Research Article Optimization of Vacuum Freeze Drying of Anthocyanins from Acanthopanax

sessiliflorus Fruits

^{1, 2}Xinru Shao, ¹Haitao Sun, ¹Yuan Dai, ¹Zhongsu Ma, ³Liping Wang and ³Jianghui Dong ¹College of Biological and Agricultural Engineering, Jilin University, Changchun 130022, ²Research Center of Changbai Mountain Food Engineering, Tonghua Normal University, Tonghua 134000, China

³School of Computer Science, Engineering and Mathematics, Flinders University, South Australia, Australia

Abstract: Acanthopanax sessiliflorus fruits anthocyanins were prepared by vacuum freeze drying technology and the particle structure was studied. On the basis of single factor test the effects of drying pressure, material thickness and heating plate temperature on the drying anthocyanins were investigated by Box-Benhnken center composite design and response surface technology. The results showed that the optimum technical conditions were as follows: drying pressure 50 Pa, material thickness 8 mm and heating plate temperature 50°C. Under the optimum conditions, the drying rate was 19.37 g/(g·h), the anthocyanins retention rate was 82.88% and the moisture content was 4.13%. Scanning Electron Microscope (SEM) analyses showed that the anthocyanins were flaky particles, uniform particle size with smooth surface.

Keywords: Acanthopanax sessiliflorus fruits, anthocyanins, vacuum freeze drying

INTRODUCTION

Acauthopauax sessiliflorus belongs to Araliaceae, which grows in bushes with height of 2-5 m. The purple-black fruits become ripe in September with ovoid in shape and 1-1.5 cm in size (Jeong et al., 2007; Woo et al., 2003). Acauthopauax sessiliflorus fruits are known to be a new type of wild berries resources, which contains a large amount of bioactive ingredient (Choi et al., 2002; Jiang et al., 2006), such as flavonoids, polysaccharides, triterpenoids, coumarin and lignins (Yang et al., 2009; Lee et al., 2002a). Acauthopauax sessiliflorus fruits have high nutritional value and pharmacological effects, including immunity enhancement, anti-fatigue, central nervous system and cardiovascular system regulation, anticancer, lowering blood pressure and improving liver detoxification function (Jung et al., 2012; Jun et al., 2007).

Anthocyanins are a class of water-soluble natural pigments, widespread in nature fruits, flowers, seeds and leaves (Wang and Lin, 2000). Anthocyanins are flavonoid polyphenolic compounds mainly in the plants cell sap, combined by anthocyanidins and various sugars. The common aglycon forms of anthocyanidins, are found pelargonidin, cyanidin, delphinidin, peonidin, petunidin, malvidin, etc., (Hong *et al.*, 2001; Kim *et al.*, 2006). The combined sugars mainly are glucose, rhamnose, galactose, xylose and fructose, which

provide a range of colors (e.g., red, purple and blue) to the plants (Lee et al., 2002b). Globally, anthocyanins have been widely used in disease treatment and the production of health care and cosmetics products, due to its excellent curative effects, such as anti-rheumatoid arthritis (Lee et al., 2013), anti-inflammatory (Jaouad et al., 2007; Lee et al., 2003), enhancing immunity (Lee et al., 2012), anti-cancer (Yang et al., 2013), anticardiovascular disease (Yang et al., 2013), inhibitory pancreatic lipase activity (Yoshizumi et al., 2006), antioxidant (Wang et al., 1999; Viljanen et al., 2005) and anti-radiation (Jung et al., 2012). In the daily diet, anthocyanins can be obtained by ingesting vegetables, fruits, wine and other foods. However, to achieve the physiological functions in vivo, anthocyanins must be highly bioavailable, effectively absorbed from the intestine into the blood circulation and delivered to the appropriate location in body.

