



Efficacy of Extraction and Microencapsulation of Betacyanin extracted and Freeze-Dried from Red Dragon Fruit (*Hylocereus polyrhizus*) Peel

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Abstract

Microencapsulation is utilized to prevent chemically sensitive bioactive components from degradation due to harsh environmental conditions and is also applicable to handle the emission of the encapsulate. One of the key factors to be considered in the microencapsulation process is the coating material. Dragon fruit peel derived from processing companies contains an abundant of betacyanin, a valuable pigment. In our research, different parameters of solvent extraction (kind of solvent, pH, ratio of solvent: material); microencapsulation (kind and concentration of coating agent, synergistic effect of combined coating agents) were thoroughly investigated. Our results showed that betacyanin can achieve the highest yield by extraction with water at pH 5.0 with dissolving ratio 5.5: 1.0 (water: material) within 8 hours at ambient temperature. The filtrates were then mixed with 20% maltodextrin+10% β -cyclodextrin as microencapsulating agent during the freeze-drying to get dried powder (6.5% moisture content). By this approach, the highest betacyanin could be maintained at utmost level.

Keywords: *Dragon fruit peel, Betacyanin, Extraction, Microencapsulation, Maltodextrin, β -cyclodextrin.*

Introduction

Dragon fruit (*Hylocereus polyrhizus*) is widely cultivated in Southeast Asia. The dragon fruit peel which makes up 20-25% of the fruit is normally discarded [1]. This will create waste that could be transformed into a value-added product to extract pigment. Dragon fruit peels contain phenolic compounds betalain [2]. Betalain is a water-soluble pigment consisting of two stratial groups of red-violet betacyanine and betaxantin orange-yellow [3].

Spray-drying is popularly applied in the food processing sector due to its rapidity and low cost [4]. Freeze-drying is another method which can be applicable for the coating of sensitive components, especially for those that are susceptible to high thermal degradation [5].

Encapsulation effectiveness of pigment micro particles may be affected by the encapsulation processes applied, as well as by kind of the coating agents [6, 7]. A wide range of microencapsulating agents can be applicable for the coating of pigments, including maltodextrin, β -cyclodextrin, carrageenan, gum arabic, polydextrose and proteins [5, 7, 8]. Maltodextrins are D-glucose polymers that are normally applied for the coating of antioxidants due to their high solubility, low viscosity, and good gel formation attributes [9, 10].

Cyclodextrins are cyclic oligosaccharides consisting of glucose units linked by α -(1, 4) glycosidic bonds derived from the enzymatic degradation of starch by certain bacteria, and they are chemically and physically stable molecules [11].

Some factors may affect inclusion complex formation, such as type of cyclodextrin, cavity size, pH and ionization state, temperature, and method of preparation [12]. Carrageenans are sulfated polysaccharides extracted from algae and have been extensively used in the food industry as gelling agents, stabilizers and texture enhancers and microencapsulation [13, 14].

Soyprotein has been mentioned as beneficial microencapsulating agent owing to their film forming characteristics [15]. Objective of our study focused on different parameters such as solvent extraction (kind of solvent, pH, ratio of solvent: material); microencapsulation (kind and concentration of coating agent, synergistic effect of combined coating agents) affecting to the betacyanin extracted and microencapsulated in freeze-dried powder.

Materials and Method

Material

Dragon fruit peel was utilized from processing factories around Mekong river delta, Vietnam. After collecting, they should be moved to laboratory as soon as possible for experiments. They were subjected to destruct and blend, then extract in polar solvent (ethanol, methanol, water) in different pH values (4.0, 4.5, 5.0, 5.5, 6.0) and in different ratio (solvent: material, 4.0:1.0, 4.5:1.0, 5.0:1.0, 5.5:1.0, 6.0:1.0). Extraction was done with maceration at ambient temperature for 8 hours.

The filtrate was homogenized and centrifuged for 10 minutes at a rate of 5000 rpm. The filtrate was coated by different coating agents (maltodextrin, β -cyclodextrin, carrageenan, soybean protein) in different ratio (15%, 20%, 25%, 30%, 35%) and freeze dried into powder at 6.5% moisture content. The synergistic effect of combined coating agents was also examined.

Researching Procedure

Effect of pH and Solvent in betacyanin Extraction

Different polar solvents (ethanol, methanol, water) in different pH values (4.0, 4.5, 5.0, 5.5, 6.0) were verified to demonstrate the suitable solvent at right pH for betacyanin extraction. Efficiency of solvent extraction was evaluated on the yield of betacyanin ($\mu\text{g}/100\text{g}$).

