STUDIES ON MICROENCAPSULATION OF VANILLA EXTRACT.

by

SARIGA S

Department of Food and Agricultural Process Engineering

KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY

TAVANUR - 679573, MALAPPURAM

KERALA, INDIA

2015

STUDIES ON MICROENCAPSULATION OF VANILLA EXTRACT.

by

SARIGA S.

(2013 - 18 - 102)

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 & Technology

Kerala Agricultural University

Department of Food and Agricultural Process Engineering

KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY

TAVANUR , MALAPPURAM -679573

KERALA, INDIA

2015

DECLARATION

I, hereby declare that this thesis entitled **"STUDIES ON MICROENCAPSULATION OF VANILLA EXTRACT"** 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 to me of any degree, diploma, associateship, fellowship or other similar title, of any other University or Society.

Place: Tavanur **Sariga S.**

Date: **(2013-18-102)**

CERTIFICATE

Certified that this thesis entitled **"STUDIES ON MICROENCAPSULATION OF VANILLA EXTRACT"** is a record of research work done independently by **Ms. Sariga S. (2013-18-102)** under my guidance and supervision and that it has not previously formed the basis for the award of any degree, diploma, fellowship or associateship to her.

Tavanur,

Date:

Dr. Prince M.V.
(Major Advisor, Advisory Committee)
Associate Professor,
Department of Food and Agricultural Process Engineering,
KCAET, Tavanur

ACKNOWLEDGEMENT

I surrender myself and humbly bow before the ***Almighty*** *whose grace had endowed me the inner strength and confidence and blessed me with a helping hand at each step during this work.*

I must give my high, respectful and deep sense of gratitude and indebtedness to my Guide as well as Chairman of the Advisory Committee, ***Dr.Prince, M.V.****, Associate Professor, Department of Food and Agricultural Process Engineering, K.C.A.E.T, Tavanur for his valuable guidance and constant encouragement during the course of this research work. I consider myself very fortunate for being able to work with a very considerate, encouraging and helping guide like him.*

It is my pleasure to pay tribute to ***Dr. M.S. Hajilal****, Dean, K.C.A.E.T, Tavanur, for his advice and guidance rendered during this study.*

I would like to express my heartfelt gratitude to ***Dr. Sivaswami, M.****, former Dean, K.C.A.E.T, Tavanur, for his interest and kind advice given to me at all stages of my study.*

I have boundless pleasure to ***Dr. Santhi Mary Mathew,*** *Professor & Head, Department of Food and Agricultural Process Engineering, K.C.A.E.T, Tavanur and member of advisory committee for her sustained guidance, constant encouragement, untiring help and insightful consults throughout the period of the study.*

I express my deep gratitude to ***Dr. Sudheer, K.P.,*** *Associate Professor, Department of Food and Agricultural Process Engineering, K.C.A.E.T, Tavanur and* ***Dr. Jayan, P.R.,*** *Associate Professor and head , Department of Farm Power Machinery and Energy, K.C.A.E.T, Tavanur as a members of advisory committee for their esteemed help and encouragement offered during the entire course of study.*

*I place my special thanks to **Er. George Mathew**, Associate Professor, Department of Food and Agricultural Process Engineering, K.C.A.E.T, Tavanur, and **Dr. Rajesh, G.K.,** Associate Professor, Department of Food and Agricultural Process Engineering, K.C.A.E.T, Tavanur, for their unreserved help, incessant motivation and encouragement during the tenure of my study.*

*It is my pleasure to offer whole hearted thanks to **Mrs. Sreeja, R.**, Assistant Professor, Department of Food and Agricultural Process Engineering, K.C.A.E.T, Tavanur, for giving valuable suggestions and constant backing at all stages of this research work.*

*With deep sense of graduate and obligation, I would like to remember the helps rendered to me by **Dr. C.N. Ravishankar,** Head, Fish Processing Division, Central Institute of Fisheries Technology, **Dr. J. Bindu,** Senior Scientist, Fish Processing Division, Central Institute of Fisheries Technology and **Mr. G. Omanakuttan Nair,** Technical Officer of the Department of Food Packaging Division, CIFT, for their passionate co-operation for the completion of a part of my research work at CIFT.*

*I would like to acknowledge the valuable help rendered by **Dr. Nisha, P**., NIIST, Trivandrum for her sincere help, support and co-operation for the completion of a part of my research work.*

*I wish to express my gratitude to **Dr. Krishnan, S.**, Associate Professor and Head, Dept. of Agricultural Statistics, College of Horticulture, Vellanikkara and **Dr. Berin Pathrose,** Associate Professor, KVK, Malappuram for explaining me the statistical techniques required for the study.*

*My completion of this project could not have been accomplished without the support of my classmates especially, **Dileep Sean., Rakhi J.F., Padmavathi, D., Manasa, K., Yunous, A.,** and **Sheeja, P.S.,** for their constant and invaluable support rendered during the conduct of project work.*

I express my profound sense of gratitude to all the staff members of Dept. of FAPE, K.C.A.E.T, Tavanur, especially ***Mr. Rdhakrishanan,M.V.,*** *Lab Assistant for his immense help.*

I wish to express my hearted thanks to my seniors and juniors especially, ***Rana Salva, Sankalapa, K.B., Rachana, C., Chinnu, S.R., Anila, P.*** *and* ***Rajani, S.,*** *for their esteemed help and support offered during the study.*

I am immensely thankful to my friends, ***Dr. Anzeena Usman., Harikrishnan, M., Sonu, K. Shaji., and Mobin, M.*** *for their constant encouragement, cooperation, support and help throughout the study.*

My heartfelt thanks to ***Kerala Agricultural University*** *for providing me the KAU merit scholarship and for having proffered me a chance to study in this institution.*

On my personal note, I place every success of my life span at feet of my loving family for their patience, forbearance and assistance during the period of investigation.

Above all, I humbly acknowledge the grace and blessings of the supreme power for enlighten my life and fulfilling this well nurtured dream.

Sariga S

Dedicated to

My Parents and

Teachers

CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF PLATES

LIST OF SYMBOLS AND ABBREVATIONS

ºB	:	Brix
ºC	:	Degree Celsius
%	:	per cent
&	:	and
/	:	per
=	:	equal to
≈	:	approximate
±	:	plus or minus
$	:	dollar
µg	:	micro gram
µm	:	micro metre
ANOVA	:	analysis of variance
a_w	:	water activity
cfu	:	colony forming unit
cm	:	centimetre
CRD	:	completely randomized design
d.b	:	dry basis
DC	:	direct current
DE	:	dextrose equivalent
DMRT	:	Duncan's multiple range test
EE	:	Encapsulation efficiency
et al.	:	and others
etc.	:	etcetera
EB	:	ethyl butyrate
EE	:	encapsulation efficiency
Fig.	:	figure
g	:	gram
GA	:	gum arabic

h	:	hour (s)
ha	:	hectare
HP	:	horse power
I.D	:	inner diametre
i.e.	:	that is
KAU	:	Kerala Agricultural University
K.C.A.E.T	:	Kelappaji College of Agricultural Engineering and Technology
kg	:	kilogram
kW	:	kiloWatt
LCD	:	Liquid crystal display
m	:	metre
m.c	:	moisture content
MD	:	maltodextrin
min	:	minute (s)
ml	:	milli litre
mm	:	millimeter
MS	:	maize starch
Mt	:	million tonne
nm	:	nanometer
No.	:	number
O.D	:	outer diameter
rpm	:	revolution per minute
Rs.	:	Rupees
Sl.	:	serial
SPSS	:	Statistical Package for the Social Sciences
SS	:	stainless steel
USA	:	United States of America
viz.,	:	namely
WHO	:	World Health Organization

WPC	:	whey protein concentrate
WPI	:	whey protein isolate
w.b	:	wet basis
wt.	:	weight

CHAPTER I

INTRODUCTION

Spices are forming a major class of ingredients used in most of the food products. Spices and condiments can be defined as dried seed, fruit, bark and vegetable used for flavouring, colour, aroma and preservation of food or beverages. Spices or their extracts found its application in food, medicine, perfumery, cosmetics and other industries. The functional properties of spices or spice extracts as preservatives, antioxidants, anti-microbial, antibiotic and medicinal value increases their demand worldwide. Flavour is the main attraction of spices which includes a range of olfactory and taste sensation.

India is called as 'The Land of Spices' and Indian spices are known throughout the world. Spices in India contribute around 3% of the total agricultural commodities exported from India and contributes a share of around 0.27 % to the total export from India (Parthasarathy and Kandiannan, 2007). Vanilla is the world's most popular flavouring agent used in food, perfumery and cosmetics business.

Vanilla is the most expensive spice in the global market after Saffron (Bleu, 2009). Vanilla, the only member of *Orchidaceae* family has a real economic value in the food and related industries, because of its unique flavour and pleasant aroma. Vanillin in vanilla is responsible for the flavour and aroma. Vanillin is obtained primarily from the grown but unripe fruits or 'beans' of a climbing orchid *Vanilla planifolia* (*V. fragrans*).

The cultivation and processing of vanilla depends on market price and the fluctuation in price is affecting its cultivation. India has produced 92 MT of vanilla during 2002-03 with total area of 2545 ha; this is hardly 1.90% of total average world production. For the period ending 2007 the targets have been fixed at 15,000 ha (Parthasarathy and Kandiannan, 2007).

Vanilla flavour is used as flavourings in ice-creams, soft drinks, candy, chocolate, confectionary, baked foods, puddings, cakes, cookies etc. It is also used

in odour fixatives, deodorants, perfumes. There are many compounds present in the extracts of vanilla. The chemical compound vanillin (4-hydroxy-3-methoxy-benzaldehyde) is primarily responsible for the flavour and smell of vanilla. Another minor component of vanilla essential oil is piperonal (heliotropin). Piperonal and other substances affect the odour of natural vanilla.

Consumers prefer natural vanillin as the essence of natural flavour is no way comparable to that of its synthetic vanillin. Vanilla which is essential to some foods, play important roles in consumer satisfaction in terms of flavour and aroma and can promote the consumption of those products. Vanillin flavour is highly volatile and therefore flavour retention is limited. Therefore techniques such as microencapsulation would increase the retention and therefore its functionality and stability.

Microencapsulation is a process by which solids, liquids or even gases may be enclosed in thin coatings or wall material around the substances. It provides the means of converting liquids to solids, of altering colloidal and surface properties, of providing environmental protection and creation of a barrier to avoid chemical reactions and/or to enable the controlled release of the coated materials (Vilstrup, 2001). Microencapsulation with suitable wall material can protect the flavour from undesirable interactions with food, reduces off-flavour, minimize the oxidation, increase shelf-life, allow a controlled release and retain aroma in a food product during storage. The microencapsulation of active component may be carried out by different techniques such as spray-drying, spray freeze drying, spray-cooling, spray-chilling, freeze-drying, centrifugal extrusion, air suspension coating, extrusion, coacervation, co-crystallization, rotational suspension separation, liposome entrapment, interfacial polymerization, molecular inclusion, etc. Among the various methods, spray drying is the most common and economical method to carry out microencapsulation process since the process is simple, relatively inexpensive, rapid, continuous and produces particles of good quality. This method is especially suited for large volume low cost requirements such as in food industry.

Microencapsulation by spray drying is a fast and continuous dehydration process in which the feed solution is transformed into a solid powder forms a continuous matrix surrounding the active substances of micro particles after short drying period. The feed liquid (solution, emulsion or suspension) containing wall material and core material are sprayed into heated air in the spray drier. The solvent (almost always water) from the feed is evaporated to give instantaneous powder. The heated air supplies the latent heat of vaporization required to remove the solvent from the wall material, thus forming the microencapsulated product (Khawla *et al*., 1996).

The wall material coat the core material (flavour load) and it protects the active component from undesirable changes. The selection of appropriate wall material (coating material, shell material) decides the physico-chemical properties of the resultant microencapsulated product. The properties of wall material *i.e*., stabilization, volatility, release characteristics, oxidation, environmental conditions, etc. should be taken into consideration for efficient encapsulation. The wall material should possess the properties as capable of forming a film that is cohesive with the core material, chemically compatible and inert with the core material.

The process parameters which have been stated as influencing the volatile retention during the process and which can be controlled are type of wall material, concentration of the active substance to be retained (flavour load), concentration and viscosity of the emulsion, emulsion stability and size, dryer inlet temperature and capsule morphology (Re, 1998).

Though there have been reports of a number of encapsulating agents used as wall material, a judicious choice according to the desired application is an important task. Maltodextrin and maize starch are viable option as they are not only a good matrix former but also provides protection against oxidation, permits increased solid content with low viscosity which improves encapsulation efficiency, and is economical besides other advantages. The proportion of the wall

material, therefore, needs to be optimized based on emulsion and product characteristics.

Though there have been studies on various parameters that affect retention of active ingredient during encapsulation, a generalisation could not be arrived at in many cases. The optimised values differ for each material encapsulated. In this context, it may be noted that such optimisation studies pertaining to vanilla extract have not been found reported. This study focus on the standardization of wall material and flavour load (core concentration) and optimization of the operating parameters of the encapsulation process (inlet air temperature) by spray drying technique. Also, the microstructural and storage stability of the product would be analysed. Vanilla extract, being such a valuable and sensitive product, which possess significant scope for value addition for domestic consumption and export, such studies can improve the utilization potential, storage stability, convenience and effectiveness of use and thus contribute to its overall quality improvement.

It is in the backdrop of all the above factors that a study was undertaken with the following objectives.

1. To standardize the wall materials and flavour load (core concentration) for microencapsulation.
2. To optimize the operating parameters (inlet air temperature) of the encapsulation process by spray drying technique.
3. To carryout microstructural and storage characteristics of the optimally produced encapsulated vanilla extract powder.

CHAPTER II

REVIEW OF LITERATURE

This chapter provides the background information relevant to the understanding of the microencapsulation of vanilla extract. In order to carry out better and systematic research work, the earlier studies conducted on the effect of wall material and its concentration on emulsifying properties, spray drying parameters, microencapsulation efficiency, microstructural characteristics and storage conditions involved in efficient encapsulation and improving the quality of microencapsulated vanillin powder produced has been reviewed in a detailed manner.

2.1 VANILLA

Vanilla (*Vanilla planifolia*) is known as the 'flavour queen', which is a crop of great commercial importance as the source of natural vanillin, a major component of flavor industry. The major natural vanilla flavor used in food, beverages, cosmetics and tobacco come from the pods of *V. planifolia*. This species has its origin in Mexico where the pods had been used for medicinal purposes as well as flavoring for hot chocolate drinks at the court of Emperor Montezuma (Bruman, 1948).

The genus Vanilla comprises of about 110 species, distributed in tropical parts of the world (Purseglove *et al*., 1981) and commercial vanilla, an important and popular flavouring material and spice, is obtained from the fully grown and processed fruit of tropical climbing orchid.

Authentic vanilla extracts are prepared from the pods of *Vanilla planifolia*. Cultivation of the pods is expensive and synthetic or artificial vanilla extracts are widely used. Although the major flavor constituent of vanilla extract is vanillin (4-hydroxy-3-methoxybenzaldehyde), many other volatile compounds such as guaiacol, p-anisaldehyde, and methyl cinnamate have been reported to contribute to its flavour (Lamprecht *et al*., 1994).

Ranadive (1994) reported that over one hundred volatile compounds have been detected in vanilla, including aromatic carbonyls, aromatic alcohols, aromatic acids, aromatic esters, phenols and phenols ethers, aliphatic alcohols, carbonyls, acids, esters and lactones, of which the aldehyde vanillin is the most abundant.

Natural vanilla flavour, obtained from cured beans of *Vanilla planifolia* forms the highest priced flavour ingredient in food (60%), cosmetics (33%) and aromatherapy (7%) (Priefert *et al.*, 2001). Green vanilla beans are odourless and flavourless. Characteristic vanilla flavour in beans is only formed during curing process, which results in 2% vanillin and over 170 other compounds with delicate sweet fragrance (Mark *et al.*, 2002).

Vanilla is widely used as a flavouring compound in bakery, beverage, and ice-cream industries. Vanillin (3-methoxy-4-hydroxybenzaldehyde) is extracted from the pods of *Vanilla planifolia* through an expensive process. Approximately 50% of the world production of vanillin is used as an intermediate in the production of herbicides, antifoaming agents or drugs (Walton *et al*., 2003).

Kumar (2004) studied the demand-supply of vanilla and reported that in the international market, world demand for vanilla was around 32,000 tonnes and the demand for natural vanillin is increasing at 7–10 per annum as the world is shifting towards herbal products.

Twenty six odour-active compounds were detected by Gas Chromatography–Olfactometry analysis of organic extract from cured vanilla. They included shikimate derivatives (11), organic acids (5), aldehydes (4), esters (3), ketones (2) and one aliphatic alcohol (Pérez *et al*., 2006). Sinha *et al.* (2008) reported the uses of vanillin as an antioxidant, anticarcinogenic, antimutagenic, and antisickling agent.

Despite the synthetic production of vanillin, the major compound of vanilla extract, vanilla pods are the only source of the more complex vanilla flavour. World production of vanilla cured pods varies between 2000 and 3000 tonnes per year with a price of around $20-30 per kilogram (Gleason, 2009). Due

to phenolic character of vanillin, it is used as a chemical intermediate during the production of fine chemicals and pharmaceuticals (Claudio *et al*., 2010).

According to Xia *et al.* (2011) flavours are essential to some foods, play important roles in consumer satisfaction and can promote the consumption of those products. However, the stability of flavours in foods, especially during processing of food, has attracted intense attention due to its relationship to the quality and acceptability.

Yang *et al.* (2014) conducted a study on production of flavour microcapsules containing vanilla oil by the complex coacervation approach for the purpose of controlling vanilla oil release and improving its thermostability. 94.2% of maximum encapsulation efficiency was obtained in vanilla oil /Chitosan ratio of 2:1 and the thermostability and long residual action of vanilla oil were effectively enhanced after being encapsulated in the microcapsules.

Hundre *et al*. (2015) suggested that vanillin flavour is highly volatile in nature and due to this application as food incorporation is limited. It was recommended that techniques such as microencapsulation of vanillin are an ideal technique to increase its stability and functionality.

2.2 MICROENCAPSULATION

According to Rosenberg *et al.* (1985), microencapsulation is a processing method in which small quantities of solid, liquid and gaseous materials are packed into a wall matrix; which forms microcapsules.

Shahidi and Han (1993) proposed six reasons for applying microencapsulation in food industry to reduce the core reactivity with environmental factors; to decrease the transfer rate of the core material to the outside environment; to promote easier handling; to control the release of the core material; to mask the core taste; and finally to dilute the core material when it should be used in only very small amounts.

Lin *et al.* (1995) conducted a study on microencapsulation of squid oil with hydrophilic macromolecules for oxidative and thermal stabilization. Gelatin,

sodium caseinate, and maltodextrin were the wall materials used. The oxidative and thermal stabilities of crude squid oil have been effectively enhanced by spray-drying microencapsulation.

According to Sheu and Rosenberg (1995), microencapsulation was a process where droplets core were coated by thin films which protect the core until it needed. During microencapsulation the entrapment of sensitive ingredients within a continuous film or coating could protect them from environmental factors such as moisture, air or light (Onwulata *et al.,* 1995).

Jain *et al*. (1997) stated that microencapsulation is a technology of packaging solids, liquids or gaseous material in miniature sealed capsules that could release their contents at controlled rates at specific conditions. The miniature packs in packages, called 'microcapsules', may range from micron to several millimeters in size and was ideally spherical.

Gibbs *et al*. (1999) narrated three precautions for developing microcapsules: formation of the wall around the material, ensuring that leakage does not occur and ensuring that undesired materials are kept out for which encapsulation techniques such as spray drying, spray chilling or spray cooling, extrusion coating, fluidized-bed coating, liposomal entrapment, lyophilization, coacervation, centrifugal suspension separation, co-crystallization and inclusion complexation could be used.

Microencapsulation is the technique by which one material or a mixture of materials is coated with or entrapped within another material or system. The coated material is called active or core material, and the coating material is called shell, wall material, carrier or encapsulant. The simplest of the microcapsules may consist of a core surrounded by a wall or barrier of uniform or non-uniform thickness. The core may be composed of just one or several types of ingredients and the wall may be single or double-layered (Augustin *et al.*, 2001).

According to Desai and Park (2005) microencapsulation process is used in food industry for several reasons: 1) encapsulation (entrapment) to protect the core material from degradation by reducing its reactivity to its outside

environment (e.g., heat, moisture, air, and light); 2) to decrease/retard the evaporation or transfer rate of the core material to the outside environment; 3) to avoid alterations in the physical characteristics of the original material, that can be modified easier to handle; 4) the product can be tailored to either release slowly over time or at a certain point (*i.e.*, to control the release of the core material to achieve the property delay until the right stimulus); 5) to mask the flavour of some substances that are microencapsulated and used as the core material; 6) the core material can be diluted when only very small amounts are required, yet still achieve a uniform dispersion in the host material, and to separate components in a mixture that would otherwise react with one another.

Shaw *et al.* (2007) conducted a study on spray dried multilayered emulsions as a delivery method for ω-3 fatty acids into food systems. It was found that microencapsulation of menhaden oil-in-water emulsions stabilized with a multilayer system consisting of lecithin and chitosan could be an effective technology to produce ω-3 fatty acid delivery system for use in functional foods.

Parize *et al.* (2008) microencapsulated the natural urucum pigment with chitosan by spray drying in different solvents. This study demonstrates that urucum pigment can be successfully incorporated into chitosan by means of a spray-drying process, resulting in dry and colorful powders which are water soluble.

Lezama *et al.* (2012) microencapsulated non-aqueous extracts from chilli (*Capsicum annuum L*.) by spray drying. They reported that approximately 80% of the antioxidant activity of the non-aqueous extracts was preserved in microencapsulates. The microcapsules did not contain fractures, and this finding may have contributed to the protective action against oxidation.

Balasubramani *et al.* (2013) conducted a study on microencapsulation of garlic (*Allium sativum L*.) oleoresin by spray drying. The study was carried out with variable core material concentrations (10, 20 and 30%), drying inlet air temperatures (180, 200 and 220^{0}C) and different wall material concentrations (40, 50 and 60%). The optimum conditions obtained for microencapsulation of garlic

oleoresin were, core material at 10% concentration, wall material at 60% concentration and drying inlet air temperature of 220^0C.

Hundre *et al.* (2015) microencapsulated vanillin extract with different wall materials like β-cyclodextrin (β -cyd), whey protein isolates (WPI) and combinations of these wall materials (β -cyd + WPI). It was revealed that microencapsulation of vanillin is an ideal technique to increase its stability and functionality.

2.3 MATERIALS FOR MICROENCAPSULATION

2.3.1 Core Material

According to Jackson and Lee (1991), the core material can be released at different times depending on the properties of the coating that is applied. The coating on a core is a semipermeable membrane which protects the core from severe conditions, control substances flowing into the core and the release of the metabolites from the core.

Madene *et al.* (2005) stated that the core material was composed of one or several droplets. Core material should be of low water solubility with low rates of dissolution, which led to decrease in particle size of suspended core material. Very small core particle give rise to aggregation problems during production because of their increased surface attractive forces. Large particles can cause problems because of their rapid sedimentation. Spherical cores of very narrow size were easier to deposit with uniform coating.

2.3.2 Wall Materials and its Properties

Reineccius (1988) stated that the wall material for microencapsulation by spray-drying should possess good emulsification properties and film forming, and drying properties and the wall concentrated solutions should have low viscosity. In addition to these properties, the wall material must not have reactivity with the core material; be present in a form that is easy to handle, *i.e.* with low viscosity at high concentrations, give the maximum protection of the active ingredient against the external factors, ensure good emulsion-stabilization properties and effective

redispersion behaviour in order to release the flavour at the desired time and place.

A suitable material that can be used for microencapsulation should possess high emulsifying activity, high stability and tendency to form fine and dense network during drying. It should not permit lipid separation from the emulsion during dehydration (Tanimoto and Matsuno, 1992).

Alonso *et al.* (2003) stated that the structure formed by the microencapsulating agent around the microencapsulated compound (core) is called a "wall"; this wall protects the core compound from biological degradation and enhances its stability. Because of the direct effect of the wall on microencapsulation efficiency, microencapsulation stability, and protection efficiency of the core compound, the selection of the wall material is very important in the microencapsulation process.

Depending on the core material and the characteristics desired in the final product, wall materials can be selected from a wide variety of natural and synthetic polymers. Since almost all spray drying processes in the food industry are carried out from aqueous feed formulation, the wall material must be soluble in water at an acceptable level (Gouin, 2004).

