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Impact of Heating Temperature on the Crystallization, Structural, Pasting, and Hydration Properties of Pre-Gelatinized Adlay Flour and Its Implementation in Instant Porridge Product

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Abstract: Pre-gelatinization by using an autoclave is the simplest lab-scale method for preparing instant flour from adlay. The effect of heating temperatures (60 °C, 80 °C, and 100 °C) on the structural and pasting properties of pre-gelatinized adlay flour was studied. Moreover, the sensory acceptability of instant porridge prepared from this flour was investigated. Results showed that the shapes of starch granules of pre-gelatinized adlay flour started to disappear at a temperature of 80 °C. However, the crystallinity of the flour gelatinized at 60 °C was higher than that of flours gelatinized at other temperatures. The treatment increased water absorption, water solubility, and swelling power of pre-gelatinized adlay flour. It changed the pasting properties of pre-gelatinized adlay flour and decreased the lightness of pre-gelatinized adlay flour. Overall, the panelists preferred the instant porridge made from pre-gelatinized adlay flour prepared from 100 °C.

Keywords: pre-gelatinized flour; instant porridge; adlay; autoclave method; heating

1. Introduction

The Fourth Industrial Revolution has forced us to change our habits and follow an instant lifestyle, including the trend of consuming instant food [1]. Various instant and ready-to-eat food products are widely available in the market for example instant porridge. It is reported that the global market for instant food was valued at USD 44.11 billion in 2019, and it is projected to reach USD 72.69 billion by 2027 [2]. Therefore, research to develop new pre-gelatinized food products is needed to fulfill the global demand of the pre-gelatinized food market. Instant porridge can be prepared from pre-gelatinized flours of cereals such as adlay. Adlay (*Coix lachrymal-jobi L.*) has been widely known as an ingredient of food and traditional medicines in Asian countries [3]. This grain contains an active compound, namely coixenolide, which has been indicated by tests to have the ability to act as an antioxidant agent to prevent cancer [4–6]. Therefore, adlay can be used as an ingredient of functional food products. Based on pasting characteristics, starch varieties that are suitable to be ingredients of instant porridges are clustered among C-type starches. These starches exhibit limited granule swelling, no peak viscosity, short gelatinization times, and high water absorption indexes [7,8]. Mulyono et al. [9] reported that adlay flour has a relatively high peak viscosity (2767–3958 cP), a long gelatinization time (356–438 s), and high breakdown viscosity values (1442–1585 cP). Moreover, adlay flour shows a low water-holding capacity [10]. Based on these characteristics, native adlay flour is not suitable to be an ingredient of instant porridge. Therefore, native adlay flour should be modified prior to being implemented as an ingredient of instant products.

Instant flour can be prepared by using the pre-gelatinized starch method. This method is considered to be the simplest method to prepare instant flour [11] and is also environmentally friendly since it does not use any chemicals [12]. Pre-gelatinized starch shows a high ability to absorb water, yielding high viscosity at ambient temperature. Therefore, it has potential to be applied as an ingredient of ready-to-eat foods [11,13]. Loypimai and Moongngarm [14] explored pre-gelatinized banana flour as an ingredient of instant porridge. This product is sensorially acceptable and similar to other commercial instant porridge products. Heating by autoclave has been used to prepare pre-gelatinized flour from some starch sources. The properties of the flour are affected by the processing parameters during autoclaving. Generally, the gelatinization process converts the diffraction pattern of the native starch from A- or B-type into weak B- or V-type [15]. Moreover, higher autoclaving temperatures tend to decrease the crystallinity of the modified starch [16]. However, the degree of gelatinization depends on the extent of the heating parameters, i.e., time, temperature, and the sample's moisture content. Therefore, the properties of pre-gelatinized flours prepared by autoclaving are affected by the starch's origin and the autoclaving parameters.

In this study, the effects of different autoclaving temperatures on the physicochemical properties of pre-gelatinized adlay flour, including color, hydration, morphological, pasting, and structural properties, were investigated. Moreover, the sensory acceptability of instant porridge prepared from the pre-gelatinized adlay flours was examined.

