

Browning and Moisture Sorption Characteristics of *Rubus coreanus* Prepared by Different Drying Methods

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건조방법에 따른 복분자 분말의 갈변 및 흡습 특성

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Abstract

The effects of drying methods on the browning and moisture sorption characteristics of *Rubus coreanus* were studied. Fruits were steamed for 5 min at 100°C, dried by sun drying, infrared drying, or freeze drying, and powdered to a size of 20 mesh. Color values were measured and equilibrium moisture contents (EMC) were determined at 20°C, over a range of water activity (a_w) from 0.11 to 0.90. The browning indices L^* and a^* values were higher and lower, respectively, in freeze-dried *Rubus coreanus* compared with other samples. The b^* value was greatest in freeze-dried *Rubus coreanus*. EMC tended to increase with increasing a_w values, and a particularly sharp increment was observed above 0.75 a_w . The EMC of freeze-dried *Rubus coreanus* was significantly higher compared with the EMC of sun-dried and infrared-dried fruit at constant a_w . The moisture sorption isotherms showed a typical sigmoid shape, and the Halsey, Kuhn, and Oswin models were the best fits for the sun-dried, infrared-dried, and freeze-dried powder isotherms, respectively. With respect to monolayer moisture content, the Guggenheim-Anderson-Boer (GAB) equation showed that the various drying methods yielded very different results, with monolayer moisture contents of 0.005 g H₂O/g dry solid in infrared-dried and 0.019 g H₂O/g dry solid in sun- and freeze-dried powders, respectively. These results indicate that the drying method affects the browning and moisture sorption characteristics of *Rubus coreanus*.

Key words : *Rubus coreanus*, browning, sorption isotherm, modelling, monolayer moisture content

Introduction

Rubus coreanus are commonly preserved and distributed in dried form due to the poor storability of fresh fruit, and used as a material for processed food and traditional medicine in Korea (1). Its biological activities and therapeutic effects such as antimicrobials and similar action of estrogen(1,2), antioxidation(3), immunomodulation(4), antitumour(5),

antinociceptive and antiinflammatory(6) have been reported. Due to their perceived health-stimulating properties and medicinal effects, today the use of *Rubus coreanus* is widespread and growing in the healthy food industry.

Rubus coreanus has been generally dried under the sun or hot-air drying after harvest. However, these conventionally dried products have some undesirable quality properties in comparison with infrared and freeze drying(7). Efficient drying process of *Rubus coreanus* which can restrained the loss of quality is demanded when processing of *Rubus coreanus*. Drying technology in food processing is done by

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lowering the moisture content related to microbial spoilage and enzymatic deterioration, but also for the development of new food types(8). In spite of these advantages, the drying process may cause physicochemical alterations of the food and finally degrade its quality. The properties such as chemical components, texture, density, porosity, color and sorption characteristics are known to be readily affected by the drying process(9-12). These property changes induced by drying process depend upon drying methods as well as plant materials. Thus, previous studies have to be conducted to determine the optimum methods for each plant materials.

The quality properties of dried food are affected by their physical, chemical and microbiological stability. This stability is mainly a consequence of the relationship between the equilibrium moisture content(EMC) and water activity(a_w) at a constant temperature and pressure. Color change such as browning may be the major quality deterioration of foods and considered as index of severity of the developed chemical reactions during drying(13). The moisture sorption isotherms, consisting of a graphical representation of a_w against EMC, are unique for individual foods, and the knowledge of that is useful to solve food processing design problems, predict energy requirements, and determine proper storage and transportation conditions. Prediction of moisture sorption is based on fitting various sorption models to the experimental data. Many mathematical models are developed to describe the sorption behaviour of fruits and vegetables including apples(12), garlic(14), raisins, currants, figs, prunes and apricots(15), potatoes(16), onions(17), and ginger(18). However, investigations on the color changes and the moisture sorption isotherms of *Rubus coreanus* as affected by the drying methods have not been carried out.

The objectives of this study were to determine the browning degree and the moisture sorption isotherms of *Rubus coreanus*, to establish a sorption isotherms model to fit the data in order to predict the moisture sorption isotherms and to calculate the content of water in the monomolecular layer.

Materials and Methods

Plant materials

Unripe fruits of *Rubus coreanus* were harvested on 15 July 2007 from a hill in the Jiri mountain region of Korea, transported immediately to the laboratory, graded according to size and defects, and kept at 4°C until use.

