analysis of variance (ANOVA) for wellability of spray dried neera (Kaiparasa) powder					<u> </u>	
			Mean		p-value	
Source	Sum of Squares	df	Square	F Value	Prob > F	
Model	3634.83	9	403.87	24.15	0.0002	significant
A-concentration	480.50	1	480.50	28.74	0.0011	
B-temperature	3081.12	1	3081.12	184.26	< 0.0001	
C-feed rate	0.13	1	0.13	7.475E-003	0.9335	
AB	6.25	1	6.25	0.37	0.5603	
AC	0.25	1	0.25	0.015	0.9061	
BC	0.000	1	0.000	0.000	1.0000	
$A^2$	3.04	1	3.04	0.18	0.6825	
B <sup>2</sup>	64.04	1	64.04	3.83	0.0912	
$C^2$	1.52	1	1.52	0.091	0.7721	
Residual	117.05	7	16.72			
Lack of Fit	48.25	3	16.08	0.94	0.5020	not significant
Pure Error	68.80	4	17.20			
Cor Total	3751.88	16				
Std. Dev.	3.6	50		R-Squared	0.9767	
Mean	83.9	14		Adj R-Squared	0.9468	
C.V. %	4.2	28		Pred R-Squared	0.8829	
PRESS	455.5	0		Adeq Precision	20.754	

Table A7 Effect of process parameters on bulk density of Spray dried kalparasa powder

Run	Feed	Inlet air	Feed	Bulk density
	concentration (%)	temperature (°C)	rate (rpm)	(g/ml)
1	5.00	170.00	5.00	0.416
2	2.50	165.00	5.00	0.438
3	5.00	170.00	5.00	0.401
4	5.00	175.00	6.00	0.312
5	5.00	170.00	5.00	0.398
6	5.00	170.00	5.00	0.385
7	7.50	170.00	6.00	0.362
8	7.50	170.00	4.00	0.353
9	7.50	175.00	5.00	0.294
10	2.50	170.00	6.00	0.352
11	5.00	165.00	4.00	0.417
12	5.00	170.00	5.00	0.376
13	7.50	165.00	5.00	0.413
14	2.50	175.00	5.00	0.3
15	2.50	170.00	4.00	0.392
16	5.00	165.00	6.00	0.401
17	5.00	175.00	4.00	0.296

Table A8 Analysis of variance (ANOVA) for bulk density of spray dried neera (Kalparasa) powder

Source	Sum of Squares	df	Square	F Value	p-value Prob > F	
Model	0.021	9	2.318E-003	3.35	0.0626	not significant
A-concentration	4.351E-004	1	4.351E-004	0.63	0.4539	
B-temperature	0.015	1	0.015	21.12	0.0025	
C-feed rate	1.201E-004	1	1.201E-004	0.17	0.6895	
AB	1.323E-004	1	1.323E-004	0.19	0.6752	
AC	1.210E-004	1	1.210E-004	0.17	0.6884	
BC	2.025E-005	1	2.025E-005	0.029	0.8690	
$A^2$	3.878E-003	1	3.878E-003	5.60	0.0498	
$B^2$	2.123E-004	1	2.123E-004	0.31	0.5970	
$C^2$	9.285E-004	1	9.285E-004	1.34	0.2848	
Residual	4.845E-003	7	6.921E-004			
Lack of Fit	3.686E-003	3	1.229E-003	4.24	0.0983	not significant
Pure Error	1.159E-003	4	2.897E-004			
Cor Total	0.026	16				
Std. Dev.	0.026			R-Squared	0.8115	
Mean	0.37			Adj R-Squared	0.5692	
C.V. %	7.12			Pred R-Squared	-1.3648	
PRESS	0.061			Adeq Precision	5.167	

Table A9 Effect of process parameters on cold water solubility of Spray dried kalparasa powder

Run	Feed concentration (%)	Inlet air temperature (°C)	Feed rate (rpm)	Cold water solubility (%)
1	5.00	170.00	5.00	74.3
2	2.50	165.00	5.00	82
3	5.00	170.00	5.00	75.1
4	5.00	175.00	6.00	67
5	5.00	170.00	5.00	76.5
6	5.00	170.00	5.00	76
7	7.50	170.00	6.00	78
8	7.50	170.00	4.00	80
9	7.50	175.00	5.00	70
10	2.50	170.00	6.00	69.12
11	5.00	165.00	4.00	84
12	5.00	170.00	5.00	70.9
13	7.50	165.00	5.00	96
14	2.50	175.00	5.00	60
15	2.50	170.00	4.00	70.34
16	5.00	165.00	6.00	88
17	5.00	175.00	4.00	69

Table A10 Analysis of variance (ANOVA) for cold water solubility of spray dried neera (Kalparasa) powder

