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## Microencapsulation and Stability Analysis of Blueberry Anthocyanins

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# Microencapsulation and Stability Analysis of Blueberry Anthocyanins

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**Abstract.** In order to improve the stability of blueberry anthocyanins, microencapsulation of blueberry anthocyanins was investigated by spray drying method and the stability of the native and microencapsulated anthocyanins was evaluated. The optimal microencapsulation parameters were as follows:  $\beta$ -cyclodextrin-whey protein-arabicgum with the quality ratio of 16:8:1 as wall material, the blueberry anthocyanins as core material, core-to-wall quality ratio of 1:2.5, the emulsion concentration was 250 g/L, rotary speed 2000 r/min, emulsification time 10 min, feed flow rate 350 mL/h, inlet air temperature 160 °C, outlet air temperature 80 °C. The encapsulation efficiency of 95% was obtained under the optimized conditions. The microcapsulated blueberry anthocyanins were dark-red powder with smooth spherical shape, and were more stable than the native anthocyanins when exposed to light and temperature.

## 1. Introduction

Blueberries are rich in anthocyanins which possess a lot of biological activities including antioxidant, anticancer, protection cardiovascular, improving vision, and so on [1]. But the blueberry anthocyanins affected by pH, oxidation, light, temperature, etc., have poor stability during storage, which is a big obstacle for their application [2]. It would be helpful to expand the applications of blueberry anthocyanins if the stability of them could be increased.

To enhance the stability of blueberry anthocyanins, physical and chemical methods have been done. Some studies reported that the association and copigmentation by adding various sugars, flavonoids and organic acids could increase the stability of blueberry anthocyanins [3, 4]. Some studies reported that the acylation of blueberry anthocyanins by the chemical synthesis method to introduce the aliphatic carbonyl acids or aromatic acids to blueberry anthocyanins had high stability [5-7].

Microencapsulation is an alternative for improving the stability of anthocyanins. It can protect sensitive food ingredients against heat, pH, and moisture and mitigate unpleasant smell and taste, thus enhancing stability during storage by suppressing the emission of the core [8]. Flores et al. prepared blueberry anthocyanins microencapsulation by using whey protein isolate and gum arabic as the wall materials respectively, and found that rehydrated gum arabic microcapsules retained more total anthocyanins than did whey protein microcapsules [9]. Wall materials could affect the morphology, yield, the stability of the microcapsules. Among microencapsulation techniques, spray-drying is a cost



effective method that can result in the formation of a stable, free-flowing powder. Here, we investigated the microencapsulation of blueberry anthocyanins by spray drying method using  $\beta$ -cyclodextrin-whey protein-arabic gum as wall material and blueberry anthocyanins powder as core material, and the stability of the native and microencapsulated anthocyanins was evaluated.

## 2. Materials and methods

### 2.1. Materials

Blueberry anthocyanins were prepared by ourselves as described in a previous study [10].  $\beta$ -cyclodextrin, whey protein, and arabic gum were food-grade. Deionized water was prepared from a Milli-Q Element water purification system (Millipore Co., Billerica, MA, USA).

### 2.2. Blueberry anthocyanins encapsulation

Wall materials at different ratios were mixed and dissolved in deionized water at room temperature to get wall materials. Blueberry anthocyanins were added slowly to the solution containing wall materials. The mixture was mixed under strong magnetic stirring. To ensure complete homogenization, sample was further emulsified, and the obtained dispersion was pumped to spray-dryer. The resultant powders were stored in polypropylene bottles at 4 °C.

### 2.3. Total anthocyanin content

The pH differential method was used to estimate the total anthocyanin content of the anthocyanin powders and spray-dried powders [5-7]. Specifically, take 1mL diluted sample, add 9 mL buffer solution which pH is 1.0 and 4.5 respectively, and then detect the absorbance at 510 and 700 nm after a water bath for 40 min.

### 2.4. Encapsulation efficiency (EE)

EE was determined as the following equation:  $EE (\%) = (1 - S_E/T_E) \times 100$

Where  $S_E$  is the anthocyanin content on the surface of microcapsules and  $T_E$  is the total anthocyanin content of microcapsules [17].

### 2.5. Encapsulation productivity (EP)

EP expressed as the ratio in percentage between total anthocyanin content in microcapsules ( $T_E$ ) and the amount of anthocyanins used ( $T_A$ ), was calculated using the following equation [11]:

$$EP (\%) = T_E/T_A \times 100$$

### 2.6. Powder morphology

The morphology of encapsulated powders was investigated using a multifunctional optical microscope (Axio Imager A1).

