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Research Article

Improvement of Properties of Gelatinization and Retrogradation of Adzuki Bean Starch by Adding Glycolipid

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To improve the storage quality of adzuki bean paste from starch aging, the effects of glycolipid addition on the properties of gelatinization and retrogradation properties of adzuki flour obtained by the retrogradation inside adzuki bean during storage and transportation are investigated. The results indicate the significant differences in the gelatinization and the distribution of starch granules for different varieties of beans. The higher gelatinization temperature of adzuki flour results in the lower enthalpy of gelatinization with an undulating tendency of the retrogradation due to the short-term aging of amylose and long-term aging of amylopectin within adzuki flour. In the early stage of aging, the coexistence of protein, fat, and starch inside adzuki flour hinder the aging of starch. An Avrami equation is introduced to characterize the aging process of the starch–protein–fat–water system of different varieties of adzuki flours. The nucleation mode of the crystal nucleus has a significant effect on the aging rate of different varieties of adzuki flour. The fat in the system fails to demonstrate a significant effect on the nucleation mode of starch retrogradation. The addition of glycolipids increases the gelatinization enthalpy of adzuki flour. Glycolipids can reduce the aging degree of adzuki flour, and the inhibition effect on the retrogradation of sucrose monoester is greater than that of maltose monoester. The research results provide the valuable guidance for the high-quality processing and storage of adzuki bean paste from starch aging.

1. Introduction

Adzuki bean contains rich nutritional components including 25% protein, 1% fat, and 50% starch [1]. The products and paste of adzuki bean with unique flavor and taste have a large consumer market in Asian countries due to their unique flavor and taste. The common peeled bean paste weakens the primary taste with the loss of nutrients and low yield of product. The whole bean filling processed by steam cooking may retain the bean skin and its rich components, which combines the consumption concept of "whole grain food" with "whole food nutrition." For the adzuki beans as fillings, the internal starch granules are gelatinized to form a special flavor and mouthfeel. The aging occurrence of gelatinized starch colloid is due to hydrogen bonding at low temperature (4°C), where the starch aging rate reaches the highest level [2] at storage for 24 hours. The complex content of amylose, lipid-amylose, and the amylopectin structure dominates its gelatinization and aging characteristics [1]. The starch aging of the adzuki bean paste seriously affects the quality and shelf life of bean products. Sugar esters are emulsifiers of an ester compound from the esterification reaction of sugar, fatty acid, and fatty acid ester. These esters have physiological implications in material transmission and energy transfer on the cell membrane substance, biodegradability, and safety in relation to human health and the environment [3]. Sugar esters contain at least four fatty acids with hydrophilic and lipophilic groups to form emulsifying properties, which form the spiral structure of the granule. Sugar esters are microbial surface-active molecules that are composed of a carbohydrate unit linked to a single or multiple fatty acid(s) as lipid compounds containing glycosyl ligands and amphiphilic molecule. The emulsifying properties of glycolipids impart great potential to glycolipids in areas of food preservation and processing, which can serve as sustainable substitutes for modification of food properties [4]. Sugar esters, as emulsifiers, can compete with starch for water molecules and interfere with starch molecules in parallel with antiaging role. Sugar esters may delay starch aging due to the complex of sugar esters-amylose complexes restraining the crystallization of amylose in cooling [5] leading to a soft fresh-keeping effect for starch foods.

In adzuki bean seeds, the fat or emulsifiers in starch significantly affect its dissolution, swelling, gelatinization, and rheological properties to determine the texture of food [6–9]. Considering the presence of protein and fat, the gelatinization and aging of adzuki bean starch may explain the changes in texture and rheological characteristics of bean paste from adzuki bean. Differential scanning calorimetry (DSC) has been widely applied to measure the heat flow properties of starchy foods under heating, such as lotus seed starch [10], corn starch [11], wheat starch, and wheat flour [12]. DSC may be employed to determine the gelatinization and aging characteristics of adzuki bean flour under cooling storage conditions.

Based on the above-mentioned, the research objectives were developed as follows: (1) to investigate the gelatinization and aging characteristics of adzuki bean flour, (2) to analyze the effect of sugar esters on inhibiting the aging of adzuki bean starch, and (3) to develop a method of using sugar esters to alleviate the starch aging of adzuki bean paste.

2. Materials and Methods

2.1. Materials and Reagents. The following varieties of Adzuki beans were selected as experimental samples including Longyin 09-05 (Heilongjiang, China), Zhonghong No.7 (Heilongjiang, China), Jiahong No.1 (Heilongjiang, China), Xiaofeng No.2 (Heilongjiang, China), Baoqing Red (Heilongjiang, China), Baohang Red (Heilongjiang, China), Tianjin Red (Heilongjiang, China), and Pearl Red (Heilongjiang, China). These varieties of adzuki beans have large planting areas in China with an obvious difference of components and are widely consumed in food processing, representing the typical varieties of adzuki beans used in China.

Ultrapure water was prepared in the laboratory. Maltose monoester (HLB 11) and sucrose monoester (HLB 11) were purchased from Liuzhou Qualcomm Food Chemical Co. (Liuzhou, China).

2.2. Sample Preparation. Two hundred grams of adzuki beans for each variety was immersed in water for 16 h to absorb sufficient water at room temperature (22°C), and then the peel of soaked beans was removed by hand. The peeled adzuki beans were put into an electric oven (DHG-9123A, Shanghai Yiheng Scientific Instrument Co., Ltd, Shanghai, China) at a constant temperature of 40°C for 16 h and then were pulverized using a cyclone mill (CT410, Flowserve; Millipore Corporation, US) at 80-mesh sieve to obtain adzuki bean powder for further experiments. The dimensional characteristics of the resulting powder were considered as sphericity with a diameter of 0.10-0.18 mm. It was followed by 5 g adzuki bean flour that was placed into an Erlenmeyer flask, and then, 50 mL ultrapure water was added, shaken for 30 min, and stood for 1 h. All the precipitates were transferred into a 25 mL centrifuge tube. The ultrapure water was added into the centrifuge tube with shaking for 5 min, and the mixture was centrifugated for 3000 r/min. The supernatant liquid was sucked out to scrape off the upper protein and impurities. The operation was repeated 3 times to obtain the adzuki bean starch (deproteinized) sample, then placed in an electric thermostatic drying oven (40°C) for 16 h.