Goud and Park (2005) reported that an ideal coating material should exhibit some characteristics such as good rheological properties at high concentration and easy workability during encapsulation, ability to disperse or emulsify the active material and stabilize the emulsion produced, non-reactivity with the material to be encapsulated both during processing and on prolonged storage, ability to seal and hold the active material within its structure during processing or storage, ability to completely release the solvent or other materials used during the process of encapsulation under drying or other desolventization conditions, ability to provide maximum protection to the active material against environmental conditions (e.g., oxygen, heat, light, humidity), solubility in solvents acceptable in the food industry (e.g., water, ethanol), chemical nonreactivity with the active core materials, inexpensive and food-grade status.

The selection of wall material combinations affects both the emulsion properties and the particles characteristics after drying and during storage. The emulsion characteristics such as stability, viscosity, droplets size, as well as powder properties such as surface oil, particle size, density, morphology and oxidative stability, are influenced by the type of encapsulating agent used (Jafari *et al*., 2008).

Microencapsulation through spray-drying protects the food ingredients, such as carotenoids and polyphenols from chemical deterioration and environmental factors. This protection was performed using wall materials, such as sugars, gums, proteins, natural and modified polysaccharides, lipids, and synthetic polymers (Rocha *et al*., 2012).

Balasubramani *et al.* (2013) microencapsulated garlic (*Allium sativum L.)* oleoresin by spray drying with different wall material concentrations (40, 50 and 60%). The allicin content increased with increase in wall material concentration irrespective of core material concentration and drying inlet temperature.

Garza *et al.* (2013) microencapsulated astaxanthin oleoresin from *H. pluvialis* using lecithin as an emulsifier and several wall materials. It was concluded that the encapsulation yield and the properties of the microcapsules depended on the type of wall material.

2.3.2.1 Maltodextrin

Anandaraman and Reineccius (1986) encapsulated orange peel oil by spray drying process. They reported that higher DE (dextrose equivalents) maltodextrin form a denser and more oxygen impermeable matrix providing longer shelf life for orange peel oil.

Maltodextrin is made up of partially hydrolyzing maize starch with acids or enzymes and they are supplied at different dextrose equivalents which is a measure of degree of hydrolysis of starch polymer (Kenyon and Anderson, 1988).

Raja *et al.* (1989) showed that maltodextrin with dextrose equivalence between 10 and 20 fit in for using as wall material. Those maltodextrin samples

show the highest retention of flavor because they could be dispersed in water up to 35.5% of the solution without haze formation.

According to Mckernan (1992), maltodextrin were being increasingly used in microencapsulation studies as they had acceptable food grade qualities. They were found to be non- reactive with core materials with low viscosity at high solid content and were relatively inexpensive.

Shahidi and Han (1993) reported that maltodextrin improves the shelf life of citrus oils because maltodextrin had low flavour, and can be used at high solid concentration. Blending of corn syrup solids, maltodextrin and modified starch may lead to optimal microencapsulating materials. Spray drying and extrusion process of the individual components have been used as water soluble coating techniques.

Gibbs *et al.* (1999) reported that the most commonly used materials for microencapsulation are maltodextrin of different dextrose equivalents. Maltodextrin are obtained by acid hydrolysis of several starches (corn, potato or others). Maltodextrin have high solubility in water, low viscosity, bland flavour and colourless solutions and are extensively used in the food industry.

Buffo and Reineccius (2000) optimized gum acacia/modified starches/ maltodextrin blends for the spray drying of flavours. It was revealed that maltodextrin exhibits various favourable characteristics, including high hydrosolubility, low emulsifying capacity, low retention of volatiles, and high protection against oxidation.

Usage of maltodextrin as wall material for production of *Amaranthus* betacyanin pigment significantly reduced the hygroscopicity of the betacyanin extracts enhancing their storage stability (Cai and Corke, 2000).

Belghith *et al.* (2001) conducted an experiment on stabilization of *Penicillium occitanis* cellulases by spray drying in presence of maltodextrin. They reported that 1% maltodextrin had a negative effect on enzyme recovery and it completely stabilized cellulases even after a long period (8 months) of storage at

30°C. By using maltodextrin the activity loss during spray-drying is reduced and storage stability improved.

Maltodextrin is a hydrolyzed starch which offers advantages such as low cost, neutral aroma and taste, low viscosity at high solids concentrations and good protection against oxidation (Gharsallaoui *et al*., 2007). Maltodextrin had low flavour and were non-reactive with the core component (anthocyanin) used for microencapsulation. It is improved the shelf life of black carrot anthocyanins and maintained the colour and stability during storage (Ersus and Yurdagel, 2007).

Kha *et al.* (2010) conducted a study on effects of spray drying conditions on the physicochemical and antioxidant properties of the Gac fruit aril powder. Spray drying of this material has not been successful and maltodextrin is considered as a suitable drying aid to preserve its colour and antioxidant properties. Moisture content and bulk density, colour characteristics, total carotenoid content (TCC), encapsulation efficiency and total antioxidant activity (TAA) were significantly affected by maltodextrin concentration.

The microstructural analysis of the microspheres obtained with different carriers, the use of maltodextrin resulted in more homogeneous particles, which is recommended in the spray drying microencapsulation process (Silva *et al.,* 2013).

Fernandes *et al*. (2014) microencapsulated rosemary essential oil with gum arabic, starch and maltodextrin as wall material. The mixture of modified starch and maltodextrin proved to be an effective matrix for retaining rosemary essential oil. Maltodextrin is used in combination with another encapsulating agent because it does not have an emulsifying capacity; it has the advantage of being relatively inexpensive and provides excellent protection of the encapsulated materials.

2.3.2.2 Maize Starch

Carbohydrates such as starches, corn syrup solids and maltodextrin have been usually used as encapsulating agents. These materials are considered as good encapsulating agents because they exhibit low viscosities at high solids contents and good solubility, but most of them lack the interfacial properties required for

high microencapsulation efficiency and generally associated with other encapsulating materials such as proteins or gums (DeZarn, 1995).

Singh and Singh (2003) prepared granulated cold water soluble starches from corn starch and potato starch and reported that corn starch showed higher transition temperature and lower enthalpy of gelatinization than potato starch.

Starch is a kind of abundant renewable resources from plants. Within the category of starch, corn starch, which has a long history and unique advantages, is very representative. It is a typical type starch and in its polymorphs, the double helices are closely packed together with a small amount of structural water (Bogracheva *et al.*, 2006).

Lago *et al.* (2013) conducted a study on edible coating from maize and cassava starches retain carotenoids in pumpkin during drying. They reported that maize starch coatings resulted in dehydrated products with better color and with significantly higher retention of trans-a-carotene and trans-b-carotene than the products that did not receive the pretreatment.

Corn starch occupies an important place in the preparation of pies, puddings, salad dressings and confections. It is incorporated into cakes, cookies, icings and fillings to increase moisture retention, retard crystal growth of other sugars and to improve tenderness and keeping quality. Maize starches can be used as gelling agents at lower temperature than rice starches due to their lower pasting temperature (Ali *et al*., 2014).

Xu and Zhang (2014) conducted a study on slow digestion property of microencapsulated normal corn starch. Starch is the main glycemic dietary carbohydrate, and its nutritional quality is associated with the amount of slowly digestible starch (SDS) that is beneficial to glycemic control. In this study, a microencapsulation of normal corn starch by zein protein and its slow digestion property were investigated. A significant increase of SDS and RS (resistant starch) was shown for starch capsules (weight ratio of zein to starch: 1:6) containing plasticizers of glycerol and oleic acid after high temperature (70^0C)

treatment. The acceptable sensory property makes it an ideal ingredient for specialty food preparation and glycemic control.

2.4 MICROENCAPSULATION BY SPRAY DRYING

The production of microencapsulated powders by spray-drying generally involved the formation of a stable emulsion in which the wall material acted as a stabilizer for the core material. The emulsion was then spray-dried to yield the encapsulated powder product (Reineccius, 1988).

Masters (1991) observed that spray drying has paved the way for the production of powder colorants with high storage stability, easier to handle for some applications, and minimize the weight for transportation, in comparison with liquid concentrate.

Shahidi and Han (1993) suggested that the microencapsulation by spray drying involves four stages: preparation of the dispersion or emulsion; homogenization of the dispersion; atomization of the infeed emulsion; and dehydration of the atomized particles.

Deis (1997) reported that spray drying was one of the commercial processes which was widely used in large-scale production of encapsulated flavours and pigments. Spray drying provides a very large surface area, which enhances oxidation, if the wall material is not thick or dense enough to act as a good oxygen barrier.

Desobry *et al*. (1997) compared the freeze drying and spray drying techniques and reported that spray drying is the most common and cheapest technique to produce microencapsulated food materials. Equipment is readily available and production costs are lower than most other methods. Compared to freeze drying, the cost of spray-drying method is 30–50 times cheaper.

The main compounds of the essential oils, responsible for the flavour and the functional properties, are volatiles and chemically unstable in the presence of oxygen, moisture and heat. Stability of essential oils can be enhanced through

microencapsulation by spray drying. Spray drying is the most commonly used encapsulation technique in the food industry (Re, 1998).

Cai and Corke (2000) studied on production properties of spray dried amaranthus betacyanin pigments and reported that spray drying was an economical method for preservation of natural colorants by entrapping the ingredients in coating materials. Spray drying is the most common and cheapest technique to produce microencapsulated food materials.

Tari and Singhal (2002) reported that microencapsulation by spray drying can protect the flavour from undesirable interactions with food, minimize loss against light-induced reactions and oxidation, increase the flavour shelf life, allow a controlled release and retain aroma in a food product during storage.

The spray drying process involves the dispersion of the substance to be encapsulated in a carrier material, followed by atomization and spraying of the mixture into a hot chamber (Watanabe *et al*., 2002). The resulting microcapsules were then transported to a cyclone separator for recovery.

Drusch (2007) reported that the rheological properties of the emulsion are the key parameter in the spray drying process; thus, an emulsion with high viscosity causes the formation of large droplets which affects the drying rate.

Ersus and Yurdagel (2007) studied microencapsulation of black carrot anthocyanin using spray drying and reported that maltodextrin had low flavour and were non-reactive with the core component (anthocyanin) used for microencapsulation. It improved the shelf life of black carrot anthocyanins and maintained the colour and stability during storage.

Parize *et al*. (2008) conducted a study on microencapsulation of natural urucum pigment extracted from *Bixa orellana* seed by spray drying. It was observed that urucum pigment could be successfully incorporated into chitosan by spray-drying process and obtained water soluble dry and colourful powders.

Obon *et al*. (2009) found that betacyanin pigment obtained from *Opuntia stricta* microencapsulated by co-current spray drying of *O.stricta* fruit juices with

bench-scale two fluid nozzle spray dryer showed high color strength when stored at room temperature for one month. He reported that spray dried food powders show high storage stability, good handling characteristics (for some applications) and minimized transportation weight in comparison with liquid concentrates. Spray drying is a common method of encapsulation of food ingredients in the food industry.

Spray drying produces, depending on the starting feed material and operating conditions, a very fine powder (10-50 μm) or large size particles (2-3 mm), which are separated in a cyclone after their formation (deVos *et al*., 2010).

2.4.1 Emulsion Preparation for Microencapsulation

2.4.1.1 Emulsion Droplet Size and Viscosity

The flavour retention and the stability of the encapsulated flavours will also be influenced by the emulsion droplet size and viscosity of the feed liquid. Rosenberg *et al*. (1990) conducted studies by adding sodium alginate to gum arabic/ethyl caproate infeed and monitored the effect of alginate addition on ethyl caproate retention during spray drying. It was reported that the optimum infeed viscosity ranged from 1250 to 2500 cp.

In feed emulsion viscosity exerts effect on flavour retention during microencapsulation by circulation currents within the drying droplet and time to form discrete droplets (Coumans *et al*., 1994). If the viscosity is low, internal mixing occurs during drying which delays the formation of semi permeable surface. This delay permits greater flavour loss during early drying, thus an increase in feed viscosity too much will slow the formation of discrete particles during atomization which promotes volatile losses (King, 1995).

Sheu and Rosenberg (1995) produced microcapsules of ethyl caprylate consisting of whey protein and carbohydrate as wall material. They suggested that the retention of volatile during microencapsulation by spray drying could be enhanced by reducing the mean size of the dispersed core material during emulsification. Ethyl caprylate retention was improved by reducing the mean

dispersed particle size when the combination of whey protein and maltodextrin was used as the carrier.

Hogan *et al*. (2001) conducted a study on the viscosity of the emulsion containing various levels of soy oil dispersed in whey protein concentrate solution. It was found that viscosity of the emulsion was largely unaffected by increasing oil/protein ratio from 0.25 to 0.75 but increased significantly at ratios more than one. The increase in apparent viscosity was associated with increase in the dispersed volume.

Yoshii *et al*. (2001) conducted a study on flavour release from spray dried maltodextrin/gum arabic or soy matrices as a function of storage relative humidity. Retention of emulsified ethyl butyrate during spray drying was dependent on the concentration of maltodextrin and the type of emulsifier. Addition of 1% gelatin as emulsifier in the feed liquid had a pronounced influence in increasing the retention of ethyl butyrate during spray drying, and also in controlling the release rate of the encapsulated ethyl butyrate.

Soottitantawat *et al*. (2003) conducted a study on the influence of the mean emulsion droplet size on flavour retention during spray drying of d-limonene, ethyl butyrate, and ethyl propionate. Gum arabic, soybean water-soluble polysaccharides, or modified starch blended with maltodextrin, were used as wall material. The increasing emulsion droplet size decreased the retention of flavours. The distribution curve containing large emulsion droplets shifted towards a smaller size after atomization, indicating that the larger emulsion droplets would be changed in sizing during atomization and result in decreasing flavour retention. When ethyl butyrate and ethyl propionate was used, the retention was found to be higher.

Barbosa *et al*. (2005) reported that the microcapsules containing emulsifiers, gum arabic and maltodextrin plus Tween 80 showed the highest encapsulation efficiency which was higher than bixin encapsulated with maltodextrin alone.

2.4.2 Solid Content of Feed and Core to Wall Ratio

Increasing the total solids content of the emulsion and drying temperature improved the retention of ethyl caproate in gum arabic during spray drying (Rosenberg *et al*., 1990).

Lee and Rosenberg (2000) prepared whey protein based microcapsules and found that the effects of core-to-wall ratio ranging from 1:1.5 to 5:1.5 on formation, properties and core release from whey protein-based microcapsules. Core retention and microcapsules size increased with increase in core to wall ratio and also influenced the outer topography and inner structure of microcapsules.

Yoshii *et al*. (2001) studied flavour release kinetics from spray dried maltodextrin/gum arabic or soy matrices as a function of storage relative humidity. It was observed that release of ethyl butyrate decreased as the concentration of maltodextrin in the feed liquid increased.

The higher solid content in the feed increased the retention of active ingredient during spray drying by reducing the time required to form a semi permeable membrane at the drying particle surface (Buffo *et al*., 2002).

Swapnali *et al*. (2003) studied microencapsulation of cinnamon oleoresin by spray drying using binary and ternary blends of gum arabic, maltodextrin, and modified starch as wall materials. They observed that a 4:1:1 blend of gum arabic: maltodextrin: modified starch offered a protection, better than gum.

Huezo *et al.* (2004) conducted a study on microencapsulation of red chilly carotenoid pigment with gum arabic at different core to wall ratio (1:2.6, 1:3.9 and 1:1.4). They observed that 1:3.9 core to wall ratio showed highest encapsulation efficiency.

Microencapsulation of cardamom oleoresin with gum arabic, maltodextrin and modified starch as wall materials were studied by Krishnan *et al.* (2005). The study showed that the stability of the cardamom oleoresins decreased as the quantity of gum arabic decreased in its blend. Gum arabic: maltodextrin: modified

starch (4/6, 1/6, 1/6) blend proved to be more efficient than the other blends even better than 100% gum arabic.

Kanakdande *et al.* (2006) microencapsulated cumin oleoresin by spray drying using gum arabic, maltodextrin, and modified starch. The study showed that the stability of the character impact constituents present in oleoresin decreased as the quantum of gum arabic decreased in its blend with maltodextrin and modified starch. Gum arabic/ maltodextrin/ modified starch (4/6:1/6:1/6) blend proved to be more efficient than the other blends, and even better than gum arabic itself.

Ersus and Yurdagel (2007) conducted an experiment on microencapsulation of anthocyanin pigment of black carrot by spray drying with a core to maltodextrin ratio of 1:3. It was revealed that storage at 4ºC increased the half-life of spray dried anthocyanin pigments three times when compared to 25ºC storage temperature.

2.4.3 Operating Conditions of Spray Drier

In order to obtain a high microencapsulation efficiency, optimal spray-drying conditions must be used even though suitable wall materials are employed. The main factors in spray-drying that must be optimized are feed temperature, air inlet temperature, and air outlet temperature. The air inlet temperature is usually determined by two factors: the temperature which can safely be used without damaging the product or creating operating hazards and the comparative cost of heat sources (Fogler and Kleinschmidt, 1938).

When the air inlet temperature is low, the low evaporation rate causes the formation of microcapsules with high density membranes, high water content, poor fluidity, and easiness of agglomeration. High air inlet temperature causes an excessive evaporation and results in cracks in the membrane inducing subsequent premature release and a degradation of encapsulated ingredient or also a loss of volatiles (Zakarian and King, 1982).

Zbicinski *et al.* (2002) reported that the rate of feed delivered to the atomizer is adjusted to ensure that each sprayed droplet reaches the desired drying level before it comes in contact with the surface of the drying chamber. Moreover, appropriate adjustment of the air inlet temperature and flow rate is important.

Patel *et al.* (2009) described the critical elements of a spray drying process which are the inlet air temperature, outlet air temperature, viscosity of the feed, solid content of the feed, surface tension of the feed, feed temperature, volatility of the solvent, and nozzle type.

The effects of spray drying conditions of the Gac fruit aril powder with inlet drying air temperature (120, 140, 160, 180 and 200^0C) and maltodextrin addition (10%, 20% and 30%) were analysed by Kha *et al.* (2010). Moisture content, bulk density, colour characteristics, total carotenoid content and encapsulation efficiency were significantly affected by maltodextrin concentration and the inlet air temperatures.

Haidong *et al.* (2012) microencapsulated ginkgo leaf extracts with gum arabic, maltodextrin and soybean protein as wall materials. The result indicated that, for the microcapsules, the encapsulation efficiency of 81.3% was achieved when air inlet temperature was 181^0C.

Solval *et al.* (2012) conducted a study on development of cantaloupe juice powders using spray drying technology. Cantaloupe fruit juice with 10% maltodextrin (MD) added was spray dried at inlet temperatures of 170, 180 and 190^0C. The juice powder produced at 170^0C had higher moisture content and higher vitamin C and b carotene than those produced at 180 and 190^0C. This indicated that the inlet air temperature of the spray dryer can affect vitamin C and β-carotene contents of cantaloupe juice powders.

The physical, chemical, and powder properties, except for the pH value and colour of the powders of the spray dried sumac extract powders, were significantly affected by both the inlet/outlet temperature. As a result, the extracts with 10% TSS and $160/80^0$C inlet/outlet temperature were rated as best conditions for sumac extract powder (Caliskan and NurDirim, 2013).

According to Hasheminya and Dehghannya (2013) the factors influencing the performance of spray dryers include temperature, moisture content, inlet air flow rate, the amount of solid materials, viscosity and surface tension of the processed material, type of atomizer and its related parameters such as velocity and diameter of the atomizer.

Mishra *et al.* (2014) conducted a study on the effect of maltodextrin concentration and inlet temperature during spray drying on physicochemical and antioxidant properties of amla juice powder. Maltodextrin concentration (5-9%) and drying temperature (125-200^{0}C) significantly affected moisture content, bulk density, hygroscopicity, color attributes. Amla juice powder dried at 175^{0}C and 7% maltodextrin was adequately effective to produce powder with less hygroscopicity, acceptable color in terms of L, a and b values and potent free radical scavenging activity.

2.5 Characteristics of Microencapsulated Powder

2.5.1 Moisture Content, Bulk Density and Particle Size

Papadakis and King (1988) conducted a study on air temperature and humidity profiles in spray drying. As the air temperature increased, air took more moisture from the spray thereby resulting in lower moisture content in the powder. The results concluded that the spray drier temperature largely controlled the moisture content of the final powder.

The operating temperature could influence the particle size of the microencapsulated powder. The high inlet temperature and low temperature difference between inlet and exit air led to very fast drying and also produced slightly larger particles than slow drying (Masters, 1991).

Cai and Corke (2000) conducted an experiment on microencapsulation of amaranthus pigments with maltodextrin and modified starches as coating materials with inlet air temperature from 150 to 210ºC with constant air flow rate. Higher drying air temperature resulted in faster drying and higher powder productivity. The bulk density of pigment powders decreased with the increase of

spray drying air temperature. The lower the bulk density, the more occluded was air within the powders, and therefore, there was a greater possibility for oxidative degradation of the pigments and reduced storage stability.

According to Keogh *et al.* (2003), the higher the particle median diameter, the higher is the interstitial air content between particles and, consequently, the higher is the volume occupied. This can also explain the higher bulk density of the sample produced with tapioca starch, with respect to the others.

Soottitantawat *et al*. (2003) conducted a study on the influence of emulsion size on the retention of volatile compounds of spray dried microcapsules. It was concluded that the effect of the emulsion size affected to the stability of encapsulated flavour powder. The retention is also affected by the droplet size.

Beltra *et al.* (2005) conducted a study on morphological changes of particles during spray drying. Particles change in size and shape during drying are related to moisture content of the material and operating drying temperatures. At low temperatures, final product showed the smallest size (12 μm) and intermediate and high-temperature drying, particles with mean diameters of 32 and 37 μm. Formation of thick, compact and irregular crust, was more evident for low-temperature drying (110/70^{0}C) than when drying at higher temperatures (170/145 and 200/173^{0}C) in which smooth and regular surfaces of complete and broken material were observed.

Soottitantawat *et al*. (2005) conducted a study on microencapsulation of d-limonene by spray drying with respect to the effects of emulsion droplet size, powder particle size, as well as to the effects of various kinds of matrices (gum arabic, maltodextrin, and modified starch) on its stability. The distributions of emulsion size in the powder showed an increase in the fraction of large emulsion droplets and changed to a bimodal distribution. The modified starch showed a higher stability of encapsulated d-limonene than the others. The release and the oxidation decreased deeply with an increase in powder and emulsion particle size for gum arabic and maltodextrin materials.

Erus and Yurdagel (2007) microencapsulated anthocyanin from black carrots by spray drier with maltodextrin as a carrier and coating agent. It was reported that increasing spray drying temperature reduced the moisture content of powders. Moisture content ranged from 1.09 to 3.76% for powders where the flow rates were kept constant at 5 ml/ min for the temperature ranges of 160 to 200ºC. They also reported that the size of the microencapsulated pigment powder ranged from 3 to 20 µm (by using Scanning Electron Microscope).

Goula and Adamopoulos (2010) developed a new technique for spray drying concentrated orange juice using dehumidified air as drying medium and maltodextrin as drying agent. Concentrated orange juice was spray dried at inlet air temperatures of 110, 120, 130, and 140°C and (concentrated orange juice solids) / (maltodextrin solids) ratios of 4, 2, 1, and 0.25. It was reported that bulk density increased with a decrease in inlet air temperature and maltodextrin concentration. Moisture content decreased with an increase in inlet air temperature and a decrease in maltodextrin concentration and dextrose equivalent.

Fazaeli *et al.* (2012) conducted a study on Effect of spray drying conditions and feed composition on the physical properties of black mulberry juice powder. It was revealed that inlet air temperature negatively influenced the bulk density due to the increase of powder's porosity. The solubility of the powder increased with decrease in bulk density.

2.5.2 Microencapsulation Efficiency

Buma (1971) conducted a study on particle structure of spray dried caseinate and lactose by scanning electron microscopy. He observed that microencapsulation efficiency values reflect the amount of core recovered from powder particles during the solvent extraction process, which is composed of both surface and internal fat entrapped within the wall matrix.

Microencapsulation efficiency can be increased by increasing wall solution solids concentration which can be related to the effect of wall solids concentration on the formation of surface core prior to the formation of crust around the drying droplets (Young *et al*., 1993).

Hogan *et al.* (2001) conducted a study on microencapsulation properties soy oil powder with whey protein concentrate as wall material. It was found that increasing oil/protein ratio from 0.25 to 2.0 resulted in a significant decrease in microencapsulation efficiency from 59.0% to 40.0%.

The microencapsulation efficiency is dependent on surface oil and the presence of surface oil affects the physical properties of powder such as flowability, bulk density, dispersibility and induces rapid lipid oxidation (Keogh *et al*., 2001).

Huezo *et al*. (2004) conducted an experiment on microencapsulation of red chilly pigment emulsion with 25% and 35% solid content with spray drying. Emulsions at 35% solid content showed the best morphology and highest microencapsulation efficiency and storage stability.

Shu *et al.* (2005) microencapsulated lycopene by spray drying method using a wall system consisting of gelatin and sucrose. Encapsulation efficiency was significantly affected by the ratio of core and wall materials, ratio of gelatin and sucrose, homogenization pressure, inlet temperature, feed temperature, and lycopene purity.

