Physicochemical and gelatinization properties of ethanol-treated sorghum flour

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Abstract. Granular cold-water swelling starch can be produced by ethanol-treated method. This work aims to determine the chemical, physical and gelatinization properties of ethanol-treated sorghum flour. White sorghum flour was heated in ethanol solution (45, 50 and 55 %v/v) with ratio of 1:9 at three temperature levels (60, 80 and 100 °C). The modified flour properties including moisture content, ash content, whiteness degree, and gelatinization properties were determined. The results showed that ethanol concentration had significant effect on the whiteness degree and breakdown properties. Heating temperature significantly affected the moisture content, whiteness degree, peak viscosity, final viscosity, breakdown and set-back properties of sorghum flour. Ethanol-treated sorghum flour produced from ethanol concentration of 55 % and heating temperature of 60 °C showed superior characteristics than the other treatments in which it’s properties including moisture content, ash content, whiteness degree, peak viscosity, final viscosity, breakdown and set-back properties were 10.24 %, 0.62 %, 84.85 %, 633.50 cP, 4713.50 cP, 571.00 cP, 4551.00 cP, respectively.

1. Introduction
Sorghum is known as an alternative carbohydrate source for staple food. Sorghum contains carbohydrate, protein, fat and fiber of 70.7 %, 10.4 %, 3.1 %, 2%, respectively [1]. Due to these high nutritional properties, therefore, sorghum is classified as an important ingredient for human food also for industrial applications [2]. Since sorghum is easy to grow in different soil and environments, therefore, it is easy to find this commodity in many countries [3] [4]. Moreover, sorghum is potential to be used as ingredient in many processed foods such as bakery and pasta products. Development of sorghum flour to enhance its functionality will be beneficial [1].

Now, we are living in 4th industrial era in which time for preparing food is a matter. Therefore, the demand for convenience foods is increasing. Some examples of convenience food products include frozen food, instant pasta, instant noodles, and instant porridges. Research for developing instant products are growing. For instance, many types of flour have been modified to be instant flour which is used as an ingredient of convenience food. There are several technologies to prepare instant flour such as drum drying [5], spray drying [6], and extrusion [7]. Instant flour can also be prepared by using an ethanol-heating method. Several studies have been conducted on the preparation and characterization of ethanol-treated flour [8] [9].

However, preparation and characterization of ethanol-treated flour from sorghum has not been performed. Therefore, the objective of the current study was to prepare an ethanol-treated flour from
sorghum. Moreover, the chemical, physical and gelatinization properties of the ethanol-treated sorghum flour was investigated.

2. Materials and methods

2.1. Materials

White sorghum flour was obtained from local supplier in Bandung, West Java. Analytical grade ethanol was purchased from Merck Ltd. (Merck Pte Ltd. Singapore).

2.2. Methods

2.2.1. Sample preparation. A method to prepare ethanol-treated sorghum flour (ETF) was adopted from method to prepare ethanol-treated starch as described by Sarifudin et al. [9]. Sorghum flour (15 g) was mixed with 135 ml of ethanol (45%, 50% and 55% v/v ethanol in water) in ratio of 1:9. The slurry was heated in a Schott bottle at three temperature levels (60, 80 and 100 °C) for 30 min by using a water bath. Following the thermal treatments, the samples were then cooled at room temperature for about 2 hrs. The sediment was collected and washed with absolute ethanol for three times. The wet powder then was vacuum dried at 55 °C for 24 hrs. Finally, the sample was milled by using a Phillips grinder then sieved at 40 mesh.

2.2.2. Sample analysis. The chemical properties of sample including moisture and ash content were determined according to AOAC method (AOAC, 2005). Color of the sorghum flour (L*, a*, b*) was observed using a colorimeter (3NH-NH300, China). Whiteness degree of sorghum flour was determined using following equation: 100 – [(100 – L*)² + a*² + b*²]¹⁄₄ [10]. Pasting properties of sample were investigated by using Rapid Viscosity Analyzer (RVA, Model TecMaster Newport Scientific, Australia).

