The Effect of Activated Charcoal Coffee Grounds (Coffea Sp.) as an Adsorbent on the Quality of the Liquid Sugar of Siwalan

Pengaruh Adsorben Arang Aktif Ampas Kopi (Coffea Sp.) Terhadap Kualitas Gula Cair Siwalan

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ABSTRACT

This study aimed to determine the adsorbent characteristics of coffee grounds activated charcoal (Coffea Sp.), optimum adsorption contact time, and differences in the quality of siwalan liquid sugar through adsorption and without adsorption. The characteristics of coffee grounds activated charcoal were determined by FTIR and SAA. FTIR characterization showed that activated coffee grounds charcoal contained functional groups of O-H, Csp2-H, Csp3-H, C-H2 methylene, C-O, and C=C-H. Characterization with SAA showed that the surface area of activated charcoal was 27.70 m2/g with a pore volume of 0.02 ml/g and an adsorption pore size of 1.63 nm. The optimum contact time of the adsorbent in the adsorption of impurities in the palm sap was 60 minutes. The siwalan liquid sugar, which went through adsorption, had a reducing sugar content of 6.70%, and the one without adsorption was 10.18%. While the ash content of siwalan liquid sugar through adsorption was 4.73%, and without adsorption was 6.5%. This value indicates that the quality of siwalan liquid sugar through adsorption is higher than that which does not go through the adsorption stage.

Keywords: adsorption, activated charcoal, siwalan liquid sugar

ABSTRAK

Telah dilakukan penelitian dengan judul pengaruh adsorben arang aktif ampas kopi (coffea sp.) terhadap kualitas gula cair siwalan yang bertujuan untuk mengetahui karakteristik adsorben arang aktif ampas kopi (Coffea Sp.), waktu kontak optimum adsorpsi dan perbedaan kualitas gula cair siwalan yang melalui adsorpsi dan tanpa adsorpsi. Penentuan karakteristik arang aktif ampas kopi menggunakan FTIR dan SAA. Hasil karakterisasi dilakukan menggunakan FTIR menunjukkan arang ampas kopi yang sudah diaktivasi mengandung gugus fungsi O-H, Csp2-H, Csp3-H, C-H2 metilen, C-O, dan C=C-H. Sedangkan karakterisasi menggunakan SAA menunjukkan luas permukaan arang aktif yang diperoleh sebesar 27,70 m2/g dengan volume pori sebesar 0,02 ml/g dan ukuran pori adsorpsi yang terbentuk sebesar 1,63 nm. Waktu kontak optimum adsorben dalam mengadsorpsi zat pengotor pada nira siwalan adalah 60 menit. Hasil penentuan kualitas gula cair siwalan yang meliputi kadar gula pereduksi yaitu 6,70 % kadar gula pereduksi yang melalui adsorpsi dan 10,18 % tanpa adsorpsi dan kadar abu yang melalui adsorpsi sebesar 4,73 % dan tanpa adsorpsi sebesar 6,5% yang menunjukan kualitas gula cair siwalan yang melalui adsorpsi lebih baik dari pada gula cair tanpa adsorpsi.

Kata Kunci: adsorpsi, arang aktif, gula cair siwalan

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1. INTRODUCTION
Sugar is a source of carbohydrates in the human body and is added to food or beverages. Indonesia's national sugar consumption continues to increase every year. This increase is due to the high projected growth of the food and beverages industry and changes in the quality of life, which is equivalent to the increase in population. The most widely traded sugar is solid sucrose crystals, used to sweeten foods or beverages. In Indonesia, besides cane sugar, there is also brown sugar (palm sugar) in both solid and liquid forms. Apart from being a sweetener, brown sugar contains mineral salts and is rich in nutrients beneficial for the body. One type of palm sugar that is widely used is siwalan sugar (Sahroel, 2009). According to Sahroel (2009), siwalan sap is obtained from tapping the male flowers of the siwalan tree. Siwalan sap is usually clear, slightly cloudy, and contains between 10-15% sugar. The potential production of siwalan sap in East Nusa Tenggara per tree per year with a tapping period of 184 days is 726.84 liters. When multiplied by the average number of trees of 1516,500 trees, the total production of siwalan sap is 110,498,256,000 per year (Journal of forest policy analysis, vol.7, no.1, 2010:27-45). One of the centers for siwalan sugar production is in the province of NTT.

The production of siwalan sugar in East Nusa Tenggara generally uses traditional methods resulting in a low-quality product. The production of siwalan sugar goes through several systematic stages. Filtering is one of the processes. However, this filtering process does not guarantee that the filtrate or siwalan sap is free from impurities, meaning that impurities pass through the filter, such as heavy metals or other dyes that cause a non-clear/cloudy sugar. Therefore, it is necessary to develop innovation in producing higher quality siwalan sugar. Several methods can be used to improve the quality of the sugar, including coagulation, filtration, electrododecolorization, and adsorption. According to Setyaningtias and Roy (2007), one of the most widely used methods for purification is adsorption. Adsorption using activated carbon is a potential way of separating a substance.

