Int.J.Curr.Res.Aca.Rev.2017; 5(4): 56-63

International Journal of Current Research and Academic Review

ISSN: 2347-3215 (Online) Volume 5 Number 4 (April-2017)

Journal homepage: http://www.ijcrar.com

doi: https://doi.org/10.20546/ijcrar.2017.504.009

A Natural Colourant Spray Drying of Cactus Pear (*Opuntia Ficus Indica*) Juice and Its Physico-Chemical Characteristics

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Abstract

The *Opuntia ficus indica* (OFI) fruits were obtained from Dindugal district, Tamil Nadu, India. The juice was extracted from fully ripened fruit. It had an average total soluble solids equivalent to 14.06º Brix. Before spray drying, a 1:1 dilution with distilled water was made to obtain a soluble solid content of 5.4ºBrix. Maltodextrin (MD) 32 gms and Arabic Gum 40 gms were added to the diluted juice mixed and filtered. The clear solution was spray dried using Labultima, model LU 222 advanced. The powder was obtained with the flow of air drying parallel to the material feeding. The drying parameters were Inlet temperature = 140˚C; Outlet temperature = 70˚C; Aspirator speed = 35 cu.m/hr; Feed pump speed = 2mL/min which were standardized for the experiment. The spray dried fruit powder was packed in metallized bioriented polypropylene bags at 24°C± 2° C and stored at room temperature (20-24°C) before being submitted to the physic-chemical analyses. The physic-chemical characteristics like X-ray diffraction, solubility test (0.57%), and bulk density (2.5g/ml), colour parameters and water absorption capacity (WAC) of fruit powder (29.6 ml) were determined. Hence the present study provides useful information about the spray drying of an under-utilised fruit to obtain a colourant that can be used for commercial food product development.

Article Info

Accepted: 04 April 2017
Available Online: 20 April 2017

Keywords

Opuntia ficus indica,
Maltodextrin,
Spray drying.

Introduction

Cactus pear known as chappathikalli in the local language (Tamil), a fruit of the perennial *Opuntia Ficus Indica*, belonging to the Cactaceae family is well adapted to arid and semiarid climates, where water can be a limitation for cultivation Benson (1982). Cactus pear fruit is a many seeded berry with a thick peel, enclosing a delicately flavoured seedy pulp. Cactus pear fruits are rich sources of nutrients and vitamins and can be eaten fresh, dried or preserved in jams, syrups or processed into candy-like products Hoffman, (1980). Their juices are sometimes fermented, using appropriate yeast strains, either into ethanol or wine and other beverages or used in food flavourings and colourings (Bustos, 1981). Thus, there is an increasing interest for large-scale cactus pear fruit processing for the production of coloured foodstuffs, opening new markets on functional foods (Mobhammer *et al.*, 2005).

Hence the present study was carried out with the following objectives:

To spray dry the juice of the selected fruit *Opuntia ficus indica* (OFI).

To evaluate the Physico Chemical Characteristics of Spray dried OFI powder.

Materials and Methods

Raw material

Samples of the OFI fruit were collected from Puthupatti village in Dindugal district of Tamil Nadu, India located 10°21′14.4″N 77°59′6″E. The Harvesting stage of the fruit was the fully ripened stage during the month of May to July 2012. Care was taken to exclude over-ripe and damaged fruits.

Sample Preparation

The fruits were carefully washed with running tap water by rubbing to remove the glochid and prickles. After the free water drained from the fruits, their weight was recorded. Fruits were cut longitudinally, the flesh and thick peel were separated, and the weights of these fractions were recorded. The Preethi Zodiac - MG 218, mixer grinder with a 3-in-1 Insta Fresh Fruit Juicer Jar was used to make the fresh juice of the OFI without the addition of water. The remaining pulp was separated and discarded.