2. Materials and Methods

2.1. Materials

Adlay seeds were purchased from a local supplier (UMKM Hanjeli, Sumedang, West Java, Indonesia). Coconut milk powder, creamer powder, and sugar were obtained from a local supermarket (Tokma Supermarket, Subang, West Java, Indonesia). All chemicals used in the analysis were of analytical-grade.

2.2. Preparation of Pre-Gelatinized Adlay Flour

Pre-gelatinized adlay flour was prepared according to the method of Niba and Hoffman [17] with slight modifications. Polished adlay seeds and distilled water with a ratio of 1:3 were weighed and placed in a Schott bottle. Then, the sample was heated in an autoclave (Hirayama HVE-50, Saitama, Japan) for 60 min at different temperature levels of 60 °C (H-60), 80 °C (H-80), and 100 °C (H-100) at observed pressures of 0.2, 0.5, and 1.0 bar, respectively. An untreated sample (H-0) was used as the reference sample. The samples were then dried in an oven (UM500; Memmert, Schwabach, Germany) at 50 °C for 12 h. Finally, the samples were ground by using a powder grinder (HR2115, Philips, Jakarta, Indonesia) and sieved by using a sieve (D-42757; RetschGmbh, Düsseldorf, Germany) with apertures of 40 mesh. The samples were stored at room temperature prior to analysis.

2.3. Preparation of Instant Porridge from Pre-Gelatinized Adlay Flour

Instant porridge was prepared by the dry mixing method of Khan et al. [18] with modifications. The pre-gelatinized adlay flour was thoroughly mixed with other ingredients, including coconut milk powder (10%), creamer powder (5%), and sugar (12%).

2.4. Color Analysis

The color analysis of the samples was carried out by using a color meter (Shenzhen ThreeNH Technology Co., Ltd., Shenzhen, China). Prior to the analysis, the instrument was calibrated with a white tile standard provided by the manufacturer. Each sample was placed in transparent glass, and its surface was measured with a lamp. Results were expressed as L* (lightness), a* (red–green), and b* (blue–yellow) [19].

2.5. Morphological Properties

The morphological properties of the samples were observed by using a scanning electron microscope (JEOL JSM IT300, Tokyo, Japan). Prior to observation, the samples were dried by using a vacuum oven at 70 °C for 12 h. The dried samples were sprinkled onto double-sided tape, and the remaining samples were removed by spraying them with N_2. The samples were coated with gold. The morphological properties were observed with a power of 2 kV at different magnification levels, including 100×, 500×, 1000×, and 5000× [20].

2.6. Crystalline Structure

The crystalline structure was assayed by using X-ray diffraction (XRD D8 Advance, Bruker, Berlin, Germany) according to the method of Nakorn et al. [21] with some modifications. The XRD data were collected over an angular range from 3° to 27° at 2θ. The crystallinities of the samples were calculated by the following formula.

$$\text{Relative crystallinity } (\%) = \frac{\text{area of crystalline peaks}}{\text{total area of diffractogram}} \times 100\% \quad (1)$$

2.7. Hydration Properties

The hydration properties of the samples were determined according to the method of Lai [20]. The sample was weighed (2.5 g) (*Wi*) in a centrifuge tube. Then, 30 mL of distilled water was added. The sample was mixed by using a vortex mixer (Model VM-300, Gemmy Industrial Corp., Taipei, Taiwan) for 1 min. Then, the sample was centrifuged at a speed of 5000 rpm and temperature of 20 °C for 15 min. The supernatant was dried at 105 °C for 12 h, and the dried supernatant was weighed (W_s). The sediment was weighed (W_r). The hydration parameters of water absorption index (*WAI*), water solubility index (*WSI*), and swelling power (*SP*) were calculated by using Equations (2)–(4), respectively.

$$WAI\left(\frac{\text{g}}{\text{g}}\right) = \frac{W_r}{W_i} \quad (2)$$

$$WSI\ (\%) = \frac{W_s}{W_i} \times 100\% \quad (3)$$

$$SP\left(\frac{\text{g}}{\text{g}}\right) = \frac{WAI}{1 - \frac{WSI}{100}} \quad (4)$$

2.8. Pasting Properties

The pasting characteristics of the samples were determined according to the method of Lai [20]. Sample suspensions (12% *w*/*v*) were prepared by mixing 3 g of sample with 25 mL of distilled water in an aluminum canister. The pasting properties of the samples were assessed by a Rapid Visco Analyzer (RVA-TecMaster, Macquarie Park, Sydney, Australia) following this sequence: The sample was equilibrated at 35 °C for 2 min, heated up to 95 °C at a rate of 11.8 °C/min, and then held at this temperature for 2.5 min. Finally, it was cooled down to 35 °C at a rate of 11.8 °C/min. The pasting curve of the sample was reported.

