



RESEARCH ARTICLE

Physico-chemical properties of spray dried anthocyanin extract from hibiscus flowers (*Hibiscus rosasinensis* L.) as food colourant

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Abstract

Flowers are a rich source of bio-pigments like carotenoids, anthocyanins and betalains. Among these, anthocyanins are the most abundant pigments and *Hibiscus* flowers are one source of anthocyanin pigments. The major drawback in using hibiscus anthocyanins as a food colourant is their stability. Hence, to enhance stability, microencapsulation of aqueous extract of hibiscus petals was attempted with maltodextrin encapsulation at different TSS levels and temperatures and the physico-chemical and functional properties of spray dried hibiscus powder were assessed. Microencapsulation of anthocyanin extract with maltodextrin at 15° brix and 180 °C resulted in the highest dried powder recovery (17.85%), anthocyanin content (64.81 c3g eq.mg/L) and water solubility (99.58%). Physical properties like bulk density (0.593 g/m³) and tapped density (0.695 g/m³), was significantly higher in spray dried hibiscus powder obtained with maltodextrin 20 °brix at 170 °C. Based on Hausner's ratio (1.16), Carr index (14.04) and hygroscopicity (17.85%), hibiscus spray dried powder exhibited good flowability when dried with maltodextrin 15 °brix at 180 °C. Chromometer values of spray dried encapsulation with maltodextrin 15 °brix at 180 °C proved a deep red shade with more darkness (L*38.5) and (a* 23.50 and b* -0.67) when compared to other treatments. The spray dried powder also exhibited significant colour stability when used as food colourant in aonla juice, lemon juice, curd and butter cream. This study explored the potential of spray-dried anthocyanin extract from hibiscus flowers as a bio-colourant in the food industry

Keywords

anthocyanins; colour stability; colour values; food colourant; hibiscus; maltodextrin; microencapsulation; spray drying

Introduction

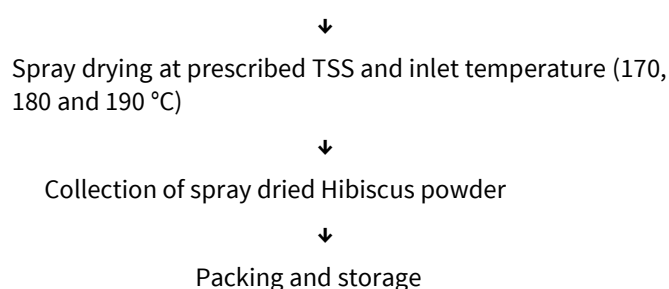
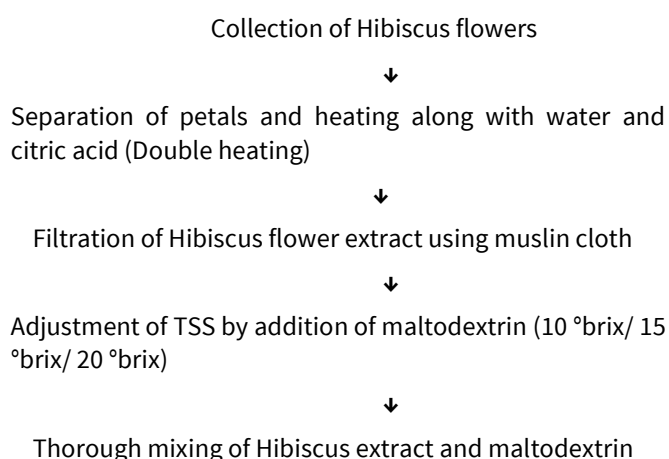
Hibiscus rosa-sinensis L., is considered as one of the most beautiful ornamental shrubs because of its vibrant showy colours, aesthetic value and significant medicinal properties in all parts of the plant. The leaves and flowers of hibiscus contain bioactive components such as glycosides, terpenoids, saponins and flavonoids and are therefore recommended as herbal alternatives to treat many diseases (1), including hypertension, high cholesterol and cancer. Fresh and dried petals of *Hibiscus* flowers also serve as potential sources of bio-pigment viz., anthocyanin. Anthocyanins serve as natural food colourants and contribute to health benefits as a rich source of antioxidants (2). The primary anthocyanin found in the red flowers of *Hibiscus rosa-sinensis* is cyanidin-3-sophoroside (3).

