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Full Length Research Article

Impact of Spray Drying on Physical Properties of *S. anguivi* L. and *Emblica officinalis* Combined Powder

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Abstract

The present study aimed to produce iron-rich spray-dried powder from *Solanum anguivi* L. (S) with enriched vitamin C from *Emblica officinalis* (E) through a spray-drying process. Fresh juice of *Solanum anguivi* L. and *Emblica officinalis* (S1:E3) were extracted and dried using three different carrier agents (Gum Acacia, Maltodextrin, β-Cyclodextrin at 15%) in a laboratory scale spray dryer with 130°C and 60°C drying temperature were maintained. The developed juice powders were analyzed for encapsulation efficiency, yield, bulk properties, reconstitution properties, hygroscopicity, degree of caking of the particles and color attributes. The results procured that Gum Acacia and Maltodextrin encapsulated powders improve drying yield, encapsulation efficiency and moisture free which is the most effective, whereas the reconstitution properties and bulk properties revealed that β-Cyclodextrin encapsulated powder obtained better results. However, the percentage for a degree of caking and hygroscopicity was elevated which was influenced by ambient temperature and resulted in stickiness. Thus, S1:E3 juice powder encapsulated with Gum acacia, Maltodextrin and β-Cyclodextrin is seen with wholesome quality in terms of its physical properties.

***Key words*:** Bulk density, Degree of caking, Hygroscopicity, *Solanum anguivi* L., Spray-drying, β-Cyclodextrin

Carbohydrates are the preferred substance for the encapsulation process due to their widespread availability and lower price. Furthermore, their superiority over other encapsulating agents is a result of their good solubility properties and capacity to generate low-viscosity solutions at larger solids concentrations. The most popular encapsulating agents include maltodextrin and β-cyclodextrin, which are starch derivatives. Because it is less hygroscopic and amphiphilic than other wall materials, β-cyclodextrin is discovered to be the ideal choice for this method. According to McNamee *et al* (1998), gum acacia is a complex polysaccharide that comprises rhamnose, galactose, glucuronic acids, arabinose, and a lower fraction of protein. The gum acacia's functional qualities are determined by this protein content (Anderson *et al.*, 1966). The majority of the food industries use Gum Arabic for their spray drying applications to prevent oxidation and volatilization of the core material (Shaikh *et al.*, 2006). In addition, compared to other hydrocolloid gums, it has a high solubility and a low viscosity in an aqueous solution, which makes spray drying efficient.

Spray drying is of the supreme drying technique that converts fluid into solid substances (Murugesan *et al.*, 2011). In the process of spray drying, a liquid product is instantly transformed into powder by being atomized in a hot gas current. Air is typically utilized, or less frequently, an inert gas, particularly nitrogen gas. An emulsion, solution, or suspension can be used as the first liquid feeding (Gharsallaoui *et al.*, 2007). Both heat-sensitive and heat-resistant products can be modified with the help of this method. The analysis reveals that there has been very little research on enhancing the synthesis of vitamin C powder by processing Solanum *anguivi* L. Insufficient engineering and lack of nutritional data for *Solanum anguivi* L. processing create a need to do more research and study in this field. Thus, the present work is aimed to encapsulate the juice of *Solanum anguivi* L. since it is a rich source of iron and it is enhanced with vitamin C from the natural source of *E. officinalis*, the carrier agent of Gum acacia, β-cyclodextrin and Maltodextrin were used for encapsulation and it was carried out with physical parameters.

MATERIALS AND METHODS

Preliminary experiment

According to the previous study by Karthika and Poongodi, 2022, juices were collected, authenticated and quantified its nutritional and antioxidant properties. Out of different combinations, S1:E3 (*Solanum anguivi* L. at 25% and *Emblica officinalis* at 75%) juice was selected for the microencapsulation process through spray drying technology to prevent oxidation.

Emulsion preparation

Emulsions were prepared with concentrated S1:E3 juice extract with total soluble solids (TSS) equivalent to 12 Brix.

The carrier substances such as Gum Arabic (GA), Maltodextrin (MD), and β-cyclodextrin (β-CD) were incorporated with a concentration of 15% (w/w) to the fruit juice extract when the total solids were consistent. The feed emulsion was immediately spray-dried after being prepared. The emulsion was agitated to homogeneity with an Ultra-Turrax T-50 basic homogenizer at a speed of 5200 rpm for 8 minutes.