Effect of Dissolving Ratio (Solvent: Material) in betacyanin Extraction

After choosing the suitable solvent at right pH, the betacyanin extraction also examined whether solvent: material ratio (4.0:1.0, 4.5:1.0, 5.0:1.0, 5.5:1.0, 6.0:1.0) affecting to the yield of betacyanin ($\mu\text{g}/100\text{g}$) or not.

Effect of Coating Agent for betacyanin Microencapsulation

Maltodextrin, β -cyclodextrin, carrageenan, soybean protein at different concentration (15%, 20%, 25%, 30%, 35%) were used to demonstrate the suitable coating agent for betacyanin microencapsulation. Efficiency of coating agent was evaluated on the yield of betacyanin ($\mu\text{g}/100\text{g}$) retention in freeze-dried powder.

A Combination of different Coating Agents on betacyanin Microencapsulation

Different formulas combined from maltodextrin and β -cyclodextrin (5%+25%, 10%+20%, 15%+15%, 20%+10%, 25%+5%) was verified.

Chemical and Statistical Analysis

Betacyanin ($\mu\text{g}/100\text{g}$) was quantified by spectrophotometer at wavelength 538 nm [16]. The experiments were run in triplicate with three different lots of samples. Statistical analysis was performed by the Stat graphics Centurion XVI.

Result & Discussion

Effect of pH and Solvent in betacyanin Extraction

Betacyanin is more soluble in water than in non-polar solvents. This property contributes to extraction and separation. Betacyanin extracted from dragon fruit peel was affected by solvent and pH [17]. In our research, different polar solvents (ethanol, methanol, water) in different pH values (4.0, 4.5, 5.0, 5.5, 6.0) were verified to demonstrate the suitable solvent at right pH for betacyanin extraction.

Efficiency of solvent extraction was evaluated on the yield of betacyanin ($\mu\text{g}/100\text{g}$). Our result showed that there was no significant difference of betacyanin yield extracted by ethanol and water. Betacyanin was dissolved much more in methanol. To ensure the food

safety in respect of betacyanin pigment as colorant, we decided to choose water as a green and safe solvent to extract betacyanin

during utilization. Betacyanin would be received the highest yield at pH 5.0. This finding was similar to other reports.

Table 1: Effect of solvent and pH in betacyanin content ($\mu\text{g}/100\text{g}$) extracted

Solvent	pH 4.0	pH 4.5	pH 5.0	pH 5.5	pH 6.0
Water	528.43 \pm 0.01 ^{bc}	531.12 \pm 0.02 ^b	575.49 \pm 0.03 ^a	536.18 \pm 0.04 ^{ab}	516.04 \pm 0.00 ^c
Methanol	541.25 \pm 0.04 ^{bc}	557.92 \pm 0.00 ^{ab}	582.15 \pm 0.02 ^a	552.48 \pm 0.03 ^b	534.25 \pm 0.01 ^c
Ethanol	530.04 \pm 0.03 ^{bc}	532.29 \pm 0.01 ^b	577.03 \pm 0.00 ^a	537.31 \pm 0.02 ^{ab}	517.75 \pm 0.03 ^c

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant ($\alpha = 5\%$)

Extraction using water is better for stabilizing pigments [18]. Betacyanin pigment is easily influenced by temperature, oxygen, light and water activity [19]. In one report, the highest efficiency of encapsulation of dragon peel extract obtained from the encapsulation composition with solvent pH 5.0 [20]. Betacyanin content was obtained from peels which extracted by methanol pH 5 (515.20 $\mu\text{g}/100\text{g}$) higher than betacyanin content in water pH 5 (491.16 $\mu\text{g}/100\text{g}$) [16].

Effect of Dissolving Ratio (Solvent: Material) in betacyanin Extraction

After choosing the suitable solvent at right pH, the betacyanin extraction also examined whether solvent: material ratio (4.0:1.0, 4.5:1.0, 5.0:1.0, 5.5:1.0, 6.0:1.0) affecting to the yield of betacyanin ($\mu\text{g}/100\text{g}$) or not. Our result showed that the highest content of betacyanin ($\mu\text{g}/100\text{g}$) could be achieved by dissolving ratio 5.5:1.0. This finding was quite similar to another report. M. N. Handayani et al [20]. Confirmed that extract ratio to suspension of 5:1, more efficient than ratio of 3:1.