Statistical analysis by using One-way Analysis of Variance (ANOVA) method was performed to determine the mean difference between samples (IBM SPSS Statistics 25, 2018). A post hoc by using a Duncan Multiple Range Test (DMRT) method was conducted to confirm the significant differences between samples at a confidence level of 95% (p≤0.05).

3. Result and discussion

3.1. Chemical characteristics of sorghum flour

The chemical properties of the ethanol-treated sorghum flour were presented in Table 1. The result showed that ethanol concentration did not significantly affected to the moisture and ash content of ethanol-treated sorghum flour. Heating temperature affected to the moisture content, but did not affected to the ash content.

The moisture content of ethanol-treated sorghum flour ranged from 10.13% to 12.09%. The moisture content of sorghum flour increased with increasing heating temperature. Sorghum flours were heated at 100 °C has the highest moisture than other temperature and it has no significant differ with native sorghum flour which has 11.34% of moisture. Based on Codex Standard 173-1989, the moisture content of sorghum flour should not exceed 15%.

The ash content of ethanol-treated sorghum flour ranged from 0.54% to 0.63%, significantly lower than the native sorghum flour which has 0.76% of ash. During ethanol treatment process, sorghum flour was washed several times which can removed impurities materials. It induced the ethanol-treated sorghum flour has a lower ash content than native sorghum flour that still contained impurities materials. The ash content of ethanol-treated sorghum flour still corresponds to Codex Standard (max 1.5%).
Table 1. Moisture and ash content of ethanol-treated sorghum flour

| Ethanol concentration (%) | Heating temperature (°C) | Moisture (%) | Ash (%) |
|---------------------------|--------------------------|--------------|---------|
| 45                        | 60                       | 10.13 ± 0.76<sup>Ab</sup> | 0.54 ± 0.05<sup>Aab</sup> |
| 80                        | 60                       | 10.21 ± 0.03<sup>Ab</sup> | 0.61 ± 0.02<sup>Ab</sup> |
| 100                       | 60                       | 12.09 ± 0.11<sup>Aa</sup> | 0.56 ± 0.07<sup>Aab</sup> |
| 50                        | 60                       | 10.39 ± 0.25<sup>Aa</sup> | 0.63 ± 0.03<sup>Aa</sup> |
| 80                        | 60                       | 10.93 ± 0.14<sup>Ab</sup> | 0.62 ± 0.04<sup>Ab</sup> |
| 100                       | 60                       | 11.42 ± 0.72<sup>Aa</sup> | 0.60 ± 0.02<sup>Aa</sup> |
| 55                        | 60                       | 10.24 ± 0.46<sup>Ab</sup> | 0.62 ± 0.01<sup>Ab</sup> |
| 80                        | 60                       | 10.78 ± 0.67<sup>Aa</sup> | 0.59 ± 0.03<sup>Aa</sup> |
| Control                   |                           | 10.59 ± 0.73<sup>Ba</sup> | 0.60 ± 0.03<sup>Ba</sup> |

The capital letters denote significant difference (p<0.05) between the different ethanol concentration. The small letters denote significant difference (p<0.05) between different heating temperature.

3.2. Physical characteristics of sorghum flour

Whiteness degree of the sorghum flour was determined to evaluate its physical characteristics. It was one of the important parameter to evaluate flour quality. Whiteness degree represent the ability of flour to reflect light coming to its surface [11]. The whiteness degree of the ethanol-treated sorghum flour were presented in Figure 1.

![Figure 1. Whiteness degree of ethanol-treated sorghum flour](image)

Based on Figure 1, ethanol concentration and heating temperature significantly affected to the whiteness degree of ethanol-treated sorghum flour. Generally, ethanol-treated sorghum flour had the higher whiteness degree than native sorghum flour. Sorghum flour which treated in 55% ethanol and heated at 80 °C had the highest whiteness degree (86.87%). Modification treatment on sorghum flour
known affected to the whiteness degree increased. As reported by Wulandari et al. [12] and Kinanti et al. [13] who modified sorghum flour by amylase treatment and lactic acid submersion treatment.