Due to it's difficult to storage anthocyanins after extraction and purification and the anthocyanins are sensitive to high temperature, there is significant demand for a highly efficient and precisely controlled drying process to retain the maximum activity of anthocyanins. Vacuum freeze drying is a method of frozen materials and placed in a high vacuum container and water is sublimated vapor state from solid state by low-temperature heating (Wang *et al.*, 2010). Due to the low temperature of freeze, microbial activity is

Corresponding Author: Zhongsu Ma, College of Biological and Agricultural Engineering, Jilin University, Changchun 130022, China

This work is licensed under a Creative Commons Attribution 4.0 International License (URL: http://creativecommons.org/licenses/by/4.0/).

preserved and the products for a long time without deterioration. In addition, the products prepared by vacuum freeze drying have the advantages of minimum loss of nutrients, good rehydration ratio and good color with low drying temperature. Vacuum freeze drying has been widely used to obtain high-quality and high-value dehydrated fruit and vegetables (Ji *et al.*, 2012), which is one of the best methods to remove the water molecules from biological materials and the biological active substance is maximally retained (Sagar and Suresh, 2010). In the present study, we report the optimum conditions of anthocyanins derived from *Acanthopanax sessiliflorus* fruits by vacuum freeze drying and the particle structure was studied.

MATERIALS AND METHODS

Materials: The *Acanthopanax sessiliflorus* fruits were ripe and picked from Changbai mountain (Tonghua, China) in September, were cleaned gently, dried at 50°C, crushed and preserved in -20°C used for further analysis in laboratory.

Ethanol, concentrated Hydrochloric Acid (HCl) and sodium Hydroxide (NaOH) were of analytical grade and purchased from Shanghai Chemical Works (Shanghai, China).

Extraction and purification of anthocyanins from *Acanthopanax sessiliflorus* fruits: The defrosted *Acanthopanax sessiliflorus* fruits powder was added to solid-liquid ratio 1:10 pH 2.0, 40% ethanol solution. The extraction was carried out at 50°C for 20 min, ultrasonic power 200 w by KQ-200KDB CNC highpower ultrasonic cleaner (Kunshan Ultrasonic Instrument Co., Ltd.). The extract was filtered, centrifuged at 4000 rpm for 10 min, the supernatant was concentrated at 50°C by RE-5298 rotary evaporator (Shanghai Ya Rong Biochemical Instrument Co., Ltd.) and the *Acanthopanax sessiliflorus* fruits anthocyanins concentrate were obtained.

The Acanthopanax sessiliflorus fruits anthocyanins concentrate were purified by AB-8 macroporous resin. The optimal conditions were as follows: adsorption equilibrium 4 h, desorption equilibrium 2 h, adsorption equilibrium concentration 750 mg/L and pH 3.0 60% ethanol solution was used as desorption solution. The optimal conditions of dynamic desorption was adsorption flow rate of 1 mL/min, the sample solution concentration 3.0 mg/mL and 5 volumes of pH 3.0 60% ethanol solution was used as eluent, with the flow rate of 1 mL/min. Then the purification of Acanthopanax

sessiliflorus fruits anthocyanins was obtained by concentrated under reduced pressure at 50°C.

Vacuum freeze drying of *Acanthopanax sessiliflorus* **fruits anthocyanins:** Experiments were performed in LGJ-10 vacuum freeze dryer (Shanghai Institute of centrifugal Mechanical). The purification concentrate of *Acanthopanax sessiliflorus* fruits anthocyanins were pre-frozen in the cold trap at -40°C, followed by sublimation and desorption drying (Wu *et al.*, 2010).

Single factor tests: The effect of drying pressure, material thickness, heating plate temperature on drying rate, anthocyanins retention rate and moisture content were investigated. The single factor tests were performed under the condition of material thickness 6 mm, heating plate temperature 50°C, drying pressure 20 Pa, 40, 60, 80 and 100 Pa, respectively; drying pressure 60 Pa, heating plate temperature 50°C, material thickness 2, 6, 10, 14 and 18 mm; material thickness 6 mm, drying pressure 60 Pa, heating plate temperature 30°C, 40, 50, 60 and 70°C, respectively.

Response surface optimization tests: Based on single factor tests results, according to the central composite experimental design principles of Box-Behnken, the effect of three independent variables drying pressure (X_1) , material thickness (X_2) , heating plate temperature (X_3) on the drying rate (Y_1) , anthocyanins retention rate (Y_2) and moisture content (Y_3) were investigated, the factors and levels were shown in Table 1.

Calculation of the composite indicator: Weighting method was used to given weights for each index, the weight coefficient of drying rate, anthocyanins retention rate and moisture content were 0.3, 0.4 and 0.3, respectively. The maximum of drying rate and anthocyanins retention were considered to be 100 points, the minimum of water content was considered to be 100 points, the comprehensive evaluation value was calculated by each index multiplied the weighting coefficient and summed.