The use of anthocyanin pigments in food products is hindered by their poor stability, making their incorporation into foods challenging (4). Anthocyanin stability is affected by several factors, including pH, light, storage temperature, extraction solvent and the presence of flavonoids, enzymes, proteins and oxygen. Therefore, encapsulation is an efficient method to improve the stability of sensitive ingredients like anthocyanins in the food products. Encapsulating agents such as maltodextrin, gum arabic and starch are used to prevent pigment degradation from environmental factors such as light, temperature, moisture and oxygen. Among the different techniques used for improving stability, spray drying is typically employed for encapsulating phenolics such as anthocyanins. There is significant potential and a growing demand for natural bio-pigments derived from flowers. To explore the potential uses of hibiscus flowers as natural food colourant, the present study on physico-chemical properties of spray dried anthocyanin extract from Hibiscus flowers (*Hibiscus rosasinensis* L.) as food colourant was undertaken at the Department of Floriculture and Landscaping, Horticultural College and Research Institute, Tamil Nadu Agricultural University, Coimbatore, Tamil Nadu, India. The study aimed to develop a spray-dried formulation of hibiscus flowers encapsulated with maltodextrin and to assess its physico-chemical properties and suitability as a food colourant.

Materials and Methods

The study was conducted using Hibiscus flowers collected from germplasm at the Department of Floriculture and Landscape Architecture, Tamil Nadu Agricultural University, Coimbatore. To optimize the spray drying process for the production of powder from Hibiscus extract, parameters such as total soluble solids of extract (TSS), inlet hot air-drying temperature, outlet air temperature and feed rate in spray-drying were chosen based on information in literature (13, 14). The experiment was conducted under FCRD with nine treatments and 3 replications. The 9 treatments comprised of T₁ - Maltodextrin 10 °brix + 170 °C, T₂ - Maltodextrin 10 °brix + 180 °C, T₃ - Maltodextrin 10 °brix + 190 °C, T₄ - Maltodextrin 15 °brix + 170 °C, T₅ - Maltodextrin 15 °brix + 180 °C, T₆ - Maltodextrin 15 °brix + 190 °C, T₇ - Maltodextrin 20 °brix + 170 °C, T₈ - Maltodextrin 20 °brix + 180 °C and T₉ - Maltodextrin 20 °brix + 190 °C.

PROCESS FLOWCHART OF HIBISCUS SPRAY DRYING



The quantity of maltodextrin added was based on the final TSS of the extract adjusted to 10 °brix, 15 °brix and 20 °brix. A pilot model spray drier (M/s. GOMA Engineering private Ltd, Mumbai, India) with a capacity of 25 L available at the Center for Post Harvest Technology of Tamil Nadu Agricultural University, Coimbatore, was used to spray-dry the aqueous Hibiscus extract into powder. The spray-drier was turned on according to the operating method; the inlet temperature (170, 180 and 190 °C) and outlet temperature (80 °C) were set as needed and the extract was fed into the drying chamber at the feed rate of 25 mL/min. The spray-dried extract was collected as a fine powder in the outlet and packed in airtight containers and the observations were recorded.

