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Research Article

Physicochemical and Morphological Properties of Extruded Adlay (*Coix lachryma-jobi* L) Flour

Gaopeng Zhang, Yangyue Ding, Chunlei Ni, Qingfeng Ban, Li Xu, Liandong Guo, and Jianjun Cheng

College of Food Science, Northeast Agricultural University, Harbin, China

Correspondence should be addressed to Jianjun Cheng; jjcheng@neau.edu.cn

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The effects of extrusion treatment on the structure and properties of adlay (Job's tears) were investigated. Adlay flour was extruded through a twin-screw extruder with different parameters, including barrel temperature (80–160°C), moisture content (19–27%), and screw speed (170–330 rpm). The results showed that although the expansion index increased with increasing temperature, an increase in moisture content significantly decreased the EI ($p < 0.05$). Extrusion improved the water solubility index and water absorption index of adlay flour ($p < 0.05$). Furthermore, analysis of the gelating properties revealed that the structure and function of adlay flour had radically changed. After extrusion, the viscosity of the adlay flour decreased (peak viscosity decreased by more than 1000 cP), and its fluidity increased. The rheological data were modeled by the Herschel–Bulkley model. X-ray diffraction experiments showed that extrusion contributed to a decrease in relative crystallinity. Scanning electron microscopy revealed that extrusion damaged the basic structure of adlay flour, causing holes and pits on the extrudate surface. Compared to the native adlay flour, the extrusion resulted in significantly changing the pasting, gelating, thermal, rheological, and morphological properties of adlay flour. In conclusion, the extrusion can alter adlay characteristics, but it is necessary to choose appropriate conditions to attain the desired properties.

1. Introduction

Adlay (*Coix lachryma-jobi* L. var. *ma-yuen* Stapf) has been applied as a dietary supplement for humans and used as a nourishing food for more than a thousand years. Recently, a considerable body of literature has accumulated regarding the nutritional ingredients and physicochemical properties of adlay [1]. Some studies have demonstrated that adlay is beneficial to human health [2–4]. Adlay is rich in starch, protein, lipids, and nonstarch polysaccharides and can function as both medicine and food [5]. However, adlay use has some drawbacks, including cooking difficulty, poor mouthfeel, and poor flowability of the adlay paste. Extrusion is a convenient and effective method used to improve adlay properties.

Extrusion is a versatile and efficient technology that uses high-temperature and short-time physical treatment and has a wide range of applications in grain processing. However, only a few studies have reported the impact of extrusion parameters on the physicochemical properties of adlay flour [6]. Therefore, it is necessary to further characterize the effect of extrusion on adlay flour. The extrusion process includes combining, mixing, cooking, and formation of solid materials under high temperature and pressure. Extrusion technology has been increasingly utilized to produce convenience products such as puffed snack foods, instant rice, and breakfast cereals. Extrusion technology also provides options for further processing of a variety of adlay foods.

Extrusion allows starch conversion, protein denaturation, lipid oxidation, increase of the soluble dietary fiber content, deepened color, decreased enzyme activation, and microbial reduction, all of which are dependent upon the extrusion conditions and varieties of the raw materials [7–10]. Moisture content, barrel temperature, and material residence time are factors that determine the density, porosity, and expansion of the extrudate, which can be altered to improve the edibility of the extrudates [11]. Scanning electron microscopy observation of whole-grain barley extrudates produced with lower shear force showed a less dense structure and less ungelatinized starch [12]. Extrusion can increase the extrudate swelling and water absorption capacities when oats are added at levels above 15% [13]. Several studies have shown that extrusion can modify starch-based food materials to produce various functional food products. The effect of extrusion parameters on the production quality of starch-based materials has been widely studied [14, 15].

The present study is aimed at providing a basis for the systematic production of adlay flour for breakfast cereals and as an ingredient for functional foods, which could give indications on the final extrudate properties. To achieve different properties of adlay flour, the flour was extruded with diverse conditions during the processing treatment, including barrel temperature, screw speed, and moisture content. Afterward, native adlay and extrudates were analyzed for pasting, rheology, and retrogradation properties using a rapid viscosity analyzer (RVA), rotational rheometer, and differential scanning calorimetry (DSC), respectively. This study will contribute to a better understanding of the mechanisms of extrusion as they apply to adlay flour to improve the utilization efficiency of adlay in industrial production.

2. Materials and Methods

2.1. Material. Adlay seeds (*Coix lachryma-jobi* L. var. *ma-yuen* Stapf) were obtained from Liaoyang, Liao Ning Province, China (N41°16′, E123°12′). All the chemicals used in this study are of analytical grade.

2.2. Twin-Screw Extrusion. Extrusion was performed using a corotating intermixing twin-screw extruder (DS56-III Experiment-type Screw Extruder, Jinan Saixin Machinery Co., Ltd.). The barrel diameter and L/D ratio were 25 mm and 16 : 1, respectively. Materials were fed into the feed port through a screw feeder (Model Jd180, China). The screw installed in the barrel performs the function of mixing and grinding. The feeder was calibrated to obtain a feed rate of 25 kg/h (wet basis) for the trials. In the single-factor experiments, one factor changed, while the other factors were unchanged. The process moisture content conditions were 19, 21, 23, 25, and 27% (wt/wt) at the same feed rate. The sample was conditioned with different amounts of moisture in the first zone of the barrel by manually adjusting the precalibrated water feed pump. The feeder speed and screw speed were each maintained at 170, 210, 250, 290, and 330 rpm. The temperature in the third- and fourth-barrel sections was set to 80, 100, 120, 140, and 160°C.

