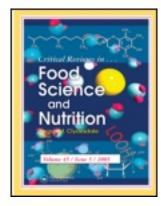
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A Critical Review on the Spray Drying of Fruit Extract: Effect of Additives on Physicochemical Properties

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A Critical Review on the Spray Drying of Fruit Extract: Effect of Additives on Physicochemical Properties

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Spray drying accomplishes drying while particles are suspended in the air and is one method in the family of suspended particle processing systems, along with fluid-bed drying, flash drying, spray granulation, spray agglomeration, spray reaction, spray cooling, and spray absorption. This drying process is unique because it involves both particle formation and drying. The present paper reviews spray drying of fruit extracts, such as acai, acerola pomace, gac, mango, orange, cactus pear, opuntia stricta fruit, watermelon, and durian, and the effects of additives on physicochemical properties such as antioxidant activity, total carotenoid content, lycopene and β -carotene content, hygroscopy, moisture content, volatile retention, stickiness, color, solubility, glass transition temperature, bulk density, rehydration, caking, appearance under electron microscopy, and X-ray powder diffraction. The literature clearly demonstrates that the effect of additives and encapsulation play a vital role in determining the physicochemical properties of fruit extract powder. The technical difficulties in spray drying of fruit extracts can be overcome by modifying the spray dryer design. It also reveals that spray drying is a novel technology for converting fruit extract into powder form.

Keywords Spray drying, fruit extract, antioxidant, additives, encapsulation

INTRODUCTION

Fruits that easily deteriorate during storage have to be preserved. The storage period of fruits must be prolonged to maintain their nutritional value and enable offseason supply (Hsu et al., 2003). The advantages of a dried extract over conventional liquid forms are lower storage costs, a higher concentration, and the stability of active substances. Spray drying has been adopted to manufacture powders due to its ability to generate a product with precise quality specifications in continuous operation.

Spray drying is a well-established and widely used technique to turn liquid foods or suspensions into powder form in a one-step process and is also suitable for the preparation of coated crystals as intermediates for tablet manufacture. There are three main steps in the spray-drying operation: (1) atomization of the liquid feed, (2) drying of the droplets once they are formed, and (3) motion of the droplet (Shabde and Hoo, 2007).

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Spray drying refers to the removal of moisture from slurry by breaking it into small droplets in the presence of hot air to obtain a solid, dry powder. In the spray-drying process, the liquid feed is pumped into the drying chamber through an atomizing system. Inside the spraying chamber, a stream of heated gas traps the droplets and carries them from the drying chamber to the product recovery system. Evaporation takes place in a few seconds as the relatively cool droplets come in contact with the hot gas (Al-Asheh et al., 2003).

The drying time of the droplets in the spray dryer is very short compared with that of most other drying processes. The drying time for droplets depends on the process conditions such as flow rate, pump rate, aspiration rate, and heat. There are many types of dryers that use hot air flow to dry fruits, including the constant bed dryer, fluidized bed dryer, and microwave dryer. Hot air coming into contact with solid products such as grains, vegetables, and fruit pulps dries them. These dryers require a long time for the drying process. The low product temperature and short drying time suggest that the spraydrying process is appropriate for heat sensitive products such as foods, dairy products, and fruit juices (Roustapour et al., 2009).

The products to be spray dried can be categorized into two major groups: nonsticky and sticky products. Sticky products are generally difficult to use in the spray-drying process. During the drying process, they may remain as a syrup, stick on the dryer wall, or form unwanted agglomerates in the dryer chamber and conveying system, which causes lower product yields and operating problems. Some examples of such sticky products are fruit and vegetable juice powders, honey powders, and amorphous lactose powder. Nonsticky products can be dried using a simpler dryer design, and the obtained powder is relatively less hygroscopic and more free flowing (Goula and Adamopoulos, 2005).

Natural hygroscopic and thermoplastic properties are the basic problems in the transport and handling of fruit juice powder produced by spray drying. Sugar-rich materials are difficult to spray dry because they produce highly hygroscopic powders that are prone to stickiness and flow problems. Possible consequences include impaired product stability, decreased yields (because of stickiness on the drier chamber walls), and even operational problems with the spray drier. The sticky behavior is attributed to a high concentration of low molecular weight sugars and organic acids, which have low glass transition temperatures $(T_{\rm g})$ and are rubbery and thermoplastic at the temperatures in the chamber. The fast moisture removal during spray-drying results in an amorphous and highly hygroscopic product. Stickiness does not occur when higher molecular weight carbohydrates such as maltodextrins (MDs) are spray dried. Instead, MDs facilitate drying of sugar-rich foods because their T_g increasing effect reduces the hygroscopicity of powders. Thus, they are frequently used as drying aids (Moreira et al., 2008).

Another issue is the complex chemical composition of juices. About 90% of dry substances in juices consist of hydrocarbons such as monosaccharides (glucose, fructose), disaccharides (sucrose), and polysaccharides. Bhandari et al. (1993) used a variety of methods to obtain powder from concentrated juices. According to their experiments, the results were obtained for a juice to MD ratio of 65/35 for blackcurrant, 60/40 for apricot, and 55/45 for raspberry at inlet air temperatures between 90 and 160°C (Roustapour et al., 2009).

Spray drying is also a useful technique for producing powders suitable for inhalation (Corrigan et al., 2006). The physicochemical properties of powders produced by spray drying depend on process variables such as the characteristics of the liquid feed (viscosity, particle size, flow rate) and of the drying air (temperature, pressure), as well as the type of atomizer. Therefore, it is important to optimize the drying process to obtain products with better sensory and nutritional characteristics and to optimize process yield. Evaluating the surface properties of these particles can improve our understanding of the production process and assist in the optimization of the powder composition.

Although thermal degradation can potentially be avoided in spray drying, degradation during atomization may constitute a problem to biopharmaceuticals. Atomization requires high shear rates that can denature proteins. The large surface area of the droplets improves mass transfer but can sometimes serve as a site

for the degradation of biopharmaceuticals. The use of suitable excipients can reduce denaturation during spray drying. For instance, polysorbate-20 has been used to reduce spray-drying-induced denaturation of human growth hormone.

Careful selection of operating parameters is critical to obtain a high-quality product during spray drying. Spray drying has relatively low cyclone collection efficiency for particles smaller than 2 μ m. The typical yield from a spray dryer is between 20 and 50%, but the new high-performance cyclone from Buchi in Switzerland has improved the yield to greater than 70%.

Another important issue in spray drying is the lack of control over the mean droplet size, which leads to a potentially broad distribution of droplet sizes and the risk of clogging if pneumatic nozzles are used. Using an ultrasonic nozzle generates more uniform droplets and thus a relatively homogenous size distribution of the produced powder. However, the energy needed for atomization may further contribute to the denaturation of proteins.

Few researchers have succeeded in producing hollow protein microcapsules using spray drying. Sutton et al. (2000) prepared a fine powder intended for ultrasonic imaging contrast enhancement, but many of the prepared particles had dimensions in the respirable range. The above author was also able to show the effect of nozzle type on the dimensions of particle obtained. When a two-fluid nozzle was used for atomization, the particle size ranged from 4 to 6 μ m, while particles between 1 and 8 μ m were obtained when a centrifugal nozzle was used.

Some problems such as stickiness, hygroscopy, and solubility can be solved by adding carrier agents such as biopolymers and gums to the product before atomization. These agents are normally used for microencapsulation and can reduce powder hygroscopicity, protect sensitive food components from unfavorable ambient conditions, reduce the volatility and reactivity of the food components, and make the product more attractive for consumption (Tonon et al., 2008).

Microencapsulation techniques offer protection and controlled release of food ingredients. Generally, encapsulation techniques can be divided into three classes: chemical processes like molecular inclusion or interfacial polymerization; physicochemical techniques like coacervation and liposome encapsulation; and physical processes like spray drying, spray chilling/cooling, co-crystallization, extrusion, or fluidized bed coating (Drusch and Berg, 2008).

The encapsulation of active components in powders has become an attractive process in recent decades not only in the food industry but also in the pharmaceutical industry. The main objective is to entrap a sensitive ingredient within a capsule or wall to physically isolate the ingredient from the environment. For example, this barrier may confer protection against oxygen, water, or light, allow controlled release of the entrapped ingredient, or prevent contact with other constituents in a mixture. The wall material is generally made of compounds that create a network-like structure. These compounds usually harbor hydrophilic or hydrophobic groups (e.g., starches, gums, gelatins, or polymers; Turchiuli et al., 2005).

The effect of various excipients on the biological activity and aerosol performance of spray-dried lysozyme and catalase has also been studied (Liao et al., 2005). Sucrose, trehalose, and polyvinyl alcohol (PVA) were studied as potential stabilizers for proteins during spray drying. Pure lysozyme lost just over 10% of its original biological activity, while the incorporation of sucrose, trehalose, or a combination of trehalose and PVA with lysozyme preserved approximately 100% of its original biological activity.

Pure catalase retained 55% of its biological activity upon spray drying, but this retention of activity increased to approximately 93% when either sucrose or trehalose was used to stabilize the enzyme during the manufacturing process. Catalase retained almost full activity when it was stabilized using a mixture of PVA—trehalose. Scanning electron microscopy (SEM) showed that the morphology of the spray-dried powders appeared to be similar regardless of the type and quantities of excipients used to stabilize the protein (Shoyele and Cawthorne, 2006).

ACAI (Euterpe oleracea Mart.) JUICE

Tonon et al. (2010) studied the spray drying of acai juice. Acai (*Euterpe oleracea* Mart.) is a fruit from Amazonia with great abundance and economic importance in the Brazilian state of Para. Beyond its high energy content, acai has been recognized to have functional properties and is considered an important source of anthocyanins, which have high antioxidant activity. This natural antioxidant potential can serve as preventive medicine (Krishnaiah et al., 2011).

Spray drying is an alternative process for producing acai powder with high anthocyanin content. This technique has been widely used in the microencapsulation of food ingredients susceptible to deterioration by external agents and consists of entrapping an active agent (solid particles, liquid droplets, or gaseous compounds) in a polymeric matrix to protect it from adverse conditions. Microencapsulation has been used by the food industry to protect sensitive food ingredients during storage, to mask or preserve aromas and flavors, to protect food against nutritional losses, or even to add nutritive materials to foods after processing (Re, 1998).

MDs and Arabic gum (AG) are normally used as the carrier agents in the spray drying of fruit juices (Cano-Chauca et al., 2005; Gabas et al., 2007; Quek et al., 2007) because of their high solubility and low viscosity, which are important conditions for the spray-drying process. Tapioca starch is also used by some Brazilian companies in the production of powdered fruit juices to ensure that no genetically modified material is used in the powder production, a condition required by some importing countries. These carrier agents, which have a high molecular weight, are useful for increasing the product's glass transition temperature. A higher temperature reduces operational problems encountered in spray drying such as stickiness on the dryer chamber wall and structural transformations such as collapse and crystallization during food processing and storage,

an especially important issue in the case of sugar-rich products such as fruit juices.

In the case of acai juice, spray drying with carrier agents such as MD 10 DE, MD 20 DE, AG, and tapioca starch results in a nutritionally rich product with the potential for preservation of antioxidant activity upon storage.

Spray-Drying Conditions

The spray-drying process of acai was performed in a laboratory scale spray dryer Labplant SD-05 (Huddersfield, England) with a 1.5 mm diameter nozzle and a main spray chamber that is $500 \text{ mm} \times 215 \text{ mm}$. The mixture was kept under magnetic agitation at room temperature and fed into the main chamber through a peristaltic pump with a drying air flow rate of 73 m³/hour and a compressor air pressure of 0.06 MPa. A feed flow of 15 g/minute was used, and the inlet and outlet air temperatures were $140 \pm 2 \text{ and } 78 \pm 2^{\circ}\text{C}$, respectively (Table 1).

Antioxidant Activity

According to Tonon et al. (2010), the antioxidant activity in the filtered pulp of acai before spray drying was equal to $1230.43 \pm 59.20 \mu \text{mol TE/g}$ dried matter. The antioxidant activity after spray drying with MD 10 DE, MD 20 DE, AG, and tapioca starch as carrier agents were found to be 1165.84 \pm 35.29, 1101.73 ± 36.11 , 1118.37 ± 48.02 , and 1010.87 ± 48.02 41.97 μ mol TE/g juice-dried matter (Table 2). Powders produced with MD and AG did not significantly differ with respect to antioxidant activity, while the powder produced with tapioca starch showed the lowest 1,1-diphenyl-2-picrylhydrazyl (DPPH) scavenging activity after the spray-drying process. MDs and AG are highly soluble materials, and thus, when the feed mixture passes through the spray dryer, the resulting powder forms hollow particles in which the crust is a matrix containing both the carrier agent and the entrapped juice, which can be considered a "microencapsulated juice." Tapioca starch is highly insoluble, and the resulting powder probably consists of particles of the dried juice and separate particles of tapioca starch. In this case the juice is not encapsulated, and the carrier agent is only used to facilitate the drying process. This lack of encapsulation is a likely reason for the lower antioxidant activity when this agent was used.