The test samples of adzuki bean starch were prepared using the following procedure: (1) adding ethanol into the deproteinized starch of adzuki bean shaking for 5 min, then centrifuging at 3000 r/min for 10 min, and removing the water in the sample; (2) adding petroleum ether into the dehydrated starch and then shaking for 5 min; furtherly adding ethanol and shaking for 5 min, then centrifuging at 3000 r/min for 10 min to gain the deproteinized and degreased sample of adzuki bean starch. (3) The bean starch sample was placed into a drying oven at 40°C for 16 h. The dried starch samples were removed for further experiments.

2.3. Measurement of Properties

2.3.1. Measurement of Element Components of Adzuki Bean Flour. The element components of selected adzuki bean, including crude protein, fat content, starch content, amylose, and amylopectin content, were measured by using the AOAC Official Method (2002) [13].

2.3.2. Measurement of the Gelatinization and Retrogradation (Aging) of Adzuki Bean Flour via DSC Method. A differential scanning calorimeter (DSC-Q2000, TA Company, US) was employed to measure the gelatinization and aging of adzuki bean flour. The DSC pan used was a TA T-ZERO solid aluminum crucible ($\varphi 5.4 \times 2.6$ mm, Shanghai Qunhong Instrument Equipment Co., Ltd, Shanghai, China). The DSC measurement procedure was as follows: (1) A total of 0.003 ± 0.0001 g of each sample, including adzuki bean flour, deproteinized starch, and deproteinized and degreased starch, was placed in the DSC crucible; (2) the crucible loading sample was sealed by using a supporting aluminum coat to avoid water evaporation; (3) the scanning temperature range was determined to be within 30~95°C with a scanning rate of 10°C/min; (4) the gelatinized sample was stored in a refrigerator at 4°C for the setting duration, and retrogradation parameters were measured for the different adzuki bean flours (with bean coat removed), starch, and defatted starch; (5) a scan with the same temperature of gelatinization was performed, where three samples of same starch placed in the refrigerator once for retrogradation were measured, and the average value was reported as the final value. The starch retrogradation (aging) degree was calculated by using

$$\text{Aging degree}/\% = \frac{\text{starch aging enthalpy at time } t}{\text{gelatinization enthalpy required for starch gelatinization}} \times 100. \quad (1)$$

2.3.3. Fitting Treatment of the Avrami Equation. The change of the crystallization rate of polymer under isothermal conditions can be described by using the Avrami equation [14] as shown in

$$1 - \theta = \frac{E_L - E_t}{E_L - E_0} = \exp\left(-kt^n\right). \quad (2)$$

According to Eq. (2), an improved Avrami equation was developed as shown in

$$\log\left(-\ln\frac{E_L - E_t}{E_L - E_0}\right) = \log k + n \log t, \quad (3)$$

where E_0 is the enthalpy value at time zero (J/g); E_t is the enthalpy value at time t(J/g); E_L is the maximum aging enthalpy value (14 d in the study) (J/g); θ is the crystallinity at a certain time t(%); t is the crystallization time (d); n (Avrami exponent) —an integer representing nucleation and nucleation growth patterns, as the slope of the line, is calculated by plotting $\log\left[-\ln\left(E_L - E_t\right)/\left(E_L - E_0\right)\right]$ vs. $\log t$; k—crystallization rate constant, as the intercept of the curve, is related to the density of the nucleus and the one-dimensional growth rate of the crystal (min^{-1}) [15].

2.4. Experimental Design. To analyze the effects of sugar esters on the gelatinization and aging characteristics of adzuki bean flour, Longyin 09-05 adzuki bean flour with obvious starch aging was selected as the research material. A total of 0.003 ± 0.0001 g of the whole powder of the selected variety was placed into the DSC test crucible with 0.25% sucrose ester/maltose ester solution and ultrapure water in the crucible. The total mass of the sample reached 0.015 ± 0.0001 g, and the suspension was prepared by adding the ultrapure water in sugar ester content of 0, 0.03%, 0.06%, 0.09%, 0.12%, and 0.15%. The sealed sample was stored in a 4°C refrigerator for further experiments.

2.5. Statistical Analysis. The experimental data were expressed as mean ± standard deviation ($n = 3$). Correlation analysis for data t was conducted by using Pearson's method in SPSS software (V. 22.0, SPSS Inc., Chicago, US), and correlation significance was defined at the 0.05 level. The data plotting of this study and curve fitting were performed by Origin software (V. 9.1, Origin LabCorp., North Hampton, MA, US).

3. Results and Analysis

3.1. Gelatinization Characteristics of Different Varieties of Adzuki Bean Flour, Starch, and Defatted Starch. Table 1 and Table 2 indicate the gelatinization and aging DSC parameters of different varieties of adzuki bean flour, starch, and defatted starch. The DSC thermogram of eight kinds of adzuki bean flour is provided in Figure 1.

According to the results from Table 1 and Table 2, the characteristic temperatures of different varieties of adzuki bean flour related to gelatinization and the aging process are obtained as an initial gelatinization temperature of 62.22~68.60°C and the peak temperature in 68.40~75.45°C, the final temperature of gelatinization in 77.97~82.57°C, the gelatinization enthalpy value in 2.35~5.49 J/g, and the initial gelatinization temperature range of starch in 58.93~62.43°C. These results indicate that the gelatinization and aging of adzuki bean is dependent on varieties [16]. The beginning temperature of starch gelatinization of the selected adzuki bean varieties is 62.22-68.60°C. The high protein and low fat within adzuki bean influence the gelatinization of its interior starch. The protein content has a significant effect on the heat transfer of starch paste to determine gelatinization degree. The result is attributed to the greater proportion of gluten protein in the starch system, resulting in the lower gelatinization enthalpy of starch and the higher gelatinization peak temperature [17]. Gluten protein can reduce starch gelatinization enthalpy and increase the initial gelatinization temperature and peak temperature of starch [18]. The formation of a complex structure of protein and starch, coupled with the water absorption of the protein, affects the movement of the free water in the starch gelatinization system [19].