### 2.7. Thermal stability

The blueberry anthocyanins of before and after microencapsulation were taken in a porcelain crucible and put them in a constant temperature oven at 60, 80 and 100 °C, respectively. After 2, 4, 6, 8, 10 hours, a certain amount of each sample was weighed and dissolved in the buffer solution of pH 1.0 and 4.5 respectively. The absorbance at 510 and 700 nm after a water bath for 40 min was measured, and the retention rate was calculated by the following formula:

$$\text{Retention rate (\%)} = (A_t/A_0) \times 100$$

Where  $A_t$  is the anthocyanin content at different times and  $A_0$  is the anthocyanin content before treatment.

### 2.8. Light stability

The blueberry anthocyanins before and after microencapsulation were taken in white jar and put them in outdoor natural light and indoor avoiding light, respectively. After 2, 4, 6, 8 10 days, a certain

amount of each sample was weighed and dissolved in the buffer solution of pH 1.0 and 4.5 respectively. The absorbance at 510 and 700 nm after a water bath for 40 min was measured, and the retention rate was calculated by the following formula:

$$\text{Retention rate (\%)} = (A_t/A_0) \times 100$$

Where  $A_t$  is the anthocyanin content at different times and  $A_0$  is the anthocyanin content before treatment.

### 2.9. Statistical analysis

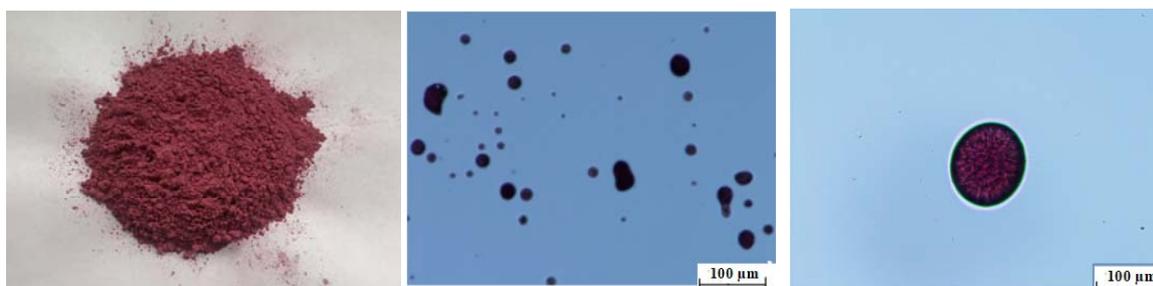
Experiments were performed at repeating three times, and the results were expressed as means standard deviation. Statistical analysis was performed using SPSS 16.0. Differences between groups were considered significant at  $P < 0.05$ .

## 3. Result and discussion

### 3.1. Blueberry anthocyanins microencapsulation

The effects of arabicgum content,  $\beta$ -cyclodextrin content, whey protein content, core-wall ratio, emulsion concentration, rotary speed, emulsification time, feed flow rate, inlet air temperature, outlet air temperature and total solids content on the microencapsulation of blueberry anthocyanins were investigated, and the EP was analyzed by variance analysis. The experimental results showed that the  $\beta$ -cyclodextrin content, whey protein content, core-wall ratio, inlet air temperature and solid content had a significant effect on the EP. And the optimal microencapsulation parameters were:  $\beta$ -cyclodextrin-whey protein-arabicgum with the quality ratio of 16:8:1 as wall material, the blueberry anthocyanins as core material, core-to-wall quality ratio of 1:2.5, the emulsion concentration 250 g/L, rotary speed 2000 r/min, emulsification time 10 min, feed flow rate 350 mL/h, inlet air temperature 160 °C, outlet air temperature 80 °C. Under the optimized conditions, 80% EP and 95% EE could be obtained.