Determination of *Acanthopanax sessiliflorus* **fruits anthocyanins content:** The absorbance was measured by TV-1901 UV-visible spectrophotometer (Beijing PERSEE Co., Ltd.). The maximum absorption wavelength of *Acanthopanax sessiliflorus* fruits anthocyanins was 525 nm in pH 1 and the anthocyanins

Table 1: Factors and levels of response surface design

	Factors		
Levels	X ₁ drying pressure/Pa	X ₂ material thickness/mm	X_3 heating plate temperature/°C
-1	50	6	45
0	60	8	50
1	70	10	55
		747	

were changed to colorless chalcone. The content *Acanthopanax sessiliflorus* fruits anthocyanins was calculated by pH-differential method (Shao *et al.*, 2014).

Determination of drying rate:

$$\mathbf{V}_{\mathrm{d}} = (\mathbf{m}_{\mathrm{w}} \cdot \mathbf{m}_{\mathrm{d}}) / (\mathbf{m}_{\mathrm{d}} \cdot \mathbf{t}_{\mathrm{d}})$$

where,

 $\begin{array}{ll} V_d &= Drying \ rate \ (g/g \cdot h) \\ m_w &= The \ material \ weight \ before \ drying \ (g) \\ m_d &= The \ material \ weight \ after \ drying \ (g) \\ t_d &= Drying \ time \ (h) \end{array}$

Data were analyzed using the Design Expert 8.0.6 software, p-value of < 0.05 was considered significant. All experiments were repeated in triplicates.

Observation structure: Acanthopanax sessiliflorus fruits anthocyanins after vacuum freeze drying were fixed on slide, the samples were coated with conductive gold film by a ion sputtering instrument. Then the surface morphology and particle structure of Acanthopanax sessiliflorus fruits anthocyanins were observed by S-3000N scanning electron microscope (SEM, Japan Hitachi Ltd.)

RESULTS AND DISCUSSION

Single factor tests: Figure 1 showed that the drying rate and anthocyanins retention rate reached maximum value and moisture content reached minimum value at the drying pressure of 60 Pa. It's due to the vacuum freeze drying is a process of heat and mass transfer (Wu *et al.*, 2010). The drying time can be shortened and drying rate can be improved by increasing the drying pressure. However, it was difficult to reduce the heat and drying rate when the pressure was higher than 60 Pa. With the increasing of drying pressure, the heating temperature and the loss of heat-sensitive anthocyanins can be reduced and anthocyanins retention rate can be improved. Furthermore, ice sublimation can be accelerated and the moisture content can be reduced by increasing the drying pressure.

The results (Fig. 2) showed that the effect of material thickness on drying rate was significant, drying rate decreased with the increase of material thickness, especially when material thickness was 14-18 mm. This was because the mass transfer resistance was great as well as the moisture migration was slow from the inside to the surface in a thicker the material, leading to a smaller drying rate. Anthocyanins retention was increased with the increase of material thickness and reached maximum value at ~10 mm (Wang *et al.*, 2010). When the thickness >10 mm, temperature difference between the inside and outside became larger



Fig. 1: Effect of drying pressure on drying rate, anthocyanins retention rate and moisture content



Fig. 2: Effect of material thickness on drying rate, anthocyanins retention rate and moisture content



Fig. 3: Effect of heating plate temperature on drying rate, anthocyanins retention rate and moisture content

and the more damage occurred to the outer anthocyanins cell tissue, therefore the anthocyanins retention rate was lower. In addition, the moisture content was increases with the material thickness. It was appropriate to select material thickness at 6-10 mm as the scope of study. The process of sublimation was maintained a product must be further dried to remove the bound water (Ji *et al.*, 2012). As was shown in Fig. 3, the drying rate increased with the increasing heating plate temperature and the anthocyanins retention rate reached

certain time and the ice crystals disappeared and the maximum at 50° C. In lower heating plate temperature (30-50°C), the drying rate was low and the drying time was long, which causes more damage to the outer anthocyanins cell tissue and anthocyanins retention rate