According to the results from Table 1, the differences in the gelatinization characteristics of eight adzuki bean flours are mainly reflected in the gelatinization temperature range and the gelatinization enthalpy value. The initial gelatinization temperature of adzuki bean varieties from low (62.22°C) to high (68.60°C) is as Zhong red, Pearl Red, Longyin, Tianjin Red, Baohang Red, Baoqing Red, Xiaofeng, and Jiahong. The enthalpy of different varieties of adzuki bean from low (2.35 J/g) to high (5.49 J/g) was Jiahong, Baoqinghong, Tianjin Red, Xiaofeng, Longyin, Zhonghong, Baohanghong, and Pearl Red. These differences in gelatinization temperatures for the different varieties are attributed to the difference in the content of components in each variety as shown in Table 3 and Table 4. The amylose content and protein content have a significantly negative correlation with the initial gelatinization temperature and gelatinization enthalpy, respectively. However, no significant correlation of the components was found for the different varieties with peak gelatinization temperature in DSC properties in view of statistical analysis.

3.2. Correlation Analysis of the Gelatinization and Aging Parameters of Adzuki Bean Flour and Components. To explain the correlation between the content of each component and the gelatinization characteristics in different varieties of adzuki bean flour, Table 3 and Table 4 show the protein, fat, starch, amylose, and amylopectin content of adzuki bean flour and their correlation analysis with DSC gelatinization parameters.

As shown in Table 4, the amylose content has a significantly negative correlation with the initial gelatinization

Table 1: Gelatinization parameters of different adzuki bean flour (bean coat removed), starch, and defatted starch under DSC measurement.

Sample category	Adzuki bean flour				Starch				Defatted starch			
	T_0/°C	T_P/°C	T_C/°C	ΔH/J/g	T_0/°C	T_P/°C	T_C/°C	ΔH/J/g	T_0/°C	T_P/°C	T_C/°C	ΔH/J/g
Longyin 09-05	63.71 ± 0.45^{c}	69.36 ± 0.32^{c}	79.27 ± 0.65^{c}	3.66 ± 0.13^{c}	60.60 ± 0.57^{c}	66.60 ± 0.62^{d}	72.4 ± 0.83^{d}	9.57 ± 0.16^{c}	61.55 ± 0.48^{c}	67.09 ± 0.28^{c}	73.59 ± 0.68^{c}	9.96 ± 0.22^{a}
Zhonghong No.7	62.22 ± 0.74^{d}	68.40 ± 0.54^{c}	78.80 ± 0.63^{c}	3.91 ± 0.11^{b}	59.23 ± 0.46^{d}	66.35 ± 0.58^{d}	72.93 ± 0.72^{d}	9.41 ± 0.30^{d}	60.44 ± 0.82^{d}	66.47 ± 0.75^{d}	71.40 ± 0.57^{d}	9.08 ± 0.19^{c}
Jiahong No.1	68.60 ± 0.59^{a}	75.36 ± 0.65^{a}	80.87 ± 0.42^{b}	2.35 ± 0.09^{e}	62.33 ± 0.32^{a}	72.11 ± 0.89^{a}	77.16 ± 0.59^{a}	10.43 ± 0.11^{a}	62.90 ± 0.25^{a}	72.46 ± 0.21^{a}	78.02 ± 1.03^{a}	9.91 ± 0.23^{ab}
Xiaofeng No.2	68.31 ± 0.31^{a}	75.45 ± 0.72^{a}	82.57 ± 0.47^{a}	3.44 ± 0.16^{d}	58.93 ± 0.57^{d}	65.60 ± 0.73^{e}	74.40 ± 0.82^{bc}	8.96 ± 0.53^{d}	59.77 ± 0.74^{e}	66.11 ± 0.57^{d}	74.72 ± 0.89^{b}	8.74 ± 0.61^{c}
Baoqing Red	67.63 ± 0.59^{a}	75.01 ± 0.49^{a}	80.72 ± 0.51^{b}	2.40 ± 0.14^{e}	59.34 ± 0.69^{d}	67.54 ± 0.41^{b}	75.71 ± 0.26^{a}	10.23 ± 0.26^{b}	60.12 ± 0.43^{e}	68.72 ± 0.64^{b}	76.91 ± 0.72^{a}	9.34 ± 0.52^{b}
Baohang Red	65.95 ± 0.62^{b}	72.11 ± 0.63^{b}	78.56 ± 0.73^{c}	4.08 ± 0.18^{b}	62.43 ± 0.43^{a}	67.90 ± 0.57^{b}	73.76 ± 0.37^{c}	9.90 ± 0.19^{b}	62.54 ± 0.16^{a}	68.03 ± 0.73^{bc}	73.70 ± 0.23^{c}	10.51 ± 0.12^{a}
Tianjin Red	64.56 ± 0.43^{c}	71.51 ± 0.36^{b}	77.97 ± 0.89^{d}	2.47 ± 0.20^{e}	60.99 ± 0.87^{c}	68.01 ± 0.32^{b}	75.33 ± 0.72^{a}	8.71 ± 0.23^{e}	61.17 ± 0.37^{c}	68.62 ± 0.24^{b}	75.29 ± 0.67^{b}	9.89 ± 0.67^{ab}
Pearl Red	63.52 ± 0.72^{c}	71.68 ± 0.66^{b}	79.75 ± 0.69^{bc}	5.49 ± 0.29^{a}	61.32 ± 0.76^{b}	67.20 ± 0.13^{c}	75.12 ± 0.63^{ab}	9.67 ± 0.31^{c}	61.84 ± 0.56^{b}	68.06 ± 0.61^{b}	73.74 ± 0.82^{c}	10.55 ± 0.24^{a}

Note: T_0, T_P, and T_C represent the starting temperature, peak temperature, and ending temperature of the gelatinization process, respectively, and ΔH is the enthalpy value. The superscript letters represent the significance of the difference.

Table 2: Retrogradation parameters of different adzuki bean flour (bean coat removed), starch, and defatted starch under DSC measurement.