To produce flours, the extrudates and native adlay were each dried at a constant temperature oven at 40°C for 12 h and then smashed with a pulverizer (Huangcheng HC-150T2, Lvke Food & Machinery Co., Ltd., China) until the granular size was less than 200 μm. The flours were sealed in airtight low-density polyethylene (LDPE) bags and preserved at 4°C until further research [16]. All experiments were performed in duplicate.

2.3. Expansion Index (EI). The EI was obtained via dividing the extrudate diameter by a 3.0 mm die diameter. The extrudate diameter was obtained at 3 distinct locations for 10 random samples with a precise ruler by vernier [17].

2.4. Water Absorption Index (WAI) and Water Solubility Index (WSI). The WAI and WSI were calculated by the same method as that used for grains [18]. First, 2.5 g flour (m_1) was added to 25 mL deionized water, stirred at 25°C for 30 min, and then centrifuged at 3000 $\times g$ for 15 min. The WAI is defined as the weight (m_2) of the solid gel after removing the weight of the sample supernatant. The supernatant was then placed in a glass beaker and dried to a constant weight at 105°C. The WSI was calculated by recording the dry solid weight (m_3) recovered from the supernatant and calculating the percentage of the dry initial sample [19]. The WAI and WSI were calculated by the following formulas:

$$\begin{aligned} \text{WAI}\left(\text{g}\cdot\text{g}^{-1}\right) &= \frac{m_2}{m_1}, \\ \text{WSI}\,(\%) &= \frac{m_3}{m_1}, \end{aligned} \qquad (1)$$

where m_1 = the original weight of the sample, m_2 = the weight of the solid gel after removal of the supernatant, and m_3 = the weight of dry solids recovered from the supernatant.

2.5. Pasting Properties. The pasting properties of the native adlay and extrudates were measured using the method in [20], by a rapid viscosity analyzer (RVA 4500, Perten Instruments Inc., Sydney, Australia). Each sample (3.0 g, 14% wt/wt) was weighed in the canister, and distilled water was added so that a total weight of 28 g was attained. The slurry was maintained at 50°C for 1 min, heated to 95°C at a heating rate of 10.0°C/min, held at 95°C for 3 min, and then cooled to 50°C at a rate of 10.0°C/min. The pasting temperature and peak, trough, final, breakdown, and setback viscosity values were obtained. Three measurements were taken for each sample.

2.6. Thermal Properties. The thermal properties of the native adlay and extrudates were measured with a differential scanning calorimeter (DSC1, Mettler Toledo, Switzerland). The test was performed using the method described by Zhou et al. [21] with slight modifications. For analysis, 3.0 mg flour and 6.0 mL deionized water were placed in an aluminum box and balanced at 4°C for 12 h. Samples were measured from 30 to 100°C at a heating rate of 10°C/min. The thermal transitions for onset temperature (T_o), peak temperature (T_p), conclusion temperature (T_c), and gelatinization temperature range (ΔT) were calculated for the samples. All experiments were performed in triplicate.

2.7. Static Rheological Properties. The static rheological properties of the flour suspension were measured with a 60 mm parallel-plate rotational rheometer (Haake Mars 40, Thermo Fisher Scientific, USA). The rheological parameters were modified [22]. Each sample was mixed with deionized water to prepare a suspension having a mass fraction of 10%. Before the measurement, the gap of the parallel plates was sealed with silicone oil. The rheological properties were analyzed with a shear rate range from 1 s^{-1} to 1000 s^{-1} over 1 min, and the slit gap was 1 mm. The procedure was identically performed three times, and the average value was recorded as the measured result.

2.8. X-Ray Diffraction (XRD) Spectroscopy. XRD analysis of the native adlay and extrudates was carried out using an X-ray diffractometer (D8-focus, Bruker, Karlsruhe, Germany). Operation conditions included: a voltage of 30 kV, current of 10 mA, a 2θ range of 5–40°, a step size of 0.026°, and a rate of 0.33°/s. Crystallinity was calculated as the ratio of total peak area to total diffraction area [23] using the Jade version 6.0 software (Materials Data Inc., California, USA).

2.9. Microstructure. The microstructure of native adlay and extrudates was measured using a scanning electron microscope (Hitachi S-3400N, Japan). The samples were fixed on an aluminum stub with double-sided tape and coated with a gold film before observation. All samples were evaluated at an acceleration potential of 5 kV with 2000× magnification [24].

2.10. Data Analysis. In this study, extrusion was studied as a single-factor test, and the effects of the interaction of different conditions were not studied. All data are presented as the mean ± standard deviation. Mean values were compared using a one-way ANOVA test, using SPSS version 21 software (IBM Corp, New York, USA). The values were considered significant at $p < 0.05$.

3. Results and Discussion

3.1. Expansion Index. The expansion index (EI) of adlay flour through different extrusion conditions is presented in Figure 1. The main factors influencing the expansion index of extrudates include moisture, barrel temperature, shear force, and resident time. The EI rises by an increased barrel temperature, which reaches a maximum of 3.30 at 140°C. When the temperature rises, the degree of starch conversion of adlay flour increases. However, EI is slightly decreased from 3.30 to 2.27 when the temperature reached 160°C. The high temperature might cause water loss, and because water is a primary reactant in the gelatinization reaction, this led to the decrease of gelatinization degree and expansion index.