The physicochemical properties of the spray-dried powder like powder recovery, bulk and tap density (5), and water solubility index (6), were assessed. The Carr Index (7) and Hausner ratio (8) were calculated from the bulk and tap densities of the powders. Further, the Carr index and Hausner ratio were assessed to study the flowability of the powder (9). The specifications of flowability ranging from excellent to very poor were based on Carr index and Hausner ratio (10). Water solubility index was determined by the method outlined by (6) and the hygroscopicity of the spray dried Hibiscus powder was determined based on the method described (11). The total monomeric anthocyanin content was measured using a spectrophotometric pH differential protocol given by (12) and the anthocyanin content was reported as cyanidin-3-glucoside equivalent in mg/L. The spray dried powders were visually observed and their corresponding colour values were recorded using 3 nh chromometer (3 nh is the series of chromometers used to measure the colour and illuminance of products).

To assess the colour stability, the spray dried Hibiscus powder obtained from the best treatment (maltodextrin 15 °brix + 180 °C) was used as food colorant in various processed products. Measured quantity of spray dried powder (based on preference of colour) was added to commercially available processed products as given below,

The colour value obtained and the consumer acceptance of processed products coloured with Hibiscus anthocyanin colorant were assessed based on organoleptic scoring. The results obtained from all the above experiments were carried out in triplicates and the values are expressed in Mean ± SD. The critical difference was worked out at 5% (0.05) probability level. The data recorded were subjected to statistical analysis with AGRES software package and MS Excel® spreadsheet.

Sl. No.	Products	Quantity of spray dried Hibiscus powder added (g)
1	Lemon juice	200 mL
2	Aonla juice	200 mL
3	Butter cream	100 g
4	Curd	160 g
5	Milk shake	180 mL

Results and discussion

Powder recovery

Powder recovery was obtained by the effect of carrier agent concentration and inlet air temperature of spray dried powders. The highest recovery of 17.85% was obtained by the encapsulation of hibiscus extract with maltodextrin 15 °brix + 180 °C (Table 1). This finding indicates that higher inlet air temperatures resulted in higher recovery through the increased efficiency of heat and mass transfer processes when the extracts were dried at higher inlet temperatures. This result is in agreement with the findings (13) and (14). The lowest powder recovery of 6.69% was obtained in the treatment maltodextrin 10 °brix + 170 °C which might be due to the presence of increased amount of mucilage content with decreased amount of maltodextrin concentration in the feed solution. The extract with the TSS of 20 °brix showed a tendency to stick onto the internal wall surfaces of drying chamber due to the presence of higher amount total solids and sugars present in the feed solution that resulted in minimum powder yield. Similar results have been reported (15-17) in the powders of *Hibiscus sabdariffa*, raisin juice and black mulberry juice respectively. Moreover, the higher inlet air temperature of 190 °C resulted in the least powder recovery.

Water solubility

Spray dried powders are intended for dehydration and the ideal qualities of powder are quick and thorough wetting along with capacity to dissolve/disperse without lumps (18). The water solubility index of hibiscus extract spray dried with maltodextrin 15 °brix at 180 °C was nearly 100% (Table 1). This indicated that the solubility of powders increased with increasing inlet air temperatures. Similar results were reported (19, 20) for spray dried ginger juice and green banana starch. Moreover, the solubility of powders increased with simultaneous increase in the concentration of maltodextrin. These results are in accordance with the findings of (21) in which the solubility index for spray dried roselle (*Hibiscus sabdariffa*) powder was 99.22%.

Bulk density and tapped density

The bulk density and tap density of spray-dried hibiscus powder ranged from 0.516 to 0.554 g/m³ and 0.619 to 0.627 g/m³ in 10 °brix, 0.518 to 0.592 g/m³ and 0.634 to 0.677 g/m³ in 15 °brix and 0.526 to 0.593 g/m³ and 0.652 to 0.695 g/m³ in 20 °brix of maltodextrin at 170 °C, 180 °C and 180 °C respectively (Table 1; Fig. 1). Bulk density and tap density were the most critical factors in spray dried powders that determine the storage, transport and packaging considerations (22). It was observed that the increase in inlet air temperature resulted in lower bulk density due to faster evaporation rates by feed solution. Hence the powder tends to become porous and fragmented by reducing the bulk density. Further, with an increase in the inlet temperature, it becomes hollow due to the particle inflation-ballooning with decrease in bulk density (23). The moisture content of the powder was altered due to the effect of inlet air temperature (5). Increase in inlet air temperature (180 °C and 190 °C) resulted in the decreased bulk and tap densities thereby attributing to the rapid moisture removal in the powders or air trapped in the particles.

