# Effect of using different clarifying agents and temperature on physicochemical and sensory properties of sweet sorghum syrup extract

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#### Abstract

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The utilization of sorghum crops to produce value-added products such as alternative natural sweeteners has been increasing. Knowledge of the sweet sorghum juice clarification process to yield sweet sorghum syrup as an alternative sweetener is essential to get the best result and improve the quality of the syrup. This research aimed to study the sorghum syrup extraction process by using clarifying agents: Ca(OH)<sub>2</sub>, bentonite, and activated carbon, followed by the heating process at 70°C or 80°C. Sorghum stalks were milled to produce sorghum juice then clarified and heated up to make sweet sorghum syrup to reach 68.78-76.78% Bx. The clarification process was done to reduce particles in sorghum juice before the heating process. In this work, the psycho-chemical (pH, colour, turbidity, reducing sugar) and sensory properties (flavour and aroma) of sorghum syrup after clarification and heating process were analyzed. A randomized block design was used as the experimental design. Adding Ca(OH)<sub>2</sub> and heating at 80°C resulted in sorghum syrup with higher pH and lower turbidity than that heated at 70°C. The evaluation revealed that the combination of Ca(OH)<sub>2</sub> and 5% bentonite has the sweetest flavour, caramel aroma, and preferred psycho-chemical properties showed in pH (5.28), turbidity (35.56 NTU), and reducing sugar (57.52%).

#### 1. Introduction

Sorghum (Sorghum bicolour (L.) Moench) is a crop within the grass family which can grow in semi-arid or dry conditions. Originally from East Africa but it is widely grown throughout the world, particularly in developing countries in the area of Asia, Africa, and South America, especially in the tropical and subtropical areas. Almost every part of the plant can be utilized as a raw material in making derivative products such as flour, syrup, and sugar. Sweet sorghum stalk was found profitable in the making of sweet sorghum syrup (Kumar *et al.*, 2013; Noerhartati and Rahayuningsih, 2013).

A sorghum stalk is a serial sequence of internodes and nodes that are cylindrical in shape, and has no cambium. The last four nodes of the sorghum stalk have a high amount of starch and sucrose, while the beginning of the nodes has a high amount of glucose and fructose. Sorghum syrup came from the sweet sorghum juice extracted by milling the mature stalk. The quality of sweet sorghum juice may be equivalent to sugarcane juice. Its sugar content ranges from 10 to 25%, and consists of 70% sucrose, 20% glucose, and 10% fructose (Shoemaker and Bransby, 2010). Generally, the amount of sweet sorghum juice yielded from the milling process ranges from 50-60% of the stalk's total weight (Wu *et al.*, 2010).

To deliver a good quality sweet sorghum syrup, a clarification process is required. The clarification process is used to separate the impurities, such as pigments and protein, from the sweet sorghum juice prior to the evaporation process for producing a sweet sorghum syrup (Ratnavathi *et al.*, 2016). Furthermore, the temperature and agitation during the evaporation process also has an impact on the yielded syrup. A total solid content analysis was done to ensure that the yielded syrup met the desired psycho-chemical and sensory properties.

The sorghum juice clarification process can be achieved by adding flocculants, adsorbents, or both combined. The adsorbents used were similar to those applied in sugarcane extraction, such as activated carbon, bentonite, and zeolite. Prati and Moretti (2010) purified sugarcane by adding 1.25% calcium hydroxide and aluminum polychloride (APC) to the sugarcane juice FULL PAPER

until it reached the desired pH, it was followed by evaporation process at 65°C for 50 mins. Their finding indicates that a combination of the addition of 60 ppm APC at the pH of 8 was the best clarification method to get the desired sensory properties and turbidity (Prati and Moretti, 2010). Laksameethanasan et al. (2012) reported that the clarification process using  $Ca(OH)_2$  as a flocculating agent combined with bentonite and/or activated carbon affected the physicochemical properties of sweet sorghum syrup, the addition of 3% bentonite and 0.3% activated carbon achieved the best result. Adsorbent addition is a simple clarification process that can improve the properties of sweet sorghum syrup. The adsorbent bind to the unwanted organic acids, flavonoids, polyphenols, or other impurities which cause the undesired dark colour to develop into a much clearer and less turbid syrup (Laksameethanasan et al., 2012).

The short crop rotation of sweet sorghum is beneficial for producing a high amount of sweet sorghum syrup. However, an effective clarification process should be undertaken to develop the possibility of sweet sorghum syrup becoming an alternative natural sweetener for food products. Therefore, this research aims to find a simple way of clarification by utilizing flocculating agents and adsorbents to produce sweet sorghum syrup with preferable physicochemical and sensory properties. The results may lead to sweet sorghum syrup broad utilization as an alternative natural sweetener in the future.