Table 2: Experimental design and results of response surface design Factors

	1 401015								
				Y_1 drying rate/(g/(g·h))		Y ₂ anthocyanins retention rate/%		Y ₃ moisture content/%	
Run	X ₁ drying pressure/Pa	X ₂ material thickness/mm	X ₃ heating plate temperature/°C	Actual value	Predictive value	Actual value	Predictive value	Actual value	Predictive value
1	-1	-1	0	15.95	15.93	83.52	83.59	4.82	4.79
2	1	1	0	15.02	15.04	75.47	75.40	4.89	4.92
3	-1	1	0	15.02	15.21	79.05	79.04	4.55	4.52
4	0	0	0	19.98	19.70	82.60	82.52	4.20	4.16
5	1	-1	0	15.33	15.14	80.91	80.92	4.95	4.98
6	0	1	-1	15.64	15.67	69.61	69.55	4.93	4.94
7	-1	0	-1	19.05	18.83	78.21	78.28	4.50	4.52
8	1	0	-1	16.88	16.83	74.13	74.26	4.64	4.59
9	0	0	0	19.36	19.70	82.65	82.52	4.14	4.16
10	0	-1	-1	15.33	15.57	76.91	76.77	5.15	5.16
11	0	1	1	16.26	16.02	70.77	70.91	4.85	4.83
12	0	0	0	19.59	19.70	82.32	82.52	4.19	4.16
13	1	0	1	18.98	19.20	74.38	74.31	4.65	4.64
14	-1	0	1	18.12	18.17	76.74	76.60	4.08	4.12
15	0	-1	1	16.95	16.92	73.71	73.77	4.94	4.93
16	0	0	0	19.73	19.70	82.61	82.52	4.17	4.16
17	0	0	0	19.84	19.70	82.41	82.52	4.13	4.16

Table 3: Analysis of variance for each term of the fitted regression model

ANOVA for response surface quadratic model of Y1

ANOVA for response surface quadratic model of Y₂

Source	S.S.	df	M.S.	F value	p-value prob>F	S.S.	df	M.S.	F value	p-value prob>F	
Model	58.670	9	6.520	87.48	< 0.0001**	320.340	9	35.590	1302.75	< 0.0001**	
X_1	0.470	1	0.470	6.25	0.0410*	19.920	1	19.920	729.12	< 0.0001**	
X_2	0.330	1	0.330	4.40	0.0741	50.750	1	50.750	1857.61	< 0.0001**	
X ₃	1.450	1	1.450	19.50	0.0031**	1.340	1	1.340	49.82	0.0002**	
X_1X_2	0.096	1	0.096	1.29	0.2935	0.240	1	0.240	8.68	0.0215*	
X_1X_3	2.300	1	2.300	30.80	0.0009**	0.740	1	0.740	27.01	0.0013**	
X_2X_3	0.250	1	0.250	3.35	0.1097	4.730	1	4.730	173.31	< 0.0001**	
X_{1}^{2}	4.900	1	4.900	65.75	< 0.0001**	0.120	1	0.120	4.22	0.0790	
X_2^2	45.610	1	45.610	612.01	< 0.0001**	36.590	1	36.590	1339.32	< 0.0001**	
X_{3}^{2}	0.560	1	0.560	7.48	0.0292*	195.840	1	195.840	7168.02	< 0.0001**	
Residual	0.520	7	0.075			0.190	7	0.027			
Lack of fit	0.30	3	0.098	1.74	0.2974	0.110	3	0.036	1.69	0.3061	
Pure error	0.230	4	0.057			0.084	4	0.021			
Cor total	59.200	16				320.530	16				

ANOVA for response surface quadratic model of \mathbf{Y}_3

					p-value
Source	S.S.	df	M.S.	F value	prob>F
Model	2.050	9	0.230	116.81	<0.0001**
X_1	0.170	1	0.170	88.08	<0.0001**
X_2	0.053	1	0.053	26.94	0.0013**
X ₃	0.060	1	0.060	30.69	0.0009**
X_1X_2	0.011	1	0.011	5.44	0.0525
X_1X_3	0.048	1	0.048	24.81	0.0016**
X_2X_3	3.745×10-3	1	3.745×10-3	1.92	0.2084
X_{1}^{2}	0.020	1	0.020	10.31	0.0148*
X_{2}^{2}	1.360	1	1.360	694.69	<0.0001**
X_{3}^{2}	0.230	1	0.230	118.20	<0.0001**
Residual	0.014	7	1.951×10-3		
Lack of fit	9.522×10-3	3	3.174×10-3	3.07	0.1533
Pure error	4.133×10-3	4	1.033×10-3		
Cor total	2.060	16			