Sample category	Adzuki bean flour				Starch				Defatted starch			
	T_0/°C	T_P/°C	T_C/°C	ΔH/J/g	T_0/°C	T_P/°C	T_C/°C	ΔH/J/g	T_0/°C	T_P/°C	T_C/°C	ΔH/J/g
Longyin 09-05	53.51 ± 0.62^c	62.89 ± 0.31^b	71.13 ± 0.24^a	1.27 ± 0.12^a	48.55 ± 0.32^d	58.13 ± 0.23^c	64.98 ± 0.41^c	3.47 ± 0.31^e	51.58 ± 0.51^c	60.30 ± 0.31^d	67.45 ± 0.19^a	$4.98 \pm .12^d$
Zhonghong No.7	48.76 ± 1.02^e	59.96 ± 0.67^c	71.24 ± 0.11^a	0.98 ± 0.04^b	47.86 ± 0.67^e	57.95 ± 0.60^d	65.05 ± 0.32^c	3.89 ± 0.15^d	52.13 ± 0.43^c	60.63 ± 0.23^c	67.28 ± 0.24^b	4.85 ± 0.51^c
Jiahong No.1	57.93 ± 0.62^a	65.40 ± 0.26^a	71.00 ± 0.32^b	1.12 ± 0.03^a	48.50 ± 0.19^d	59.29 ± 0.49^b	68.43 ± 0.54^a	4.72 ± 0.11^b	51.93 ± 0.72^c	57.59 ± 0.67^e	65.30 ± 0.56^c	5.49 ± 0.13^b
Xiaofeng No.2	51.82 ± 0.84^d	59.41 ± 0.79^c	70.48 ± 0.19^b	0.37 ± 0.01^d	49.24 ± 0.41^c	58.78 ± 0.61^b	65.40 ± 0.21^b	3.14 ± 0.43^e	53.06 ± 0.34^b	61.04 ± 0.29^b	65.09 ± 0.63^c	3.79 ± 0.41^e
Baoqing Red	52.94 ± 0.67^c	63.61 ± 0.14^b	69.56 ± 0.44^c	0.29 ± 0.09^d	49.83 ± 0.32^c	59.09 ± 0.37^b	65.48 ± 0.57^b	4.01 ± 0.13^c	53.22 ± 0.17^b	61.44 ± 0.41^b	65.55 ± 0.47^c	4.30 ± 0.27^e
Baohang Red	56.90 ± 0.41^a	63.62 ± 0.29^b	70.63 ± 0.82^b	0.42 ± 0.12^d	49.48 ± 0.24^c	59.41 ± 0.42^b	64.42 ± 0.82^c	5.30 ± 0.08^a	53.02 ± 0.15^b	62.70 ± 0.13^a	66.62 ± 0.34^b	6.38 ± 0.11^a
Tianjin Red	55.66 ± 0.82^b	63.57 ± 0.38^b	71.05 ± 0.23^b	0.40 ± 0.11^d	51.01 ± 0.11^b	60.23 ± 0.11^a	65.50 ± 0.44^b	4.33 ± 0.14^c	53.29 ± 0.21^a	62.38 ± 0.16^a	65.64 ± 0.30^c	5.35 ± 0.07^b
Pearl Red	54.11 ± 0.35^c	63.36 ± 0.42^b	70.94 ± 0.20^b	0.80 ± 0.19^c	51.62 ± 0.17^a	60.46 ± 0.19^a	65.27 ± 0.61^b	4.26 ± 0.17^c	53.77 ± 0.14^a	62.13 ± 0.21^a	67.45 ± 0.21^a	4.77 ± 0.39^c

Note: T_0, T_P, and T_C represent the starting temperature, peak temperature, and ending temperature of the gelatinization process, respectively, and ΔH is the enthalpy value. The superscript letters represent the significance of the difference.