### 3.2. Powder morphology



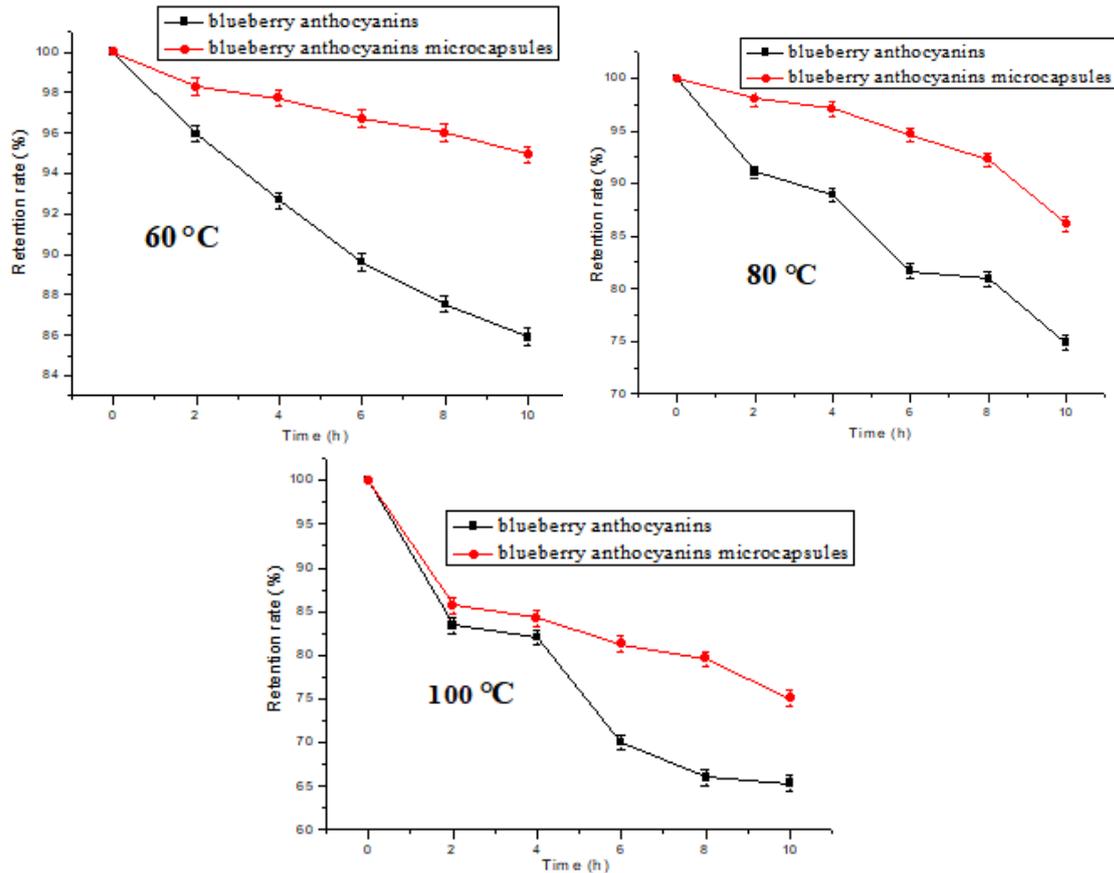
**Figure 1.** Microphotograph of blueberry anthocyanins microcapsules ( $\times 10$ ).

According to the optimized process, the blueberry anthocyanins microcapsules were dark-red powder and no special odor. Under the microscope, the shape of the capsules was spherical or nearly spherical (Figure 1).

### 3.3. Thermal stability

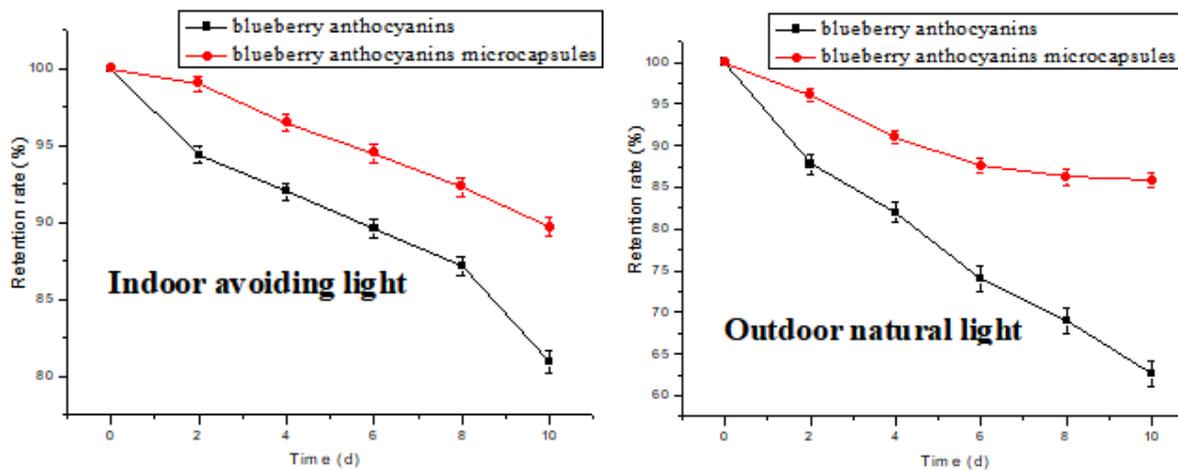
Figure 2 showed the retention rates of blueberry anthocyanins before and after microencapsulation with increasing temperature and extension of heating time. Anthocyanins after microencapsulation were found to possess higher heat resistance than the blueberry anthocyanins at different temperatures. With increasing temperature, the effect of heat resistance became more obvious. After being heated 10h at 60, 80 and 100 °C, the retention rates of blueberry anthocyanins after microencapsulation were all over 75%, which was obviously higher than the native anthocyanins. It might be that the

microcapsule wall material had a protective effect on blueberry anthocyanins and increased its heat resistance.



