PRODUCTION OF KOKUM JUICE POWDER USING SPRAY DRYING TECHNOLOGY

By

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PROJECT REPORT

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DECLARATION

We hereby declare that this project report entitled **“PRODUCTION OF KOKUM JUICE POWDER USING SPRAY DRYING TECHNOLOGY”** is a bonafide record of project work done by us during the course of study and that the report has not previously formed the basis for the award of any degree, diploma, associateship, fellowship or other similar title of any other University or Society.

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Place: Tavanur

Date: 5-02-2016

CERTIFICATE

Certified that this project report, entitled, **"PRODUCTION OF KOKUM JICE POWDER USING SPRAY DRYING TECHNOLOGY"** is a record of project work done jointly by Ms. Alfonsa James, Ms. Amrutha K under my guidance and supervision and that it has not previously formed the basis for the award of any degree, fellowship, associate ship or other similar title of any other University or Society.

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Alfonsa James

Amrutha k

DEDICATED TO THE

FOOD ENGINEERING PROFESSION

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CHAPTER I

INTRODUCTION

Spices are mainly used for preserving, colouring and flavouring. Kokum or *Garcinia indica* is a minor spice and one of the 200 species in the genus Garcinia found in the Afro-Asian countries and one of the 300 species found in India. Kokum is a slender evergreen tree found in the tropical rainforests of India, particularly Maharashtra, Karnataka, Kerala, Western Ghats, Nilgiris in Tamil Nadu, South Gujarat, Goa, West Bengal and Assam. Kokum is important member of the family Clusiaceae. The flowers of kokum are unisexual, auxillary or terminal and solitary. The fruits are generally spherical, dark purple when ripen, encloses 5 to 8 large seeds. Kokum tree starts flowering during the month of November to December and fruits are harvested during April-May. The fruits of the tree have an agreeable flavor and a sweetish acid taste. Fruit have very short life of 2-3 days after ripening. Rind also has much commercial application such as colour pigment (2-3% anthocyanin), wine, and concentrate. Addition of kokum is supposed to enhance the taste of coconut based curries and to remove the unpleasant smell of mackerel and sardines. They are also used in some vegetable dishes and to prepare chutneys and pickles. Nowadays Kokum is valued for its numerous medicinal properties. The fruit is antihelminthics, cardiotonic, and useful in piles, dysentery, tumors, pains and heart complains. Syrup from juice is driven in bilious infections. The important compounds of Garcinia indica are hydroxycitric acid (HCA) and garcinol. Plant organic acids are hydroxycitric acid, oxalic, malic, citric, tartaric and acetic acid. Hydroxycitric acid (HCA) is a potent anti-obesity compound and reduces lipid and sugar level in blood. HCA lowers body fat level with no loss of body protein or lean mass in test animals that had been experimentally made obese.

Juice extracted from this fruit is sweet and sour and thus liked by many. A glass of cold juice is refreshing and it also improves the digestive system. Since it is natural fruit extract it is preferred by many people. Kokum juice is very popular in India especially in hot summer. It is delicious and has a cooling effect on body. It is refreshing drink. It not only quenches thirst, but also helps to prevent dehydration and sunstrokes due to heat. Kokum juice can be converted to powder form. This can add a new dimension in use of kokum juice as an alternative to kokum juice, reducing volume for transportation, increasing shelf life and convenience and usefulness in medicinal applications.

Nowadays, the fast economic development has changed the trend of food consumption from calories assurance to diet nutrient enrichment. The consumers today are well aware of the importance of vitamins. High moisture content in the food leads to having high water activity which leads the quality loss in foods by increasing the enzyme activity and microbial growth. Therefore, the reducing moisture content and water activity in fruits is always desirable in maintaing the quality. Drying is done to preserve food by removing moisture content and water activity. Among the drying techniques, spray drying is usually applied to produce the fruit juice powder. Dehydration by spray drying is used in the wide range of products in food industries to produce dry powders and agglomerates. Economic considerations of this method include hygienic conditions during processing, operational costs, and short contact time. The quality of spray dried food depends on the different factors of spray dryer operating systems.

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, by altering colloidal and surface properties, provides environmental protection and act as a barrier to avoid chemical reactions and to enable the controlled release of the coated materials. Microencapsulation with suitable wall material can protect the flavour from undesirable interactions with food, reduces off flavour, minimize the oxidation, increase shelf life, allows 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, 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.

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 and efficient. During this drying process, the evaporation of solvent, that is most often water, is rapid and the entrapment of the interest compound occurs quasi instantaneously. Spray drying microencapsulation process must rather be considered as an art than a science because of the many factors to optimize and the complexity of the heat and mass transfer phenomena that take place during the microcapsule formation (Gharsallaoui *et al*., 2007).

The wall material encapsulate 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.

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 corn 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. The inlet air temperature of spray drying also influences various physico-chemical characteristics of the product.

Considering the above cited facts, a study was undertaken with the following objectives:

1. Development of process protocol for microencapsulated kokum juice powder.

2. Standardization of spray drying parameters for kokum juice powder production.

3. Quality analysis of the kokum juice powder.

4. Storage studies of kokum juice powder.

CHAPTER II

REVIEW OF LITERATURE

This chapter deals with comprehensive review of the research work done by various research workers related to the present studies that gives the general information on *Garcinia indica*, its physiochemical characteristics, drying and powder optimization and its storage studies. This chapter provides the background information relevant to the understanding of the microencapsulation of kokum juice powder.

2.1 KOKUM (*Garcinia indica*)

2.1.1 *Origin and geographical distribution*

The kokum tree originated from the tropical rain forests of the Western Ghats of Kerala and Malaysia. Kokum is a tropical evergreen tree moderate to large in size. It is found at an altitude of about 800 meters from sea level. It is a slender tree with drooping branches. It grows to a height of 15-20 m. Its cultivation is confined to the costal hilly regions of Maharashtra and Goa states and is popularly known as ´Ratamba`. Some kokum trees are also observed in Tamil Nadu, the Western Ghats of Karnataka and Kerala, as well as part of West Bengal, Assam and Gujarat (Parle and Dhamija, 2013). It also flourished well on the lower slopes of the Nilgiri hills. Kokum butter is exported to Japan, Taiwan, USA, etc from India for use in confectionery.

2.1.2 *Botanical aspects*

The kokum fruit is globose or spherical, 2.5 to 3.75 cm in diameter, dark purple when ripe, enclosing 5-8 large seeds, and the pulp is juicy, white, and delicious in taste and odour. The berry encapsulated by a tough rind, sits on top of the calyx. The seed is about one fourth of total weight of fruit. An average kokum tree bears hundreds of fruits, during summer. When they are tender, they are green in colour. The beautiful purple colour is attained on maturation. The fruit has 7-10 ridges and it takes 4-5 months to ripen. The tree takes about 8-10 years to reach the commercial bearing stage, when grown from seeds. The fruits are edible with an agreeable flavour and sour- sweetish taste inducing a cool feeling (Parle and Dhamija, 2013).

2.1.3 *Fruit composition*

The important compounds of Garcinia indica are hydroxycitric acid (HCA) and garcinol. Plant organic acids are hydroxycitric acid, oxalic, malic, citric, tartaric and acetic acids as given in Table 2.1 (Parthasarathy *et al*., 2012).

Compound	**gm wt %**	**Boiling point (^{0}C)**	**Melting point (^{0}C)**
Hydroxycitric acid	6.13	393	176-178
Malic acid	1.67	322	101-104
Oxalic acid	0.63	149-160	101-102
Citric acid	0.79	175	153
Tartaric acid	0.51	399.3	168-170
Acetic acid	0.31	118	16

Table 2.1 Organic acid profile of *Garcinia indica*

Studies have shown that the rind contains moisture (80.0 g/100 g), protein (1%), tannin (1.7%), pectin (0.9%), Total sugars (4.1%) and fat (1.4%). The seed is very rich in stearic, oleic and stearic triglycerides Phytochemical studies have shown that when compared with any other natural sources, kokum rind contains the highest concentration of anthocyanins (2.4 g/100 g of kokum fruit). The composition of fresh Kokum rind is shown in Table 2.2. (Lamture and Bhatkhande, 2014).

Components	Percentage
Moisture content	80.0
Protein	1.9
Crude fibre	14.3
Total ash	2.6
Tannin	2.9
Pectin	5.7
Starch	1.0
Crude fat	10.0
Acid	22.8
Pigment	2.4
Ascorbic acid	0.06
Carbohydrate	35.0

Table 2.2 Composition of fresh kokum rind

2.1.4 *Utility*

Pritam (2013) studied about the utility of kokum as a culinary agent and is used as an acidulant for curries by people living in south India especially Maharashtra, costal Karnataka and Goa. In summer the ripe rinds are ground in a blender with sugar and cardamom and consumed as a cooling drink. Addition of kokum is supposed to enhance the taste of coconut-based curries and to remove the unpleasant smell of mackerel and sardines. They are also used in some vegetable dishes and to prepare chutneys and pickles.

In the ayurvedic system of medicine, kokum is used to treat illness related to obesity and multiple studies have shown that hydroxycitric acid (also known as Garcinia acid) a component of kokum is reported to possess anti obesity effects. Studies have shown that consumption of hydroxycitric acid reduces appetite, inhibits fat synthesis, lipogenesis, decreases food intake and reduces body weight (Jayaprakasha and Sakariah, 2002). It also inhibits synthesis of fatty acid and lipogenesis from various precursors (Jena, 2003). Concomitantly, it also increases the synthesis of hepatic glycogen thereby activating the glucoreceptors and causing a sensation of reduced appetite and fullness (Preussa *et al*., 2004). Hydroxycitric acid is non toxic as experimental studies have shown that by oral route it did not cause death or systemic or behavioral toxicity even at high dose (Jena, 2003). Kokum is an underutilized fruit, it can be preserved by making it as a pulp to use it for the preparation of RTS (Ready- To- Serve) Beverage.