2.9. Sensory Acceptance of Instant Porridge

Sensory acceptance evaluation was carried out by using a hedonic scoring test following the method of Netshiheni et al. [22] with slight modifications. Hot water at a temperature of 70 °C and pre-gelatinized porridges were homogenously mixed at a water-to-sample ratio of 10:4. The sensory acceptability parameters of samples, including color, flavor, taste, grain texture, viscosity, and overall acceptance, were evaluated by 35 untrained panelists with 1–7 hedonic scales, in which 1 represents maximum dislike, and 7 represents maximum like.

2.10. Statistical Design and Data Analysis

Data were processed by using a one-way analysis of variance test (ANOVA) at the 5% significance level [23]. Post-hoc testing was carried out using Duncan's multiple range test.

3. Results and Discussion

3.1. Color

The color parameters, including lightness (L*), redness (a*), and yellowness (b*) values, of native and pre-gelatinized adlay flours prepared at different temperatures are shown in Table 1. The lightness of pre-gelatinized adlay flour prepared at a temperature of 60 °C was higher than that of native adlay flour. This might be due to the pigments of adlay flour leaching out into the medium during heating [23,24].

Table 1. Color properties (L*, a*, and b*) of native adlay flour (H-0) and pre-gelatinized adlay flours prepared at different temperatures (60 °C (H-60), 80 °C (H-80), and 100 °C (H-100)).

Sample	L*	a*	b*
H-0	75.38 ± 0.00 * c **	1.26 ± 0.02 d	3.89 ± 0.01 d
H-60	79.39 ± 0.05 d	0.58 ± 0.02 c	3.18 ± 0.04 c
H-80	73.14 ± 0.01 b	0.45 ± 0.01 b	2.92 ± 0.01 b
H-100	72.48 ± 0.00 a	0.38 ± 0.01 a	2.74 ± 0.02 a

* The mean value of three repetitions ± standard deviations. ** The letter difference in the same column expresses significantly differently at $p \leq 0.05$.

Heating adlay flour at higher temperatures (80 and 100 °C) tended to intensify the color of pre-gelatinized adlay flour, as indicated by the decrease in L*, a*, and b* values. The color of pre-gelatinized adlay flour is browner than that of the native adlay flour. The brown color of pre-gelatinized adlay flour might be caused by the Maillard reaction occurring during heating at high temperatures [25]. The Maillard reaction is initiated by a condensation of amino groups of proteins, peptides, and amino acids with carbonyl groups of reducing sugars. Eventually, large polymeric compounds called melanoidins, which are brown in color, are formed [26].

3.2. Morphological Properties

The scanning electron micrographs (SEM) of native and pre-gelatinized adlay flours prepared at different temperatures are presented in Figure 1. The native adlay flour (H-0) exhibited granules with relatively smooth surfaces, as well as intact and round or polygonal shapes, as previously reported [27,28].