Researchers have suggested that acai contains some compounds that have not yet been identified that are responsible for its high antioxidant activity. Wu et al. (2004) evaluated the relationship between antioxidant activity and the polyphenolic content of various fruits and vegetables and found that most had a ratio of antioxidant activity to polyphenolic content of about 10. However, Schauss et al. (2006) found that the ratio for acai pulp was about five times greater than that found for the other fruits. According to the researchers, this "unusual" ratio raises questions of whether acai contains much stronger antioxidants than those found in other berries, and research

 Table 1
 Spray-drying conditions, carrier agents, and physicochemical properties of spray-dried fruit extract

S. no.	o. Fruit extract	Spray-drying conditions	Carrier agent	Maximum value of physicochemical properties	Physicochemical properties of powder	Effect of the spray variables on the product yield and the powder properties	References
_	Acai (Euterpe oleracea Mart.) juice	Air flow rate: 73 m³/hour; Compressor air pressure: 0.06 MPa; Feed flow rate: 15 g/minute; Inlet temperature: 140 ± 2°C; Outlet temperature: 78 ± 2°C	Maltodextrin 10 DE, maltodextrin 20 DE, Arabic gum, and tapioca starch	Antioxidant activity: 1165.84 ± 35.29 μmol TE/g juice-dried powder (maltodextrin 10 DE)	Bulk density, absolute density, porosity, antho cyanin content, and antioxidant activities were found out for different carrier agents (data shown in Table 2)	Both temperature and water activity negatively affected anthocyanin stability. Antioxidant activity was also decreased with increasing water activity for the powders stored at 25°C but was higher for the powders stored at 35°C.	Tonon et al., 2010
6	Acerola pomace	Feed rate: 0.49 kg/hour; Peristaltic pump rate: 1.23 kg/hour; Aspirator flow rate: 5.51 × 10 ⁴ kg/hour; Inlet temperature > 194°C	Maltodextrin and cashew tree gum	Hygroscopicity: 34.72 ± 1.93 (g absorbed water/ 100 g powder after 7 day storage at 25°C, 90% RH)	Moisture content and hygroscopicity were found out for different inlet temperature as per Table 3	Higher inlet temperatures favored the desired physical properties of the powders, decreasing their moisture contents and hygroscopicity	Moreira et al., 2008
С	Gac (Momordica cochinchinensis) fruit	Drying air flow rate: $56 \pm 2 \text{ m}^3\text{/hour}$; Compressor air pressure: 0.06 MPa; Feed rate: 12–14 ml/minute	Maltodextrin	Total carotenoid content (TCC): 2.8 mg/g (10% maltodextrin); Total antioxidant activity (TAA): 0.14 mmol TE/g of powder (spray-drying temperature 120°C and 10% maltodextrin (w/v))	Moisture content, color characteristic, total carotenoid content, and total antioxidant activity were found under different drying conditions as per Figs. 1 and 2	Moisture content, color characteristic, total carotenoid content, and total antioxidant activities were significantly affected by maltodextrin concentration and the inlet air temperatures	Kha et al., 2010
4	Mango juice	Inlet air temperature: 160°C; Outlet air temperature: 70–75°C; Liquid feed: 10 ml/minute; Drying air flow rate: 0.7 m³/minute	Maltodextrin, Arabic gum, and waxy starch	Stickiness: 0.15 kg-f (maltodextrin + 9% cellulose); Stickiness: 0.22 kg-f (Arabic gum + 9% cellulose); Stickiness: 0.11 kg-f (waxy starch + 9% cellulose); Solubility: 90% (maltodextrin + 0% cellulose)	Electron microscopic photographs and X-ray powder diffraction of spray-dried mango juice powder with and without cellulose concentration for different drying aids were discussed. Stickiness and solubility were determined as a function of cellulose concentration for different drying aids such as maltodextrin, Arabic gum, and waxy starch as shown in Figs. 3 and 4	Value of stickiness decreases as a function of cellulose concentration for all the different drying aids tested. Solubility decreases as a function of cellulose concentration	Cano-Chauca et al., 2005

sed as Goula et al., ce, 2009 adextrin proved y of nation. rature noisture inlet air todextrin creases ose gher Tg gher Tg	Saenz et al., 2009 pulp lin were et the nd type nt did	ariables Obon et al., powder 2009 gth and	ate, Quek et al., he spray 2007 ased with ad outlet igher color of darker. creased	durian Chin et al., antly 2010 3f ss and ure
Dehumidified air was used as drying medium. Hence, combination of maltodextrin addition and use of dehumidified air was proved to be an effective way of reducing residue formation. Increase in air temperature leads to decrease in moisture content. Increases in inlet air temperature and maltodextrin concentration and decreases in maltodextrin dextrose equivalent led to a higher Tg and a lower degree of caking and lower degree of caking	Sin	O	At constant feed flow rate, moisture content of the spray dried powders decreased with the increased inlet and outlet air temperature. At higher inlet temperature the color of the powders became darker. Lycopene content decreased with increased inlet	The carporation The retention of target durian volatiles was significantly affected by the type of incorporated additives and inlet drying temperature
Relationship between glass transition temperature and moisture content of spray-dried orange juice concentrate were determined by Goula et al., (2009) as per Fig. 5 and 6. Bulk density, rehydration, hygroscopy, and caking degree were also discussed by the authors	Electron microscopic photographs of microcapsules were discussed in Fig. 7. Antioxidant activity was also found	Color strength and SEM were determined by Obon et al., (2009). Bulk density and moisture content were also found and results are shown in Table 5	Color measurement, moisture content, lycopene, and β -carotene content were determined (Tables 6–8; Fig. 8)	Surface morphology of the spray-dried powder was discussed by the author. Retention of volatiles in spray-dried powder was also tabulated in Table 9.
Powder hygroscopy: 0.04 (g/g solids) (inlet temperature: 140°C; Dextrose equivalent: 6 and concentrated orange juice solids/ maltodextrin solids: 0.25)	Antioxidant activity of cactus pulp-maltodextrin at optimal conditions: 34 ± 1.23 mmol TEAC/g	Bulk density: 0.53–0.59 g/ml; Moisture content: 3.1– 3.9%	Moisture content: 1.49–2.78% (MD 3%), 1.49–1.62% (MD 5%); Lycopene content: 907.66 \pm 2.15 μ g/g and β -carotene content: 29.47 \pm 0.61 μ g/g (155°C inlet temperature, 5% maltodextrin)	Volatile retention: propanethiol: $62 \pm 10\%$, Ethyl propanoate: $52 \pm 15\%$, E2 MB: $76 \pm 10\%$, Diethyl disulfide: $77 \pm 1\%$ (MG at 130° C T;)
Maltodextrin	Maltodextrin and inulin	Glucose syrup	Maltodextrin (3% and 5%)	Gum Arabic; Maltodextrin; N-Lok starches
Atomizer pressure: 5 ± 0.1 bar; Feed rate: 1.8 ± 0.1 g/minute; Inlet temperature: $110-140$ ($\pm 1^{\circ}$ C)	Inlet temperature: 140–160 ± 5°C (maltodextrin); Inlet temperature: 120–140 ± 5°C (inulin); Air flow rate: 600 I/hour; Feed flow rate: 10 ml/minute; Atomization pressure: 20 psi	Inlet temperature: 80–160°C; Outlet temperature: 50–68°C; Air flow rate: 0.47 m³/hour; Liquid feed rate: 0.36–0.72 Whour	Atomizer pressure: 4.5 bar; Inlet temperature: 145–175°C; Air flow rate: 0.6 m³/hour	Atomizer pressure: 2 bar, Inlet temperature: 130° C; Outlet temperature: $88 \pm 3^{\circ}$ C
5 Orange juice	6 Cactus pear (Opuntia ficus-indica)	7 Opuntia stricta fruit	8 Watermelon	9 Durian

Table 2 Bulk density, absolute density, and porosity of powders produced with different carrier agents (adapted from Tonon et al., 2010)

Carrier agent	Bulk density (g/cm ³)	Absolute density (g/cm ³)	Porosity (%)	Anthocyanin content (mg/100 g juice-dried matter)	Antioxidant activity (μ mol TE/g juice-dried matter)
Maltodextrin 10 DE	0.390 ± 0.015^{a}	1.531 ± 0.004^{a}	74.50 ± 1.01^{a}	3436.85 ± 79.18^{a}	1165.84 ± 35.29^{a}
Maltodextrin 20 DE	0.370 ± 0.016^{a}	1.511 ± 0.004^{b}	75.49 ± 1.07^{a}	3402.30 ± 167.33^{a}	1101.73 ± 36.11^{a}
Gum Arabic	0.377 ± 0.011^{a}	1.491 ± 0.008^{c}	74.70 ± 0.72^{a}	3415.96 ± 68.08^{a}	1118.37 ± 48.02^{a}
Tapioca starch	0.480 ± 0.005^{b}	1.514 ± 0.001^{b}	68.33 ± 0.30^{b}	3247.15 ± 69.70^{b}	1010.87 ± 41.97^{b}

a-c Means with different superscript letters in the same column indicate significant differences ($p \le 0.05$).

should be conducted to determine which antioxidants contribute to this unusual ratio. Lichtenthaler et al. (2005) evaluated the antioxidant capacity of some acai pulp samples and compared it to the antioxidant capacity of standard cyanidin-3-glucoside and cyanidin-3-rutinoside standard solutions (the predominant anthocyanins in acai) to estimate the contribution of these individual anthocyanins to the overall antioxidant activity. The authors verified that the antioxidant capacity of the acai samples was about 11-fold higher than what would be expected from standard anthocyanin solutions and concluded that the antioxidant capacities of acai fruit must be due to other compounds that have not yet been identified.

Using MD 10 DE as the carrier agent yielded particles with the highest degradation half-lives, followed by AG. Tonon et al. (2010) concluded that antioxidant activity also decreased with increasing water activity, which is analogous to anthocyanin content. However, the increase in temperature caused higher antioxidant activity, which was explained by two hypotheses: the presence of unidentified compounds in acai that contribute to its antioxidant capacity and the Malliard reaction, which produced compounds with antioxidant activity.

ACEROLA POMACE

Moreira et al. (2008) studied spray drying of acerola pomace extract. Acerola (Malpighia emarginata D.C.), also known as the Barbados cherry or West-Indian cherry, is present in South and Central America as well as in some southern regions of North America (Johnson, 2003). Acerola is a round red fruit with a diameter that varies from 1 to 4 cm and a thin, easily bruised skin. Demand for this fruit has increased in recent decades because of its high ascorbic acid content, which ranges from 1000 to 4500 mg/100 g (Leung and Foster, 1996; Johnson, 2003; Mezadri et al., 2006). Brazil is currently the world's leading producer and exporter of acerola (Mezadri et al., 2006), especially as a frozen puree and juice (Yamashita et al., 2003). Acerola processing involves crushing and pressing the whole fruit, which generates red pomace as a byproduct, which is usually discarded despite its high-value compounds (particularly vitamin C and flavonoids that have known antioxidant properties).

Some studies have also indicated that AG has higher $T_{\rm g}$ values than those of MD with a dextrose equivalent (DE) of 10 or higher (Righetto and Netto, 2000; Collares et al., 2004), which suggests that AG may reduce powder hygroscopicity more effective.

tively than MD. However, the high cost and availability problems associated with AG have motivated researchers to look for replacement materials (Mcnamee et al., 1998). Cashew tree gum (CTG), a complex water soluble heteropolysaccharide extracted from a cashew tree (*Anacardium occidentale*), has been demonstrated to be a very promising material due to its structural similarity to AG (Zakaria and Rahman, 1996; De Paula et al., 1998; Paula et al., 2002). The replacement of AG by CTG, previously suggested by some authors (Rosenthal, 1951; Owusu et al., 2005), could reduce costs with AG importation and would be a boon for the cashew tree business, whose only high-value-added product is currently the cashew nut.

Spray-Drying Conditions

The spray-drying process of acerola pomace extract was conducted in a mini spray dryer Buchi B-290 (Buchi Labortechnik AG, Flawil, Switzerland) under the following operational conditions: feed rate, 0.49 kg/hour; peristaltic pump rate, 1.23 kg/hour; aspirator flow rate, 5.51×10^4 kg/hour.