#### 2. Materials and methods

#### 2.1 Materials

The research was carried out at the Laboratory of Food Processing, Faculty of Agricultural Technologies, Widya Mandala Catholic University Surabaya. The sweet sorghum variety was Numbu, planted by the farmer on Wuluhan, Jember (latitude -8.328253°N and longitude 113.547255°E), East Java, Indonesia in December and harvested in March. Ca(OH)<sub>2</sub>, bentonite, and activated carbon were purchased from the local chemical supplier in East Java. After chopping and washing, the sorghum stalks were milled to produce raw sorghum juice. Following that, it was filtered using a strainer and muslin cloth before being kept frozen at -20°C. The frozen juice was thawed at room temperature about 29°C prior to the experiments. In this experiment, three clarification methods were carried out.

#### 2.2 Clarification methods

#### 2.2.1 Clarification by adding flocculant Ca(OH)<sub>2</sub>

After adding the flocculant  $Ca(OH)_2$  (1 g/L), the juice was kept for one day at 5°C, and then clarified juice was strained with the filter paper. The resulting juice

then underwent either an evaporation process or was further treated with bentonite or activated carbon.

# 2.2.2 Clarification by adding flocculant $Ca(OH)_2$ and bentonite

Bentonite in the concentration of 3% or 5% was then added to the Ca(OH)<sub>2</sub> treated juice (see section 2.2.1) and was left to sit there for 30 min. Afterward, it was filtered with ashless filtered paper (Whatmann grade 40 diameter 125 mm) and followed on with the evaporation process.

# 2.2.3 Clarification by adding flocculant $Ca(OH)_2$ and activated carbon

Activated carbon in the concentration of 0.5% or 2.5% was then added to the Ca(OH)<sub>2</sub> treated juice (refer section 2.2.1) juice and was left to sit there for 30 mins. Afterward, it was filtered with ashless filtered paper (Whatmann grade 40 diameter 125 mm) and followed on with the evaporation process.

#### 2.2.4 Evaporation process on treated juice

The clarified juices were evaporated in an oven at 70°C and 80°C to obtain a minimum of 68% Bx syrup.

#### 2.3 Analytical methods

The physicochemical analysis was done by evaluating the proximate analysis, pH, total soluble solids, turbidity, colour, and reducing sugar content. Proximate analysis was done on the raw juice obtained from the milling of sweet sorghum stalk prior to the clarification process. It consisted of protein, fat, moisture, ash, and carbohydrate content which were determined with the procedure recommended by AOAC (2005). Meanwhile, the other analysis was done on the sweet sorghum syrup yielded from the experiments.

The pH was analyzed using a pH meter (Schoot LAB 850, Camlab, UK), while the total soluble solid content analysis was performed by using a hand refractometer (Atago N3E, Cole-Palmer, India) and presented in%Brix. The colour of the sweet sorghum syrups was evaluated using a colour reader (Minolta CR400 Co. Ltd., Osaka, Japan) which was calibrated with a standard white plate at the beginning of each analyzing section. The data was explained in colour space indexes L, a\*, b\*. The values of Chroma (C) are described as square root  $a^2 + b^2$  while hue (h) describe as arc tan-1 b\*/a\* (Teixeira et al., 2012). Meanwhile, the turbidity of the sweet sorghum syrup was analyzed using a turbidimeter (Obeco Hellige 966-IR, Florida, USA) and counted in the Nephelometric Turbidity Unit (NTU). Finally, the total reducing sugar was carried out using the Somogyi-Nelson method (Nelson, 1944) and the data presented in percentage.

Sorghum syrup with the highest content of total reducing sugar from each experiment was selected to be analyzed for its fructose and glucose content by using HPLC – RI. Methods of analysis using HPLC-RI was based on the experiment by Zaky *et al.* (2017) and AOAC (1977).

#### 2.4 Sensory analysis

#### 2.4.1 Selection of panel members

The initial criteria for the sensory panels were as follows: age 19-20 years old, their interest and willingness, their sensory test-related experience, and their ability to follow the training sessions carried out later. A questionnaire was used to collect the data. Information related to the experiments was announced to the candidates. This included the aim of the research, the origin of the sample to eat, and the testing schedule. Candidates who agreed to be involved in this research were asked to declare their compliance in the informed consent form. After this, the initial sensory panel members undertoon an odor and basic taste recognition test (Civille and Carr, 2016).