Coffee grounds are the final waste of the coffee brewing and are one of the raw materials for producing carbon. Coffee grounds contain carbon, nitrogen, lipophilic compounds, ethanol, lignin, alkaloids, polyphenolic compounds, tannins, polysaccharides, and chlorogenic acid (Pujol et al., 2013). The hydrocarbon content in coffee beans is relatively high (19.9%). This high hydrocarbon content can produce carbon when coffee beans are roasted or heated. Therefore, ground coffee that have been brewed can be used as activated charcoal.

Research on activated carbon from coffee grounds as an adsorbent has been carried out by Nasution et al. (2002), namely the use of coffee grounds activated charcoal as an adsorbent of iron and mercury ions in drinking water with an absorption yield of 90.34%. Baryatik et al. (2016) used coffee grounds activated charcoal as an adsorbent for chromium (Cr) metal in batik liquid waste with an absorption yield of 95%. Moelyaningrum et al. (2019) reported the utilization of coffee grounds activated charcoal as cadmium adsorbent in well water with an absorption yield of 95%. Asutik et al. (2015) used coffee grounds as an adsorbent to reduce BOD and COD in domestic wastewater with an absorption yield of 99%. Coffee grounds have not been widely used in the community, where coffee grounds are usually seen as waste, garbage, or compost for plants. Coffee grounds can also be used for air freshener/anti-odor, ink for painting, and coffee oil for grooming products. In addition, studies on the use of coffee grounds as adsorbents for dyes or impurities have not been carried out.
2. MATERIALS AND METHODS

2.1 Materials

The materials used were coffee grounds, siwalan sap, distilled water, Luff Schrool solution, 0.1M HCl, Na$_2$S$_2$O$_3$, 20% KI, 25% H$_2$SO$_4$, Al(OH)$_2$, and 5% starch indicator. The tools used were Surface Area Analyzer (SSA) to determine the surface area of coffee grounds activated charcoal, Fourier Transform Infrared Spectroscopy (FTIR) to determine the functional groups of coffee grounds activated charcoal, kiln to carbonize coffee grounds charcoal, magnetic stirrer, shaker, 60 mesh sieve, oven, funnel, Whatman filter paper no.40, analytical balance, pH meter, and various glassware.

2.2 Preparation of the adsorbent

This research begun with preparing the adsorbent by collecting brewed coffee grounds. The collected coffee grounds were dried in the oven. The dried coffee grounds were then carbonized in a kiln at 400ºC for 3 hours. The charcoal were ground and sieved through a 60 mesh sieve. The charcoal powder was chemically activated. A total of 50 grams of coffee grounds charcoal was mixed with 100 mL of 0.1 M HCl. The immersion results were filtered, washed with distilled water until the pH was neutral, filtered, and dried in an oven at 110ºC for 3 hours. The obtained charcoal was characterized using a Surface Area Analyzer (SAA) and Fourier Transform Infrared Spectroscopy (FTIR) characterization.

2.3 Determination of optimum adsorption contact time in reducing sugar content

Fresh siwalan sap that was tapped for 12 hours from the siwalan tree was put in a bottle and stored in a cool box containing ice cubes to prevent fermentation during transportation to the laboratory. A total of 10 grams of activated charcoal of coffee grounds and 500 mL of siwalan sap were put into an Erlenmeyer flask and stirred using a shaker with variations of 30, 60, and 90 minutes. The solution was filtered with filter paper, and the filtrate was cooked to obtain liquid palm sugar.

A total of 2 mL of siwalan sap was put into a 100 mL volumetric flask and added with 50 mL of distilled water. Al(OH)$_2$ was added until the effect was no longer there (no cloudy solution and other impurities were formed). Furthermore, distilled water was added to the mark and filtered. A total of 20 mL of the filtered filtrate was added with 20 mL of Luff Schrool solution in an Erlenmeyer. The blank solution was made by mixing 20 ml of Luff Schrool solution with 20 mL of distilled water. The sample and blank solutions were heated for 10 minutes while stirring using a magnetic stirrer. Then cooled and added with 10 mL of KI 20% and 25% H$_2$SO$_4$. The solution was then titrated with 0.1 N Na$_2$S$_2$O$_3$ solution using a 5% starch indicator.

2.4 Determination of optimum contact time against ash content of siwalan liquid sugar

A total of 2 grams of samples from each time variation and samples without adsorption were burned in a kiln at 550ºC to a constant weight, then cooled and weighed as ash content.