**: p<0.01, extremely significant difference; *: p<0.05, significant difference; S.S.: Sum of square; M.S.: Mean square

was low. However, due to the heat sensitive of anthocyanins, damaging effects of structure can be enhanced at a higher temperature (> 50° C) and the anthocyanins retention rate decreased. With the increase of heating plate temperature, moisture content was also decreased. It was appropriate to select the heating plate temperature at about 50° C.

Mathematical models and analysis of significant: The results of response surface analysis were shown in Table 2, Analysis of Variance (ANOVA) results of the regression model were shown in Table 3.

Quadratic multiple regression of drying rate was obtained by using design Expert 8.0.6 software:

 $Y_{I} = 19.70 - 0.24X_{I} - 0.20X_{2} + 0.43X_{3} + 0.15X_{I}X_{2}$ $+ 0.76X_{I}X_{3} - 0.25X_{2}X_{3} - 1.08X_{I}^{2} - 3.29X_{2}^{2} - 0.36X_{3}^{2}$

The significance test and variance analysis of regression equation at the significance level $\alpha = 0.05$, F = 87.48, p<0.0001, regression models extremely significant. Determination coefficient $R^2 = 0.9799$ and 97.99% of the drying rate can be explained by the model. Lack of fit p = 0.2974 > 0.05 and not significant, showed that the fitting degree of predictive value and actual value was high. Heating plate temperature (X_3) on the drying rate was extremely significant, drying pressure (X_1) on the drying rate was significant, material thickness (X_2) on the drying rate was not significant. Interaction of drying pressure (X_1) and heating plate temperature (X_3) on the drying rate was extremely significant (p = 0.0009). Interaction of drying pressure (X_1) and material thickness (X_2) , material thickness (X_2) and heating plate temperature (X_3) on the drying rate was not significant. Removed the not significant $\alpha = 0.05$, The simplified regression equation:

$$Y_{I} = 19.70 - 0.24X_{I} + 0.43X_{3} + 0.76X_{I}X_{3} - 1.08X_{I}^{2}$$

- 3.29X₂² - 0.36X₃²

Quadratic multiple regression of anthocyanins retention rate was obtained:

$$Y_{2} = 82.52 - 1.58X_{1} - 2.52X_{2} - 0.41X_{3} - 0.24X_{1}X_{2} + 0.43X_{1}X_{3} + 1.09X_{2}X_{3} + 0.17X_{1}^{2} - 2.95X_{2}^{2} - 2.82X_{3}^{2}$$

The significance test and variance analysis of regression equation at the significance level $\alpha = 0.05$, F = 1302.75, p<0.0001, regression model was extremely significant. Determination coefficient $R^2 = 0.9986$ and 99.89% of the anthocyanins retention rate can be explained by the model. Lack of fit p = 0.3061>0.05 and not significant, showed that the fitting degree of predictive value and actual value was

high. Drying pressure (X_1) , material thickness (X_2) and heating plate temperature (X_3) on the anthocyanins retention rate was extremely significant. Interaction of material thickness (X_2) and heating plate temperature (X_3) , drying pressure (X_1) and heating plate temperature (X_3) on the anthocyanins retention rate was extremely significant (p<0.0001). Interaction of drying pressure (X_1) and material thickness (X_2) on the anthocyanins retention rate was significant (p = 0.0215). Removed not significant $\alpha = 0.05$, The simplified regression equation:

$$Y_2 = 82.52 - 1.58X_1 - 2.52X_2 - 0.41X_3 - 0.24X_1X_2 + 0.43X_1X_3 + 1.09X_2X_3 - 2.95X_2^2 - 2.82X_3^2$$

Quadratic multiple regression of moisture content was obtained:

$$Y_{3} = 4.16 + 0.15X_{1} - 0.081X_{2} - 0.086X_{3} + 0.051X_{1}X_{2} + 0.11X_{1}X_{3} + 0.031X_{2}X_{3} + 0.069X_{1}^{2} + 0.57X_{2}^{2} + 0.23X_{3}^{2}$$