Carneiro *et al*. (2013) evaluated the potential of maltodextrin combination with different wall materials in the microencapsulation of flaxseed oil by spray drying. The study showed that encapsulation efficiency values varied from 62.3% to 95.7%.

2.5.3 Stability of the Microencapsulated Powders

Reineccius *et al.* (2002) evaluated the incorporation and retention on storage of a variety of flavour compounds spray-dried in α, β, and γ cyclodextrins. Cyclodextrin/flavour complexes were stored at 20 or 40°C at 65 or 80% relative humidity, and losses during both the inclusion process and the subsequent storage period were monitored analytically. γ-cyclodextrin generally functioned best in terms of initial flavour retention. On storage, losses of volatiles were greatest for γ-cyclodextrin and least in the case of α-cyclodextrin. The results suggested that

cyclodextrin encapsulation via spray-drying involved matrix entrapment as well as molecular inclusion.

Barbosa *et al*. (2005) conducted a study on the stability of microencapsulated bixin with maltodextrin and mixture of maltodextrin with sucrose. The bixin stability was observed to be 10 times greater for both wall materials when compared to the non-encapsulated bixin.

Krishnan *et al.* (2005) conducted a study on the microencapsulation of cardamom oleoresin by spray drying using binary and ternary blends of gum arabic, maltodextrin, and modified starch as wall materials. A 4/6, 1/6, 1/6 blend of gum arabic: maltodextrin: modified starch offered a better stability than gum arabic alone.

Soottitantawat *et al.* (2005) analyzed the effect of dispersed particle size, powder particle size and various kinds of matrices (gum arabic, maltodextrin, and modified starch) on the stability of microencapsulated L-menthol by spray drying during its storage period. The rate of oxidation decreased deeply with an increase in powder stability and dispersed particle size irrespective of the wall material used.

Sheikh *et al.* (2006) studied microencapsulation of black pepper oleoresin by spray drying, using gum arabic and modified starch as wall materials. Gum arabic showed a greater protection to the pepper oleoresin than modified starch.

Erus and Yurdagel (2007) conducted an experiment on microencapsulation of anthocyanin pigment of carrot by spray drying using maltodextrin as wall material. They reported that maltodextrin had low flavour and were non-reactive with the core component (anthocyanin) used for microencapsulation. It improved the shelf life of black carrot anthocyanins and maintained the colour and stability during storage. The anthocyanin powder had more stability with anthocyanin content retaining up to 64 days at 25°C.

2.5.4 Microstructural Characteristics of Microencapsulated Powder

Rosenberg *et al*. (1985) conducted a scanning electron microscopy study of microencapsulation. They reported that the presence of surface dents adversely affects powder flowability and reconstitution properties.

Rosenberg *et al.* (1990) investigated the inner structure of microcapsules of ethyl caprylate produced by spray drying using scanning electron microscope and has shown that the encapsulated core material was dispersed in the thick walls of the capsules.

Rosenberg and Lee (1993) reported that microstructure of spray dried capsules was affected by wall composition and properties, flavour to wall ratio, atomization and drying parameters, uneven shrinkage at early stages of drying, surface tension-driven viscous flow and storage conditions.

Sankarikutty *et al*. (1988) conducted studies on microencapsulation of cardamom oil by spray drying technique and reported that spray dried cardamom oil microcapsules with wall material consisting of polysaccharides exhibits notable surface indentation.

Scanning Electron Microscope images of microencapsulated orange oil powder using maltodextrin showed relatively spherical and smooth particles (Kim and Morr, 1996). Formation of particles with dented surface might be due to rapid shrinking of the liquid droplets during early stages of the drying process.

Lee and Rosenberg (2000) prepared whey protein-based microcapsules by double emulsification and heat. It showed that outer topography and inner structure of the microcapsules were affected by pH of the core-in-wall emulsion. Microcapsules prepared from pH 7.2 core-in-wall emulsions exhibited smooth and dent free surfaces.

2.6 PACKAGING AND STORAGE OF MICROENCAPSULATED POWDER

Desorby *et al.* (1997) microencapsulated beta carotene in maltodextrin by spray drying and stored in petridishes at 25°C. There were slight changes in

colour values during storage due to oxidation. Beta carotene content reduced to 11% compared to initial content (80%) when stored at 25°C.

Pineapple powder was prepared using foam mat drying process and the dehydrated product was packaged in 300 gauge high-density polyethylene (HDPE) packages and aluminium foil and the samples were kept at room temperature. The dried product was shelf stable for six months and the foam mat dried pineapple powder was better in colour, flavour, flowability, solubility and overall acceptability than the spray dried powder (Hassan and Ahmed, 1998)

Kanakdande *et al*. (2006) conducted experiments on microencapsulation of cumin oleoresin powder and packed in polyethylene pouches for storage studies. Flavour components were found to be safe against oxidation as well as volatilization for six weeks.

CHAPTER III
MATERIALS AND METHODS

This chapter describes the materials used in the study, the experimental methods and instrumentation used for achieving the outlined objectives. Also outlined are the process of production of microencapsulated vanilla extract powder, the experimental plan and procedure for optimisation of the process parameters in terms of emulsion characteristics and powder quality characteristics.

3.1 RAW MATERIAL

3.1.1 Core Material

In this study vanilla extract was used as the core material for encapsulation which was procured from M/s.Silver Wings Agencies, Wayanad. The vanilla extract was well packed in an air tight aluminium container and kept out of sunlight and stored at room temperature (Plate 3.1).

3.1.2 Wall Material (Carrier)

Maltodextrin (DE 20) (MD) and maize starch (MS) were used as wall materials for encapsulation. These were procured from M/s. Viveka agencies, Coimbatore. All the other chemicals and reagents used in this study were of analytical grade and were procured from M/s. Chemind, Thrissur.

3.2 PHYSICO-CHEMICAL PROPERTIES

3.2.1 Physico-Chemical Properties of Vanilla Extract

3.2.1.1 Colour

The colour of the vanilla extract was found using 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 consists of measurement (sample) port, opaque cover and display unit. This colour flex meter works on the principle of focusing the light and measuring energy reflected from the sample across the entire visible spectrum. For matching a series of colour across the visible spectrum, primary

lights are required and describes the colour by mathematical model called as Hunter model. It reads the colour of sample in terms of L, a and b values where, luminance (L) forms the vertical axis, which indicates whiteness to darkness. Chromatic portion of the solids is defined by: a (+) redness, a (-) greenness, b (+) yellowness, and b (-) blueness.

A transparent glass cup filled with sample was 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. Colour was calibrated by fixing the definite colours like white and black tiles. After calibration, the sample was placed over the port and values of 'L', 'a' and 'b' were recorded.

3.2.1.2 pH Measurement

The pH of the vanilla extract was determined using a digital pH meter (M/s. Systronics; Model MK VI) (Plate 3.3). The pH meter was standardized with double distilled water of pH 7.0 and standards of pH 4.0, 7.0 and 9.0 before finding the pH of the extract.

3.2.1.3 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 vanilla extract was measured by hydrometer (Plate 3.4). The hydrometer was placed in a jar, three forth filled with vanilla extract taking care that it does not touch the sides and bottom of the jar. The hydrometer should not touch the bottom side of the jar. The specific gravity was measured by taking the reading on the hydrometer which coincides with the liquid surface of the liquid.

3.2.1.4 Refractive Index

The refractive index of vanilla extract was determined by using a hand refractometer (M/s. Erma, Japan) with a range of 0-65° Brix (Plate 3.5). One or two drops of the vanilla extract were placed on the hand refractometer and the readings corresponding to the cross mark was noted and expressed as degree Brix (Ranganna, 1991).

Plate 3.1 Vanilla extract

Plate 3.2 Hunter Lab colourimeter

Plate 3.3 pH meter

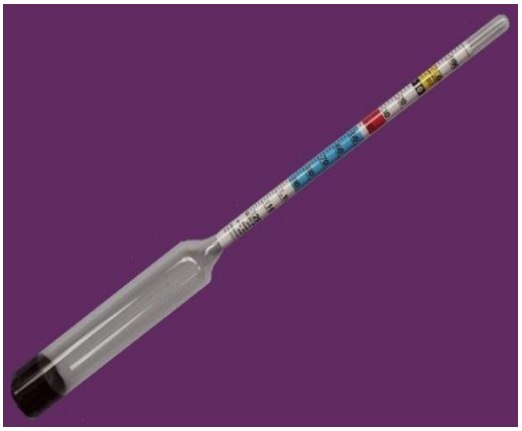


Plate 3.4 Hydrometer

Plate 3.5 Hand refractometer

Plate 3.6 Water activity meter

3.2.1.5 Water Activity

The water activity of vanilla extract emulsion was carried out using Aqua lab water activity meter (M/s. Aqua Lab, U.S.A; model: Series 3TE) (Plate 3.6). For determining the water activity, the vanilla extract was filled in the disposable cups of the water activity meter (cup with over-filled sample may contaminate the chamber‘s sensors) and the sample drawer knob is turned to OPEN position. After opening the drawer, the disposable cup with vanilla extract was then placed in the drawer and closed. The sample drawer knob was then turned to the READ position and the water activity of vanilla extract was noted from the LCD display of the water activity meter.

3.2.1.6 Viscosity

The viscosity of the prepared emulsion was determined using a rotational viscometer (Brook field DV-E Viscometer, USA) (Plate 3.8) as per the experimental procedure explained in section 3.9.1.

3.2.2 Physico-chemical Properties of Wall Materials

3.2.2.1 Moisture Content

The moisture content of maize starch and maltodextrin (DE 20) was determined as per the experimental procedure explained in section 3.10.1.

3.2.2.2 Water Activity

The water activity of maltodextrin and maize starch was determined using Aqua Lab water activity meter (M/s. Aqua Lab, USA; model: Series 3TE) (Plate 3.6) as explained in section 3.2.1.5.

3.2.2.3 Bulk Density

Bulk density of the maltodextrin and maize starch was determined by tapping method (Bhandari *et al.* 1992). The bulk density of the wall materials were determined as explained in section 3.10.2.

3.2.2.4 Solubility

The solubility of maltodextrin and maize starch were carried out based on the method proposed by Loksuwan (2007) as explained in section 3.10.4.

3.2.2.5 pH Measurement

The pH of maltodextrin and maize starch were determined using a digital pH meter (M/s. Systronics; Model MK VI) (Plate 3.3). Before determining the pH of the maltodextrin and maize starch, the pH meter was standardized with double distilled water of pH 7.0 and standards of pH 4.0, 7.0 and 9.0. The pH was determined for 20% solid content solution of wall materials.

3.2.2.6 Colour

The colour of maltodextrin and maize starch were measured using Hunter lab colour flex meter (Hunter Association laboratory, Inc., Reston, Virgina, USA) (Plate 3.2). Colour of the sample was obtained by measuring 'L', 'a' and 'b' colour values. Maltodextrin and maize starch were placed over the colour measuring port of the flex meter and 'L', 'a', 'b' colour values were recorded.

3.3 EXPERIMENTAL DESIGN

Based on a thorough review of literature and the preliminary studies conducted, the process parameters which would influence the product quality characteristics, utilization potential and storage stability were chosen as independent variables. The product quality characteristics which are characteristics of these parameters were selected as dependent variables.

3.3.1 Independent Variables

Wall material concentration (maltodextrin: maize starch) (w/w):

(a) D1: 100% maltodextrin

(b) D 2: 75% maltodextrin and 25% maize starch

(c) D3: 25% maltodextrin and 75% maize starch

(d) D4: 100% maize starch

Flavour load (core concentration) (percent w/w):

(a) C1: 10%

(b) C2: 20%

(c) C3: 30%

Spray drier inlet air temperature:

(a) 170ºC

(b) 180ºC

(c) 190ºC

3.3.2 Dependent Variables

Emulsion characteristics:

(a) Emulsion viscosity

(b) Emulsion stability

(c) Emulsion droplet size

Product characteristics:

(a) Moisture content

(b) Bulk density

(c) Powder wettability

(d) Cold water solubility

(e) Colour

(f) Encapsulation efficiency

3.3.3 Characteristics of Optimally Produced Encapsulated Vanilla Extract Powder

(a) Micro structural characteristics

i) Morphology ii) Inner structure

(b) Active component (vanillin) retention

(c) Microbial quality

3.4 STATISTICAL ANALYSIS

The effect on different parameters of microencapsulation of vanilla extract powder with respect to flavour load, wall material concentration and inlet air temperature were statistically analysed as CRD. Post Hoc test (DMRT) was performed to identify the subgroups of treatments. Wherever the number of subgroups exceeded beyond five, the subgroups were collapsed on a logical basis. The statistical analysis of data was carried out using SPSS software (Version 16.0; SPSS Inc. Chicago).

3.5 ENCAPSULATION OF VANILLA EXTRACT

Encapsulation of vanilla extract was carried out using spray drying process. There are two stages in this process. The first stage is the formation of stable emulsion of the core material (vanilla extract) in a solution of wall material with which it is immiscible with the addition of an emulsifier. The second stage is the atomisation of the obtained emulsion into spray drying chamber and drying. The flow chart of the process is shown in Fig 3.1.

3.6 PREPARATION OF EMULSION

Two hundred and fifty gram of wall material in the required proportion as mentioned in section 3.3.1 are dissolved in 300 ml of distilled water by continuously stirring it and after complete dispersion, the final volume was made up to 500 ml by adding distilled water. The resultant 50% solid wall material solutions were filtered using muslin cloth to remove the foreign materials. The resultant solutions were fortified with 25, 50 and 75 g of vanilla extract to obtain a flavour load (core concentration) of 10, 20 and 30% (w/w) of the wall solids respectively (Fernandes *et al*., 2008). Two drops of Tween-20 was added to enhance the emulsifying and film forming properties (Krishnan *et al*., 2005). The mixer was then emulsified in a high-speed mixer until the vanilla extract was dispersed completely.

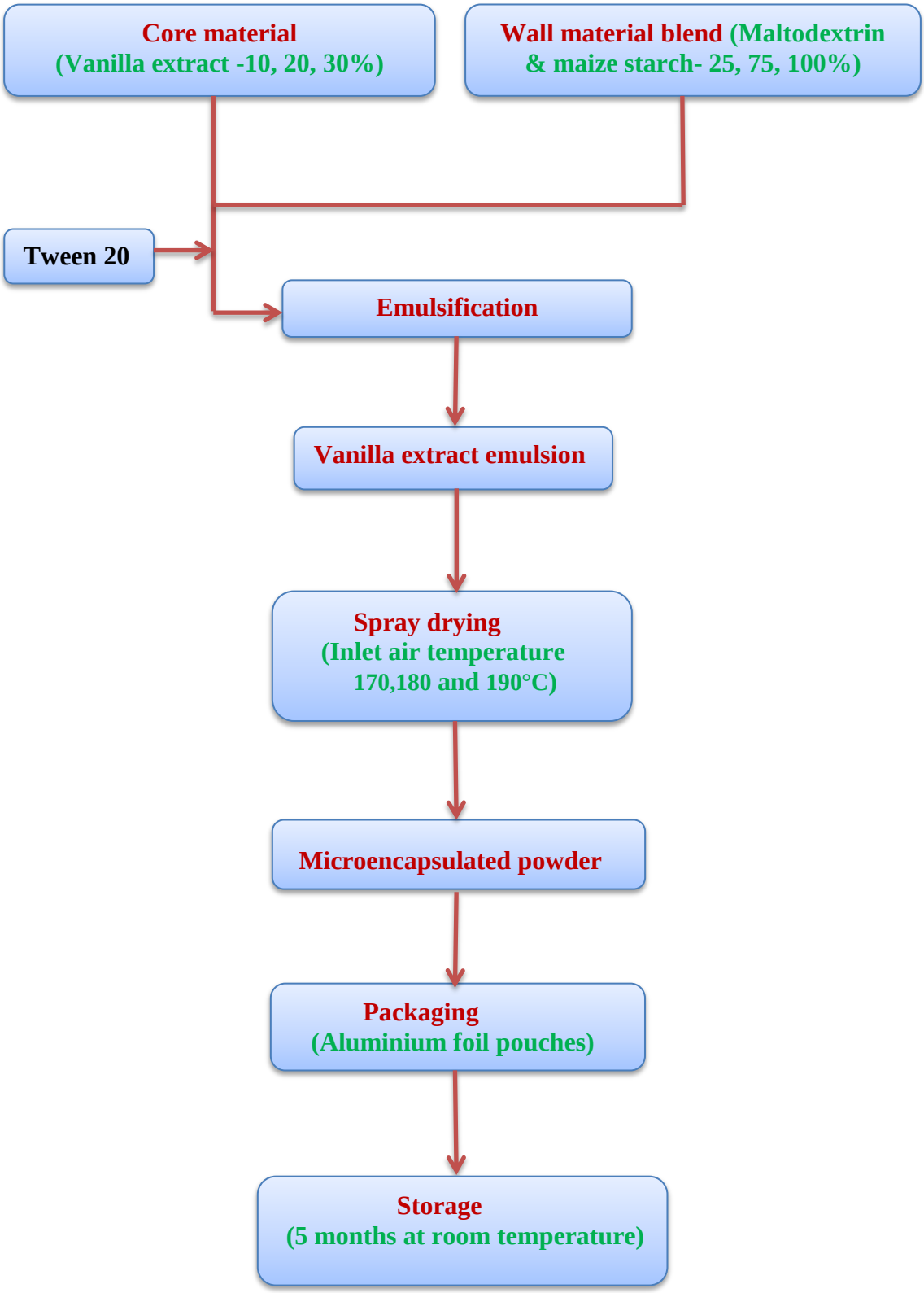


Fig.3.1 Process flow chart of spray drying encapsulation of vanilla extract

3.7 SPRAY DRYING TECHNOLOGY

Spray drying process widely used in food industry for producing dried powdered products could be effectively used for microencapsulation. Spray drying technique for microencapsulation is advantageous in terms of producing free flowing powders with a controlled particle size range at a fast drying rate at comparatively economical rates enabling scale up of the process for industrial production once the process parameters are standardised.

In this research work, a tall type spray drier with two fluid nozzle having a water evaporation capacity of 1 l/h (M/s S.M. Scientech, Kolkata) (Plate 3.7) was used for the production of microencapsulation of vanilla extract powder. The major components of the spray drier are: air supply system, feed supply system, atomizer, drying chamber, powder recovery system and control panel.

Plate 3.7 Tall type spray drier

3.7.1 Hot Air Supply System

Air supply system consists of compressor, air filter and air heater. The air is compressed by a compressor and this compressed air is introduced into twin fluid pressure nozzle atomizer after passing through an air filter and heater. The compressed air disintegrates the feed emulsion into a fine mist. An air filter is

essential to cease the entry of microorganism. The air is heated through electric heating coils to get a maximum temperature upto 350^0C.

3.7.2 Feed Supply System

The feed supply system consists of a peristaltic pump and a feed source. A five hundred millilitre beaker with emulsion was considered as a feed source, the feed which is pumped into the atomizer at the top of the spray drier by a peristaltic pump consisting of five rollers, which squeeze the hypalon natural rubber tube (6 mm diameter) against the walls of the pump, thereby the feed solution inside the tube was pumped forward in the pumping direction due to the vacuum created sucking the feed solution from the beaker. The motorised peristaltic pump has variable speed arrangement to control the flow. The motor is DC operated and its rpm is controlled by a rotary knob.

3.7.3 Atomizer

The feed solution was introduced into the main chamber in the form of fine spray by means of a two fluid nozzle from the ceiling of main chamber in downward direction. Compressed air was also introduced into the two fluid nozzle. The kinetic energy of compressed air is utilized to disperse the feed solution in the form of a fine mist. Two fluid nozzle atomizer has ability to produce a wide range of flow rate and droplet size. It produces a cone shaped spray pattern. The pressure of the compressed air for the flow of the spray was around 2 kg/cm^2. The feed coming from the peristaltic pump was brought into contact with the heated air after atomization for the evaporation of moisture to take place uniformly from the surface of all droplets within the drying chamber.

3.7.4 Drying Chamber

The drying chamber of the spray drier is made up of SS304 stainless steel and is cylindrical in shape and the bottom portion is conical for easy flow of dried powder. The atomizer is placed at the top most portion of the drying chamber. Two inspection window glasses are provided at two sides of wall, of which one with 100 W light to see the operation 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.

3.7.5 Powder Recovery System

Fine powder particles from the drying chamber were carried along with the hot air and enter into the cyclone separator where they are separated. Air along with particles swirl in a spiral direction down the cyclone and, due to density difference, air leaves the cyclone through a duct pipe since it is less/denser than the particles. The fine powder separated from the cyclone separator is collected in glass bottle attached at the bottom of the cyclone through threaded flange with a teflon gasket.

3.7.6 Control Panel

The blower speed, feed rate and inlet and outlet temperature were controlled through an electrical control panel with appropriate regulators, ON/OFF push buttons and indicators. In addition to this an automatic and manual de-blocking knob is also connected to solve the clogging of atomizer.

3.8 PREPARATION OF MICROENCAPSULATED POWDER

The prepared emulsion was pumped into the drying chamber of the spray drier through twin fluid pressure nozzle. The feed pump was adjusted to 12 rpm and the air pressure for the twin fluid pressure nozzle was adjusted to 2 kg/cm^2. The inlet air temperature was varied from 170^0C to 190^0C as per the technical programme and the blower speed was adjusted to 1500 rpm. The hot air and the fine mist of emulsion solution will mix intimately in the drying chamber resulting in the heat and mass transfer which will produce the microencapsulated product with the wall material enclosing the active ingredients thus forming microcapsule like particles. The microencapsulated vanilla extract powder was then collected from glass bottles of both drying chamber and cyclone separator which are then mixed thoroughly and packed in aluminium foil pouches, sealed air tight using a hand sealer and stored at room temperature for further analysis.

3.9 QUALITY CHARACTERISTICS OF EMULSION

The quality characteristics of the vanilla extract emulsion prepared with various combination of wall material and flavour load as per the technical program explained in section 3.3.1 such as emulsion viscosity, emulsion stability, and emulsion particle size were then determined.

3.9.1 Emulsion Viscosity

The viscosity of the prepared emulsion was determined using a rotational viscometer (Brook field DV-E Viscometer, USA) (Plate 3.8). The instrument was first calibrated by an auto test without spindle from 0 to 100 rpm. The principle of the viscometer is based on the torque necessary to overcome the resistance offered by viscosity of emulsion during the rotation of the cylinder or a submerged disc in the emulsion. Five hundred millilitres of emulsion was taken in a beaker. After preliminary tests, the spindle suitable for viscosity of emulsion under study was fitted to the viscometer and was run at 20 rpm with spindle submerged in the emulsion not touching the beaker walls. After attaining a steady state, viscosity values were recorded.

3.9.2 Emulsion Stability

The stability of the emulsion prepared as explained in section 3.6 were found as detailed by Beristain *et al*. (2002). Hundred millilitres of emulsion was taken in a 250 ml measuring cylinder and stored without disturbance at ambient temperature (28 ± 2^0C). During this storage period, vanilla extract moved to the top surface and wall material forming clear distinct bottom layer of separation. The time taken for formation of distinct layers of wall material and vanilla extract is recorded as stability.

3.9.3 Emulsion Particle Size

Emulsion particle size was determined as described by Soottitantawat *et al*. (2003). Emulsion smears of about 1 mm thick were made on glass slides after complete emulsification and it was shielded with a glass cover. This slide was kept under light microscope (Leica MZ 16 Stereo Microscope, Germany) (Plate

3.8). The emulsion image was analysed using MATLAB image processing software for the determination of number median diameter of the emulsion particle.

Plate 3.8 Rotational viscometer

Plate3.9 Stereo microscope

3.10 QUALITY ANALYSIS OF MICROENCAPSULATED POWDER

3.10.1 Moisture Content

Moisture content of encapsulated vanilla extract powder was determined by the method AOAC (1999). Samples of 10 g of encapsulated powder were dried for 16 h in an electric oven at 70^{o}C, cooled in a desiccator and weighed. The process was repeated until the differences between the two consecutive weights were not more than 0.5-1 mg. The observed moisture contents of the powder were recorded. The moisture content was calculated using the formula

$$\text{Moisture content (M), \% (w.b.)} = \frac{W_w}{W_w + W_d} \times 100 \qquad \ldots\ldots (3.1)$$

where,

M = moisture content, % (wet basis)

W_w = weight of water evaporated, g

W_d = weight of dry matter, g

3.10.2 Bulk Density

Bulk density of the encapsulated vanilla extract powder was determined by tapping method (Bhandari *et al.* 1992). Two grams of microencapsulated vanilla powder was loosely weighed into a 10 ml graduated cylinder. The cylinder containing the powder was tapped on a flat surface to a constant volume. The final volume of the vanilla powder was recorded and the bulk density was calculated by dividing the sample weight by volume.

$$\text{Bulk density } (g/cm^3) = \frac{\text{Weight of the microencapsulated vanilla powder}}{\text{Volume of the sample}} \quad \text{.....(3.2)}$$

3.10.3 Wettability

Wettability is the time taken for the complete wetting of the powder. It was determined by taking 1.5 g of encapsulated vanilla extract powder which was then gently placed on the surface of 100 ml water at 30°C. The time for the powder to get completely wet was recorded (A/S Niro Atomizer *et al*., 1978).