Drying methods

Rubus coreanus was steamed for 5 min and dried using sun, infrared or freeze drying methods. In sun drying, the raw materials were exposed to sun for about 20 days until the moisture content reached a constant level. For infrared and freeze drying, the raw materials were put into a cabinet-type infrared dryer(IRD-250, Woori Sci., Korea) kept at 60°C air temperature with constant airflow rate or a freeze dryer(FreeZone, Labconco., USA), respectively, and dried until the moisture content of the samples reached 1 to 3%. The dried products were ground by a mill(J-NCM, JISICO, Korea) and sieved to size of 20 mesh.

Color measurement

The color of powdered samples was measured with a colorimeter(CR-200, Minolta Co., Osaka, Japan) fitted with CIE illuminant C and 8 mm diameter measuring aperture, which had been calibrated with a standard white plate($L^*=97.79$, $a^*=-0.38$, $b^*=2.05$). Three reading of L^* (lightness), a^* (+, redness; -, greenness), and b^* (+, yellowness; -, blueness) were recorded for each sample.

Experimental procedure for sorption isotherms

The equilibrium moisture contents of *Rubus coreanus* were determined at 20°C by a static gravimetric method. A sample of 2 g was placed in a petri-dish inside desiccators containing saturated salt solution of water activity from 0.11 to 0.90. The desiccators were placed in a temperature controlled incubator. The samples were allowed to equilibrate until there was no discernible mass change, as evidenced by constant mass values. The moisture content of each sample was determined gravimetrically by drying in a convection oven at 105°C for 24 hr. Each experiment was carried out in triplicate.

Analysis of data

The relationship between the experimental data for the equilibrium moisture content and water activity was adjusted to the following six equations(19-24). The coefficients in each equation were obtained using linear and non-linear least squares regression analyses.

$$\text{Bradley equation} \quad \ln \frac{1}{a_w} = K_2 K_1^M$$

$$\text{Caurie equation} \quad \ln M = \ln A - r a_w$$

$$\text{Halsey equation} \quad a_w = \exp\left(-\frac{a}{M^n}\right)$$

$$\text{Henderson equation} \quad 1 - a_w = \exp(-KM^n)$$

$$\text{Kuhn equation } M = \frac{a}{\ln a_w} + b$$

$$\text{Oswin equation } M = a \left(\frac{a_w}{1 - a_w} \right)^n$$

where M is the equilibrium moisture content, a_w is the water activity, and other alphabets are the constants.

The suitability of the equations has been evaluated and compared using the coefficient of determination (R^2) and the mean relative percentage deviation modulus (P) was used, P is defined by

$$P(\%) = \frac{100}{n} \sum_{i=1}^n \frac{|M_i - M_{pi}|}{M_i}$$

where M_i is the experiment equilibrium moisture content, M_{pi} is the predicted equilibrium moisture content, and n is the number of data points. In general, when the value of P is below 10%, the model should be considered as acceptable(25).

The moisture content in the monolayer was determined by the following Brunauer-Emmett-Teller (BET) equation(26) and Guggenheim-Anderson-Boer (GAB) equation(27).

$$\text{BET equation } \frac{a_w}{m(1 - a_w)} = \frac{1}{m_1 C} + \frac{C - 1}{m_1 C} a_w$$

$$\text{GAB equation } m = \frac{m_1 C k a_w}{(1 - k a_w)(1 - k a_w + C k a_w)}$$

where a_w is the water activity, m is the equilibrium moisture content, and m_1 is the monolayer moisture content. While C and k are the constants.

Results and discussion

Comparison of browning

The color of foods is important to their acceptability. It is well known that the measurement of L^* and a^* values can be used as a suitable method for the evaluation of browning degree in fruits and vegetables(28). Lower L^* and higher a^* values indicate that the produce is more brownish. The L^* , a^* and b^* values of sun-dried, infrared-dried, or freeze-dried *Rubus coreanus* are shown in Fig. 1. The L^* value of the freeze-dried powder was largely higher when compared to the values of both infrared-dried and sun-dried powder. And the L^* value of infrared-dried powder was

slightly higher than that of sun-dried powder. The a^* value showed a minus value in the freeze-dried powder but a plus value in sun-dried and infrared-dried powder. The a^* value of sun-dried powder was higher than that of infrared-dried powder. These results may be due to preventing of certain reactions related to browning development such as enzymatic browning and non-enzymatic browning reactions by a freeze drying(13). The b^* value of the freeze-dried *Rubus coreanus* was about two times higher in comparison with the values of both sun-dried and infrared-dried *Rubus coreanus*. This result is similar to that of *Ponciri fructus* in relation to drying methods(29). The above results suggested that the drying method significantly affected the browning reactions that occurred during sun drying, and freeze drying had prevented the effect of browning development during the drying of *Rubus coreanus*.