Source	Sum of Squares	df	Square	F Value	p-value Prob > F	
Model	1404.74	9	156.08	25.89	0.0001	significant
A-concentration	0.50	1	0.50	0.083	0.7817	
B-temperature	800.00	1	800.00	132.70	< 0.0001	
C-feed rate	60.50	1	60.50	10.04	0.0158	
AB	16.00	1	16.00	2.65	0.1473	
AC	289.00	1	289.00	47.94	0.0002	
BC	25.00	1	25.00	4.15	0.0811	
$A^2$	74.27	1	74.27	12.32	0.0099	
$\mathbf{B}^2$	74.27	1	74.27	12.32	0.0099	
$\mathbb{C}^2$	43.12	1	43.12	7.15	0.0318	
Residual	42.20	7	6.03			
Lack of Fit	33.00	3	11.00	4.78	0.0823	not significant
Pure Error	9.20	4	2.30			
Cor Total	1446.94	16				
Std. Dev.	2.46			R-Squared	0.9708	
Mean	79.94			Adj R-Squared	0.9333	
C.V. %	3.07			Pred R-Squared	0.6252	
PRESS	542.38			Adeq Precision	14.869	

Table A11 Effect of process parameters on colour  $(L^*)$  of Spray dried kalparasa powder

Colour (L*)
85.84
84.88
87.03
85.87
89.49
86.05
87.22
90.69
88.91
87.14
85.83
86.25
85.86
86.36
83.48
87.2
86.69

Table A12 Analysis of variance (ANOVA) for colour (L\*) of spray dried neera (Kalparasa) powder

Source	Sum of Squares	df	Square	F Value	p-value Prob > F	
Model	43.64	9	4.85	12.49	0.0015	significant
A-concentration	10.86	1	10.86	27.98	0.0011	C
B-temperature	1.57	1	1.57	4.04	0.0845	
C-feed rate	6.06	1	6.06	15.60	0.0055	
AB	0.37	1	0.37	0.96	0.3601	
AC	6.81	1	6.81	17.55	0.0041	
BC	3.72	1	3.72	9.60	0.0174	
$\mathbf{A}^2$	12.85	1	12.85	33.11	0.0007	
$\mathbf{B}^2$	0.61	1	0.61	1.58	0.2486	
$\mathbb{C}^2$	0.18	1	0.18	0.46	0.5173	
Residual	2.72	7	0.39			
Lack of Fit	1.96	3	0.65	3.47	0.1302	not significant
Pure Error	0.75	4	0.19			
Cor Total	46.35	16				
Std. Dev.	0.62			R-Squared	0.9414	
Mean	86.76		Adj	R-Squared	0.8660	
C.V. %	0.72		Pred	R-Squared	0.2971	
PRESS	32.58		Ade	q Precision	14.614	

Table A13 Effect of process parameters on colour (a\*) of Spray dried kalparasa powder

Run	Feed	Inlet air	Feed	Colour (a*)
	concentration	temperature	rate	
	(%)	(°C)	(rpm)	
1	5.00	170.00	5.00	0.85
2	2.50	165.00	5.00	0.71
3	5.00	170.00	5.00	1.29
4	5.00	175.00	6.00	0.65
5	5.00	170.00	5.00	1.11
6	5.00	170.00	5.00	1.38
7	7.50	170.00	6.00	1.25
8	7.50	170.00	4.00	0.82
9	7.50	175.00	5.00	0.94
10	2.50	170.00	6.00	0.98
11	5.00	165.00	4.00	0.48
12	5.00	170.00	5.00	0.94
13	7.50	165.00	5.00	0.36
14	2.50	175.00	5.00	0.86
15	2.50	170.00	4.00	1.29
16	5.00	165.00	6.00	1.02
17	5.00	175.00	4.00	1.23

Table A14 Analysis of variance (ANOVA) for colour (a\*) of spray dried neera (Kalparasa) powder

Source	Sum of Squares	df	Square	F Value	p-value Prob > F	
Model	1.20	9	0.13	5.52	0.0174	significant
A-feed concentration	n 0.080	1	0.080	3.31	0.1115	C
B-temperature	0.038	1	0.038	1.57	0.2510	
C-feed rate	0.019	1	0.019	0.79	0.4043	
AB	1.000E-004	1	1.000E-004	4.141E-003	0.9505	
AC	0.000	1	0.000	0.000	1.0000	
BC	0.013	1	0.013	0.55	0.4833	
$A^2$	0.062	1	0.062	2.56	0.1534	
$B^2$	0.79	1	0.79	32.81	0.0007	
$C^2$	0.12	1	0.12	4.97	0.0611	
Residual	0.17	7	0.024			
Lack of Fit	0.020	3	6.808E-003	0.18	0.9026	not significant
Pure Error	0.15	4	0.037			-
Cor Total	1.37	16				
Std. Dev.		0.16		R-Squared	0.876	 64
Mean		0.95		Adj R-Square		
C.V. %		16.35		Pred R-Squar		
PRESS		0.56		Adeq Precision		

Table A15 Effect of process parameters on colour (b\*) of Spray dried kalparasa powder

Run	Feed concentration (%)	Inlet air temperature (°C)	Feed rate (rpm)	Colour (b*)
1	5.00	170.00	5.00	7.98
2	2.50	165.00	5.00	8.72
3	5.00	170.00	5.00	7.17
4	5.00	175.00	6.00	8.79
5	5.00	170.00	5.00	9.09
6	5.00	170.00	5.00	11.36
7	7.50	170.00	6.00	9.33
8	7.50	170.00	4.00	7.42
9	7.50	175.00	5.00	5.92
10	2.50	170.00	6.00	8.96
11	5.00	165.00	4.00	8.82
12	5.00	170.00	5.00	8.96
13	7.50	165.00	5.00	8.88
14	2.50	175.00	5.00	7.16
15	2.50	170.00	4.00	10.07
16	5.00	165.00	6.00	7.08
17	5.00	175.00	4.00	7.18