Water plays a key role in the EI of the extrudates. The presence of water is known to decrease viscosity, as it has a lower molecular weight when compared to starch. As EI decreases, water acted as a lubricant reducing the molecular weight and the melt viscosity of the extrudates. When the moisture level was increased between 19% and 27%, a resulting decrease in the EI from 4.31 to 2.65 was observed. Increased screw speed increases the shear force and reduces the residence time in the barrel. An increase in the screw speed from 170 to 330 rpm led to a nonsignificant decrease in the EI of the adlay flour. Similar results have been studied by Thymi et al. [25]. Therefore, the EIs of the extrudates were related to barrel temperature and moisture content.

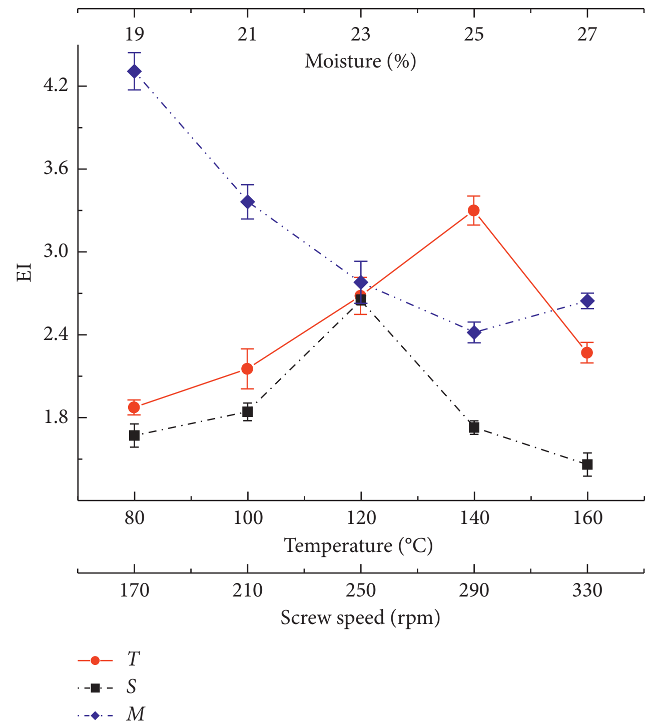


Figure 1: Expansion index of adlay flour treated by different extrusion conditions *T* (barrel temperature), *S* (screw speed), and *M* (moisture content).

3.2. WAI and WSI of Extrudates. There was a significant decrease in the WAI value when the temperature increased from 80 to 160°C ($p < 0.05$) (Figure 2(a)), while the WSI value was significantly increased with increasing temperature. Increasing the temperature from 100 to 160°C resulted in a 31.40% decrease (from 2.93 to 2.01) in the WAI value of the extrudate when moisture is at 27%, and the screw speed is 250 rpm. The WAI of the extrudate had a maximum peak at 100°C, after which it decreased. The WSI of the extrudate increased from 11.08 to 22.60 when the barrel temperature was increased from 80 to 160°C. Increasing the barrel temperature, which led to starch degradation, increased the WSI value.

Increasing the moisture content resulted in the extrudates with higher WAI and lower WSI values. The WAI of extrudates directly varied with the moisture content. Changing the moisture content from 19 to 27% resulted in 58.2% (from 1.84 to 2.91) in the WAI value of the extrudate at 120°C with 250 rpm (Figure 2(b)). It has been reported that an increase of moisture content causes a decrease of

