Physical properties of microencapsulated anthocyanin obtained by spray drying of Red *Amaranthus* extract with maltodextrin

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ABSTRACT

Introduction: Anthocyanins are water-soluble plant pigments responsible for bright red, purple and blue colours in fruits and vegetables. Extraction of anthocyanins from plant cells becomes an important task closely related to the need of preservation of their bioactivity. Therefore, encapsulation by spray drying is a technique used to retain maximum anthocyanin and colour. Methods: The study was designed to investigate the physical properties of spray drying of Red Amaranthus extract with maltodextrin. The extract was prepared by soaking washed and finely chopped red spinach (Red Amaranthus) leaves in water for 72 h at 4°C of with potable water (solvent), followed by storage at refrigerated condition at 4°C for 72 h. The extract was mixed with maltodextrin solution in three different ratios: 1:1, 1:2 and 1:3. Powder properties were studied for the three different extract and maltodextrin ratios that were fed in the spray dryer. All analyses were conducted in triplicates. **Results**: The 1:1 ratio retains maximum anthocyanin 93.03 mg/100g of spray dried powder compared to the other ratios; the moisture content of the 1:1 ratio was 0.44% (dry basis). The color chroma value of a* of 1:1 ratio was 26.24 and density was 0.55 g/cm^3 whereas the water activity of the sample was 0.62±0.01. By comparing the three ratios, the 1:1 ratio of extract and maltodextrin was the optimum combination for encapsulation of anthocyanin using spray-drying technique. Conclusion: Encapsulation with maltodextrin at 1:1 ratio had a significant impact on retention of anthocyanin and colour in the final product.

Keywords: Red Amaranthus, maltodextrin, spray drying, anthocyanin, colour

INTRODUCTION

Microencapsulation is a process in which tiny particles or droplets are surrounded by a coating or embedded in a homogeneous or heterogeneous matrix to give small capsules with several beneficial properties. Spray drying method is used widely as a microencapsulation technology, employed to produce commercially engineered powders from liquid feed in a single step (Bazaria & Kumar, 2016). It offers short contact times at relatively low temperatures, allowing the properties of foods such as colour, flavour, and nutrients to be retained in considerable percentages. The powders obtained by spray-drying are amorphous materials, susceptible to glass-transition related changes including stickiness,

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caking, and collapse as well as colour changes leading to low product yield operational problems. and Surface modification of droplets with protein is a novel way to minimise stickiness in sprays dried powder. The elementary operational conditions of spray drying namely temperature of drving air and feed flow rate of feed are crucial in explaining the quality characteristics of product. The most conventional carrier agents used in spray drying is maltodextrin mainly due to its high solubility and low viscosity (Mahdavi et al., 2016). This carrier agent has a high molecular weight and is also useful for increasing the product's glass transition temperature, aiming at avoiding spray drving operational problems such as sticking to the chamber wall, as well as structural transformations during food processing and storage (Tonon et al., 2008).

Fruit juice powders have various benefits and economic potentials over their liquid counterparts such as reduced weight, reduced packaging, easier handling and transportation, and extended shelf life. Their physical state provides a stable and natural ingredient which generally finds usage in many foods products such as flavouring and colouring agents (Shrestha et al., 2007). Freeze drying is the best way to dry sensitive plant pigments. However, spray drying, if feasible, would be a more applied and cost-effective method of producing powdered sensitive colorants as the processing cost is 30-50 times less than for freeze drying (Cai & Corke, 2000).

Research studies have shown that anthocyanins are unstable and are easily oxidized under various conditions such as pH, temperature, enzymes resulting in colour change and degradation (Santos & Williamson, 2003). Extracted plant pigment (anthocyanin) is good for health since they have numerous beneficial effects associated with their antimutagenic and antioxidant properties (Edenharder *et al.*, 2002). Many studies showed that red colour juices such as those of pomegranates, grapes, and different berries have beneficial effects on human health due to their high anthocyanin content and antioxidant activity (Lin & Tang, 2007).

There is a swelling interest to minimise the use of synthetic colours used in the food industries on a large scale and to replace it with natural pigments obtained from fruits and vegetables (Cai *et al.*, 2000).

Red spinach is one of the common vegetables cultivated around leafy the world and it comes under the Amaranthaceae family. It grows well in warm climates and well aerated sandy soil. It has red leaves and bright red stem. Red spinach is rich in vitamin A, C, iron, calcium, phosphorous, sodium, potassium and other essential amino acids. It helps in digestion and is a rich source of fibre. The role of anthocyanin as a food-colouring agent is established in the food industry. However, stability is an important aspect to consider for the use of these pigments as a colourant in food products. Based on the above reasons, this study is undertaken to investigate the feasibility of spray drying of Red Amaranthus extract and to evaluate the physical properties of the powder produced, including the content of anthocyanin, moisture, water activity, colour and density analysis.