Table A16 Analysis of variance (ANOVA) for colour (b\*) of spray dried neera (Kalparasa) powder

Source	Sum of Squares	df	Square	F Value	p-value Prob > F	
Model	26.44	9	2.94	15.67	0.0008	significant
A-feed concentra	ation 5.90	1	5.90	31.46	0.0008	C
B-temperature	19.53	1	19.53	104.17	< 0.0001	
C-feed rate	0.041	1	0.041	0.22	0.6558	
AB	0.26	1	0.26	1.36	0.2817	
AC	0.017	1	0.017	0.090	0.7727	
BC	0.11	1	0.11	0.56	0.4774	
$A^2$	0.301	0.30	1.62	0.2431		
$B^2$	1.441E-003	1	1.441E-003	7.686E-003	0.9326	
$C^2$	0.25	1	0.25	1.34	0.2855	
Residual	1.31	7	0.19			
Lack of Fit	0.76	3	0.25	1.82	0.2830	not significant
Pure Error	0.55	4	0.14			
Cor Total	27.75	16				
Std. Dev.		0.43		R-Squared	0.9527	
Mean		8.41		Adj R-Squai		19
C.V. %		5.15		Pred R-Squa		17
PRESS		12.99		Adeq Precis		

Table A17 Effect of process parameters on hue angle (°) of Spray dried kalparasa powder

Run	Feed	Inlet air	Feed	Hue angle (°)
	concentration	temperature	rate	
	(%)	(°C)	(rpm)	
1	5.00	170.00	5.00	83.91
2	2.50	165.00	5.00	85.34
3	5.00	170.00	5.00	81.8
4	5.00	175.00	6.00	85.77
5	5.00	170.00	5.00	83.03
6	5.00	170.00	5.00	83.07
7	7.50	170.00	6.00	83.12
8	7.50	170.00	4.00	83.15
9	7.50	175.00	5.00	80.97
10	2.50	170.00	6.00	84.01
11	5.00	165.00	4.00	86.88
12	5.00	170.00	5.00	83.75
13	7.50	165.00	5.00	87.67
14	2.50	175.00	5.00	83.69
15	2.50	170.00	4.00	83.07
16	5.00	165.00	6.00	79.8
17	5.00	175.00	4.00	80.27

Table A18 Analysis of variance (ANOVA) for hue angle (°) of spray dried neera (Kalparasa) powder

Source	Sum of Squares	df	Squa	re F Value	p-value	
					Prob>	f
Model	63.28	9	7.03	5.550.0171		significant
A-feed concentra	ation8.450E-003	1	8.450E-003	6.669E-003	0.9372	_
B-temperature	11.16	1	11.16	8.81	0.0209	
C-feed rate	0.95	1	0.95	0.75	0.4163	
AB	0.14	1	0.14	0.11	0.7520	
AC	0.020	1	0.020	0.015	0.9045	
BC	1.05	1	1.05	0.83	0.3928	
$A^2$	5.95	1	5.95	4.69	0.0670	
$B^2$	35.18	1	35.18	27.77	0.0012	
$C^2$	4.87	1	4.87	3.85	0.0907	
Residual	8.87	7	1.27			
Lack of Fit	0.49	3	0.16	0.0770.9689	1	not significant
Pure Error	8.38	4	2.10			
Cor Total	72.15	16				
Std. Dev.	1.13	R-Squ	ared 0	.8771		
Mean	83.49	Adj R	-Squared 0	.7190		
C.V. %	1.35	Pred R	R-Squared 0	.7106		
PRESS	20.88	Adeq	Precision 6	.955		

## 

Run	Feed	Inlet air	Feed rate	Moisture content
	concentration	temperature	(rpm)	(% wb)
1	(%) 8.00	(°C) 175.00	4.00	2.47
2	8.00	170.00	5.00	3.35
3	8.00	165.00	4.00	3.59
4	7.00	175.00	5.00	2.7
5	9.00	170.00	6.00	3.51
6	8.00	170.00	5.00	3.29
7	8.00	170.00	5.00	3.3
8	7.00	165.00	5.00	3.72
9	8.00	170.00	5.00	3.22
10	8.00	165.00	6.00	3.53
11	7.00	170.00	4.00	2.91
12	7.00	170.00	6.00	3.44
13	8.00	175.00	6.00	2.64
14	9.00	165.00	5.00	3.51
15	9.00	170.00	4.00	2.79
16	9.00	175.00	5.00	2.39
17	8.00	170.00	5.00	3.08

Table B2 Effect of process parameters on moisture content of spray dried neera (Keramrutham) powder

Source	Sum of Squares	df	Square	e F Value	p-value Prob > F	
Model	2.55	9	0.28	8.41	0.0052	significant
A-feed concentrat	ion 0.041	1	0.041	1.20	0.3088	
B-temperature	2.15	1	2.15	63.84	< 0.0001	
C-feed rate	0.23	1	0.23	6.86	0.0345	
AB	2.500E-003	1	2.500E-003	0.074	0.7933	
AC	9.025E-003	1	9.025E-003	0.27	0.6209	
BC	0.013	1	0.013	0.39	0.5510	
A^2	4.178E-003	1	4.178E-003	0.12	0.7352	
B^2	0.078	1	0.078	2.33	0.1710	
C^2	0.012	1	0.012	0.36	0.5653	
Residual	0.24	7	0.034			
Lack of Fit	0.19	3	0.064	5.84	0.0606	not significant
Pure Error	0.044	4	0.011			_
Cor Total	2.79	16				
Std. Dev.		0.18		R-Squared	0.9153	
Mean		3.14		Adj R-Squared	0.8064	
C.V. %		5.84		Pred R-Squared	- 0.1278	
PRESS		3.14		Adeq Precision	9.780	