The common carrier agents used for fruit juices are maltodextrins and gum Arabic. Maltodextrins are low cost and very useful for spray drying process on food materials. Maltodextrins are products of starch hydrolysis, consisting of D-glucose units linked mainly by α(1→4) glycosidic bonds. Gum Arabic is a natural plant exudates of Acacia trees, which consists of a complex heteropolysaccharide with highly ramified structure. It is the only gum used in food products that shows high solubility and low viscosity in aqueous solution, making easier the spray drying process (Gabas *et al.,* 2007; Rodriguez-Hernandez *et al.,* 2005). Maltodextrin (dextrose equivalent 12; Tg of about 150°C) and Arabic gum (AG) (Sigma-Aldrich Corp., St. Louis, Mo., U.S.A) were used as carriers.

One Kilogram of the edible portion of the fruit was used to obtain 300ml of extract with a total soluble solids equivalent to 14.06º Brix.. This extract was diluted with distilled water in the ratio 1:1 to obtain a total soluble solid content of 5.4º Brix. Then Malto Dextrin (32 gms) and Arabic Gum (40 gms) were added and mixed for thirty minutes. A Magnetic Strirrer with stainless steel top housing and Permanent Magnet DC (PMDC) motor to give high torque even at lower speeds and maintain speed stability despite viscosity was used for this purpose.

The next step was the filtration process. Filtration is also a mechanical process designed for clarification by removing insoluble solids from a high-value liquid food, by the passage of most of the fluid through a porous barrier, which retains most of the solid particulates contained in the food. In our study the OFI fruit extract had mucilage which had to be removed before spray drying. Filtration was performed using a filter medium, in this case a muslin cloth. Filter acts as a barrier that lets the liquid pass while most of the solids are retained. The liquid that passes through the filter medium is called the filtrate. A clear solution containing the pigment of OFI was obtained.

The main challenge in the separation of the red pigment from the cactus pear pulp is the mucilage, a complex mixture of polysaccharides, less than 50% of which corresponds to a pectin-like polymer (Sáenz, 2004; Matsuhiro *et al.,* 2006). The traditional process used to remove it involves precipitation of the mucilage using ethanol (Sáenz, 2004). However, the use of ethanol in food applications is considered increasingly unfavourable (Mohamed and Mansoori, 2002), and hence a membrane process was used to separate the mucilage and to obtain a clarified juice permeate which has a good potential as a food colourant.

Optimization of the Process Parameters for Spray Drying of OFI Extract

Spray drying involves 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 physicochemical properties of the final product mainly depend on inlet temperature, air flow rate, feed flow rate, atomizer speed, types of carrier agent and their concentration. Spray drying is often selected as it can process material very rapidly while providing relative control of the particle size distribution (Obon *et al.,* 2009).

Optimization of the three process variables; inlet drying temperature, feed rate and air flow rate was performed using the Response Surface Methodology (RSM) of Design Expert Software 6.0. The objective of the optimization was to obtain the combinations of the three process parameters, which would produce the desired powder quality.

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. The powders produced at higher inlet temperatures were more hygroscopic due to the presence of moisture content in the powder. When the drying temperature is higher moisture content is lower resulting in an increase in the hygroscopicity of the fruit powder (its capacity to absorb ambient moisture).

Spray dried powder of *Opuntia ficus indica* fruit powder (OFIFP) was obtained by using the Labultima spray dryer, (LU222 Advanced), with the flow of air drying parallel to the material feeding. After several trials the drying parameters were fixed at Inlet temperature = 140˚C; Outlet temperature = 70˚C; Aspirator speed = 35cu.m/hr; Feed pump speed = 2mL/min. The powders obtained by the spray drying process were packaged in sealed metallized bioriented polypropylene bags at 24°C± 2° C and stored at room temperature (20-24°C) before being submitted to the physic-chemical analyses.