Parle *et al*. (2013) studied the use of kokum in pharmacological activities. Hexane and benzene extracts of the rinds of kokum and its active constituent Garcinol possess powerful antibacterial activity of its own. Aqueous extract of kokum rind is reported to have highest antibacterial activity against *Baccilus subtilis,* followed by E coli, *Enterobacter aerogenes* and *staphylococcus aureus*.

Varalakshmi *et al*. (2010) demonstrated antifungal activity of aqueous extract of kokum rind against *Candida albicans and penicillium sp.* Chloroform extract of kokum rind inhibited the growth of *Aspergillus flavus* and production of aflatoxin.

Kokum pigments are useful in treating skin disorders, due to UV light absorbing properties. Methanolic extract of kokum fruit exhibited significant neuroprotective potential against 6-OHDA, indicating its anti Parkinson’s activity in rats (Khatib *et al*., 2010). It was also found to have anti cholinesterase property.

Aqueous and Ethanolic extract of kokum rind elicited ulcer protective activity against indomethacin the whole fruit extract of kokum significantly lowered fasting blood glucose levels in streptozotocin induced hyperglyceimic rats in acute as well as chronic study. The methanolic extract of the dried fruit of kokum showed remarkable anti hyperlipidemic activity in rats using cholesterol induced hyperlipidemic model.

Parle *et al*. (2013) reported the traditional uses of kokum as Digestive, Antacid, Anti dysentery, Anti diarrheal, Anti piles, Anti ulcer, Anti colic, Anti obesity, Anti helmintic, Anti asthmatic, Cardiotonic, Hepatoprotective, Anti tumour, Anti hyperplasia, Wound healer, Analgesic, Anti inflammatory, Anti dermatitis, Anti perspirant, Astringent, Demulcent, and Emllient.

Krishnamurthy *et al*. (1982) conducted study on industrial uses of kokum fruit. Kokum fruit appears to be a promising industrial raw material for commercial exploitation, due to its chemical constituents. Kokum fats have been reported to be used in chocolate and confectionary preparations. It is also used in manufacture of soaps, candles, and ointments.

Darji *et al*. (2010) analyzed culinary effects of kokum. Dried kokum rinds are widely used in seasoning, as they impart a sweetish-tangy flavour to the food. The kokum is extracted by soaking the rind in hot water. This juice is either consumed alone or mixed with spices. Dry rind of kokum is used as a substitute of tamarind, vinegar, and lime juice. It gives sour taste to curries and gravies.

2.3 Spray drying

The first spray dryers were manufactured in USA in 1933. Spray drying is one of the best drying methods to convert directly the fluid materials into solid or semi solid particles (Dolinsky, 2001). Spray drying is a unit operation by which a liquid product is atomized in a hot gas current to instantaneously obtain a powder. The gas generally used is air or more rarely an inert gas, particularly nitrogen gas.

The initial liquid feeding can be a solution, an emulsion or a suspension (Gharasallaoui, 2007). It can be used to both heat resistant and heat sensitive products. It is one of the best drying methods to convert directly the fluid materials into solid or semi-solid particles (Murugesan and Orsat, 2011). Spray drying is a unit operation by which a liquid product is atomized in a hot gas current to instantaneously obtain a powder. Spray drying is a technique widely used in the food industry to produce food powder due to its effectiveness under the optimum condition.

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

According to (Barbosa-Canovas *et al*., 2005; Masters, 1991), Spray drying belongs to the family of suspended particles systems as drying is accomplished while the particles are suspended in air. Spray drying is by definition the transformation of a feed from a fluid state into dry particulate form by spraying the feed into a hot drying medium. It is a one step, continuous particle processing involving drying. The drying of liquid food is often accomplished in a spray dryer. Moisture removal from a liquid food occurs after the liquid is atomized or sprayed into heated air within a drying chamber, various configuration of the chamber are used.

The spray drying parameters such as inlet temperature, air flow rate, feed flow rate, atomizer speed, types of carrier agent and their concentration are influencing as particle size, bulk density, moisture content, yield and hygroscopicity in spray dried foods (Chegini and Ghobadian, 2008). In the spray drying process, due to the large surface area of the small droplets, drying takes place rapidly (1-10 seconds). As a result, it is highly recommended for heat sensitive foods (Fellows, 2000; Tang and Yang, 2004; Ramaswamy and Marcotte, 2006). Furthermore, several other advantages of spray drying can be found in that the drying process is continuous, easy and entirely automatically controlled. Importantly, the quality of final powders will not be variable from one batch to another when spray drying conditions remain unchanged (Masters, 1991). However, installation costs, thermal efficiency, waste heat and exhaust-air handling are the key drawbacks of the spray drying process (Barbosa-Canovas and Vega-Mercado, 1996).

Among various methods of preparing dehydrated products, spray drying is the most important one. Spray drying delivers a powder of specific particle size and moisture content in relation to the drier capacity or product's heat sensitivity. In a continuous operation it delivers a highly controlled powder quality with relatively easy manipulation. Accordingly the objective of spray drying is to produce a spray of high surface to mass ratio droplets (ideally of equal size) and then to uniformly and quickly evaporate the water Evaporation keeps the products temperature to a minimum, which in turn reduces the chance of high temperature deterioration of the products. Further, spray drying minimizes loss of volatile flavours as against other dehydration techniques. Spray drying consists of five process stages.

1. Concentration of fruit juice

2. Atomization

3. Droplet-air contact (mixing and flow)

4. Drying of droplets (moisture/volatiles evaporation)

5. Separation of dried product from the air

Huntington (2004) stated that the flexibility of drier designs provides opportunities to produce the powders that consistently meet industrial specifications. The production capacity can be expanded to over 25 tonnes of product per hour (Masters, 2004). The process is continuous and easily automated which can reduce labour cost (William, 1971). There are less sticking and corrosion problems in spray drying if the material does not come in contact with the equipment walls until it is dry (Gupta, 1978).

Spray drying involves in the complex interactions of process, apparatus and feed parameters which all have an influence on the final product quality (Chegini *et al*., 2008). The spray drying process can produce a good quality final product with low water activity and reduce the weight, resulting in easy storage and transportation. The solid is usually collected in a drum or cyclone (Maltini, 1986). Spray drying is used to produce a wide range of products including heat sensitive materials (Mahendran, 2010).

The products produced by spray drying include: pharmaceutical, such as antibiotics, analgesics, vaccines, vitamins and catalysts; chemicals, such as, carbides, ferrite, nitrides, tannins, fine organic/inorganic chemicals detergent and dye stuffs; ceramic, including advanced ceramic formulations; and foods such as, milk and milk products, food colour, food supplement, soup mixes, spice and herb extracts, coffee, tea and sweetener. Masters (2004) spray dried food products are appealing, retain nutritional qualities and are convenient to Consumer.

According to Masters (2004) spray drying has been considered as a solution for conventional drying problems because the process has usually proved not only efficient but also economic. The main factors in spray drying that must be optimized are feed temperature, inlet air temperature, and outlet air temperature (Liu *et al*., 2006).

Powder properties such as moisture content, bulk density, particle size, hygroscopicity and morphology were affected by inlet temperature. Normally, the inlet temperature uses for spray drying technique for food powder is 150 - 220^{o}C. Chegini and Ghobadian (2008) studied the effect of inlet temperature (110-190^{o}C) on the moisture content of orange juice powder. It was found that at a constant feed flow rate, increasing the inlet air temperature reduced the residual moisture content.

The inlet air temperature has a role on the hygroscopicity of the powder. Tonon *et al*. (2008) studied the effect of inlet temperature (140, 170, 200^{o}C) on the hygroscopicity of acai juice powder. An increase on inlet air temperature increases the time of wettability, insoluble solids and decreases the bulk density and moisture content of the powder. Density of food powders is so fundamental to its storage, processing, and distribution that it merits particular consideration.

Fruit juice powder obtained by spray drying might have some problems with their property, such as stickiness, hygroscopicity and solubility, due to the presence of low molecular weight sugars and acids, which have a low glass transition temperature (Bhandari *et al*., 1993).

Sahin *et al*. (2013) suggested that, higher inlet temperature produced lighter product than lower inlet temperature. Higher degree of lightness of spray dried powder at higher inlet temperature indicates that the pigments have been lost due to oxidation.

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.

Brennan *et al*. (2007) studied on spray drying of concentrated orange juice, on a laboratory scale and some of the factors affecting the process. In his study concentrated orange juice (a) without additives and containing (b) sodium carboxymethyl cellulose, (c) gum acacia, and (d) liquid glucose as additives were spray dried in a laboratory drier. Liquid glucose was found to be the most satisfactory additive, producing a powder with good percentage, free-flowing characteristics and a minimum of wall deposition. Variations in air inlet temperature, feed temperature and rate and atomizer speed, within a limited range, resulted in no significant changes in the bulk density and particle size of the product. The higher temperatures resulted in some change in colour and an increase in insoluble solids. They concluded that cooled plate experiments indicated that the problem of wall deposition is related to wall temperature and is minimized when the wall temperature is below the sticky point temperature of the product.