Spray drying

Using Lab spray dryer LSD-48, which has a drying chamber of 150 mm diameter, the prepared emulsions were dried at 130°C and 60°C with inlet and outlet temperatures respectively. Consequently, the airflow at 200 ml/hr at an atomization pressure of 2 bars and the size of the nozzle aperture was 1.5 mm. The compressor pressure, air flow rate and feed rate were constant at 0.12 MPa, and 3.3 mL/min respectively.

Physical properties of spray-dried powder

1. *Microencapsulation efficiency*

The percentage of vitamin C present in 100g of S1:E3 powder were used to calculate the effectiveness of microencapsulation (Eq. 1).

Encapsulation Efficiency % = (vitamin C in S1:E3 / total vitamin C before drying) 100 ……. Eq. 1

2. *Total yield*

The amount of powder that was collected was used to determine the spray drying yield (Eq. 2)

$$\%YE = W_2 / W_1 \text{ X } 100 \text{ ……. Eq. 2}$$

Where YE is the yield (g/100 g^{-1}), W_1 is the weight (g) of the non-solvent mass in the feed, and W_2 is the weight (g) of the final product.

3. *Reconstitution properties*

i. Dispersibility

The dispersion time required for the powder was determined with a slight modification by Quek *et al*., 2007. 10 g of each microencapsulated powder was added to a 250 mL beaker containing 100 mL of double distilled water in a 25°C water bath. The time (s) was immediately recorded when the stirring began until all samples were dispersed (Quek *et al*., 2007).

ii. Wettability

The procedure of Fuchs *et al* (2006) was employed to absorb the powders' wettability. Without agitation, one gram of each microencapsulated powder was dropped at 20°C in 100 mL of double distilled water. The amount of wettability of the samples was compared using the time taken for the powder particles to settle, sink, immerse, and disappear from the water's surface.

iii. Solubility

100 ml of distilled water was taken to dissolve one gram of each microencapsulated powder and it was stirred for five minutes in a vortex before being centrifuged at 3780 X g for another 5 minutes. Besides that, a 25ml aliquot of the supernatant was weighed, transferred to a petri dish, and dried at 105°C for 5 hours. The solubility (g/100ml^{-1}) was computed based on the weight difference after the solids were retrieved and weighed (Shittu and Lawal, 2007).

4. *Bulk properties*

i. Bulk and tapped density

The bulk density of S1:E3 powders was estimated by Gallo *et al.,* 2011 with slight modifications. A ten gram of powdered sample was poured into a 10 cm^3 graduated cylinder and the volume occupied by the powder was observed. For the tapped density, the cylinder was mechanically tapped after the initial volume was perceived, and the volume was monitored till it achieved recurring volume. This formula is used to determine tap density.

Bulk density (g/ml) = Mass of the powder/volume of the powder (ml) …….. Eq. 3

Tap density (g/ml) = Mass of the powder/final tapped volume …….. Eq. 4

ii. True Density

The sand displacement method was adopted for calculating the true density of each microencapsulated powder. One gram of powder was added to a 10 ml graduated measuring cylinder which contains a fixed volume of (5 ml) sand and the volume increase was taken into account. The particle size of the sand plays a pivotal role and hence, the sand was sieved in a 75µ sieve to have uniform particle size.

True Density (g/ml) = (Mass of the powder (g)/ volume of powder displaced by the sand …… Eq. 5

iii. Flowability

The flowability of each microencapsulated powder was estimated in terms of the Carr's index (CI) and Hausner ratio (HR) as per the study of Jinapong *et al.,* 2008 Bulk and tapped densities of the powders were considered to derive CI and HR value:

CI = (Tapped density – Bulk density) / Tapped density X 100…….Eq. 6

HR = Tapped density / Bulk density……. Eq. 7

iv. Hygroscopicity

The Tonon *et al*., (2008) approach was used to determine the hygroscopicity. The sample was added with NaCl-saturated solution to a container at 25°C with 2 g of the microencapsulated powder. After a week, the percentage of hygroscopicity (absorbed moisture) is calculated (g/100 g^{-1}).