Physico-chemical Characteristics of spray dried *Opuntia ficus indica* fruit powder (OFIFP)

X ray diffraction microstructure analysis

Morphology is one delicate aspect of spray drying which makes it versatile as well as intricate. Morphology affects the key quality characteristics of spray dried products such as particle size distribution, flowability, friability, moisture content, bulk and particle density. Hence, it is vital to understand the variables which decide the morphology, and the frequently occurring morphology patterns in the spray dried products. X-ray diffraction analyses were performed adapting the method described by Ronkart *et al.*, (2006). X-ray diffraction (DRX) of powdered OFI was carried out using a X-RDA 6000 model Shimadzu. Measurement conditions were: Cu anode, 40 kV, 30 mA, divergence slit 1.00 deg, scatter slit 1.00 deg, receiving slit 0.30 mm, scan range 2.5-120.0, continuous scan, scan speed 0.50 deg/min, sampling pitch 0.02 deg, preset time 2.40 s. The characteristic x-ray diffraction pattern generated in a typical XRD analysis provides a unique "fingerprint" of the crystals present in the sample. When properly interpreted, by comparison with standard reference patterns and measurements, this fingerprint allows identification of the crystalline form.

Solubility Analysis

To 100 ml of distilled H_2O, one gram of powder was added by mixing at high velocity in a mixer for 5 min. The solution was placed in a tube and centrifuged at 3000 rpm for five minutes. An aliquot of 25 ml of the supernatant was placed in previously weighed Petri dishes and immediately oven-dried at 105 ºC, for five hours. Solubility (%) was calculated by the difference in weight (Eastman and Moore, 1984).

Bulk density

The bulk density (BD) of the fruit powder was determined using the method described by Okezie and Bello (1988). A ten ml graduated cylinder, previously tarred was gently filled with ten gram of fruit powder sample. The bottom of the cylinder was gently tapped on a laboratory slab ten times (Nwabuze *et al.*, 2006).

$$\text{Bulk Density (BD)} = \frac{\text{Weight of sample (g/ml or g/cm}^3\text{)}}{\text{Volume of sample after tapping}}$$

Water Absorption Capacity

This property was determined according to the method given by Smith and Circle (1972). Exactly five gram of fruit powder was mixed well with 30ml distilled water at room temperature in a centrifuge tube using a glass rod. After five minutes, the content was centrifuged at 2000 rpm for five minutes. The supernatant was measured using a graduated cylinder.

Volume of water absorbed = 30 – Supernatant

$$\text{Water absorption} = \frac{\text{Volume of water absorbed x 100}}{\text{Weight of sample}}$$

Colour Measurement

Colour is an important quality attribute in the food and bioprocess industries, and it influences consumer's choice and preferences. Food colour of the fruit powder is governed by the chemical, biochemical, microbial and physical changes which occur during growth, maturation, postharvest handling and processing. Colour measurement of food products has been used as an indirect measure of other quality attributes such as flavour and contents of pigments because it is simpler, faster and correlates well with other physicochemical properties.

The colour of an object can be described by several colour coordinate systems. Some of the colour systems used in the food industries are Munshell Colour System, RGB system, Hunter L a b, Commission Internationale de l'Eclairage's (CIE) L*a*b*, CIE XYZ, CIE L*u*v*, CIE Yxy, and CIE LCH. These differ in the symmetry of the colour space and in the coordinate system used to define points within that space.

Colour of the spray dried OFIFP was measured using the LOVIBOND LVDV-RT 200model. The parameter a* takes positive values for reddish colours and negative values for the greenish ones, whereas b* takes positive values for yellowish colours and negative values for the bluish ones. L* is an approximate measurement of luminosity, which is the property according to which each colour can be considered as equivalent to a member of the greyscale, between black and white (Pankaj *et al.*, 2013).

All estimations to determine physic chemical characteristics were carried out thrice to obtain the mean value.

Results and Discussion

Physico-chemical characteristics of spray dried *Opuntia ficus indica* fruit powder (OFIFP)

The properties of powders can be classified into three categories. They are the fundamental, functional and detective properties. The physical properties such as moisture content, bulk density, particle density, wetting, dispersion, solubility, and particle size are influenced by the nature of the feed (solid content, viscosity and temperature) and operational conditions. These properties are also dependent on their structure. Hence the microstructure analysis was carried out.

X-ray diffraction microstructure analysis

Depending on the nature of food powder (crystalline or amorphous), the isotherms shape is different. Most food powders have complex structures. Spray-dried powders usually have a small particle size of 10-100 µm, with poor handling and reconstitution properties Fuchs (2006). The residence time of particles in the spray dryer does not usually exceed 30 seconds.