3.10.4 Cold Water Solubility

The method proposed by Loksuwan (2007) was used to analyze cold water solubility of spray dried encapsulated powder. One gram of microencapsulated vanilla powder was mixed with 100 ml of water at room temperature for 30 min. A 10 ml aliquot of the supernatant solution was transferred to a 15 ml centrifuge tube and centrifuged for 15 min. The aliquot of the supernatant was then taken in a pre-weighed aluminum moisture dish, evaporated on a steam bath and dried in an oven at 110°C overnight. The cold water solubility was calculated as:

$$\text{Cold water solubility, \%} = \frac{10 \times \text{Grams of solid in supernatent}}{\text{Grams of sample}} \times 100 \quad \text{.....(3.3)}$$

3.10.5 Colour

The colour of the vanilla powder was measured using Hunter lab colour flex meter (Hunter Association laboratory, Inc., Reston, Virgina, USA) (Plate 3.2). Colour of the sample was measured by measuring 'L', 'a' and 'b' values.

The encapsulated vanilla powder samples were placed over the colour measuring port of the flex meter and 'L', 'a', 'b' values were recorded.

3.10.6 Encapsulation Efficiency

Total extract content of the powder sample was estimated by standard ASTA method (ASTA, 1968). About 10 g of encapsulated powder was taken in a glass column and about 50 ml of solvent acetone was added. The sample in the column was kept undisturbed overnight. The extract was drained into a pre-weighed beaker and the solvent was evaporated to dryness in a water bath. The difference in the weight of the beaker, gave the total amount of extract present in the sample.

Surface (non-encapsulated) extract content was determined by a modified method as suggested by Varavinit *et al*. (2001). Hexane (50 ml) was added to an accurately weighed quantity (5 g) of encapsulated powder followed by stirring for 2 min. The suspension was then filtered and the residue rinsed twice by passing 20 ml of hexane through each time. The residual powder was then air dried for 30 min. and weighed. The amount of surface extract content was calculated by the difference in weight of the encapsulated powder, before and after washing. The encapsulation efficiency was calculated using following equation.

$$\text{Encapsulation efficiency}, \% = \frac{\text{Total amount of extract - surface extract conent}}{\text{Total extract}} \times 100 \quad \ldots\ldots\ldots (3.4)$$

3.11 CHARACTERISATION OF OPTIMALLY PRODUCED ENCAPSULATED POWDER

3.11.1 Microstructural Analysis

Scanning electron microscopy (SEM) was used to investigate the microstructural characteristics including both morphology and inner structure of the encapsulated vanilla extract powder. In this study SEM was carried out in NIIST, Trivandrum. The scanning electron microscope determines the particle size of a powder by using a beam of high energy electrons and electromagnet. The signals that derive from electron-sample interactions reveal information about the

sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. SEM can produce a largely magnified image by using electrons.

Scanning Electron Microscopy analysis of the samples was carried out using a JSM-6400 scanning electron microscope (JEOL, Tokyo, Japan) (Plate 3.10). Prior to examination, the samples were uniformly spread on a sample holding stub made of aluminium. A carbon tape was stuck to the sample holding side of the stub and then a thin uniform layer of samples were formed.

3.11.1.1 Outer Morphology

The morphology (outer structure) of the encapsulated vanilla extract powder was determined as per the procedure suggested by Rosenberg and Young (1993). Microencapsulated powder was mounted directly to the specimen stub using a double adhesive tape. These samples being non-conductive were sputter coated with gold to render them electrically conductive by using HUMMLE VII Sputter Coating Device (Anatech Electronics, Garfield, N.J., USA). The sputter coated samples were examined at a SEM voltage of 15 kV. The micrographs were taken at a magnification of 500X.

3.11.1.2 Inner Structure

Inner structure of the encapsulated vanilla extract powder was determined as explained by Rosenberg and Young (1993). Encapsulated powders (attached to the stub) were fractured by attaching a second piece of adhesive tape on the top of the powder and then by quick ripping, the weak hollow particles get cut. In all cases, the specimen was subsequently coated with gold. The micrographs were taken at magnification of 500X.

3.11.2 Active Component Retention

The main active component (vanillin) of the optimally produced vanilla extract powder was determined using Gas chromatography (Shimadzu GC-17A, Japan) (Plate 3.11). The procedure for finding the vanillin content of encapsulated vanilla extract powder using gas chromatograph method was adopted from Jager

et al. (2007). Vanillin (99%) standard was obtained from M/s.Sigma, St. Louis, MO, USA.

The standard solution for gas chromatograph was made by accurately weighing 0.05 g (±0.0025 g) of the standard and dissolving it into 50 ml of ethanol in a volumetric flask. The sample prepared by dissolving 0.1 g of optimally produced microencapsulated vanilla extract powder into a 50 ml of ethanol in volumetric flask. The standard solution was first injected and the chromatograph of the standard was obtained. Then the sample solution was injected and its chromatograph was recorded following the same procedure. The injection were made with an initial split ratio of 10:1 followed by a 100:1 split after 2.5 min with the injection port temperature of 250^0C. The initial temperature was set to 100^0C with an initial 1.0 min hold followed by programmed temperature (increment) of 10^0C/min to 150^0 C and hold of 1.0 min followed by a final temperature ramp of 40^0 C/min to 250^0 C. The chromatographs were then analysed for vanillin content.

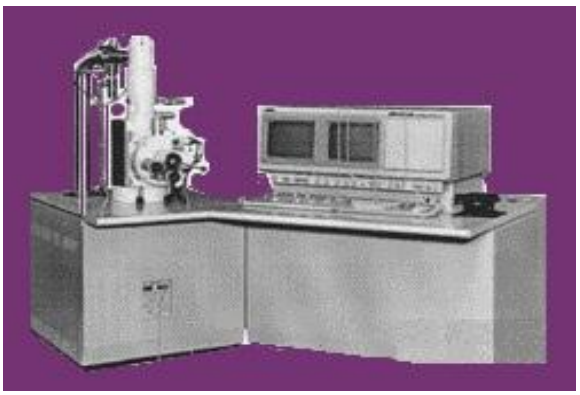


Plate 3.10 Scanning electron microscope

Plate 3.11 Gas chromatograph

3.11.3 Microbial Load of Encapsulated Powder

The quality of optimally produced encapsulated vanilla extract powder was obtained by the numbers and kind of microorganisms present, which were found by serial dilution and plating method for the differential enumeration of bacteria and fungi.

Nutrient agar media was used for the enumeration of bacteria and Martin's Rose Bengal Agar Medium was used for the enumeration of fungi. The agar and Martin's rose Bengal medium, glasswares needed for the plate count method were sterilized by autoclaving at 121°C for 15 min. One gram of the microencapsulated vanilla extract powder was taken and added to 100 ml of sterile water blank. Emulsion was shaken well for 10 to 15 min to obtain homogenized suspension of microorganisms and this gave a dilution of 1:100 (10^{-2}). One ml from (10^{-2}) this dilution was transferred to 9 ml of sterile water blank with a sterile one ml pipette, which gave a dilution of 10^{-3}. The process was repeated up to 10^{-6} dilutions with the sterile water blank. Sterile 1 ml aliquots from 10^{-3} and 10^{-6} dilutions were transferred to the sterile petridishes with Nutrient agar and Martin's Rose Bengal agar medium for the enumeration of bacteria and fungi respectively. The experiments were carried out in triplicate for greater accuracy. Approximately, 15-20 ml of molten and cooled medium (45ºC) for the respective organisms were added to each petri dish and the plates were rotated in clockwise and anticlockwise directions to have uniform dispersion of colonies. The plates were then incubated at room temperature for 24- 48 h for bacteria and three days for fungi. After the incubation period, the colonies were counted and the number of organisms (bacteria and fungi) per gram of sample was calculated by applying the formula,

Number of colony forming units (CFU's) per gram of the sample

$$\text{CFU} = \frac{\text{Mean number of Cfu's x Dilution factor}}{\text{Quantity of sample on weight basis}} \quad \ldots\ldots\ldots (3.5)$$

3.12 COST ANALYSIS OF MICROENCAPSULATED VANILLA EXTRACT POWDER

The cost analysis of production of encapsulated vanilla extract powder by spray drying technique was estimated by considering the costs *viz*, cost of building, spray drier, raw material, processing, labour, electricity and other related costs. The fixed cost and variable cost was used for determining the cost of operation. The following assumptions and relationships were used for calculating

the cost of encapsulated powder. The fixed cost was calculated by using the following relationship as described by Palanisami *et al*. (1997).

Fixed cost of the drier / year = Fixed cost × Capital Recovery Factor

Where,

Capital Recovery Factor (CRF) was derived by using the equation

$$CRF = \frac{Ri \times (1+Ri)^n}{(1+Ri)^n - 1} \quad \text{..... (3.6)}$$

where,

R_i = existing rate of interest for long term (10.5%) bank loans, %

n = life period of the spray drier, year

The variable costs, which are incurred on wages, electricity charges, repairs and maintenance, raw materials *etc*., were calculated by collecting data during the operation of equipment and assuming certain data reasonably wherever necessary.

CHAPTER IV

RESULTS AND DISCUSSION

This chapter deals with the results of the investigations carried out on the encapsulation of vanilla extract. The effect of various process variables on the quality characteristics of the encapsulated powder leading to their standardization and results of microstructural and storage studies of the optimally produced encapsulated vanilla extract powder are analysed and discussed.

4.1 PHYSICO-CHEMICAL PROPERTIES

4.1.1 Physico-chemical Properties of Vanilla Extract

The physico-chemical properties of vanilla extract used as the core material for encapsulation relevant to the study were found and are presented in Table 4.1.

Table 4.1 Physico-chemical properties of vanilla extract

Parameters		Values ± SD
Specific gravity (at 25^0C)		1.06 ± 0.20
Refractive index (^{0}Brix)		54.50 ± 0.50
pH		4.30 ± 0.87
Colour	L	31.72 ± 1.03
	a	-1.10 ± 0.01
	b	21.23 ± 0.49
Water activity (a_w)		0.702 ± 0.003
Viscosity (cp)		4370 ± 50

SD - Standard deviation

4.1.2 Physico-Chemical Properties of Wall Materials

The physico-chemical properties of maltodextrin (MD) and maize starch (MS) as wall materials for encapsulation relevant to the study were found and are presented in Table 4.2.

Table 4.2 Physico-chemical properties of wall materials

Parameters		Maltodextrin	Maize starch
Moisture content, w.b (%)		3.89 ± 0.02	3.18 ± 0.02
Water activity (a_w)		0.336 ± 0.003	0.301 ± 0.003
Bulk density (g/cm^3)		0.392 ± 0.01	0.313 ± 0.01
Solubility (%)		98.93 ± 0.50	73.14 ± 0.50
pH		7.13 ± 0.03	6.73 ± 0.04
Colour	L	94.32 ± 1.25	97.04 ± 1.31
	a	0.05 ±0.001	0.02 ± 0.004
	b	7.89 ± 0.75	2.35 ± 0.54

4.2 CHARACTERISTICS OF VANILLA EXTRACT EMULSION

The emulsion for encapsulation of vanilla extract by spray drying was prepared with MD and MS as wall materials as stated in section 3.6 and as per the experimental design as stated in section 3.3. The effect of the process variables such as wall material concentration and flavour load on the emulsion characteristics such as emulsion viscosity, emulsion stability and emulsion droplet size are presented and discussed below.

4.2.1 Viscosity of Vanilla Extract Emulsion

The apparent viscosity of the emulsion obtained after emulsification of vanilla extract with wall materials such as MD and MS are shown in Fig. 4.1. The viscosity of vanilla extract emulsion ranged between 2135 and 3514 cp. It may be observed from the figure that for all the wall material concentration studied, with increase in flavour load, the viscosity was found to increase significantly. Among the wall material concentration studied, it was found that with increase in MS composition, viscosity increased implying that maize starch imparts viscous function in the combination studied. The highest viscosity of 3514 cp was observed for a flavour load of 30% and wall material concentration of 100% MS where as a minimum viscosity of 2135 cp was noticed for 10% flavour load with a wall material composition of 100% MD.

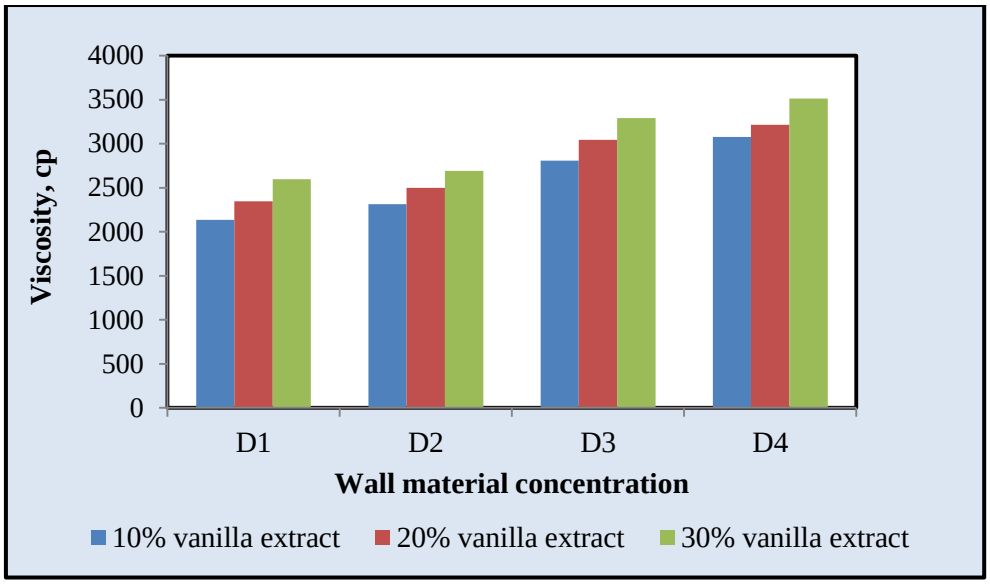


Fig. 4.1 Viscosity of vanilla extract emulsion

Increase in viscosity at same total solids content of 50% for all emulsion, might suppress internal circulation inside droplets, and permits rapid formation of semi permeable membrane around the droplet causing selective diffusion during early stages of drying which might improve retention (Reineccius, 2004). However increasing viscosity beyond an optimum limit causes a decrease of retention due to slow formation of droplets during atomization, rapid crust formation, which hinders water reaching the surface, building up internal pressure which results in irregular particles (Bhandari *et al*., 1992). On the other hand a wall material which provides minimum viscosity at maximum solid content is advantages provided the encapsulation efficiency and other quality properties are acceptable on commercial point of view.

4.2.2 Stability of Vanilla Extract Emulsion

The stabilities of the vanilla extract emulsion obtained after emulsification are depicted in Fig. 4.2. It was revealed that the emulsion stability values varied between 28 and 89 min. with combination of 30% flavour load, 100% MS as wall material registering minimum value and 10% flavour load and 100% MD as wall material recording highest value. With increase in flavour load from 10 to 30%, for all wall material combination studied, the stability was found to decrease

significantly. This is clear from the fact that the time required for the separation of the two phases decreased with increased vanilla extract concentration resulting in reduced stability. With the increase in MD content in the wall material mix, the stability values showed an increasing trend.

In general stability decreased with the increase in flavour load and MS content. With the increase in flavour load, the availability of functional groups of both MD and MS to act as emulsifying agent is reduced which could be the reason for the reduced emulsifying effect and decrease in stability (Re, 1998).

MD dissolves in the solution completely and takes long time to break into separate fractions. Therefore, increased stability was observed with wall material blend having high MD content. Though some findings claim that increased viscosity leads to increased stability, there are some findings which showed otherwise as reported by Liu *et al.* (2001) indicating that some other factors such as molecular weight, volatility and solubility of the flavours as well as wall material could have influenced stability and therefore retention. Similar results were also reported by Imagi *et al*. (1992) that stability values were found to decrease with increase in viscosity reflecting the complex nature of the structure of wall materials.

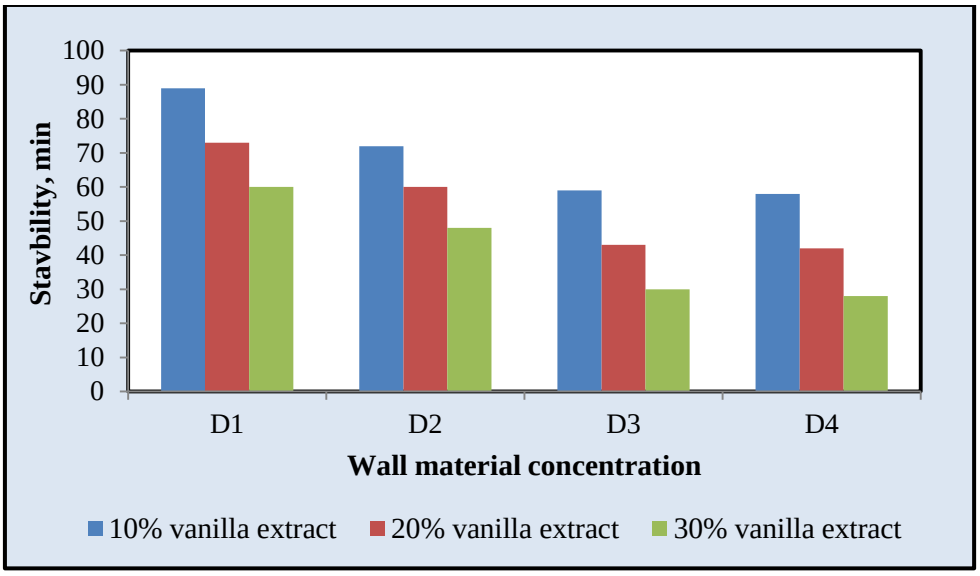


Fig. 4.2 Stability of vanilla extract emulsion

In our case since the emulsion produced with all wall material and flavour loads were encapsulated by spray drying before the phase separation occurs, the variation in stability in terms of the phase separation time would have only limited influence on encapsulation efficiency.

4.2.3 Emulsion Particle Size

The emulsion particle size of vanilla extract emulsion with wall materials MD and MS expressed in terms of mean diameter (µm) are presented in Fig.4.3. It was observed that the emulsion particle size ranged from 4.8 to 7.5 µm. The emulsion with 100% MD and 30% flavour load showed highest emulsion particle size of 7.5 µm. The emulsion particle size increased significantly with increase of in flavour load irrespective of the wall material combinations. With the increase in MS concentration in the wall material concentration, the emulsion particle size was found to decrease significantly ($P<0.05$). For a flavour load of 20%, when MS concentration increased from 0 to 100%, the emulsion particle size decreased from 7.1 to 5.5 µm.

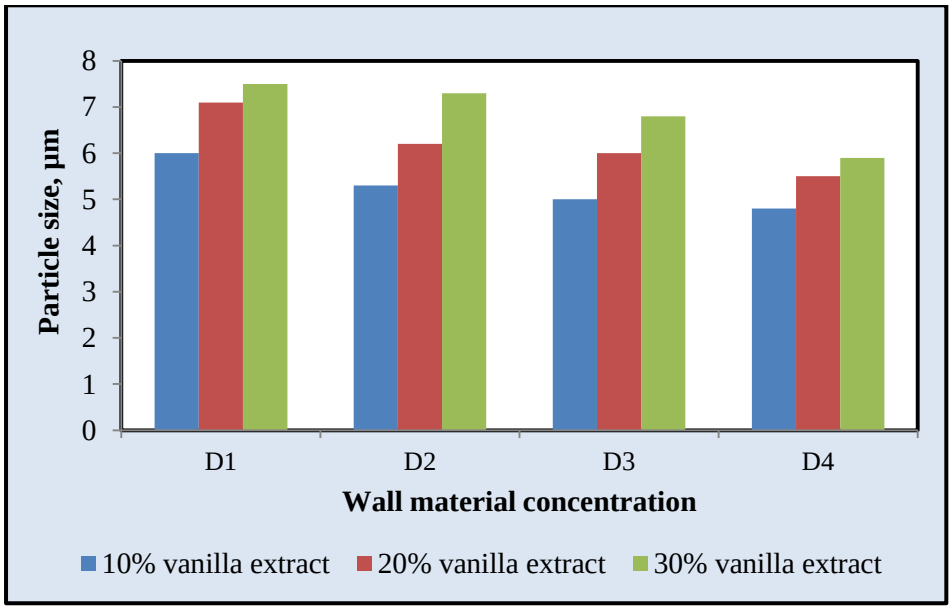


Fig. 4.3 Emulsion particle size of vanilla extract

The decrease in particle size with increase in MS content could be due to the increase in viscosity of emulsion. Increase in viscosity resists droplet

coalescence and agglomeration and retains decreased size of vanilla extract droplets. It was reported that smaller size of emulsion droplet could result in increased encapsulation efficiency (Liu *et al*., 2001; Soottitantawat *et al*., 2003).

On the other hand, with the increase in flavour load in the blend, the viscosity of the dispersed phase increases and resists the breaking down of the particle size resulting in larger oil droplets. Similar results were also reported by Bae and Lee (2008).

4.3 MICROENCAPSULATION OF VANILLA EXTRACT

Vanilla extract was microencapsulated with wall material combinations of MD and MS in a vertical co-current two fluid nozzle lab model spray drier as described in section 3.5 and 3.8. The effect of the process variables such as flavour load, wall material concentration and spray drier inlet air temperature on the quality attributes such as moisture content, bulk density, powder wettability, cold water solubility, colour and encapsulation efficiency were experimentally determined and statistically analysed for the extend of variation. The results of this analysis are discussed below.

4.3.1 Moisture Content of Microencapsulated Vanilla Extract Powder

The variation in moisture content of spray dried microencapsulated vanilla extract powder at different process parameter is shown in Fig. 4.4. It was observed during the study that the moisture content varied from 3.06 to 5.52% (w.b.), with 30% flavour load, 100% MS and inlet air temperature 190^0C recorded minimum moisture content.

With increase in MS concentration in the wall material blend, the moisture content was found to decrease. For a flavour load of 20% and inlet temperature of 180^0C, when MS concentration increased from 0 to 100%, the moisture content was found to decrease from 4.9 to 3.12% (w.b.). With increase in MS concentration, the viscosity of the resultant emulsion was found to increase resulting in decreased emulsion particle size and consequently the powder particle

size. This could result in increased surface area exposed to the drying air causing decrease of moisture content of the powder.

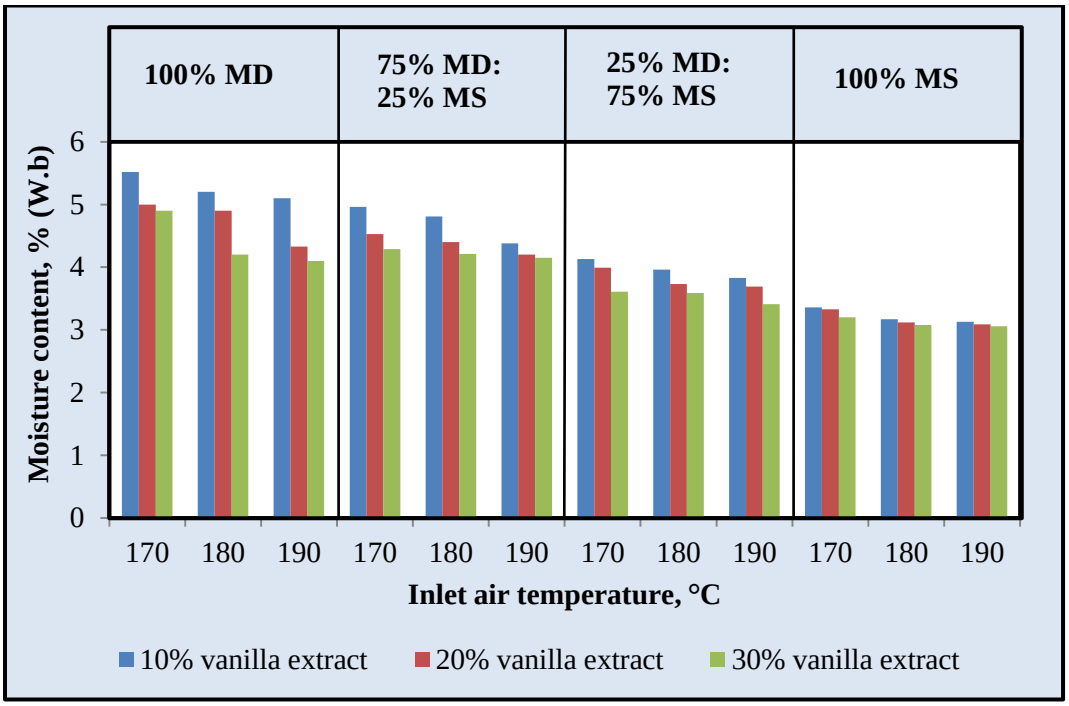


Fig. 4.4 Moisture content of microencapsulated vanilla extract powder

With the increase in flavour load, the moisture content was found to decrease. Though it was found that with increase in flavour load the viscosity of the emulsion increased, the emulsion particle size increased as stated in section 4.2.3. Therefore a correlation could not be established between the moisture content, particle size and the flavour load. Such findings were also reported by Kneifel *et al.* (1991) and Anker and Reineccius (1985) that other than viscosity and particle size, the structure and porosity of the particles, the drying rate and inlet and outlet air temperature differentials could also affect the water holding capacity of the encapsulated powder.