Table B3 Effect of process parameters on water activity of Spray dried keramrutham powder

Feed concentration	Inlet air temperature	Feed rate (rpm)	Water activity (aw)
8.00	175.00	4.00	0.211
8.00	170.00	5.00	0.249
8.00	165.00	4.00	0.252
7.00	175.00	5.00	0.262
9.00	170.00	6.00	0.201
8.00	170.00	5.00	0.245
8.00	170.00	5.00	0.239
7.00	165.00	5.00	0.288
8.00	170.00	5.00	0.232
8.00	165.00	6.00	0.258
7.00	170.00	4.00	0.272
7.00	170.00	6.00	0.281
8.00	175.00	6.00	0.221
9.00	165.00	5.00	0.21
9.00	170.00	4.00	0.193
9.00	175.00	5.00	0.163
8.00	170.00	5.00	0.228
	concentration (%)  8.00  8.00  8.00  7.00  9.00  8.00  7.00  8.00  7.00  8.00  7.00  9.00  9.00  9.00  9.00	concentration (%)         temperature (°C)           8.00         175.00           8.00         170.00           8.00         165.00           7.00         175.00           9.00         170.00           8.00         170.00           8.00         170.00           8.00         170.00           8.00         170.00           7.00         170.00           7.00         175.00           9.00         165.00           9.00         175.00           9.00         175.00	concentration (%)         temperature (°C)         (rpm)           8.00         175.00         4.00           8.00         170.00         5.00           8.00         165.00         4.00           7.00         175.00         5.00           9.00         170.00         6.00           8.00         170.00         5.00           8.00         170.00         5.00           8.00         170.00         5.00           8.00         170.00         5.00           8.00         170.00         6.00           7.00         170.00         4.00           7.00         175.00         6.00           8.00         175.00         5.00           9.00         165.00         5.00           9.00         170.00         4.00           9.00         170.00         5.00

Table B4 Effect of process parameters on water activity of spray dried neera (Keramrutham) powder

Source	Sum of Squares	df	Squar	re F Value	p-value Prob > F	
Model	0.017	9	1.929E-003	38.69	< 0.0001	significant
A-feed concentra	otion 0.014	1	0.014	283.09	< 0.0001	
B-temperature	2.850E-003	1	2.850E-003	57.17	0.0001	
C-feed rate	1.361E-004	1	1.361E-004	2.73	0.1424	
AB	1.103E-004	1	1.103E-004	2.21	0.1806	
AC	2.500E-007	1	2.500E-007	5.015E-003	0.9455	
BC	4.000E-006	1	4.000E-006	0.080	0.7852	
$A^2$	4.585E-005	1	4.585E-005	0.92	0.3695	
$\mathbf{B}^2$	8.717E-005	1	8.717E-005	1.75	0.2276	
$\mathbb{C}^2$	8.853E-006	1	8.853E-006	0.18	0.6861	
Residual	3.489E-004	7	4.985E-005			
Lack of Fit	4.375E-005	3	1.458E-005	0.19	0.8974	not significant
Pure Error	3.052E-004	4	7.630E-005			_
Cor Total	0.018	16				
Std. Dev.	7.060E-003			R-Squared	0.9803	
Mean	0.24		A	dj R-Squared	0.9550	
C.V. %	3.00			red R-Squared	0.9335	
PRESS	1.177E-003			Adeq Precision	22.483	

Table B5 Effect of process parameters on wettability of Spray dried keramrutham powder

Run	Feed concentration	Inlet air temperature	Feed rate	Wettability (s)
	(%)	(°C)	(rpm)	
1	8.00	175.00	4.00	103
2	8.00	170.00	5.00	76
3	8.00	165.00	4.00	63
4	7.00	175.00	5.00	99
5	9.00	170.00	6.00	89
6	8.00	170.00	5.00	79
7	8.00	170.00	5.00	82
8	7.00	165.00	5.00	58
9	8.00	170.00	5.00	84
10	8.00	165.00	6.00	66
11	7.00	170.00	4.00	71
12	7.00	170.00	6.00	70
13	8.00	175.00	6.00	106
14	9.00	165.00	5.00	72
15	9.00	170.00	4.00	91
16	9.00	175.00	5.00	108
17	8.00	170.00	5.00	87

Table B6 Effect of process parameters on wettability of spray dried neera (Keramrutham) powder