Jaya and Das (2007) stated relationship of moisture content, glass transition temperature and sticky point temperature of vacuum dried mango, pineapple and tomato with added maltodextrin and tricalcium phosphate). In that study, the ratios of maltodextrin (DE 38): fruit pulps were at 0.093:1, 0.065:1 and 0.033:1 respectively. The tricalcium phosphate at 0.015:1 was used for ant caking in the three types of vacuum dried powder. The difference between glass transition temperature and sticky point temperature however were found from 2.5 to 15.5°C depended on the nature of raw materials and amount of maltodextrin. The difference of these two temperatures was also found to vary with moisture contents. For pineapple powder, the glass transition and sticky point temperature appeared to be very close to each other (minimum difference of around 2.5°C).

Chegini *et al*. (2008) reported that the sticky point temperature of orange juice powder using maltodextrin, liquid glucose and methylcellulose as carriers was found to be at around 44°C at 2% moisture.

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 influence the bulk density due to the increase of powder’s porosity. The solubility of the powder increased with decrease in bulk density.

Deis (1997) stated 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.

2.4 Microencapsulation by spray drying

According to Reineccius (1988), 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.

Spray drying is the most common and cheapest technique to produce microencapsulated food materials. Compared to freeze drying, the cost of spray drying method is 30–50 times cheaper (Desobry *et al*., 1999). Spray drying has been considered as a solution for conventional drying problems because the process has usually proved not only efficient but also economic (Masters, 1968).

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 feed emulsion, and dehydration of the atomized particles.

Tari and Singhal (2002) has mentioned 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.

According to Masters (1991), 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

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 anthocyanin and maintained the colour and stability during storage.

Currently maltodextrin is the most widely used additive to obtain fruit juice powders, since it satisfies the demands and is reasonably cheap (Bhandari *et al*., 1993). Stickiness is a major reason which has limited the use of spray drying for sugar rich and acid rich foods. On the other hand, the stickiness was not encountered when less hydrolyzed starch derivatives such as maltodextrin were spray dried; instead, they facilitated the spray drying process of the sugar rich foods. Hence, they are frequently used as drying aids.

Maltodextrins are digestible carbohydrates made from several different cereal sources, including corn, potato, rice and tapioca. The processes to produce maltodextrins involve cooking of starch followed by acid and/or enzymatic hydrolysis to break the starch into smaller chains. These chains are composed of several oligosaccharides molecules along with polysaccharides of larger molecular weight (Avaltroni *et al*., 2004).

Cai and Corke (2000) studied about the usage of maltodextrin as wall material for production of Amaranths betacyanin pigment significantly reduced the hygroscopicity of the betacyanin extracts enhancing their storage stability.

Jaya (2004) mentioned that, maltodextrin addition improved mango juice powder hygroscopicity, caking, and solubility, whereas it deteriorated slightly its moisture content and density. However, in experiments conducted using humidified air, powder moisture content was much higher and bulk density was much lower.

Dolinsky *et al*. (2000) suggested that 30-55% maltodextrin should be added to the fruit and vegetable juice in order to obtain the powder. Adhikari *et al.* (2003) reported that the addition of maltodextrins significantly reduced the stickiness of fructose solutions, showing its use as an effective drying aid.

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.

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).

2.5 Quality characteristics

Poduval (2002) suggested that the quality standards are of great importance in facilitating both national and international trade. Quality is an increasingly important factor in the production and marketing of biological products. The term ´Quality` is one of the most defined terms in use in food industry today.

2.5.1 Moisture content

According to (Pomeranz and Meloan, 1994), drying is the standard method for determining the moisture content of materials. The material is heated under carefully specified conditions and the loss of weight is taken as a measure of the moisture content of the sample. Drying methods are simple, relatively rapid, and provide the simultaneous analyses of large numbers of samples.

The residual moisture content of spray-dried samples was determined by oven drying the powders at 102°C, determining the difference in weight, and expressing the weight loss as a percent of the initial powder weight ((IS: SP: 18 (part XI), 1981). An analysis of moisture content is necessary for quality control of dry food products.

According to Mishra (2004), higher MD concentrations shows a sound effect to down the moisture trend of spray dried powder owing to the fact that the addition of MD that increased the total solids of the feed and reduced the amount of water vaporization. It was found that at a constant feed flow rate, increasing the inlet air temperature reduced the residual moisture content.

2.5.2 pH and titrable acidity

pH values give a measure of the acidity or alkalinity of a product, while titrable acidity gives a measure of the amount of acid present. Assessment of pH and titrable acidity of banana, cooked banana and plantain are used primarily to estimate consumption quality and hidden attributes. They could be considered as indicators of fruit maturity or ripeness.

Dadzie and Orchard (1996) reported that acids make an important contribution to the post harvest quality of the fruit, as taste is mainly a balance between the sugar and acid contents, hence post-harvest assessment of acidity is important in the evaluation of the taste of the fruit.

Ingham and Uljas (1999) stated that temperature and pH interact to form barriers to the survival of certain pathogens.

According to (Anjineyulu *et al*. 2014), addition of maltodextrin has significant role in increasing the pH.

(Luh and Woodruff, 1975) concluded that the acid hydrolysis of polysaccharides can cause changes in pH during storage. Temperature has no considerable effect in pH on storage days.

2.5.3 Wettability

The reconstitution properties (wettability, solubility and dispersibility) of skim milk powder is having particular importance to manufacturers and consumers as a benchmark of consumption quality and also has a direct impact on their perception of the overall product quality. Wettability (or wetting time) was determined by placing 3 g of dried powder around a pestle inside a funnel so that the pestle blocks the funnel opening. Then, the pestle was lifted to allow the powder to flow though the stem into a beaker of water. As soon as all the powder had flown into the beaker of water, a stop watch was started. The time (s) taken for complete wetting of the powder was noted as the wetting time. The experiment was done in triplicate (Falade and Omojola, 2010). As the temperature increases, the time required for wetting of the powder increases which implies the wettability of the powder decreases. This may be due to reduced product residual moisture content. This was in accordance with the findings of Bhandari *et al.* (1993) and Jumah *et al*. (2000). The changes in wettability can be due to the difference in particle size with the addition of maltodextrin (Anjineyulu *et al*., 2014).

2.5.4 TSS

Total soluble solids (TSS) indicates soluble solid content of banana flour, and high TSS has been associated with high sucrose content in banana pulp. The addition of the maltodextrin has increased the total solids of the feed and reduced the amount of water evaporation. This was in accordance with the findings of Anjineyulu *et al*. (2014) in spray dried dahi powder. Maltodextrins could alter the surface stickiness of low molecular weight sugars such as glucose, sucrose and fructose and organic acids, therefore, facilitated drying, reduced the stickiness and increased the soluble solids of the spray dried product.

Luh and Woodrff (1975) reported that the increase in TSS during storage may be due to acid hydrolysis of polysaccharides especially gums and pectin. The addition of the maltodextrin has increased the total solids of the feed and reduced the amount of water evaporation.

2.5.5 Bulk density

Goula and Adamapoulos (2008) described a method for the bulk density in which Bulk density (g/ml) was determined by gently adding 2 g of powder into an empty 10 ml graduated cylinder and holding the cylinder on a vortex vibrator for 1 minute. The ratio of the mass of the powder and the volume occupied in the cylinder determines the bulk density value.

Cai and Corke (2000) conducted an experiment on microencapsulation of amaranths 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.

2.5.6 Water activity

Water activity (a_w) is defined as the ratio of vapour pressure of water in a system to the vapour pressure of pure water or the equilibrium relative humidity of the air surrounding the system at the same temperature. It is a function of moisture and temperature of food. Most fresh foods can be considered as high-moisture foods and their shelf life is largely controlled by the growth of microorganisms. High moisture foods have a_w of 0.90 to 0.99 and they usually contain more than 50% w/w water (Guzman *et al*., 1974).

2.5.7 Particle size

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).

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^{0}C and 200/173^{0}C) in which smooth and regular surfaces of complete and broken material were observed.

2.5.8 Colour

Colour is the important factor which affects the quality of the product. The addition of bulking agents can affect the colour of the product. According to Grabowski *et al*. (2006), the maltodextrin concentration can impart whiteness to the powder. Abadio *et al*. (2004), based on the work done in pineapple juice powder concluded that maltodextrin because of its white color imparts a greater lightness of powders, represented by a higher L* value, was obtained at higher concentrations of maltodextrin. .

2.5.9 Packaging and storage of spray dried products

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.

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 Kokum juice powder, the experimental plan and procedure for optimization of the process parameters.

3.1 Collection of sample

The fresh kokum fruits were collected from Anakayam research station and CPCRI, Kasargod.

3.2 Pre-Preparation of the Selected Kokum

Grading of fruit was done based on soundness, firmness, cleanliness, size, maturity, weight, color, shape and freedom from foreign matters, insect damage and mechanical injury. Sorting and grading is essential to get suitable quality of fruit which was done by hand. The fruits were first washed to remove the dirt.

3.3 Extraction and preparation of juice

From the graded kokum the juice was extracted manually. Rinds and pulp were selected for juice extraction. It was homogenized in a mixer to obtain fine juice. Then the juice was filtered and mixed with maltodextrin. Maltodextrin was used as an encapsulating agent.

3.4 Micro encapsulation by Spray drying

The Microencapsulation was carried out in Tall type spray dryer with two fluid nozzles. 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 190^oC to 200^oC and the blower speed was adjusted to 1700 rpm. Compressed air was introduced in the two fluid nozzles.