v. Degree of caking

Following the hygroscopicity assessment, the wet sample is dried at 102°C in a hot air oven. After cooling, the dried material was weighed and sieved in a sieve of 500-µm size using a shaking apparatus for 5 minutes. The microencapsulated powder that was left in the sieve was weighed (Jaya and Das 2004).

$$CD\ \% = b/a \text{ X } 100 \text{ …… Eq. 8}$$

where CD represents the percentage of the degree of caking, a indicated the total amount of microencapsulated powder for the sieving process and b indicates the amount of the powder left in the sieve after sieving.

vi. Color attributes

Color attributes for S1:E3 powders were assessed using (Model No. A60-1012-312, Hunter Associates Laboratory, Reston, VA), in terms of CIE (Commission Internationale de L'Eclairage) 'L*' (lightness and darkness), 'a*' (redness and greenness), and 'b*' (brightness) (yellowness and blueness). The sensor was first calibrated using a black-and-white reference tile for assessing color. Calculations were determined using the samples' total color difference (E), hue angle, and color intensity (chroma) (Duangmal *et al.*, 2008).

Statistical analysis

The software Statistical 17.1 was used to perform the statistical analyses of experimental results. A one-way ANOVA test was used to determine among the physical parameters

whether there was a significant difference between the carrier agents.

RESULTS AND DISCUSSION

The ratio of entrapped vitamin C content in microencapsulated powder (theoretical loadings) is the percentage of Microencapsulation efficiency. The encapsulation efficiency was significantly greater in GA (93.8±1.88%) powder followed by MD (84.24%) and β-CD (83.18%) juice powders (Table 1). Similarly, GA powder procured significantly ($p<0.05$) highest value at 83.18±2.04% than the MD (81.45±1.89%); and β-CD (54.39±2.19%) powders. The time required to disperse the particle of GA powder (Table 1) was significantly higher (88.33±1.50 sec) whereas MD (49.33±1.15 sec) and β-CD (34.66±3.05 sec), powders were dispersed quickly and wet thoroughly, sinks rather than float without any lump's formation. In the case of wettability, the β-CD powder obtained a lower value ($p<0.05$) than the GA and MD powders, whereas no significant difference was observed in their solubility.

The bulk density, tapped density and true density (Table 1) of GA and MD powders were significantly lower ($p<0.05$) than β-CD powder. According to the classification given by Lebrun *et al* (2012), GA and MD powders significantly acquired poor flowability, however, the β-CD powder (Carr's index – 17.27±2.52%; Hausner's ratio – 1.20±0.03) obtained fair flowability and intermediate cohesiveness.

Hygroscopicity (Table 1) of GA (48.33±0.57%) and MD (48.66±0.57%) powders were significantly low when compared to β-CD (55.83±0.76%) powder. Although the degree of caking did not show a significant difference among GA (10.50±0.61) and MD (14.96±0.61) powders while β-CD powder obtained a significantly ($p<0.05$) higher degree of caking (44.92±1.98).

Table 1 Physical properties of microencapsulated spray-dried powders

S. No.	Particulars		GA	β-CD	MD
1.	Encapsulation Efficiency		93.8±1.8^{c}	83.1±1.6^{a}	84.2±0.9^{b}
2.	Total Yield		83.1±2.0^{c}	54.3±2.1^{a}	81.4±1.8^{b}
3.	Dispersibility (sec)		88.33±1.50^{c}	34.66±3.05^{a}	49.33±1.15^{b}
4.	Wettability (sec)		103.33±1.5^{b}	53±2.64^{a}	62.33±2.08^{a}
5.	Solubility Index (%)		85.33±1.10*	86.80±0.72*	85.73±1.10*
6.	Bulk density (g/mL)		0.61±0.02^{a}	1.40±0.03^{c}	0.73±0.02^{b}
7.	Tapped density (g/ml)		0.87±0.02^{a}	1.69±0.05^{b}	0.84±0.01^{a}
8.	Porosity (%)		39.94±2.11^{c}	9.29±0.55^{a}	31.58±0.35^{b}
9.	Flowability	Carr's Index (%)	29.63±3.6^{c}	17.27±2.52^{a}	26.67±2.31^{b}
		Hausner's Ratio (%)	1.42±0.07^{b}	1.20±0.03^{a}	1.36±0.04^{a}
10.	Hygroscopicity		48.33±0.5^{a}	55.83±0.7^{b}	48.66±0.5^{a}
11.	Degree of Caking		10.50±0.6^{a}	44.92±1.9^{c}	14.96±0.6^{b}

‡Values are the average of three determinants. The a,b,c alphabets indicate the significant mean difference suggested by LSD
*No significant difference

The L value (Table 2) indicates the degree of lightness, significantly highest value in the β-CD (102.95±0.03) powder than other powders represented more lightness; β-CD also revealed high redness (a) and yellowness (b). GA powder was significantly lower in lightness and redness, and MD powder predicted significantly lower yellowness at $p<0.05$.