Hausner's ratio (HR) and Carr index (CI)

The flowability characters of powders are decided by the values of Carr index and Hausner's ratio. In the experiment, the Carr index values ranged from 11.64 to 19.32 and the Hausner's values of spray dried Hibiscus powder ranged from 1.13 to 1.23. The lowest Carr index and Hausner's values exhibited better flowability whereas, highest CI and HR values exhibited poor flowability. From the results, it was observed that Hibiscus spray dried powder exhibited good flowability when dried at the inlet air temperature of 170 °C and 180 °C and exhibited fair flowability at the temperature of 190 °C (Table 1; Fig. 1). HR and CI values of the powder increased with an increase in inlet air temperature and maltodextrin concentration. Similar results were reported in pink guava powder with Hausner's ratio of 1.18 and exhibited good flowability at concentration of 15% maltodextrin. Similar findings were also reported in elderberry, blackberry and raspberry powders with Carr index values of 5.89, 15.35 and 18.45 respectively along with Hausner's ratio of 1.08, 1.15 and 1.23 which exhibited excellent and fair flowability characters (24). On the other hand, Carr index and Hausner's ratio increased with decrease in particle size, and it was reported that, reduced particle size exhibited poor flowability (9). According to (25), HR values were low

Table 1. Physico-chemical properties of spray dried Hibiscus powder

Treatment details	Powder recovery (%)	Water solubility (%)	Hygroscopicity (%)	Bulk density (g/m ³)	Tap density (g/m ³)	Hausner's ratio	Carr index (%)
T ₁ - Maltodextrin 10 °brix + 170 °C	6.69 ± 0.04	96.25 ± 0.02	16.45 ± 0.45	0.554 ± 0.01	0.627 ± 0.01	1.13 ± 0.03	11.64 ± 0.24
T ₂ - Maltodextrin 10 °brix + 180 °C	9.87 ± 0.07	96.67 ± 3.64	17.72 ± 0.51	0.543 ± 0.01	0.625 ± 0.01	1.15 ± 0.03	13.12 ± 0.18
T ₃ - Maltodextrin 10 °brix + 190 °C	7.36 ± 0.21	97.09 ± 1.95	19.12 ± 0.61	0.516 ± 0.02	0.619 ± 0.02	1.19 ± 0.04	16.63 ± 0.65
T ₄ - Maltodextrin 15 °brix + 170 °C	15.88 ± 0.65	97.34 ± 3.81	17.33 ± 0.35	0.592 ± 0.02	0.677 ± 0.02	1.14 ± 0.03	12.55 ± 0.26
T ₅ - Maltodextrin 15 °brix + 180 °C	17.85 ± 0.77	99.58 ± 0.44	17.85 ± 0.40	0.557 ± 0.02	0.648 ± 0.01	1.16 ± 0.04	14.04 ± 0.29
T ₆ - Maltodextrin 15 °brix + 190 °C	14.45 ± 0.37	98.15 ± 2.12	19.18 ± 0.79	0.518 ± 0.01	0.634 ± 0.02	1.12 ± 0.01	18.29 ± 0.37
T ₇ - Maltodextrin 20 °brix + 170 °C	12.87 ± 0.35	98.49 ± 2.56	17.68 ± 0.38	0.593 ± 0.02	0.695 ± 0.02	1.17 ± 0.02	14.67 ± 0.08
T ₈ - Maltodextrin 20 °brix + 180 °C	14.16 ± 0.49	99.13 ± 3.03	18.10 ± 0.11	0.580 ± 0.01	0.685 ± 0.01	1.18 ± 0.01	15.32 ± 0.05
T ₉ - Maltodextrin 20 °brix + 190 °C	10.92 ± 0.36	98.80 ± 3.82	19.55 ± 0.22	0.526 ± 0.01	0.652 ± 0.01	1.23 ± 0.03	19.32 ± 0.08
Mean	12.22	97.94	18.10	0.553	0.651	1.16	15.06
S.Ed	0.167	3.254	0.249	0.016	0.014	0.022	0.309
CD	0.353**	2.026**	0.528**	0.034**	0.031**	0.046**	0.653**