The function of compressed air is to disperse the solution or slurry in fine mist. Hot air is sucked by blower through the main chamber from the ceiling which is intermixed with the mist resulting in evaporation of water and formation of dry particles. The tiny dried particles during its moist stage agglomerates to larger particles and drop in the bottom. Smaller particles is collected in the cyclone and collected in the glass product jar placed below the cyclone. Moist humid air is finally sucked by the blower and exits to atmosphere. The microencapsulated kokum juice 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.

Plate 3.1 Spray dryer

3.5 Physicochemical analysis

3.5.1 Moisture content

Moisture content of the sample was determined by weighing accurately about 5g of Kokum powder into a flat bottomed glass or aluminium dish (with a cover) previously dried and weighed and heat the dish containing the material (after uncovering) in an electric oven maintained at 100^0C for about 3h. Cool the sample in a dessiccator and weigh with the cover on. Repeat the process of drying, cooling and weighing at 30 min intervals until the difference between two consecutive weighing is less than 1mg. Preserve the dish containing this dried material in a dessicator for the determination of other contents. The moisture content of the samples was calculated on a percent wet basis, and the average value of the triplicate measurements was used.

Moisture content in % wet basis = (M initial – M final)/M initial

3.5.2 Total soluble solids

Total soluble solid (TSS) was measured using a hand refractometer. It is expressed in degree Brix. Kokum powder was mixed with water and allows the sample to settle. For TSS measurement one or two drops of the prepared sample were placed on the hand refractometer and reading was noted.

Plate 3.2 Hand refractometer for TSS measurement

3.5.3 pH

pH being the logarithm of the reciprocal of hydrogen ion concentration, is a measure of active acidity which influence the flavour or palatability of a product and also affects its processing requirements. The pH of the kokum powder samples was determined using a digital pH meter. The pH of maltodextrin and maize starch were determined using a digital pH meter. Before determining pH, the pH meter was standardized with double distilled water of pH 7.0 and standards of pH 4.0, 7.0 and 9.0.

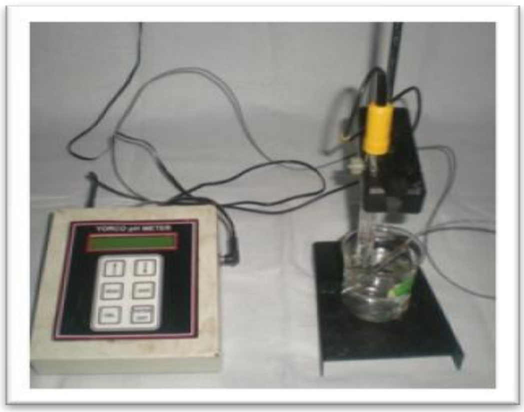


Plate 3.3 YORCO pH meter, model: YSI – 601

3.5.4 Water activity

The water activity of kokum juice powder was determined using Aqua Lab water activity meter. It is the fastest instrument for measuring water activity. Its readings are precise, providing ± 0.003 accuracy.

For the water activity determination, kokum powder is filled in the disposable cups of the water activity meter and the sample drawer knob is turned to OPEN position and the drawer is opened. The prepared sample is then placed in the drawer. Checked the top lip of the cup to make sure it is free from sample residue (an over filled sample cup may contaminate the chamber‘s sensors). After placing the sample, turned the sample drawer knob to the READ position. The a_w of samples were noted from the LCD display of the water activity meter.

Plate 3.4 Aqua lab water activity meter

3.5.5 Wettability

Wettability is the time taken for the complete wetting of the powder. It was determined by placing 3 g of kokum powder around a pestle inside a funnel so that the pestle blocks the funnel opening. There after the powder was allowed to flow into a beaker of water by lifting the pestle and the time taken for the complete wetting of the powder in the beaker was noted and recorded as the wetting time. The experiment was done in triplicate.

3.5.6 Bulk density

Bulk density of the kokum juice powder was determined by tapping method. Two grams of microencapsulated kokum juice 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 powder was recorded and the bulk density was calculated by dividing the sample weight by volume.

Bulk density (g/cm^3) = Weight of the powder / Volume of the powder

3.5.7 Colour

The colour of the kokum juice extract and powder were measured using Hunter lab colour flex meter (Hunter Association laboratory, Inc., Reston, Virgina, USA). Colour of the sample was obtained by measuring ‘L’, ‘a’ and ‘b’ colour values. Kokum juice and powder were placed over the colour measuring port of the flex meter and ‘L’, ‘a’, ‘b’ colour values were recorded.

3.5.8 Microstructural Analysis

Scanning electron microscopy (SEM) was used to investigate the microstructural characteristics including both morphology and inner structure of the encapsulated kokum juice extract powder. 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).

3.6 Proximate analysis

3.6.1 Determination of Total Carbohydrate by Anthrone Method

Carbohydrates are the important components of storage and structural materials in the plants. The carbohydrate content can be measured by hydrolysing the polysaccharides into simple sugars by acid hydrolysis and estimating the resultant monosaccharides. Carbohydrates are first hydrolysed into simple sugars using dilute hydrochloric acid. In hot acidic medium glucose is dehydrated to hydroxyl methyl furfural. This compound forms with anthrone a green coloured product with an absorption maximum at 630 nm (Ranganna, S. 1991).

Procedure 1. Weigh 100 mg of the sample into a boiling tube.

2. Hydrolyse by keeping it in a boiling water bath for three hours with 5 ml of

2.5 N HCl and cool to room temperature.

3. Neutralise it with solid sodium carbonate until the effervescence ceases.

4. Make up the volume to 100 ml and centrifuge.

5. Collect the supernatant and take 0.5 and 1 ml aliquots for analysis.

6. Prepare the standards by taking 0, 0.2, 0.4, 0.6, 0.8 and 1 ml of the working standard.'0' serves as blank.

7. Make up the volume to 1 ml in all the tubes including the sample tubes by adding distilled water.

8. Then add 4 ml of anthrone reagent.

9. Heat for eight minutes in a boiling water bath.

10. Cool rapidly and read the green to dark green colour at 630 nm.

11. Draw a standard graph by plotting concentration of the standard on the X-axis versus absorbance on the Y-axis.

12. From the graph calculate the amount of carbohydrate present in the sample tube.

Calculation

Amount of carbohydrate present in 100 mg of the sample = (mg of glucose) ×100/volume of test sample.

3.6.2 Determination of protein

The protein content was determined from the organic nitrogen content by Micro Kjeldal method. The KEL PLUS Automatic Nitrogen /protein estimation system by pelican equipments, Trivandrum, India was used for this estimation .The various nitrogenous compounds were used for this estimation. The various nitrogenous compounds were converted into ammonium sulphate by boiling with concentrated sulphuric acid. The ammonium sulphate formed was decomposed with concentrated sulphuric acid. The ammonium sulphate formed was decomposed with an alkali (NaOH) and the ammonia liberated was absorbed in excess of standard solution of acid and then back titrated with standard alkali. The nitrogen value was multiplied by 6.25 to obtain the protein content. (Ranganna, S.1991).

The protein content was calculated as:

Protien (%) = (14×titre value×Normality of alkali×6.25) / Sample weight

The percent nitrogen was multiplied by a factor of 6.25 to obtain percent "protein" on a total nitrogen basis.

3.6.3 Total ash

Note the tare weight of three silica dishes (7-8cm dia). Weigh 5-10g (or more if minerals are to be estimated, see under minerals) of the sample into each. If moist, dry on a water bath. (After determination of moisture content, the same dishes may be used for ashing) ignite the dish and the content on a muffle furnace. Ash the material at not more than 525^{0}C for 4 to 6 hr. If need be, ash overnight. Cool the dishes and weigh. The difference in weights gives the total ash content and is expressed as percentage.

3.6.4 Vitamin C

The direct colorimetric method is based on measurement of extent to which a 2, 6-dichlorophenol–indophenol solution is decolorized by ascorbic acid in sample extracts and in standard ascorbic acid solutions. Since inferring substances reduce the dye slowly, rapid determination would be measuring mainly the ascorbic acid.

Procedure

Preparation of sample: prepare the sample as in visual dye titration method but using 2% HPO_3. If the sample is solid, to blend 50 to 100 g of the sample with an equal weight of 6% HPO_3 and make an aliquot of the macerate to 100ml. Standard curve: to dry cuvettes or test tubes, pipette the requisite volume of standard ascorbic acid solution- 1, 2, 2. 5, 3, 4 and 5ml and make up to 5ml with the requisite amount of 2% HPO_3. Add 10ml of dye with a rapid delivery pipette shake and take the reading within 15 to 20s. Set the instrument to 100% transmission using a blank consisting of 5ml of 2% HPO_3 solution and 10ml of water. Measure the red colour at 518 nm or a wavelength nearest to the required wavelength using a suitable filter. Plot the absorbance against concentration. Sample: place 5 ml of extract in dry curette or test tube, add 10 ml of dye and measure as in standard.

3.6.5 Packing of microencapsulated powder

The powder obtained spray dryer is packed for shelf life studies. The samples were packed using a hand sealing machine and stored in ambient condition (temp 29-30^{o}C with 40- 50% RH) and the different quality parameters of the powder were evaluated. A double layer packing was done for storing the powder. The powder was packed in poly propylene (PP) cover and again packed with Aluminium foil. It was further stored in an air tight glass container.

3.6.6 Storage study

Bio-chemical analysis of powder obtained by spray drying were carried out to evaluate the quality deterioration during drying and storage. The moisture content, total solids, pH, bulk density, and wettability were estimated in every two week using the standard procedures.