The chroma values revealed redness was significantly ($p<0.05$) high in β-CD powder and showed more vividness and brightness along with significantly high white index and yellow index than in GA and MD powders. Least saturation was acquired by GA and MD powders that exhibited a dull or grayish color. According to total color difference, the least difference was noticed in MD powder (13.92±0.009) followed by GA (15.00±0.07) powder and a significantly high difference was observed in β-CD (21.26±0.01) powder.

Variations in the microencapsulation efficiency might be related to the low viscosity, high solubility, and high glass transition temperature of its emulsion feed as reported by Goula and Adamopoulos (2008). Similar microencapsulation efficiency was reported by Kadam *et al* (2010) in Ginger oil (91%); Haidong *et al* (2012) in ginkgo leaf extract (82.4%). Denaturation and structural changes that happened in the feed at the time of the spray drying process reduce the ability to bind and encapsulate vitamin C which is similar to the report of Al-Ismail *et al* (2016). Besides, the dextrose equivalent (DE) also plays a major role in encapsulation efficiency; the dextrose equivalent of MD is 16.5 – 19.5 (Gavaric *et al.*, 2019) and β-cyclodextrin is 8 (Pishtiyski and Zhekova, 2006), which may reflect in the fair encapsulation efficiency when compared to GA powder.

Table 2 Color analysis of microencapsulated spray-dried powders

Color value	L	a	b	Chroma	Hue	Saturation	Yellow index	White Index	Total color difference
GA	94.12±0.02^{a}	4.07±0.01^{a}	14.53±0.06^{b}	15.09±0.05a	1.30±0.00^{d}	0.16±0.00^{a}	22.06±0.09^{b}	94.80±0.01^{a}	15.00±0.07^{b}
β-CD	102.95^{d}±0.03	11.28±0.01^{d}	19.44±0.04^{d}	22.48±0.03^{d}	1.04±0.00^{b}	1.99±0.00^{c}	26.98±0.05^{d}	104.90±0.03^{d}	21.26±0.01^{d}
MD	98.82±0.02^{b}	9.37±0.01^{c}	12.55±0.05^{a}	15.66±0.03^{b}	0.93±0.00^{a}	0.16±0.00^{a}	18.14±0.07^{a}	99.55±0.02^{b}	13.92±0.009^{a}

‡Values are the average of four determinants. a,b,c,d alphabets indicate the significant mean difference suggested by LSD test

In microencapsulation, encapsulation efficiency and total yield are considered vital aspects. Experimental conditions such as inlet temperature, flow rate and compressed air flow determine the yield of microspheres by spray drying. According to the report of Sharifi *et al* (2015) in barberry *(Berberis vulgaris)* the yield was 78% with GA-MD blend at 180°C inlet temperature; Shishir *et al* (2014) in pink guava powder was 52% and 55% yield at the respective level of 15% and 20% maltodextrin, the yield noted in the present study was greater and compatible for commercialization. The lower yield could be due to lower concentrations of the encapsulating agents and higher ratios of core material resulting in a lower yield of spray-