Source	Sum of Squares	df	Squar	e F Value	p-value Prob > F	
Model	3621.67	9	402.41	24.19	0.0002	significant
A-feed concentration	1 480.50	1	480.50	28.88	0.0010	
B-temperature	3081.13	1	3081.13	185.21	< 0.0001	
C-feed rate	1.13	1	1.13	0.068	0.8023	
AB	6.25	1	6.25	0.38	0.5593	
AC	0.25	1	0.25	0.015	0.9059	
BC	0.000	1	0.000	0.000	1.0000	
$A^2$	2.69	1	2.69	0.16	0.6993	
$\mathbf{B}^2$	50.12	1	50.12	3.01	0.1262	
$\mathbb{C}^2$	1.27	1	1.27	0.077	0.7900	
Residual	116.45	7	16.64			
Lack of Fit	43.25	3	14.42	0.79	0.5602	not significant
Pure Error	73.20	4	18.30			-
Cor Total	3738.12	16				
Std. Dev.	4.08			R-Squared	0.9688	
Mean	82.59			Adj R-Squared	0.9288	
C.V. %	4.94			Pred R-Squared	0.7843	
PRESS	806.38			Adeq Precision	17.502	

Run	Feed concentration (%)	Inlet air temperature (°C)	Feed rate (rpm)	Bulk density (g/ml)
1	8.00	175.00	4.00	0.297
2	8.00	170.00	5.00	0.418
3	8.00	165.00	4.00	0.42
4	7.00	175.00	5.00	0.31
5	9.00	170.00	6.00	0.364
6	8.00	170.00	5.00	0.403
7	8.00	170.00	5.00	0.399
8	7.00	165.00	5.00	0.44
9	8.00	170.00	5.00	0.386
10	8.00	165.00	6.00	0.402
11	7.00	170.00	4.00	0.393
12	7.00	170.00	6.00	0.357
13	8.00	175.00	6.00	0.315
14	9.00	165.00	5.00	0.415
15	9.00	170.00	4.00	0.356
16	9.00	175.00	5.00	0.296
17	8.00	170.00	5.00	0.378

Table B8 Effect of process parameters on bulk density of spray dried neera (Keramrutham) powder

Source	Sum of Squares	df	Square	F Value	p-value Prob > F	
Model	0.032	9	3.544E-003	19.67	0.0004	significant
A-feed concentra	ation 5.951E-004	1	5.951E-004	3.30	0.1120	
B-temperature	0.026	1	0.026	146.18	< 0.0001	
C-feed rate	9.800E-005	1	9.800E-005	0.54	0.4848	
AB	3.025E-005	1	3.025E-005	0.17	0.6942	
AC	4.840E-004	1	4.840E-004	2.69	0.1452	
BC	3.240E-004	1	3.240E-004	1.80	0.2218	
$A^2$	5.353E-004	1	5.353E-004	2.97	0.1284	
$\mathbf{B}^2$	1.731E-003	1	1.731E-003	9.61	0.0173	
$\mathbb{C}^2$	1.368E-003	1	1.368E-003	7.59	0.0283	
Residual	1.261E-003	7	1.801E-004			
Lack of Fit	2.982E-004	3	9.942E-005	0.41	0.7533	not significant
Pure Error	9.628E-004	4	2.407E-004			
Cor Total	0.033	16				
Std. Dev.	0.013			R-Squared	0.9620	
Mean	0.37		Ad	lj R-Squared	0.9131	
C.V. %	3.59		Pred	l R-Squared	0.8107	
PRESS	6.276E-003		Ad	eq Precision	13.442	

Table B9 Effect of process parameters on Cold water solubility of keramrutham powder

Run	Feed concentration	Inlet air temperature	Feed rate	Cold water solubility (%)
1	(%)	(°C)	(rpm)	70
1	8.00	175.00	4.00	70
2	8.00	170.00	5.00	76
3	8.00	165.00	4.00	89
4	7.00	175.00	5.00	64
5	9.00	170.00	6.00	83
6	8.00	170.00	5.00	78
7	8.00	170.00	5.00	81
8	7.00	165.00	5.00	87
9	8.00	170.00	5.00	80
10	8.00	165.00	6.00	92
11	7.00	170.00	4.00	73
12	7.00	170.00	6.00	74
13	8.00	175.00	6.00	68
14	9.00	165.00	5.00	96
15	9.00	170.00	4.00	85
16	9.00	175.00	5.00	72
17	8.00	170.00	5.00	82

Table B10 Effect of process parameters on cold water solubility spray dried neera (Keramrutham) powder

Source	Sum of Squares	df	Square	F Value	p-value Prob > F	
Model	1204.42	9	133.82	33.82	< 0.0001	significant
A-feed concentration	on 180.50	1	180.50	45.61	0.0003	_
B-temperature	1012.50	1	1012.50	255.87	< 0.0001	
C-feed rate	0.000	1	0.000	0.000	1.0000	
AB0.25	1	0.25	0.063	0.8088		
AC2.25	1	2.25	0.57	0.4754		
BC6.25	1	6.25	1.58	0.2492		
$A^20.44$	1	0.44	0.11	0.7473		
$B^21.92$	1	1.92	0.48	0.5087		
$C^20.44$	1	0.44	0.11	0.7473		
Residual	27.70	7	3.96			
Lack of Fit	4.50	3	1.50	0.26	0.8523	not significant
Pure Error	23.20	4	5.80			_
Cor Total	1232.12	16				
Std. Dev.	1.99			R-Squared	0.9775	
Mean	79.41		A	Adj R-Squared	0.9486	
C.V. %	2.50			red R-Squared	0.9121	
PRESS	108.25			deq Precision	20.974	