In general, the moisture content was found to decrease with increase in inlet air temperature. At a flavour load of 20% and 100% MD content, the moisture content decreased from 5 to 4.33% (w.b.) when inlet air temperature was increased from 170^0C to 190^0C. With increase in inlet air temperature the temperature differential in spray drier chamber increased resulting in decreased

relative humidity and the drying air could hold more moisture during the drying process leading to the lowering of moisture content of the encapsulating powder. Similar results were reported by Erus and Yurdagel (2007).

The effect of flavour load, wall material concentration and inlet air temperature on moisture content of encapsulated of vanilla extract powder was statistically analysed by analysis of variance (ANOVA) and presented in Table B.4 (Appendix B). The treatments with 100% maize starch, flavour load of 10, 20 and 30% and inlet air temperature of 170, 180 and 190^{0}C on moisture content were significantly superior and recorded as group 'a'.

4.3.2 Bulk Density of Microencapsulated Vanilla Extract Powder

The variation in bulk density of encapsulated vanilla extract powder with different wall material blend, flavour load and inlet air temperature are presented in Fig.4.5.

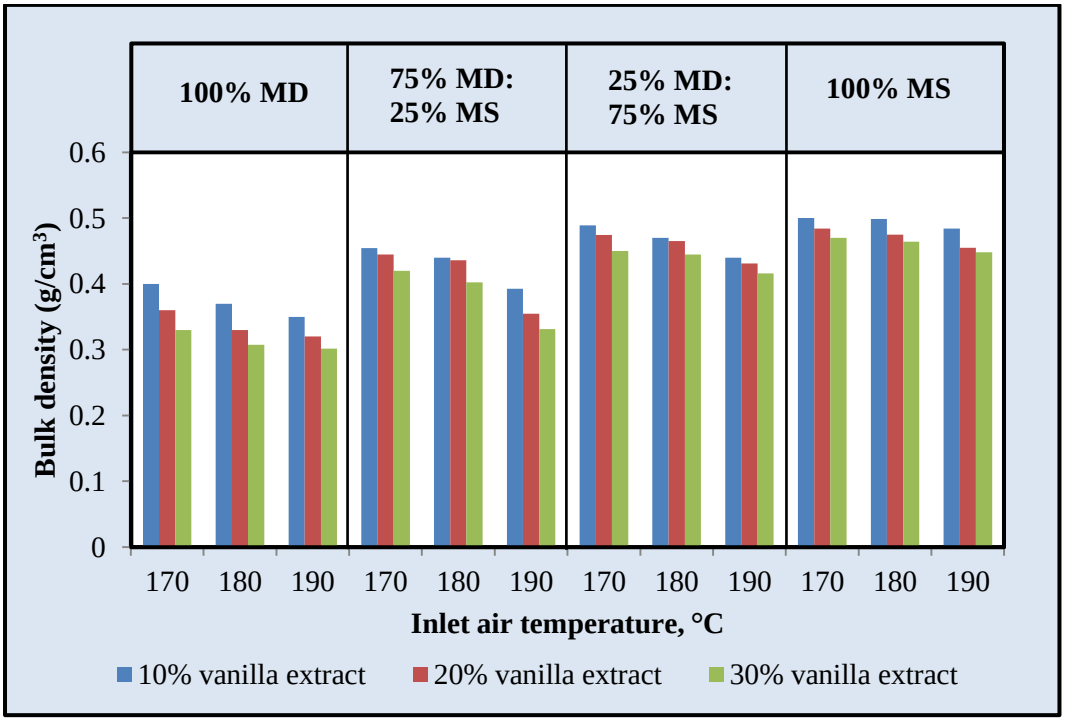


Fig. 4.5 Bulk density of microencapsulated vanilla extract powder

At the same temperature, for a given flavour load, bulk density increased with increase in MS content in the wall blend. For a flavour load of 20% and inlet

air temperature of 180^0C, the bulk density increased from 0.33 to 0.475 g/cm^3, when the MS content increased from 0 to 100%. With increase in MS content in the blend, the apparent viscosity of the emulsion increased which would result in formation of smaller sized particles with increased bulk density. This trend could also be attributed to the lower moisture content of the MS rich powder.

In general, with increase in flavour load, the bulk density of the powder was found to decrease. For a wall material concentration of 100% MD and inlet air temperature of 180^0C, the bulk density decreased from 0.37 to 0.307 g/cm^3 when the flavour load was increased from 10 to 30%. As explained in section 4.2.3 on emulsion particle size, with increase in flavour load, the emulsion particle size increased resulting in larger sized particles causing a decrease in bulk density of the encapsulated powder.

The influence of inlet air temperature on bulk density of the encapsulated powder may also be observed from the Fig. 4.5. It was revealed that with increase in inlet air temperature, the bulk density was found to decrease irrespective of the wall material concentration and flavour load. High inlet air temperature results in lower density powders, which is partially due to more rapid drying of the particle and fixing of particle size much before the removal of water and secondly due to the steam formation in the drying droplet, causing expansion of particle. Similar results were also reported by Anker and Reineccius (1998).

Bulk density is an important parameter which is prominent for the storage, packing and distribution of the microencapsulated powder. Higher bulk density leads to lesser packaging volume and space.

The effect of flavour load, wall material concentration and inlet air temperature on bulk density of encapsulated vanilla extract powder was statistically analysed by analysis of variance (ANOVA) is presented in Table B.5 (Appendix B). The treatments with 100% maize starch, 10% vanilla concentration and 170^0C and 180^0C on bulk density were significantly superior and recorded as group ‘a’.

4.3.3 Wettability of Microencapsulated Vanilla Extract Powder

Apart from surface oil measurements and encapsulation efficiency the ability of the spray dried powder to reconstitute in water is also very important which could be determined by the powder wettability. The variation in wettability of spray dried microencapsulated vanilla extract powder at different process parameters are shown in Fig. 4.6. It was observed that the wettability varied from 34.99 to 98.16 s. With 10% flavour load, 100% maize starch and inlet air temperature of 190^{0}C recorded maximum wettability (minimum wetting time).

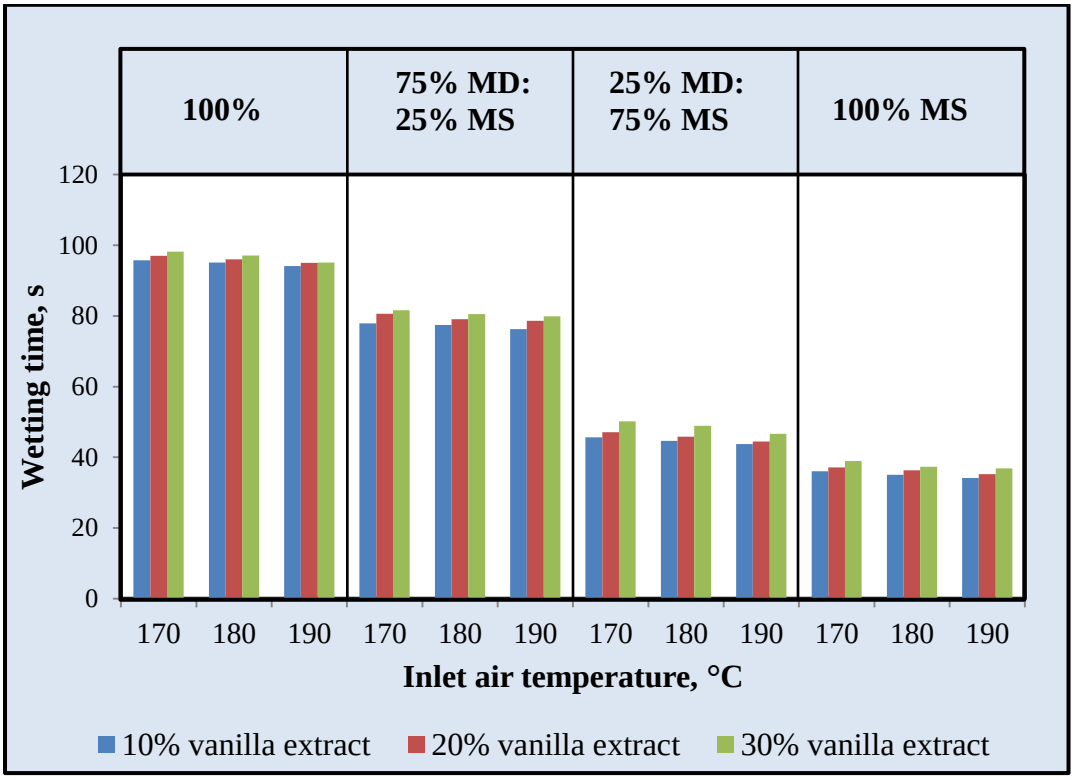


Fig. 4.6 Wettability of microencapsulated vanilla extract powder

With increase in MS concentration in the wall material blend, the wettability was found to increase. For a flavour load of 20% and inlet temperature of 180^{0}C, when MS concentration increased from 0 to 100%, the wettability was found to increase (wetting time decreased) from 96.97 to 35.1 s. Wettability is influenced by the carrier matrix used, apart from particle size and density (Reineccius, 2004). MS is hydrophilic in nature, and the short chain amorphous starch in MS facilitates the penetration of water into the powder. This is also

accompanied by increase in bulk density of the powder with increase in MS content.

With the increase in flavour load, the wettability was found to decrease. For a wall material concentration of 100% MS and inlet air temperature of 180^0C, the wettability decreased (wetting time increased) from 34.99 to 35.13 s when the flavour was increased from 10 to 30%. With increase in flavour load, the bulk density decreased, which would have reduced the wettability. Also with increase in flavour load, the surface oil content would have increased resulting in a decrease of wettability. According to Vega and Roos (2006), the wettability depends on powder particle size, density, porosity, surface area and surface activity of powders.

Wettability was found to increase with increase in inlet air temperature. At a flavour load of 20% and 100% MD content, the wettability increased from 96.97 to 95.00 s when inlet air temperature was increased from 170^0C to 190^0C. This could be due to the decreased moisture content of the product that the powder absorbs water and wet the surface fast.

The effect of flavour load, wall material concentration and inlet air temperature on wettability of encapsulated vanilla extract powder was statistically analysed by analysis of variance (ANOVA) is presented Table B.6 (Appendix B). The treatments with 100% maize starch, 10 % vanilla concentration and inlet air temperature of 180^0C and 190^0C on wettability were significantly superior and recorded as group ‘a’.

4.3.4 Cold Water Solubility of Microencapsulated Vanilla Extract Powder

The variation in cold water solubility of encapsulated vanilla extract powder with different wall material blend, flavour load and inlet air temperature are presented in Fig. 4.7. At the same temperature, for a given flavour load, solubility decreased with increase of MS concentration. For a flavour load of 20% and inlet air temperature of 180^0C, the solubility decreased from 81.72 to 52.99%, when the MS content increased from 0 to 100%. The composition of hydrolysed starches has much higher ratios of amylase to amylopectin than native starches so

that modified starches have higher solubility than native starches (Mukerjea *et al*., 2007). Since MD is a hydrolysed starch and MS is a native starch, with the increase in MS content in the wall material blend, the amylase to amylopectin ratio would be decreased resulting in decreased solubility.

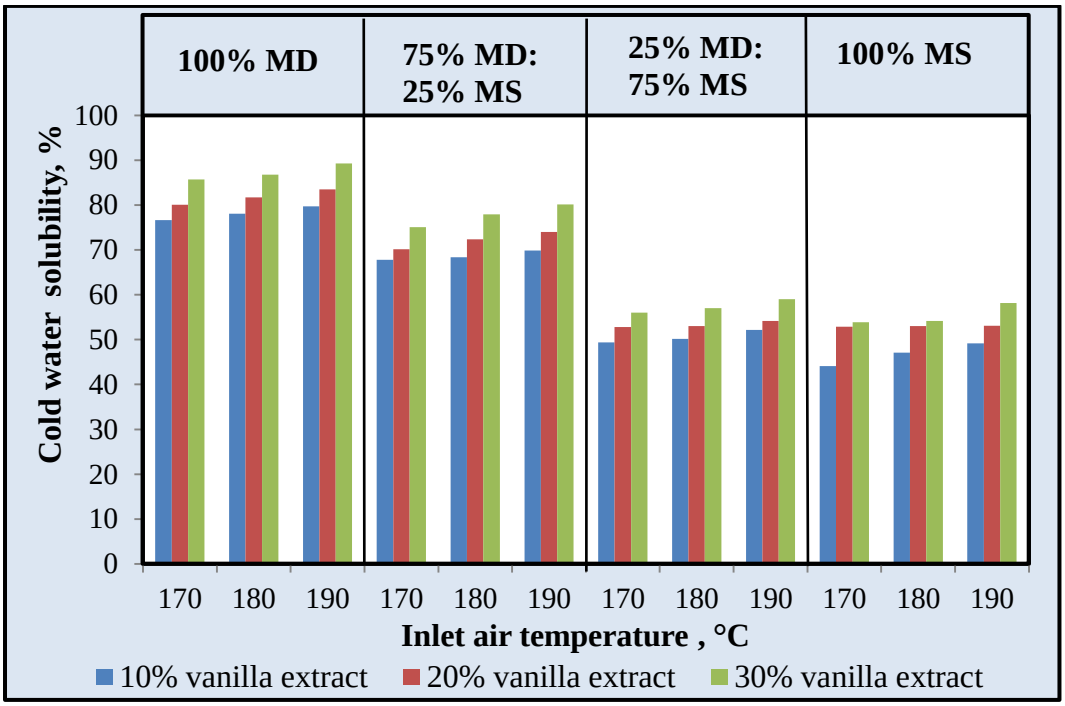


Fig. 4.7 Solubility of microencapsulated vanilla extract powder

With increase in flavour load, the solubility of the encapsulated vanilla extract powder was found to increase. For a wall material concentration of 100% MD and inlet air temperature of 180^0C, the solubility increased from 78.11 to 86.82% when the flavour load was increased from 10 to 30%. With the increase in flavour load, the hydrophilic wall material available would be limited to produce a strong structural matrix resulting in thinner layers of wall material between encapsulated oil droplets (McNamee *et al*., 1998). This could have resulted in easy solubilisation of wall material.

The influence of inlet air temperature on solubility of the encapsulated powder may also be observed from the Fig. 4.7. It was revealed that with increase in inlet air temperature, the solubility was found to increase irrespective of the wall material concentration and flavour load. For a wall material concentration of

100% MD and flavour load of 20% the solubility increased from 80.10 to 83.48% when the inlet air temperature was increased from 170 to 180^{0}C. When emulsion blend is heated in the spray drier the tight organization of starch granules which is the main constituent of the wall material constituents gets disrupted facilitating the migration of water into the granules (Loksuwan, 2007). Also, with the increase in inlet air temperature, the amylopectin branched chain length gets broken and reduced contributing to the increased solubility (Singh and Singh, 2003).

The effect of vanilla extract concentration, wall material concentration and inlet air temperature on cold water solubility of encapsulated vanilla extract powder was statistically analysed by analysis of variance (ANOVA) is presented in Table B.7 (Appendix B). The treatments with 100% maltodextrin, 30% vanilla concentration and 180 and 190^{0}C inlet air temperature on cold water solubility were significantly superior and recorded as group 'a'.

4.3.5 Colour of Microencapsulated Vanilla Extract Powder

Since whiteness of the powder is the most important quality parameter in terms of its food applications, the variation of Hunter 'L' values with wall material concentration, flavour load and inlet air temperature are plotted and presented in Fig.4.8. The Hunter 'L', 'a' and 'b' values of the encapsulated vanilla extract powder are given in Table A.8, A.9 and A.10 (Appendix A). It was observed that the Hunter 'L' value varied from 92.69 to 96.33, with 10% flavour load, 100% maize starch and inlet air temperature of 170^{0}C recorded maximum Hunter 'L' value.

With increase in MS concentration in the wall material blend, Hunter 'L' value was found to increase. For a flavour load of 20% and inlet temperature of 180^{0}C, the Hunter 'L' value increased from 93.24 to 96.01 when MS concentration increased from 0 to 100%. With the increase in flavour load, the Hunter 'L' value was found to decrease. For a wall material concentration of 100% MS and inlet air temperature of 180^{0}C, the Hunter 'L' value decreased from 96.30 to 95.55 when the flavour load was increased from 10 to 30%. In general,

the ‘L’ value was found to decrease with increase in inlet air temperature. At a flavour load of 20% and 100% MD content, Hunter ‘L’ value decreased from 93.47 to 93.01 when inlet air temperature was increased from 170^0C to 190^0C.

The increase in ‘L’ value with increase in MS content is obvious from the fact that the raw MS has higher ‘L’ values compared to MD. Similarly, the decrease in ‘L’ values with increase in flavour load could also be attributed to the low ‘L’ values of the vanilla extract. At higher temperature, the emulsion will be subjected to high intensity heat which could lead to burning, structural changes and increase in retention rates (Falade and Omojola, 2010) which could have resulted in the lowering of ‘L’ values with increase in inlet air temperature.

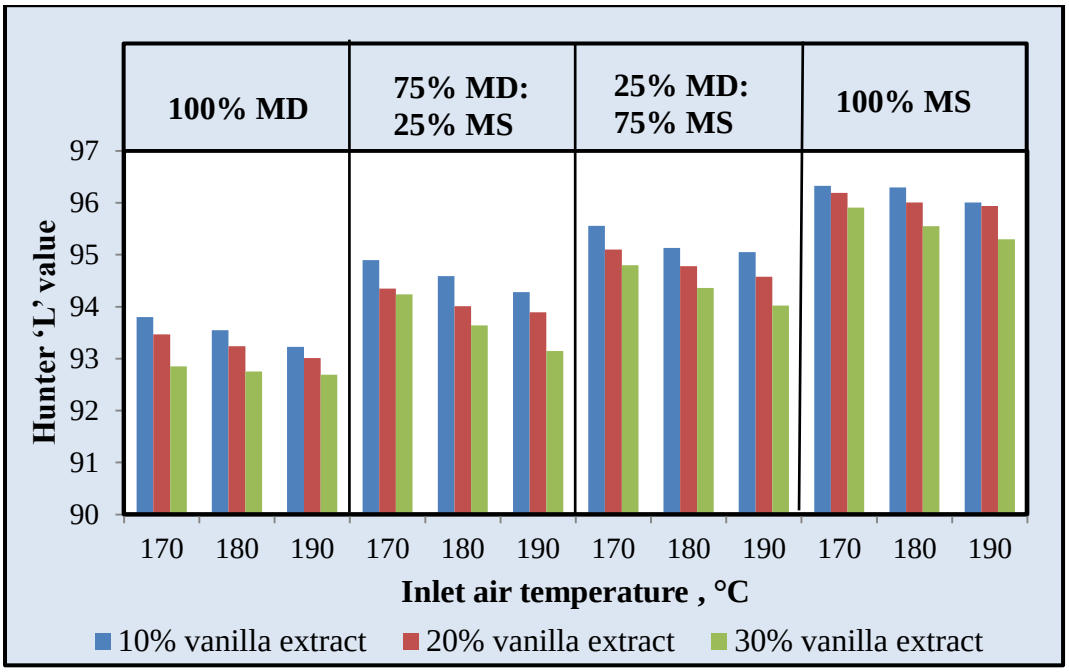


Fig. 4.8 Colour of microencapsulated vanilla extract powder

The effect of vanilla extract concentration, wall material concentration and inlet air temperature on Hunter L, a and b colour value of microencapsulated vanilla extract powder was statistically analysed by analysis of variance (ANOVA) and presented in Table B.8, B.9 and B.10 (Appendix B). The treatment with 100% maize starch, 10% vanilla concentration and 170^0C and 180^0C inlet air temperatures on Hunter L Colour value were significantly superior and recorded as group ‘a’. In addition, the treatment with 100% maize starch, 10% vanilla

concentration and 170^{0}C and 180^{0}C inlet air temperatures on both Hunter 'a' and 'b' Colour values were significantly superior and recorded as group 'a'. The subgroups in ascending alphabetical order show the lesser desirable values of the members comprising the same.

4.3.6 Encapsulation Efficiency of Microencapsulated Vanilla Extract Powder

Encapsulation efficiency (EE) defined by Rosenberg and Sheu (1996) as the proportion (expressed as %) of the encapsulated flavour that cannot be extracted by a suitable solvent from one gram of microcapsule. This is the most reliable parameter to assess efficiency of the process rather than 'retention percentage' which is the ratio expressed in percentage of the encapsulated flavour to the initial flavour load in the emulsion.

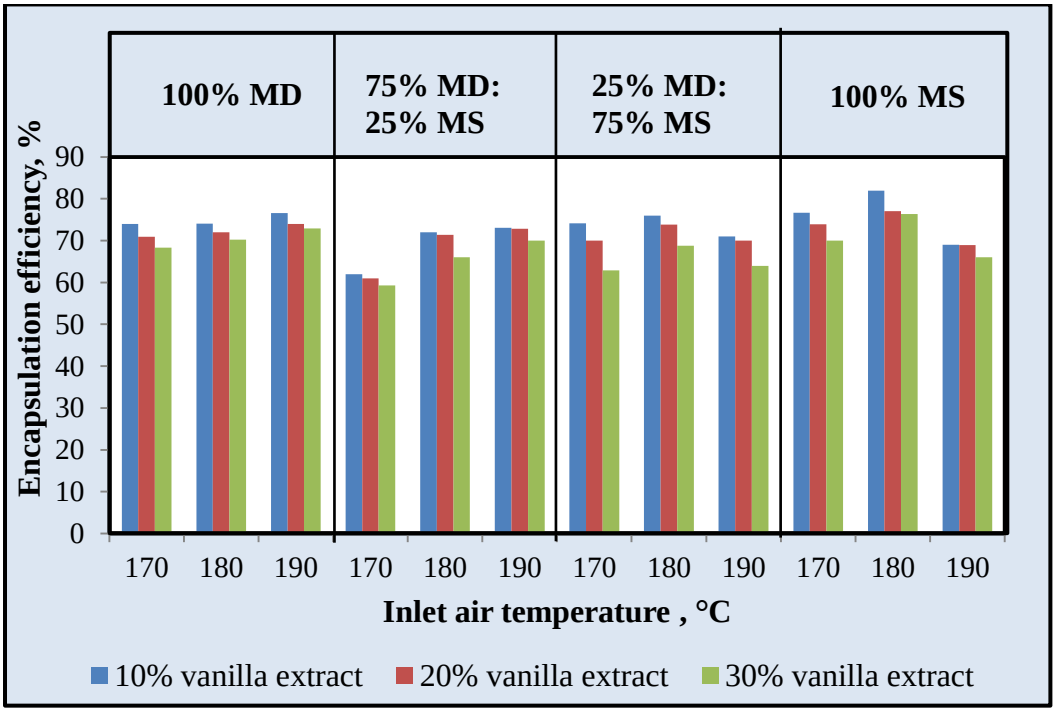


Fig. 4.9 Encapsulation efficiency of microencapsulated vanilla extract powder

The effect of wall material concentration, flavour load and inlet air temperature on the encapsulation efficiency of the vanilla extract powder are presented in Fig. 4.9 and the obtained data is tabulated in Table A.11 (Appendix A). As mentioned, the EE values show the presence of flavour extract on the

powder particle surfaces and the degree to which the wall material can prevent the extraction of internal oil through leaching process.

The encapsulation efficiency varied in the range of 59.32 to 81.93%. For a given flavour load, with increase in MS content and inlet air temperature, the encapsulation efficiency was found to increase when MS concentration increased from 0 to 25%. But when the MS concentration increased further, it was observed that the efficiency increased when temperature increased from 170 to 180°C followed by a decrease in EE, when the drying air temperature was increased from 180 to 190°C. This indicates that temperature plays a role in MS structure and its capacity to retain the flavour. The same trend was also observed at all flavour loads studied. For flavour load of 20%, the EE increased from 70.9 to 74% and 61.01 to 72.85% when MS concentration increased from 0 to 25% between the temperatures of 170 to 190°C. But on further increase of MS percentage, and when temperature was incremented from 170 to 190°C at the rate of 10°C, it was revealed that the EE increased from 70.01 to 73.83% and further decreased to 70.01%. Similar trend was observed for 100% MS concentration also. These results indicated that in general MS is better encapsulant than MD as the highest efficiencies were reported by 100% MS concentrations. The increase in EE with increase in MS concentrations could be attributed to the better emulsifying/ stabilizing properties of it compared to MD. The increase in viscosity with increase in MS content results in formation of small sized droplets and increased retention as mentioned in section 3.4.