MATERIALS AND METHODS

Processing of sample

Fresh bunches of Red *Amaranthus* (red spinach) were collected from the local market in Chennai. The samples were cleaned thoroughly to remove excess soil and debris. The non-edible part was separated and the leaves were used for further experimental analysis.

Preparation of extract

Potable water was used as a solvent to carry out different extraction methods to obtain maximum extract from the sample. The extraction was done using mortar and pestle (Sivasankar et al., 2011), magnetic stirrer (Tomsone et al., 2012) and soaking the samples in water refrigerated at 4°C (Davang Norulfairuz et al., 2014). 10 g of fresh leaves were treated with varying volumes of potable water in the ratio of 1:1, 1:2, 1:3 and a contact time of 5, 10, 15 minutes. The mixture was macerated using a mortar and pestle and was centrifuged at 10,000 rpm for 10 min. For extraction using magnetic stirrer, 10 g of fresh leaves were added to varying volumes of potable water in the ratio of 1:1, 1:2, 1:3 and extraction was done at 700 rpm for 30, 60 and 90 minutes at room temperature.

Preparation of feed suspension for spray drying

The extract was prepared by soaking the washed and finely chopped red spinach leaves in water for 72 h at 4°C. The extract was collected using a muslin cloth and filtered before mixing with maltodextrin. For each run, three different ratios (1:1, 1:2 and 1:3) of the extract and wall material (maltodextrin) were used. The extract was fed into the spray drier with an inlet temperature of 110°C and outlet temperature varying between 65-75°C.

Experimental design

The extract was prepared by soaking the washed and finely chopped red spinach (Red *Amaranthus*) leaves in water for 72 h at 4°C of with potable water (solvent) and stored at refrigerated condition at 4°C for 72 h. The extract obtained from Red *Amaranthus* was mixed with the solution of maltodextrin in three different ratios: 1:1, 1:2 and 1:3 was carried out. The extract was fed into the spray drier

with an inlet temperature maintained at 110°C and outlet temperature varying between 65°C-75°C. The physical properties of the obtained powder were determined and all the analysis were done in triplicates and reported as mean±standard deviation.

Analysis of the spray-dried powder

The spray-dried powders were analysed for their total anthocyanin content (TAC), moisture content, water activity, colour and density parameters.

Total Anthocyanin Content (TAC)

TAC was quantified in accordance to the method described by Sutharut *et al.*, (2012). 1 ml extracted solution was transferred into 10 ml volumetric flask for preparing two dilutions of the sample, one volume adjusted with potassium chloride buffer pH 1.0 and the other with sodium acetate buffer pH 4.5. These dilutions were equilibrated for 15 min. The absorbance of each dilution was measured at 510 nm and 700 nm against a blank cell filled with distilled water.

Moisture content

The moisture content was determined based on Association of Analytical Communities (AOAC) method. Triplicate samples of red spinach powder (1 g each) were weighed and then dried in a hot air oven at 105°C. The drying was continued until a constant weight was obtained and moisture loss was expressed in terms of percent dry basis (d.b).

Water activity

Water activity was measured using the water activity meter. The average of the triplicate values was recorded (Lab Touch, Novasina).

Colour determination

The colorimeter is used to obtain the values of the spray dried Red *Amaranthus*

powder. The results were obtained as L^* , a^* , b^* values which determine the different colour range and brightness, darkness of the sample. The L^* value determines the brightness (white at 100) to darkness (black at 0), while a^* measures green when negative and red when positive. Similarly, b^* measures yellow when positive and blue when negative. Calibration was done using the white tile prior to the sample analysis. Colour analysis was performed using Hunter Colorimeter (ColorQuest XE) in triplicates.

Density analysis

The parameters were determined as per Chang *et al.* (2012). Bulk density: 2 g of sample was added to a pre-weighed volumetric cylinder and the volume was read as V1. Tapped density: 2 g of sample was placed into a measuring cylinder and tapped until a consistent volume (V2) is reached which corresponds to the maximum packing density of the material. By measuring bulk and tapped volume, the following parameters were determined.

Statistical analysis

Data were expressed as mean±standard deviation of three replications and statistical analysis (ANOVA) was done using Minitab 17. The values were considered to be significant with p<0.05.