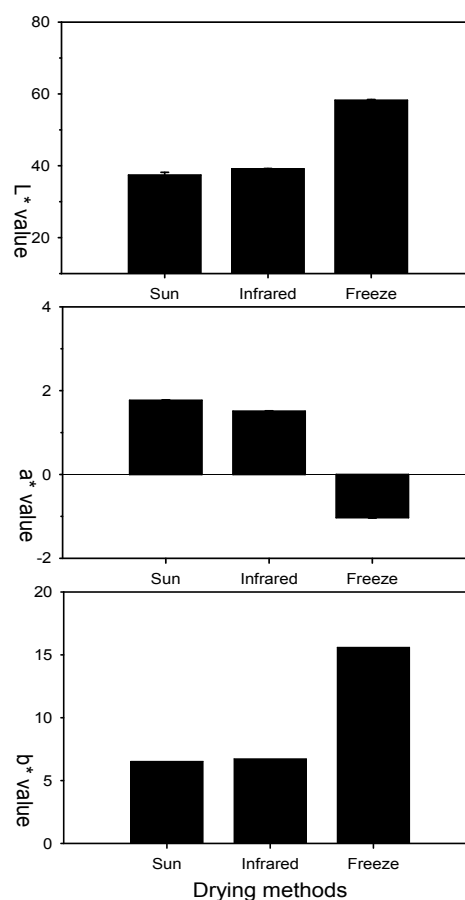


Fig. 1. Color values of *Rubus coreanus* in relation to drying methods.

Sorption isotherms

The sorption isotherms relating a_w and EMC of *Rubus coreanus* at 20°C as affected by drying methods are plotted

in Fig. 2. The sorption isotherms were typical sigmoid shape and resembled the isotherms observed in fruits and vegetables such as apples, garlic, raisins, currants, figs, prunes and apricots, potatoes, onion, and ginger(12,14-18). The EMC of all samples tended to increase with increasing a_w , especially the EMC sharply increased at above 0.75 a_w . These may be due to an inability of the food materials to maintain the vapor pressure at unity with decreasing moisture content. Rao *et al.*(30) reported that the steep acceleration of sorption during high water activities in case of *chhana podo* could be due to the solubilization of sugars. At constant water activity, the EMC of the freeze-dried *Rubus coreanus* was significantly higher when compared to the values of the sun-dried and infrared-dried. That is, the EMC of the freeze-dried, infrared-dried and sun-dried increased from 0.03, 0.02 and 0.01(d.b.) at 0.11 a_w to 0.22, 0.18 and 0.18(d.b.) at 0.90 a_w , respectively. These results may be caused by more porous structure of the freeze-dried *Rubus coreanus* than the sun-dried and infrared-dried. It is well known that the freeze-dried foods have strong water binding capacity for high pore texture(9).

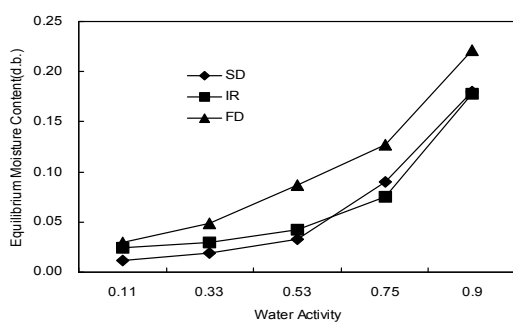


Fig. 2. Experimental sorption isotherms for *Rubus coreanus*.

SD: sun drying, IR: infrared drying, FD: freeze drying.

Fitting of sorption isotherm models

Bradley, Caurie, Halsey, Henderson, Kuhn, and Oswin models were fitted for describing the moisture sorption isotherm of *Rubus coreanus* at 20°C. The values of coefficients obtained by a regression analysis in each equation are presented in Table 1. Among the fitted models, Caurie, Halsey, Kuhn and Oswin equations were found to be good in predicting the experimental moisture sorption data and to characterize the sorption behaviour for the entire range of water activity and the *Rubus coreanus* dried by all drying methods, as exhibited by high R^2 values. Generally the fitness of the models depends upon the chemical components of foods(31). In case of the dried *Rubus coreanus*, we suggest

that the suitability of the models is related to drying methods.