Hygroscopicity

Hygroscopicity is defined as the moisture mass (g) absorbed by 100 g of a powder during seven days of storage at 25°C and 90% relative humidity (RH; in a desiccator with a barium chloride saturated solution), which is a modification of the method described by Callahan et al. (1982). Moisture content is one of the major factors affecting powder stability because a small amount of water is able to depress the $T_{\rm g}$ far enough to increase the mobility of the matrix during storage (Roos and Karel, 1992; Roos, 2002; Bhandari and Hartel, 2005). Increasing the drying aid/acerola ratio reduced the powder hygroscopicity, which confirms the behavior described in previous studies (Peleg and Hollenbech, 1984; Bhandari et al., 1993, 1997a; Bhandari and Hartel, 2005; Silva et al., 2006). High proportions of the drying agent tended to slightly decrease the solubility of the powders, which confirms results described by Abadio et al. (2004) and Cano-Chauca et al. (2005).

Higher proportions of CTG enhanced the powder flowability and decreased hygroscopicity, which suggests that CTG increases the $T_{\rm g}$ of the powders more effectively than MD. Although no other studies have characterized the effects of CTG on $T_{\rm g}$, the similarity between the structures of CTG and AG

 38.70 ± 2.03

 47.34 ± 2.81

 46.76 ± 2.42

 48.53 ± 2.39

14

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16

17

Drying aid/acerola Degree of Hygroscopicity solid ratio (g of replacement of Moisture content (g absorbed water/ Inlet temperature drying aid per g of maltodextrin by of the powder 100 g powder after 7 day (g/100 g)storage at 25°C) Treatment (°C) acerola solids) cashew tree gum (%) 176 20.2 4.52 ± 0.21 51.16 ± 2.44 1 2.6 2 194 2.6 20.2 3.70 ± 0.17 43.91 ± 2.14 3 176 4.4 20.2 4.31 ± 0.19 47.12 ± 2.38 4 194 4.4 20.2 3.62 ± 0.15 38.73 ± 1.82 5 176 79.8 46.45 ± 2.22 2.6 4.95 ± 0.22 6 194 2.6 79.8 4.22 ± 0.20 41.52 ± 2.03 176 4.4 79.8 3.91 ± 0.18 40.31 ± 1.90 8 194 4.4 79.8 3.09 ± 0.16 34.72 ± 1.93 170 3.5 56.44 ± 2.71 50.0 5.43 ± 0.25 10 200 3.5 50.0 3.96 ± 0.20 40.04 ± 1.92 11 185 2.0 50.0 5.31 ± 0.22 49.10 ± 2.42 12 185 5.0 50.0 3.8 ± 0.15 34.97 ± 1.79 13 185 3.5 0.0 4.65 ± 0.21 49.36 ± 2.49

100.0

50.0

50.0

50.0

Table 3 Experimental conditions and responses of the spray-drying treatments (adapted from Moreira et al., 2008)

3.5

3.5

3.5

3.5

suggest that both would have similar $T_{\rm g}$ effects. Thus, the results of Moreira et al. (2008) can be compared with those reported by Righetto and Netto (2000) and Collares et al. (2004), who reported that the $T_{\rm g}$ -increasing effect of AG is higher than that of MD.

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Hygroscopicity must not be evaluated by its absolute value because these studies exposed the powders to abnormally abusive conditions. The powders produced at higher temperatures, with a higher drying aid/acerola ratio and a high degree of replacement of MD by CTG, were the least hygroscopic. Among the conditions studied by Moreira et al. (2008), the best processing conditions to obtain a free-flowing and least hygroscopic acerola pomace extract powder by spray drying were an inlet temperature above 194°C and a drying aid/acerola ratio of 4, in which the drying aid is at least 80% CTG. Under these conditions, the hygroscopicity was found to be 34.72 ± 1.93 (g absorbed water/100 g powder after seven-day storage at 25°C, 90% RH) as in Table 3.

The CTG is abundant and inexpensive in several tropical developing countries. Moreira et al. (2008) concluded that the assessment of new applications for this virtually unexploited polysaccharide may be important to motivate industry to recognize its potential. If properly exploited, CTG can greatly impact the cashew tree business and bring socio-economic benefits to countries with cashew tree growth.

GAC (Momordica cochinchinensis) FRUIT ARIL POWDER

Kha et al. (2010) studied spray drying of Gac fruit. Gac fruit, *Momordica cochinchinensis* Spreng, contains extraordinarily high levels of carotenoids, especially β -carotene (more than 16 mg/100 g) and lycopene (more than 50 mg/100 g),

and these compounds are primarily located in the red aril (Bauernfield, 1971; Vuong, 2000; Aoki et al., 2002). Consumption of carotenoid-rich Gac fruit can increase plasma β -carotene and retinol levels (Vuong et al., 2002) and has been linked with a lower risk of prostate cancer (Guns and Cowell, 2005; Chan et al., 2009) and coronary heart disease (Rao and Agarwal, 1999). Moreover, Gac fruit contains comparatively high concentrations of other nutrients such as Vitamin E (7.6 mg/100 g), unsaturated, and polyunsaturated fatty acids (852 mg/100 g of edible portion) (Vuong, 2000; Vuong et al., 2006) that are known to be beneficial for human health. The presence of fat in the Gac pulp plays an important role in the absorption of carotenes, vitamin E, and other fat-soluble nutrients (Kuhnlein, 2004). It is desirable for this fruit to be in powder form for ease of consumption, and the resulting powder has a high nutrient content and an attractive red color.

 3.48 ± 0.21

 4.88 ± 0.24

 4.35 ± 0.21

 5.17 ± 0.27

Spray drying has been widely utilized for commercial production of dried fruits and vegetables. Spray-dried powders have good reconstitutional characteristics and low water activity and are suitable for transport and storage.

Several additives can be used as drying aids such as MD, AG, and gelatin. MD is currently one of the most common drying aids for spray drying because of its beneficial role as a carrier or an encapsulating agent that increases the stability of carotenoids; moreover, it is reasonably cheap and commercially available. The addition of MD before spray drying has been reported to effectively preserve carotenoids such as β -carotene (Desobry et al., 1997); carrot carotenes (Wagner and Warthesen, 1995); black currant, apricot, and raspberry juices (Bhandari et al., 1993); guava juice (Chopda and Barrett, 2001); and pineapple juice (Abadio et al., 2004). Furthermore, the color of foods is one of the most important sensory attributes that are affected by many factors during spray drying such as the inlet temperature and additives (Abadio et al., 2004).

Spray-Drying Conditions

The feed mixtures containing added MD and red flesh Gac fruit juice were spray-dried in a lab plant SD-05 spray dryer (Lab plant Ltd., England). The inlet temperatures/measured outlet temperatures were 120/83°C, 140/94°C, 160/103°C, 180/112°C, and 200/125°C. The drying air flow rate, compressor air pressure, and feed rate were constant at 56 ± 2 m³/hour, 0.06 MPa gauge, and 12–14 ml/minute, respectively. After the spraying process, the Gac fruit powder was collected in a glass collection vessel wrapped with aluminium foil and immediately stored in a dessicator containing silica gel for equilibration to room temperature. The spray-drying processes were all carried out in duplicate.

Moisture Content

Kha et al. (2010) showed that increasing the MD concentration and drying temperature resulted in a lower moisture content. As the MD concentration increased from 10 to 20%, the moisture content of the samples was significantly reduced from 4.87 to 4.06%. A similar trend was observed when the drying temperatures were increased from 120 to 200°C, which caused a significant drop in moisture content from 5.29 to 3.88%.

For spray drying in general, increasing the drying temperature resulted in a greater loss of water from the resultant powder because of the higher rate of heat transfer into the particles, which caused faster water removal. Similarly, Goula et al. (2004); Chegini and Ghobadian (2005); Rodriguez-Hernandez et al. (2005); and Ersus and Yurdagel (2007) reported that the moisture content in tomato powder, orange juice powder, cactus pear juice powder, and black carrot powder, respectively, decreased as the drying temperature increased.

Moreover, as per Kha et al. (2010) a decrease in the moisture content of Gac powder was also obtained when the MD concentration increased. Similarly, Abadio et al. (2004) found that an increase in the concentration of MD 10 DE from 10 to 15% reduced the moisture content of the resulting pineapple juice powders. Grabowski et al. (2006) also reported a similar result in tests on sweet potato puree powder. These findings can be explained by the fact that higher concentrations of MD increased feed solids and reduced total moisture for evaporation. The values of pH, water activity $a_{\rm w}$, and the water solubility index (WSI) of the Gac powders were not significantly affected by inlet air-drying temperature or MD concentration (Kha et al., 2010).

Color Characteristics

The color characteristics of spray-dried powders were significantly impacted by the MD concentration and drying temperature. The lightness of the product color was significantly affected by MD concentration. An increase in the lightness of

the products was obtained by increasing the MD concentration from 10 to 20%. However, there was no significant difference in the lightness of samples when the concentration increased from 20 to 30%. The MD concentration affected the chroma value of the samples. Increasing the MD from 10 to 20% decreased the chroma from 34.18 ± 5.97 to 25.94 ± 3.01 (Kha et al., 2010).

Total Carotenoid Content (TCC)

The total carotenoid content (TCC) in powder samples was reduced from 1.95 mg/g to 0.61 mg/g of powder as the MD concentration increased from 10 to 30% (Fig. 1). The TCC was significantly decreased when the MD concentration was increased from 10 to 30% because the high MD concentration causes lower TCC when the feed juice is constant (Kha et al., 2010). An increase in encapsulation efficiency (EE) was observed because of the increasing MD concentration. The carotenoid content in powder is well known to be protected in a high initial feed solid. Similar observations were also found in other studies (Wagner and Warthesen, 1995; Rodriguez-Huezo et al., 2004).

Total Antioxidant Activity (TAA)

The MD concentration and the drying temperature significantly affected the TAA of powders. There was no significant difference in TAA between the samples of Gac fruit when MD was added at concentrations of 10 and 20% (Fig. 2). However, when the concentration increased from 20 to 30%, a decrease in TAA was observed. Overall, as the drying temperature increased from 120 to 200°C, a significant decrease in TAA was observed

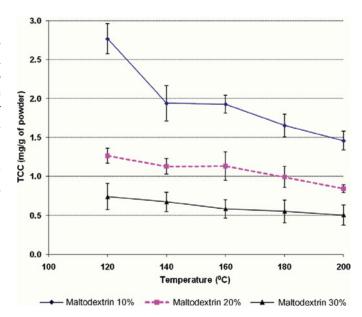


Figure 1 Total carotenoid content of spray-dried powders as a result of different drying conditions (adapted from Kha et al., 2010). (Color figure available online.)

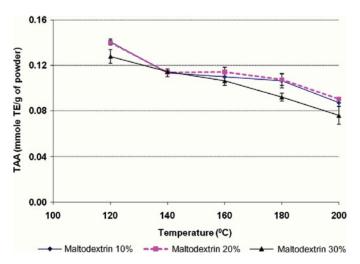


Figure 2 Total antioxidant activity of spray-dried powder under different drying conditions (adapted from Kha et al., 2010). (Color figure available online.)

(from 0.14 to 0.08 mmol TE/g powder). However, there was no significant difference between the TAA of samples spray-dried at temperatures of 140 and 160°C (Kha et al., 2010).

In a similar pattern to the TCC results, increasing the MD concentration and drying temperature decreased the TAA of powder samples because the loss of TCC at higher drying temperatures, a major antioxidant compound in spray-dried Gac powder, leads to a decrease in TAA. Similarly, the decrease in TAA results from the increasing MD content, which also lowers TCC. Kha et al. (2010) showed that the highest TAA was obtained with a spray-drying temperature of 120°C and 10% w/v MD.

The recommended daily intake level of carotenoids is 0.7–16.5 mg (Muller, 1996). Many studies also indicate that consumption of carotenoid-rich fruits and vegetables, especially lycopene, has been linked with lower risk of prostate cancer (Guns and Cowell, 2005; Chan et al., 2009). Furthermore, the TCC of Gac powder (2.8 mg/g) is much higher than that of other carotenoid-rich powders such as hot-air-dried pumpkin (0.14 mg/g) and carrot (1.1 mg/g) (Muratore et al., 2008) and spray-dried watermelon powder (1 mg/g) (Quek et al., 2007). Therefore, Gac powder is highly recommended due to its high levels of carotenoids and TAA.

The TAA in the powder is not only based on the TCC but also on other antioxidative components in Gac powders such as α -tocopherol (vitamin E) and fatty acids, which also benefit from encapsulation and exhibit synergistic effects.

Kha et al. (2010) demonstrated that there is a strong correlation between TCC and TAA (0.915 $\leq R^2 \leq$ 0.948), with the highest correlation found at the lowest MD concentration. In contrast, Thaipong et al. (2006) observed a negative correlation between total carotenoids and antioxidant activity in guava fruit extract. The difference between those results is likely due to the different raw materials used in their studies and the effects of encapsulation.