According to [14] that whiteness degree of flour affected by protein content. The higher protein content of the material, whiteness degree of flour produced might be lower. This study used heating as one of treatment. Heating process can induced denaturation of protein, which caused protein content decreased. Hence, ethanol-treated sorghum flour which used heating in the process might had the higher whiteness degree than native sorghum flour. Eskin et al. at Kinanti et al. [13] stated that enzymatic browning reaction can prevented by oxygen removal. Oxygen can be reduced by submersion. Therefore, ethanol-treated sorghum flour had the higher whiteness level than native flour because had submersion by ethanol during the process.

3.3. Gelatinization profile of sorghum flour
Gelatinization profile is required to identification the changes in amylographic response, predicting starch properties during processing and initial data identification for processing starch/flour equipment set up [15]. The gelatinization parameters were observed are peak viscosity, final viscosity, breakdown and setback. As presented in Table 2, ethanol concentration only had an significantly effect on the breakdown parameter, whereas heating temperature significantly affecting all gelatinization parameters tested.

Table 2. Gelatinization parameters of ethanol-treated sorghum flour

| Ethanol con. (%) | Heating temp. (°C) | Peak viscosity (cP) | Final viscosity (cP) | Breakdown (cP) | Setback (cP) |
|-----------------|-------------------|---------------------|----------------------|----------------|--------------|
| 45              | 60                | 427.50 ± 16.87<sup>ab</sup> | 4196.50 ± 27.58<sup>ad</sup> | 381.50 ± 14.04<sup>Bc</sup> | 4150.50 ± 14.76<sup>Ac</sup> |
| 80              |                   | 257.00 ± 15.45<sup>Aa</sup> | 3303.00 ± 23.44<sup>Ac</sup> | 226.50 ± 11.92<sup>Bb</sup> | 3272.50 ± 16.97<sup>Ab</sup> |
| 100             |                   | 412.50 ± 4.95<sup>Aab</sup> | 1595.50 ± 19.81<sup>Ab</sup> | 215.50 ± 8.79<sup>Bb</sup> | 1198.50 ± 13.65<sup>Aa</sup> |
| 50              | 60                | 552.00 ± 12.73<sup>Ab</sup> | 4461.50 ± 17.22<sup>Ad</sup> | 498.00 ± 9.91<sup>BCc</sup> | 4407.50 ± 14.39<sup>Ac</sup> |
| 80              |                   | 285.00 ± 17.68<sup>Aa</sup> | 3430.50 ± 21.32<sup>Ac</sup> | 235.00 ± 13.54<sup>BCb</sup> | 3380.50 ± 17.17<sup>Ab</sup> |
| 100             |                   | 359.50 ± 16.87<sup>Aa</sup> | 1713.00 ± 20.15<sup>Ab</sup> | 217.50 ± 12.93<sup>BCb</sup> | 1571.00 ± 16.21<sup>Aa</sup> |
| 55              | 60                | 633.50 ± 10.50<sup>Ab</sup> | 4713.50 ± 16.26<sup>Ad</sup> | 571.00 ± 12.73<sup>Cc</sup> | 4551.00 ± 14.04<sup>Ac</sup> |
| 80              |                   | 517.00 ± 11.11<sup>Ab</sup> | 3332.50 ± 12.85<sup>Ac</sup> | 417.00 ± 5.66<sup>Ch</sup> | 3232.50 ± 18.31<sup>Ab</sup> |
| 100             |                   | 285.00 ± 7.07<sup>Aa</sup> | 1924.00 ± 8.25<sup>Ab</sup> | 227.50 ± 7.71<sup>Ch</sup> | 1033.50 ± 5.47<sup>Aa</sup> |
| Control         |                   | 409.00 ± 10.91<sup>Ab</sup> | 1543.00 ± 12.23<sup>AAa</sup> | 160.50 ± 1.92<sup>AAa</sup> | 4934.00 ± 4.96<sup>Bd</sup> |

The capital letters denote significant difference (p<0.05) between the different ethanol concentration. The small letters denote significant difference (p<0.05) between different heating temperature.