The P values of the Caurie, Halsey, Kuhn and Oswin models were calculated by the above mentioned equations and are shown in Table 2. Generally, Halsey model was the best model to describe the experimental sorption data for *Rubus coreanus* throughout the entire range of water activity.

Table 1. Estimated coefficients of sorption equations fitted to experimental sorption data of *Rubus coreanus*

Drying method	Bradley				Caurie				Halsey			
	ln K ₂	ln K ₁	SigF	R ²	ln A	r	SigF	R ²	ln a	n	SigF	R ²
Sun	0.46	-15.99	0.01	0.90	-4.99	3.45	0.00	0.97	-3.98	-1.06	0.00	0.99
Infrared	0.58	-17.10	0.03	0.85	-4.22	2.44	0.01	0.91	-4.86	-1.44	0.00	0.98
Freeze	0.96	-15.25	0.00	0.96	-3.81	2.47	0.00	0.99	-4.35	-1.49	0.00	0.98
Drying method	Henderson				Kuhn				Oswin			
	ln K	n	SigF	R ²	b	a	SigF	R ²	ln a	n	SigF	R ²
Sun	2.72	0.99	0.01	0.90	0.01	-0.02	0.00	0.98	-3.27	0.66	0.00	0.96
Infrared	3.40	1.30	0.03	0.83	0.02	-0.02	0.00	1.00	-3.01	0.47	0.01	0.92
Freeze	3.20	1.45	0.00	0.96	0.04	-0.02	0.00	0.95	-2.57	0.48	0.00	0.99

Table 2. Mean relative percentage deviation modules of sorption equations fitted to experimental sorption data of *Rubus coreanus*

Drying method	P (%)			
	Caurie	Halsey	Kuhn	Oswin
Sun	6.85	3.63	6.03	6.13
Infrared	9.25	5.03	0.74	8.87
Freeze	1.88	3.25	12.20	1.36
Average	5.99	3.97	6.32	5.45

Halsey model gave P values ranging from 3.25% to 5.03%, with average values of 3.97%. There was difference in the P values among the drying methods. That is, P values of Caurie, Halsey and Oswin models were lowest in the freeze-dried *Rubus coreanus* and highest in the infrared-dried. However, the P value of Kuhn model was the lowest in the infrared-dried *Rubus coreanus* and highest in the freeze-dried. The P value of 12.20% in the case of freeze drying means that the Kuhn model cannot represent the sorption isotherm of the freeze-dried *Rubus coreanus*. In comparison of the sorption isotherm models according to P values, Halsey model was the best fit model in the case of the sun-dried *Rubus coreanus*, Kuhn model was the best fit model in case of the infrared-dried, and Oswin model was the best fit sorption model in case of the freeze-dried.

Experimental data for *Rubus coreanus* were compared to sorption isotherms predicted by the Caurie, Halsey, Kuhn

and Oswin models as shown in Fig. 3. All models, with the exception of the Kuhn model in case of freeze drying, graphically gave good fits to experimental data over the range of water activities investigated regardless of drying methods.