MANGO JUICE

Cano-Chauca et al. (2005) studied spray drying of mango juice. Spray drying of sugar-rich foods such as fruit juice has great economic potential. However, fruit juice powders obtained by spray drying have some drawbacks in their functional properties such as stickiness, solubility, and hygroscopy that make their packaging and utilization difficult. The possibility of creating a highly organized structure during spray drying could reduce the stickiness, considering that crystalline sugar has a lower water sorption potential. The author aimed to crystallize powdered mango juice during the process of spray drying.

Tropical and subtropical countries produce an enormous amount of commercially attractive fruit and vegetables; a typical example is the mango. However, in association with the seasonal problem, most of these products have a high water content in their mature state, which makes them more susceptible to decomposition by microorganisms and chemical and enzymatic reactions. Therefore, these products are extremely perishable and cannot be marketed or exported as fresh produce, resulting in post-harvest losses in excess of 20 and 30% (Agrianual, 2003).

According to Bhandari et al. (1997a), the sticky behavior of sugar- and acid-rich materials is attributed to low molecular weight sugars such as fructose, glucose, and sucrose, and organic acids such as citric, malic, and tartaric acid, which constitute more than 90% of the solids in fruit juices and purees. These materials have low glass transition temperatures (sucrose: 62°C; fructose: -5°C; glucose: 32°C). These compounds are very hygroscopic in their amorphous state and have a loose free-flowing nature at high moisture contents (Roos and Karel, 1991).

According to Sebhatu et al. (1994), fruit juice powder obtained by spray drying favors the yield of high sugar content solids, most of them present in the amorphous state. These sugars are very hygroscopic, which affects the functional characteristics of the dehydrated material, mainly its tendency to become sticky (stickiness) and form high agglomerates. This tendency to agglomerate may become accentuated as the amorphous state sugar transforms into crystalline sugar through the adsorption of small amounts of water.

Obtaining powders containing crystalline sugars is of fundamental importance for their stability. According to Bunn (1972), the crystalline state is the most stable state with the lowest free energy (available energy). This state is characterized by the fact that its component parts (atoms, molecules, or ions) are arranged into spatial nets, where the distances between the atoms of a crystal of any concrete material are constant and characteristic of that material.

The glassy or amorphous state is characterized by a disorderly molecule state with a metastable configuration because a small energy gain leads to a more stable state, that is, liquid or crystalline. However, on a kinetic level, the glassy state is considered to be more stable (Genin and Rene, 1995). According to Goff (1992), the glossy or amorphous state is characterized as a liquid with a high viscosity of 10^{12} – 10^{14} Pa·s that flows

at a very low molecular diffusion velocity. An amorphous solid substance does not have a natural geometric shape or a regular internal structure; its particles are distributed irregularly as in liquid substances. Thus, amorphous solid bodies are considered to be molten liquids that are mainly characterized by the lack of a clear fusion temperature.

The possibility of achieving a highly organized structure during spray drying could reduce the stickiness phenomenon considering that crystalline sugar has a lower water sorption potential. Crystalline and amorphous forms of the same powder show differences in particle size, particle shape, physicochemical properties, chemical stability, water solubility, hygroscopy, flow properties, and compatibility (Nakai et al., 1990).

Spray-Drying Conditions

Powder was obtained with a Mini Spray Dryer (Buchi, B_191, laboratory-Techniques LTD, Flawil-Switzerland). The spray dryer has a spray nozzle with an orifice of 1 mm in diameter. The inlet air temperature was 160°C for all of the solutions investigated, and the outlet air temperature was 70–75°C. The liquid feed to the dryer was about 10 ml/minute. The flow of the drying air was about 0.7 m³/minute. The experiments were performed under constant process conditions. The obtained material was placed into commercial bags (approximately 100 g) that were stored in a dessicator containing silica gel until use.

Electronic Microscopy

The three-dimensional characteristics of the surfaces of powder particles obtained from mango juice by spray drying were analyzed using electronic microscopy. When MD was used as a carrier with 0 and 3% of cellulose, the particles were larger, amorphous, aggregated, and strongly attracted to each other. The microstructure of the powders obtained using an AG carrier with added cellulose revealed a more uniform shape and a better distribution of particles with smooth and intact surfaces; varying sizes and disfigured particles were rarely observed. The microstructures obtained using waxy starch carriers with or without added cellulose contained some hexagonal particles among the spherical particles (Cano-Chauca et al., 2005).

X-Ray Powder Diffraction

The crystalline state is of great importance for the stability of powdered juice, and its presence can be determined through X-ray diffraction (XRD). Amorphous materials display diffuse and large XRD peaks because molecules in the amorphous state yield wide bands whereas crystalline materials yield sharp and defined peaks because they are presented in a highly ordered state. The XRD profiles of the XRD of standard sucrose, cellulose, glucose, and crystalline fructose were studied to compare the crystalline structures.

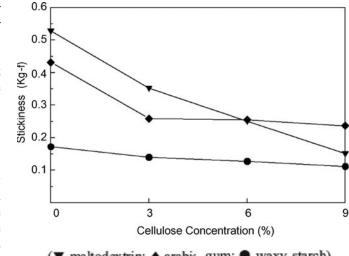
XRD profiles of the particles using MD as a treatment and cellulose at 0, 3, 6, and 9% verify that the system without added cellulose presents a totally amorphous surface, which is confirmed by the presence of large, nondefined peaks with abundant noise (Cano-Chauca et al., 2005). The XRD profile of powdered particles prepared using AG as a carrier and added cellulose was also examined. These particles were amorphous and partially crystalline materials, that is, peaks with considerable noise and semidefined peaks were observed, which means that the addition of cellulose influenced the formation of partially crystalline structures. Amorphous material may present because the material did not reach the conditions necessary for crystallization during drying. According to Reineccius and Risch (1995) the high molecular weight and high viscosity of AG increase the glass transition temperature, which favors conditions for the amorphous state. The change from the amorphous state to the crystalline state occurs above the glass transition temperature.

The crystalline material may occur because the waxy starch is formed by amylase and amylopectin, where the latter is considered a crystalline structure and represents about 90% of the XRD profile.

Stickiness

Spray drying is a dynamic process, and physical changes may occur during drying. Stickiness is considered the biggest problem in fruit juice spray drying, and this phenomenon is related to a low glass transition temperature (T_g).

The stickiness of MD was observed by Cano-Chauca et al. (2005) to decrease with cellulose concentration until reaching 0.15 kg-f at 9% cellulose (Fig. 3). Higher stickiness values may be related to the characteristics of the microstructure of the powder when compared with other additives. Using AG



(▼ maltodextrin; ◆ arabic gum; ● waxy starch).

Figure 3 Behavior of powdered mango juice stickiness as a function of cellulose concentration for the treatments used (adapted from Cano-Chauca et al., 2005).

as the carrier with 9% cellulose yielded a stickiness value of 0.22 kg-f. Similar behavior was observed in samples treated with MD, that is, decreasing stickiness as a function of the concentration of cellulose. However, these carriers presented the greatest values of cohesive force, higher than that obtained with MD. For the waxy starch treatment with 9% cellulose, the stickiness was 0.11 kg-f. The value of stickiness decreases slightly as a function of the concentration of cellulose. Thus, adding cellulose had little effect on stickiness. Stickiness occurs because the particles are better dispersed, which decreases the cohesive force among them.

Solubility

The solubility of powdered mango juice was studied as a function of cellulose concentration. In all treatments, the solubility of powdered mango decreases as a function of cellulose concentration. MD treatment yields a highly soluble powder with a solubility greater than 90% without added cellulose, whereas adding 9% cellulose decreases the solubility to around 72% (Fig. 4). MD is the most frequently used carrier in spray drying due to its physical properties such as its high solubility in water. For the AG treatment, the solubility greater than 90% without added cellulose and decreased to 71% when 9% cellulose was added (Cano-Chauca et al., 2005). Several researchers recommend the use of AG as the carrier in fruit juice spray drying because of its emulsification ability and its high water solubility. AG treatment with different levels of cellulose results in a highly soluble powder. The waxy starch treatment resulted in solubility values of around 31% for 9% added cellulose. The solubility of powdered mango juice decreases as a function of the concentration of cellulose, and this decrease is

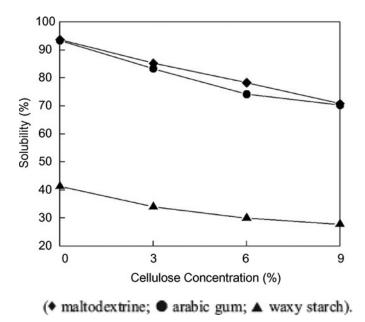


Figure 4 Solubility of powdered mango juice in function of cellulose concentration (adapted from Cano-Chauca et al., 2005).

more pronounced in the starch treatments for two reasons: first, the starches have low solubility in cold water (around 35–40%), and second, the presence of crystalline surfaces in the material may result in greater particle organization.

Cellulose was not suitable for inducing the crystallization of sugar, but it did affect the powder microstructure and therefore influenced the functional properties. Carriers used in mango juice dehydration also have an independent effect on the functional properties of the dehydrated material. Adding cellulose to the juice led to higher stickiness stability; however, the functional property of solubility was also affected.

ORANGE JUICE

Goula et al. (2009) studied spray drying of orange juice. Orange juice powder has many benefits over its liquid counterparts and thus a significant economic potential, and it represents a stable, natural, easily dosable ingredient for use in many foods and pharmaceutical products, including as a flavoring and coloring agent. However, dehydration of orange juice is a difficult task. The low transition temperature of the main juice components (low molecular weight sugars and organic acids) as well as their high hygroscopy, low melting point, and high water solubility result in a highly sticky product when spray dried.

Retention of product on the chamber wall for a long period of time is undesirable. This retention affects product quality because deposits can become scorched and then contaminate the entire product when they are dislodged. Furthermore, drying orange juice is not cost effective due to the frequent shutdowns of the dryer required for cleaning.

Various methods capable of producing a free-flowing fruit juice powder have been proposed, including the addition of drying aids such as MDs, glucose, soybean protein, sodium chloride, and skim milk powder (Lazar et al., 1956; Brennan et al., 1971; Tsourouflis et al., 1976; Bhandari et al., 1993; Bhandari et al., 1997a; Rao and Gupta, 2002; Adhikari et al., 2003, 2004; Jaya and Das, 2004; Chegini and Ghobadian, 2005; Papadakis et al., 2006; Roustapour et al., 2006; Quek et al., 2007; Shrestha et al., 2007; Chegini et al., 2008).

According to Goula et al. (2009), the major components of orange juice such as fructose, glucose, and citric acid have very low $T_{\rm g}$ values in the pure, dry state of 5, 31, and 16°C, respectively, and these values decrease drastically when moisture is absorbed. As a result, spray-drying orange juice is a complex process. Shrestha et al. (2007) produced orange juice powder by spray drying a mixture of juice and MD with a DE of 6 at 160°C. Tsourouflis et al. (1976) mentioned that using low-DE MDs as orange juice drying aids resulted in higher collapse temperatures than high-DE MDs at the same concentrations. Chegini and Ghobadian (2005) carried out spray drying of orange juice using additives (MD, glucose, and methyl cellulose) at high concentrations and a modified dryer with a jacketed wall for air cooling.

The major limitations of the use of drying aids are the changes that they induce in the product properties and the cost. To prevent sticking, a cool wall is favorable to minimize the number of thermoplastic particles that stick because a cold wall can cool and solidify the outer surface of the thermoplastic particles as they come into contact with the wall. This method, however, was found to improve the process but not to resolve the problem because the cold chamber wall also cools the surrounding environment and increases the RH of the air close to the wall surface.

Spray-Drying Operating Conditions

pilot-scale spray dryer (Buchi, B-191, Buchi Laboratoriums-Technik, Flawil, Switzerland) with a cocurrent regime and a two-fluid nozzle atomizer was used for the spray-drying process of orange juice. The atomizer had an inner diameter of 0.5 mm and used compressed air with a flow rate controlled by a variable area flow meter. The feed was metered into the dryer through a peristaltic pump. Inlet drying air, after passing through an electrical heater, flowed concurrently with the spray through the main chamber. The main chamber was made of thick transparent glass and had an inner diameter of 10.5 cm and a total height of 52.5 cm. The distance between the tip of the atomizer and the axis of the side exit tube was 34.9 cm. The bottom of the chamber was cone shaped and formed an angle of 60° with the walls. A cyclone air separator/powder recovery system was used. Dried powder samples were collected from the base of the cyclone. The dryer was designed so that while the inlet temperature could not be set with a temperature regulator, the outlet air temperature resulted from a combination of the inlet temperature, the aspirator setting, the pump setting, and the concentration of the feed. The modification to the original design consisted of connecting the spray dryer inlet air intake nipple to an air drying unit with a flexible plastic air duct. The compressed air was also dehumidified before being supplied to the two-fluid nozzle. An ultrapac 2000 adsorption dryer (Model 0005, ultrafilter International AG, Haan, Germany) with two desiccant cartridges was used to dry the air to 0.01 g of water per kg of dry air.