An odor recognition and description test were held using the matching test method (Ciappini *et al.*, 2013). 20% solution came from three different samples were made and put in nine small sensory glassware. Then, it was put randomly in the rows of three. Each candidate was asked to match samples in the second and third rows to the ones in the first row. Candidates who matched the samples correctly were to pass the aroma recognition test.

Ranking tests (Lawless and Heymann, 2010) were used in order to select the panels through basic taste recognition. For such screening arrays, an arrangement of sweet, sour, and bitter tastes was designed. Five levels of sucrose and citric acid solution with the concentration of 0-10% and 0-1.5% respectively were given to analyze the sweetness and sourness levels of the candidates. Moreover, the bitterness test was done using a caffeine solution with a concentration of 0-2.6%. Candidates able to rank samples correctly or invert only adjacent pairs were selected as a panel. Sixteen-panel members have been selected as they fulfilled the criteria.

#### 2.4.2 Panels training sessions

The training was organized in three sessions in which panel members become familiar with the sample and its sensory attributes. Training sessions were given to the selected panels to develop their abilities to recognize and identify flavour and aroma, improve the sensitivity, and perform consistent analysis. Instruction was given on how to smell and taste the samples, wash the mouth between samples, and dissolve the sample in the mouth. The aroma definition is presented in Table 1.

# 2.4.3 Actual sensory analysis

The actual analysis has been conducted after all the training sessions have been completed and the panels were capable of recognizing at least 75% of the samples. The test was carried out conferring the general direction of the sensory test (ISO 6658, 2005) with individual rooms free of odors and strange noises (ISO 8589, 2005). Sweet sorghum syrup was diluted in warm water to obtain the concentration of 8% in the solution. Flavour analysis was conducted by using the triangle test method. Coded samples in a single tray for each panel were prepared, placing between 35 to 40 mL of solution in a glass of 50 mL. The glasses should be clean before use and stored at room temperature  $(25\pm2^{\circ}C)$ . The results were interpreted by calculating the number of correct responses to match the 5% risk of error (Civille and Carr, 2016). A similar sample was used to define the aroma analysis. It was put in the small glass jar as being done in the flavour analysis, covered with aluminum foil with three small holes to catch the first odor impression by smelling the samples after rotating the container. The panels have to explain the aroma of the sample by the family (Table 1) to which belongs the odor perception and describe its intensity on 1 to 8 scale. A similar scale was also used to describe the intensity of sweetness, sourness, and bitterness in the flavour analysis.

Family	Reference
Citrus, lemony	Lemon, orange, bergamot
Sweet	Sugar, candy
Burned caramel	Black sugar, caramel, molasses
Spicy	Clove, nutmeg, cinnamon, thyme
Sour, fermented	Wine, alcoholic beverage
Sour, vinegary	Acetic acid
Floral	Roses, orange blossom, jasmine
Wet, earthy	Wet grass, raw mushroom, wet wood
Rancid	Over-oxidized oil
Burnt, scorced	Smoke cigarette ash
Green, grassy	grass clipping, fresh leaves crushed

#### 2.5 Statistical analysis

For each physicochemical property, the average and standard deviation achieved from repetitions of the same samples were calculated. Analysis of variance (ANOVA) was conducted to define the difference between samples with a 5% significant level (p<0.05), followed by Duncan Multiple Range Test (DMRT). For the sensory properties, the aroma defined by the panels was presented in the spider web, while the flavour analysis was statistically analyzed by counting the correct response. If the number of correct responses was at least matched the critical number of correct responses, then the sample was assumed as different from the others.

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#### 3. Results and discussion

### 3.1 Sweet sorghum juice chemical composition

The sweet sorghum stalk has sat for three days after being harvested to give a chance for enzymatic reaction in developing reducing sugar more before it has been milled using a crusher. The stalks were planted for 3 months before it was ready to be harvested. Leaves and other parts of the plant were removed before the milling process have been carried out to produce raw green sweet sorghum juice. This process may lead to an increase in yield and taste (Ratnavathi et al., 2016). The extracted sorghum juice was then filtered by using a muslin cloth to separate it from bagasse or other impurities that may be followed in the milling process. The pH of the sorghum stalk juice was 4.04±0.02 with the total soluble solid as high as 8.7%Brix. Its turbidity reached 1165.67±1.53 NTU. From the proximate analysis, it was found that the sorghum stalk juice contained 0.91±0.09 ash (g/100 g), 0.03±0.00 fat (g/100 g), 0.43±0.09 protein (g/100 g), and 4.55±0.19 carbohydrate (g/100 g). Sweet sorghum stalk has a waxy surface that provides the fat composition of the sweet sorghum syrup (Eggleston et al., 2022). The sweet sorghum juice was found to have a high moisture content as well, reaching about 94.09 g/100 g. The juice generally contained more than 70% water, depending on the way of milling and the tools used (Ratnavathi et al., 2016). After the first filtration process using a muslin cloth, the sweet sorghum juice still contained high amounts of impurities. Following that, the clarification process was done by using 1 g/L CaOH<sub>2</sub>(F) only or followed by 0.5% activated carbon (A1), 2.5% activated carbon (A2), 3% bentonite (B1), or 5% bentonite (B2), and continued to evaporation process in 70°C (S1) or 80°C (S2).