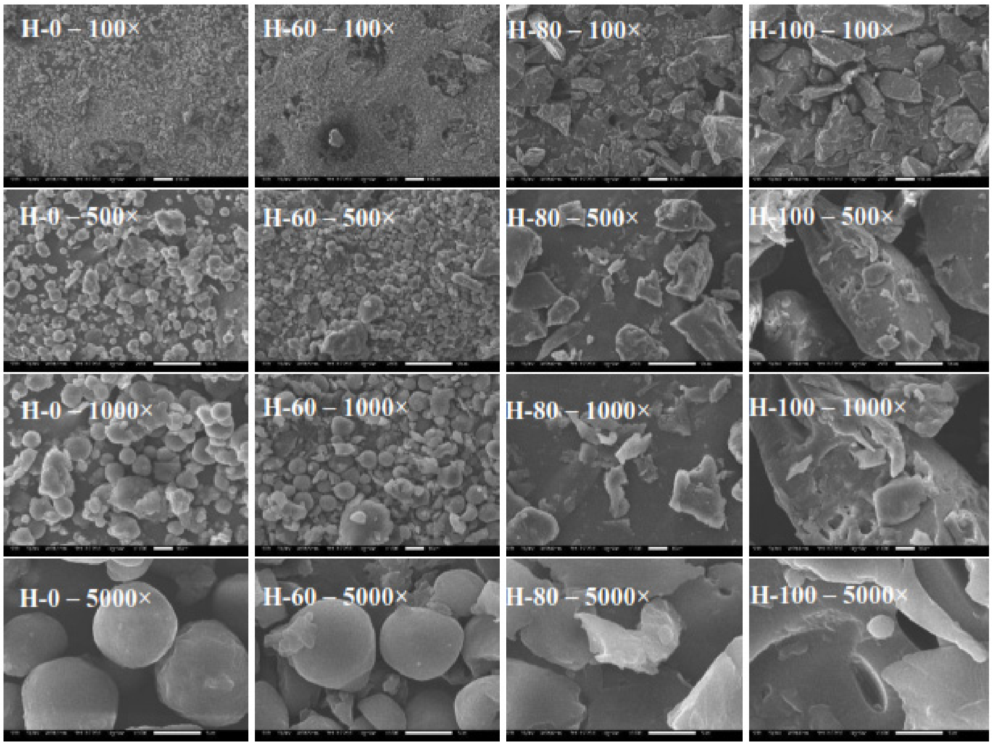

Figure 1. Micrograph of native adlay flour (H-0) and pre-gelatinized adlay flours prepared at different temperatures (60 °C (H-60), 80 °C (H-80), and 100 °C (H-100)) observed at magnifications of 100×, 500×, 1000×, and 5000×.

Heating at a temperature of 60 °C did not alter the granule morphology of adlay flour. Furthermore, heating at temperatures at above 80 °C destroyed the granule's shapes, yielding particles with irregular shapes. These alterations were indicative of the complete gelatinization of starch as result of heating adlay flour in an excess of water at high temperatures [29]. During gelatinization, high temperature breaks the hydrogen bonds within starch molecules and destroys the double helix structure of the starch granules [30], which results in breaking starch granules into irregular shapes. Xu et al. [31] and Sun et al. [32] also reported that the pre-gelatinized starch exhibited no intact starch granules. The morphological changes in pre-gelatinized adlay flour may cause alterations in the physical properties of the flour.

3.3. Crystalline Structure

The X-ray diffraction patterns of native and pre-gelatinized adlay flours prepared at different temperatures are shown in Figure 2. The native adlay flour (H-0) and pre-gelatinized adlay flour at 60 °C (H-60) showed the pattern of an A-type starch, as indicated by the characteristic peaks of A-type crystal at 2θ of 15°, 17°, 18°, 20°, and 23° [33].

Heating at temperatures above 80 °C destroyed the crystal structure of adlay flour, as indicated by the disappearance of identifiable peaks of A-type crystal. Instead, the broad Gaussian peak of an amorphous structure [34] was observed. The destruction of the native structure of adlay flour was also observed in the morphology of broken granules of the starches of pre-gelatinized adlay flours produced by heating at temperatures of 80 °C (H-80) and 100 °C (H-100) (Figure 1).

The relative crystallinities of native and pre-gelatinized adlay flours are presented in Table 2. Surprisingly, the relative crystallinity of pre-gelatinized adlay flour heated at 60 °C (H-60) was higher (30.14%) than that of the native sample (H-0) (26.87). This might be due to the crystalline lamellae moving toward perfect configuration, which is caused by hydrothermal pressure that occurs during autoclaving [35]. However, further investigation is required to confirm the phenomenon by using thermal (DSC) analysis. Moreover, the relative crystallinities of pre-gelatinized adlay flours heated at 80 °C and 100 °C were 0, indicating fully gelatinized starches [29].