Goula et al. (2009) used 21 DE, 12 DE, and 6 DE MDs as drying agents. Orange juice concentrate was spray dried at inlet air temperatures of 110, 120, 130, and 140°C (\pm 1°C) and (concentrated orange juice solids)/(MD solids) ratios of 4, 2, 1, and 0.25. In all experiments, the atomizer pressure, the feed rate, the feed solids concentration, and the feed temperature were kept at 5 \pm 0.1 bar, 1.8 \pm 0.1 g/minute, 35 \pm 0.2%, and 32 \pm 0.5°C, respectively. In a previous work, the author found that the lowest wall deposition rate was achieved at a drying air flow rate of 22.8 \pm 0.2 m³/hour and a compressed air flow rate of 800 \pm 20 l/hour (Goula and Adamopoulos, 2005).

Glass Transition Temperature

According to Goula et al. (2009), $T_{\rm g}$ decreased by increasing moisture content due to the plasticizing effect of water (Fig. 5).

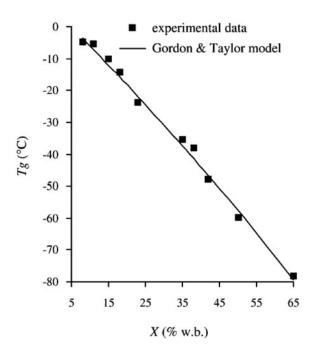


Figure 5 Relationship between glass transition temperature (T_g) and moisture content (X) of spray-dried orange juice concentrate (adapted from Goula et al., 2009).

Low-DE MDs were found to give higher glass transition temperatures than high-DE MDs with the same moisture content. Hence, the above researchers found the relationship between $T_{\rm g}$ and moisture content for MD 6 DE (Fig. 6). According to Werner et al. (2007), the glass transition occurs over a 10–20°C

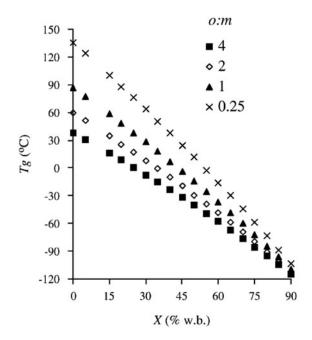


Figure 6 Relationship between glass transition temperature (T_g) and moisture content (X) of concentrated orange juice-maltodextrin 6 DE mixture for various (concentrated orange juice solids)/(maltodextrin solids) (o:m) ratios (adapted from Goula et al., 2009).

range, but usually only a single temperature is quoted as $T_{\rm g}$. In addition, sample preparation can lead to discrepancies in $T_{\rm g}$ data, and high molecular weight polymers also have wide thermal peaks that make it difficult to identify $T_{\rm g}$ exactly.

Moisture Content

The moisture content of the orange juice powders varied from 1.9 to 7.0%. According to the data described by Goula et al. (2009), an increase in air inlet temperature leads to a decrease in moisture content. The greater the temperature difference between the drying medium and the particles, the greater the rate of heat transfer into the particles, which provides the driving force for moisture removal.

The moisture content increases as the MD concentration increases because it is difficult for water molecules to diffuse past the larger MD molecules (Adhikari et al., 2004). In addition, the higher DE MD causes the powder moisture content to increase because high-DE MDs develop stickiness slower and reach a state of nonadhesion slower than low-DE MDs (Goula and Adamopoulos, 2008a). As the stickiness of a material increases, its drying rate decreases.

Bulk Density

Using the combination of added MD and dehumidified air as the drying medium proved to be a more effective way to minimize the number of particles that stick. The sticky nature of a powder is associated with a high bulk density (Shrestha et al., 2007; Goula and Adamopoulos, 2008b) because the particles that tend to stick together leave less space between them and consequently result in a smaller bulk volume.

The bulk density decreases as the MD concentration increases because adding MD minimizes the number of thermoplastic particles that stick. In addition, an increase in MD concentration may cause an increase in the volume of air trapped in the particles because MD is skin forming and spray-dried particles often contain air bubbles, which can occur as a result of the deposition of air that was initially present in the liquid feed or was absorbed during atomization. Generally, an increase in the volume of trapped air causes a decrease in the apparent density of the particles, and this apparent density primarily determines the powder bulk density.

An increase in MD DE leads to an increase in bulk density. This increase can be attributed to the fact that when the MD DE is high, its glass transition temperature is lower, lowering the elevation of the $T_{\rm g}$ of the orange juice concentrate—MD mixture and resulting in a stickier mixture (Adhikari et al., 2004; Goula and Adamopoulos, 2008a).

Rehydration

The effect of MD DE on powder rehydration depends on its effect on powder moisture content because a low moisture content seems to be associated with fast rehydration (Goula and Adamopoulos, 2008b) because lower moisture content implies less stickiness, thus increasing the surface area in contact with the rehydration water. An increase in MD DE leads to an increase in powder moisture content and a decrease in the ability of the powder to rehydrate. However, an increase in MD concentration does not reduce the rehydration ability of the powder, although it increases the moisture content. This dependence may be attributed to the fact that MD has superior water solubility, and it is mainly used in spray drying because of its physical properties such as its high solubility in water (Cano-Chauca et al., 2005; Grabowski et al., 2006).

Hygroscopy

Increases in inlet air temperature and MD concentration and decreases in MD DE led to a higher $T_{\rm g}$ and a lower hygroscopicity. This observation is in accordance with those of other researchers (Jaya and Das, 2004; Phanindrakumar et al., 2005).

Caking Degree

Increases in inlet air temperature and MD concentration and decreases in MD DE led to lower hygroscopy and a lower degree of caking. A similar trend was reported during spray drying of the tomato pulp–MD mixture (Goula and Adamopoulos, 2008b).

According to Goula et al. (2009), residue formation decreases as MD concentration decreases and the inlet air temperature and DE decrease. The moisture content decreases as the inlet air temperature increases and the MD concentration and DE decrease. The bulk density increases as the DE increases and the inlet air temperature and MD concentration decrease. The rehydration ability increases as the inlet air temperature and MD concentration increase and the DE decreases. The hygroscopy and degree of caking decrease as the inlet air temperature and MD concentration increase and the MD DE decreases.

CACTUS PEAR (Opuntia ficus-indica)

Opuntia spp. is an interesting crop from semiarid regions, and its fruits, known as prickly pear or cactus pear, are an excellent source of betalain natural colorants and functional compounds. The use of Opuntia spp. fruits as a betalain source is preferred to red beets. Opuntia spp. fruits are at lower risk of microbial contamination, have no nitrate content, are highly flavored, show adequate nutritional properties (e.g., high levels of calcium, magnesium, and vitamin C), and contain interesting functional compounds such as quercetin (Butera et al., 2002; Piga, 2004). In addition, Opuntia spp. extracts have shown analgesic, antiinflammatory, hypoglycaemic, physiological antioxidant, cancer chemoprevention, and neuroprotective effects (Zou et al., 2005; Kim et al., 2006). Thus, there has been increasing interest in the large-scale processing of cactus pear fruit for the production of

coloring foodstuffs, with the potential of opening up new markets for functional foods to the dairy and beverage industries (Mo β hammer et al., 2005, 2006).

Saenz et al. (2009) studied spray drying of cactus pear. Cactus pear (*Opuntia spp.*) is a tropical fruit tree native to America that grows in arid and semiarid regions (Pimienta-Barrios and del Castillo, 2002). It has green and collared fruits (red, yellow, or purple) due to the presence of various pigments such as betalains and carotenes (Tesoriere et al., 2005; Castellar et al., 2006; Diaz et al., 2006).

Cactus pear is one of the few sources of betalains in nature and therefore is an attractive alternative to replace synthetic additives, and it is also produced for direct consumption. Cactus pear could be used as a natural coloring agent and to provide health benefits by its antioxidants (Stintzing and Carle, 2004; Tesoriere et al., 2005).

Bioactive compounds of pulp (CP) and ethanolic (CE) extracts of the cactus pear (*Opuntia ficus-indica*) were encapsulated with MD or inulin (I) (Saenz et al., 2009). Different types of encapsulating agents have been used for spray drying and include polysaccharides (starches, MDs, corn syrups, and AG), lipids (stearic acid, mono and diglycerides), and proteins (gelatin, casein, milk serum, soy, and wheat) (Gibbs et al., 1999). The most commonly used materials for microencapsulation are MDs of different DEs. MDs are obtained by acid hydrolysis of several starches (e.g., corn or potato). In general, MDs have high solubility in water, low viscosity, bland flavor, and no color (Gibbs et al., 1999), so they are extensively used in the food industry.

Inulin may be an interesting possible encapsulation agent due to its technical and nutritive properties (Stevens et al., 2001). Inulin is a fructooligosaccharide obtained commercially from chicory (*Cichorium intybus*) root, Dahlia (*Dahlia pinuata* Cav.), and Jerusalem artichoke (*Helianthus tuberosus*); the inulin obtained from those three sources has an average degree of polymerization (DP) of 10–14, 20, and 6, respectively. Inulin is composed of fructose units with β (2-1) links with glucose at the end of the chain.

Spray-Drying Operating Conditions

Saenz et al. (2009) used a B-191 spray dryer (Buchi, Switzerland) for the processing of cactus pear. The spray dryer was operated at an inlet temperature ranging from $140-160 \pm 5$ to $120-160 \pm 5^{\circ}$ C for MD and I, respectively. The air flow, rate of feeding, and atomization pressure were 600 l/hour, 10 ml/minute, and 20 psi, respectively, for both encapsulating agents. The powders obtained were stored to exclude light and were kept at -20° C until they were analyzed.

Antioxidant Activity

According to Saenz et al. (2009), the antioxidant activities were $3.30 \,\mu$ mol TEAC/g and $2.87 \,\mu$ mol TEAC/g for CP and CE,

respectively. Butera et al. (2002) found 4.2 μ mol TEAC/g in red cactus pear pulp, and Stintzing et al. (2005) reported 3.64 μ mol TEAC/g with another method. This antioxidant activity is similar to those reported for other fruits such as pineapple, passion fruit, and blackberry (Kukoski et al., 2005). The lower antioxidant activity in CE compared with CP corresponds to the lower content of bioactive components found in the extract.

Saenz et al. (2009) reported the concentration values of bioactive compounds of pulp and ethanol extract before and after encapsulation. The recovery of betacyanins and indicaxanthins in the pulp encapsulated with MD and I were both 100%, which indicates that the drying temperature and type of encapsulating agent did not affect the recovery of the bioactive compound. In the ethanol extract, the recoveries of betacyanins and indicaxanthins were lower in MD at 62 and 67%, respectively, and in I at 81 and 86%, respectively. The higher degradation of bioactive compounds in MD could be attributed to the high inlet air temperature (140°C).

The recoveries of polyphenols were over 100%, which could be a consequence of the hydrolysis of the cactus pear polyphenol conjugates during the preparation of the samples or during the drying process (Turkmen et al., 2005). Saenz et al. (2009) also reported the characterization of pulp and ethanolic extract of cactus pear microcapsules obtained under optimal conditions with MD and I.

Scanning Electron Microscopy (SEM)

CP-MD, CP-I, CE-MD, and CE-I microcapsules were imaged using SEM (Fig. 7). The morphology of microcapsules with both encapsulating agents was irregularly spherical in shape with an extensively dented surface. The dented surfaces of the spray-dried particles were attributed to the shrinkage of the particles during the drying process. A similar morphology was

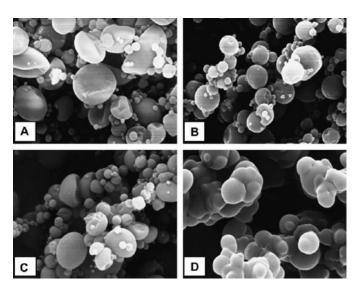


Figure 7 Scanning electron microscopic photographs of microcapsules for the CP-MD (A), CP-I (B), CE-MD (C), and CE-I (D) designs, respectively (adapted from Saenz et al., 2009).

observed in microcapsules of other cactus pear cultivar (*Opuntia lasiachanta*) pigments with MD (10 DE) (Diaz et al., 2006), *Amaranthus* (Cai and Corke, 2000) using MD of different DEs (10 DE; 20–23 DE; and 28–31 DE), and β -carotene using modified tapioca starch and MD (24 DE) as encapsulating agents (Loksuwan, 2007). Nevertheless, smooth spheres have primarily been observed in microcapsules of black carrot pigments (*Daucuscarota* L.) with MD (10 DE and 20–23 DE) (Ersus and Yurdagel, 2007).

Saenz et al. (2009) demonstrated that CP microcapsules obtained under the optimal conditions showed a higher recovery of bioactive compounds in the drying process. The cactus pear microcapsules represent a promising food additive for incorporation into functional foods because of their antioxidant content and potential use as a red colorant.