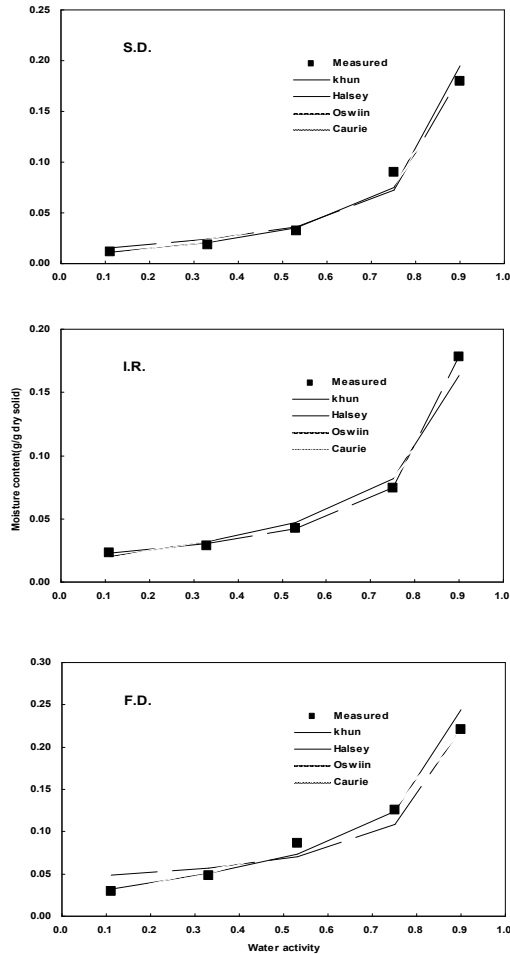


Fig. 3. Comparison of the experimental sorption data and sorption isotherms of *Rubus coreanus* fitted by sorption models. Symbols represent experimental values, lines represent the predicted isotherm.

SD: sun drying, IR: infrared drying, FD: freeze drying.

Content of monolayer moisture content

The moisture content in the monolayer is determined as index of optimum water content for the stability of various dried foods. Monolayer moisture contents calculated from BET and GAB equations are presented in Table 3. According to R² values, BET equation could be used only for infrared-dried *Rubus coreanus*. However, GAB equation could be used for all *Rubus coreanus* dried by three methods. Monolayer moisture content of infrared-dried *Rubus coreanus* was slightly lower than the value from the both sun-dried and freeze-dried *Rubus coreanus*. This lower in monolayer

moisture contents can be explained by considering the structural difference induced by the drying methods(32). Iglesias and Chirife(33) reported that monolayer moisture content is closely related with the number of active sites available of foods for binding water. This result suggested that drying methods affect the monolayer moisture content of *Rubus coreanus*.

Table 3. Coefficients and monolayer moisture contents of *Rubus coreanus* determined by BET and GAB equations

Drying Method	BET equation				GAB equation				
	C	m1	SigF	R ²	C	k	m1	SigF	R ²
Sun	6.18	0.02	0.02	0.86	-1.89	0.64	0.02	0.00	0.99
Infrared	-28.01	0.02	0.00	0.99	-1.88	0.75	0.01	0.02	0.98
Freeze	-9.85	0.03	0.02	0.88	-1.92	0.65	0.02	0.00	0.99

In conclusions, the browning degree of freeze-dried *Rubus coreanus* was lower compared to that of dried with other methods. The moisture sorption isotherms of *Rubus coreanus* obtained at 20°C showed a sigmoid shape depend on the drying methods. The best fit model was affected by the drying methods. That is, Halsey, Khun, and Oswin models gave the best fit with experimentally determined data of sun-dried *Rubus coreanus*, infrared-dried, and freeze-dried, respectively. Monolayer moisture contents determined from GAB model were lower in infrared-dried *Rubus coreanus* than in the both sun-dried and freeze-dried.

요 약

건조방법이 복분자 분말의 갈변 및 흡습 특성에 미치는 영향을 조사하기 위하여, 신선 복분자를 천일, 적외선 및 동결 건조한 다음 분쇄하고 이 분말의 색도와 수분활성도 (a_w) 0.11-0.90 범위에서 평형수분함량(EMC)을 각각 측정하였다. 갈변지표인 L*값과 a*값은 동결 건조한 복분자 분말이 다른 방법으로 건조한 분말보다 높고 낮은 값을 나타내었고, 황색도인 b*값은 동결 건조 분말에서 가장 높은 수준을 보였다. EMC는 a_w의 증가와 함께 증가하는 경향을 보였으며 특히, 0.75 a_w 이상에서 급격하게 증가하였다. 동일한 a_w에서 분말의 EMC는 동결건조품이 천일과 적외선 건조품 보다 유의적으로 높게 나타났다. 복분자 분말의 흡습곡선은 전형적인 sigmoid 형태를 보였고, 천일건조 분말은 Halsey model, 적외선 건조 분말은 Kuhn model, 그리고 동결 건조 분말은 Oswin model이 적합도가 가장 높았다. 단분자층 수분함량은 GAB 방정식으로 설명할 수 있었으며, 적외선 건조 분말에서 0.005 g H₂O/g dry solid, 천일

및 동결 건조 분말에서 0.019 g H₂O/g dry solid를 각각 나타내었다. 이로써 건조방법은 복분자 분말의 갈변과 흡습 특성에 영향을 미치는 것으로 확인되었다.

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