### 3.2 pH analysis

The pH of raw sweet sorghum juice after filtration was 4.02. The addition of Ca(OH)<sub>2</sub> increased the pH up to 4.75. Further clarification process by adding the flocculant and adsorbents brought a sweet sorghum syrup with a pH of 4.86-5.85 (Figure 1). Sweet sorghum juice naturally contains a high amount of organic acid, which resulted in a low pH (Castro-Munoz et al., 2022). However, the clarification method would lead to pH elevation (Willis et al., 2013). The pH of the groups that evaporated at 70°C was relatively higher than the ones that evaporated at 80°C, except for the groups which only got flocculant addition (FS1 and FS2), as shown in Figure 1. In the lower pH, enzyme activity which turns sucrose into glucose or fructose was higher than in the high pH condition which may affect the quality of sweet sorghum syrup yielded (Andrzejewski et al., 2013).

The addition of 2.5% activated carbon resulted in lower pH compared to those with 0.5%. A similar trend was shown in the addition of bentonite followed by evaporation at 70°C. This did not occur in the ones evaporated at 80°C but the result of both evaporation temperatures was not statistically different (Figure 1). The pH of sweet sorghum syrup clarified by using 0.5% activated carbon then evaporated in 70°C (A1S1) was 3.5% higher than FS1, while the pH of a similar group evaporated in 80°C (A1S2) was 10.5% lower than FS1. By the addition of 2.5% activated carbon followed evaporation at 70°C (A2S1) and 80°C (A2S2), the pH of extracted sweet sorghum syrup was 16.9% and 17% lower than FS2, respectively. Nevertheless, the pH of FS1 was not statistically different from A1S1 and A2S1. The different trends appeared in those evaporated at 80°C, where the FS2 were shown statistically different from A1S2 and A2S2 (p<0.05) (Figure 1).



Figure 1. pH analysis for sweet sorghum syrup clarified by using 1 g/L CaOH2 (F), 0.5% activated carbon (A1), 2.5% activated carbon (A2), 3% bentonite (B1), or 5% bentonite (B2), then evaporated in 70°C (S1) or 80°C (S2). Values with different superscripts are statistically significantly different ( $\alpha = 0.05$ ).

Syrup clarified using bentonite had higher pH than activated carbon. The addition of 3% (B1S1) and 5% bentonite (B2S1) followed by evaporation at 70°C increased the pH by 7.4% and 3.5%. However, a similar clarification method followed by evaporation at 80°C (B1S2 and B2S2) decreased the pH by 11% and 10%. There were no significant differences between B1S1 and B2S1 in terms of pH. However, another trend appeared in the ones evaporated at 80°C, which showed that the pH of FS2 was significantly different if compared to B1S2.

Adsorption was influenced by many parameters such as temperature, pH, and adsorbent concentration (Jahed *et al.*, 2014). The evaporation process may lead to increasing acidity because of the reaction between unstable organic substances with the reducing sugar. Decomposition of reducing sugar may lead to the formation of organic acid which may cause a pH decrease (Khalil et al., 2018). This can explain the analysis shown in Figure 1, where most of the samples that evaporated at 80°C were found to have lower pH than those that evaporated at 70°C.

#### 3.3 Total soluble solid analysis

A previous study showed that TSS in raw sorghum syrup was 6.2-21.4%, depending on the physiological stages of growth (Atokple et al., 2014). The highest sugar percentages in the stalk juice were achieved in the later phase of physiological growth. Before clarification, the total soluble solids (TSS) content of raw sweet sorghum juice was 9.3%. After flocculation, the TSS was reduced to 8.7%. The juice was then clarified and evaporated until reached 68.78-76.78%Brix (Figure 2). The data showed that the TSS content of the sweet sorghum syrup evaporated at 80°C was higher than that evaporated at 70°C, except for the A2S1 and B1S1. The higher reducing sugar content in a sample will contribute to its TSS (Eggleston et al., 2022).