3.6.7 Microbial Load of Encapsulated Powder

The quality of optimally produced encapsulated kokum juice 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, glass wares needed for the plate count method were sterilized by autoclaving at 121ºC for 15 min. One gram of the microencapsulated kokum juice 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, number of colony forming units (CFU's) per gram of the sample was estimated using the given formulae,

$$\text{CFU} = \frac{\text{(Mean no of CFU x Dilution factor)}}{\text{Quantity of sample on wt basis.}}$$

3.6.8 Sensory evaluation

Sensory quality is the ultimate measure of product quality and success. The fresh kokum juice with sugar was kept as control. The juice prepared from spray dried powder T4 (25% MD, 200^{0}C) with cardamom flavour was kept as sample 1 and sample 2 prepared from spray dried powder T4 was mint flavoured. Sample 3 and sample 4 was prepared from spray dried powder T6 (30% MD, 200^{0}C) with cardamom and mint flavour respectively. Sugar was added in all samples, irrespective of treatments.

SENSORY EVALUATION CARD

Name of the panelist: Date:

SAMPLE NO	COLOUR	TASTE	TEXTURE	OVERALL ACCEPTABILITY

Table 3.1 Sensory evaluation card

5-Like very much, 4-Like, 3-Nither like nor dislike

2-Dislike, 1-Dislike very much

Signature of the panelist

3.7.9 Standardisation of drying temperature and treatment

About 500 ml of juice was taken for spray drying. The drying was carried out at two different inlet temperatures and at different concentrations of maltodextrin.

Outlet temperature -94^{0}C, Blower speed -1700 rpm, Feed rate – 600 ml/h

TRIAL NO	SAMPLE	INLET TEMERATURE
T1	20% MD	190°C
T2	20% MD	200°C
T3	25% MD	190°C
T4	25% MD	200°C
T5	30% MD	190°C
T6	30% MD	200°C

Table 3.2 Standardisation of drying temperature and treatment

Process protocol for kokum juice powder

Fresh Kokum
⇩
Splitting ⟶ rind + seeds
⇩
Citric acid (1.5 %) ⟶ Pulping & staining
⇩
Maltodextrin ⟶ Blending
⇩
Spray drying
⇩
Packaging
⇩
Storage

Fig 3.1 Flow chart for kokum powder preparation

CHAPTER IV

RESULTS AND DISCUSSION

This chapter deals with the results of the investigations carried out on the encapsulation of Kokum juice powder. 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 Kokum juice powder are analyzed and discussed.

4.1 Standardization of treatments

4.1.1 Water activity

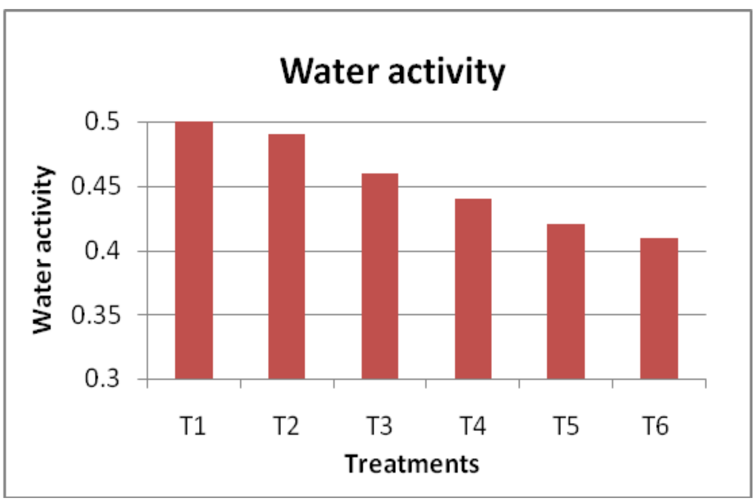


Fig 4.1 Variation in Water activity in various treatments

From the fig.4.1 it is observed that with increase in maltodextrin content and temperature, water activity of the powder decreases. Food stability usually decreases with increase in water activity (Anjineyulu *et al*., 2014). Water activity is maximum for treatment T1 (spray drying with 20% MD, 190^oC) and minimum for treatment T6 (spray drying with 30%, 200^oC). In general the water activity is in the permissible limit. Microbial growth, and in some cases the production of microbial metabolites, may be particularly sensitive to alteration in water activity.

4.1.2 Moisture content

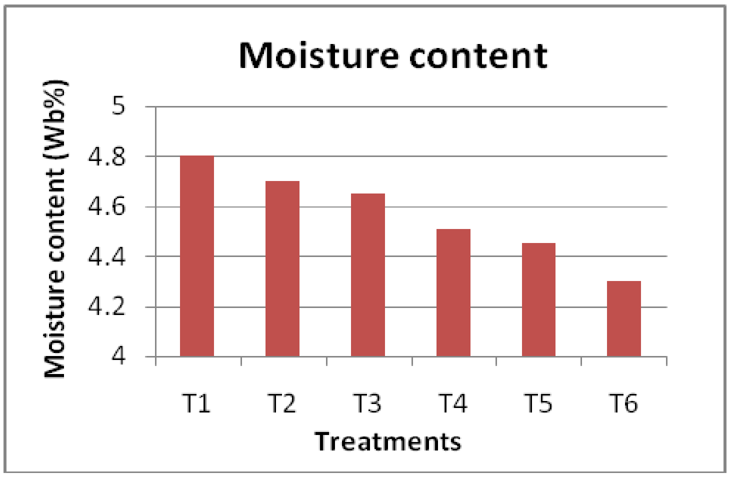


Fig 4.2 Variation in Moisture content in various treatments

From the Fig.4.2 it is evident that the moisture content is maximum for sample T1 (spray drying with 20% MD, 190^oC) and minimum for sample T6 (spray drying with 30% MD, 200^oC). The higher the inlet air temperature and MD concentration, the lower the moisture content of the spray dried kokum powder. Similarly according to Mishra *et al*. (2014), higher MD concentrations showed a sound effect to down the moisture trend of spray dried powder owing to the fact that the addition of MD that increased the total solids of the feed and reduced the amount of water vaporization. It was found that at a constant feed flow rate, increasing the inlet air temperature reduced the residual moisture content.

4.1.3 Wettability

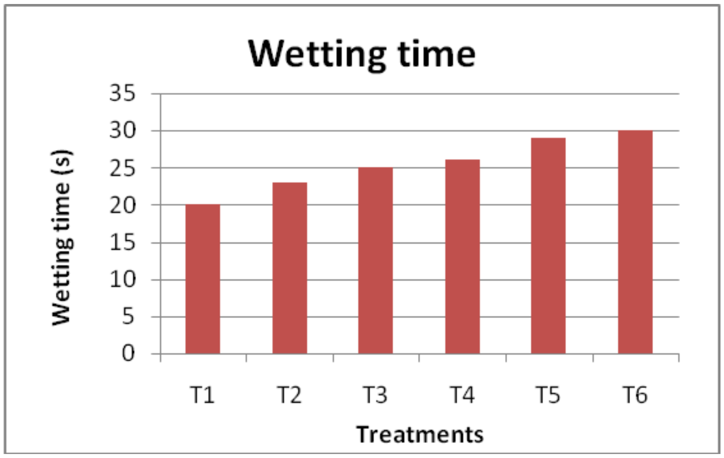


Fig 4.3 Variation in Wettability in various treatments

The term wettability refers to the ability of agglomerates to be penetrated by the liquid. Fig.4.3 show that sample T1 (20% MD, 190^{o}C) has the least wetting time while sample T6 (30% MD, 200^{o}C) has the highest wetting time. Wettability decreased with the maltodextrin concentration and temperature. The changes in wettability can be due to the difference in particle size with the addition of maltodextrin (Anjineyulu *et al*., 2014).

4.1.4 TSS

As shown in Fig.4.4, sample T6 (30% MD, 200^{o}C) showed highest TSS value while sample T1 (20% MD, 190^{o}C) showed the least. This was in accordance with the findings of Anjineyulu *et al*. (2014) in spray dried dahi powder. The addition of the maltodextrin has increased the total solids of the feed and reduced the amount of water evaporation. The temperature variation has also effect on the change in TSS. Maltodextrins could alter the surface stickiness of low molecular weight sugars such as glucose, sucrose and fructose and organic acids, therefore, facilitated drying, reduced the stickiness and increased the soluble solids of the spray dried product.