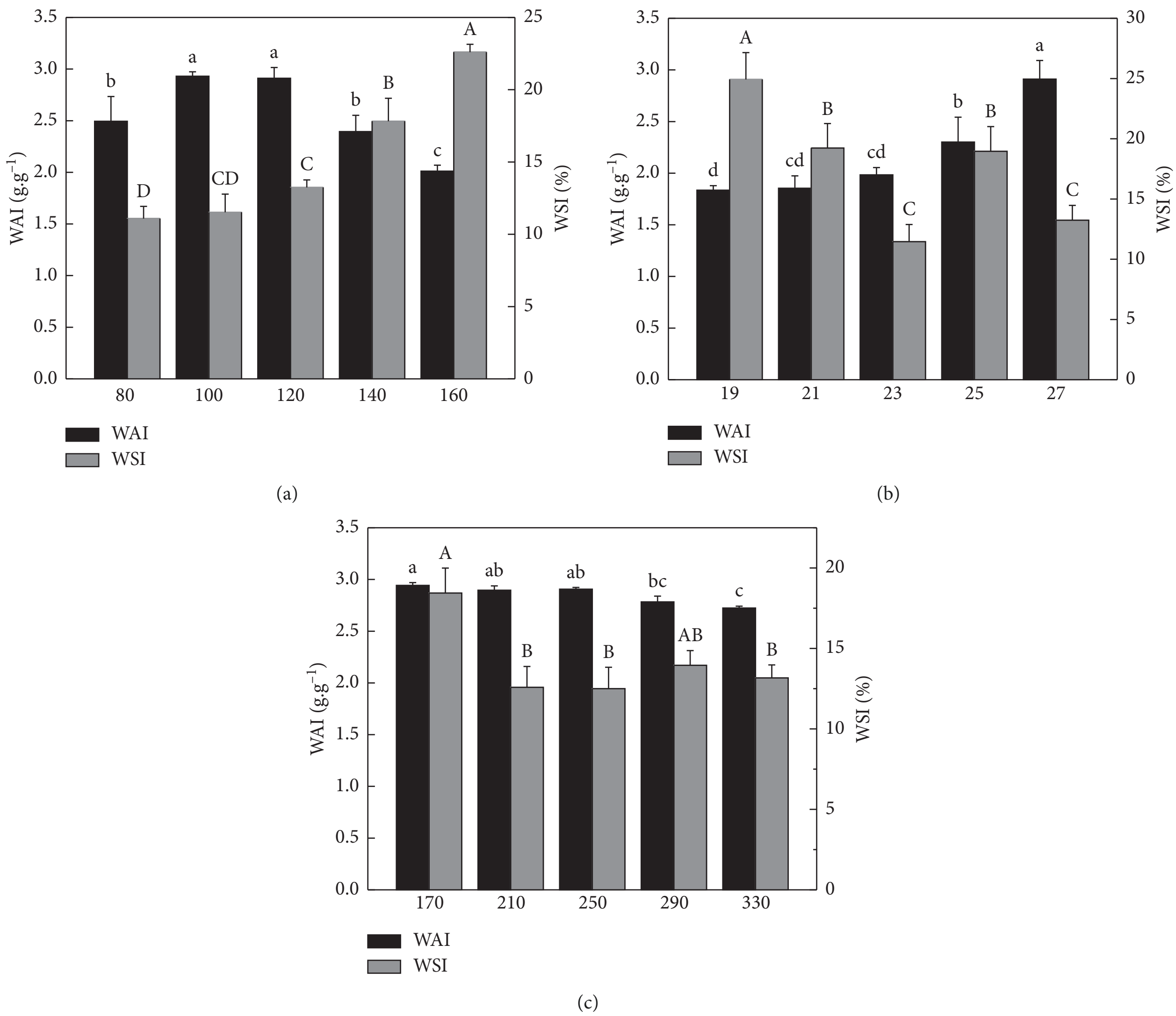


Figure 2: WAI and WSI of adlay flours treated by extrusion at different extrusion conditions. Uppercase and lowercase letters represent WSI and WAI, respectively. Different letters indicate significant differences ($p < 0.05$). (a) Temperature (°C). (b) Moisture (%). (c) Screw speed (rpm).

starch damage, and the damaged starch absorbs considerably low water than native starch [26]. Furthermore, the WSI of the extrudate was affected by moisture content. Increasing the moisture content from 19 to 27% led to a 46.9% decrease (from 24.92 to 13.24) in the WSI values of extrudates when the temperature and screw speed remained the same (Figure 2(b)). A higher moisture content led to a lower degradation rate and resulted in a lower WSI value for the flour.

Figure 2(c) shows the WAI and WSI values of adlay flours treated by extrusion at different screw speeds. It was observed that the screw speed increase did not result in a distinct influence on the WAI values. Screw speeds from 170 to 210 rpm significantly influenced the WSI values of the extrudate ($p < 0.05$). The WSI value is considered as an index of starch degradation and is used to evaluate the grade of the modified starch [27]. The increase in the screw speed reduces the residence time of the flour during extrusion, resulting in a decrease in the WSI values.

3.3. Pasting and Thermal Characteristics. The pasting curves of native adlay and extrudates are shown in Figure 3(a). The native adlay flour had a pasting temperature value of 73.50°C, ranging between T_o (69.57°C) and T_c (82.84°C) (Figure 3(b)). Also, the native adlay flour had a higher peak viscosity (1664 cP) and the final viscosity (2080 cP) and the lower setback (498 cP) and the breakdown viscosities (82 cP). These results illustrated that the native adlay flour is suitable for long-term storage due to the low retrogradation. When the adlay flour was heated in the RVA for examination, it was apparent that the extrusion process had a major influence. Compared with the native flour, the viscosity curve of extrudates exhibited a completely different trend. Extrusion usually causes extensive and nonreversible damage to the starch amylopectin crystal structure. Some researchers attribute this damage to a change in the order of the crystal alignment and the interaction of the amorphous region among starch chains [28]. Extrusion may also lead to

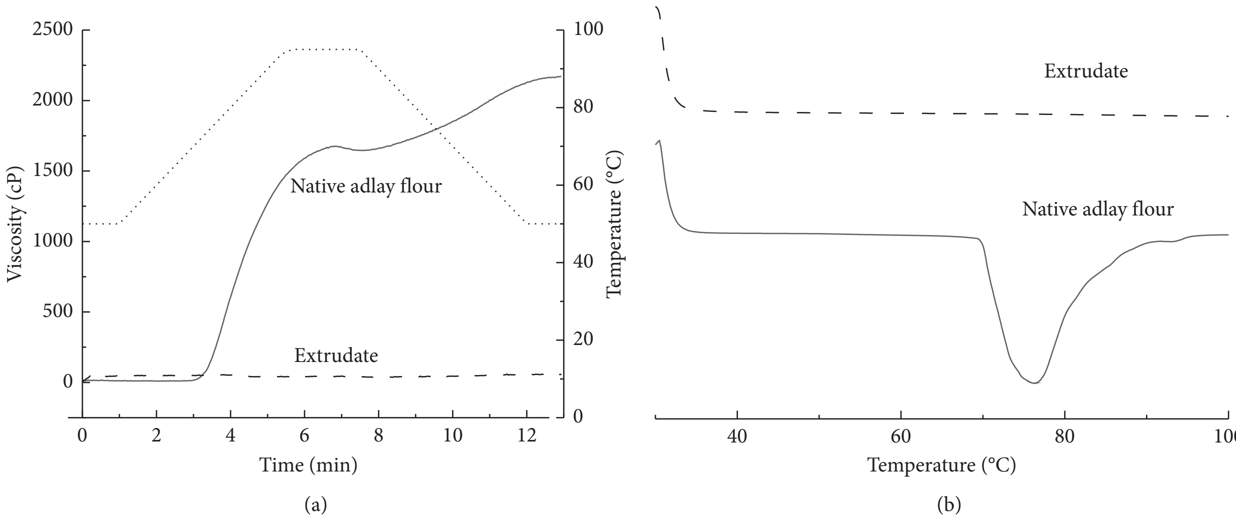


Figure 3: RVA curves (a) and thermal properties (b) of native adlay flour and adlay flour treated by extrusion.