The hygroscopicity of fruit powders with high levels of sugar is attributed to the amorphous state of sugars. MD is an adjuvant widely used in drying processes. MD is especially used in hard drying materials, such as fruit juices in order to reduce the problems of agglomeration during storage and to improve product stability (Silva, 2006).

We obtained the following graphical presentation of the spray dried OFI fruit powder. We conclude that the powder was in the amorphous state because the graph line had abundant broad background pattern.

It is generally agreed that the peak breadth of a specific phase of material is directly proportional to the mean crystallite size of that material. Quantitatively speaking, sharper XRD peaks are typically indicative of high nanocrystalline nature and larger crystallite materials. From our XRD data, a peak broadening of the nanoparticles was noticed. The X-ray scattering from amorphous materials leads to broad features in the powder pattern. These features can be analyzed using old mathematics (the Debye function), or more rigorously using new approaches and high-energy synchrotron or neutron diffraction. The results of either analysis are a radial distribution function (pair correlation function), which gives the electron density weighted distribution of inter atomic vectors in the material. Such functions can be hard to interpret on an absolute basis, but changes in catalyst supports during use can be detected and quantified.

It is important to state that the X-ray diffraction profile behavior in this work was similar to those observed by Cano-Chauca *et al.*, (2005), which showed that the powders of the mango juices obtained through spray drying process using the carriers' maltodextrin and arabic gum presented surfaces of amorphous particles. The presence of amorphous material could be due to the fact that during the drying process, the material did not crystallize because the arabic gum had high molecular weight and high viscosity that increased the glass transition temperature, turning the surface amorphous. Such change from this state to crystalline state occurred above the glass transition temperature. Microstructure analyses reported by Cano- Chauca (2005) showed that the powders of mango juices obtained through spray drying using MD, gum arabic, and waxy starch presented characteristics of amorphous particles.

Solubility analysis

In our study the dried powder was stable at room temperature of 25±2°C. The fruit powders were reconstituted after blending with water at the ratio of 1:100 at the room temperature of 25±2°C. Solubility problems occur when foods are subjected to higher temperatures, especially in products with high concentration of solids. In general, a reduction in MD concentration improved the solubility. Mahendran (2010) reported that when 30% MD was added to guava juice, the solubility of powder was 95% whereas adding60% MD decreased the solubility to 86%. The result of solubility test was 57%. Solubility test was to find amount of powder that dissolve in water.

Bulk Density

BD is a measure of heaviness of flour or powders. In this study the spray dried OFIFP had a BD of 2.5g/ml. It is an important parameter that determines the suitability of the ease of packaging and transportation of particulate foods (Shittu *et al.*, 2005).

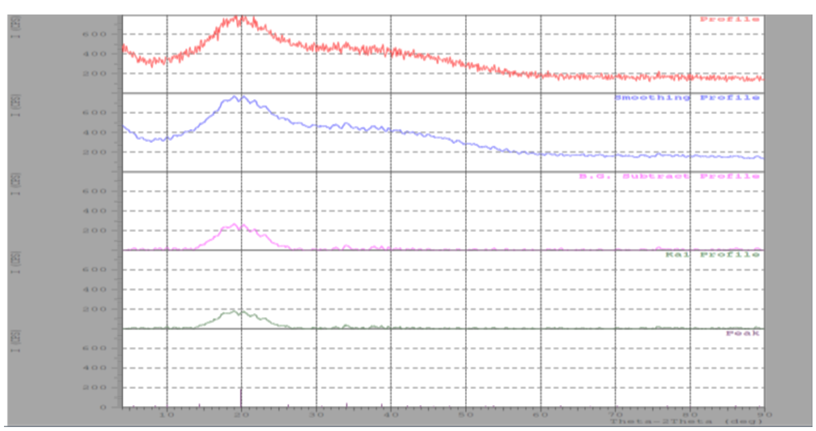
In another study by Borges *et al* (2002) the bulk and particle densities of the dried guava powders were also compared. The tunnel dried powder had significantly higher (p<0.05) bulk density than spray dried powder. However, the values found for both densities were lower viz. 0.75 and 2.47g/cm3 for bulk density and particle density, respectively. Bhandari *et al.*, 1993 found values for bulk density of 0.47– 0.58g/cm3 for temperate climate fruit powders. An increase in MD concentration during spray drying led to a significant decrease (p<0.05) in both bulk and particle densities of the powder compared to freeze and tunnel drying.