OPUNTIA STRICTA FRUITS

Obon et al. (2009) studied the spray drying of Opuntia stricta fruits. The fruits are pear-shaped, with an average size of 2.5×6 cm, and they are a red-purple color. *Opuntia stricta* fruits had the highest level of betalains (80 mg/100 g fresh fruit) compared with different Opuntia spp., with betanin and isobetanin as the main colorant components, while no betaxanthins were detected. The levels of betanin and isobetanin are about five times those of the red-purple fruits of O. ficus-indica (14–19 mg/100 g fresh fruit), O. undulate (20 mg/100 g fresh fruit), or O. lasiacantha (19-28 mg/100 g fresh fruit), and they are even higher than those found in some commercial red beets (40-60 mg/100 g fresh fruit) (Fernandez-Lopez et al., 2002; Castellar et al., 2003; Diaz et al., 2006). A concentrated liquid extract from O. stricta can be used as a commercial food colorant because of its high colorant capacity (color strength: 3.9), low viscosity (59 cP), and high storage stability ($t_{1/2} = 236$ days, 4°C). Opuntia stricta colorant presents a vivid red-purple color that is clearly distinguishable from the colors shown by other commercialized natural red food colorants (Castellar et al., 2006).

Spray drying has paved the way for the production of powder colorants with high storage stability, ease of handling, and minimal weight compared with liquid concentrates. Advantages of spray drying include the ability to quickly produce a dry powder (e.g., as compared with lyophilization) and the ability to control the particle size distribution. Even though many studies of the spray-drying process have been undertaken, it still remains a step with uncertainties and difficulties. Reasons for these difficulties include the substantial effect of material properties on the drying behavior and the complex fluid dynamics in the spray dryer.

The most widely used additives in fruit juice powders are partially hydrolyzed starch products. These saccharide polymers of D-glucose have a neutral taste, white color, and an absence of odor, and they are easily digested and well tolerated. They are usually classified according to their degree of hydrolysis, expressed as DE. MD has a DE of <20, according to the United

States Food and Drug Administration (direct food substances affirmed as GRAS; 21 CFR paragraph 184.14444), while saccharide polymers with DE higher than 20 are considered to be dried glucose syrup (DGS; sweeteners and table syrups; 21 CFR paragraph 168.121) (Marchal et al., 1999).

Spray-Drying Operating Conditions

Opuntia stricta fruit juice was spray dried using a Buchi Mini Spray Dryer (B-290, Flawil, Switzerland). The spray dryer worked in an open cycle, with co-current flow of hot air and sprayed material. Atomization was done with a two-fluid nozzle, using compressed air. The spray nozzle had a nozzle tip of 0.7 mm in diameter and a nozzle cap of 1.4 mm diameter. A glass cylinder (50×15 cm) was used as the drying chamber. The feed temperature was thermostatized at 20°C, and the inlet air temperature varied within the range of 80–160°C. The liquid feed rate to the dryer was in the range of 0.36-0.72 l/hour. The spray air flow rate was fixed at 0.47 m³/hour, which allowed for good atomization at the liquid feed rate studied, and the aspirator flow rate of drying air was fixed at 36 m³/hour (90%). The design of the dryer chamber and the airflow rate provide a droplet residence time of about 1 second. The maximum evaporative capacity was 1 l/hour at an inlet air temperature of 220°C.

The physicochemical properties of *O. stricta* fruit juice are shown in Table 4. The high color strength of the juice was obtained from its high levels of betacyanins (Castellar et al., 2006). The color parameters show the dark red-purple color of the juice, which turns into a vivid pink-purple color after dilution. The juice of *O. stricta* has a lower pH than other *Opuntia species*, and foods with acidic substances are expected to be more difficult to dry (Bhandari et al., 1993). Furthermore, *O. stricta* juice is viscous due to its high mucilage content, which makes spray-drying process more difficult.

Initial experiments performed by Obon et al. (2009) with O. stricta juice using an inlet air temperature of 120°C, a feed rate of 0.72 l/hour (40% maximum pump speed), and with no

Table 4 Analysis of *Opuntia stricta* juice (adapted from Obon et al., 2009)

Parameter	Value
Color strength (OD 535 nm, 1% v/v sol)	0.85 ± 0.05
pН	3.8 ± 0.1
Total soluble solids (°Brix)	11.5 ± 0.5
Glucose (g/l)	55 ± 1
Fructose (g/l)	38 ± 1
Water content (%)	88 ± 0.5
Viscosity at 25°C (cP)	107 ± 1
Density at 25°C (g/ml)	1.040 ± 0.002
Color parameters	
L^* (darkness–lightness) (0, 100)	13.99 ± 0.02
a* (redness–greenness)	7.98 ± 0.04
<i>b</i> * (yellowness–blueness)	0.57 ± 0.03
C* (chroma)	8.00 ± 0.06
h° (hue, 0° red, 90° yellow, 180° green, 270° blue	0.07 ± 0.05

Mean \pm standard deviation, n = 3.

Table 5 Effect of juice concentration and glucose syrup to juice concentration ratio on bulk density and moisture of the powders, and outlet temperature of the drying process (adapted from Obon et al., 2009)

Juice (°Brix)	Dried glucose syrup (°Brix)/ juice (°Brix)	Bulk density (g/ml)	Moisture (%)	Outlet temperature (°C)
1.2	1.66	0.53 ± 0.02^{a}	3.8 ± 0.2^{b}	50 ± 1^{a}
1.2	2.50	0.55 ± 0.01^{a}	3.9 ± 0.1^{b}	50 ± 1^{a}
1.2	3.33	0.59 ± 0.02^{b}	3.1 ± 0.2^{a}	51 ± 1^{a}
2.4	1.66	0.55 ± 0.01^{a}	3.4 ± 0.2^{b}	59 ± 1^{b}
2.4	2.50	0.53 ± 0.01^{a}	3.5 ± 0.3^{b}	60 ± 2^{b}
2.4	3.33	0.57 ± 0.01^{b}	3.2 ± 0.2^{a}	61 ± 1^{b}
9.6	1.66	0.54 ± 0.01^{a}	3.9 ± 0.2^{b}	63 ± 2^{c}
9.6	2.50	0.55 ± 0.02^{a}	3.7 ± 0.3^{b}	67 ± 1^{c}
9.6	3.33	0.57 ± 0.01^{b}	3.1 ± 0.2^{a}	68 ± 2^{c}

Note: Means having same letter within the column did not differ significantly from each other according to Tukey's test at p < 0.05.

addition of drying aid, showed that although a powder was obtained, it rapidly becomes sticky when collected. DGS of 29 DE was selected as a drying aid by the above researchers because although MDs have the advantages of higher transition temperature ($T_{\rm g}$) and lower hygroscopicity, DGSs have lower viscosity and higher solubility, are much cheaper, and their labeling as glucose syrup is preferred by customers over MDs. In addition, it could form a dense and highly oxygen-impermeable wall system, providing better storage stability for pigments (Delgado-Vargas and Paredes-Lopez, 2003). Initial assays, changing the ratio of DGS to juice, were run at three different final juice concentrations (expressed as $^{\circ}$ Brix), as shown in Table 5.

Color Strength and Bulk Density

The color strength of the powders, along with the drying yield and color yield of the drying process, was calculated by Obon et al. (2009). Sticky powders were obtained when the ratio (DGS/J) was less than or equal to one; thus, color strength, drying yield, and color yield could not be measured. Nonsticky powders were only obtained when the ratio (DGS/J) was greater than one. This was observed at the three different juice concentration assayed by Obon et al. (2009), suggesting that a minimum amount of drying aid is required to avoid sticky powders. Increasing the ratio to 3.33 enhanced neither the color strength of the obtained powders nor the drying yield or color yield of the drying process. The values for color yield were always close to those of the drying yield, which means that betacyanin levels were retained during the drying process. Juice concentration is an important parameter to take into account because high concentration values lead to lower values of color strength, drying yield, and color yield when using the optimum spray-drying conditions. Juice concentrations lower than 1.2 °Brix were assayed, with no better results. High juice concentrations led to the formation of sticky particles on the drier cylindrical chamber walls. Drying yields fell short of 100%, also due to the difficulties in smallest particle collection, which cannot be recovered in the lab-scale apparatus because they do not effectively deposit on the cyclone and because their low masses cause them to be drawn up. The bulk density of the obtained powders ranged from 0.53 to 0.59 g/ml, and the moisture of the powders was between 3.1 and 3.9%. The values did not differ significantly when using a drying aid/juice ratio of 1.66 or 2.55, while a small increase in bulk density and a small decrease in moisture were observed for a ratio of 3.33. These parameters were not affected by the juice concentration used. The values for outlet temperature were higher when the solid content of the feed was increased. Similarly, Abadio et al. (2004) obtained particles with 0.59 g/ml bulk density and 1.18% moisture content using 10 DE MD and pineapple juice at a 1.2 ratio.

Scanning Electron Microscopy

According to Obon et al. (2009), the scanning electron micrograph of the powders revealed that the nonsticky powders obtained in all cases showed a spherical shape. The diameters of the particles varied between 2 and 10 μ m, with similar crust shrinkage. Using the same juice concentration (1.2 °Brix), an increase in the (DGS/J) ratio led to a slight decrease in average diameter of the particles (images), as per Obon et al. (2009). In contrast, when the juice concentration was increased at a constant (DGS/J) ratio, an increase in average particle diameter was observed. The larger particle size was found in samples with higher concentrations of solids. These results are logical because the high concentration increased the amount of solid in each droplet exiting the nozzle. Therefore, when the water in the droplet evaporates, more of the particle remains. These trends were expected on the basis of the established spray-drying theory. Holes were observed in the crust of a few individual particles, suggesting that hollow spheres were formed in the spray-drying process.

Obon et al. (2009) also concluded that low juice concentrations optimize color strength, drying yield, and color yield, and that under the conditions tested, a (DGS/J) value of 1.66 was enough to obtain nonsticky powders. Hence, spray drying is a good technique for producing a red-purple powder food colorant from O. stricta fruit juice. Using dry glucose syrup (Glucidex 29) as a drying aid and fixing the spray-drying process variables, a high color strength (4.0) nonsticky powder was produced with a drying yield of 58%, as per Obon et al. (2009). This powder colorant, stored at room temperature, maintained 98% of its color after one month. A yogurt and a soft drink were colored with this powder colorant to produce foods of vivid red-purple tonalities. These foods were stored under refrigeration for one month and did not show any evident change in their color ($\Delta E > 5$).

WATERMELON

Quek et al. (2007) studied the spray drying of watermelon. Watermelon (*Citrulus lanatus*) is native to tropical Africa and it is a popular thirst quencher during hot summer weather.

Watermelon juice has proven to be a very concentrated source of carotenoid, namely, lycopene. The lycopene content of watermelon was found to be higher than that of many other fruits and vegetables (Edwards et al., 2003; Perkins-Veazie and Collins, 2004). Tomato is traditionally thought to be the richest source of lycopene. However, the study of Edwards et al. (2003) showed that the lycopene concentration of watermelon is 4868 μ g/100 g fresh watermelon, which is 40% higher than the lycopene concentration of raw tomato (3025 μ g/100 g tomato). In addition, watermelon is also an excellent source of vitamin C and a good source of vitamin A, notably through its β -carotene content. Both of the above vitamins are important antioxidants needed by the human body to neutralize free radicals. 100 g of watermelon provides 8.1 mg of vitamin C and 569 IU of vitamin A. corresponding to 13.5% of the daily value (DV) for vitamin C and 11.38% of the DV for vitamin A. In addition, it is also a good source of B vitamins, especially B1 and B6, as well as minerals such as potassium and magnesium.

Carotenoids are widely believed to protect human health due to their antioxidant properties. Lycopene is a vibrant red tetraterpenic carotenoid with a molecular formula of $C_{40}H_{56}$. It is the open-chain analogue of β -carotene and contains 13 conjugated double bonds (Fuhrman et al., 2000; Lin and Chen, 2003). Lycopene has proven to be the most effective oxygen radical scavenger among the carotenoids. Research shows that the oxygen free-radical-quenching ability of lycopene is more than twice that of β -carotene and 10 times that of α -tocopherol. Lycopene is relatively stable during cooking and processing, and the bioavailability of lycopene is influenced by heat.

Watermelon is a seasonal fruit and is thus not available year-round. Because of its high nutritional value, it would be beneficial if a watermelon product could be produced and made available throughout the year. Spray drying can be used to turn the watermelon juice into a powder that has longer shelf life and is readily available. Ideally, the spray-dried watermelon powder should have instant properties or serve as a lycopene-rich functional food ingredient for incorporation into food products. Since the main dietary lycopene source is currently tomato, the development of lycopene-rich watermelon powder will provide consumers with an alternative choice, according to Quek et al. (2007).