Table 2: Effect of dissolving ratio (water:material) in betacyanin content ($\mu\text{g}/100\text{g}$) extracted

Water: material ratio	4.0:1.0	4.5:1.0	5.0:1.0	5.5:1.0	6.0:1.0
Betacyanin ($\mu\text{g}/100\text{g}$)	579.26 \pm 0.04 ^b	584.25 \pm 0.02 ^{ab}	597.12 \pm 0.01 ^{ab}	605.08 \pm 0.03 ^a	605.14 \pm 0.05 ^a

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant ($\alpha = 5\%$)

Effect of Coating Agent for betacyanin Microencapsulation

Maltodextrin, β -cyclodextrin, carrageenan, soybean protein at different concentration (15%, 20%, 25%, 30%, 35%) were used to demonstrate the suitable coating agent for

betacyanin microencapsulation. Efficiency of coating agent was evaluated on the yield of betacyanin ($\mu\text{g}/100\text{g}$) retention in freeze-dried powder. Our result showed that the highest content of betacyanin ($\mu\text{g}/100\text{g}$) retention by microencapsulation of filtrate with 30% maltodextrin.

Table 3: Effect of coating agent in betacyanin content ($\mu\text{g}/100\text{g}$) retention in freeze-dried powder

Coating agent	15%	20%	25%	30%	35%
Maltodextrin	401.35 \pm 0.03 ^c	425.39 \pm 0.01 ^b	435.29 \pm 0.02 ^{ab}	439.40 \pm 0.03 ^a	439.58 \pm 0.03 ^a
β -cyclodextrin	362.19 \pm 0.02 ^c	401.22 \pm 0.00 ^b	416.34 \pm 0.01 ^{ab}	427.78 \pm 0.01 ^a	427.90 \pm 0.01 ^a
Carrageenan	317.19 \pm 0.00 ^c	348.36 \pm 0.03 ^b	357.19 \pm 0.02 ^{ab}	379.03 \pm 0.04 ^a	379.17 \pm 0.02 ^a
Soybean protein	331.25 \pm 0.05 ^c	374.15 \pm 0.02 ^{bc}	384.13 \pm 0.04 ^b	395.29 \pm 0.02 ^{ab}	403.17 \pm 0.01 ^a

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant ($\alpha = 5\%$)

In one report, microencapsulations of dragon fruit peel extract using maltodextrin [20]. Adding maltodextrins and starches significantly reduced the hygroscopicity of the betacyanin extracts and enhanced storage stability [19].

A Combination of different Coating Agents on betacyanin Microencapsulation

Microencapsulation efficiency is highly depend on the composition of the encapsulated coating material [21].

In our research, different formulas combined from maltodextrin and β -cyclodextrin (5%+25%, 10%+20%, 15%+15%, 20%+10%, 25%+5%) was verified. Our results noted that

20% maltodextrin + 10% β -cyclodextrin was the best formula for microencapsulation of betacyanin during freeze-drying.

Table 4: Combination of maltodextrin and β -cyclodextrin in betacyanin ($\mu\text{g}/100\text{g}$) retention in freeze-dried powder

Maltodextrin % + β -cyclodextrin %	5%+25%	10%+20%	15%+15%	20%+10%	25%+5%
Betacyanin ($\mu\text{g}/100\text{g}$)	406.79 \pm 0.03 ^d	442.17 \pm 0.04 ^{bc}	459.27 \pm 0.03 ^b	494.13 \pm 0.02 ^a	438.04 \pm 0.04 ^c

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant ($\alpha = 5\%$)

The effect of different combinations of maltodextrin (MD) coating agents (MD, MD + soybean protein, and MD + ι -carrageenan) on the encapsulation of lemon by-product aqueous extracts using freeze-drying and spray-drying were investigated. Freeze-drying with the mixture of MD + soybean protein resulted in the highest retention of TPC, TFC, and FRAP.

The combination of maltodextrin with soybean protein as the microencapsulating agent resulted in the formation of spherical porous materials during freeze-drying [22]. The powders of the blueberry anthocyanin extracts manufactured by freeze-drying using combinations of different microencapsulating

carriers, including maltodextrin, β -cyclodextrin, and whey protein isolate, and gum arabic, were in the amorphous phase, irrespective of the formulations [23].

Conclusion

Both extraction and microencapsulation technique were found to significantly affect the stability of betacyanin pigment powder extracted and freeze-dried from dragon fruit peel waste. Microencapsulation is defined as the trapping process of both liquid, solid and gas particles in thin films that can provide a physical barrier between core compounds and other components. Freeze drying is one of the best strategies in microencapsulation technology for preserving the stability of sensitive pigments.

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