the formation of amylose (amorphous region) and lipid complexes. The combination of heating and expansion of the amorphous starch renders the crystalline region unstable, so adjusting the severity of the extrusion conditions may be a feasible method for controlling the degree of adlay modification. The reduction in the final and setback viscosities of adlay indicates its effect on the amylose chains, which might lose the ability to retrograde.

The thermal properties of native adlay and extrudates are displayed in Figure 3(b). The gelatinization of the native adlay flour was more noticeable than that of the extrudates. The ΔH value of the native adlay flour was examined (8.34 J/g). Due to the gelatinization of amylopectin, the native adlay flour presented an endothermic peak. Thus, the application of extrusion conditions was enough to change the properties of adlay starch, including the transformation of starch gelatinization. Regarding the extrudates, there was no endothermic peak, which confirmed that most of the starch granules had gelatinized. This result is consistent with the previous research results. During extrusion, the force is propelled from the outer and amorphous portions of the starch granules to the center of the starch granules until no crystalline structure remains [29]. This result agrees with the viscosity curves obtained by RVA (Figure 3(a)), which are due to the integrity loss and disintegration of the starch granules. Compared with the native flour, the gelatinization enthalpy of the extrudate was prominently decreased by the extruding strength. Extrusion could cause starch gelatinization and increase the damaged starch, resulting in a subsequent decrease in the ability of the starch granules to gelatinize [30].

3.4. Static Rheological Properties. Figures 4(a)–4(c) show the viscosity profiles of adlay flours treated by extrusion at different screw speeds, temperatures, and moisture content. All the results illustrate that the extrudates were pseudoplastic fluids that exhibited thixotropism and shear thinning properties [31]. The fluids (the extrudates in water) followed the Herschel–Bulkley mathematical model, which described the type of behavior. The rheological parameters were obtained according to the Herschel–Bulkley model, where τ and γ are the shear stress and shear rate, respectively:

$$\text{Herschel} - \text{Bulkley model } \tau = \tau_o + K(\gamma)^n. \quad (2)$$

As shown in Figure 4(a), non-Newtonian behavior occurred when the increase in the shear rate caused a decrease in the viscosity of the extrudates. The moisture and screw speed also influenced the steady shear viscosity. At a low shear rate, the change of the shear viscosity was more significant due to the strong repulsion between the adlay starch chains. In contrast, the change in the shear viscosity was not significant at high shear rates because the adlay starch chains align themselves along the direction of the flow. As the temperature is increased, the shear viscosity of the extrudates decreased, and at higher temperatures, the activity of the starch molecules is increased [32]. In Figures 4(b)-4(c), the same trend of the shear viscosity of extrudates was observed when the flour was subject to different moisture contents and screw speeds. In summary, the lower viscosity was attributed to the stronger shear force.

3.5. XRD Properties. The crystalline structure and relative crystallinity can be analyzed by XRD [33]. The results for XRD patterns and the relative crystallinities of adlay flours treated by extrusion are given in Figure 5. The starches from different botanical origins may show varied crystalline types [34]. The diffractogram of native adlay flour displayed peaks at 15.1°, 17.2°, 18.0°, and 23.1° (Figure 5(a)). These peaks are typical for the A-type crystallinity pattern of starch [35], and the degree of relative crystallinity was 24.59%. Comparable results to our findings have been reported [36, 37], but the diffraction peak at 5.6° was not observed in this research. The possible reasons for this effect are the determination of adlay flour instead of adlay starch and the difference of adlay varieties.

The amylopectin crystal structure can be partially or totally disrupted. It is shown that all the extrudates underwent total disruption of the original amylopectin structure. The