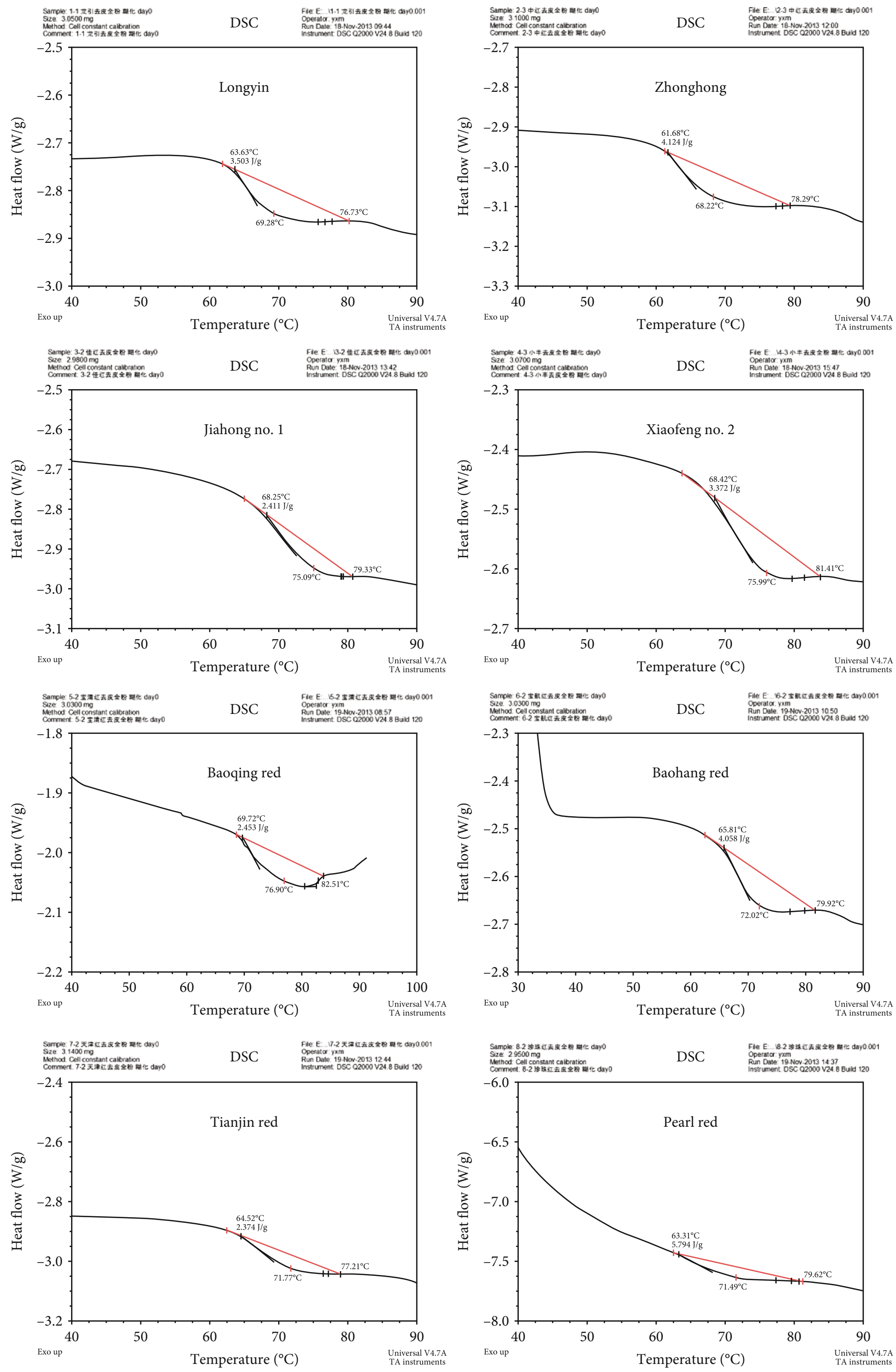

Figure 1: DSC thermogram of 8 kinds of adzuki bean flour.

temperature at the correlation coefficient of -0.65 at $p < 0.05$. This indicates that the higher the amylose content in the sample, which may result in the lower initial gelatinization temperature, the easier the gelatinization of the bean when cooking. The protein content has a significant negative correlation with the gelatinization enthalpy value at the correlation coefficient of -0.70 at $p < 0.05$. The higher the protein content, the lower the endothermic enthalpy value of starch gelatinization. This result was attributed to the ratio reduction of protein content to starch content in the same mass to reduce gelatinization enthalpy. Starch gelatinization is an endothermic process. The heat energy is consumed through the disintegration of the starch crystal structure, the swelling of starch granules, and the diffusion of starch molecules in the starch granules of the starch gelatinization.

Figure 2 shows the changing trend of the aging degree of adzuki bean flour after storage at 4°C for 14 days. The aging degree of adzuki bean flour for selected varieties increased

Table 3: Component contents of different adzuki bean varieties (species) flour with bean coat removed.

Variety	Protein (drybase, %)	Fat (dry base, %)	Starch (dry base, %)	Amylose (dry base, %)	Amylopectin (dry base, %)
Longyin 09-05	27.73 ± 0.52^b	1.08 ± 0.01^c	56.20 ± 0.41^d	11.60 ± 0.71^d	44.60 ± 0.41^d
Zhonghong No.7	23.80 ± 0.63^d	0.82 ± 0.11^e	59.28 ± 0.13^a	13.94 ± 0.24^a	45.34 ± 0.23^c
Jiahong No.1	25.78 ± 0.25^c	0.95 ± 0.03^d	56.82 ± 0.27^c	12.37 ± 0.12^b	44.45 ± 0.44^d
Xiaofeng No.2	28.67 ± 0.17^a	1.14 ± 0.12^c	55.52 ± 0.64^e	11.77 ± 0.56^c	43.75 ± 0.21^e
Baoqing Red	28.04 ± 0.20^b	1.14 ± 0.02^b	57.35 ± 0.21^b	10.86 ± 0.79^d	46.49 ± 0.24^b
Baohang Red	25.91 ± 0.54^c	1.38 ± 0.04^a	59.20 ± 0.07^a	12.25 ± 0.19^c	46.95 ± 0.18^a
Tianjin Red	28.75 ± 0.11^a	1.24 ± 0.09^b	56.48 ± 0.43^d	12.71 ± 0.13^b	43.77 ± 0.82^e
Pearl Red	22.83 ± 0.76^e	1.37 ± 0.05^a	59.10 ± 0.15^a	13.04 ± 0.21^b	46.06 ± 0.11^b

Note: The superscript letters represent the significance of the difference.

Table 4: Pearson's correlations between basic contents and DSC parameters.

Parameter	Initial gelatinization temperature (T_0/°C)	Peak gelatinization temperature (T_P/°C)	Gelatinization enthalpy ΔH (J/g)
Protein content	0.49	0.38	-0.70*
Fat content	0.04	0.16	0.39
Starch content	-0.51	-0.43	0.60
Amylose content	-0.65*	-0.58	0.44
Amylopectin content	-0.12	-0.08	0.40

Note: *Significant correlation at 0.05 level; **very significant correlation at 0.01 level (variety of Longyin 09-05).

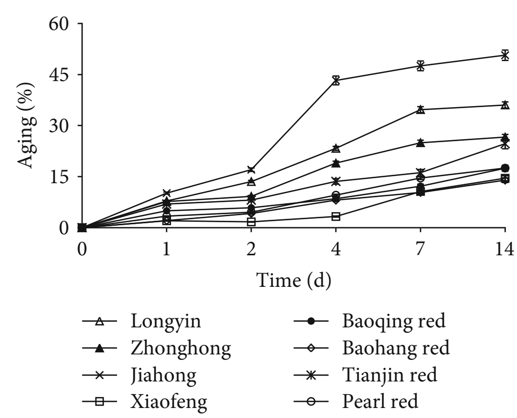

Figure 2: The retrogradation (aging) degree of different varieties with storage time at 4°C.