**Figure 2.** The stability of blueberry anthocyanins before and after microencapsulation at different temperatures.

3.4. Light stability



**Figure 3.** The stability of blueberry anthocyanins before and after microencapsulation in outdoor natural light and indoor avoiding light.

It could be seen from Figure 3 that the blueberry anthocyanins were more resistant to light after microencapsulation. Compared with the microcapsules, native anthocyanins had poor stability to light. When exposed to indoor natural light for a period of time, the retention rate of native anthocyanins decreased rapidly. After being lighted 10 days in outdoor natural light, the retention rate of microencapsulated blueberry anthocyanins was over 85%, which was obviously higher than the native anthocyanins. The blueberry anthocyanins after microencapsulation might get protection from the wall material and their stability to light could be improved.

#### 4. Conclusion

Microencapsulation of blueberry anthocyanins was successfully prepared by spray drying method using  $\beta$ -cyclodextrin-whey protein-arabic gum as wall material. Under the optimized conditions, the anthocyanins microcapsules were dark-red powder with smooth spherical shape and were more stable than the native anthocyanins when exposed to light and temperature. Microencapsulated blueberry anthocyanins with high stability were conducive to the storage of anthocyanins and could improve the utilization rate of anthocyanins. This study would provide a theoretical basis for the development of functional food of anthocyanins.

#### Acknowledgments

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#### References

- [1] Q. Xu, Y.J. Zhou, Y. Wu, Q. Jia, F. Nie, G. Gao, Bioactivities of blueberry anthocyanins, *Biotech. Med. Sci.* (2016) 104-109.
- [2] P.M. Reque, R.S. Steffens, A. Jablonski, S.H. Flôres, A.D.O. Rios, V.D.J. Erna, Cold storage of blueberry (*Vaccinium* spp.) fruits and juice: Anthocyanin stability and antioxidant activity, *Subtropical Plant Sc.* 33 (2014) 111-116.
- [3] R. Jamei, F. Babaloo, Stability of blueberry (*Cornus mas*–Yulyush) anthocyanin pigment under pH and co-pigment treatments, *Int. J. Food. Prop.* 9 (2017) 2128-2133
- [4] Y.Z. Pan, Y. Guan, Z.F. Wei, X. Peng, T.T. Li, X.L. Qi, Y.G. Zu, Y.J. Fu, Flavonoid C-glycosides from pigeon pea leaves as color and anthocyanin stabilizing agent in blueberry juice, *Ind. Crop. Prod.* 58 (2014) 142-147
- [5] Q. Xu, Y.J. Zhou, L.P. Luo, Z. Huang, F. Nie, G.L. Gao, Acylation of blueberry anthocyanins with aliphatic carbonyl acids and their stability analysis, *Advances Engineering Res.* 143(2017) 904-907.
- [6] Q. Xu, Y.J. Zhou, L.P. Luo, F. Nie, G.L. Gao, Acylation and stability analysis of blueberry anthocyanins, *Advances Material Energy & Environmental Engineering*, 2017, 378-381.
- [7] Q. Xu, Y.J. Zhou, D.B. Wang, B.J. Li, L.P. Luo, F. Nie, G.L. Gao, Study on improving the stability of blueberry anthocyanins, *Advances in Engineering Res.* 163 (2018) 1747-1750.
- [8] M.A. Augustin, Y. Hemar, Nano- and microstructured assemblies for encapsulation of food ingredients. *Chem. Soc. Rev.* 38 (2009) 902-912.
- [9] F.P. Flores, R.K. Singh, W.L. Kerr, R.B. Pegg, F. Kong, Total phenolics content and antioxidant capacities of microencapsulated blueberry anthocyanins during in vitro digestion, *Food. Chem.* 153 (2014) 272-278.
- [10] Q. Xu, Y.J. Zhou, Y.X. Wu, Q. Jia, G.L. Gao, F. Nie, Enzyme-assisted solvent extraction for extraction of blueberry, *Int. J. Food. Sci. Tech.* 51 (2016) 2567-2573
- [11] G. Davidov-Pardo, I. Arozarena, M.R. Marín-Arroyo, Optimization of a wall material formulation to microencapsulate a grape seed extract using a mixture design of experiments, *Food Bioprocess Tech.* 6 (2013) 941-951.