Though the EE increased with increase in inlet air temperature for low MS concentration, with further increase in MS content EE was found to increase and then decrease when temperature was increased from 170 to 190°C at the rate of 10°C. When temperature was increased from 170 to 180°C, rapid drying occurred which permits quicker formation of semipermeable membrane and accompanied selective diffusion and increased retention. However as postulated by Rulkens and Thijssen (1972), further increase in temperature would have resulted in higher

vapour pressure than atmosphere leading to formation of vapour bubbles and puffing causing flavour loss.

For a given wall material concentration, regardless of the inlet air temperature, the EE decreased with increase in flavour load. For a wall material concentration of 100% MD at 180^0C inlet air temperature, the EE values decreased significantly from 74 to 68.3% when flavour load was increased from 10 to 30%. This general trend could be due to the insufficient wall material to produce a sufficiently strong structural matrix and thin layers of wall material between encapsulated oil droplets (McNamee, *et al*., 1998; Young *et al*., 1993).

The effect of flavour load, wall material concentration and inlet air temperature on encapsulation efficiency of microencapsulated vanilla extract powder was statistically analysed by analysis of variance (ANOVA) is presented in Table B.11 (Appendix B). The treatment with 100% maize starch, 10% vanilla concentration and 180^0C inlet air temperature on encapsulation efficiency was significantly superior and recorded as group 'a'.

4.4 PROCESS OPTIMIZATION FOR ENCAPSULATED VANILLA EXTRACT POWDER

Microencapsulation of vanilla extract was carried out by spray drying technique and the various process parameters which could influence the quality characteristics of the encapsulated powder were studied as per the experimental design charted out. The effect of flavour load, wall material concentration (MD and MS) and inlet air temperature, on the quality aspects relevant to their use efficiency, application potential and storage stability of the encapsulated powder were determined and discussed in section 4.2 and 4.3. Based on the results and related discussions and the statistical analysis carried out, the process parameters responsible for yielding maximum product quality are summarised in Table 4.3 (a) and (b).

Table 4.3 (a) Process parameters for obtaining optimum product quality characteristics for encapsulated vanilla extract powder

Sl. No.	Quality characteristics	Parameters for obtaining optimum product characteristics
i. Emulsion characteristics		
1	Emulsion viscosity	Emulsions with 100% MS concentration and flavour concentration of 10, 20 and 30% showed higher viscosity than other combinations.
2	Emulsion stability	Higher stability values were observed in the emulsion with 100% MD and 10% flavour concentration.
3	Emulsion droplet size	Lower emulsion particle size was observed in the emulsion with flavour concentration of 10% and wall material concentration of 100% MS.
ii. Powder characteristics		
1	Moisture content	MS coated encapsulated vanilla extract showed minimum moisture content than MD and combination of MD and MS coated powder. 100% MS with flavour load of 10, 20 and 30% at inlet air temperature of 170, 180 and 190^0C resulted in minimum moisture contents.
2	Bulk density	Encapsulated samples coated with MS showed higher bulk density than other wall material combinations. 10% flavour emulsified with 100% MS and spray dried at 170 and 180^0C gave higher bulk density.

Table 4.3 (b) Process parameters for obtaining optimum product quality characteristics for encapsulated vanilla extract powder

Sl. No.	Quality characteristics	Parameters for obtaining optimum product characteristics.
3	Powder wettability	MS coated vanilla extract showed higher wettability than other wall material combinations. Vanilla extract emulsified with 100% MS, 10 % flavour load and spray dried at 180 and 190^0C showed maximum wettability.
4	Cold water solubility	MD coated encapsulated vanilla extract showed higher solubility than MS. A flavour load of 30% emulsified with 100% MD as wall material and spray dried at inlet air temperatures of 180 and 190^0C provided powders with higher solubilities.
5	Colour	Higher 'L' values were observed in MS coated encapsulated vanilla extract powder. A flavour load of 10% emulsified with 100% MS and spray dried at inlet air temperatures of 170 and 180^0C showed higher Hunter 'L' values and therefore high whiteness.
6	Encapsulation efficiency	100% MS coated encapsulated powder showed higher encapsulation efficiency than other wall material combinations. A flavour load of 10%, emulsified with 100% MS and spray dried at an inlet air temperature of 180^0C resulted in highest encapsulation efficiency.

It may be derived from the Table 4.3 that a wall material concentration of 100% MS, flavour load of 10% and inlet air temperature of 180^0C, may be chosen as the best, based on their influence on emulsion and powder characteristics studied except in the case of solubility which showed 100% MD and flavour load

of 30% the best. A 30% flavour load increases solubility since there exists inefficient wall material for solubilisation. A 30% flavour load drastically reduces the EE as high surface oil content was observed.

The decreased solubility of MS in cold water is the other reason for reduced solubility. This is not a major disadvantage since in most food applications; the food is heated to at least 60^0C which will increase the solubility further. Also since MS is a food component, it may be consumed along with the other component releasing the flavour. Therefore the optimal parameters chosen for the production of microencapsulated vanilla extract powder are as follows:

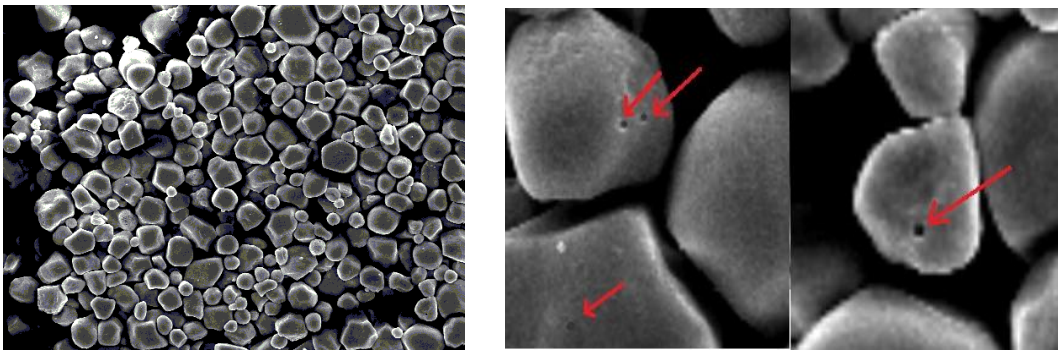
Wall material concentration	: 100% Maize starch
Flavour load	: 10% vanilla extract (w/w)
Inlet air temperature	: 180^0C

4.5 MICROSTRUCTURAL CHARACTERISTICS OF THE ENCAPSULATED POWDER

The microstructural characteristics such as morphology (outer structure) and inner structure of the optimally produced encapsulated vanilla extract powder were determined by Scanning Electron Microscopy (SEM). The SEM micrographs of morphology and inner structure of encapsulated vanilla extract powder with the wall material concentration of 100% MS, flavour load of 10% and inlet air temperature of 180^0C shown are in Fig. 4.10 (a) and (b).

The SEM micrographs of the encapsulated powder appeared to be spherical but with surface dents. Surface indentations are more prevalent in larger particles than smaller ones. Presence of dents could be due to the slow drying at the initial drying period because of which particles undergo shrinkages in latter stages of drying. Even though surface dents were present, absence of surface cracks and pores indicated that particles had not undergone puffing action which is not desired for microcapsule stability. Similar results were also observed by Krishnan *et al.* (2005). The SEM micrographs also revealed that encapsulated powder had a particle size ranged from 5 to 50 µm. It may be observed from the

micrographs that encapsulated powder existed as single discrete particles with no agglomerations indicating efficient encapsulation and better flowability.



(a) (b)

Fig. 4.10 SEM micrographs of optimally produced encapsulated vanilla extract powder (a) outer surface (b) inner surface.

SEM micrographs of the inner structure of the encapsulated powder indicated that the flavour particles were in the form of small droplets embedded and distributed in the wall matrix. The central void related to expansion of the particle (puffing) during drying was not observed.

4.6 MICROBIAL QUALITY OF THE ENCAPSULATED VANILLA EXTRACT POWDER

Optimally produced microencapsulated vanilla extract powder was analysed for its microbial quality (bacteria and fungi) and the results are presented in Table 4.4. The number of colony forming units (cfu) per gram of bacteria and fungi of optimized microencapsulated vanilla extract powder was determined by serial dilution method.

The microbial load of the optimised microencapsulated vanilla extract powder were analysed at different dilutions such as 10^{-3} and 10^{-6} for both bacterial and fungal growth. It may be revealed from the results the microbial load (bacteria and fungi) in encapsulated vanilla extract powder sample was found to be nil. This could be due to the sterile conditions of drying and the high temperature to which the powder was subjected to during drying.

Table 4.4 Microbial load of encapsulated vanilla extract powder

Material	Microbial load (cfu/g)			
	Bacteria		Fungi	
	10^{-3}	10^{-6}	10^{-3}	10^{-6}
Microencapsulated vanilla extract powder	NIL	NIL	NIL	NIL

4.7 RETENTION OF ACTIVE COMPONENT IN ENCAPSULATED OF VANILLA EXTRACT POWDER

Vanillin is the active component in the vanilla extract and the presence of vanillin content in optimized encapsulated vanilla extract powder was determined by gas chromatography method. The presence of vanillin was analysed by comparing with the chromatograph of vanillin standard. The obtained gas chromatograph of the optimally produced encapsulated vanilla extract powder is shown in Fig 4.11.

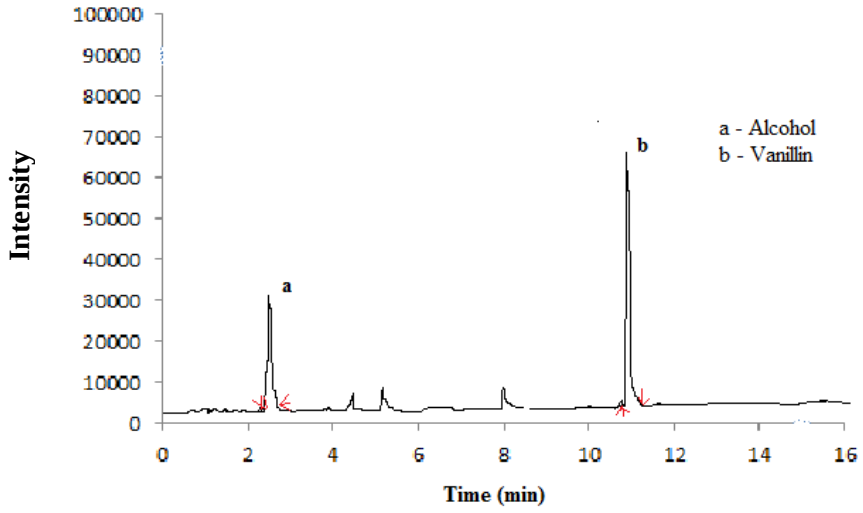


Fig. 4.11 Gas chromatograph of optimized microencapsulated vanilla extract powder

From the figure, it is clear that the peak of vanillin in the chromatograph of the optimized sample similar to that obtained for the standard showed the presence of vanillin in the encapsulated vanilla extract powder.

4.8 CHANGES IN QUALITY ATTRIBUTES OF ENCAPSULATED VANILLA EXTRACT POWDER DURING STORAGE

The optimally produced microencapsulated vanilla extract powder was well packed in aluminium foil pouches and stored at room temperature. The changes in the main quality attributes of the optimally produced encapsulated vanilla extract powder for a period of five months are presented in Table. 4.5. From the table, it was revealed that there occurred a slight increase of moisture content from 3.17 to 3.44% (w.b.) during the storage period of five months. This could be due to the absorption of moisture from package environment.

Table 4.5 Changes in quality attributes of the encapsulated vanilla extract powder during five month storage

SI. No.	Quality characteristics	Microencapsulated vanilla extract					
		Storage period (month)					
		Fresh	1st	2nd	3rd	4th	5th
1.	Moisture content,% (w.b)	3.17	3.20	3.31	3.36	3.40	3.44
2.	Bulk density, g/cm^3	0.495	0.484	0.450	0.440	0.441	0.431
3.	Wettability, s	35.03	37.01	39.59	41.53	43.18	45.13
4.	Solubility, %	47.11	46.16	45.89	45.78	43.25	42.98
5.	Encapsulation efficiency, %	81.93	81.70	81.59	81.00	80.28	80.96
6.	Microbial load, cfu/g	NIL	NIL	NIL	NIL	NIL	NIL

Similarly slight differences in values of wettability, bulk density, solubility, and encapsulation efficiency were noticed during the five months storage period, but these changes would not contribute for any appreciable

changes in the quality of the product. It may be noted that the microbial load in cfu/g of the powder remained nil during the entire storage periods indicating the microbial stability of the product.

4.9 COST ANALYSIS

The computation of cost of production of the one kilogram of microencapsulated vanilla extract powder is given in Appendix C. The computed cost of one kilogram of microencapsulated vanilla extract powder using a tall type lab model spray drier with twin fluid nozzle is found to be Rs. 850. The cost of the production could be further scaled down once the production is taken up on a commercial scale.

CHAPTER V

SUMMARY AND CONCLUSION

Spices are mainly used for preserving, colouring and flavouring. Flavour is the sensory impression of food or other substance. Vanilla is one of the minor spices and is the most popular flavouring agent and second most expensive spice in the world. Vanilla flavour is widely used as flavour in custard, cake, candy, and pudding and to enhance the flavour of beverages and sauces and especially by the ice cream industry and thus most people consider it to be the default flavour.

Vanillin flavour is highly volatile and heat sensitive and application in food incorporation is limited; this can be minimized by encapsulation technique with suitable wall material. This microencapsulation technique protects the vanilla extract from undesirable changes and coverts into a free flowing powder besides several other advantages. There are different techniques available for microencapsulation; spray drying is the most common, economical and commercial method for microencapsulation process.

Spray drying is a simple, fast and continuous process in which a liquid or paste is transformed in a powder of micro particles after a relatively short drying period and can be used for the heat liable material because of the low temperature that the wall material reaches. The microencapsulation of vanilla extract could be carried out with suitable wall material. The intention of the present study was to develop a free flowing and high stable encapsulated vanilla extract powder and standardization of suitable wall material combinations, flavour load and inlet air temperature of spray drier for the microencapsulation process by spray drying technique.

In this study, microencapsulation of vanilla extract was carried out in tall type spray drier with twin fluid atomizer. Maltodextrin, maize starch and their combinations were used as wall material to encapsulate the vanilla extract. The physico-chemical characteristics of vanilla extract and wall materials were carried out. Different proportion of wall materials were used for the emulsification such as 100% maltodextrin, combination of 75% maltodextrin and 25% maize starch,

25% maltodextrin and 75% maize starch and 100% maize starch. The wall material combinations were emulsified with 10, 20 and 30% of vanilla extract (flavour load) and emulsified in a high speed mixture. The emulsion characteristics such as emulsion viscosity, stability and emulsion particle size were then determined.

The emulsion with core concentration of 30% and wall material concentration of 100% maize starch showed significantly higher viscosity of 3514 cp. With increase of MS concentration from 0 to 100% and increase in flavour load, the emulsion viscosity increased. The emulsion with 100% MD and 10% flavour load showed significantly higher emulsion stability. Lower emulsion particle size was observed in the emulsion with flavour load of 10% and wall material concentration of 100% MS.

The emulsions were then spray dried at different inlet temperatures of 170, 180 and 190^0C. The encapsulated vanilla extract powder were collected, packed in aluminum foil pouches and stored in room temperature for five months. The microencapsulated powder characteristics such as moisture content, bulk density, wettability, cold water solubility, and encapsulation efficiency were carried out and analyzed. The moisture content varied from 3.01 % to 5.52 % (wet basis). The moisture content decreased with increase of maize starch content and flavour load and decreased inlet air temperature. Powders with 100% maize starch, flavour load of 10, 20 and 30% and inlet air temperature of 170, 180 and 190^0C showed significantly lower in moisture content than other combinations. The bulk density varied from 0.3016 to 0.5000 g/cm^3. The bulk density increased with increase of maize starch concentration and increase of inlet temperature, and decreased with increase in flavour load. Encapsulated powder with 100% maize starch, 10% flavour load with inlet air temperature of 170, 180 and 190°C showed significantly higher bulk densities than other combinations. The wettability of encapsulated vanilla extract powder varied from 34.99 to 98.16 s. Wettability was increased with increase of maize starch content, inlet air temperature and decreased with increase of flavour load. Encapsulated powder from 100% maize

starch, flavour load of 10% and inlet air temperatures of 180 and 190^{0}C showed significantly higher wettability. The cold water solubility ranged from 49.14 to 85.72%. The solubility decreased with increase of maize starch content and increased with increase of inlet air temperature and flavour load. The colours of all the microencapsulated powder were sparingly different from white. Hunter 'L' value increased with increase in maize starch concentration and decreased with inlet air temperature and flavour load. Encapsulated powder from 100% maize starch, 10% flavour load and 170 and 180^{0}C showed significantly higher 'L' value. The encapsulation efficiency of encapsulated vanilla extract ranged from 59.32 to 81.93%. The encapsulation efficiency increased with increase of maize starch content whereas it was observed that the efficiency increased when temperature increased from 170 to 180°C followed by a decrease in encapsulation efficiency, when the drying air temperature was further increased to 190°C. Encapsulation efficiency decreased with the increase of flavour load. The encapsulated powder with 100% maize starch concentration, flavour load of 10% spray dried at an inlet air temperature of 180^{0}C showed significantly higher encapsulation efficiency.

Wall material concentration of 100% MS, flavour load of 10% and inlet air temperature of 180^{0}C were chosen as best optimum parameters for the production of microencapsulated vanilla extract powder , based on their influence on emulsion and powder characteristics studied.

The morphology and inner microstructural characteristics of optimally produced encapsulated vanilla extract were carried out by using scanning electron microscopy (SEM). SEM micrographs revealed that the particle size ranged between 5 to 50 µm. The encapsulated powder was in spherical shape with surface dents, single, discrete with no agglomerations with flavour droplets distributed with wall matrix. The retention of vanillin in optimized sample of microencapsulated vanilla extract was also carried out employing gas chromatography. The chromatographs indicated the presence of vanillin. The powder characteristics such as moisture content, bulk density, wettability, cold

water solubility, encapsulation efficiency and microbial load of the optimally produced encapsulated vanilla extract powder were carried out for a five month storage period to analyse the changes in the quality attributes. No bacterial and fungal growth were observed in the optimized microencapsulated vanilla extract powder sample during five month of storage. No significant variations in moisture content, bulk density, wettability, cold water solubility and encapsulation efficiency were noticed during the storage period indicating the storage stability of the product.

The cost of production of one kilogram of microencapsulated vanilla extract powder using the pilot model spray drier was estimated to be Rs.850.

The following are the suggestions for future research work on the microencapsulation of vanilla extract.

1. Studies may be carried out with different combinations of wall material for the encapsulation to improve the stability of the encapsulated powder.
2. Studies may be carried out using different drying methods for the encapsulation.
3. Studies may be carried out with different wall materials foe encapsulating vanilla extract.

REFERENCES

Ali, A., Wani, T.A., Wani, I.A., and Masoodi, F.A. 2014. Comparative study of the physico-chemical properties of rice and corn starches grown in Indian temperate climate. *J. Saudi. Soc. Agric. Sci*. 33(6) (in press).

Alonso, P.C., Gonzalez, B.J.G., Beristain, C.I., Carter, V.E.J., and Mendoza, V.M.G. 2003. Estimation of the activation energy of carbohydrate polymers blends as selection criteria for their use as wall material for spray-dried microcapsules. *Carbohyd. Polym.* 53(2):197-203.

Anandaraman, S. and Reineccius, G.A. 1986. Stability of encapsulated orange peel oil. *Food. Technol.* 40: 88-93.

Anker, M.H. and Reineccius, G.A. 1988. Encapsulated orange oil: Influence of spray dryer air temperature on retention and shelf life. In flavour encapsulation. In: Risch, S.J. and Reineccius, G.A. (eds), *Flavour encapsulation*. Washington DC: American Chemical Society. pp. 78-86.

AOAC. 1999. Moisture in Flour and Cereal Foods. Methods of Analysis of the Association of the Official Analytical Chemists. AOAC International, Vol EE.

ASTA. 1968. Official Analytical Methods. 2nd edn. New Jersey: American Spice Trade Association.

A/S Niro Atomizer.1978. Determination of wettability. In: Sorensen, I.H., Krag. J., Pisecky, J., and Westergaard, V. (eds.), *.Analytical methods for dry milk product- 4th Edition*. De Forenede Trykkerier A/S, Copenhagen, Denmark. pp. 26-27.

Augustin, M.A., Sanguansri, L., Margetts, C., and Young, B. 2001. Microen-capsulation of food ingredients. *Food. Aust.* 53: 220-223.

Bae, E.K. and Lee, S.J. 2008. Microencapsulation of avocado oil by spray drying using whey protein and maltodextrin. *J. Microencapsul.* 25(8): 549-560.

Balasubramani, P., Viswanathan, R., and Vairamani, M. 2013. Response surface optimisation of process variables for microencapsulation of garlic (Allium sativum L.) oleoresin by spray drying. *Biosyst. Eng*. 114: 205-213.

Barbosa, M.I., Borsarelli, C.D., and Mercadante, A.Z. 2005. Light stability of spray-dried bixin encapsulated with different edible polysaccharide preparations. *Food Res. Int*. 38(9): 989-994.

Belghith, H., Chaabouni, S. E., and Gargouri, A. 2001. Stabilization of *Penicillium occitanis* cellulases by spray drying in presence of Maltodextrin. *Enzym. Microb. Technol*. 28: 253-258.

Beltra, A.L., Perez, C.J.J., Aparicio, J.A.R., and Lopez, G.G.F. 2005. Description of morphological changes of particles along spray drying. *J. Food Eng*. 67: 179-184.

Beristain, C.I., Anzuara, E., and Vernon, E.J. 2002. Effect of water activity on the stability to oxidation of spray dried encapsulated orange peel oil using gum arabic as wall material. *J. Food Sci.: Food Eng. Phys. Prop.* 67(1): 206-211.

Bhandari, B.R., Dumoulin, E.D., Richard, H.M.J., Noleau, I., and Lebert, A.M. 1992. Flavour encapsulation by spray drying. *J. Agric. Food Chem.* 49: 780-785.

Bleu, L.C. 2009. *Le Cordon Bleu Cuisine Foundations*. Cengage learning. p.213.

Bogracheva, T.Y., Meares, C., and Hedley, C.L. 2006. The effect of heating on the thermo-dynamic characteristics of potato starch. *Carbohyd. Polym.* 63: 323-330.

Bruman, H. 1948. The culture history of Mexican vanilla. *Hisp. Am. Hist. Rev*. 2: 360-376.

Buffo, R.A. and Reineccius, G.A. 2000. Optimization of gum acacia/modified starches/ maltodextrin blends for the spray drying of flavours. *Perfumer. Flavorist*. 25: 37-49.

Buffo, R.A., Probst, K., Zehentbauer, G., Luo, Z., and Reineccius, G.A. 2002. Effects of agglomeration on the properties of spray dried encapsulated flavours. *Flavour. Fragr. J.* 17: 292-299.

Buma, T.J. 1971. Particle structure of spray dried caseinate and spray dried lactose as observed by scanning electron microscopy. *Neth. Milk Dairy J.* 25: 278-283.

Cai, Y.Z. and Corke, V. 2000. Production and properties of spray- dried *Amarnthus* Betacyanin Pigments. *J. Food Sci.* 65(6): 1248-1252.

Caliskan, G. and NurDirim, S. 2013. The effects of the different drying conditions and the amounts of maltodextrin addition during spray drying of sumac extract. *Food Bioprod. Process.* 91:539-548.

Carneiro, H.C.F., Tonon, R.V., Grosso, C.R.F., and Hubinger, M.D. 2013. Encapsulation efficiency and oxidative stability of flaxseed oil microencapsulated by spray drying using different combinations of wall materials. *J. Food Eng.* 115: 443-451.

Claudio, A.F.M., Freire, M.G., Freire, C.S.R., Silvestre, A.J.D., and Coutinho, J.A.P. 2010. Extraction of vanillin using ionic-liquid-based aqueous two-phase systems. *Sep. Purifi. Technol*. 75: 39-47.

Coumans, W.J., Kerkhof, P., and Bruin, S. 1994. Theoretical and practical aspects of aroma retention in spray drying and freeze drying. *Dry. Technol.* 12(1/2):99-149.