Table B11 Effect of process parameters on  $colour(L^*)$  of Spray dried keramrutham powder

Feed concentration (%)	Inlet air temperature (°C)	Feed rate (rpm)	Colour (L*)
8.00	175.00	4.00	87.94
8.00	170.00	5.00	88.89
8.00	165.00	4.00	87.54
7.00	175.00	5.00	87.65
9.00	170.00	6.00	89.01
8.00	170.00	5.00	87.85
8.00	170.00	5.00	86.87
7.00	165.00	5.00	87.64
8.00	170.00	5.00	86.58
8.00	165.00	6.00	87.57
7.00	170.00	4.00	87.75
7.00	170.00	6.00	87.32
8.00	175.00	6.00	84.62
9.00	165.00	5.00	88.98
9.00	170.00	4.00	91.34
9.00	175.00	5.00	89.69
8.00	170.00	5.00	87.54
	concentration (%)  8.00  8.00  8.00  7.00  9.00  8.00  7.00  8.00  7.00  8.00  7.00  7.00  9.00  9.00  9.00  9.00	concentration (%)         temperature (°C)           8.00         175.00           8.00         170.00           8.00         165.00           7.00         175.00           9.00         170.00           8.00         170.00           8.00         170.00           8.00         170.00           8.00         170.00           7.00         170.00           7.00         175.00           9.00         165.00           9.00         175.00           9.00         175.00	concentration (%)         temperature (°C)         (rpm)           8.00         175.00         4.00           8.00         170.00         5.00           8.00         165.00         4.00           7.00         175.00         5.00           9.00         170.00         6.00           8.00         170.00         5.00           8.00         170.00         5.00           8.00         170.00         5.00           8.00         165.00         6.00           7.00         170.00         4.00           7.00         170.00         6.00           8.00         175.00         6.00           9.00         165.00         5.00           9.00         175.00         5.00

Table B12 Effect of process parameters on colour (L\*) of spray dried neera (Keramrutham) powder

Source	Sum of Squares	df	Square	e F Value	p-value Prob > F	
Model	27.71	9	3.08	4.22	0.0355	significant
A-feed concentrati	on 9.37	1	9.37	12.84	0.0089	_
B-temperature	0.42	1	0.42	0.57	0.4737	
C-feed rate	4.58	1	4.58	6.27	0.0408	
AB	0.12	1	0.12	0.17	0.6944	
AC	0.90	1	0.90	1.24	0.3030	
BC	2.81	1	2.81	3.84	0.0908	
$A^2$	8.74	1	8.74	11.97	0.0106	
$\mathbf{B}^2$	1.04	1	1.04	1.42	0.2718	
$\mathbb{C}^2$	0.073	1	0.073	0.10	0.7610	
Residual	5.11	7	0.73			
Lack of Fit	1.82	3	0.61	0.74	0.5815	not significant
Pure Error	3.29	4	0.82			-
Cor Total	32.83	16				
Std. Dev.	0.85			R-Squared	0.8443	
Mean	87.93			Adj R-Squared	0.6440	
C.V. %	0.97			Pred R-Squared	-0.0451	
PRESS	34.31			Adeq Precision	9.267	

Table B13 Effect of process parameters on colour  $(a^*)$  of Spray dried keramrutham powder

Run	Feed concentration (%)	Inlet air temperature (°C)	Feed rate (rpm)	Colour (a*)
1	8.00	175.00	4.00	1.24
2	8.00	170.00	5.00	0.81
3	8.00	165.00	4.00	1.25
4	7.00	175.00	5.00	1.34
5	9.00	170.00	6.00	0.79
6	8.00	170.00	5.00	0.7
7	8.00	170.00	5.00	0.64
8	7.00	165.00	5.00	1.11
9	8.00	170.00	5.00	0.34
10	8.00	165.00	6.00	0.99
11	7.00	170.00	4.00	0.92
12	7.00	170.00	6.00	0.89
13	8.00	175.00	6.00	1.21
14	9.00	165.00	5.00	0.9
15	9.00	170.00	4.00	0.82
16	9.00	175.00	5.00	1.03
17	8.00	170.00	5.00	0.46

Table B14 Effect of process parameters on colour(a\*) of spray dried neera (Keramrutham) powder

Source	Sum of Squares	df	Square	F Value	p-value Prob > F	
Model	1.07	9	0.12	5.07	0.0219	significant
A-feed concentra	tion 0.065	1	0.065	2.76	0.1409	_
B-temperature	0.041	1	0.041	1.73	0.2302	
C-feed rate	0.015	1	0.015	0.65	0.4463	
AB	2.500E-003	1	2.500E-003	0.11	0.7539	
AC	0.000	1	0.000	0.000	1.0000	
BC	0.013	1	0.013	0.56	0.4778	
$A^2$	0.037	1	0.037	1.57	0.2500	
$B^2$	0.71	1	0.71	30.28	0.0009	
$C^2$	0.12	1	0.12	5.25	0.0557	
Residual	0.16	7	0.024			
Lack of Fit	0.022	3	7.408E-003	0.21	0.8860	not significant
Pure Error	0.14	4	0.036			_
Cor Total	1.24	16				
Std. Dev.	0.15		R-Squared	0.8670		
Mean	0.91		dj R-Squared	0.6960		
C.V. %	16.88	Pre	ed R-Squared	0.5329		
PRESS	0.58		deq Precision	5.877		