3. RESULTS AND DISCUSSION

3.1 Characteristics of coffee ground activated charcoal

a. Characteristics based on Fourier Transform Infrared Spectroscopy (FTIR) analysis

The results of the FTIR spectrum of the activated carbon of coffee grounds (Fig.1) showed an absorption with strong intensity at a wavelength of 3464.15 cm$^{-1}$, which was side by side with absorption at a wavelength of 3425.58 cm$^{-1}$ due to the absorption of the O-H group from phenol. The absorption at wavelengths of 3070.68 cm$^{-1}$ and 3001.24 cm$^{-1}$ was due to the absorption of the Csp$_2$-H group. The absorption at wavelengths of 2900.94 cm$^{-1}$ and 2862.36 cm$^{-1}$ was due to the absorption of the Csp3-H group. The absorption at a wavelength of 1465.90 cm$^{-1}$ was due to C-H$_2$ methylene groups. There
was also absorption at wavelength 1265.30 cm\(^{-1}\) and 1165.00 cm\(^{-1}\), which was side by side with absorption at wavelength 1080.14 cm\(^{-1}\) due to absorption of C-O groups from carboxyl. Furthermore, there was an absorption band at wavelengths of 925.83 cm\(^{-1}\), 856.39 cm\(^{-1}\), 748.38 cm\(^{-1}\), 694.37 cm\(^{-1}\), which was the absorption of aromatic C=C-H groups. FTIR analysis data of coffee grounds activated carbon presented in Table 1.

![Figure 1. FTIR spectrum of coffee grounds activated charcoal after activation](image)

**Table 1. FTIR analysis data of coffee grounds activated carbon**

| Wavelength cm\(^{-1}\) | Functional Group |
|------------------------|------------------|
| Literature             | Coffee Ground Activated Carbon | |
| 3473.3 (Sari,2017)     | 3464.15, 3425.58 | OH |
| 2924.09-2854.65 (Sari,2017) | 2900.94, 2862.36 | C-H |
| 1442.75-1381.03 (Sari,2017) | 1465.90 | C-H Bending |
| 1165.00-1118.71 (Sari,2017) | 1265.90, 1165, 1080.14 | C-O |
| 1000-675 (Sari,2017)    | 925.83, 856.39, 748.38, 694.37 | C=H Bending |

b. Characteristics Based on Surface Area Analyzer (SAA)

Table 2 shows that the surface area of the coffee grounds activated charcoal obtained was 27.70 m\(^2\)/g with a pore volume of 0.023 mL/g and an adsorption pore size of 1.63 nm. Based on these results, the pore size of coffee grounds activated charcoal is classified as microporous. It is based on the classification of pore size according to the International Union of Pure and Applied Chemistry (IUPAC), namely micropore size d<2 nm. The smaller the particle size of the adsorbent, the larger the surface area. The micropore type indicates that in adsorption, the adsorbate particles will stick around the adsorbent wall to form a strong bond (Sugesti, 2018).
3.2 Optimum adsorption contact time on reducing sugar content

According to Baharudin et al. (2007), the higher the reducing sugar content in brown sugar, the lower the quality of the sugar. And vice versa, the lower the reducing sugar content, the higher the quality. High levels of reducing sugar might be caused by contaminated raw materials (sap) with microbes during tapping and production. The maximum quality standard for reducing sugar according to SNI is 6%. The reducing sugar level in palm sugar liquid using the Luff Schoorl method are presented in Table 3.

Graph of reducing sugar concentration from siwalan liquid sugar presented in Fig. 2.

Table 2. SAA karakter characterization data

| Surface Area (m²/g) | Pore Volume (cc/g) | Pore Size (nm) |
|---------------------|-------------------|----------------|
| 27.70               | 0.023             | 1.63           |

Table 3. Data analysis of reducing sugars using the Luff Schoorl method

| No | Siwalan liquid sugar | Volume Na₂S₂O₃ 01 N (mL) | Reducing Sugar (%) |
|----|----------------------|--------------------------|-------------------|
|    |                      | Sample | Blanko |                   |
| 1  | Contact time 30 minutes | 19.3  | 31     | 7.33              |
| 2  | Contact time 60 minutes | 20.3  | 31     | 6.70              |
| 3  | Contact time 90 minutes | 19.7  | 31     | 7.008             |
| 4  | Without adsorption   | 13.2   | 29     | 10.18             |

Figure 2. Graph of reducing sugar concentration from siwalan liquid sugar with variations in contact time
The titration of Luff Schoorl's solution on siwalan liquid sugar through adsorption with variations in contact time and without adsorption showed that the four samples contained reducing sugars. There were differences in reducing sugar content in the samples that went through the adsorption process and those that did not. The liquid sugar of siwalan through adsorption had reducing sugar content which decreases with the increasing of contact time. At the contact time of 30 minutes, the reducing sugar content was higher than other contact times, 7.33%. It is caused by the presence of empty spaces on the carbon that has not been occupied by impurities that affect the increase in reducing sugar in the siwalan liquid sugar.