RESULTS AND DISCUSSION

Total Anthocyanin Content

Anthocyanins are very sensitive compounds and unstable during processing and storage. The internal properties such as pН, chemical structure, and anthocyanin concentration of the product, available enzymes and processing conditions also play а role in their stability. The initial total anthocyanin content of fresh leaves is 169.22 mg/100 g fresh weight and

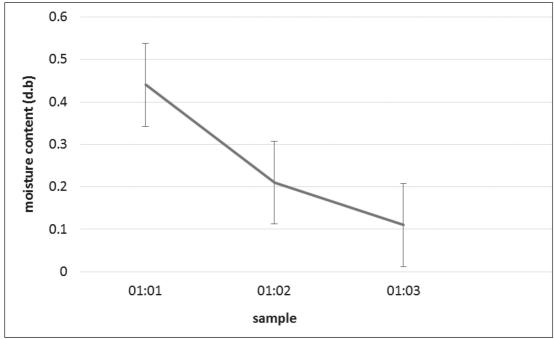
whereas the total anthocyanin content the final product ranged from in 43.11-98.03 mg/100 g sample weight. From Figure 1 it was observed that raising maltodextrin levels reduced anthocyanin rate of the product (extract: maltodextrin). Khazaeia et al. (2014) reported this decrease too, stating that in these cases, the juice was not really encapsulated and the carrier agent acted merely as an aid for facilitating the drying process, which is probably a reason for the lower anthocyanin content when this agent was used. The same phenomenon was reported by Tonon et al. (2008) who used different carrier agents including maltodextrin for spray drying of acai juice.

Moisture content

Moisture in powder plays a significant role in determining its flowability, stickiness and storage stability due to its effect on glass transition and crystallisation behaviour. The moisture content of spray dried red spinach extract was done with triplicate samples and ranged from 0.11% to 0.44% (on dry basis). The results (Figure 2) show that as the amount of maltodextrin increases, there is a decrease in the moisture content. In a spray drying system, the water content of the feed influences the final moisture content of the powder produced. The results show similarity to the study conducted by Quek et al., (2007), where addition of maltodextrin to the feed prior to spray drying increased the total solid content and also increases the amount of water for evaporation. Hence, decreased the moisture contents of the powder produced. Powders with lower moisture content could be obtained by increasing the amount of maltodextrin in the feed.

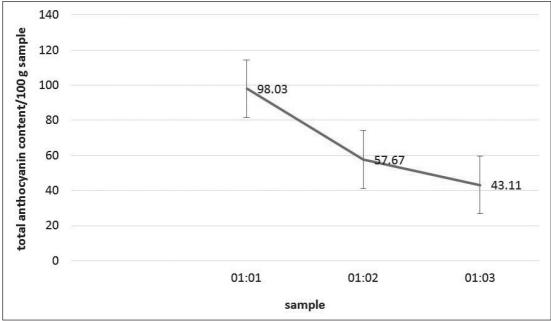
Water activity

Water activity (a_w) is an important parameter for spray-dried powder because it can significantly affect the



Data represent the mean±standard deviation of triplicate readings.

Figure 1. Total anthocyanin content of the spray dried powder with varying volume of maltodextrin (extract: maltodextrin)



Data represent the mean±standard deviation of triplicate readings.

Figure 2. Moisture content of spray dried Red amaranthus extract with maltodextrin

Extract : maltodextrin	Water activity, a_w	Temperature	
1:1	0.629±0.01	31.3°C	
1:2	0.605±0.01	31.4°C	
1:3	0.586±0.01	32.1°C	

Table 1. Water activity of spray dried powder obtained from different ratios of extract and maltodextrin

Data represent the mean±standard deviation of triplicate readings.

shelf life of the powder produced. Water activity is different from moisture content as it measures the availability of free water in a food system that is accountable for biochemical reactions, whereas the moisture content represents the water composition in a food system. High water activity indicates more free water available for biochemical reactions and results in a shorter shelf life. Generally, food with a_w <0.6 is considered as microbiologically stable and if there is any spoilage it is attributed to chemical reactions rather than by micro-organism.

From Table 1, the water activities of the obtained powders were in the range of 0.5–0.6. This shows that the spray-dried powders produced were relatively stable microbiologically. However, the storage conditions also played an important role in this matter. As the spray-dried powder contains maltodextrin, they are highly hygroscopic and should be stored properly in air-tight containers and kept in a cool dry place. the lightness of the sample and $+a^*$ measures the red colour. The a* value ranged from 14-26 and the maximum value was observed in 1:1 spray dried sample as increased quantities of maltodextrin reduced the colour intensity of a^* that correlates with the total anthocyanin present in the final product. The positive correlation with the a* value and anthocyanin pigment in the present study is in correlation with the experiments carried out using beet juice by Bazaria *et al.* (2016).

