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MICROENCAPSULATION OF ROSELLE (*HIBISCUS SABDARIFFA* L.) THROUGH SPRAY DRYING

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ABSTRACT

Roselle calyces are known for their rich nutritional content, bioactive compounds and therapeutic properties. However, these calyces have a limited seasonal availability and are highly perishable. As a result, processing of roselle calyces through spray drying is crucial in increasing the availability of the product during the off-season. Thus, an attempt was made to optimise the processing variables viz., inlet temperature (170, 180 and 190 °C) and maltodextrin concentration (5, 10 and 15%). The results of different parameters varied moisture content 5.74-6.14%, water activity 0.37-0.50, swelling capacity 13.33-12.46 ml/g, water absorption capacity 9.45-8.51 ml/g, anthocyanin content 716.26-697.29 mg/100g, total phenols 39.06-38.79 mg GAE/100g and antioxidant activity 92.22-87.31%. In conclusion, the high-quality spray dried roselle powder can be obtained at inlet temperature of 190°C and maltodextrin concentration 15 per cent resulted in minimum moisture content, water activity, swelling capacity and water absorption capacity that could be stable for the storage.

Keywords : Spray drying, moisture content, water activity and total phenols.

Introduction

Roselle (*Hibiscus sabdariffa* L.) belonging to family Malvaceae is likely native to tropical central and West Africa. It is primarily cultivated in tropical and subtropical regions for its appealing edible calyces. In India it is named in various languages as Lal ambari, Patwa, Gongura (Hindi), Chukar, Lal-Mista (Bengali), Chukiar (Assam), Lal-ambadi (Marathi), Pulichachai, Polechi (Malyalam), Pulachekari, Pundibija (Kannada), Yerragogura (Telugu) and in Tamil called as Pulichchaikerai (Gautam, 2004).

Roselle calyces are perishable in nature and susceptible to post harvest losses. Glut of seasonal crops during the peak production periods not only lowers the market price, but also leads to heavy losses. Storage of juice or conversion of juice into powder

would be a correct approach for long term preservation.

The advancement of processing technology plays a pivotal role in maximizing the utilization of roselle calyces by creating value-added products. This approach helps to ensure the availability of roselle calyces for an extended period. By transforming them into processed products, their benefits can reach consumers for a more extended period, enhancing their economic and nutritional value for rural communities.

In addition to pulp preservation, roselle calyces can be preserved using various other methods. One such method involves converting the roselle juice into powder through techniques like freeze drying, foam mat drying, spray drying and tunnel drying. Among these, spray drying is a widely employed technique for

producing fruit juice powders. Consequently, there is a significant potential for preparing roselle powder using spray drying with maltodextrin as a carrier agent. The aim of this study was to optimise the spray drying technique in roselle.

Material and Methods

Materials: Roselle (*Hibiscus sabdariffa* L.) flowers were sourced from Horticultural Research and Extension Centre (HREC) Badakundri (Hidkal Dam) in Karnataka and the flowers were brought to the laboratory to facilitate subsequent experimental procedures.

Methodology for the production of roselle powder. The experiment was conducted using a laboratory spray dryer (LSD-48) manufactured by Techno search and systems Pvt. Ltd., located in Thane, Mumbai.

Spray drying process: Feed solution is prepared by diluting 100 ml of extracted roselle juice with water in 1:1 ratio for which maltodextrin was added and continuously homogenized for 8-10 minutes in the mixer and filtered with the double sieve, inlet air temperature (170, 180 and 190°C) and maltodextrin concentration (5, 10 and 15%), outlet temperature of 80 °C, feed flow rate 2 ml/min was maintained. The obtained powder was collected and packed in 50-micron aluminium pouches and stored at an ambient room temperature. The assessments were conducted on samples taken to analyze alterations in both physical and chemical characteristics.

Statistical analysis: The interpretation of data was carried out in accordance with Panse and Sukhatme (1985). The level of significance used in the 'F' test was $p=0.01$.

Analysis of physicochemical properties

Moisture content (%)

The moisture content of the powdered roselle calyces was measured using a moisture analyzer.

Water activity (a_w)

The water activity of spray-dried roselle calyces powder was measured using a water activity meter (Model: Novasia AG, Switzerland).

Swelling capacity (ml/g)

The swelling capacity was determined by placing the sample in 100 ml graduated cylinder until it reached the 10 ml mark. Distilled water was added to make a total volume of 50 ml. The top of the graded cylinder was tightly sealed and mixed by flipping it, method as suggested by Okaka and Potter, 1979.