According to Kamakar et al. [15], peak viscosity is the top point of the paste consistency during the heating process or the condition while starch granules attained the maximum expansion and broked subsequently. It is used as convenient cooked starch indicator during heating and as paste strength indicator. Sorghum flours were heated at 60 °C at all ethanol concentrations had the higher peak viscosity than other flours. Meanwhile, most of sorghum flours were heated at 80 °C and 100 °C had the lower peak than native sorghum flour. According to Zu and Liu [16] it is mainly caused by the crystalline structure of starch granules was destroyed in the process of alcoholic-alkaline treatment.

Peak viscosity decreased on heating temperature treatment more than 60 °C might caused by amylase and amylpectin bonds of the starch/flours were hydrolized and its content were decreased. Amylopectin has branched structure, double helix formed and contributed to starch expansion. Hydrogen bonds can broken off while starch was heated. Because amylase and amylpectin bonds were hydrolyzed, the ability of starch to absorb water and swell was reduced and peak viscosity became lower [12].
Final viscosity is defined as the ability of the starch to form a lumpy paste or gel after heating or cooling phase [17]. In this study, final viscosity of ethanol-treated sorghum flours were higher than native sorghum flour. It is might caused by the starch of native sorghum flour was not hydrolized and the amylose of the starch was not disintegrate. According to Kinanti et al. [13], unhydrolyzed amylose induced a small amount of short-chain saccharides formed. Amylose which is a long-chain saccharides inclined to be difficult to bind a water when not heated. Hence, native sorghum flour and water were inclined to be segregate and causes a low flour viscosity.

Based on Table 2, as heating temperature treatment was increased the final viscosity of sorghum flours were decreased. Kurakake et al. [18] reported that the swelling power and solubility of maize starch was decreased with increasing treatment temperatures. It is due to the starch granules become more rigid during treatment at the higher temperature. These results might in line with viscosity parameter. Generally, swelling power and solubility of starch proportionate to starch/flour viscosity. A high final viscosity can be achieved if the swelling power and solubility of the starch/flour also high.

Breakdown constitutes a very important parameter describing the stability of starch granules during heating and paste consistency [19]. Ethanol-treated sorghum flour had the higher breakdown than native sorghum flour. This indicates that paste of ethanol-treated sorghum flours were unstable during heating. High breakdown would not be a problem for instant product because it was expected not to used excessive heat during cooking.

Setback parameter is indicator of retrogradation and syneresis of paste [20]. As shown in Table 2, the setback of ethanol-treated sorghum flours were decreased as heating temperature treatment was increased. The higher value of setback, the higher tendency to form a gel during cooling. The high value of setback indicate a tendency for the occurrence of retrogradation. According to Beta and Corke [21] the setback can used to measure the recrystallization capability during cooling, related to retrogradation and starch rearrangement. Singh et al. [22] reported that at the time of gelatinization, amylose out of the starch granules and can form amylose-fat complexes. This complex formation can also reduce the tendency of amylose to bind, form a gel and being retrogradation thus inhibiting the hardening rate during heating.

4. Conclusion
In summary, ethanol treatment largely alter the physicochemical and gelatinization properties of white sorghum flour. Ethanol-treated on sorghum flour increased whiteness degree, peak viscosity, final viscosity and breakdown also decreased ash content and setback viscosity than its native flour. Ethanol concentration had significant effect on whiteness degree and breakdown parameter. While heating temperature affected significantly on moisture content, whiteness degree, peak viscosity, final viscosity, breakdown and setback of sorghum flour. Ethanol-treated sorghum flour produced from ethanol concentration of 55 % and heating temperature of 60 °C showed superior characteristics than the other treatments in which it’s properties including moisture content, ash content, whiteness degree, peak viscosity, final viscosity, breakdown and set-back properties were 10.24 %, 0.62 %, 84.85 %, 633.50 cP, 4713.50 cP, 571.00 cP, 4551.00 cP, respectively.

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