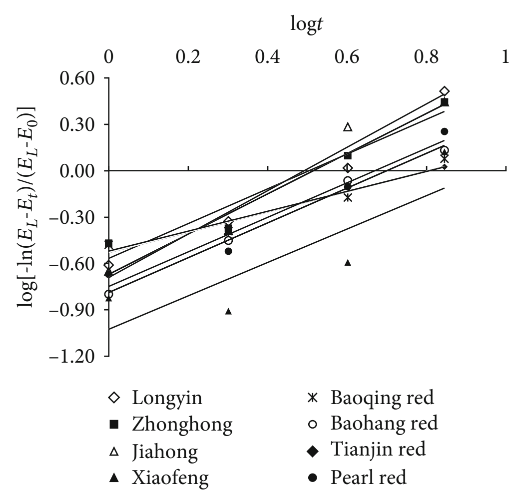

Figure 3: Plot of $\log[-\ln(E_L - E_t)/(E_L - E_0)]$ vs. $\log t$ at 4°C of different adzuki bean flours (bean coat removed).

slowly from the beginning of storage till 2 days, followed by increasing significantly till the seventh day, and then kept slowly aging trends ($p < 0.01$). These results were explained as the temperature gradually decreases, the molecular motion of the gelatinized starch weakens, and the molecules tend to automatically arrange in order, close together, and tightly aggregate through hydrogen bonding. The microcrystalline bundles no longer present their original state but form a disordered combination, forming particles larger than the colloid and precipitate. Starch aging is divided into two stages: short-term aging and long-term aging. Short-term starch aging, caused by the recrystallization of amylose, generally occurs within 10 h; long-term aging is a relatively slow process taking place over several days due to the recrystallization of amylopectin side chains [20]. The amylose of adzuki bean starch is prone to aging [21]. According to the short-term aging stage for adzuki bean flours from eight varieties, the lower average aging degree (around 10%) at $p < 0.05$ indicates the greater inhibition of the adzuki bean of the protein and fat in powdered adzuki bean on the starch aging.

The data from Figure 2 were fitted based on Equation (3) as the plots of $\log[-\ln(E_L - E_t)/(E_L - E_0)]$ vs. $\log t$ for different adzuki bean flours shown in Figure 3. The fitted results are presented in Table 5. From Table 5, the different n-values (n is the Avrami index) indicate that the varieties of adzuki bean powder showed significant differences in nucleation and growth mode. Under normal circumstances, $n \leq 1$ corresponds to the one-dimensional, two-dimensional, and three-dimensional crystal growth methods, and the nucleation

Table 5: Avrami parameters of different adzuki bean flour (bean coat removed).

Variety	n	k	R^2
Longyin 09-05	1.30	0.512	0.930
Zhonghong No.7	1.12	0.567	0.972
Jiahong No.1	1.40	0.501	0.959
Xiaofeng No.2	1.08	0.359	0.840
Baoqing Red	0.66	0.589	0.978
Baohang Red	1.13	0.455	0.993
Tianjin Red	0.64	0.595	0.951
Pearl Red	1.12	0.473	0.974

Note: n, k, and R^2 represent Avrami index, rate constant, and regression correlation square, respectively (variety of Longyin 09-05).

Table 6: Pearson's correlations between basic component contents and Avrami parameters.

Parameter	n	k	Gelatinization enthalpy $\varDelta H$ (J/g)	Enthalpy of aging $\varDelta H$ (J/g)
n	1			
k	0.795**	1		
$\varDelta H$	0.307	0.038	1	
Enthalpy of aging	0.755*	0.951**	0.168	1
Protein content	-0.439	-0.378	-0.725*	-0.444
Fat content	-0.305	-0.653*	0.404	-0.472
Starch content	0.075	0.038	0.633*	0.055
Amylose content	0.220	0.273	0.465	0.358
Amylopectin content	-0.078	-0.172	0.416	-0.208

Note: *Significantly correlated at the 0.05 level; **very significantly correlated at the 0.01 level (variety of Longyin 09-05).

method is instant nucleation. At $1 < n \leq 2$, the corresponding nucleation method is mainly spontaneous nucleation [22, 23]. The crystallization rate constant k value of each sample has an obvious difference, suggesting that the k value could well amplify the crystallization rate of starch in the complex system of starch–protein–fat–water by means of the characterization of the Avrami equation for the aging process of starch.

Table 6 presents the Pearson bivariate correlation analysis between the protein, fat, starch, amylose, and amylopectin content of eight adzuki bean flours and Avrami parameters. The Avrami index n shows a very significant positive correlation with the rate constant k, with a correlation coefficient of 0.795, which indicates that the nucleation method of crystal nucleation has a very significant effect on the aging rate. Both the n and k values have a significantly positive correlation with the aging enthalpy, with the correlation coefficients of 0.755 at $p < 0.05$ and 0.951 at $p < 0.01$, respectively. The gelatinization enthalpy value has a very significant negative correlation with the protein content with a correlation coefficient of -0.725, which is consistent with existing results [24, 25]. The gelatinization enthalpy is positively correlated with starch content with a correlation coefficient of 0.633. The content of amylose has a weak positive correlation with the values of n and k [26]. Although the proportion of fat content in the system is low, a negative correlation with the n value indicates that fat has a significant effect on adzuki bean flour. The nucleation mode of starch aging in the powder has an inhibitory effect on the aging of starch [27].

3.3. Effect of Sugar Esters on the Gelatinization and Aging Characteristics of Adzuki Bean Flour. Sugar esters are a mixture formed by the esterification reaction of sucrose and fatty acids. Sucrose contains 8 -OH groups and can be esterified to produce various products from monoesters to 8-esters [28]. Due to sugar as the hydrophilic group and fatty acids as the lipophilic group, sugar ester biosurfactant molecules are composed of hydrophobic and hydrophilic groups [22, 23]. The essence of starch retrogradation is that the molecular movement of gelatinized starch molecules slows down when the temperature drops. At this time, the branches of amylose molecules and amylopectin molecules tend to be parallel and close under the interaction of hydrogen bonds to reform the structure of mixed microcrystalline bundles. The structure is very similar to the structure of the original starch granules in a disorderly combination, not in a radial form. Due to the existence of hydrogen bonds in the molecules, firm intermolecular association occurs in the starch paste of adzuki beans to weak water solubility. If the cooling rate of starch paste is too fast to rearrange amylose molecules, the starch gel forms with a bundle structure called by starch retrogradation [29]. However, sugar ester molecular weight (molecular weight of 317.507) is lower than that of starch, which is easy to enter α-chains in amylose molecules as the D-hexacyclic glucose connection through 1,4-neneneba glycosidic bond and the branch position of amylopectin through α-1,6-neneneba glycosidic bond [30]. The hydrogen bonds between the 1,4 glycosidic chains

Table 7: Effects of different addition of sugar esters on the gelatinization temperature and enthalpy of adzuki bean flour (bean coat removed).