Water absorption capacity

The water absorption capacity was determined using a modified Lin and Zayas (1987) method. Two grams of the sample (W_1) were mixed with 30 ml of 70°C distilled water, agitated for 30 minutes and settled for 10 minutes. Residue was scraped and washed with 10 ml of hot water, then centrifuged at 1165 rpm for 25 minutes at 50°C. The cooled sample was weighed (W_2).

$$\text{Water absorption capacity (ml/g)} = \frac{W_2 - W_1 \text{ (ml)}}{\text{Weight of the sample (g)}}$$

Chemical parameters

Antioxidant activity (%)

The antioxidant activity of spray dried roselle powder was determined using 2,2-diphenyl-2-picrylhydrazyl (DPPH) free radical as per the procedure suggested by Eghdami *et al.* (2010).

$$\text{Antioxidant activity (\%)} = \frac{A_{517} \text{ nm of control} - A_{517} \text{ nm of sample}}{A_{517} \text{ nm of control}} \times 100$$

Anthocyanin (mg/100 g)

Total anthocyanin content of spray dried roselle powder was determined according to the pH differential spectroscopic method reported by Lee *et al.*, 2005.

$$\text{Total OD/100 g} = \frac{\text{OD}_{535} \times \text{Volume made up} \times 100}{\text{Weight of sample}}$$

Total phenols (mg GAE/100 g)

The total phenol content of spray dried roselle powder was determined using the Folin-Ciocalteu Reagent (FCR) method as studied by Sadasivam and Manickam, 2005.

Result and Discussion

Effect of spray drying condition on moisture content and water activity

Moisture content and water activity are important powder properties as they are related to the drying efficiency. Moisture content and water activity of micro encapsulated powder plays an important role in determining the flowability, stickiness and storage stability. Moisture content and water activity of spray dried roselle powder were in the range of 5.74 to 6.14 per cent and 0.37 to 0.50, respectively, which was sufficient to make powder microbiologically safe. Similar result was reported by Tze *et al.* (2012) in pitaya fruit powder where moisture content of the powder was less than 10 per cent. During storage, moisture content and water activity of the powder were

increased due to hygroscopic nature of the spray dried powder (Shrestha *et al.*, 2007).

Generally, increasing the inlet temperature and maltodextrin decreased the moisture content and water activity of the powder. The lowest moisture content (5.24%) and water activity (0.24) were noticed in the treatment T₉ with 190 °C inlet temperature and 15 per cent maltodextrin during initial period and upon storage a meagre increase was recorded (Table 1). This

might be due to the greater loss of water from powder because of the higher rate of heat transfer into sprayed small particles (more surface area), which caused faster water removal. The results in the present investigation were following similar trend and are in line with Ersus and Yurdagel (2007) in black carrot powder, Chegini and Ghobadian (2005) in orange juice powder, Rodriguez *et al.* (2005) in cactus pear juice and Goula *et al.* (2004) in tomato powder.

Table 1 : Influence of treatments on moisture content and water activity of spray dried roselle powder during storage

Treatment	Moisture content (%)				Water activity (a _w)			
	Initial	1 MAS	2 MAS	3 MAS	Initial	1 MAS	2 MAS	3 MAS
T ₁	6.68	6.71	6.78	6.82	0.42	0.50	0.56	0.65
T ₂	6.04	6.07	6.10	6.17	0.39	0.43	0.50	0.56
T ₃	5.59	5.61	5.65	5.73	0.37	0.39	0.44	0.48
T ₄	5.88	5.91	5.97	6.05	0.41	0.44	0.51	0.61
T ₅	5.66	5.69	5.76	5.83	0.38	0.41	0.45	0.52
T ₆	5.53	5.56	5.60	5.64	0.37	0.40	0.42	0.47
T ₇	5.66	5.69	5.75	8.00	0.38	0.42	0.46	0.52
T ₈	5.42	5.44	5.52	5.59	0.35	0.39	0.41	0.44
T ₉	5.24	5.26	5.38	5.46	0.24	0.25	0.25	0.26
Mean	5.74	5.77	5.83	6.14	0.37	0.40	0.44	0.50
S.Em±	0.17	0.22	0.17	0.18	0.06	0.03	0.03	0.04
C.D. @ 1%	0.49	0.62	0.50	0.53	NS	0.08	0.08	0.10