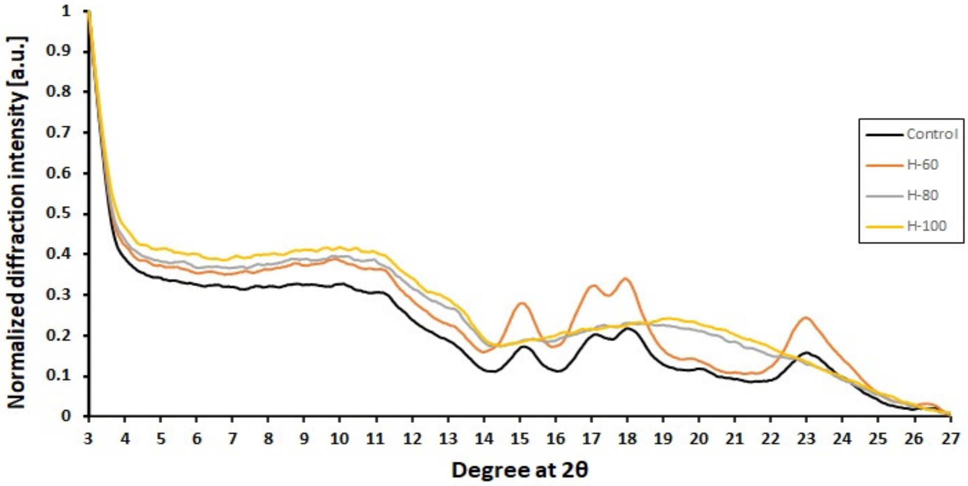

Figure 2. X-ray diffraction pattern of native adlay flour (H-0) and pre-gelatinized adlay flours prepared at different temperatures (60 °C (H-60), 80 °C (H-80), and 100 °C (H-100)).

Table 2. Relative crystallinities of native adlay flour (H-0) and pre-gelatinized adlay flours prepared at different temperatures (60 °C (H-60), 80 °C (H-80), and 100 °C (H-100)).

Sample	Relative Crystallinity (%)
H-0	26.87
H-60	30.14
H-80	0.00
H-100	0.00

3.4. Hydration Properties

The water absorption index (*WAI*), water solubility index (*WSI*), and swelling power (*SP*) of native and pre-gelatinized adlay flours prepared at different temperatures are summarized in Table 3. The treated samples exhibited higher *WAI* values than the native adlay flour. Moreover, pre-gelatinized adlay flour produced at higher temperatures showed higher *WAI* values than that produced at lower temperatures. Heating adlay flour in an excess of water and at higher temperatures destroys the hydrogen bonds within the starch structures at both levels of crystalline and amorphous structures [36]. Consequently, water more easily diffuses into broken starch structures [37,38], which results in the high *WAI* values of pre-gelatinized adlay flours.

Table 3. Hydration properties (water absorption index (*WAI*), water solubility index (*WSI*), and swelling properties (*SP*)) of native adlay flour (H-0) and pre-gelatinized adlay flours prepared at different temperatures (60 °C (H-60), 80 °C (H-80), and 100 °C (H-100)).

Sample	*WAI* (g/g)	*WSI* (%)	*SP* (g/g)
H-0	2.15 ± 0.02 * a **	8.86 ± 0.87 bc	2.36 ± 0.03 a
H-60	2.37 ± 0.01 a	2.49 ± 0.83 a	2.43 ± 0.01 a
H-80	3.62 ± 0.18 b	9.51 ± 1.59 c	4.00 ± 0.26 b
H-100	5.61 ± 0.69 c	6.94 ± 1.08 b	6.02 ± 0.74 c

* The mean value of three repetitions ± standard deviations. ** The letter difference in the same column expresses significantly differently at $p \leq 0.05$.