Volume of addition	Sucrose monoester				Maltose monoester			
	T_0/°C	T_P/°C	T_C/°C	ΔH/J/g	T_0/°C	T_P/°C	T_C/°C	ΔH/J/g
Gelatinization	66.79 ± 0.09^b	73.56 ± 0.23^a	77.41 ± 0.57^c	3.66 ± 0.14^b	66.79 ± 0.03^b	73.56 ± 0.22^a	77.41 ± 0.57^d	3.66 ± 0.12^c
0.03%	66.79 ± 0.24^b	73.36 ± 0.44^b	80.38 ± 0.45^a	4.17 ± 0.53^b	67.19 ± 0.31^a	73.64 ± 0.72^a	79.81 ± 0.38^b	4.28 ± 0.33^b
0.06%	66.74 ± 0.56^b	73.59 ± 0.73^a	79.95 ± 0.38^a	4.14 ± 0.25^b	67.80 ± 0.04^a	73.45 ± 1.03^a	78.55 ± 0.45^c	4.39 ± 0.14^b
0.09%	67.27 ± 0.11^a	73.65 ± 0.24^a	79.94 ± 0.51^a	4.84 ± 0.31^a	66.78 ± 0.71^b	73.55 ± 0.11^a	79.68 ± 0.21^b	4.31 ± 0.17^b
0.12%	67.04 ± 0.41^b	73.01 ± 0.09^b	78.80 ± 0.13^b	4.96 ± 0.22^a	67.10 ± 0.62^a	73.28 ± 0.87^a	79.84 ± 0.44^b	5.14 ± 0.10^a
0.15%	67.70 ± 0.17^a	73.63 ± 0.17^a	77.98 ± 0.14^c	5.12 ± 0.10^a	67.00 ± 0.39^b	73.74 ± 0.92^a	81.42 ± 0.51^a	5.33 ± 0.22^a

Note: T_0, T_P, and T_C represent the starting temperature, peak temperature, and ending temperature of the gelatinization process, respectively, and ΔH is the enthalpy value (variety of Longyin 09-05).

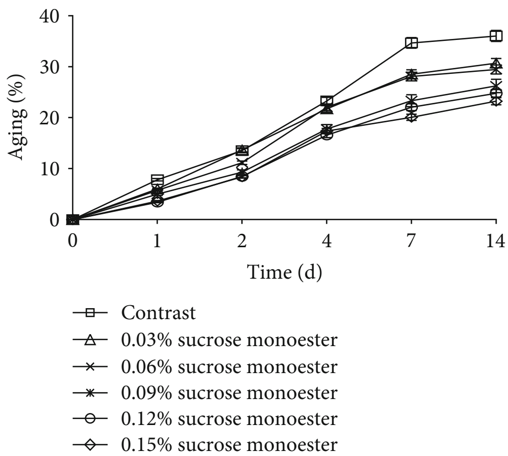

Figure 4: Effects of sucrose monoester on the retrogradation degree of adzuki bean flour (bean coat removed) with storage time at 4°C (variety of Longyin 09-05).

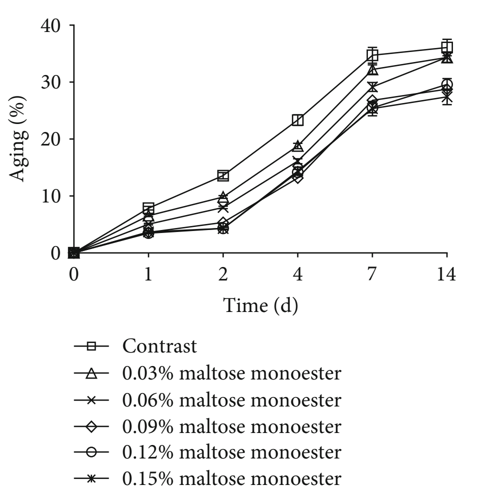

Figure 5: Effects of maltose monoester on retrogradation degree of adzuki bean flour (bean coat removed) with storage time at 4°C (variety of Longyin 09-05).

in adzuki bean paste are easy to bind with the starch molecule, which prevents the amylose and amylopectin molecule from aging or retrogradation [31]. Table 7 shows the thermal properties of adzuki bean powder with the

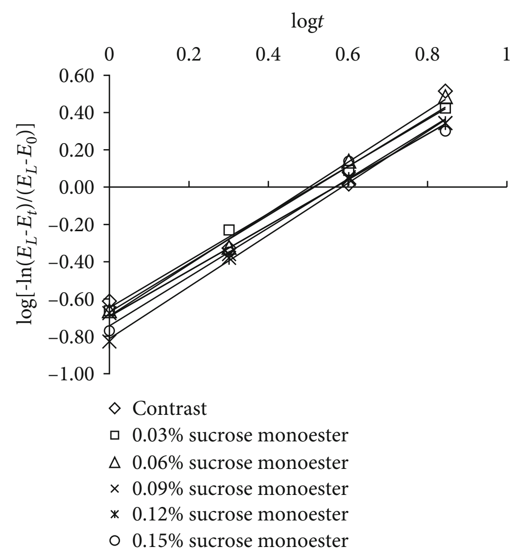

Figure 6: Plots of log $[-\ln (E_L - E_t)/(E_L - E_0)]$ against log storage time at 4°C of adzuki bean flour (bean coat removed) with the amount of sucrose monoester added (Variety of Longyin 09-05).