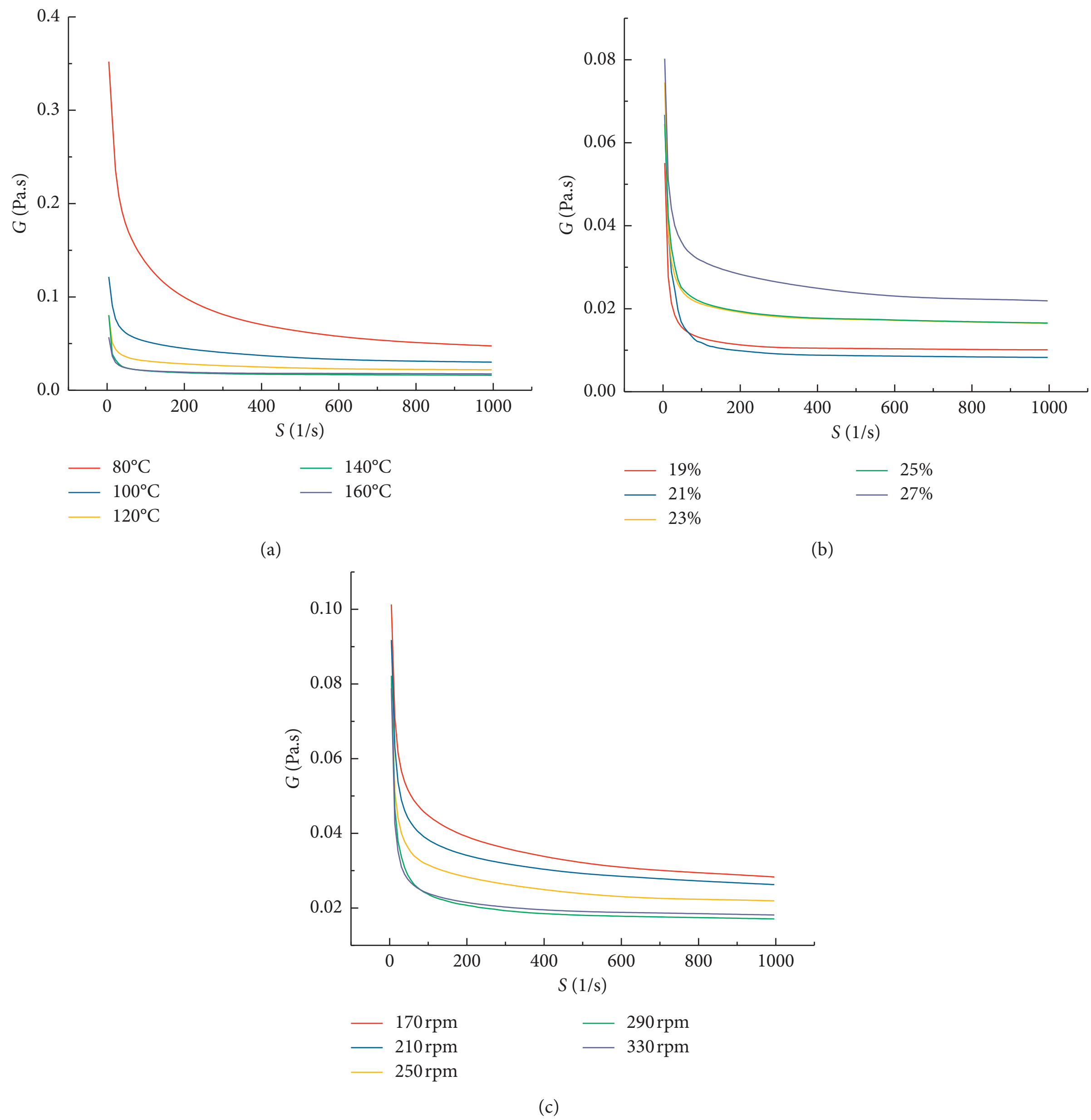


FIGURE 4: Steady shear viscosity of adlay flours treated by extrusion at different extrusion conditions. (a) Barrel temperature, (b) moisture content, and (c) screw speed.

degree of relative crystallinity decreased from 24.59% of the native adlay to less than 4.32% of the extrudates (Figure 5). For the extrudates, these peaks gradually disappear, which suggested that the crystalline area of starch was destroyed. This result agrees with the values of RVA and DSC. The extrudates showed different diffraction patterns compared to the native adlay flour. From Figure 5(a), it is possible to see that it is almost similar to the native starch granule diffraction profile, allowing saying that mild modification was seen in the original amylopectin structure at 80°C. The relative crystallinity increased with increasing barrel temperature (Figure 5(a)). High temperature accelerated the gelatinization of adlay flour, resulting in the destruction of crystal structure in starch. However, the relative crystallinity decreased with increasing moisture content and screw speed in Figures 5(b)-5(c). Water can protect the starch granules from destroying, and higher screw speed reduced the heating time of the adlay flour. The decrease in relative crystallinity was owing to damage to the ordered structure of crystalline and amorphous areas in adlay starch granules.

3.6. Microscopic Structures. The microscopic structures of the extrudates at 2000× magnification were obtained at different barrel temperatures (Figure 6(a)), moisture content (Figure 6(b)), and screw speeds (Figure 6(c)). It was observed that most of the native starch granules were spherical, elliptical, and polygonal, with a particle distribution size of 5–16 μm, similar to that of most other grains. Scanning