TSS content of A1S1 and A2S1 were 5.6% and 4.1% lower than the TSS content of FS1 respectively. The TSS content of A1S1 was significantly different from FS1, but consider similar if compared to A2S1 (p<0.05). On the other hand, A1S2 and A2S2 had lower TSS content than FS2. However, The TSS content of FS2 was statistically different from A2S2 but it was not the case if compared to A1S1 (Figure 2).



Figure 2. Total soluble solid analysis for sweet sorghum syrup clarified by using 1 g/L CaOH2 (F), 0.5% activated carbon (A1), 2.5% activated carbon (A2), 3% bentonite (B1), or 5% bentonite (B2), then evaporated in 70°C (S1) or 80°C (S2). Values with different superscripts are statistically significantly different ( $\alpha = 0.05$ ).

Clarification by bentonite resulted in higher TSS content than clarification by activated carbon. The TSS content of B1S1 and B2S1 were 2.2% higher and 4.9% lower than the TSS content of FS1 respectively. However, the TSS content of B1S2 and B2S2 were 8.7% and 3.7% lower than FS2. While evaporated at 70°C, the TSS content of B2S1 was not significantly different from FS1. On the other hand, the TSS content of B2S1 is statistically difference from B1S1. A similar trend appeared in the ones that evaporated at 80°C.

# 3.4 Turbidity analysis

The turbidity of raw sweet sorghum juice was 1165.67 $\pm$ 1.53 NTU. After the addition of Ca(OH)<sub>2</sub>, the turbidity was reduced to 24.37±0.47 NTU. The turbidity of the final products after the complete clarification and evaporation process were 35.56-272.44 NTU (Figure 3a). The turbidity of sweet sorghum syrup evaporated at 80°C was relatively lower than that evaporated at 70°C. This showed that higher evaporation temperature combined with the addition of flocculant and adsorbent would improve the separation and deposition of the nonsugar soluble compounds from the sweet sorghum juice. The effectiveness of the clarification methods as indicated by the amounts of impurities that could be separated from the juice which led to a clear and less turbid solution (Andrzejewski et al., 2013). By the end of the evaporation process, non-sugar soluble compounds such as protein and pigments will coagulate and a less turbid solution was produced (Atokple et al., 2014; Khalil et al., 2018).



Figure 3. Turbidity (left) and viscosity (right) analysis for sweet sorghum syrup clarified by using 1 g/L CaOH<sub>2</sub> (F), 0.5% activated carbon (A1), 2.5% activated carbon (A2), 3% bentonite (B1), or 5% bentonite (B2), then evaporated in 70°C (S1) or 80°C (S2). Values with different superscripts are statistically significantly different ( $\alpha = 0.05$ ).

Turbidity level for A1S1 and A2S1 was 32.9% and 64.5% lower than FS1. After evaporation at 70°C, it was clear that the turbidity level of A1S1 was significantly different from FS1 and A2S1. However, after evaporation at 80°C, the turbidity levels of A1S2 and

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A2S2 were 13.9% and 19.7% lower than FS2. There was no significant difference between the turbidity level of A1S2 and A2S2 while both were statistically different from FS2 (Figure 3a).

The turbidity level of sweet sorghum syrup clarified using bentonite and evaporated at 70°C was higher than that evaporated using activated carbon. However, the evaporation process at 80°C after the addition of bentonite resulted in a lower turbidity level, especially if compared to the FS2. The turbidity levels of B1S2 and B2S2 were 83.2% and 85.1% lower than FS2 respectively. A combination of clarification by 5% bentonite followed by evaporation at 70°C brought improvement in syrup turbidity. However, a similar trend did not appear in ones evaporated at 80°C, which showed that there was no significant difference between the turbidity level of B1S2 and B2S2. However, the value of pH could be related to the turbidity of the syrup. B1S2 and B2S2 had no significant difference in pH value, but both had lower pH values compared to B1S1 and B1S2. Turbidity reduction for samples clarified using bentonite were more pronounced on the lower pH, as the result of bentonite positively charged edge surface on low pH enables better adsorption of negatively charge nonsugars compounds (Jahed et al., 2014; Djordjevic et al., 2018).