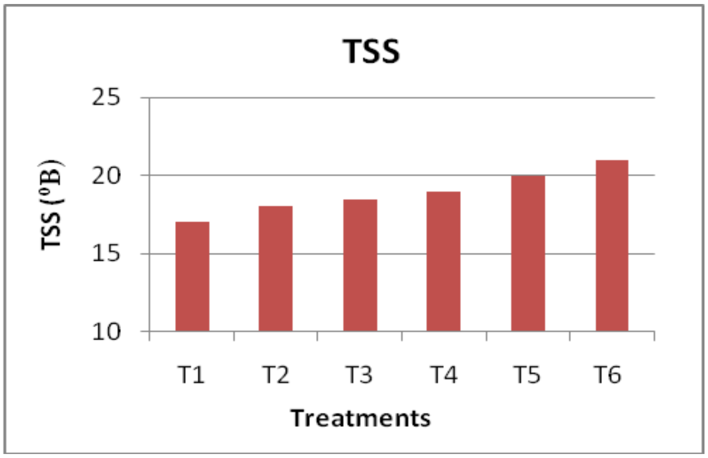


Fig 4.4 Variation in TSS in various treatments

4.1.5 Bulk density

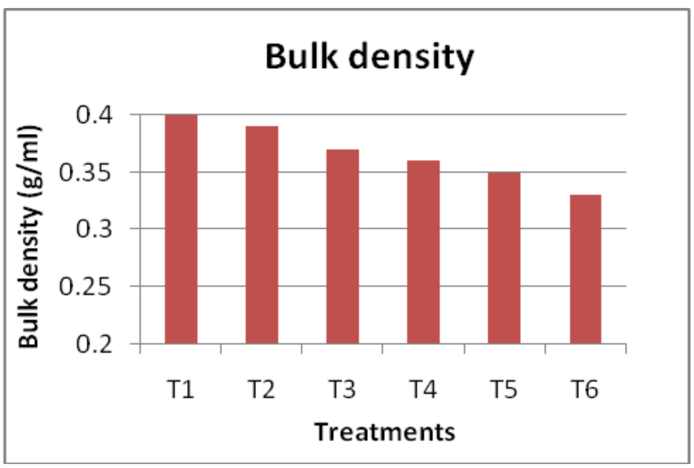


Fig 4.5 Variation in Bulk density in various treatments

In this study, bulk density of the kokum powders reduced with increasing inlet temperature and maltodextrin concentration. Bulk density was higher for sample T1 (20% MD, 190^{o}C) and least for sample T6 (30% MD, 200^{o}C) and is shown in Fig. 4.5. It is due to the less droplet shrinkage of the powder. This was similar to the findings of Cai and Corke (2000) in the microencapsulation of amaranthus. An increase in the inlet air temperature often results in a rapid formation of dried layer on the droplet surface and particle size and it causes the skinning over or casehardening on the droplets at the higher temperatures. Higher inlet air temperatures result in powders with lower density, which is due to the higher drying temperature that causes faster particle drying with less droplet shrinkage giving lower powder density.

4.1.6 pH

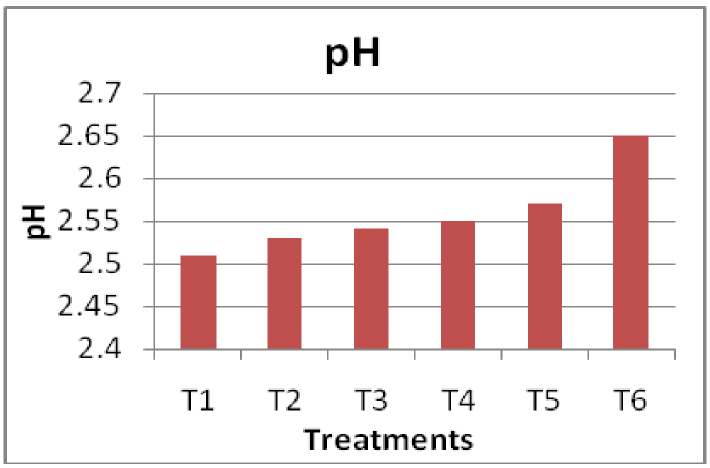


Fig 4.6 Variation in pH in various treatments

From Fig.4.6 it is evident that the sample T1 (20% MD, 190^{o}C) has low pH when compared to that of sample T6 (30% MD, 200^{o} C) due to the low maltodextrin concentration (Anjineyulu *et al*., 2014). pH of the solutions were significantly higher than those of the fresh kokum juice.

4.1.7 Acidity

As shown in Fig.4.7 sample T1 (20% MD, 190^{o}C) has high acidity when compared to that of sample T6 (30% MD, 200^{o}C) due to the low maltodextrin concentration. It was also found that the temperature variations have no considerable effect on pH and acidity (Luh and Woodruff, 1975).

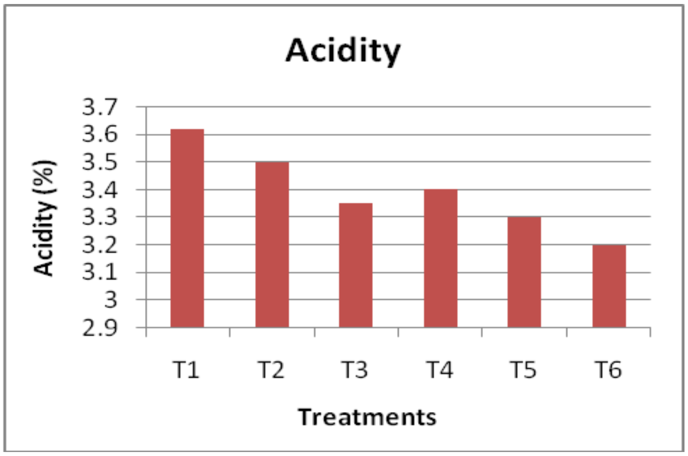


Fig 4.7 Variation in Acidity in various treatments

4.1.8 Colour

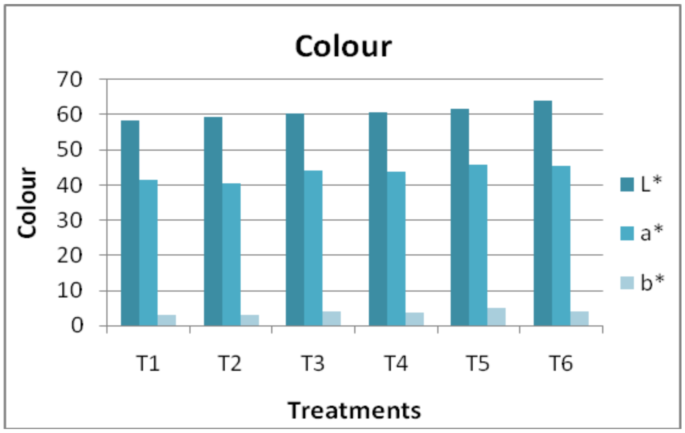


Fig 4.8 Variation in Colour in various treatments

The lightness, redness and yellowness of the solutions of spray dried Kokum powder and reconstituted juice were significantly different from the fresh sample. It is observed from Fig.4.8, the lightness of the powders was high for T6 (30% MD, 200^{o}C) and less for sample T1 (20% MD, 190^{o}C). The cause of the more lightness was due to the effect of the maltodextrin. Because of white color of maltodextrin a greater lightness of powders, represented by a higher L value, was obtained at higher concentrations of maltodextrin. However, if the added maltodextrin was more than 20%, the resulted powders lost their attractive purple red colour. Similar results were also found in spray dried sweet potato powders by Grabowski *et al*. (2006).

4.2 Standardized parameters of spray drying

Spray drying parameters	Result
Feed pump rpm	5 rpm
Main blower rpm	1700 rpm
Inlet temperature	200^{o}C
Outer temperature	85^{o}C
Pressure	2kg/cm^2

Table 4.1 Standardized parameters of spray drying

The spray drying parameters were optimized based on the yield and external appearance of the powder. Temperature and other process parameters were adjusted by considering above attributes. At 200^{0}C more yield and better appearance were observed. The feed pump speed was adjusted to 5 rpm. A substantial reduction in powder was observed at higher pump feed rate (>10 rpm). The pressure and blower speed of spray dryer were adjusted to 2 kg/cm^2 and 1700 rpm, respectively in order to obtain good quality powder with above attributes.

4.3 Sensory analysis

Sensory quality is the ultimate measure of product quality and success. Sensory analysis comprises a variety of powerful and sensitive tools to measure human responses to foods and other products. The fresh kokum juice with sugar was kept as control. The juice prepared from spray dried powder T4 (25% MD, 200^{0}C) with cardamom flavour was kept as sample 1 and sample 2 prepared from spray dried powder T4 was mint flavoured. Sample 3 and sample 4 was prepared from spray dried powder T6 (30% MD, 200^{0}C) with cardamom and mint flavour respectively. Sugar was added in all samples, irrespective of treatments.

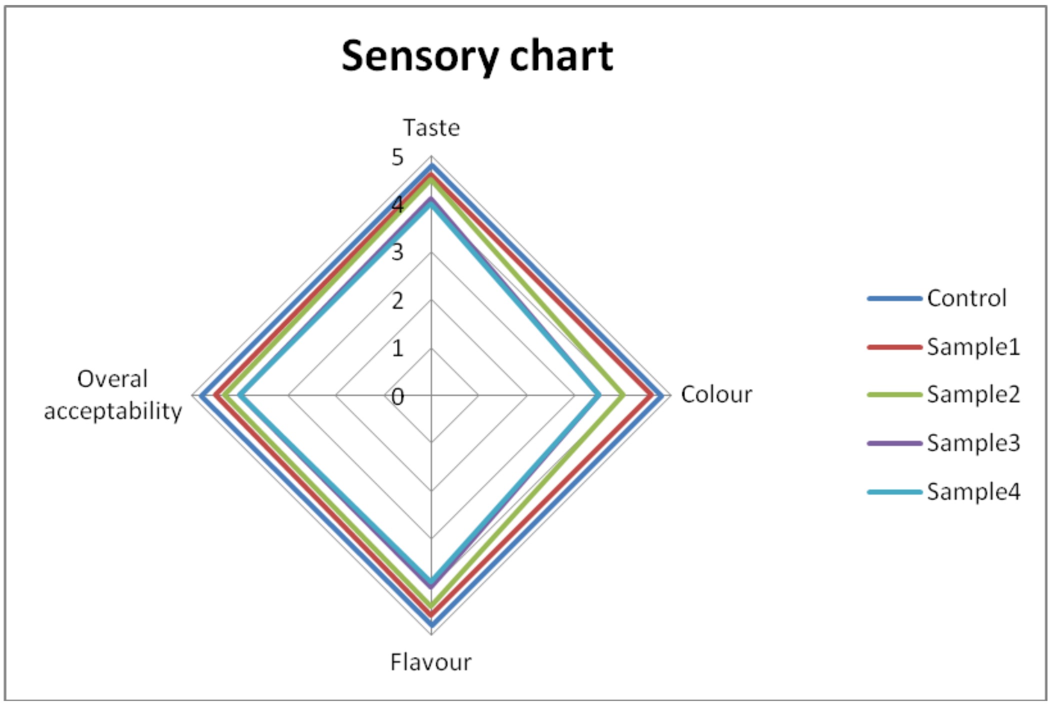


Fig 4.9 Sensory analysis of spray dried kokum powder

It was observed that taste, flavor and overall acceptability were high for the control. However, the colour of juice prepared from the powder T4 was marked as better by sensory panelists. Sample 1 (T4 with cardamom flavour) fetched more sensory score than other samples. Colour of the juice made from spray dried powder T6 was less compared to that of T4. It may be due to the increased concentration of maltodextrin in T6. Based on quality analysis and sensory analysis spray dried powder T4 was optimized as best powder.