dried fruit powder. In addition to that, the chemical structure and less dextrin equivalent of β-CD which stuck on the drying chamber during the spray drying process was directly related to the interfacial surface energy of contacting material, as resulted in lower yield Muzaffar *et al* (2015). Solubility is the dissolving of soluble particles in the liquid. Dispersibility is particle dispersion with slight stirring (Koc *et al*., 2014). The greater seconds to disperse GA encapsulated powder might be due to lumps formed during water adsorption than other encapsulating agents and also related to the temperature of the medium to disperse as stated by Koc *et al* (2014) in spray-dried yogurt powder. The low percentage of wettability in β-CD powder has a high moisture content which tends to form agglomerations and hence, the penetration of fluid into the pores is easier (Ochoa *et al.,* 2015). Correspondingly, the result was obtained by Ferrari *et al* (2012), who found a significant difference in wettability when using 7% of MD and 7% of GA as encapsulating agents, at 82.20 and 134.20 seconds respectively. Solubility is the main quality indicator to evaluate the behavior of powder products in an aqueous solution and it is the final step of powder dissolution and is taken as the major determinant for the overall reconstitution quality (Fang *et al.,* 2007). Higher Hydrophilic components in encapsulating agents of β-CD and MD leads to higher solubility in S1:E3 powder.

Bulk density and tapped density are strongly linked with the moisture content of powder particles with less moisture content and are lighter in density. The higher bulk density and tapped density of β-CD encapsulated powder could be due to heavier material that accommodates easier into the spaces between the particles as revealed by Ferrari *et al* (2012). The true density of microencapsulated spray dried fruit powders was comparable with the level reported by Patil *et al* (2014) (1.13 – 1.24 g/ml) in guava fruit powder; Tze *et al* (2012) in pitaya fruit powder at 20% (1.30–1.48 g/ml) and 30% (1.14-1.44 g/ml) of maltodextrin. It revealed that true density reflected as real solid density present in the particles opted by Tonon *et al* (2010). The poor flowability attributed to the liquid bridges and capillary forces acting flanked by powder particles reduced the flowability and raised the cohesiveness of the spray-dried fruit powder (Kim *et al.,* 2009). However, the β-CD encapsulated powder had fair flowability and intermediate cohesiveness because of the diminution of friction and binding caused by surface roughness since moisture acted as a lubricant (Mobhammer *et al.,* 2005). The hygroscopicity of spray-dried powders reflected the capacity to absorb the environmental moisture that directly affects the quality of the powder (Wang *et al.,* 2020). This could be endorsed by the biopolymers used for the encapsulation of fruit juice which had a hydrophilic character related to the immense water concentration gradient amidst the powder and the atmospheric temperature. Akin findings were denoted by Moreira *et al* (2009) in acerola pomace extract during spray drying technology (34.72±1.93 – 51.16±2.44%). The degree of caking of β-CD and MD encapsulated powders were identical to the result reported by Fontes *et al* (2014) in prebiotic fruit powder that varied from 23.87±1.92; 35.33±1.57; 67.02±0.23 of pineapple, melon, orange juice respectively; Koc *et al* (2014) in yogurt powder (30.32±3.37); Goula and Adamopoulos (2008) in tomato powders (8.9 to 22.3%) and Martinelli *et al* (2007) in lemon powder (21.11±2.12 – 34.77±2.43).

The color of the fruit powder is an important factor in determining the color of the reconstituted powders. The obtained L, a, and b values were significantly higher than the level reported by Shrestha *et al* (2007) in spray-dried orange juice powder. The observed L and b values were lower; redness (a) was higher than the level reported by Caparino *et al* (2012) in mango powder. The chroma values revealed redness was significantly ($p<0.05$) high in β–CD and low in GA and MD encapsulated powders. The hue value is the intensity range of dark color from redness followed by grey > white > yellowness (0-100 units) (Tolvaj and Nemeth, 2008). Osorio *et al* (2010) reported that chroma and hue values were extremely high in corozo (*Bactris guineensis*) fruit. Saturation refers the color intensity and purity (Stone *et al.,* 2008). Significantly high white index and yellow index in β–CD encapsulated powder as a reflection of L and b values. A similar trend was produced by the studies of Quek *et al* (2007) in spray-dried watermelon powder and Shishir *et al* (2014) in spray-dried guava powder.

CONCLUSION

This study demonstrated the economic viability of spray drying as a method for concentrating *Solanum anguivi* L. and *Emblica officinalis* juice into a powder that can then be reconstituted to create an instant beverage. It was discovered that the parameters of inlet and outlet air temperature for spray drying had a significant impact on product yield. The ideal operating conditions are 130°C for the input air, 60°C for the output, and 12° Brix for the total soluble solids. The physical characteristics of different encapsulated powders were found to be good in quality in every evaluated aspect. To infer, spray drying is used for preserving juice, and the use of this method will set up new market opportunities.

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