The significance test and variance analysis of regression equation at the significance level $\alpha = 0.05$, F = 116.81, p<0.0001, regression model was extremely significant. Determination coefficient $R^2 = 0.9849$ and 98.49% of the moisture content can be explained by the model. Lack of fit p = 0.1533 > 0.05 and not significant, showed that the fitting degree of predictive value and actual value was high. Drying pressure (X_1) , material thickness (X_2) and heating plate temperature (X_3) on the moisture content was extremely significant. Interaction of drying pressure (X_1) and heating plate temperature (X_3) on the moisture content was extremely significant (p = 0.0016). Interaction of drying pressure (X_1) and material thickness (X_2) , material thickness (X_2) and heating plate temperature (X_3) on the moisture content was not significant. Removed not significant $\alpha = 0.05$, The simplified regression equation:

$$Y_3 = 4.16 + 0.15X_1 - 0.081X_2 - 0.086X_3 + 0.11X_1X_3 + 0.069X_1^2 + 0.57X_2^2 + 0.23X_3^2$$

Interaction analysis: Data in Table 2 were response surface analyzed using Design Expert 8.0.6 software and the effects of various factors interaction on the drying rate were shown in Fig. 4. In the range of test level, at a constant heating plate temperature, the drying rate increased with the increasing drying pressure and material thickness. Continued to increase the material thickness and drying pressure, the drying rate decreased after reached the maximum. When the material thickness was 8 mm and the heating plate temperature was constant, the drying rate increased with the increasing of drying pressure, continued to improve drying pressure, the drying rate decreased rapidly after



(a) Drying pressure and material thickness



(b) Drying pressure and heating plate temperature



(c) Material thickness and heating plate temperature

Fig. 4: Response surface of drying rate



Adv. J. Food Sci. Technol., 7(10): 746-755, 2015





(b) Drying pressure and heating plate temperature



(c) Material thickness and heating plate temperature

Fig. 5: Response surface of the anthocyanins retention rate



(a) Drying pressure and material thickness



(b) Drying pressure and heating plate temperature



(c) Material thickness and heating plate temperature

Fig. 6: Response surface of the moisture content

reaches the maximum; At a constant drying pressure, with the heating plate temperature increased, the drying rate first increased and then decreased. When the drying pressure was 60 Pa and the heating plate temperature was constant, the drying rate increased with the increasing of material thickness, continued to increase material thickness, the drying rate decrease rapidly after reaches the maximum; At a constant material thickness, drying rate was increased with the increasing of heating plate temperature. Comprehensive analysis of the interaction between the various factors, drying pressure (X_1) and heating plate temperature (X_3) on the drying rate were extremely significant. The effects of various factors on the drying rate were heating plate temperature>material thickness.

The effects of various factors interaction on the anthocyanins retention rate were shown in Fig. 5. Using the same method described above, interaction of drying pressure (X_1) and heating plate temperature (X_3) , material thickness (X_2) and heating plate temperature (X_3) on the anthocyanins retention rate were extremely significant. The effects of various factors on the anthocyanins retention rate were: material thickness>heating plate temperature>drying pressure.

The effects of various factors interaction on the moisture content were shown in Fig. 6. Using the same method described above, interaction of drying pressure (X_1) and heating plate temperature (X_3) on the moisture content was extremely significant. The effects of various factors from strong to weak on the anthocyanins retention rate were drying pressure, heating plate temperature and material thickness.

Comprehensive optimization conditions of vacuum freeze drying: Combined with regression model mathematical analysis, the optimum conditions were analyzed by using Design-Expert 8.0.6 Trial: drying pressure 53.2 Pa, material thickness 7.882 mm and heating plate temperature 50.2° C and the predictive value of drying rate, anthocyanins retention rate and moisture content was 19.51 g/(g·h), 83.50 and 4.11%.

In order to verify the reliability of the model and consider the actual value of operation, the optimum conditions were modified as follows: drying pressure 50 Pa, material thickness 8 mm and heating plate temperature 50°C. The experiment was repeated three times in this condition and the average value of drying rate, anthocyanins retention rate and moisture content was 19.37 g/(g·h), 82.88 and 4.13% and the predictive value of drying rate, anthocyanins retention rate and moisture content was 19.51 g/(g·h), 83.50 and 4.11%. Deviation of actual value and predictive value was small and the vacuum freeze drying parameter was reliable by using response surface methodology.