4.4 Proximate composition of the powder

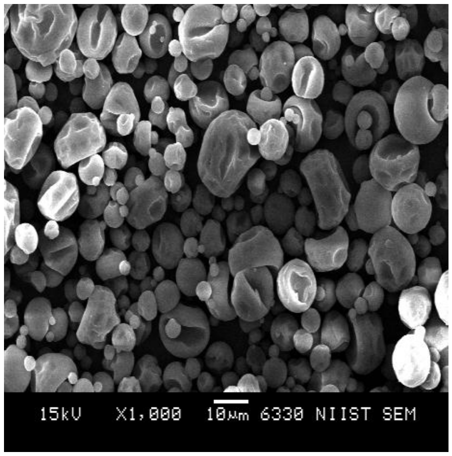
Table 4.3 shows the proximate composition of kokum powder. The carbohydrate content of powder was found to be 70%. The powder contains 1.5% protein, 216 kcal of energy and 3.4% of total ash.

Sl No	COMPOSITION	
1	Carbohydrate	70%
2	Protein	1.5%
3	Vitamin C	0.06%
4	Energy	216 kcal
5	Ash	3.4%

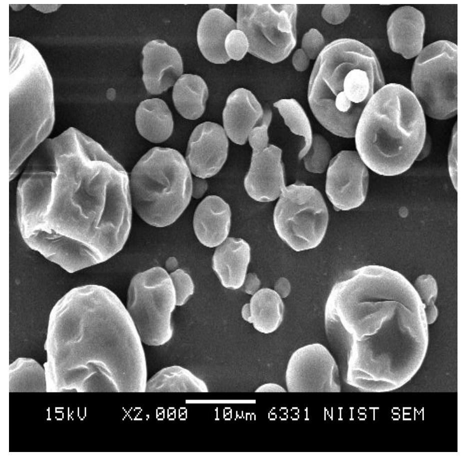
Table 4.2 Proximate composition of the powder

4.5 Particle size analysis

The morphology of the spray dried powder which was produced at optimized process conditions was analyzed with the help of Scanning Electron Microscope (SEM). The spray dried powder sample was having an average particle size of 10 µm.



a



b

Plate 4.1 SEM micrographs of kokum powder

4.6 Physico-chemical analysis

Moisture content of optimized spray dried powder was found to be 5%. This moisture content was found to be safe for storage and further processing. The pH and titrable acidity of the juice was found to be 2.5 and 3.4% respectively. TSS of fresh juice was found to be 11˚Brix and the spray dried powder was 19˚Brix. Water activity of the powder was found to be 0.4 which is in the permissible limit. It indicates that the powder is safe for storage. The wetting time of the powder should be low for good reconstitution. The wetting time of the powder was 26s. It can be seen that as the temperature increases, the time required for wetting of the powder increases which implies the wettability of the powder decreases. This may be due to reduced product residual moisture content. Similar results were found by Bhandari *et al*. (1993) and Jumah *et al*. (2000). The bulk density of powder was found to be 0.36 g/ml.

The results of various physico-chemical analysis of optimized sample (T4) is shown in the table 4.3.

PHYSICO-CHEMICAL ANALYSIS	KOKUM POWDER
Ph	2.5
Titrable acidity	3.4%
TSS	19^0B
Moisture content	5%
Water activity	0.4
Wettability	26 s
Bulk density	0.36 g/ml

Table 4.3 Physico-chemical analysis of the optimized sample

Plate.4.2 Kokum juice powder

4.7 Shelf life study of the powder

The optimized sample was further subjected to shelf life study of storage. The changes in the main quality attributes of the optimally produced encapsulated kokum juice powder for a period of two months were analyzed. The results obtained are discussed below.

4.7.1 Changes in Moisture content on storage

The change in moisture content with storage is shown in Fig.4.10. This increase in moisture content could be due to the water vapour permeability of packaging material during storage. Similar results of increase in moisture content were reported in dahi powder (Anjineyulu Kothakota *et al*., 2014).

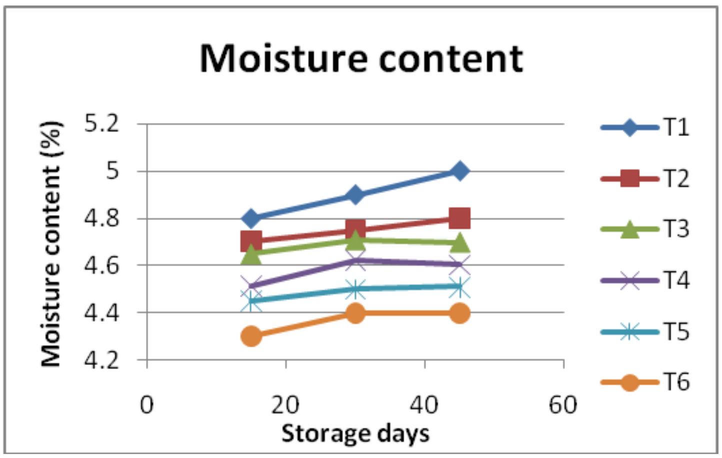


Fig.4.10 Changes in moisture content on storage

4.7.2 Changes in Water activity on storage

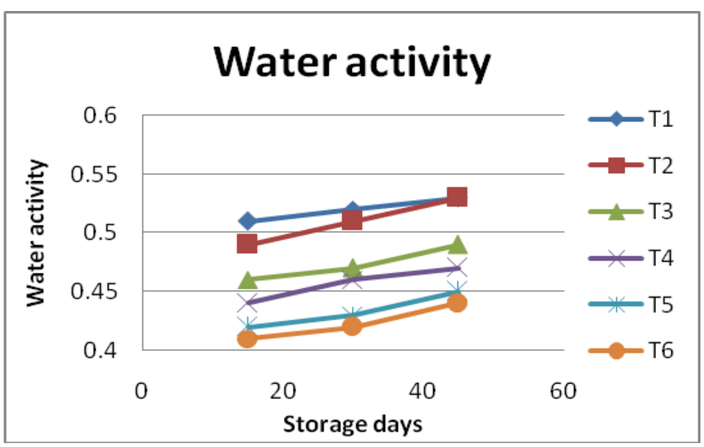


Fig.4.11 Changes in water activity on storage

The water activity of the samples were in the range of 0.4-0.5 and further increase in storage period resulted in slight increase of water activity and it is shown in Fig.4.11. It may be due to improper sealing and water vapour migration through packaging material during storage.

4.7.3 Changes in TSS on storage

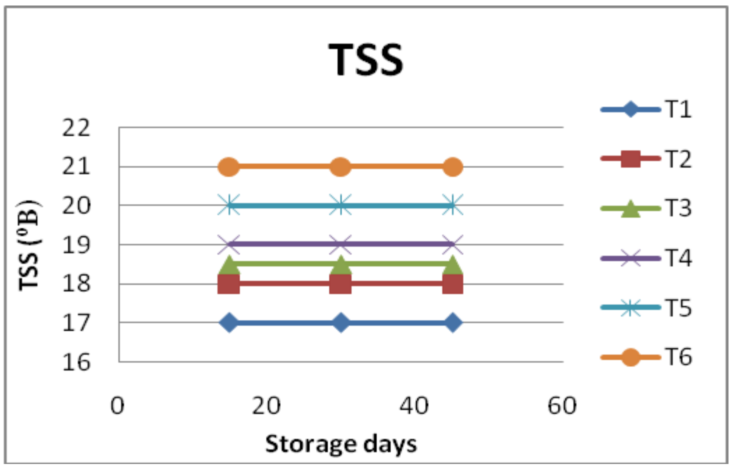


Fig.4.12 Changes in TSS on storage

It is clear from the Fig.4.12 that there is no increase in TSS concentration during storage. (Luh and Woodruff, 1975) studied the change of TSS in storage and concluded that the increase in TSS during storage may be due to acid hydrolysis of polysaccharides especially gums and pectin.

4.7.4 Changes in bulk density on storage

Fig. 4.13 shows that the bulk density is increasing in storage. This is due to the increase in moisture content of the powder particles which resulted in increased cohesiveness thus reducing the volume Cai and Corke (2000).