At the contact time of 60 minutes, there was an increase in adsorption, indicated by a decrease in the reducing sugar content, which was 6.70%. It indicates that the empty spaces in the activated charcoal of coffee grounds have been filled with impurities of the liquid sugar of siwalan. Meanwhile, at the contact time of 90 minutes, the absorption of activated charcoal from coffee grounds decreased, as indicated by an increase in reducing sugar content, which was 7.008%. It happens because the prolonged contact time between the adsorbent and the adsorbate resulting in a reduce adsorption rate. Longer contact time can also result in desorption, namely the release of impurities that the adsorbent has bound. It is in accordance with Bernard et al. (2013), who states that after the adsorption reaches an equilibrium state at the optimum contact time, the addition of the contact time between the adsorbent and the adsorbate will not have a significant effect on the adsorption process.

Siwalan liquid sugar without adsorption has a higher reducing sugar content than siwalan liquid sugar that undergo adsorption. It proves that adsorption can improve the quality of palm sugar liquid by decreasing the reducing sugar content by 3.48%. The adsorption mechanism is that the activated charcoal of the coffee grounds releases water molecules from the surface cavity and then interacts with the molecules to be adsorbed from the siwalan sap in the form of unwanted impurities. Impurities in siwalan sap might come from the air, the bamboo container for tapping, or other contaminants during the tapping process. These impurities can stimulate the growth of the yeast *Saccharomyces cerevisiae* and bacteria of the acetobacter genus (Mulyawanti et al., 2011). According to Marsigit (2005), *Saccharomyces cerevisiae* can hydrolyze sucrose to produce reducing sugars (fructose and glucose).

### Table 4. Ash content

| Adsorption contact time          | Sample weight (gram) | Cup weight (gram) | Weight of cup + ash (gram) | % Ash content |
|---------------------------------|----------------------|------------------|---------------------------|--------------|
| Contact time 30 minutes         | 2                    | 13.3             | 13.397                    | 4.85         |
| Contact time 60 minutes         | 2                    | 13.3             | 13.395                    | 4.73         |
| Contact time 90 minutes         | 2                    | 13.3             | 13.396                    | 4.8          |
| Without adsorption              | 2                    | 13.3             | 14.6                      | 6.5          |

![Figure 3. Graph of ash content of siwalan liquid sugar](image)

*The Effect Activated Charcoal Coffee...*
3.3 Effect of optimum adsorption contact time on ash content

The ash content of siwalan liquid sugar through the adsorption process and without adsorption is presented in Fig. 3. Analysis of the ash content of the siwalan liquid sugar through adsorption with variations in contact time and without adsorption (Table 4) showed that the four samples had different ash content. Samples that went through the adsorption process showed that as the adsorption contact time increased, the ash content decreased until the adsorption reached a state of equilibrium. At the 30 minutes contact time, the adsorption was still low at 4.85%. At the 60 minutes contact time, there was an increase in absorption, which was indicated by a decrease in the ash content of the liquid sugar of palm sugar, which was 4.73%. Meanwhile, at the 90 minutes contact time, the absorption of activated charcoal from coffee grounds decreased, which was indicated by an increase in the ash content of the siwalan liquid sugar, which was 4.8%. It proves that adsorption can reduce the ash content in the siwalan liquid sugar by 1.7%.

The decrease in ash content occurred due to decreasing of reducing sugar in the liquid sugar due to adsorption. The increase in reducing sugar can increase the ash content in liquid sugar, which results in a decrease in sugar quality. The mechanism of adsorption of activated charcoal carbon from coffee grounds releases water molecules from the surface cavity. It effectively interacts with organic acid molecules that will be adsorbed from the siwalan sap. Organic acids are formed due to the hydrolysis of sucrose into reducing sugars (Waluyo, 2005). Organic acids significantly affect the increase in ash content during the cooking of sap so that the quality of the sugar produced decreases.

4. CONCLUSIONS

On the basis of the study's findings, it can be concluded that:

1. FTIR analysis showed that the charcoal after activation contained functional groups O-H, Csp2-H, Csp3-H, C-H2 methylene, C-O, C=C-H. Analysis of activated charcoal with SAA showed that the surface area obtained was 27.70 m²/g with a pore volume of 0.02 mL/g, and the adsorption pore size formed was 1.63 nm.
2. The optimum condition of the adsorbent to adsorb impurities in the water cave occurs at a contact time of 60 minutes.
3. Siwalan liquid sugar that went through the adsorption process had a higher quality than without adsorption. It can be seen from the smaller content of reducing sugar and ash content than siwalan liquid sugar without adsorption.
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