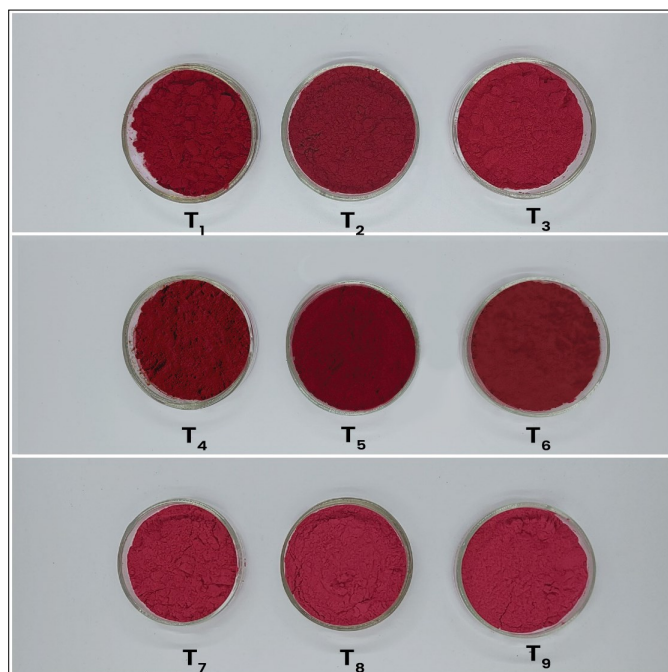


Fig. 1. Spray dried hibiscus powder encapsulated with maltodextrin at different TSS levels.

(<1.2) in soymilk spray dried powders and CI value was significantly good (<15%) in jamun fruit powder as reported (26).

Hygroscopicity (%)

Hygroscopicity is the process that attracts water molecules from the surrounding environment through absorption or adsorption. The hygroscopicity value of the hibiscus spray dried powders ranged from 16.45 to 19.12% in 10 °brix, 17.33 to 19.18% in 15 °brix and 17.68 to 19.55% in 20 °brix of maltodextrin at 170 °C, 180 °C and 190 °C respectively (Table 1). It was indicated that the highest concentration of maltodextrin resulted in the lowest hygroscopicity value. Similarly, the lowest hygroscopicity values were obtained when the inlet air temperature decreased. In order to minimize hygroscopicity, food additives like maltodextrin are encapsulated with the extracts (27, 28) and also to get higher powder recovery. Even though the hygroscopicity values were reduced due to the addition of maltodextrin, higher amount of maltodextrin led to higher feed viscosity, larger droplets of extracts during drying thereby resulting in larger particle size (25). The highest hygroscopicity values were obtained at 20 DE (dextrose equivalent) and 25 DE maltodextrin of amaranthus spray dried powder (11). In addition, the lowest hygroscopicity values were observed with decrease in inlet air temperature. Similar results were

reported (29) in spray dried fruit of *Euterpe oleracea* Mart. Palm (acai) powder. In the experiment, hygroscopicity values ranged from 16.45 to 19.55% with the exhibition of more hygroscopy in the powder thereby attracting more moisture from the environment with the formation of clumps. Similar results were reported in spray dried *Hibiscus sabdariffa* (21) with hygroscopicity values ranged between 10.84 and 15.42 and in acai spray dried powder (29).