Water Absorption Capacity

WAC is important in bulking and consistency of product as well as in baking applications (Nlba *et al.*, 2001). The method described by Smith and Circle (1972) was used to determine WAC of OFIFP. The result of WAC of OFIFP was 29.6 ml.

Fig.1&2 Fresh OFI & Spray dried OFIFP

Fig.3 X-ray Diffraction Microstructure Analysis



The WAC is important in the development of ready to eat foods, and a high absorption capacity may assure product cohesiveness (Houson and Ayenor, 2002). Water holding capacity is the ability to retain water against gravity, and includes bound water, hydrodynamic water, capillary water and physically entrapped water (Moure *et al.,* 2006).

Colour Measurement

The colour of the spray dried powder was measured with the help of Lovibond Tintometer. The results of colour are expressed in value of L*, a*, and b* values. The L* value indicates the measure of lightness of a sample and is considered to be an expression of the sample's whiteness. The value ranges from 0 (black) to 100 (perfect white); with higher values indicating brighter samples (Hutchings 1994, Kurimoto and Shelton 1988). a* measures redness (+a*=red, -a*=green), meanwhile b* is related to yellowness (+b*=yellow, -b*=blue).

In the present study the values for colour measurement were found to be L* 20.3, a* 3.2 and b* 0.08. The powder was light pink in colour. The spray dried OFIFP may be incorporated into dairy or soya beverages to give an attractive product.

In a study by Mahendran (2010) the guava juice had high sugar content with low in particulates allowing satisfactory puffing during freeze drying which is responsible for the colour and shiny nature of the powders. All the powders produced by spray drying were a light yellow colour in appearance irrespective of the colour of the feed material. There were no significant differences ($p>0.05$) between the L* and a* (redness) values for powders prepared using different MD combinations. Chopda and Banrett (2004) reported the production of bright white guava powder after spray drying of puree with MD. During spray drying a* decreased as L* increased because oxidation of pigments increasingly revealed the white colour of the MD.

Cactus pear is one of the few natural sources of betalains, which are also found in beetroot, amaranth and some other Cactaceae such as the cactus pear, pitaya (*Stenocereus sp.),* pitahaya (*Hylocereus undatus*) and garambullo (*Myrtillocactus geometrizans*). The current source of commercial betalains, which have been used as natural food colours for many years, is beetroot (*Beta vulgaris*). Betanin (also termed "beetroot-red") is accepted amongst the natural pigments that are classified as additives E-162 (European Union) and 73.40 (FDA). It is used mainly in foods that do not require thermal treatment, such as yogurt, confectionery, ice cream, syrups and sausages (Vergara *et al,* 2015).

Conclusions

The present study has shown the successful process for spray drying of *opuntia ficus indica*. The evaluation of the raw materials and product showed that it will have a viable market. This study clearly revealed that the Fruit juice powders have many benefits and economic potentials over their liquid counterparts such as reduced volume or weight, reduced packaging, easier handling and transportation, and much longer shelf life. The quality of spray dried food is quite dependent on the operating parameters. Thus, an understanding of factors affecting the product properties is required for the process optimization. The developed product may be suitable for value addition. The microcapsules described in this study represent an interesting food additive for incorporation into functional foods, due to both the presence of antioxidants and as a red colourant.

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How to cite this article:

Sujitha, S., and Jemima Beryl Mohankumar. 2017. A Natural Colourant Spray Drying of Cactus Pear (*Opuntia Ficus Indica*) Juice and Its Physico-Chemical Characteristics. *Int.J.Curr.Res.Aca.Rev*. 5(4), 56-63.
doi: https://doi.org/10.20546/ijcrar.2017.504.009