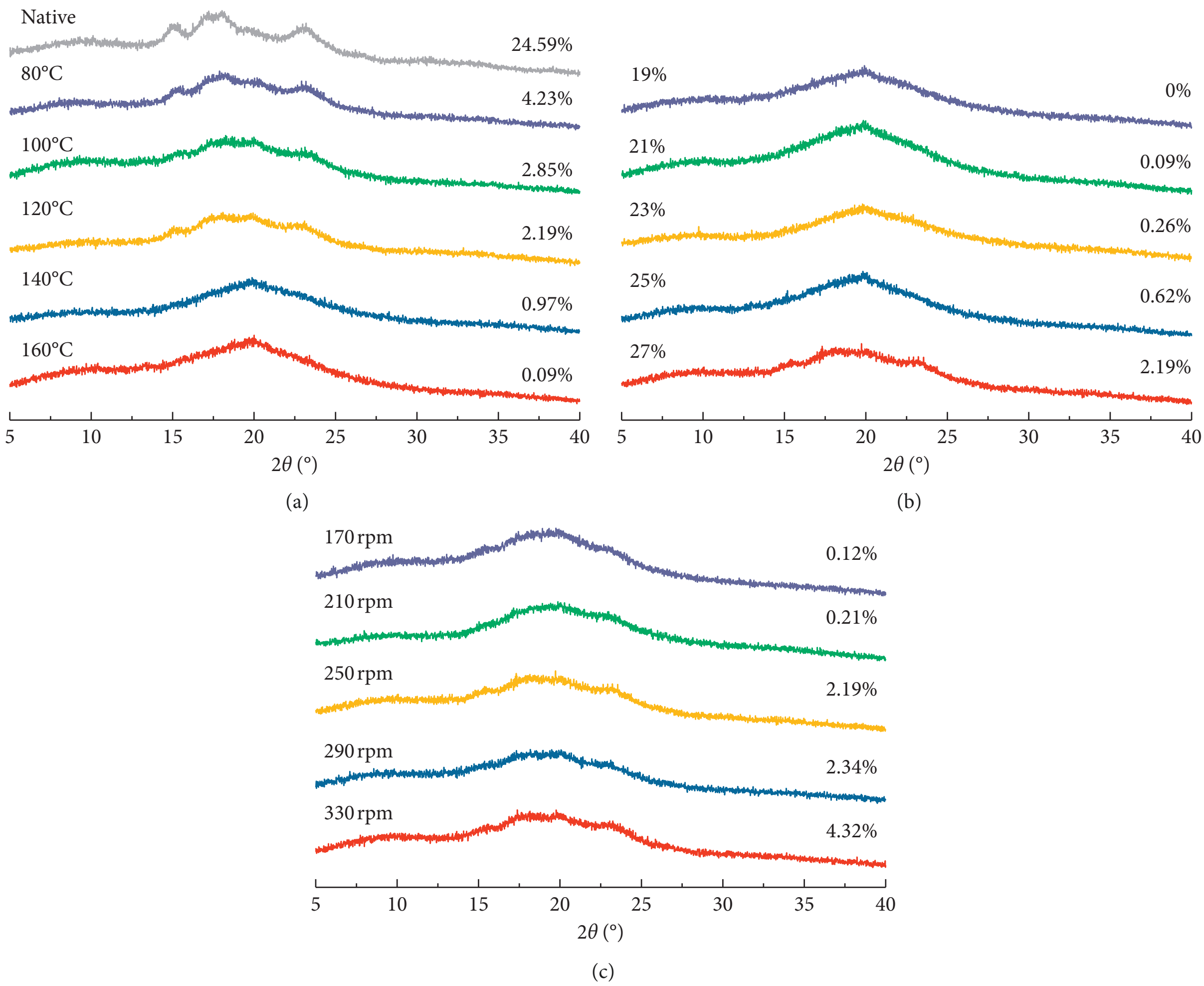
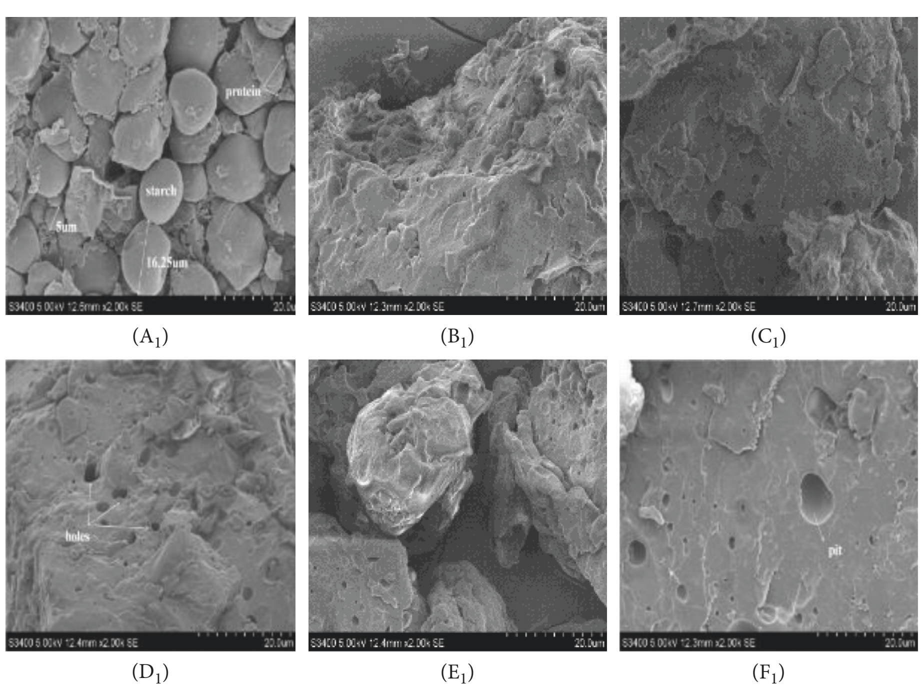


Figure 5: XRD spectra of adlay flours treated by extrusion at different extrusion conditions. (a) Barrel temperature, (b) moisture content, and (c) screw speed.



(a)

Figure 6: Continued.