Deis, R.C. 1997. Spray drying - innovative use of an old process. *Food Prod. Des*. 7(2): 136-148.

Desai, K.G.H. and Park, H.J. 2005. Recent developments in microencapsulation of food ingredients. *Dry. Technol*. 23: 1361-1394.

Desobry, S., Netto, F.M., and Labuza, T.P. 1997. Comparison of spray-drying, drum-drying and freeze-drying for β-carotene encapsulation and preservation. *J. Food Sci.* 62: 1158-1162.

deVos, P., Faas, M.M., Spasojevic, M., and Sikkema, J. 2010. Encapsulation for preservation of functionality and targeted delivery of bioactive food components. *Int. Dairy J.* 20(4): 292-302.

deZarn, T.G. 1995. Food ingredients encapsulation: An overview. In: Risch, S.J. and Reineccius, G.A. (eds), *Encapsulation and controlled release of food ingredients*. ACS symposium series, American Chemical Society, Washington, DC, pp.74-86.

Drusch, S. 2007. Sugar beet pectin: A novel emulsifying wall component for microencapsulation of lipophilic food ingredients by spray-drying. *Food Hydrocolloid.* 21(7): 1223-1228.

Ersus, S. and Yurdagel, U. 2007. Microencapsulation of anthocyanin pigments of black carrot by spray drier. *J. Food Eng*. 80: 805-812.

Falade, K.O. and Omojola, B.S. 2010. Effect of processing methods on physical, chemical, rheological, and sensory properties of okra (Abelmoschus esculentus). *Food Bioprocess. Technol*. 3 (3): 387-394.

Fazaeli, M., Djomeh, E.Z., Ashtari, A.K., and Omid, M. 2012. Effect of spray drying conditions and feed composition on the physical properties of black mulberry juice powder. *Food Bioprod. Process.* 90: 667-675.

Fernandes, L. P., Turatti, I.C., Lopes, N.P., Ferreria, J C., and Oliveira, W.P. 2008. Volatile retention and antifungal properties of spray-dried microparticles of Lippia sidoides essential oil. *Dry. Technol*. 26(12): 1534-1542.

Fernandes, R.V.B., Borges, S.V., and Botrel, D.A. 2014. Gum arabic/starch/ maltodextrin/inulin as wall materials on the microencapsulation of rosemary essential oil. *Carbohyd. Polym*. 101: 524-532.

Fogler, B.B. and Kleinschmidt, R.V. 1938. Spray drying. *Ind. Eng. Chem.* 30: 1372-1384.

Garza, B.C., Fernandez, Y.J., and Huerta, B.B.E. 2013. Thermal and pH stability of spray-dried encapsulated astaxanthin oleoresin from Haematococcus pluvialis using several encapsulation wall materials. *Food Res. Int.* 54:641-49.

Gharsallaoui, A., Roudaut, G., Chambin, O., Voilley, A., and Saurel, R. 2007. Applications of spray drying in microencapsulation of food ingredients: an overview. *Food Res. Int.* 40(9): 1107-1121.

Gibbs, B.F., Kermasha, S., Ali, I., and Mulligan, C.N. 1999. Encapsulation in the food industry: a review. *Int. J. Food Sci. Nutr.* 50:213-224.

Gleason, A.J. 2009. The state of Vanilla: challenges and opportunities. *Perfum. Flavor*. 34: 19-22.

Goud, K. and Park, H.J. 2005. Recent Developments in Microencapsulation of Food Ingredients. *Dry. Technol.* 23: 1361-1394.

Gouin, S. 2004. Microencapsulation: Industrial appraisal of existing technologies and trends. *Trends Food Sci. Technol*. 15: 330-347.

Goula, A.M. and Adamopoulos, K.G. 2010. A new technique for spray drying orange juice concentrate. *Innov. Food Sci. Emer. Technol*. 11: 342-351.

Hasheminya, S.M. and Dehghannya, J. 2013. Spray dryers: Applications, performance, essentialparts and classifications. *Int. J. Farming Allied Sci.* 19: 756-759.

Hassan, M. and Ahmed, J. 1998. Sensory quality of foam- mat dried pineapple juice powder. *Indian Food Pack.* 52 (7): 31-33.

Haidong, L., Fang, Y., Zhihong, T., Huanwei, S., and Tiehui, Z. 2012. Use of combinations of gum arabic, maltodextrin and soybean protein to microencapsulate ginkgo leaf extracts and its inhibitory effect on skeletal muscle injury. *Carbohyd. Polym.* 88: 435- 440.

Hogan, S.A., McNamee, B.F., O'Riordan, E.D., and O'Sullivan, M. 2001. Microencapsulation properties of sodium caseinate. *J. Agric Food Chem*. 49: 1934-1938.

Huezo, R.M.E., Islas, P.R., Barragan, P.L.A., Beristain, C.I., and Carter, V.E.J. 2004. Microencapsulation by spray drying of multiple emulsions containing caroteinoids. *J. Food sci. Food Eng. Phys. Prop.*. 69: 351-359.

Hundre, S.Y., Karthik, P., and Anandharamakrishnan, C. 2015. Effect of whey protein isolate and b-cyclodextrin wall systems on stability of microencapsulated vanillin by spray freeze drying method. *Food Chem*. 174: 16-24.

Imagi, J., Yamanouchi, T., Okada, K., Tanimoto, M., and Matsuno, R. 1992. Properties of agents that effectively entrap liquid lipids. *Biosci. Biotech. Biochem*. 56: 477-480.

Jackson, L.S. and Lee, K. 1991. Microencapsulation and the food industry. *Lebenesm Wissu. u. Technol*. 24: 289-297.

Jafari, S.M., Assadpoor, E., He, Y., and Bhandari, B. 2008. Encapsulation efficiency of food flavours and oils during spray drying. *Dry. Technol*. 26(7): 816–835.

Jager, L.S.D., Perfetti, G.A., and Diachenko, G.W. 2008. Comparison of headspace SPME-GC-MS and LC-MS for the detection and quantification of Coumarin, Vanillin, and ethyl vanillin in vanilla extract products. *Food Chem.* 107: 1701-1709.

Jain, P., Arora, S., and Rai, T. 1997. Flavour encapsulation and its application. *Beverage Food World*. 24(4): 21-24.

Kanakdande, D., Bhosale, R., and Singhal, R.S. 2006. Stability of cumin oleoresin microencapsulated in different combination of gum arabic, maltodextrin and modified starch. *Carbohyd. Polym.* 67: 536–541.

Kenyon, M.M. and Anderson, R.J. 1988. Maltodextrin and low dextrose equivalence corn syrup solids in flavour encapsulation. In: Risch, S.J. and Reinceccius, G.A. (eds), *Flavor encapsulation.* American Chemical Society, Washington, pp.7-11.

Keogh, M., Murray, C.A., and O'Kennedy, B.T. 2003. Effects of ultrafiltration of whole milk on some properties of spray-dried milk powders. *Int. Dairy J.* 13(12): 995-1002.

Keogh, M., O'Kennedy, B., Kelly, J., Auty, M.A., Kelly, P.M., and Fureby, A. 2001. Stability to oxidation of spray-dried fish oil powder microencapsulated using milk ingredients. *J. Food Sci.* 66: 217–224.

Kha, T.C., Nguyen, M.H., and Roach, P.D. 2010. Effects of spray drying conditions on the physicochemical and antioxidant properties of the Gac (Momordica cochinchinensis) fruit aril powder. *J. Food Eng.* 98: 385–392.

Khawla, A., Abuizza., Contreras, G.L., and Lu, R.D. 1996. Selection of better methed for the preparation of microspheres by applying hierarchy process. *J. Pharm. Sci*. 85: 572-575.

Kim, Y.D. and Morr, C.V. 1996. Microencapsulation of gum Arabic and several food proteins: Spray dried orange oil emulsion particles. *J. Agric. Food Chem*. 44(5): 1314-1320.

King, C.J. 1995. Spray drying: retention of volatile compound revisited. *Dry. Technol*. 13(5): 1221-1240.

Kneifel, W., Paquin, P., Abert, T., and Richard, J.P. 1991. Water holding capacity of proteins with special regard to milk proteins and methodological aspects: A review. *J. Dairy Sci.* 74(7):2027-2041.

Krishnan, S., Bhosale, R., and Singhal, R.S. 2005. Microencapsulation of cardamom oleoresin: Evaluation of blends of gum arabic, maltodextrin and a modified starch as wall materials. *Carbohyd. Polym.* 61: 95-102.

Kumar, A.S. 2004. *Vanilla*. In: Kerala Calling pp. 26-30.

Lago, V.E.S., do Nascimento, P., Fontes, E.A.F., Mauro, M.A., and Kimura, M. 2013. Edible coatings from native and modified starches retain carotenoids in pumpkin during drying. *Food Sci.Technol*. 50: 420-425.

Lamprecht, T., Pichlmayer, F., and Schmid, E.R. 1994. Determination of the authenticity of vanilla extracts by stable-isotope ratio analysis and component analysis by HPLC. *J. Agric. Food Chem*. 42(8): 1722-1727.

Lee, S.L. and Rosenberg, M. 2000. Whey protein-based microcapsules prepared by double emulsification and heat gelation. *Food Sci. Technol*. 33: 80-88.

Lezama, G.A.Y., Alvarez, D.L., Flores, J.M.E., Alonso, P.C., Niranjan, K., López, G.G.F., and Beltrán, A.L. 2012. Preparation and characterization of non-aqueous extracts from chilli (Capsicum annuum L.) and their microencapsulates obtained by spray-drying. *J. Food Eng*. 112: 29-37.

Lin, C.C., Lin, S.Y., and Hwang, L.S. 1995. Microencapsulation of squid oil with hydrophilic macromolecules for oxidative and thermal stabilization. *J. Food Sci.* 60: 36-39.

Liu, X.D., Atarashi, T., Furuta, T., Yoshii, H., Aishima, S., Ohkawara, M., and Linko, P. 2001. Microencapsulation of emulsified hydrophobic flavors by spray drying. *Dry. Technol*. 19 (7): 1361–1374.

Loksuwan, J. 2007. Characteristics of microencapsulated b-carotene formed by spray drying with modified tapioca starch, native tapioca starch and maltodextrin. *Food Hydrocoll.* 21: 928-935.

Madene, A., Jacquot, M., Scher, J., and Desobry, S. 2005. Flavour encapsulation and controlled release – a review. *Int. J. Food Sci. Technol*. 41: 1-21.

Mark, J.W., Dignum, J.K., and Rob, V. 2002. Vanilla curing under laboratory conditions. *Food Chem.* 79(2): 165-171.

Masters, K. 1991. *Spray Drying Handbook*. Longman Scientific and Technical, UK, 100p.

McKernan, W.M. 1992. Microencapsulation in the flavor industry. *Flavour Ind*. 4: 72-78.

McNamee, B., O'Riordan, E., and O'Sullivian, M. 1998. Emulsification and microencapsulation properties of gum arabic. *J. Agric. Food Chem.* 46(11): 4551-4555.

Mishra, P., Mishra, S., and Mahanta, C.L. 2014. Effect of maltodextrin concentration and inlet temperature during spray drying on physicochemical and antioxidant properties of amla (Emblica officinalis) juice powder. *Food Bioprod. Process.* 92: 252-258.

Mukerjea, R., Slocum., G., and Robyt, J.F. 2007. Determination of the maximum water solubility of eight native starches and the solubility of their acidic-methanol and -ethanol modified analogues. *Carbohyd. Res.* 342: 103–110.

Obon, J.M., Castellar, M.R., Alacid, M., and Lopez, F. J.A. 2009. Production of a red-purple food colorant from Opuntia stricta fruits by spray drying and its application in food model systems. *J. Food Eng.* 90: 471-479.

Onwulata, C I., Smith, P.W., and Holsinger, V.H. 1995. Flow and compaction of spray–dried powders of anhydrous butter oil and high melting milk fat encapsulated in disaccharides. *J. Food Sci.* 60: 836-840.

Palanisami, K., Paulraj, C., and Ali, A.M. 1997. National short term training on irrigation in Agriculture planning and budgeting. Water Technology Centre, Tamil Nadu Agricultural University, Coimbatore.

Papadakis, S.E. and King, C.J. 1988. Air temperature and humidity profiles in spray drying. Experimental measurements. *Ind. Eng. Chem. Res.* 27: 2116-2123.

Parize, A.L., Souza, T.C.R., Favere, I.M.V., Laranjeira, M.C.M., Spinelli, V., and Longo, E. 2008. Microencapsulation of the natural urucum pigment with chitosan by spray drying in different solvents. *Afr. J. Biotechnol.* 7(17): 3107-3114.

Parthasarathy, V.A. and Kandiannan, K. 2007. *Spices and Condiments [on-line]*. http://nsdlniscair.res.in/jspui/bitstream/123456789/478/1/revised%20spices%20and%20condiments.pdf [21 September 2007].

Patel, R.P., Patel, M.P., and Suthar, A.M. 2009. Spray drying technology: an overview. *Indian J. Sci. Technol.* 2(10): 44-47.

Perez, S.A., Odoux, E., Brat, P., Ribeyre, F., Jimenez, R.G., and Olvera, R.V. 2006. GC–MS and GC–Olfactometry analysis of aroma compounds in a representative organic aroma extract from cured vanilla (Vanilla planifolia G. Jackson) beans. *Food Chem.* 99: 728-735.

Priefert, H., Rabenhorst, J., and Steinbiichel, A. 2001. Biotechnological production of vanillin. *Appl. Microbiol. Biotechnol.* 56: 296-314.

Purseglove, J.W., Brown, E.G., Green, C.I., and Robbins, S. R. J. 1981. *Tropical Agricultural Series, vol. 2*. Longman Inc., New York, 735p.

Raja, K.C.M., Sankarikutty, B., Sreekumar, M., Jayalekshmy, A., and Narayanan, C.S. 1989. Material characterization studies of maltodextrin samples for the use of wall material. *Starch/ starke*. 41: 298-303.

Ranadive, A.S. 1994. Vanilla cultivation, curing, chemistry, technology and commercial products. In: Charalambous, G. (ed.), *Spices, herbs and edible fungi.* Elsevier Science, Amsterdam, pp. 517–575.

Ranganna, S. 1991. *Handbook of analysis and quality control for fruit and vegetable products*. Tata Mc-Graw Hill Publishing Co. Ltd, New Delhi, 56p.

Re, M.I. 1998. Microencapsulation by spray drying. *Dry. Technol.* 16: 1195-1236.

Reineccius, G.A. 1988. Spray-drying of food flavors. In: Reineccius, G.A. and Risch, S.J. (eds), *Flavor encapsulation*. American Chemical Society, Washington, pp.55-66.

Reineccius, G.A. 2004. The spray drying of food flavors. *Dry. Technol*. 22 (6): 1289-1324.

Reineccius, T.A., Reineccius, G.A., and Peppard, T.L. 2002. Encapsulation of flavors using cyclodextrins: comparison of flavor retention in alpha, beta and gamma types. *J. Food Sci*. 67: 3271-3279.

Rocha, G.A., Trindade, F C.S., and Grosso, F.C.R. 2012. Microencapsulation of lycopene by spray drying: characterization, stability and application of microcapsules. *Food Bioprod. Process.* 90: 37-42.

Rosenberg, M and Lee, S.L. 1993. Microstructure of whey protein/anhydrous milk fat emulsion. *Food Struct.* 12:267-274.

Rosenberg, M. and Sheu, T.Y. 1996. Microencapsulation of volatiles by spray drying in whey protein based wall systems. *Int. Dairy J.* 6: 273-284.

Rosenberg, M., Kopelman, I.J., and Talman, Y. 1985. A scanning electron microscopy study of microencapsulation. *J. Food Sci.* 50:130-144.

Rosenberg, M., Kopelman, I.J., and Talmon, Y. 1990. Factors affecting retention in spray drying microencapsulation of volatile materials. *J. Agric. Food Chem*. 38: 1288-1294.

Rosenberg, M. and Young, S. 1993. Whey proteins as microencapsulating agents. Microencapsulation of anhydrous milk fat. Structure evaluation. *Food. Struct*. 12(1): 31-41.

Rulkens, W.H. and Thijssen, H.A. C. 1972. The retention of organic volatiles in spray-drying aqueous carbohydrate solutions. *J. Food. Technol.* 7(1): 95-105.

Sankarikutty, B., Sreekumar, M.M., Narayanan, C.S., and Mathew, A.G. 1988. Studies on microencapsulation of cardamom oil by spray drying technique. *J. Food Sci. Technol*. 25: 352-357.

Shahidi, F. and Han, X.Q. 1993. Encapsulation of food ingredients. *Crit .Rev. Food Sci*. *Nutr*. 33: 501-547.

Shaw, L.A., McClements, D.J., and Decker, E.A. 2007. Spray-dried multilayered emulsions as a delivery method for ω-3 fatty acids into food systems. *J. Agric. Food chem.* 55: 3112-3119.

Sheu, T.Y. and Rosenberg, M. 1995. Microencapsules consisting of Whey proteins and Carbohydrates. *J. Food Sci. Eng. Process.* 63(3): 491-494.

Sheikh, J., Bhosale, R., and Singhal, R. 2006. Microencapsulation of black pepper oleoresin. *Food chem.* pp.105-110.

Shu, B., Wenli, Y., Yaping, Z., and Liu, X. 2005. Study on microencapsulation of lycopene by spray-drying. *J. Food Eng.* 86: 85-94.

Silva, P.I., Stringheta, P.C., Teófilo, R.F., and deOliveira, I.R.N. 2013. Parameter optimization for spray-drying microencapsulation of jaboticaba (Myrciaria jaboticaba) peel extracts using simultaneous analysis of responses. *J. Food Eng*. 117: 538-544.

Singh, J. and Singh, N. 2003. Studies on the morphological and rheological properties of granular cold water soluble corn and potato starches. *J. Food Hydrocoll.* 17: 63-72.

Sinha, A.K., Sharma, U.K., and Sharma, N. 2008. A comprehensive review on vanilla flavor: Extraction, isolation and quantification of vanillin and other constituents. *Int. J. Food Sci.Nutr*. 59: 299-326.

Solval, K.M., Sundararajan, S., Alfaro, L., and Sathivel, S. 2012. Development of cantaloupe (Cucumis melo) juice powders using spray drying Technology. *Food Sci. Technol*. 46: 287-293.

Soottitantawat, A., Ohkawara, F.B.M., Yoshii, H., Linko, P., and Furuta, T. 2005. Influence of emulsion and powder size on the stability of encapsulated D-limonene by spray drying. *Innovative Food Sci. Emerging Technol*. 6: 107-114.

Soottitantawat, A., Takayama, K., Okamura, K., Muranaka,D., Yoshii, H., and Furuta, T. 2005. Microencapsulation of L-menthol by spray drying and its release characteristics. *Innov. Food Sci. Emerg. Technol.* 6: 163-170.

Soottitantawat, A., Yoshii, H., Furuta, T., Ohgawara, M., and Linko, P. 2003. Micro-encapsulation by spray drying: Influence of emulsion size on the retention of volatile compounds. *J. Food Sci.* 68: 2256-2262.

Swapnali, V., Rajesh, B., and Rekha, S.S. 2003. Microencapsulation of cinnamon oleoresin by spray drying using different wall materials. *Dry. Technol.* 24: 983-992.

Tanimoto, M. and Matsuno, R. 1992. Properties of agents that effectively entrap liquid lipids. *Biosci. Biotechnol. Biochem*. 56: 477-480.

Tari, T.A. and Singhal, R.S. 2002. Starch based spherical aggregates: reconfirmation of the role of amylose on the stability of a model flavouring compound, vanillin. *Carbohyd. Polym*. 50: 279-282.

Varavinit, S., Chaokasem, N., and Shobsngob, S. 2001. Studies of flavor encapsulation by agents produced from modified sago and tapioca starches. *Starch Starke*. 53(6): 281-287.

Vega, C. and Roos, Y.H. 2006. Spray-dried dairy and dairy like emulsions compositional considerations. *J. Dairy Sci.* 89(2): 383-401.

Vilstrup, P. 2001. *Microencapsulation of food ingredients*. Leatherhead Publishing, Leatherhead, England. 56p.

Walton, N.J., Mayer, M.J., and Narbad, A. 2003. Molecules of interest Vanillin. *Phytochem*. 63: 505-515.

Watanabe, Y., Fang, X., Minemoto, Y., Adachi, S., and Matsuno, R. 2002. Suppressive effect of saturated acyl L-ascorbate on the oxidation of linoleic acid encapsulated with maltodextrin or gum arabic by spray-drying. *J. Agric. Food Chem.* 50: 3984-3987.

Xia, J.X., Yan, H.Y., and Jian, Y. 2011. Microencapsulation of sweet orange oil by complex coacervation with soybean protein isolate/gum arabic. *Food Chem.* 125: 1267-1272.

Xu, H. and Zhang, G. 2014. Slow digestion property of microencapsulated normal corn starch. *J. Cereal Sci.* 60: 99-104.

Yang, Z., Peng, Z., Li, J., Li, S., Kong, L., Li, P., and Wang, Q. 2014. Development and evaluation of novel flavour microcapsules containing vanilla oil using complex coacervation approach. *Food Chem.* 145: 272-277.

Yoshii, H., Soottitantawat, A., Liu, X., Atarashi, T., Furuta, T., Aishima, S., Ohgawara, M., Linko, P. 2001. Flavour release from spray dried maltodextrin /gum arabic or soy matrices as a function of storage relative humidity. *Innov. Food Sci. Emerg. Technol*. 2: 55-61.

Young, S.L., Sarda, X., and Rosenberg, M. 1993. Microencapsulation properties of whey proteins. 1. Microencapsulation of anhydrous milk fat. *J. Dairy Sci*. 76: 2868-2877.

Zakarian, A.J. and King, C.J. 1982. Volatiles loss in the zone during spray drying of emulsions. *Ind. Eng. Chem. Process Des. Dev.* 21: 107-113.

Zbicinski, I., Delag, A., Strumillo, C., and Adamiec, J. 2002. Advanced experimental analysis of drying kinetics in spray drying. *Chem. Eng. J.* 86: 207-216.