Spray-Drying Operating Conditions

A Buchi mini spray dryer (Model B-191, Buchi Laboratoriums-Technik, Flawil, Switzerland) was used for the spray-drying process. The spray dryer was equipped with a water bath (Model W22, Grant Instrument, Cambridge). Spray drying was carried out at an aspirator rate of 60%, flow rate of 600 L/hour, pressure of 4.5 bar, and feed temperature of 20°C. These conditions were chosen by Quek et al. (2007) after conducting initial trial runs. Four inlet air temperatures were investigated: 145, 155, 165, and 175°C. MD (3 and 5%) was added according to the weight of the watermelon juice. The dryer was washed with water at the desired parameter setting

for 10 minutes before and after the spray-drying process. All the spray-dried powders were collected in clean water activity container with known weight. The powders produced were then weighed, sealed in the bottle, and stored at 4°C in the dark.

Effect of Additive (Maltodextrin)

From the observations of Quek et al. (2007), very little powder accumulated in the collector if MD was not added to the feed. The particles produced were very sticky, were mainly deposited onto the wall of the drying chamber and cyclone, and could not be recovered. Therefore, MD of 3 and 5% (of the total feed solution) was added to the juice prior to spray drying to investigate its effects on the resulting product. The MD (Dridex 9) used was a low-DE MD with DE of 8-12. Other researchers have reported that low-DE MDs have better nutrient binding properties (Desobry et al., 1997; Cai and Corke, 2000; Rodriguez et al., 2005). MD has also proven to be a very good encapsulant for low molecular weight sugars such as fructose and organic acids (Adhikari et al., 2003, 2004). It was observed that the condition improved by results and powder quantity with the addition of MD. The addition of 5% MD to the feed appeared to give better results than the addition of 3% MD. These results showed that MD was a useful drying aid in the spray drying of watermelon juice, as it improved the yield of product.

It was suggested by Quek et al. (2007) that MD could alter the surface stickiness of low molecular weight sugars such as glucose, sucrose, and fructose, and organic acids, and therefore facilitate drying and reduce the stickiness of the spray-dried product (Adhikari et al., 2003, 2004). However, if more than 10% MD was added, the resulting powders lost their attractive red-orange color.

Moisture Content

The results showed that at constant feed flow rate, the moisture content of the spray-dried powders decreased with the increased inlet and outlet air temperature (Table 6). This is because at higher inlet temperatures, the rate of heat transfer to the particle is greater, providing greater driving force for moisture evaporation. Consequently, powders with reduced moisture

Table 6 Physical properties of the spray-dried powder (adapted from Quek et al., 2007)

Inlet temperature ($^{\circ}$ C)	Outlet temperature ($^{\circ}$ C)	Maltodextrin (%)	Moisture content (%)
145	94.7	3	2.78 ± 0.21
155	101.0	3	2.29 ± 0.18
165	105.0	3	1.62 ± 0.20
175	108.6	3	1.49 ± 0.32
145	95.4	5	1.62 ± 0.11
155	101.5	5	1.55 ± 0.10
165	107.6	5	1.57 ± 0.08
175	112.7	5	1.49 ± 0.21

content are formed. The results were consistent with other findings (Goula et al., 2004).

The results of Quek et al. (2007) also showed that the moisture content of the spray-dried powder decreased when more MD was added. In a spray-drying system, the water content of the feed has an effect on the final moisture content of the powder produced (Abadio et al., 2004). Addition of MD to the feed prior to spray drying increased the total solid content and reduced the amount of water for evaporation, resulting in decreased moisture content of the powder. This meant that powders with lower moisture content could be obtained by increasing the percentage of added MD. However, if the proportion of MD was very high, the powder produced would be of lower quality because the nutrients from the watermelon juice would be diluted.

Color Measurement

Quek et al.'s (2007) measurements for watermelon powders with 5% MD are shown in Table 7. *L*-value measures the lightness of the sample, $+a^*$ measures the red color, and $+b^*$ measures the yellow color. Hue angle measures the property of the color and it is the ratio of a^* and b^* (hue = $\tan^{-1} (b^*/a^*)$). Chroma indicates the color intensity or saturation (chroma = $(a^{*2} + b^{*2})^{1/2}$).

According to Quek et al. (2007), when inlet temperature increased, the $+b^*$ values increased, while the $+a^*$ values first increased then decreased at 175°C. This contributed to the changes in hue angle and chroma (Table 7). Overall, the lightness of the powders decreased and the chroma of the powders increased. This implied that the color of the powders became darker at higher inlet temperatures. One of the explanations for this phenomenon is that watermelon contains sugars that could contribute to browning of the powders at higher inlet temperatures. As the inlet temperature increased, the hue angles also increased from 47.39 to 53.49°. These figures correspond to the regions of red to yellow color where 0° is pure red and 90° is yellow. This meant that there was a decrease in the red color when the inlet temperature was increased. The changes in hue angle might be caused by the destruction of lycopene and β -carotene at higher temperature.

Lycopene and β-Carotene Content

The results (Table 8) of Quek et al. (2007) showed that the watermelon studied had an average lycopene content of

Table 7 Colorimetric results of the spray-dried powders (adapted from Quek et al., 2007)

Inlet temperature (°C)	L	a*	b^*	Hue angle (°)	Chroma
145	74.63	+14.91	+16.21	47.39	22.02
155	70.68	+18.60	+20.60	47.92	27.79
165	67.76	+20.00	+23.66	49.79	30.98
175	66.91	+18.94	+24.66	53.96	31.09

Table 8 Lycopene and β -carotene contents of the raw fruits, juices, and spray-dried powders (adapted from Quek et al., 2007)

Inlet temperature (°C)	Lycopene content	β -carotene content
Fruit $(\mu g/g)$	36.45 ± 2.05	2.80 ± 0.22
Spray-dried powders (μg/g) 145	954.02 ± 3.11	31.46 ± 0.34
155	907.66 ± 2.15	29.47 ± 0.61
165	820.35 ± 1.82	26.71 ± 0.42
175	724.48 ± 1.15	23.05 ± 0.32

 $36.45 \pm 2.05 \,\mu\text{g/g}$ of fresh fruit, which was consistent with the Perkins-Veazie et al. (2001) study. The lycopene content of the watermelon varies across cultivars and is also affected by factors such as seasons and growing environment (Perkins et al., 2001; Bang et al., 2004). The β -carotene content of watermelon was found to be $2.80 \pm 0.22 \,\mu\text{g/g}$ of fresh fruit, which was much lower than the lycopene content.

The lycopene and β -carotene contents of spray-dried powders with 5% MD are as shown in Table 8. It can be seen that the lycopene content decreased with increased inlet temperature. A similar observation was made for the spray drying of tomato pulp (Goula et al., 2004). The reduction in lycopene content was likely due to thermal degradation and oxidation. Goula et al. (2004) reported that the spray-dried powders produced at lower inlet temperature had a tendency to undergo agglomeration because of their higher moisture content. This is especially true for powders with a sticky nature, which contain a high concentration of sugars. Agglomeration would lower the exposure of powders to oxygen and therefore protect the lycopene from destruction. For β -carotene, a similar trend was observed. Carotenoids are reported to be very susceptible to heat destruction and oxidation because of their highly unsaturated chemical structure (Stefanovich and Karel, 1982). However, β -carotene could be more heat sensitive than lycopene (Regier et al., 2005). Quek et al. (2007) found that the percentage loss of β -carotene was slightly higher than lycopene when the inlet temperature increased. For example, the loss of lycopene was 24.06% compared with 27.08% for β -carotene when the inlet temperature increased from 145 to 175°C.

The lycopene and β -carotene content could both affect the color of the spray-dried powders produced. However, the effect from lycopene would predominate, as its content was much higher in watermelon. The lycopene and β -carotene contents (Table 8) could correlate with the L^* a^* b^* color analysis (Table 7). Both the lycopene and β -carotene content were found to be inversely proportional to the hue angle (Fig. 8). Thus, when the inlet temperature was increased, the lycopene and β -carotene content were reduced, reflecting in the reduction in the red-orange color of the powder.

Another observation of Quek et al. (2007) was that the spraydrying process successfully concentrated the lycopene and β carotene in solid form. Depending on the inlet temperature, lycopene and β -carotene were concentrated by approximately 20–30-fold and 2–10-fold by weight, respectively, compared with the raw fruit. Hence, MD (Dridex 9) was an effective

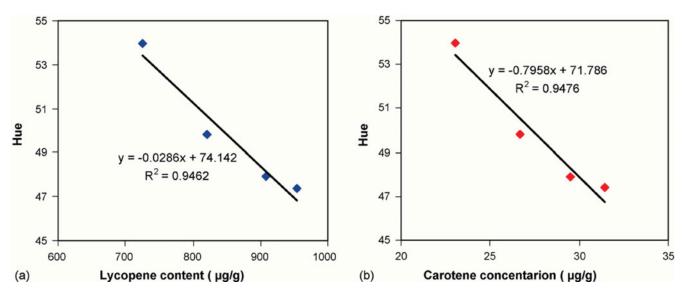


Figure 8 The correlations between lycopene content and hue (a) and carotene content and hue (b) (adapted from Quek et al., 2007). (Color figure available online.)

drying aid for spray drying of watermelon juice. The addition of MD reduced the stickiness of the products and altered the physicochemical properties of the spray-dried powders. The results of Quek et al. (2007) showed that inlet temperature has a great effect on the physicochemical properties of the spray-dried powders. As inlet temperature increased, the moisture content of the powder decreased, while the time required for reconstitution increased. The lightness and hue of the spray-dried powders decreased, which could be correlated with the loss of lycopene and β -carotene at higher inlet temperatures. The sugar content also decreased as inlet temperature increased. Overall, at the inlet temperature of 155°C, the spray-dried powders have the best colorimetric results, reasonably low moisture content, as well as good lycopene and β -carotene content. Drying the watermelon juice at a temperature above 165°C led to inferior products, overall, due to nutrient loss and changes in color. Therefore, physicochemical properties of the powders are very important for ensuring production of high-quality watermelon powders.

DURIAN

Chin et al. (2010) studied the spray drying of Durian. Durian (*Durio zibethinus*), from the Bombacacea family, possesses a distinct flavor that is highly appreciated by people in Southeastern Asia (Sapii and Nanthachai, 1994). Over 170 constituents have been reported in the volatiles fraction of durian, with thiols, sulphides, and ethyl esters indicated as the key contributors to durian aroma (Baldry et al., 1972; Weenen et al., 1996). The production of spray-dried durian powder is advantageous, as the fresh fruit has a limited storage life and is difficult to transport.

Spray-Drying Operating Conditions

The fruit was spray dried using a co-current-type spray dryer (Niro 2000 model type A; GEA Niro A/S, Soeborg Denmark)

operated with a 24-vane centrifugal atomizer at 2 bar pressure, inlet temperature ($T_{\rm i}$) of 130°C, and outlet temperature ($T_{\rm o}$) of 88 \pm 3°C. Four replicates of the drying experiment were conducted, and the average moisture content of the spray-dried product ranged between 1.49 and 1.91 g/100 g.

Retention of Volatiles in Spray-Dried Powder

Two esters (ethyl propanoate and ethyl 2-methylbutanoate (E2 MB)) and two sulphides (propanethiol and diethyl disulphide) that have been reported to be responsible for the key durian odor (Baldry et al., 1972; Weenen et al., 1996) were monitored by Chin et al. (2010). Volatile retention of spray-dried durian flavor, as shown in Table 9, was quantified by comparing the initial amount of specific volatile in the final dried products to the residual amount of the volatile after storage. Overall, the retention of target volatiles was observed within the range of 22–77% (Table 9). The retention of target durian volatiles except for E2 MB was significantly affected by the type of incorporated additives and inlet drying temperature, T_i. Also, as reported by Hassan and Mumford (1996), high-temperature exposure of fructose droplets over the boiling point (i.e., 150°C) resulted in the build-up of a more porous crust that offers less resistance to vapor diffusion during drying. A considerable amount of fructose and other minor substances, including organic acids, contained in the spray-dried durian droplet may have induced the onset of puffing or ballooning phenomena at $T_i = 170^{\circ}$ C. Although the increase in T_i improved the retention of volatiles by increasing the rate of droplet film formation (Reineccius et al., 1982), at the higher drying temperature (170°C), the volatiles retention of spray-dried durian was not improved due to the nature of the sample used by Chin et al. (2010).