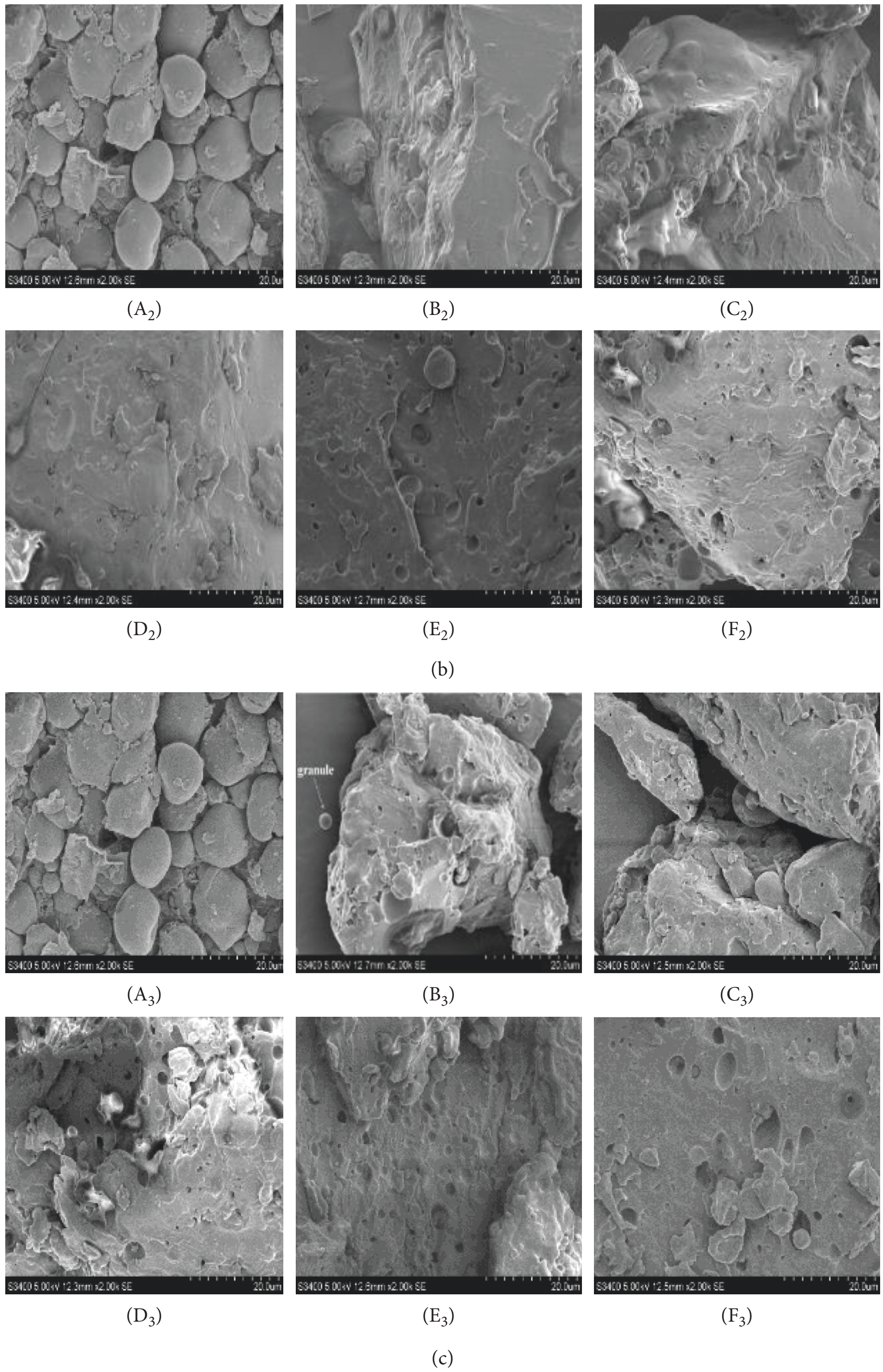


Figure 6: Scanning electron micrographs of adlay flour treated by extrusion at different extrusion conditions (2000×). (a) Barrel temperature (A1-F1): native, 80, 100, 120, 140, and 160°C; (b) moisture content (A2-F2): native, 19, 21, 23, 25, and 27 g $(100\,g)^{-1}$wb; (c) screw speed (A3-F3): native, 170, 210, 250, 290, and 330 rpm.

electron microscopy displayed cracking of starch granules with extrusion [38]. The microstructure and particle morphology of the powder may be related to changes in extrusion parameters. As the temperature increases, the structure becomes more colloidal, which is caused by the higher degrees of starch gelatinization at higher temperatures. Increasing the moisture results in more pores on the surface of the particles, particularly at 25% and 27%, as shown in Figure 6(b)-E_2 and 6(b)-F_2. At low screw speed, some intact starch granules still exist, as seen in Figure 6(c)-B_2 because screw speed provides the shearing force during extrusion. The higher the screw speed, the greater the shearing force.

SEM images show that extrusion had a significant effect on the microstructure of the adlay flour. The continuous matrix structure was destroyed by extrusion, and the extent of the destruction increased as the extrusion strength increased. After extrusion, the starch and protein particles were severely damaged, and almost no intact particles were visible. There were many uneven and irregular fragments, and some pits and holes appeared on the surface of the particles.

4. Conclusions

Twin-screw extrusion provides an alternative method for producing adlay flours with different technological properties. Extrusion resulted in significant effects on the physicochemical and morphological characteristics of adlay flour, including an increase in EI, WAI, and WSI, a decrease in paste viscosity, thermal property, and relative crystallinity, and the cracking of starch granules. Extrusion at high temperature and high shear influenced the pasting properties of the adlay flours. The change in the rheology property of the flour broadened its potential applicability to canned and frozen foods. These results may contribute to the optimization of a starch-based extruded food process to achieve better quality characteristics.

Data Availability

The data used to support the findings of this study are available from the corresponding author and zhanggaopeng@neau.edu.cn upon request.

Consent

Informed consent was obtained from all individual participants included in the study.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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Supplementary Materials

Supplementary Figure S1: a graphical abstract that visually represents the primary findings of this article, allowing readers to easily identify the article's main message. (*Supplementary Materials*)

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