Table B15 Effect of process parameters on colour (b $^{\star}$ ) of Spray dried keramrutham powder

Run	Feed concentration (%)	Inlet air temperature (°C)	Feed rate (rpm)	Colour (b*)
1	8.00	175.00	4.00	10.01
2	8.00	170.00	5.00	7.85
3	8.00	165.00	4.00	7.09
4	7.00	175.00	5.00	11.32
5	9.00	170.00	6.00	7.39
6	8.00	170.00	5.00	8.66
7	8.00	170.00	5.00	8.72
8	7.00	165.00	5.00	7.02
9	8.00	170.00	5.00	8.82
10	8.00	165.00	6.00	7.01
11	7.00	170.00	4.00	8.92
12	7.00	170.00	6.00	7.89
13	8.00	175.00	6.00	9.25
14	9.00	165.00	5.00	5.89
15	9.00	170.00	4.00	7.13
16	9.00	175.00	5.00	9.01
17	8.00	170.00	5.00	8.79

Table B16 Effect of process parameters on colour(b\*) of spray dried neera (Keramrutham) powder

Source	Sum of Squares	df	Square	F Value	p-value Prob > F	
Model	26.32	9	2.92	13.98	0.0011	significant
A-feed concentration	on 4.10	1	4.10	19.62	0.0030	C
B-temperature	19.78	1	19.78	94.57	< 0.0001	
C-feed rate	0.32	1	0.32	1.55	0.2533	
AB	0.35	1	0.35	1.66	0.2380	
AC	0.42	1	0.42	1.99	0.2013	
BC	0.12	1	0.12	0.55	0.4814	
$A^2$	0.62	1	0.62	2.95	0.1296	
$B^2$	0.066	1	0.066	0.31	0.5931	
$C^2$	0.52	1	0.52	2.50	0.1575	
Residual	1.46	7	0.21			
Lack of Fit	0.80	3	0.27	1.63	0.3176	not significant
Pure Error	0.66	4	0.16			C
Cor Total	27.79	16				
Std. Dev.		0.46		R-Squared	0.947	73
Mean		8.28		Adj R-Squar		
C.V. %		5.52		Pred R-Squa		
PRESS		13.90		Adeq Precisi		

Table B17 Effect of process parameters on hue angle (°) of Spray dried keramrutham powder

Run	Feed concentration	Inlet air temperature	Feed rate (rpm)	Hue angle (°)
	(%)	(°C)		
1	8.00	175.00	4.00	84.93
2	8.00	170.00	5.00	84.1
3	8.00	165.00	4.00	81.96
4	7.00	175.00	5.00	83.47
5	9.00	170.00	6.00	83.43
6	8.00	170.00	5.00	85.37
7	8.00	170.00	5.00	85.8
8	7.00	165.00	5.00	81.01
9	8.00	170.00	5.00	87.79
10	8.00	165.00	6.00	80
11	7.00	170.00	4.00	84.11
12	7.00	170.00	6.00	83.56
13	8.00	175.00	6.00	84.59
14	9.00	165.00	5.00	81.31
15	9.00	170.00	4.00	83.89
16	9.00	175.00	5.00	83.24
17	8.00	170.00	5.00	87

Table B18 Effect of process parameters on hue angle (°) of spray dried neera (Keramrutham) powder

Source	Sum of Squar	res df	Squai	re F Value	p-value Prob > F	
Model	57.36	9	6.37	4.58	0.0286	significant
A-feed concentra	ation9.800E-003	1	9.800E-003	7.046E-003	0.9355	
B-temperature	17.85	1	17.85	12.83	0.0089	
C-feed rate	1.37	1	1.37	0.98	0.3541	
AB	0.070	1	0.070	0.050	0.8286	
AC	2.025E-0031	2.025E-003	1.456E-003	0.9706		
BC	0.66	1	0.66	0.47	0.5143	
$A^2$	8.71	1	8.71	6.26	0.0408	
$B^2$	22.58	1	22.58	16.24	0.0050	
$C^2$	2.87	1	2.87	2.07	0.1938	
Residual	9.74	7	1.39			
Lack of Fit	1.49	3	0.50	0.24	0.8646	not significant
Pure Error	8.25	4	2.06			_
Cor Total	67.09	16				
Std. Dev.		1.18		R-Squared	0.85	49
Mean		83.86		Adj R-Squar	ed 0.66	83
C.V. %		1.41		Pred R-Squa		34
PRESS		36.67		Adeq Precisi		0

### APPENDIX C

## SENSORY SCORE CARD FOR SPRAYDRIED NEERA POWDERS

You are requested to assess the product in terms of general

Department of Food and Agricultural process engineering KCAET, KAU, TAVANUR

Date:

Name of judge:

Score system:

Comments if any:

acceptability on a 9 point hedonic scale

Like extremely 9						
Like very much 8						
Like moderately 7						
Like slightly 6						
Neither like nor dislik	e 5					
Dislike slightly 4						
Dislike moderately 3						
Dislike very much 2						
Dislike extremely 1						
Characteristics			Sam	ple		
			code	•		
	A	В	С	D	Е	F
Colour& appearance						
Flavor						
Taste						
Overall acceptability						