Observation of the surface morphology and particle structure: As was shown in Fig. 7, *Acanthopanax sessiliflorus* fruits anthocyanins after vacuum freeze



Fig. 7: SEM image of anthocyanins particles

drying forms flaky particles with uniform particle size and surface smoothly. The surface tension of the particles increased with the complete particle morphology, inter-particle agglomeration was slight, which beneficial to the adsorption of water molecules. Anthocyanins also had a good solubility and stability after dissolved in water.

CONCLUSION

In this study, anthocyanins derived from *Acanthopanax sessiliflorus* fruits were prepared by vacuum freeze drying technology. The results showed that the optimum conditions for vacuum freeze drying were: drying pressure 50 Pa, material thickness 8 mm and heating plate temperature 50° C. Under the optimum conditions, the drying rate was 19.37 g/(g·h), the anthocyanins retention rate was 82.88% and the moisture content was 4.13%. Particle structure analyses showed that the anthocyanins were flaky particles with uniform particle size and smooth surface.

ACKNOWLEDGMENT

This research was funded by Science and Technology Department of Jilin Province (No. 20140520182JH) and Education Department of Jilin Province (No. 2014556).

REFERENCES

- Choi, Y.E., S.K. Ko, K.S. Lee and E.S. Yoon, 2002. Production of plantlets of eleutherococcus sessiliflorus via somatic embryogenesis and successful transfer to soil. Plant Cell Tiss. Org., 69(2): 201-204.
- Hong, S.S., J.S. Hwang, S.A. Lee, B.Y. Hwang, K.W. Ha, K.R. Ze, R.S. Seung, J.S. Ro and K.S. Lee, 2001. Isolation and quantitative analysis of acanthoside D from a canthopanacis cortex. Saengyak Hakhoechi, 32: 316-321.

- Jaouad, B., R. Hassan and D. Amadou, 2007. Chlorogenic acid, a polyphenol from *Prunus domestica* (Mirabelle), with coupled anxiolytic and antioxidant effects. J. Neurol. Sci., 262: 77-84.
- Jeong, S., B. Yang, Y. Jeong, K.S. Rao and C. Song, 2007. Isolation and characterization of biopolymers extracted from the bark of a canthopanax sessiliflorus and their anticomplement activity. J. Microbiol. Biotechn., 17(1): 21-28.
- Ji, H., A. Du, L. Zhang, S. Li, M. Yang and B. Li, 2012. Effects of drying methods on antioxidant properties and phenolic content in white button mushroom. Int. J. Food Eng., 8(3): 1-14.
- Jiang, W., W. Li, L. Han, L. Liu, Q. Zhang, S. Zhang, T. Nikaido and K. Koike, 2006. Biologically active triterpenoid saponins from acanthopanax senticosus. J. Nat. Prod., 69(11): 1577-1581.
- Jun, W.J., H.S. Seong, H. Chun, E.J. Lim, K.I. Kim and H.Y. Cho, 2007. Determination of antioxidative potentials of a canthopanax sessiliflorus (Rupr. and Maxim.) seem. in differentiated HL-60 cells. Food Chem., 105(3): 1557-1563.
- Jung, S.K., T.G. Lim, J.E. Kim, S. Byun, G.W. Kim, J.N. Choi, C.H. Lee, B.Y. Kim, K.W. Lee and H.J. Lee, 2012. Inhibitory effect of ERK1/2 and AP-1 by hyperoside isolated from a canthopanax sessiliflorus. Food Chem., 130(3): 915-920.
- Kim, H., J.S. Kim, S.H. Lee, S.J. Lee, G.P. Lee, S.S. Kang, S.H. Cho and D.S. Cheoi, 2006. Quantitative analysis of lignans in the fruits of Acanthopanax species by HPLC. Food Sci. Biotechnol., 15: 778-780.
- Lee, D.Y., K.H. Seo, D.S. Lee, Y.C. Kim, L.S. Chung, G.W. Kim, D.S. Cheoi and N.I. Baek, 2012. Bioactive 3,4-seco-triterpenoids from the fruits of *Acanthopanax sessiliflorus*. J. Nat. Prod., 75: 1138-1144.
- Lee, D.Y., K.H. Seo, R.H. Jeong, S.M. Lee, G.S. Kim, H.J. Noh, S.Y. Kim, G.W. Kim, J.Y. Kim and N.I. Baek, 2013. Anti-inflammatory lignans from the fruits of *Acanthopanax sessiliflorus*. Molecules, 18(1): 41-49.
- Lee, S., B.K. Kim, S.H. Cho and K.H. Shin, 2002a. Phytochemical constituents from the fruits of *Acanthopanax sessiliflorus*. Arch. Pharm. Res., 25(3): 280-284.
- Lee, S., J. Ji, K.H. Shin and B.K. Kim, 2002b. Sessiline, a new nitrogenous compound from the fruits of *Acanthopanax sessiliflorus*. Planta Med., 86(10): 939-941.
- Lee, S.H., Y.S. Lee, S.H. Jung, J. Ji, K.H. Shin, B.K. Kin and S.S. Kang, 2003. Antitumor and immunostimulating activities of *Acanthopanax sessiliflorus* fruits. Nat. Prod. Sci., 9: 112-116.