Anthocyanin content in spray dried powder

Anthocyanins pigments are potential natural colourants due to their low toxicity (30). The anthocyanin content of the spray dried Hibiscus powder ranged from 54.16 to 64.81 mg/L among the treatments (Table 2). Increased inlet air temperature during spray drying leads to the degradation of the anthocyanin pigments. This might be due to higher inlet temperature and occluded air which make the powder more porous thereby increasing the oxidative degradation and reducing the storage stability (31). Additionally, the degradation of the anthocyanin content of the powder was minimized by the inclusion of maltodextrin as an encapsulating agent thereby protecting the colour and stability of the powders (11) and (34). While the addition of maltodextrin reduces anthocyanin degradation, higher amount of maltodextrin leads to colour fading during drying. Similar results were reported (32) in beetroot juice powder. In the present study, hibiscus extract spray dried at maltodextrin 15 °brix at 180 °C resulted in highest anthocyanin content. This is in accordance with the results reported (11, 29, 33) in spray dried amaranthus powder, acai powder and bayberry powder.

Colour values of the powder

The colour values of spray dried powder were assessed using 3 nh chromometer. L*, a*, b* are indicators of the CIE L*A*B* color space indicators as defined by the International Commission on Illumination. L* measures the degree of lightness and darkness of the sample (lower value=darkness; higher value=lightness). On the other hand, the shift from negative to positive values was indicated by colour change from bluish green to purplish red in a* value and from blue to yellow in b* value. Among all, spray dried powder obtained at 15 °brix showed the lowest L* value and highest a* and b* values indicating high degree of darkness along with shift towards purplish red to blue colour of the sample (Table 2; Fig. 1).

Table 2. Anthocyanin content and colour values of spray dried Hibiscus powder

Treatment details	Anthocyanin content (c3 g eq.mg/L)	Colour values		
		L*	a*	b*
T ₁ - Maltodextrin 10 °brix + 170 °C	61.82 ± 2.05	40.11	20.03	-0.55
T ₂ - Maltodextrin 10 °brix + 180 °C	63.19 ± 0.45	39.34	22.75	-0.63
T ₃ - Maltodextrin 10 °brix + 190 °C	56.23 ± 1.97	42.58	18.68	-0.45
T ₄ - Maltodextrin 15 °brix + 170 °C	62.34 ± 1.96	40.25	21.67	-0.59
T ₅ - Maltodextrin 15 °brix + 180 °C	64.81 ± 0.52	38.5	23.50	-0.67
T ₆ - Maltodextrin 15 °brix + 190 °C	58.45 ± 0.84	41.56	19.7	-0.56
T ₇ - Maltodextrin 20 °brix + 170 °C	57.01 ± 2.00	42.05	17.56	-0.50
T ₈ - Maltodextrin 20 °brix + 180 °C	59.87 ± 1.24	41.36	19.52	-0.50
T ₉ - Maltodextrin 20 °brix + 190 °C	54.16 ± 0.48	44.23	16.09	-0.43
Mean	59.76	41.10	19.94	-0.542
S.Ed	1.266	0.972	0.497	0.008
CD	2.660**	2.043**	1.044**	0.018**

The present study proved that at decreased inlet air temperature, dark coloured powder was obtained with least L* values. Similar results of dark coloured spray dried powders were obtained in tomato (35) and passion fruit (36) at least inlet air temperatures. On the contrary, increased concentration of maltodextrin at 20 °brix resulted in lighter colour powders when compared to 10 °brix and 15 °brix.