The turbidity of raw juice is mainly caused by impurities such as polysaccharides and protein (Lee *et al.*, 2006; Mazumdar *et al.*, 2012). Reducing turbidity will improve the quality of the extracted syrup. The turbidity level of all groups clarified by using adsorbents were lower than that which was only treated by flocculant. Evaporation at 80°C was enough to coagulate protein compounds that may improve the clarification process. It was indicated by the low level of turbidity (Andrzejewski *et al.*, 2013). Nevertheless, the addition of a higher concentration of adsorbents led to a low level of turbidity in samples evaporated at the same temperature.

#### 3.5 Viscosity

The dissolved components in any material and the ratio of the solvent to the solute affect its viscosity. The lower the viscosity, the higher the flow rate value. The average flow rates obtained ranged from 0.1895 cm/s to 0.9113 cm/s (Figure 3b). The difference in flow rate was caused by differences in the ability of each type of clarifier used to separate non-sugar compounds from sweet sorghum juice. Evaporation at 70°C produced a higher flow rate than at 80°C, which means the viscosity of the sweet sorghum extract evaporated at 80°C was thicker than at 70°C. Evaporation at a higher temperature causes more water to evaporate; therefore, the sweet sorghum extract liquid becomes thicker. This is different from Willis *et al.* (2013) research results in lower viscosity at higher temperatures.

CaOH<sub>2</sub> was not significantly different from that of 5% bentonite, and both resulted in lower flow rates or were denser than other adsorbents. CaOH<sub>2</sub> was not significantly different from that of 5% bentonite, and both resulted in a lower flow rate or a thicker extract than other adsorbents. These results are similar to minerals in bentonites, such as calcium, sodium, and magnesium; therefore, the clarification mechanism is similar to that of calcium flocculants. The use of 2.5% activated charcoal was not significantly different from that of 3% bentonite at the two temperatures applied. Both treatments give a higher flow rate value. This study indicates that the difference in bentonite concentration significantly affects the flow rate or viscosity of the sweet sorghum extract.

#### 3.6 Colour analysis

The natural sweet sorghum juice colour was light green, indicating high chlorophyll content. After

Experimental	Value				
Group	L	a*	b*	С	°h
FS1	23.75±0.25	0.65±0.09 <sup>c</sup>	$0.55{\pm}0.05^{d}$	$0.85{\pm}0.04^{\rm bc}$	40.47±5.89 <sup>c</sup>
FS2	$24.25 \pm 0.39$	$0.80{\pm}0.13^{de}$	$0.43{\pm}0.13^{\circ}$	$0.91{\pm}0.17^{c}$	$28.11 \pm 4.22^{b}$
A1S1	$23.62 \pm 0.35$	$0.22{\pm}0.03^{a}$	$0.37{\pm}0.06^{bc}$	$0.43{\pm}0.06^{\rm a}$	59.24±3.72 <sup>e</sup>
A1S2	$24.17 \pm 0.18$	$1.15{\pm}0.05^{\rm f}$	$0.57{\pm}0.08^{d}$	$1.28{\pm}0.05^{d}$	$26.34{\pm}3.46^{b}$
A2S1	23.20±1.00	0.67±0.13 <sup>c</sup>	$0.28{\pm}0.06^{ab}$	$0.72{\pm}0.14^{b}$	$22.93{\pm}1.85^{b}$
A2S2	$23.35 \pm 0.86$	$0.47{\pm}0.06^{b}$	$0.58{\pm}0.03^d$	$0.75{\pm}0.03^{b}$	$51.41 \pm 4.42^{d}$
B1S1	$25.15 \pm 1.91$	$0.42{\pm}0.03^{b}$	$0.75 {\pm} 0.05^{e}$	$0.86{\pm}0.05^{ m bc}$	60.93±2.25 <sup>e</sup>
B1S2	$24.08 \pm 0.38$	$1.37{\pm}0.03^{g}$	$0.60{\pm}0.05^{d}$	$1.49{\pm}0.03^{e}$	$23.73 {\pm} 1.72^{b}$
B2S1	25.58±2.12	$0.77 {\pm} 0.06^{cd}$	$2.87{\pm}0.03^{\rm f}$	$2.97{\pm}0.04^{\rm f}$	$75.04{\pm}1.02^{\rm f}$
B2S2	25.88±0.28	$0.92{\pm}0.03^{e}$	$0.23{\pm}0.03^{a}$	1.95±0.03 <sup>e</sup>	$14.21 \pm 1.65^{a}$

Table 2. Colour analysis in L, a\*, b\* parameters.