- Sagar, V.R. and P. Suresh, 2010. Recent advances in drying and dehydration of fruits and vegetables: A review. J. Food Sci. Technol., 47(1): 15-26.
- Shao, X.R., Z.S. Ma, Y.Q. Niu, Y. Dai and L.P. Wang, 2014. Optimization of ultrasonic-microwave synergistic extraction of anthocyanins from *Acanthopanax sessiliflorus* (Rupr. et Maxim.) seem. J. Chem. Pharmaceut. Res., 6(3): 959-963.
- Viljanen, K., P. Kylli, E.M. Hubbermann, K. Schwarz and M. Heinonen, 2005. Anthocyanin antioxidant activity and partition behavior in whey protein emulsion. J. Agr. Food Chem., 53(6): 2022-2027.
- Wang, H.B., M.G. Nair, G.M. Strasburg, Y.C. Chang, A.M. Booren, J.I. Gray and D.L. DeWitt, 1999. Antioxidant and antiinflammatory activities of anthocyanins and their aglycon, cyanidin, from tart cherries. J. Nat. Prod., 62(2): 294-296.
- Wang, R., M. Zhang and A.S. Mujumdar, 2010. Effects of vacuum and microwave freeze drying on microstructure and quality of potato slices. J. Food Eng., 10(1): 131-139.
- Wang, S.Y. and H.S. Lin, 2000. Antioxidant activity in fruits and leaves of blackberry, raspberry and strawberry varies with cultivar and developmental stage. J. Agr. Food Chem., 48(2): 140-146.
- Woo, S.Y., K.W. Kwon, J.C. Lee, J.H. Choi and B.S. Kang, 2003. Recovery of net photosynthetic rate after SO2 fumigation in *Quercus acutissima*, *Pinus densiflora*, *Populus alba×glandulosa*, and *Acanthopanax sessiliflorus*. Photosynthetica, 41(2): 319-320.
- Wu, M., X. Hu, B. Ge, G. Zhao and Z. Wang, 2010. Process optimisation of the bitter gourd (*Momordica charantia*) concentrated juice preparation for a freeze-dried powder. Int. J. Food Sci. Tech., 45(12): 2553-2559.
- Yang, C., Q. An, Z. Xiong, Y. Song, K. Yu and F. Li, 2009. Triterpenes from *Acanthopanax sessiliflorus* fruits and their antiplatelet aggregation activities. Planta Med., 75(6): 656-659.
- Yang, C., Z. Wang, P. Song, Y. Xiao, Y. Meng, Y. Wang, H. Jiang and H. Kuang, 2013. Monoterpenoids from *Acanthopanax sessiliflorus* fruits. Molecules, 18(3): 3043-3049.
- Yoshizumi, K., K. Hirano, H. Ando, Y. Hirai, Y. Ida, T. Tsuji, T. Tanaka, K. Satouchi and J. Terao, 2006. Lupane-type saponins from leaves of *Acanthopanax sessiliflorus* and their inhibitory activity on pancreatic lipase. J. Agr. Food Chem., 54(2): 335-341.