The *WSI* value of the untreated sample (H-0) was higher than the values of the treated samples (H-60, H-80, and H-100) (Table 3). This result indicated that native adlay flour contains many soluble materials such as soluble proteins [37,39], which dissolve into water when adlay flour is soaked with water. Increasing heating temperatures tended to increase the *WSI* values of pre-gelatinized adlay flours. Higher heating temperatures impact the greater molecular destruction of large molecules of adlay flour components, i.e., starch and proteins become smaller soluble fragments of the components, i.e., oligosaccharides and peptides [21,30]. As a result, pre-gelatinized adlay flour produced at higher heating temperatures tended to have higher *WSI* values.

The results showed that the pre-gelatinized adlay flour produced at the highest heating temperature (H-100) exhibited the highest swelling power values. This result indicates that the molecular destruction of sample H-100 due to the gelatinization process was more severe than in the other pre-gelatinized adlay flour produced at the lower temperatures (H-60 and H-80). Pre-gelatinized starch with a higher gelatinization degree exhibits higher amounts of free hydroxyl groups on the starch's structural backbone [30,40]. When pre-gelatinized starch is dissolved in water, the water more easily penetrates into this opened structure of starch; consequently, the swelling power value of pre-gelatinized starch is high [41].

3.5. Pasting Properties

The pasting properties of native and pre-gelatinized flours prepared at different temperatures are shown in Figure 3. The results showed that the pre-gelatinized adlay flour that had been heated at a temperature of 60 °C (H-60) exhibited the highest peak viscosity. This could be due to the structural arrangement of the starch in H-60 tending to have a more perfect alignment than that in H-0. This was evidenced by the higher relative crystallinity value of H-60 compared to that of H-0 (Table 2). This phenomenon was also supported by the results of the SEM analysis in which the pre-gelatinized adlay flour heated at 60 °C (H-60) still showed intact starch granule structures (Figure 1: panel H-60). Due to hydrothermal pressure occurring during the heating of starch in the presence of a solvent, the crystalline lamellae of the starch moves toward more perfect alignment until it is destroyed when heating proceeds further [35]. Therefore, H-60 showed better thermal stability than H-0, as indicated by the peak viscosity of H-60 being higher than that of H-0.

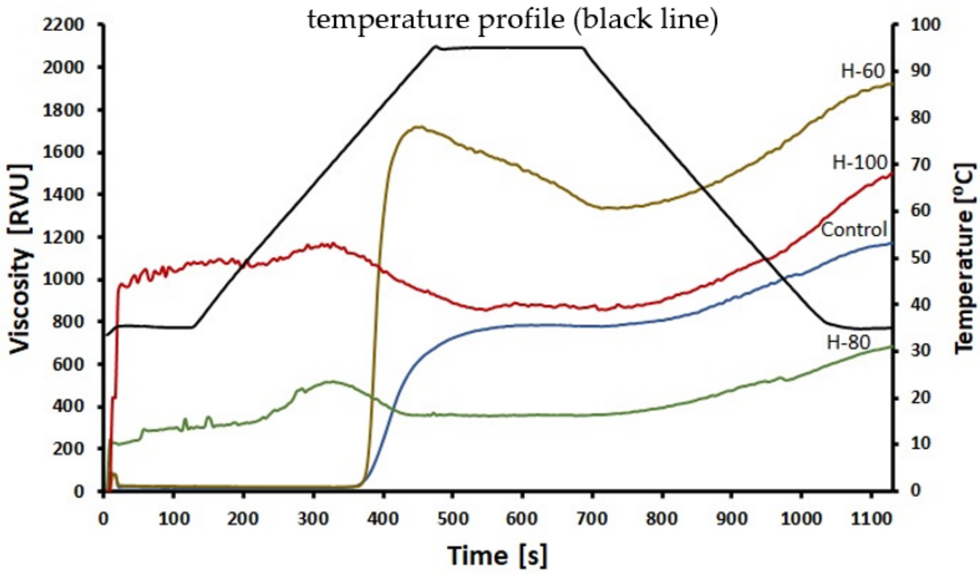

Figure 3. Pasting curve of native adlay flour (H-0) and pre-gelatinized adlay flours prepared at different temperatures (60 °C (H-60), 80 °C (H-80), and 100 °C (H-100)).