Sweet sorghum syrup was clarified using 1 g/L CaOH<sub>2</sub> (F), 0.5% activated carbon (A1), 2.5% activated carbon (A2), 3% bentonite (B1), or 5% bentonite (B2), followed by evaporation in 70°C (S1) or 80°C (S2).

flocculant addition, the colour had changed into clear brown. With the addition of adsorbent followed by filtration, the colour changed into clear yellow. Colour analysis of the sweet sorghum syrup used colour reader CR400 (Minolta), and the colour indication is determined by using the value of C and °hue (°h). The values of a\*, b\*, C, and °h are shown in Table 2. The positive value of a\* indicates red, whereas the positive value of b\* indicates yellow colour (Teixeira *et al.*, 2012). The colour measurement results showed that the L\* value is 23.30-25.58, which means sweet sorghum syrup is in the dark colour range. The value of a\* is 0.22-1.37, while the value of b\* is 0.23-2.87. From calculating the values of a\*, and b\*, the values for C and oh are 0.43 -2.97 and 14.21-75.04, respectively.

The higher the L value, the brighter the sweet sorghum extract. Based on Table 2, B1S2 had a more redness element, while B2S1 had a more yellow element. B2S1 has the highest value of yellowness, while B2S2 has the lowest. All treatment results have a colour that is classified as dark because the L value is below 50. Based on the resulting L value, A2S1 has the darkest colour while B2S1 has the lightest colour, although there is no significant difference between all groups. The darker colour represented the Maillard reaction during processing which may lead to the formation of melanoidin and 5-hydroxymethylfurfural (5-HMF) due to thermal degradation of sucrose and fructose in a more acidic nature (Solis-Fuentes et al., 2019; Castro-Munoz et al., 2022). Melanoidin formation is a product of sugar and amino acid condensation after the evaporation process using heat treatment (Simaratanamongkol and Thiravetyan, 2010). Based on the C value, A1S1 has the dullest, whereas B2S1 has the lightest colour.

#### 3.7 Reducing sugar content

Evaluation of the effect of the clarification process on the content of reducing sugars was to determine the clarification method that gives the highest yield. Figure 4 shows that the reducing sugar content in the sample is 39.45-57.62%. A1S1 has the lowest reducing sugar content, while A2S2 has the highest. Five treatment groups, i.e. FS1, A1S2, A2S2, B1S1, and B2S2 were selected to analyze their glucose and fructose content. The glucose level obtained is about 10.2 - 19%, while the fructose level is 10.3 - 17.3%. FS1 had the lowest glucose and fructose levels, while B2S2 and A1S2 had the highest glucose and fructose levels (Table 3). The sucrose content for all groups was below 5%. Sweet sorghum syrup generally has lower sucrose and total sugar than cane sugar but has higher glucose, fructose, and organic acids (Asikin et al., 2018).



Figure 4. Reducing sugar content (%) of sweet sorghum syrup clarified by using 1 g/L CaOH<sub>2</sub> (F), 0.5% activated carbon (A1), 2.5% activated carbon (A2), 3% bentonite (B1), or 5% bentonite (B2), then evaporated in 70°C (S1) or 80°C (S2). Values with different superscripts are statistically significantly different ( $\alpha = 0.05$ ).

 Table 3. Reducing sugar composition of sweet sorghum syrup

 from different extraction method

Experimental Group	Glucose (%)	Fructose (%)
FS1	10.16±0.56	$10.31 \pm 0.15$
A1S2	$14.92 \pm 2.24$	$17.27 \pm 0.17$
A2S2	17.39±1.35	$16.30 \pm 1.43$
B1S1	17.80±1.65	17.06±1.54
B2S2	$19.02 \pm 2.00$	14.14±1.46

Sweet sorghum syrup was clarified using 1 g/L CaOH<sub>2</sub>(F), 0.5% activated carbon (A1), 2.5% activated carbon (A2), 3% bentonite (B1), or 5% bentonite (B2), followed by evaporation in 70°C (S1) or 80°C (S2).

#### 3.8 Sensory properties

#### 3.8.1 Aroma profile

Descriptive analysis was to perceive the aroma profile of all groups. A total of sixteen selected panels, consisting of 6 males and ten females, were involved in the analysis. Panels were asked to identify two dominant scents that arose in the first-time testing and their intensity. The answers were recorded in a questionnaire. The smell was stated as written in Table 1. The aroma expected was sweet and caramel with a hint of sour. It was found that the group FS2, A1S1, A2S2, and B2S1 have sweet and/or caramel aroma, while the others were described as having a hint of sour scent and/or green grassy and wet/earthy. A1S1 also has burned/scorched aroma, while B1S2 has a sour vinegary scent. One of the samples (A1S2) was not being analyzed in aroma and flavour due to its sensory properties, which were very sour and acidic. Therefore, it could not be tolerated as a good product.