Density analysis

Bulk density is a measure of the heaviness of powder and an important parameter that determines the suitability of powder for the ease of packaging and transportation. The bulk density of the spray dried powder of different ratios was found to be in the range of 0.3-0.5 g/cm³. Higher maltodextrin reduced density of the final product, probably due to a decrease in its moisture content or

Extract : maltodextrin	L^*	a*
1:1	53.75	26.24
1:2	72.65	22.38
1:3	81.17	14.88

Table 2. Colour analysis of the spray dried samples

Triplicate samples were analysed and the mean was recorded.

Colour intensity

The results of the colour measurement for the spray dried powder are shown in Table 2. The L^* value measures the higher air trapped in the particles as maltodextrin is a skin forming material (Fazaeli *et al.*, 2012). This indicates that maltodextrin as a coating agent

Extract: maltodextrin	Bulk density (g/ cm³)	Tapped density (g/cm³)	Hausner ratio	Carr's index %
1:1	0.553±0.03	0.680±0.03	1.22±0.03	18.67±0.04
1:2	0.353±0.02	0.421±0.03	1.19±0.03	16.15±0.02
1:3	0.294±0.04	0.368±0.04	1.25±0.03	20.10±0.05

Table 3. Density analysis of three different ratios of the extract with maltodextrin

Data represent the mean±standard deviation of triplicate readings.

could reduce the hygroscopicity of the anthocyanin. Comparable results were observed by Goula and Adamopoulos (2005) and Abadio *et al.* (2004) when tomato and pineapple pulp were dried using maltodextrin as the carrier in a spray dryer.

The flowability of the dried powder is determined by the Hausner ratio and Carr index. Carr index shows the flowability index of the dried food product as given in the Table 3, 5-15% corresponds to excellent flowability index and poor flowability, if the value is less than 25%. From the result, it can be concluded that the powder has poor flowability property.

CONCLUSION

Different ratios of extract and maltodextrin were used to study the quality parameters of Red Amaranthus powder. The models for powder physical and functional properties were statistically significant (p < 0.05) where total anthocyanin content and a* values were considered as important parameters for incorporation into functional foods as a red colorant replacing the use of synthetic food colour used commercially. From this study, it can be concluded that the optimised combination includes equal parts of the wall material, in this case, maltodextrin and extract. The 1:1 ratio retains maximum anthocyanin 93.03 mg/100 g fresh weight, a* value of 26.24 and density of 0.55 g/cm³.

Increased quantities of maltodextrin reduced the total anthocyanin and the moisture content present in the final product. The results of density analysis show that all ratios of the spray dried powder can be considered as a medium flowing powder in accordance to the Hausner ratio. This study demonstrated the feasibility of production of spraydried Red *Amaranthus* extracts as a food grade colorant.

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Authors' contributions

Meenakshi N, carried out the research as a main author, all the experiments are carried out by this author; Sasikala Shanmugam, guided the experiment work carried out in this work; Pavithra MS, assisted in analytical works carried out in this work.

References

- Abadio FDB, Domingues AM, Borges SV & Oliveira (2004). Physical properties of powdered pineapple (*Ananascomosus*) juice-effect of malt dextrin concentration and atomization speed. *J. Food Eng.* 64(3):285-287.
- Akhavan Mahdavi S, Jafari SM, Assadpoor E & Dehnad D (2016). Microencapsulation optimization of natural anthocyanins with maltodextrin, gum Arabic and gelatin. Int. J. Biol. Macromol. 85:379-385.