Evaluation of colour stability of spray dried hibiscus powder as food colorant

In this experiment, the colour stability and consumer acceptance of spray-dried Hibiscus powder were assessed as a natural food colourant in lemon juice, aonla juice, curd, cream and milkshake (Table 3; Fig. 2). Anthocyanin stability is higher in acidic conditions and lower in alkaline conditions (37). Among the products, spray-dried powder added to lemon juice and aonla juice exhibited deep red colour due acidic nature of juices. The organoleptic score of lemon juice and aonla juice was 5.0 (high acceptance), indicating a strong consumer preference for coloured juices. Similarly, curd and butter cream with a pH range of 3.5 to 4.5 (acceptance) exhibited a pink colour due to the moderate stability of the pigment, and had a consumer acceptability score of 4.5. Furthermore, the spray-dried powder added to the milkshake exhibited light purple colour due to high pH range of 6.5 to 7.2, with an organoleptic score of 3.6 (moderate acceptance).

Conclusion

The present study concluded that Hibiscus spray-dried powder obtained at 180 °C with 15° brix maltodextrin resulted in the highest powder recovery, improved hygroscopicity,

solubility, anthocyanin content and colour values. The inlet air temperature and the concentration of encapsulating agent play a significant role in physiochemical properties of spray-dried powder. The spray-dried powder also exhibited high consumer acceptability when used as food colourant in aonla juice, lemon juice, curd and butter cream.

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Authors' contributions

AR conducted research experiments, recorded observations and analyzed data. SPT provided guidance in formulating the technical program, interpreting data and documenting research work. SK conducted confirmatory trials under the AICRP program and interpreted the data. MG reviewed documentation work and performed technical editing. SPM assisted in statistical analysis, data tabulation and documentation work. MV assisted in conducting confirmatory trials and data documentation works.

Compliance with ethical standards

Conflict of interest: Authors do not have any conflict of interest to declare.

Ethical issues: None

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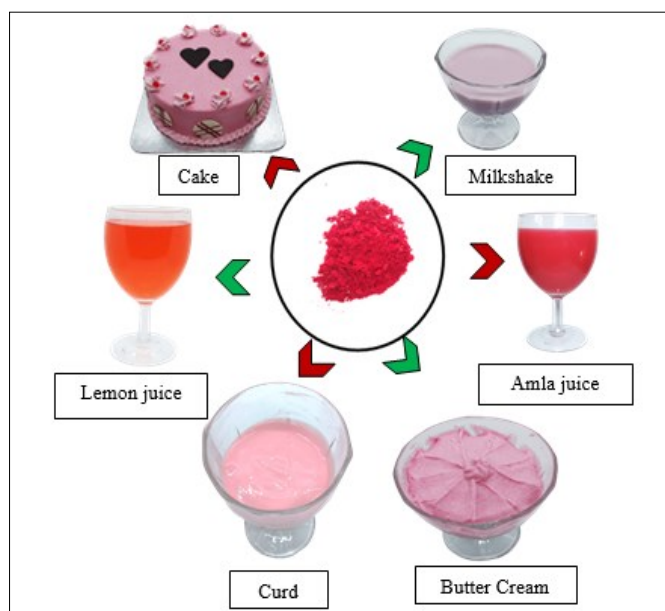


Fig. 2. Evaluation of spray dried hibiscus powder as food colorant.

Table 3. Organoleptic scoring of the product coloured with hibiscus powder

Sl. No.	Products	Colour obtained	Colour	Flavour	Taste	Overall Acceptability	Mean
1.	Lemon juice	Deep red	5.0	4.8	4.6	4.7	4.7
2.	Aonla juice	Deep red	5.0	4.4	4.5	4.4	4.5
3.	Butter cream	Pink	4.4	3.9	4.5	4.6	4.5
4.	Curd	Pink	4.2	4.6	4.5	4.5	4.5
5.	Milkshake	Light Purple	3.6	3.7	3.9	3.6	3.7

(Organoleptic scores: 4.0 to 5.0 - Highly acceptable; 3.0 to 4.0 - Acceptable; 2.0 to 3.0 - Moderately acceptable; 1.0 to 2.0 - least acceptable; 0 to 1.0 - Not acceptable)

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