MAS- Months after storage

T₁- Inlet temperature of 170oC + maltodextrin of 5%

T₂- Inlet temperature of 170oC + maltodextrin of 10%

T₃- Inlet temperature of 170oC + maltodextrin of 15%

T₄- Inlet temperature of 180oC + maltodextrin of 5%

T₅- Inlet temperature of 180oC + maltodextrin of 10%

NS- Non significant

T₆- Inlet temperature of 180oC + maltodextrin of 15%

T₇- Inlet temperature of 190oC + maltodextrin of 5%

T₈- Inlet temperature of 190oC + maltodextrin of 10%

T₉- Inlet temperature of 190oC + maltodextrin of 15%

Swelling capacity and water absorption capacity

Swelling capacity (SC) and water absorption capacity (WAC) are the important properties in deciding stability, yield, texture and sensory evaluation. SC denotes the degree of powder hydration and WAC represents the amount of water held and absorbed by the hydrated sample after an external force is applied. SC and WAC for roselle powder are shown Table 2. Swelling capacity (SC) and water absorption capacity (WAC) of spray dried roselle powder were in the range of 13.33 to 12.46 ml/g and 9.45 to 8.51 ml/g, respectively. There was a decrease in SC and WAC during storage period and SC and WAC increased with the increase in the inlet temperature and maltodextrin concentration. The highest SC (21.47 ml/g) and WAC

(16.86 ml/g) were noticed in the treatment T₉ with 190°C inlet temperature and 15 per cent maltodextrin concentration during initial period. This might be due to the breakdown of intermolecular hydrogen bonds in the amorphous region allowing gradual and irreversible water absorption which facilitated the breakdown of hydrogen bonding and swelling capacity is directly related to the particle size hence, with the increase in the size of the particle the SC also increased thereby increasing the SC and WAC of the spray dried powder. A slight reduction of SC and WAC was noticed upon storage, this could be due to the stickiness of the powder. Similar results were found by Izidoro *et al.* (2011) in spray dried banana powder and Alpizar-Reyes *et al.* (2017) in tamarind seed mucilage powder.

Table 2 : Influence of treatments on swelling capacity and water absorption capacity of spray dried roselle powder during storage

Treatment	Swelling capacity (ml/g)				Water absorption capacity (ml/g)			
	Initial	1 MAS	2 MAS	3 MAS	Initial	1 MAS	2 MAS	3 MAS
T ₁	4.86	4.64	4.31	3.95	4.66	4.22	3.97	3.72
T ₂	6.15	6.02	6.00	5.54	5.17	4.83	4.62	4.28
T ₃	8.69	8.37	7.98	7.43	6.98	6.57	6.29	5.95
T ₄	10.23	10.07	9.82	9.35	7.09	6.63	6.50	6.11
T ₅	13.45	13.12	12.76	12.47	8.47	8.24	7.92	7.72
T ₆	16.37	16.23	15.94	15.62	10.65	10.19	9.79	9.57
T ₇	18.72	18.65	18.33	17.99	11.68	11.15	10.86	10.59
T ₈	20.15	20.02	19.77	19.23	13.52	12.96	12.77	12.67
T ₉	21.47	21.29	20.97	20.58	16.86	16.47	16.21	15.98
Mean	13.33	13.17	12.88	12.46	9.45	9.03	8.77	8.51
S.Em±	0.45	0.50	0.44	0.44	0.48	0.40	0.32	0.30
C.D. @ 1%	1.32	1.45	1.28	1.27	1.42	1.16	0.95	0.89

MAS- Months after storage

T₁- Inlet temperature of 170°C + maltodextrin of 5%T₂- Inlet temperature of 170°C + maltodextrin of 10%T₃- Inlet temperature of 170°C + maltodextrin of 15%T₄- Inlet temperature of 180°C + maltodextrin of 5%T₅- Inlet temperature of 180°C + maltodextrin of 10%T₆- Inlet temperature of 180°C + maltodextrin of 15%T₇- Inlet temperature of 190°C + maltodextrin of 5%T₈- Inlet temperature of 190°C + maltodextrin of 10%T₉- Inlet temperature of 190°C + maltodextrin of 15%**Table 3 :** Influence of treatments on anthocyanin, total phenols and antioxidant activity of spray dried roselle powder during storage