Figure 3 also showed that the pre-gelatinized adlay flours resulting from heating at temperatures of 80 °C (H-80) and 100 °C (H-100) exhibited high viscosity at the beginning of RVA analysis. This result implied that those samples had undergone gelatinization prior to RVA analysis [42]. This result is in accordance with the results of the morphological analysis (Figure 1: panel H-80 and H-100), which showed that no intact starch granules were found. Moreover, the results of the X-ray diffraction pattern confirmed the amorphous structures of H-80 and H-100 (Figure 2). The initial viscosity of H-100 is higher than that of H-80 (Figure 3). This indicated that H-100 underwent a higher degree of gelatinization than H-80.

3.6. Sensory Acceptance of Instant Porridge

The sensory evaluations of instant porridge made from native and pre-gelatinized adlay flours prepared at different temperatures are illustrated in Table 4. The results showed that panelists tended to like the color of instant porridge made fromH-60 compared to the color of instant porridge from the other samples.H-60 exhibited a higher lightness value than the other samples (Table 1). Instant porridge prepared from pre-gelatinized adlay flours heated at high temperatures (H-80 and H-100) tended to have a darker brown color, which might be due to the browning effect that occurred during heating [25,43]. Moreover, panelists tended to dislike instant porridge prepared from pre-gelatinized adlay flours that had been heated at high temperatures (H-180 and H-100) (Table 4). This result implies that instant porridge with a brighter color is more acceptable [23,43].

Table 4. Sensory acceptance of instant porridge prepared from native adlay flour (H-0) and pre-gelatinized adlay flours prepared at different temperatures (60 °C (H-60), 80 °C (H-80), and 100 °C (H-100)).

Sample	Color	Aroma	Taste	Sandy Texture	Viscous Feel	Overall Acceptance
H-0	4.63 ± 1.11 * ab **	4.40 ± 1.19 ab	3.09 ± 1.27 a	2.60 ± 1.24 a	2.51 ± 1.17 a	2.86 ± 0.81 a
H-60	4.97 ± 0.92 b	4.34 ± 1.16 ab	3.57 ± 1.40 ab	3.06 ± 1.39 a	3.29 ± 1.36 b	3.46 ± 1.15 b
H-80	4.46 ± 0.89 a	3.97 ± 1.32 a	4.00 ± 1.19 bc	4.14 ± 1.29 b	4.40 ± 1.24 c	4.26 ± 1.07 c
H-100	4.51 ± 1.04 ab	4.63 ± 0.91 b	4.46 ± 1.17 c	4.80 ± 1.02 c	4.09 ± 1.29 c	4.63 ± 1.11 c

* The mean value of three repetitions ± standard deviations. ** The letter difference in the same column expresses significantly differently at $p \leq 0.05$.

Regarding the sandy texture of instant porridge, panelists tended to like smooth texture (Table 4). Instant porridge prepared from pre-gelatinized adlay flours produced at high temperatures (H-80 and H-100) tended to have a smoother texture than that from flour produced at low temperature (H-60) or native adlay flour (H-0). H-80 and H-100 are more completely gelatinized than H-60 (Figure 1: panel H-60, H-80, and H-100, and Figure 2). When H-80 and H-100 are mixed with water, the water is more easily absorbed into the destroyed structure of the fully gelatinized starch, creating a smooth viscous liquid [25]. Meanwhile, when H-0 and H-60 came into contact with water, most of the water is only absorbed on the surfaces of the starch granules, creating a coarse or sandy texture in the instant porridge prepared from these samples. Overall, instant porridge made from H-100 was the most accepted by the panelists.

4. Conclusions

Heating temperature (60, 80, or 100 °C) affected the structural and pasting properties of pre-gelatinized adlay flours, as well as the sensory acceptability of instant porridge made from the modified flours. As heating temperature increased, the lightness value of pre-gelatinized adlay flour decreased. However, its water absorption index, water solubility index, and swelling power showed a tendency to increase. The SEM results showed severe damage to the starch granule structures of adlay flours heated at high temperatures. The pasting properties of the pre-gelatinized adlay flours were different from those of the native adlay flour. The X-ray diffraction patterns of H-80 and H-100 changed from an A-type to an amorphous structure. Overall, instant porridge made from pre-gelatinized adlay flour treated at 100 °C was preferred by the panelists.

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