- Al-Hakim K & Stapley AGF (2004). Morphology of spray-dried and spray freeze-dried whey powders. Proceedings of the 14th International Drying Symposium. (pp. 1720-1726). State University of Campinas, Sao Paulo, Brazil.
- Athanasia M, Goula, Konstantinos G & Adamopoulos (2010). A new technique for spray drying orange juice concentrate. *Innov.* Food Sci. & Emerg. Technol. 11:342-351.
- Bernard, Regnault, Gendreau, Charbonneau S & Relkin P. (2011). Enhancement of emulsifying properties of whey proteins by controlling spray- drying parameters. *Food Hydrocoll.* 25, 758-763.
- Bhusari SN, Muzaffar K, & Kumar P (2014). Effect of carrier agents on physical and microstructural properties of spray dried tamarind pulp powder. *Powder Technol.* 266:354–364.
- Bindu B & Pradyuman K (2016). Effect of whey protein concentrate as drying aid and drying parameters on physicochemical and functional properties of spray dried beetroot juice concentrate. *Food Biosci.* 14(2016):21-27.
- Cai YZ & Corke H (2000). Production and properties of spray-dried Amaranthus betacyanin pigments. J. Food Sci. 65:1248–1252.
- Cano-Chauca M, Stringheta PC, Ramos AM & Cal-Vidal J (2005) Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. *Innov. Food Sci. & Emerg. Technol.* 6(4):420-428.
- Chang CH, Lin HY, Chang CY & Liu YC (2006). Comparisons on the antioxidant properties of fresh, freeze dried and hot air dried tomatoes. *J. Food Eng.* 77:478-485.
- Chegini GR & Ghobadian B (2005). Effect of spraydrying condition on physical properties of orange juice powder. *Drying Technol.* 23:657-668.
- Dayang Norulfairuz, Abang Zaidel, Nur Shakira S, Yanti Maslina MJ & Ida Idayu M (2014). Encapsulation of Anthocyanin from Roselle and Red Cabbage for Stabilization of Water-in-Oil Emulsion. *Agric. Agric. Sci. Procedia* 2:82-89.
- Edenharder RJW, Sagr H, Glatt E & Muckel PKL (2002). Protection by beverages, fruits, vegetables, herbs and flavonoids against genotoxicity of 2-acetylaminofluorene and2 amino-1-methyl-6-phenylimodazole(4,5-b) pyridine (PhIP) in metabolically competent V79 cells. *Mutat. Res.* 521:57-72.

- Hongxiang F & Bhandari (2012). Comparing the efficiency of protein and maltodextrin on spray drying of bayberry juice. *Food Res. Int.* 48:478-483.
- León-Martínez FM, Méndez-Lagunas LL & Rodríguez-Ramírez J (2010). Spray drying of nopal mucilage (Opuntiaficus-indica): Effects on powder properties and characterization. *Carbohydr. Polym.* 81:864-870.
- Goula AM, Konstantinos G, & Adamopoulos G (2005). Spray drying of tomato pulp in dehumidifed air: I. The effect on powder recovery. J. Food Eng. 66:25-34.
- Khazaei KM, Jafari SM, Ghorbani M & Hemmati Kakhki A (2014). Application of maltodextrin and gum Arabic in microencapsulation of saffron petal's anthocyanins and evaluating their storage stability and color. *Carbohydr. Polym.* 105:57-62.
- Lin JY & Tang CY (2007). Determination of total phenolic and flavonoid contents in selected fruits and vegetables as well as their stimulatory effects on mouse splenocyte proliferation. *Food Chem.* 101:140-147.
- Pitalua E, Jimenez M, Vernon-Carter E & Beristain C (2010). Antioxidative activity of microcapsules with beetroot juice using gum arabic as wall material. *Food Bioprod. Process.* 88:253-258.
- Lolita T, Zanda & Ruta (2012). Comparison of Different Solvents and Extraction Methods for Isolation of Phenolic Compounds from Horseradish Roots (Armoracia rusticana). International Journal of Biological, Biomolecular, Agricultural, Food and Biotechnological Engineering Vol:6, No:4.
- Fazaeli M, Emam-Djomeh Z, Kalbasi KA & Omid M (2012). Effect of spray drying conditions and feed composition on the physical properties of black mulberry juice powder. *Food Bioprod. Process.* 90:667-675.
- Quek SY, Chok NK & Swedlund P. (2007). The physicochemical properties of spray-dried watermelon powders. *Chem. Eng. Process.* 46:386-392.
- Santos-Buelga C & Williamson G (2003). Methods in polyphenol analysis. Cambridge: The Royal Society of Chemistry. J. Nat. Prod. 67:1077– 1078
- Sutharut J & Sudarat J (2012). Total anthocyanin content and antioxidant activity of germinated colored rice. *Int. Food Res. J.* 19(1):215-221.

- Shrestha AK, Ua-arak T, Adhikari BR, Howes T & Bhandari BR (2007). Glass transition behavior of spray dried orange juice powder measured by differential scanning calorimetry (DSC) and thermal mechanical compression test (TMCT). *Int. J. Food Prop.* 10:661–673.
- Sivasankar V, Moorthi A, Sarathi Kannan D & Suganyadevi P (2011). Anthocyanin, and its antioxidant properties in selected fruits. J. Pharm. Res. 2011, 4(3): 800-806.
- Tonon RV, Brabet C & Hubinger MD (2008). Influence of process conditions on the physicochemical properties of acai (Euterpeoleraceae Mart.) powder produced by spray drying. *J. Food Eng.* 88:411-418.