Treatment	Anthocyanin content (mg/100g)				Total phenols (mg GAE/100g)				Antioxidant activity (%)			
	Initial	1 MAS	2 MAS	3 MAS	Initial	1 MAS	2 MAS	3 MAS	Initial	1 MAS	2 MAS	3 MAS
T ₁	660.90	656.10	649.30	642.30	35.23	35.19	35.03	34.92	85.92	84.65	83.84	80.68
T ₂	720.20	715.90	709.50	700.90	37.79	37.74	37.58	37.50	91.90	90.54	89.67	86.42
T ₃	738.20	733.60	727.90	721.70	39.82	39.78	39.66	39.54	94.29	93.69	92.78	90.09
T ₄	676.40	670.40	662.70	657.80	37.37	37.33	37.23	37.12	88.97	87.72	86.72	83.24
T ₅	729.43	726.13	718.20	713.20	38.43	38.37	38.25	38.15	92.74	91.98	91.48	87.14
T ₆	746.80	740.70	733.60	726.20	40.72	40.67	40.56	40.43	94.72	93.25	92.68	90.86
T ₇	687.50	683.70	675.80	670.40	39.65	39.60	39.49	39.40	90.91	89.14	87.84	84.65
T ₈	735.60	729.30	720.40	712.50	40.54	40.49	40.38	40.29	93.10	92.78	90.56	89.48
T ₉	751.30	748.50	739.40	731.10	41.97	41.92	41.85	41.75	96.70	95.87	93.34	90.47
Mean	716.26	711.59	704.03	697.29	39.06	39.01	38.89	38.79	92.22	91.13	89.99	87.31
S.Em±	22.74	22.59	22.35	21.14	1.24	1.23	1.22	1.25	2.45	2.29	2.26	2.30
C.D. @ 1%	67.44	66.01	65.33	63.73	3.70	3.66	3.64	3.61	7.34	6.88	6.78	6.91

MAS- Months after storage

T₁- Inlet temperature of 170°C + maltodextrin of 5%T₂- Inlet temperature of 170°C + maltodextrin of 10%T₃- Inlet temperature of 170°C + maltodextrin of 15%T₄- Inlet temperature of 180°C + maltodextrin of 5%T₅- Inlet temperature of 180°C + maltodextrin of 10%T₆- Inlet temperature of 180°C + maltodextrin of 15%T₇- Inlet temperature of 190°C + maltodextrin of 5%T₈- Inlet temperature of 190°C + maltodextrin of 10%T₉- Inlet temperature of 190°C + maltodextrin of 15%

Chemical parameters

Anthocyanin, total phenols and antioxidant activity

The spray dried roselle powder exhibited an increasing in anthocyanin, total phenols and antioxidant activity. Over a storage period of up to three months, this value significantly decreased. The highest anthocyanin, total phenols and antioxidant activity content was observed with a highest inlet temperature and a higher maltodextrin concentration. This indicated that maltodextrin plays an important role in trapping anthocyanin, total phenols and antioxidant content in roselle powder.

The anthocyanin, total phenols and antioxidant activity increased as the inlet temperature and the maltodextrin concentration increased (Table-3). The highest anthocyanin (751.30 mg/100g), total phenols (41.97 mg GAE/100 g) and antioxidant activity (96.70%) was recorded in treatment T₉ which had an inlet temperature of 190°C and a maltodextrin concentration of 15 per cent. In contrast, the lowest anthocyanin (660.90 mg/100g), total phenols (35.23 mg GAE/100 g) and antioxidant activity (85.92%) was observed in treatment T₁ with an inlet temperature of 170°C and a maltodextrin concentration of five per

cent, both initially and throughout the storage period. This might be due to the faster drying process at higher temperatures, which caused shorter exposure time and thus lesser degradation of the heat-sensitive phenolic compounds (Demarchi *et al.*, 2013). In addition, it might be due to the polymerization as well as synthesis of polyphenols at higher drying temperature, which increased the TPC of the powder (Mishra *et al.*, 2014). Similar findings have been reported by Jafari *et al.* (2017) for pomegranate juice powder, Souza *et al.* (2014) in bordo grape, Silva *et al.* (2013) in spray dried jaboticaba peel extracts, Ahmed *et al.* (2010) in purple sweet potato pulp.

Conclusion

Roselle powder obtained at inlet temperature of 190°C and maltodextrin concentration of 15 per cent (T₉) was found to be good with respect to low moisture content (5.24%), water activity (0.24), while the maximum swelling capacity (21.47 ml/g), water absorption capacity (16.86 ml/g), anthocyanin (751.30 mg/100g), total phenols (41.97 mg GAE/100 g) and antioxidant activity (96.70%).

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