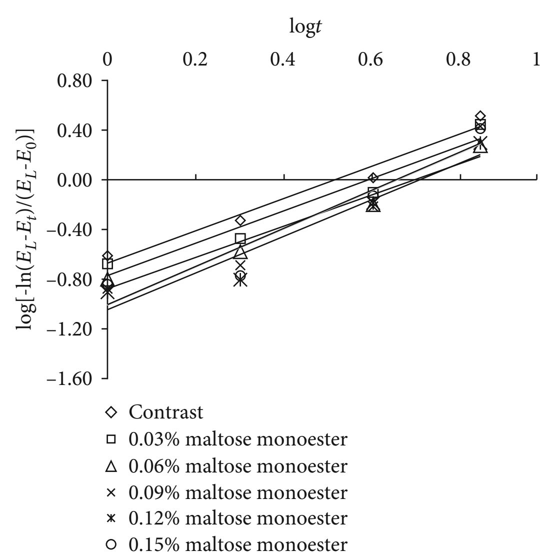

Figure 7: Plots of log $[-\ln (E_L - E_t)/(E_L - E_0)]$ against log storage time at 4°C of adzuki bean flour (bean coat removed) with maltose monoester added (variety of Longyin 09-05).

Table 8: Avrami parameters characterizing the effects of addition of sucrose monoester and maltose monoester.

Volume of addition	Sucrose monoester				Maltose monoester			
	Equation	n	k	R^2	Equation	n	k	R^2
Gelatinization	y=1.3008x-0.6702	1.3008	0.5116	0.9681	y=1.3008x-0.6702	1.3008	0.5116	0.9681
0.03%	y=1.2619x-0.6461	1.2619	0.5240	0.9966	y=1.3023x-0.7704	1.3023	0.4628	0.9388
0.06%	y=1.3751x-0.6912	1.3751	0.5009	0.9960	y=1.2589x-0.8762	1.2589	0.4163	0.9599
0.09%	y=1.2227x-0.6938	1.2227	0.4996	0.9974	y=1.5309x-1.0061	1.5309	0.3656	0.9269
0.12%	y=1.3903x-0.8125	1.3903	0.4437	0.9985	y=1.4764x-1.0453	1.4764	0.3516	0.9255
0.15%	y=1.3133x-0.7437	1.3133	0.4753	0.9809	y=1.5379x-1.0050	1.5379	0.3660	0.9087

Note: n, k and R^2 are Avrami index, rate constant and regression correlation square, respectively. Variety of Longyin 09-05.

additions of sucrose monoester and maltose monoester. Sugar esters combine starch molecules to form a complex with high thermal stability [32] and promote cross-linking and increase the starch gelatinization temperature. The addition of sugar esters in the adzuki bean powder–water system significantly increases the gelatinization enthalpy value due to the endothermic process of starch gelatinization in the system, while sugar esters hinder the progress of the gelatinization reaction.

Figures 4 and 5 present the changing trend of the aging degree of adzuki bean powder added with different mass fractions of sucrose monoester and maltose monoester under cold storage at 4°C, respectively. The results suggest that adding sugar esters can reduce the aging degree of adzuki bean flour. As the mass fraction of sugar esters in the adzuki bean flour system increases, the aging degree of adzuki bean flour decreases. Therefore, the aging inhibitory effect of sucrose monoester is greater than that of maltose monoester at the same mass of added sugar esters.

The Avrami equation is used for linear regression analysis of the thermodynamic parameters of adzuki bean powder with sugar esters added and the control group stored at 4°C for 14 days, as shown in Figures 6 and 7. The aging kinetic equation of adzuki bean powder is obtained in k from the intercept of the line and n value from the slope, as shown in Table 8. The nucleation mode of red adzuki powder with sugar esters ($1 < n \leq 2$) has a similar trend to the recrystallization behavior of amylopectin [33]. In the crystallization system, crystal growth is often multidimensional, whose growth kinetics are related to the growth dimension and nucleation mode [34]. From the point of view of the crystallization rate constant k, the k value of adzuki bean powder without sugar esters is the highest, the crystallization growth rate is the fastest, and the rejuvenation rate is the highest; however, the addition of a certain concentration of sugar ester significantly reduces the rejuvenation degree of adzuki bean powder, indicating the capability of sugar ester to delay the rejuvenation of adzuki bean powder. As the concentration of sugar ester increases, the crystallization rate constant k decreases due to the polyhydroxyl structure of sugar ester. Hydrogen bond formations through the interactions of hydroxyl group and water molecules can retain bound water in the starch molecules. The expansion of network scaffold of sugar esters can cause the spatial barrier of starch molecules, which reduce the crystalline order of starch molecules. The hydroxyl group of a sugar ester is stronger than that of the hydroxyl group of starch molecules in terms of generating hydrogen bonds [20, 35], which hinder the formation of microcrystalline bundles of starch molecule hydrogen bonds to further delay the aging of starch molecules [27]. However, the amount of sugar ester increasing to 0.15% causes the increase of the crystallization rate constant k. This may be because the interaction between sugar ester and starch is also affected by water and protein in the system. It is a more complicated process than that in the environment of pure starch, which needs further study.

4. Conclusion

For the eight representative varieties of adzuki bean, the aging degree is slow for the first two days of storage, then significantly increases in the rest of the seven days of storage with a relatively stable case. In the short-term aging stage of adzuki bean flour, the average aging degree (only about 10%) is significantly lower than that of the aging speed of adzuki bean starch. The amylose content and protein content in different varieties of adzuki bean influence their gelatinization temperatures due to the significantly negative correlation with the initial gelatinization temperature and gelatinization enthalpy, respectively. The Avrami equation may characterize the aging process within the starch–protein–fat–water system for different varieties of adzuki bean flour, which indicate that the presence of sugar esters has an aging inhibitory effect on the system. The addition of sugar esters obviously influences the aging of adzuki bean starch, which reduce the aging degree. The addition of sugar ester to 0.15% causes the increase of the crystallization rate constant of adzuki beans due to the interaction between sugar ester and starch depending on water and protein in the system. The aging inhibitory effect of sucrose monoester is greater than that of maltose monoester. The general practical recommendations for further use of adzuki bean starch adding glycolipid as the enhancement of texture quality in viscosity and consistency, extended shelf life keeping a fresh taste in filling food with adzuki bean.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors have declared no conflicts of interest for this article.

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