APPENDIX A

Quality Characteristics of Emulsion and Encapsulated Powder

Table A.1 Viscosity of vanilla extract emulsion

Wall material	Viscosity (cp)		
	Flavour load (w/w)		
	10	20	30
MD (100%)	2135	2346	2596
MD (75%): MS (25%)	2312	2498	2691
MD (25%): MS (27%)	2806	3045	3143
MS (100%)	3176	3214	3514

Table A.2 Stability of vanilla extract emulsion

Wall material	Stability (min)		
	Flavour load (w/w)		
	10	20	30
MD (100%)	89	73	60
MD (75%): MS (25%)	72	60	48
MD (25%): MS (27%)	59	43	30
MS (100%)	58	42	28

Table A.3 Emulsion particle size of vanilla extract

Wall material	Emulsion particle size (µm)		
	Flavour load (w/w)		
	10	20	30
MD (100%)	6.0	7.1	7.5
MD (75%): MS (25%)	5.3	6.2	7.3
MD (25%): MS (27%)	5.0	6.0	6.8
MS (100%)	4.8	5.5	5.9

Table A.4 Moisture content of encapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Moisture content, % (w.b.)		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	5.52	5.00	4.90
	180	5.20	4.90	4.20
	190	5.10	4.33	4.10
MD (75%): MS (25%)	170	4.96	4.53	4.29
	180	4.81	4.40	4.21
	190	4.38	4.20	4.15
MD (25%): MS (75%)	170	4.13	3.99	3.61
	180	3.96	3.73	3.59
	190	3.83	3.69	3.41
MS (100%)	170	3.36	3.33	3.20
	180	3.17	3.12	3.08
	190	3.13	3.09	3.06

Table A.5 Bulk density of encapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Bulk density (g/cm3)		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	0.4000	0.3600	0.3300
	180	0.3700	0.3300	0.3076
	190	0.3500	0.3200	0.3016
MD (75%): MS (25%)	170	0.4545	0.4445	0.4200
	180	0.4400	0.4360	0.4025
	190	0.3926	0.3545	0.3311
MD (25%): MS (75%)	170	0.4890	0.4745	0.4500
	180	0.4701	0.4650	0.4446
	190	0.4400	0.4310	0.4160
MS (100%)	170	0.5000	0.4840	0.4700
	180	0.4989	0.4750	0.4640
	190	0.4840	0.4550	0.4480

Table A.6 Wettability of encapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Wettability (s)		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	95.71	96.97	98.16
	180	95.11	96.05	97.14
	190	94.16	95.00	95.15
MD (75%): MS (25%)	170	77.90	80.65	81.62
	180	77.43	79.12	80.56
	190	76.25	78.67	79.90
MD (25%): MS (75%)	170	45.62	47.12	50.23
	180	44.65	45.81	48.92
	190	43.79	44.52	46.68
MS (100%)	170	35.15	35.18	35.20
	180	35.03	35.13	35.15
	190	34.99	35.10	35.13

Table A.7 Cold water solubility of encapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Cold water solubility (%)		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	76.68	80.10	85.72
	180	78.11	81.72	86.82
	190	79.73	83.48	89.30
MD (75%): MS (25%)	170	67.81	70.13	75.10
	180	68.38	72.37	77.93
	190	69.86	74.01	80.13
MD (25%): MS (75%)	170	49.36	52.83	56.00
	180	50.14	53.00	57.00
	190	52.15	54.17	59.00
MS (100%)	170	44.13	52.85	53.88
	180	47.11	52.99	54.19
	190	49.14	53.11	58.13

Table A.8 Hunter ‘L’ value of encapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Hunter ‘L’ value		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	93.80	93.47	92.85
	180	93.55	93.24	92.75
	190	93.23	93.01	92.69
MD (75%): MS (25%)	170	94.90	94.35	94.24
	180	94.59	94.01	93.64
	190	94.28	93.89	93.15
MD (25%): MS (75%)	170	95.56	95.1	94.80
	180	95.13	94.78	94.36
	190	95.05	94.58	94.02
MS (100%)	170	96.33	96.19	95.91
	180	96.30	96.01	95.55
	190	96.01	95.94	95.30

Table A.9 Hunter ‘a’ value of encapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Hunter ‘a’ value		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	-0.41	-0.43	-0.47
	180	-0.42	-0.45	-0.48
	190	-0.45	-0.46	-0.49
MD (75%): MS (25%)	170	-0.40	-0.45	-0.46
	180	-0.41	-0.46	-0.48
	190	-0.42	-0.48	-0.49
MD (25%): MS (75%)	170	-0.41	-0.42	-0.44
	180	-0.43	-0.45	-0.46
	190	-0.45	-0.46	-0.49
MS (100%)	170	-0.38	-0.41	-0.43
	180	-0.39	-0.42	-0.46
	190	-0.43	-0.44	-0.49

Table A.10 Hunter ‘b’ value of encapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Hunter ‘b’ value		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	5.79	6.74	6.68
	180	6.34	6.55	6.77
	190	6.38	6.57	6.82
MD (75%): MS (25%)	170	5.01	5.50	5.60
	180	5.31	5.83	6.18
	190	5.61	5.93	6.66
MD (25%): MS (75%)	170	4.33	4.77	5.06
	180	4.74	5.07	5.48
	190	4.80	5.26	5.79
MS (100%)	170	3.27	3.60	3.86
	180	3.35	3.76	4.19
	190	3.56	3.82	4.41

Table A.11 Encapsulation efficiency of encapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Encapsulation efficiency (%)		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	74.00	70.90	68.30
	180	74.05	72.00	70.20
	190	76.60	74.00	72.90
MD (75%): MS (25%)	170	62.00	61.01	59.32
	180	72.00	71.36	66.01
	190	73.10	72.85	70.00
MD (25%): MS (75%)	170	74.13	70.01	62.93
	180	75.98	73.83	68.78
	190	70.99	70.01	64.00
MS (100%)	170	76.70	73.91	70.01
	180	81.93	77.07	76.39
	190	69.00	68.93	66.00

APPENDIX B

Effect of Independent Variables on Dependent Variable

Table B.1 Effect of wall material and vanilla extract on emulsion viscosity

Wall material	Viscosity (cp)		
	Flavour load (w/w)		
	10	20	30
MD (100%)	2135^{i}	2346^{g}	2596^{f}
MD (75%): MS (25%)	2312^{h}	2498^{f}	2691^{e}
MD (25%): MS (27%)	2806^{e}	3045^{d}	3143^{c}
MS (100%)	3176^{c}	3214^{b}	3514^{a}

Treatments with even alphabets as superscript form a homogenous subgroup

Table B.2 Effect of wall material and vanilla extract on emulsion stability

Wall material	Stability (min)		
	Flavour load (w/w)		
	10	20	30
MD (100%)	89^{a}	73^{b}	60^{c}
MD (75%): MS (25%)	72^{b}	60^{c}	48^{f}
MD (25%): MS (27%)	59^{d}	43^{f}	30^{h}
MS (100%)	58^{e}	42^{g}	28^{i}

Treatments with even alphabets as superscript form a homogenous subgroup

Table B.3 Effect of wall material and vanilla extract on emulsion particle size

Wall material	Emulsion particle size (µm)		
	Flavour load (w/w)		
	10	20	30
MD (100%)	6.0^{e}	7.1^{g}	7.5^{h}
MD (75%): MS (25%)	5.3^{c}	6.2^{f}	7.3^{g}
MD (25%): MS (27%)	5.0^{b}	6.0^{e}	6.8^{f}
MS (100%)	4.8^{a}	5.5^{c}	5.9^{d}

Treatments with even alphabets as superscript form a homogenous subgroup

Table B.4 Effect of wall material, flavour load and inlet air temperature on moisture content of microencapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Moisture content, % (w.b.)		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	5.52[d]	5.00[d]	4.90[d]
	180	5.20[d]	4.90[d]	4.20[c]
	190	5.10[d]	4.33[c]	4.10[b]
MD (75%): MS (25%)	170	4.96[d]	4.53[d]	4.29[c]
	180	4.81[d]	4.40[c]	4.21[c]
	190	4.38[c]	4.20[c]	4.15[c]
MD (25%): MS (75%)	170	4.13[c]	3.99[b]	3.61[b]
	180	3.96[b]	3.73[b]	3.59[b]
	190	3.83[b]	3.69[b]	3.41[b]
MS (100%)	170	3.36[a]	3.33[a]	3.20[a]
	180	3.17[a]	3.12[a]	3.08[a]
	190	3.13[a]	3.09[a]	3.06[a]

Table B.5 Effect of wall material, flavour load and inlet air temperature on bulk density of microencapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Bulk density (g/cm3)		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	0.4000[b]	0.3600[c]	0.3300[d]
	180	0.3700[c]	0.3300[d]	0.3076[d]
	190	0.3500[c]	0.3200[d]	0.3016[d]
MD (75%): MS (25%)	170	0.4545[b]	0.4445[b]	0.4200[b]
	180	0.4400[b]	0.4360[b]	0.4025[b]
	190	0.3926[c]	0.3545[c]	0.3311[c]
MD (25%): MS (75%)	170	0.4890[b]	0.4745[b]	0.4500[b]
	180	0.4701[b]	0.4650[b]	0.4446[b]
	190	0.4400[b]	0.4310[b]	0.4160[b]
MS (100%)	170	0.5000[a]	0.4840[b]	0.4700[b]
	180	0.4989[a]	0.4750[b]	0.4640[b]
	190	0.4840[b]	0.4550[b]	0.4480[b]

Table B.6 Effect of wall material, flavour load and inlet air temperature on wettability of microencapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Wettability (s)		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	95.71^{d}	96.97^{d}	98.16^{d}
	180	95.11^{d}	96.05^{d}	97.14^{d}
	190	94.16^{d}	95.00^{d}	95.15^{d}
MD (75%): MS (25%)	170	77.90^{c}	80.65^{d}	81.62^{d}
	180	77.43^{c}	79.12^{d}	80.56^{d}
	190	76.25^{c}	78.67^{c}	79.90^{d}
MD (25%): MS (75%)	170	45.62^{b}	47.12^{c}	50.23^{c}
	180	44.65^{b}	45.81^{c}	48.92^{c}
	190	43.79^{b}	44.52^{b}	46.68^{c}
MS (100%)	170	35.15^{b}	35.18^{b}	35.20^{b}
	180	35.03^{a}	35.13^{b}	35.15^{b}
	190	34.99^{a}	35.10^{b}	35.13^{b}

Table B.7 Effect of wall material, flavour load and inlet air temperature on cold water solubility of microencapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Cold water solubility (%)		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	76.68^{b}	80.10^{b}	85.72^{b}
	180	78.11^{b}	81.72^{b}	86.82^{a}
	190	79.73^{b}	83.48^{b}	89.30^{a}
MD (75%): MS (25%)	170	67.81^{b}	70.13^{b}	75.10^{a}
	180	68.38^{b}	72.37^{a}	77.93^{a}
	190	69.86^{b}	74.01^{a}	80.13^{a}
MD (25%): MS (75%)	170	49.36^{d}	52.83^{c}	56.00^{b}
	180	50.14^{d}	53.00^{c}	57.00^{b}
	190	52.15^{c}	54.17^{c}	59.00^{b}
MS (100%)	170	44.13^{d}	52.85^{c}	53.88^{c}
	180	47.11^{d}	52.99^{c}	54.19^{c}
	190	49.14^{d}	53.11^{c}	58.13^{b}

Table B.8 Effect of wall material, flavour load and inlet air temperature on Hunter L value of microencapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Hunter 'L' value		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	93.80^{d}	93.47^{d}	92.85^{d}
	180	93.55^{d}	93.24^{d}	92.75^{d}
	190	93.23^{d}	93.01^{d}	92.69^{d}
MD (75%): MS (25%)	170	94.90^{c}	94.35^{c}	94.24^{c}
	180	94.59^{c}	94.01^{c}	93.64^{d}
	190	94.28^{c}	93.89^{d}	93.15^{d}
MD (25%): MS (75%)	170	95.56^{b}	95.11^{b}	94.80^{c}
	180	95.13^{b}	94.78^{c}	94.36^{c}
	190	95.05^{b}	94.58^{c}	94.02^{c}
MS (100%)	170	96.33^{a}	96.19^{b}	95.91^{b}
	180	96.30^{a}	96.01^{b}	95.55^{b}
	190	96.01^{b}	95.94^{b}	95.30^{b}

Table B.9 Effect of wall material, flavour load and inlet air temperature on Hunter a value of microencapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Hunter 'a' value		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	-0.41^{c}	-0.43^{d}	-0.47^{d}
	180	-0.42^{d}	-0.45^{d}	-0.48^{d}
	190	-0.45^{d}	-0.46^{d}	-0.49^{d}
MD (75%): MS (25%)	170	-0.40^{c}	-0.45^{c}	-0.46^{c}
	180	-0.41^{c}	-0.46^{c}	-0.48^{d}
	190	-0.42^{c}	-0.48^{c}	-0.49^{d}
MD (25%): MS (75%)	170	-0.41^{b}	-0.42^{b}	-0.44^{c}
	180	-0.43^{b}	-0.45^{c}	-0.46^{c}
	190	-0.45^{b}	-0.46^{c}	-0.49^{c}
MS (100%)	170	-0.38^{a}	-0.41^{b}	-0.43^{b}
	180	-0.39^{a}	-0.42^{b}	-0.46^{b}
	190	-0.43^{b}	-0.44^{b}	-0.49^{b}

Table B.10 Effect of wall material, flavour load and inlet air temperature on Hunter b value of encapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Hunter 'b' value		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	5.79^{c}	6.74^{d}	6.68^{d}
	180	6.34^{d}	6.55^{d}	6.77^{d}
	190	6.38^{d}	6.57^{d}	6.82^{d}
MD (75%): MS (25%)	170	5.01^{c}	5.50^{c}	5.60^{c}
	180	5.31^{c}	5.83^{c}	6.18^{d}
	190	5.61^{c}	5.93^{c}	6.66^{d}
MD (25%): MS (75%)	170	4.33^{b}	4.77^{b}	5.06^{b}
	180	4.74^{b}	5.07^{b}	5.48^{b}
	190	4.80^{b}	5.26^{b}	5.79^{c}
MS (100%)	170	3.27^{a}	3.60^{b}	3.86^{b}
	180	3.35^{a}	3.76b	4.19^{b}
	190	3.56^{b}	3.82^{b}	4.41^{b}

Table B.11 Effect of wall material and vanilla extract concentration and inlet air temperature on encapsulation efficiency of encapsulated vanilla extract powder

Wall material	Inlet air temperature (°C)	Encapsulation efficiency (%)		
		Flavour load (w/w)		
		10	20	30
MD (100%)	170	74.00^{b}	70.90^{c}	68.30^{c}
	180	74.05^{b}	72.00^{b}	70.20^{c}
	190	76.60^{b}	74.00^{b}	72.90^{b}
MD (75%): MS (25%)	170	62.00^{d}	61.01^{d}	59.32^{d}
	180	72.00^{b}	71.36^{c}	66.01^{d}
	190	73.10^{b}	72.85^{b}	70.00^{c}
MD (25%): MS (75%)	170	74.13^{b}	70.01^{c}	62.93^{d}
	180	75.98^{b}	73.83^{b}	68.78^{d}
	190	70.99^{c}	70.01^{c}	64.00^{d}
MS (100%)	170	76.70^{b}	73.91^{b}	70.01^{c}
	180	81.93^{a}	77.07^{b}	76.39^{b}
	190	69.00^{c}	68.93^{c}	66.00^{d}

Treatments with even alphabets as superscript form a homogenous subgroup

APPENDIX C

Estimation of Cost of Production of Microencapsulated Vanilla Extract Powder

Capacity of the Spray dryer	=	1000 g of water evaporation/ h
Working hour per shift	=	8 h
Number of shifts per day	=	2 shifts
Total capacity of the unit per day	=	16.0 kg/day
Cost of the Spray drier (S)	=	Rs. 12,00,000/-
Cost of high shear emulsifier (E)	=	Rs. 25,000/-
Cost of the electric balance (B)	=	Rs. 10,000/-
Life span of the unit (n)	=	15 years
Annual usage (A)	=	300 days
Interest rate (i)	=	11 % per annum

I. Fixed cost per year

A) Fixed cost of the Spray drier unit (C) $= \frac{i(i+1)^n}{(i+1)^n+1} \times S$

$= \frac{0.11(0.11+1)^{15}}{(0.11+1)^{15}+1} \times 12{,}00{,}000$

= Rs. 1,09,181/-

B)Fixed cost of the high shear emulsifier(E) $= \frac{i(i+1)^n}{(i+1)^n+1} \times E$

$= \frac{0.11(0.11+1)^{15}}{(0.11+1)^{15}+1} \times 25{,}000$

= Rs. 2275/-

C) Fixed cost of the electric balance (B) $= \frac{i(i+1)^n}{(i+1)^n+1} \times B$

$= \frac{0.11(0.11+1)^{15}}{(0.11+1)^{15}+1} \times 10{,}000$

= Rs. 910/-

Total fixed cost/ year = A + B + C

= 1,09,181 + 2275 + 910

= Rs. 1,12,366 /-

II. Variable cost per year

i) Repair and maintenance of spray dryer = 2 % of initial cost of the spray dryer

$= 12,00,000 \times \frac{2}{100}$

= 24000/-

ii) Repairs and maintenance of high shear emulsifier = 2 % of initial cost of the high shear emulsifier

$= 25,000 \times \frac{2}{100}$

= Rs.500/-

iii) Repairs and maintenance of balance = 2 % of initial cost of the grinder

$= 10,000 \text{ x } \frac{2}{100}$

= Rs. 200/-

Total Repair and maintenance charge = i + ii + iii

= 24000 + 500 + 200

= Rs. 24700/-

b) Cost of energy

Energy requirement (motor + heating coils) = 131.57 kWh / 16 h

Energy requirement of emulsifier and balance = 7.2 kWh / 16 h

Energy requirement

2 fan = 80 w/h

3 light = 120 w/h

2 Exhaust = 80 w/h

= 2.24 kWh / 16 h

Total energy requirement = 141.01 kWh / 16 h

Electricity charges = Rs. 5.85 / kWh

Electricity consumption charges = No. of days x Energy/day x Rate

	=	300 x 141.01 x 5.85
	=	Rs. 2,47,473

c) Labour charges

One women labour @ Rs. 400/shift	=	1 × 450 = 450
Labour charge /day	=	Rs. 900 / day
Cost of labour per year	=	900 x 300
	=	Rs. 2,70,000/-

d) Cost of raw materials

Vanilla extract

Cost of vanilla extract	=	Rs. 7500 / kg
Total quantity of vanilla extract required per day	=	1.44 kg (for two shifts)
Cost of vanilla extract for 300 days	=	1.44 x 7500 x 300
	=	Rs. 32,40,000 /-

e) Cost of maize starch

Cost of maize starch	=	Rs. 40 / kg
Total quantity of maize starch required per day	=	14.56 kg
Total quantity of maize starch required per year	=	14.56 x 300 kg
Cost of maize starch per year	=	14.56 x 40 x 300
	=	Rs. 174720 /-

Total variable cost of microencapsulated vanilla extract powder / year	=	a + b +c +d +e
	=	24,700 + 2,47,473+ 2,70,000 + 32,40,000 + 1,74,720
	=	Rs. 39,56,893 /-
Total cost for the production of micro-encapsulated vanilla powder / year (m)	=	Total fixed cost + Total variable cost
	=	1,12,366 + 39,56,893

	=	Rs. 40,69,259/-
Total production of microencapsulated vanilla extract powder / year (n)	=	16 × 300
	=	4800 kg/year
Cost of production of one kg of microenpasulated vanilla extract powder / year	=	$\frac{m}{n}$
	=	$\frac{40,69,259}{4800}$
	≈	**Rs. 850 /kg**

STUDIES ON MICROENCAPSUALTION OF VANILLA EXTRACT

by

SARIGA S

(2013 - 18 - 102)

ABSTRACT OF 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 & Technology

Kerala Agricultural University

Department of Food and Agricultural Process Engineering

KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY

TAVANUR , MALAPPURAM-679573

KERALA, INDIA

2015

ABSTRACT

Vanilla is one of the minor spices, most popular flavoring agent and second most expensive spice in the world. Vanillin flavour is highly volatile, heat sensitive and application in food incorporation is limited; this can be minimized by encapsulation technique with suitable wall material. The microencapsulation technique protects the vanilla extract from undesirable changes and coverts into a free flowing powder. Spray drying is the most common and commercial method for carry out the microencapsulation process. Therefore, to increase the storage stability of the microencapsulated vanilla extract powder, an investigation has been taken up to develop optimum process parameters to produce best quality microencapsulated vanilla extract powder. The microencapsulation of vanilla extract was carried out in tall type spray drier with twin fluid atomizer. Maltodextrin and maize starch were used as wall material. Different proportion of wall materials were used for the emulsification such as 100% maltodextrin, 100% maize starch, combination of 75% maltodextrin and 25% maize starch and 75% maize starch and 25% maltodextrin. The wall materials were emulsified with 10, 20 and 30% of vanilla extract for spray drying. The physico-chemical characteristics of vanilla extract and wall materials, and emulsion characteristics were carried out and the emulsions were spray dried at different inlet temperatures of 170, 180 and 190^{0}C. The encapsulated vanilla extract powder were collected and packed in aluminum foil and stored in room temperature for five months. The powder characteristics of encapsulated vanilla extract powders were carried out and analyzed. Based on the emulsion and powder characteristics, optimum conditions for the production of best quality encapsulated vanilla extract powder were found out. The study concluded that the encapsulated vanilla extract powder produced from 100% maize starch, vanilla extract concentration of 10% and inlet air temperature of 180^{0}C was found to be superior. The cost of one kilogram of optimised microencapsulated vanilla extract powder using spray drying technique was estimated to be Rs.850.

സംഗ്രഹം

ലോകത്തിലെ വളരെ പ്രശസ്തമായ ഒരു സുഗന്ധദ്രവ്യമാണ് വാനില. ഇവ യ്ക്ക് വിലയുടെ കാര്യത്തിൽ ലോകത്തിൽ രണ്ടാം സ്ഥാനമാണുള്ളത്. വാനിലയിലെ വാനിലിൻ എന്ന രാസവസ്തുവാണ് വനിലയിൽ നിന്നും സുഗന്ധം പുറപ്പെടുവിക്കുന്നത്. എന്നാൽ ഈ രാസവസ്തു അന്തരീക്ഷത്തിൽ അതിവേഗം അലിഞ്ഞുചേരുന്നതിനാലും താപത്തെ ചെറുത്തുനിൽക്കാൻ കഴിവില്ലാത്തതിനാലും ഭക്ഷ്യവസ്തുക്കളിൽ ഇതിന്റെ ഉപയോഗം വളരെ പരിമിതമാണ്. മൈക്രോഎൻകാപ്സുലേഷൻ എന്ന സാങ്കേതികവിദ്യ ഉപയോഗിച്ച്, അനുയോജ്യമായ ആവരണ വസ്തുക്കളാൽ ഈ പ്രശ്നം പരിഹരിക്കാവുന്നതാണ്. മൈക്രോഎൻകാപ്സുലേഷൻ എന്ന സാങ്കേതികവിദ്യ, വാനിലയുടെ സത്തിനെ അതിന്റെ ഭൗതികവും രാസപരവുമായ മാറ്റങ്ങളിൽ നിന്നും സംരക്ഷിക്കുന്നു. സാധാരണ രീതിയിൽ മൈക്രോഎൻകാപ്സുലേഷൻ എന്ന സാങ്കേതിക വിദ്യയ്ക്കായി സ്പ്രേ-ഡ്രൈയിംഗ് എന്ന പ്രക്രിയയാണ് ഉപയോഗിച്ചുവരുന്നത്. മൈക്രോഎൻകാപ്സുലേഷനിലൂടെ വാനിലയുടെ ഗുണമേന്മയുള്ള മികച്ച ഒരു ഉൽപ്പന്നം നിർമ്മിക്കുന്നതിനായി അതിന്റെ സംരക്ഷണസ്ഥിരത വർദ്ധിപ്പിക്കുന്ന മെച്ചപ്പെട്ട പ്രക്രിയാപരിധികളെ വികസിപ്പിച്ചെടുക്കാൻ ഒരു ഗവേഷണം നടത്തി. ഈ ഗവേഷണത്തിനായി ട്വിൻ-ഫ്ലൂയിട് അറ്റൊമൈസർ ഘടിപ്പിച്ചിരിക്കുന്ന ഉയരമുള്ള തരം സ്പ്രേ-ഡ്രൈയെറാണ് ഉപയോഗിച്ചിരിക്കുന്നത്. കൂടാതെ മാൾട്ടോഡെസ്ക്ടിനും ചോളത്തിൽ നിന്നുള്ള അന്നജവുമാണ് ആവരണവസ്തുക്കളായി തിരഞ്ഞെടുത്തിട്ടുള്ളത്. ആവരണവസ്തുക്കളെ അവയുടെ വത്യസ്ത അനുപാതത്തിലും (അതായത് 100% മാൾട്ടോഡെസ്ക്ടിൻ, 75% മാൾട്ടോഡെസ്ക്ടിന്റെയും 25% ചോളം-അന്നജത്തിന്റെയും മിശ്രണം, 25% മാൾട്ടോഡെസ്ക്ടിന്റെയും 75% ചോളം-അന്നജത്തിന്റെയും മിശ്രണം, 100% ചോളം-അന്നജം) 10, 20, 30% വാനില സത്തിന്റെയും മിശ്രിതമാണ് സ്പ്രേഡ്രൈയെറിലേക്ക് നൽകുന്നത്. വാനില സത്തിന്റെയും ആവരണ വസ്തുക്കളുടെയും ഭൗതികവും രാസപരവുമായ സ്വഭാവസവിശേഷതകളും ഇവയുടെ മിശ്രിത സ്വഭാവസവിശേഷതകളും കണ്ടുപിടിക്കുകയും ഈ മിശ്രിതം സ്പ്രേ-ഡ്രൈയറിൽ 170, 180, 190^{0}C എന്നീ താപനിലകളിൽ ഉണക്കി പൊടിയാക്കി വായു കടക്കാത്ത വിധം അലൂമിനിയത്തിൽ നിർമിതമായ സഞ്ചികളിലാക്കി സാധാരണ ഊഷ്മാവിൽ അഞ്ചുമാസം വരെ സംഭരിക്കുകയും ചെയ്തു. മിശ്രിതത്തിന്റെയും മൈക്രോഎൻകാപ്സുലേഷൻ വഴി ഉൽപാദിപ്പിച്ച വാനില പൊടിയുടെയും സ്വഭാവസവിശേഷതകളുടെ അടിസ്ഥാനത്തിൽ മികച്ച ഗുണമേന്മയുള്ള ഒരു ഉൽപ്പന്നം കണ്ടെത്തി. 100% ചോളം-അന്നജ ത്തിന്റെയും 10% വാനില സത്തിന്റെയും മിശ്രിതം 180^{0}C താപനിലയിൽ മൈക്രോഎൻകാപ്സുലേഷൻ എന്ന സാങ്കേതികവിദ്യയുടെ സഹായത്താൽ നിർമിക്കപ്പെട്ട ഉൽപ്പന്നമാണ് ഗുണമേന്മയിൽ മുൻപന്തിയിൽ നിൽക്കുന്നതെന്ന നിഗമനത്തിലാണ് ഈ പഠനം എത്തിച്ചേർന്നിരിക്കുന്നത്. ഈ ഉൽപ്പന്നത്തിന്റെ ഒരു കിലോഗ്രാമിന് ഏകദേശ ചിലവ് 850 രൂപയായി കണക്കാക്കപ്പെടുന്നു.