The sour vinegary scent, and fermented scent, may appear due to the increase of organic acid content because of sugar decomposition during the evaporation process by using heat (Khalil *et al.*, 2018). Otherwise, green grassy and a wet or earthy aroma are generally found in the sorghum juice from the sorghum stalk FULL PAPER

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milling process. At the same time, the sweet and caramel scent was formed after the caramelization during the heating process (Ciappini *et al.*, 2013). Therefore, based on the data of aroma profile in Figure 5, the evaporation process using a higher temperature will lead to the formation of organic acid content, which brings a sour aroma to the sweet sorghum syrup.



Figure 5. Aroma profile of sweet sorghum syrup clarified by using 1 g/L CaOH2 (F), 0.5% activated carbon (A1), 2.5% activated carbon (A2), 3% bentonite (B1), or 5% bentonite (B2), then evaporated in  $70^{\circ}$ C (S1) or  $80^{\circ}$ C (S2).

#### 3.8.2 Flavour characteristic

The predominant flavour to be analyzed in sweet sorghum syrup was sweet, sour, and bitter, while salty and umami flavours were not considered (Ciappini *et al.*, 2013). Sixteen panels selected for the sensory analysis had trained to increase their knowledge of sorghum syrup sensory profile. From the analysis of variance, it was found that there were no differences between score levels given by the panels ( $\alpha$ =0.05), and they were consistent in their answer; hence the real test could be conducted.

The differentiation test was done by using the triangle test. A set consisting of two groups that had clarified using a similar type of flocculant or adsorbent with the same amount and evaporated in different heating temperatures was put together in a row of three. One of the samples was different from the other and analyzed using a triangle test by its differences in sweetness, sourness, and bitterness. If there were a minimum of nine panels stating the correct sample, which is different from the others, both groups were considered as having significant differences (Civille *et al.*, 2016).

It was found that thirteen panels that chose the correct answer, which means that there was a significant difference between groups presented in the same set. Panels reported that FS1 has a more acidic flavour but is less sweet if compared to FS2. Both had the same intensity of bitterness. Another set, which compared A1S1 and A1S2, brought a decision that A1S1 was

sweeter, less acidic, and less bitter than A1S2. Panels thought that A1S2 had a much lower intensity of sweetness but the higher intensity of sourness and bitterness than A2S2. The high intensity of sweetness in syrup clarified by using activated carbon was not in line with its reduction sugar content (Figure 5), whereas groups that had a higher content of reduction sugar tasted less sweet than the ones with lower content of reduction sugar. This might be happened because of their different viscosity profile, which can be seen in Figure 3b. Samples with higher viscosity have tended to be sweeter than others with lower viscosity and vice versa. Viscosity profile may affect sensory perception detected by the tongue during sensory analysis (Civile *et al.*, 2013).

B2S1 was informed to have a lower sweetness intensity if compared to B1S1. However, both of them have a similar intensity of sourness and bitterness. B1S1 tasted sweeter but less sour and less bitter than B1S2, while B1S2 tasted sourer than B2S2 with lower sweetness and equal bitterness intensity. B2S2 had a similar sweetness, sourness, and bitterness intensity to B2S2.

A level of sweetness that arose in sorghum syrup was generally in line with its sweet aroma. Panels reviewed that samples with higher sweetness intensity were FS1, A1S1, B1S1, and B2S2. However, the level of sweetness in sorghum syrup was usually lower than that of sugar cane syrup because it has a lower content of reducing sugar. In addition, bitterness and sourness may arise because of minerals such as potassium, sodium, and organic acid in sorghum syrup (Asikin et al., 2018; Appiah-Nkansah et al., 2016). Panels observed that there was no sample rated as having a higher score than "slightly sweet" (score: 4) in the level of sweetness. In addition. the evaporation process using high temperatures led to the increase of organic acid content, which promotes sourness (Bansal and Goyal, 2005).

#### 4. Conclusion

Based on the study results, it can be concluded that the different clarification methods brought different biochemical and sensory profiles of the sweet sorghum extract. Clarification using 5% bentonite and evaporation at 80°C gave the most preferred sorghum syrup character. The syrup has a reducing sugar content of 57.62%, pH 5.28, turbidity 35NTU, and 0.22 cm/s flowability, sweet taste, and caramel aroma. The utilization of clarified sorghum syrup in food products should be further analyzed.

# **Conflict of interest**

The authors declare no conflict of interest.

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