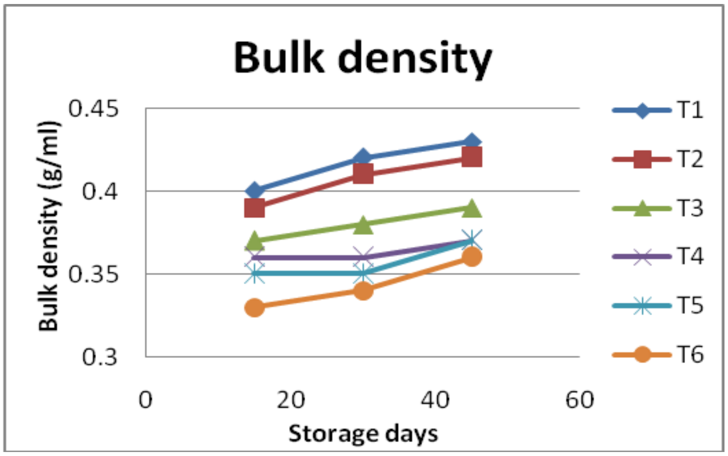


Fig.4.13 Changes in bulk density on storage

4.7.5 Changes in wettability on storage

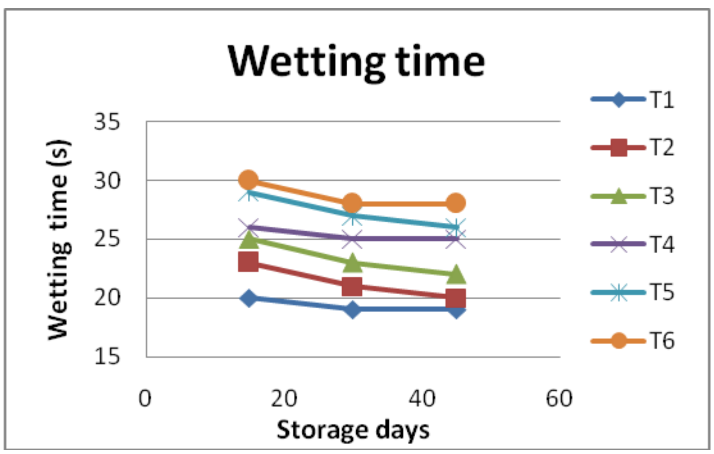


Fig.4.14 Changes in wettability on storage

From Fig.4.14 it is clear that the wetting time of the powder samples decreases during storage. This property depends largely on particle size. Small particles have a large value of specific area (i.e., the ratio of surface area to mass) and may not be wetted individually; increasing particle size and/or agglomerating particles can reduce the incidence of clumping. The nature of the particle surface can also affect wettability (Desobry *et al*., 1999).

4.7.6 Changes in pH on storage

As represented in Fig.4.15, the pH of the reconstituted kokum juice slightly increased with storage. The changes may be due to the acid hydrolysis of polysaccharides (Luh and Woodruff, 1975).

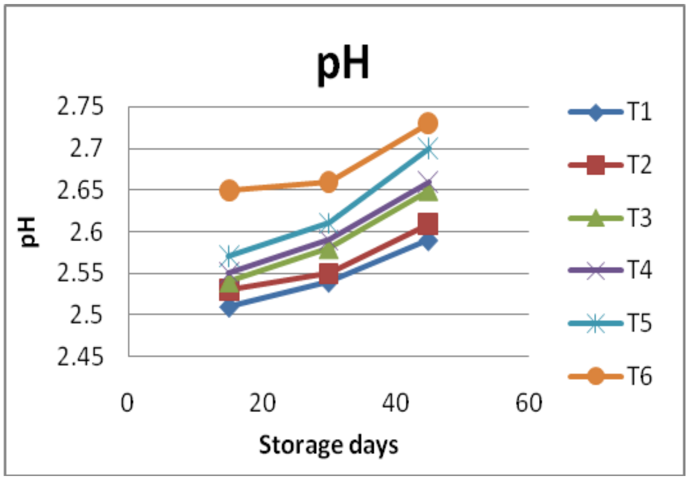


Fig.4.15 Changes in bulk density on storage

4.7.7 Microbiological Analysis

The total number of viable microbes per gram of sample was obtained by multiplying the number of colony forming units (CFU) on the plate with respective dilution factor and then was converted into logarithmic form. Microbial load of standardized sample was found to be $1X10^2$ CFU/ml which is very small. So the powder was found to be safe.

CHAPTER IV
SUMMARY AND CONCLUSION

Spices are mainly used for preserving, colouring and flavouring. Flavour is the sensory impression of food or other substance. *Garcinia indica* (kokum) is one of the minor spices and is a popular flavouring agent. In summer the ripened rinds are ground in a blender with sugar and cardamom and consumed as a cooling drink. Kokum fruit possess useful anti-oxidant, chelating, anti cancer, anti inflammatory, anti bacterial, anti fungal, cardio protective and anti ulcer activities. Realizing the importance of fruits as a significant contributor to human well being, as a cheaper and better source of protective foods, their perishable nature and seasonality in production calls for preservation of them to be supplied throughout the year for human consumption. This study was planned keeping in view the nutritional importance of kokum, to utilize it by formulating a powdered product from kokum. As kokum is an underutilized fruit, it can be preserved by making it as powder to use it for the preparation of RTS beverage and natural colours.

In this study we used spray drying technology along with micro encapsulation to obtain the powder. There are different techniques available for microencapsulation, spray drying is the most common, economical and commercial method for microencapsulation process. The microencapsulation of kokum extract could be carried out with wall material as maltodextrin. The intention of the present study was to develop a free flowing and high stable encapsulated kokum extract powder and standardization of maltodextrin concentration and inlet air temperature of spray drier for the microencapsulation process by spray drying technique. The emulsions were then spray dried at different inlet temperatures of 190 and 200^{o}C with maltodextrin concentration of 20, 25 and 30%. The encapsulated kokum extract powder were collected, packed in aluminium foil pouches and stored. The storage study of the powder was done for two months.

The present work was carried out to investigate the influence of maltodextrin concentration at different inlet air temperatures on spray dried kokum juice powder properties in terms of its physical properties, and storage life. Both of the factors had a significant effect on the powder properties. The physicochemical characteristics such as wettability, bulk density, pH, titrable acidity, TSS, MC and water activity of powder were determined and consumer acceptability of the juice prepared from the powder was determined by sensory analysis. At 200ºC inlet temperature, 94ºC outlet temperatures, feed pump rpm of 5, main blower 1700 rpm, good quality powder was obtained. The powder produced with higher concentration of maltodextrin at higher temperature was found to be more convenient than the others where the moisture content in the range of 4-5% indicates more stability with the highest bulk density that was more desired and with the maximum yield was ensured. SEM micrographs revealed that the particle size of 10 µm. Samples with higher concentrations of maltodextrin were low hygroscopic and was more stable on storage with a shelf life of more than two months. The Maltodextrin was a suitable drying material agent to increase the dryer yield. Sample T4 (25% MD at 200ºC) showed a stable shelf life with better physiological qualities. The reconstituted juice of sample T4 with cardamom flavour fetched the best results in sensory analysis. The advantage of kokum powder is as it can be easily packed in small pouches and easy to transport and was more stable on storage with a shelf life of two months. Further investigation is required to analyze some chemical compounds of the final powder to preserve the best quality, and on the storage effects towards the quality properties to enhance the shelf life of the product. Garcinia is sold in many different forms, from capsules and tablets to powder that can mix with water or juice for refreshing drink, so *Garcinia indica* juice is healthier and far more refreshing option as compared to commercial bottled drinks. Hence generation of powder can be new business opportunity for *Garcinia indica* growing farmers in India

CHAPTER V

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PRODUCTION OF KOKUM JUICE POWDER USING SPRAY DRYING TECHNOLOGY

By

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ABSTRACT OF THE THESIS REPORT

Submitted in partial fulfillment of the requirement for the degree

Bachelor of Technology
In
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ABSTRACT

Garcinia indica is popularly known as kokum in Maharashtra or Indian butter tree belongs to family Guttiferae. Kokum fruit possess useful antioxidant, chelating, anti cancer, anti inflammatory, anti bacterial, anti fungal, cardio protective and anti ulcer activities. As kokum is a highly underutilized fruit, it can be preserved by making it as a powdered product to use it during off-season.

A study was conducted on microencapsulation of kokum *(Garcinia indica)* juice by spray drying technique using maltodextrin as the wall material. The kokum juice was spray dried at inlet air temperatures of 190 and 200^{o}C with maltodextrin at a concentration of 20, 25 and 30%. The kokum powder was analyzed for physical properties viz; wettability, bulk density, pH, titrable acidity, TSS, a_w and particle size using standard procedures. The RTS prepared from powder were assessed for their sensory attributes and overall acceptability. The variations in physico-chemical properties were also analyzed during storage period. Based on the physico- chemical characteristics and sensory analysis, a maltodextrin concentration of 25% was standardized for the production of microencapsulated kokum juice by spray drying. At 200^{o}C inlet temperature, 94^{o}C outlet temperatures, feed pump rpm of 5, main blower speed 1700 rpm, good quality powder was obtained. The physico-chemical characteristics of the optimized kokum juice powder were: moisture content (4.5%), wettability (26 s), bulk density (0.36 g/ml), pH (2.5), titrable acidity (3.4%), TSS (19^{o}Brix), and water activity (0.4). The SEM micrographs showed a uniform powder with an average particle size of 10 µm. The study suggests that reconstituted kokum juice with cardamom flavour can be taken as a ready to drink beverage. The samples with higher concentrations of maltodextrin were low hygroscopic and was more stable on storage with a shelf life of two months. Microbial load of standardized sample was found to be 1X10^{2} CFU/ml which is very small. Hence, the kokum powder is safe and fit for human consumption.