A blend of MD of DE 15 (MD) with AG at a ratio of 3:1 (MG) resulted in a powder with the highest retention for propanethiol, ethyl propanoate, and diethyl disulphide volatiles, of 62, 52, and

Table 9 Retention of volatiles in spray-dried durian powder that produced under different thermal conditions (adapted from Chin et al., 2010)

		Volatile retention (%); $n = 4$				
			ID ^a			
Volatile compounds/	Volatile amount of			MG^a	NL^a	
physical properties	feedstock (μ g/g)	170°C ^b	130°C	130°C	130°C	
Propanethiol/MW 76, BP 67°C	6.54 ± 5.06	$52 \pm 30\%$ ab	$28 \pm 9\%$ ab	$62 \pm 10\%$ b	22 ± 8%a	
Ethyl propanoate/MW 102, BP 99°C	31.15 ± 36.83	$28 \pm 4\%a$	$29 \pm 5\%$ a	$52 \pm 15\%$ b	$30 \pm 6\%$ a	
E2 MB/MW 130, BP 133°C	38.75 ± 26.93	$67 \pm 12\%$ a	$46 \pm 7\%a$	$76 \pm 10\%$ b	$63 \pm 22\%a$	
Diethyl disulfide/MW 122, BP 153°C	52.25 ± 45.57	$76 \pm 7\%$ b	$41\pm20\%a$	$77 \pm 1\%$ b	$50 \pm 8\%$ a	

MW means molecular weight; BP means boiling point.

77%, respectively (Table 9), in comparison to MD and modified starch N-Lok (NL) powder matrices produced at $T_i = 130^{\circ}\text{C}$. Partial replacement of MD with AG produced better EE, which was likely due to the rapid formation of surface film by AG (Bangs and Reineccius, 1990). Since volatiles evaporated from the droplet surface at a faster rate than water molecules during the constant stage in drying, the loss of a certain amount of volatiles was inevitable. However, the residual amount of key flavor volatiles in MG-prepared powder, which ranged from 46 to 78%, was similar to the level of volatiles retained in freezedried fruit products such as pear (Komes et al., 2007), apricot (Komes et al., 2005), and banana (Mui et al., 2002).

Chin et al. (2010) demonstrated that volatiles with larger molecular weight tended to retain better during spray drying, confirming previous findings (Rulkens and Thijssen, 1972; Clarke, 1987; Rosenberg and Sheu, 1996). According to the selective diffusion concept, volatiles retention is generally dependent on the diffusivity of the volatile compounds after the formation of the semipermeable surface. Due to the decrease in diffusivity coefficient of volatile compounds and increasing molecular size, volatiles with high molecular weight were retained better than volatiles with smaller molecular weight.

Scanning Electron Microscopy

The surface morphology of the spray-dried powder was viewed with SEM at magnifications ranging from 200 to $1000 \times$. Spherical particles were formed in the spray-dried microcapsules prepared with MD, MG, and NL particles. The MD-prepared powder exhibited a smooth particle surface, but strong adherence took place among the particles to form the bulk of agglomerates when dried at $T_i = 170^{\circ}$ C. Results of Chin et al. (2010) may imply that further depletion of volatile compounds could occur during the adhesion/agglomeration stage prior to the powder being collected from the dryer's cyclone, since stresses appeared on the surfaces of the powder and caused the particle structure to collapse (Whorton and Reineccius, 1995). Meanwhile, MD microcapsules processed at $T_i = 130^{\circ}$ C showed a higher degree of surface undulation. This effect could be expected since the slow drying rate at low T_i may result in the

formation of an outer crust or the particle shape becoming sluggish, and subsequently result in shrinkage of the particle, as reported previously by Finney et al. (2002).

According to Chin et al. (2010), broken capsules and cracks were observed on the surface of NL-containing microcapsules. The shell of NL-containing microcapsules was comprised primarily of ocetenyl succinate starch and fructose. According to Hassan and Mumford (1996), such matrices were comparatively rigid, forming a porous crust that ruptured easily under high-temperature treatment. As a consequence, relatively low volatiles retention in NL-containing microcapsules was probably attributed to cracks, which acted as channels that allowed volatiles to escape from the interior of the microcapsule during and after drying. Relatively higher feedstock viscosity and rapid film-forming behaviors exhibited by AG would accelerate the speed of shell crust formation (Thevenet, 1988). This eventually triggers the onset of the selective diffusion mechanism in the earlier drying stage and thus further reduces the loss of volatiles during drying. At the slower drying rate, volatiles retention in MG-containing microcapsules was relatively higher than that produced with MD. Hence, the surface of MG-containing microcapsules was characterized by dents and free of surface cracks.

Hence, according to Chin et al. (2010), volatiles retention of the spray-dried powder comprising of propanethiol, ethyl propanoate, E2 MB, and diethyl disulfide were significantly (p > 0.05) affected by the inlet drying temperature and types of drying aids applied during spray drying. Spray-dried durian powder prepared using MG showed greater volatiles retention than those prepared with MD and NL.

TECHNICAL PROBLEMS IN SPRAY DRYING OF FRUITS AND SOLUTIONS

Major components present in fruits are sucrose, glucose, fructose, and citric acid. The technical difficulties of drying such sugar-rich foods are associated with the basic physical characteristics of the mixture of the low molecular weight sugars present, especially sucrose, maltose, glucose, and fructose. The fast removal of moisture during spray-drying results in either a

abMean values in the same row with different superscript indicate that there are significant differences among the treatments (p < 0.05).

^aMD refers to Maltodextrin DE 15 (Maldex 150), MG refers to blend of MD with gum Arabic AS IRX40830 at a ratio of 3:1, and NL refers to modified starch N-Lok.

^bIndicates inlet temperature T_i.

completely amorphous product or with some microcrystalline regions dispersed in the amorphous mass. The amorphous form is a nonequilibrium metastable state. Such amorphous products also show high degree of hygroscopicity.

Stickiness is a phenomenon frequently encountered during production and storage of dried powders. This property of fruit juice powders can be related to glass transition temperature (T_{σ}) of the components and sticky point temperature (T_s) . Stickiness of fruit juice powders was reduced by adding higher T_{g} components to the fruit juices and by modifying the spray-dryer design. Hence, additives are added which could reduce the hygroscopic and thermoplastic properties without altering the quality and solubility of the powder produced (Chegini et al., 2008). Sticky products are difficult to spray dry. During the drying process they may either remain as syrup or stick on the drier chamber wall. This can lead to lower product yield and operating problems. Some of these products are so sensitive to the drying conditions that a few degree celsius variations in temperature during drying turn them into a sticky and nonflowing mass (Bhandari et al., 1993).

Hence, to overcome these issues, various methods are available to produce free-flowing fruit juice powder by addition of drying aids (MDs with different DE, AG, tapioca starch, CTG, waxy starch, inulin, glucose syrup, and NL starches) and modification of spray-drying system, which was proved efficient over the standard laboratory spray dryer in many cases. And also several researchers obtained free-flowing fruit juice powder by making some modifications in the spray dryer such as cooling of the drying chamber walls (Chegini and Gobadian, 2005; Chegini et al., 2008), scrapping of dryer surfaces (Karatas and Esin, 1994), and admission of atmospheric air near the chamber bottom, allowing transport of the powder to a collector having a low humidity atmosphere (Ponting et al., 1973).

As far as the cooling of the drying chamber walls is concerned, the cold wall will be favorable to minimize the thermoplastic particles from sticking, as the wall will be cold enough to cool and solidify the outer surface of the thermoplastic particles coming in contact. This method, however, was found to improve the process but not to resolve the problem. The reason is that the cold chamber wall will also cool the surrounding environment and cause an increase in the RH of the air close to the wall surface (Goula et al., 2009). The same researchers Goula and others used dehumidified air as drying medium and MD as drying agent in order to produce free-flowing orange juice powder using spray drying, which was discussed earlier.

Goula and Adamopoulos (2005) also modified the spraydrying system for spray drying of tomato pulp. The modification made on the original design consisted of connecting the spray dryer inlet air intake to an absorption air dryer. Preliminary air dehumidification reduced residue accumulation on the walls in spray drying of tomato pulp, and also allowing the product to be dried at lower air outlet temperatures. The much lower outlet temperatures and humidities of drying air in the modified system resulted in the formation of a solid particle surface and so, decreased the residue accumulation or dryer fouling, minimizing the thermoplastic particles from sticking to the dryer wall (Goula and Adamopoulos, 2005). Preliminary air dehumidification improved not only product recovery, but also product properties of spray-dried tomato pulp powder, which was substantiated by the author.

An alternative approach to the cooling of drying chamber walls has been the Bris Tower process. The drying takes place in a very tall tower into which the tomato juice is introduced as a spray at a predetermined height. The whole drying process relies on the time-delayed fall of the product droplets and the very low temperature (not exceeding 30°C) of the upward airflow. In this way, explosion-type evaporation is avoided and the particles are not exposed to high temperatures likely to damage their organoleptic properties (Goose and Binsted, 1964). The cost of building and operating such towers is so high today that this design of tomato dryer is no longer realistic (Bhandari et al., 1997b).

Another system capable of producing a free-flowing product is a scraped surface drying chamber. This method can be very useful for relatively less thermoplastic sugar such as lactose or sucrose. Karatas (1989) developed an experimental spray dryer with a chamber wall scrapper specifically to dry tomato juice. The product recovery was up to 77% with a low inlet air temperature (115°C) (Goula and Adamopoulos, 2005).

Introduction to cool air at the lower part of the dryer chamber resulting in the formation of a solid particle surface can also reduce the stickiness of the powder particles (Ponting et al., 1973). However, limited amount of air can only be introduced because the cooling process will also raise the RH of the air that can once again aggravate the situation by increasing the surface moisture level (Bhandari et al., 1997b).

Lazar et al. (1956) reported that sticking of particle in the drier was decreased by cooling them with atmospheric air admitted near the bottom cone of the drier; this allowed transport of the powder to a collector having low humidity atmosphere. If dehumidified air was not used in transporting the powder to the collector, moisture content increased up to 0.5–0.8%. In spray drying, stickiness occurs when particles are insufficiently dry, collide with one another or with the dryer walls, and become stuck.

Masters (1985) stated that if the dryer is designed to accomplish product drying and agglomeration in one step (straight process), after dryer it is necessary to employ vibrated fluidized bed to finish the drying and cooling of the product. Wall deposition can lead to lower product yield, operating problems, and powder handling difficulties. Chambers with air brooms, which rotate slowly close to the wall, can also cool the wall surface to prevent stickiness of powders. The air broom arm contains a row of nozzles that direct compressed air on to the wall surface. An intermediate sweeping of chamber wall with dehumidified cold air can remove loosely adhered particles on the chamber wall. This method may be useful for relatively less thermoplastic sugars such as lactose and sucrose. The researcher Mani et al. (2002) also claimed that modified spray dryer with rotating air broom system reduced the stickiness problem by 30% to produce banana powder.

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Papadakis and Babu (1992) summarized that for products that are characterized by stickiness and are usually spray dried, common approaches involve cooling the dryer walls, using special dryer chamber designs and modifying the sticky characteristics of the material most frequently through the addition of drying aid agents. They also stated that the problem of stickiness in dryers could be dealt by trial and error experimentation in order to find conditions, which avoid or limit the sticky characteristics of the given material.

CONCLUSIONS

Spray drying has moved into all major industries, from food and pharmaceutical manufacturing in the most delicate of conditions to high-tonnage outputs from heavy chemical fields such as mineral ores and clays. This process is pervasive and is used to produce many products and articles that are used in daily life. Spray dryers are unique in that they can produce powders of a specific particle size and moisture content independent of the dryer capacity and product heat sensitivity. This paper reviewed the spray drying of fruit extracts and the effects of additives on the physicochemical properties of the products. Microcapsules obtained under optimal spray-drying conditions showed a higher recovery of bioactive compounds and, thus, can be used as additives in the food processing or pharmaceutical industries. Moreover, if proper additives such as CTG are used to retain the bioactive compounds in the powder, countries that produce those additives will experience socio-economic benefits. This review also demonstrates that the additive concentration and inlet temperature of spray drying influence physicochemical properties such as residue formation, moisture content, bulk density, rehydration, hygroscopy, and degree of caking. Besides, the problems of stickiness, residue accumulation or dryer fouling can be solved by modification of spray-drying system.

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GLOSSARY

 $T_{\rm g}$ = Glass transition temperature

TE = Trolox equivalent
DE = Dextrose equivalent

AG = Arabic gum
CTG = Cashew tree gum
MD = Maltodextrin

TCC = Total carotenoid content

TAA = Total antioxidant activity

FOS = Fructooligosaccharide DP = Polymerization degree

CP = Cactus pulp

= Cactus ethanolic extract

I = Inulin

RH = Relative humidity

TEAC = Trolox equivalent antioxidant capacity

 $a_{\rm w}$ = Water activity

WSI = Water solubility index EE = Encapsulation efficiency

XRD = X-ray diffraction

SEM = Scanning electron microscopy

MG = Blend of MD with AG at a ratio of 3:1

 $T_{\rm o}$ = Outlet temperature $T_{\rm i}$ = Inlet temperature DV = Daily value

DGS = Dried glucose syrup

J = Juice

E2 MB = Ethyl 2-methylbutanoate NL = Modified starch N-Lok

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