Signature

## APPENDIX D

## Estimation of Cost of Production of spray dried neera Powder

Cost of machineries		
Cost of spray dryer	=	Rs.12,00,000/-
Cost of sealing machine	=	Rs.10,000/-
Floor space 5 m <sup>2</sup> and building	=	Rs. 3,230/-
Miscellaneous item	=	Rs. 6,000/-
Cost of electric balance	=	Rs. 10,000/-
Total cost	=	Rs. 1,29,230/-
Assumptions		
Life span (L)	=	20 years
Annual working hours (H)	=	300 days (16 hours)
		= 4800
Salvage value (S)	=	8% of initial cost
Interest on initial cost (i)	=	13% annually
Repair and maintenance	=	5% of initial cost
Insurance and taxes	=	2% of initial cost
Electricity charge	=	Rs. 7/unit
Labour wages/person	=	Rs. 300/-

1. Total fixed cost per year	
i. Depreciation	$\frac{\text{C-S}}{\text{L} \times \text{H}} = \frac{1229230 - 98338.4}{20 \times 4800} = \text{Rs.}11.8/\text{h}$
ii. Interest	$\frac{C+S}{2} \times \frac{i}{H} = \frac{1229230 + 98338.4}{2} \times \frac{13}{100 \times 4800}$ Rs. 18/h
iii. Insurance & taxes	2% of initial cost $\frac{2}{100 \times 4800} \times 1229230 = \text{Rs.} 5.12/\text{h}$
Total fixed cost	i + ii + iii = 35/h = Rs. 168000/-
2. Total variable cost per year	
i. Repair & maintenance	5% of initial cost $\frac{5}{100 \times 4800} \times 1229230 = 12.8 / h$ Rs.61440/year

ii. Electricity cost		
Energy consumed by the spray dryer	=	5.8 kwh
Energy consumed by, sealing machine	=	0.2 kwh
and weighing balance		
Total energy consumption	=	6.0 kwh
Cost of energy consumption/h	=	Power × duration × cost of 1 unit
		$6 \times 4800 \times 7 = \text{Rs. } 201660/\text{year}$
iii. Labour cost (2 persons)	=	Rs. 500/day
		Rs. =150000/-
iv. Cost of raw materials		
Quantity of neera required	=	1000L/year
Cost of neera	=	Rs. 60000/-
Quantity of gum Arabic required	=	5 kg/year
Cost of gum Arabic	=	Rs. 5×1000=5000/-
Quantity of maltodextrin	=	50 kg/year
required		
Cost of maltodextrin	=	50×90=Rs. 4500/-
Total variable cost	=	i+ii+iii+iv+v= Rs. 482,600/-
Total cost for production of one	=	Fixed cost + Variable cost
kg of spray dried neera powder (		= Rs.650600/-
one kg per day)		= Rs. 2168.6 / kg

# DEVELOPMENT OF NEERA POWDER USING SPRAY DRYING PROCESS

By

ANJALI A V

(2017-18-002)

#### **ABSTRACT OF THE THESIS**

Submitted in partial fulfillment of the requirement for the degree of MASTER OF TECHNOLOGY

IN

#### AGRICULTURAL ENGINEERING

(Agricultural Processing and Food Engineering)

Faculty of Agricultural Engineering and Technology

Kerala Agricultural University



DEPARTMENT OF PROCESSING AND FOOD ENGINEERING
KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND
TECHNOLOGY, TAVANUR – 679573, KERALA, INDIA

#### Abstract

The Coconut Palm (Cocos nucifera) is one of the most important crops grown in humid tropics, cultivated for its multiple utilities, belongs to Arecaceae family. Since most of the components of coconut palm is getting transformed to useful products, it is referred to as "Tree of life". Neera is a delicious health drink collected from the immature unopened inflorescence of coconut palm. It is a nector like liquid having slightly alkaline pH and translucent in color. Neera is a rich source of natural sugars, minerals and vitamins and it contains substantial amounts of iron, phosphorus and ascorbic acid. The major difficulty associated with neera production is its natural fermentation. The product undergoes fermentation within 2-3 hours under ambient temperature. Thus the shelf life of the sap is identified as a major issue in the long distance transport of neera. The only solution is the development of neera powder by spray drying technology, so that it will arrest the fermentation by reducing the available water. This study mainly concentrated on development of a process protocol for spray dried neera powder, standardization of the spray drying parameters and quality analysis of neera powder. The neera used in the study was collected from two different sources, CPCRI (Kalparasa) and KAU (Keramrutham). The optimum parameters obtained for Kalparasa powder was 3.53% MD+0.353% GA as feed material combination, an inlet air temperature of 168°C and 4.25 rpm feed flow rate. Whereas for the Keramrutham powder the obtained optimum condition is 7.58% MD+0.758% GA as feed material combination and 171.89°C inlet air temperature with 4.82 rpm feed flow rate. The blower speed of 1200 rpm and air pressure 2kg/cm<sup>2</sup> were kept constant for developing the products. The physico chemical characteristics such as pH, TSS, Colour, moisture content, and reconstitution properties were determined. The optimally produced products were packed, stored in retort pouches and the quality characteristics such as pH, moisture content, vitamine C, antioxidant activity and phenolic content were also analysed up to 5 months. Both powders showed good acceptance in the sensory evaluation. The cost analysis of the product was done and cost of one kilogram was estimated as Rs 2168.6/-.