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Utilization of Spent Arabica Coffee Grounds as Raw Material for Activated Charcoal in Liquid Bath Soap Formulation

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ABSTRACT

Arabica coffee (*Coffea arabica*) is one of the most popular coffees among Acehnese, an ethnic group from Aceh, Indonesia. The amount of coffee consumed is directly proportional to the amount of coffee waste produced. Spent coffee grounds are the residue obtained during the brewing process. Spent coffee grounds can be utilized by converting them into active charcoal adsorbents. This study aimed to produce activated charcoal from spent arabica coffee grounds (SACG), to utilize it as an active ingredient in the liquid bath soap formulations, and to determine the best formula. The characterization of activated charcoal was conducted and compared to the Indonesian National Standard (SNI). The liquid bath soap contains activated charcoal from SACG was divided into five formulas with various concentrations of cocamidopropyl betaine (CAPB) and sodium lauryl sulphate (SLS) surfactants. Furthermore, the evaluation of liquid bath soap formula was performed. The characterization results showed that activated charcoal produced from SACG met the SNI requirements with a yield of 86.3%, volatile substance content of 10.67%, water content of 5.67%, ash content of 1.7%, pure carbon content of 81.96%, and iodium absorption of 1522.8 mg/g. The evaluation results revealed that liquid bath soap formula with activated charcoal from SACG containing a combination of 5% CAPB and 5% SLS (F5) was the best formula that met the requirements. SACG can be used as an excellent raw material for activated charcoal in liquid bath soap formulations.

Keywords: *arabica coffee, activated charcoal, cocamidopropyl betaine, liquid bath soap*

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INTRODUCTION

Indonesia, the world's fourth largest coffee producer after Brazil, Vietnam, and Colombia (FAOSTAT, 2018), produced 685.8 thousand tons of coffee in 2018, with arabica coffee (*Coffea arabica*) accounting for 116.6 thousand tons of that total (BPS-Statistics Indonesia, 2019). Aceh is Indonesia's leading producer of arabica coffee, with 59,489 tons produced on an area of 100,590 acres in 2018 (BPS-Statistics Indonesia, 2019). Coffee is a popular beverage and the world's second-most traded commodity (ICO, 2019; Shahbandhen, 2020; Czarniecka-Skubina et al., 2021), which will increase the amount of spent coffee grounds.

Spent coffee grounds are typically solely viewed as common solid waste that is thrown away and hardly used (Franca & Oliveira, 2019). The spent coffee grounds can be used to produce activated charcoal, which is a material that can be used as an absorbent or adsorbent due to its total carbon content being relatively high (47.8 to 58.9%). Activated charcoal typically absorbs a large amount of chemicals and poisons because of its effective adsorbent properties. Additionally, activated charcoal has been frequently used as an additional component in items like soaps, scrubs, shampoos, and masks that keep skin and hair clean and smooth (Lempang, 2014).

Activated charcoal created from spent arabica coffee grounds (SACG) absorbs substances better than charcoal made from spent robusta coffee grounds because the pores of the former have a denser texture (Suhariyanto et al., 2020). Based on its ability to absorb substances, activated charcoal has the potential to be further developed as an ingredient in skin-cleansing preparations like liquid bath soap. This study aimed to recycle the SACG by converting them into activated charcoal. Activated charcoal was then formulated as a functional ingredient in liquid bath soap formulations.

METHODS

Materials

Spent arabica coffee grounds (SACG) was collected from several coffee shops around the city of Banda Aceh, Aceh province, Indonesia. Hydrogen chloride (HCl), potassium hydroxide (KOH), butyl hydroxide toluene (BHT), sodium thiosulphate, and iodine were obtained from Merck® (Germany). Sodium carboxymethyl cellulose (Na CMC) was obtained from Sigma® (USA), cocamidopropyl betaine (CAPB) was obtained from Evachem® (Malaysia), sodium lauryl sulphate (SLS) was obtained from Qualigens® (India). Starch, stearic acid, glycerin, fresh mint fragrance, and distilled water were used. The glassware, obtained from Pyrex® (England,

UK), were: burette, beaker glass, volumetric glass, glass funnel. A hotplate magnetic stirrer was purchased from Thermo Scientific® (USA), and an analytical balance was purchased from Matrix® (Canada). Muffle furnace, oven, desiccator, filter paper, masks and gloves, coffee grounds containers, stirring rod, thermometer, pH meter, porcelain dish, petri dish, pestle and mortar were used.

Production of Activated Charcoal

The SACG (40 g) was dried in the sunlight. Then, SACG was burned in a furnace at a temperature of 450°C for approximately 45 minutes. Afterward, the charcoal was allowed to stand for a while to cool, and then stored in a desiccator. Furthermore, the charcoal was immersed in a HCl solution (0.1 M) for 48 hours to perform the charcoal activation procedure chemically. Finally, the charcoal was drained and washed with water before drying in the oven at 100°C for 4 hours (Huda et al., 2015).

Characterization of Activated Charcoal

The characterization of the activated charcoal was carried out by calculating the yield, volatile matter content, water content, ash content, pure activated carbon content and iodine absorption (Huda et al., 2015).

Yield value

The yield of activated charcoal was measured by comparing the weight of the raw material before carbonization with the weight of the activated charcoal after carbonization (BSN, 1995).

Volatile levels

Activated charcoal (1 g) was heated for 15 minutes in a furnace at 950°C, then cooled in a desiccator and weighed. The levels of volatile activated carbon were calculated as follows (BSN, 1995).

$$\textbf{Volatile Levels} = \frac{\mathbf{C-D}}{\mathbf{C}} \times \mathbf{100\%}$$

C = Initial weight of activated charcoal before heating (g)
D = Final weight of activated charcoal after heating (g)

Water Content

In a previously weighed porcelain crucible, 1 g of activated charcoal was placed. After that, it was transferred to an oven (105°C) for 3 hours, then cooled in a desiccator and weighed. Then, the water content was calculated as follows (BSN, 1995).

$$\textbf{Water Content} = \frac{\mathbf{E}}{\mathbf{F}} \times \mathbf{100\%}$$

E = Initial weight of activated charcoal before heating (g)
F = Final weight of activated charcoal after heating (g)

Ash content

One gram of activated charcoal was added to a porcelain crucible that had previously been weighed. It was then moved to a furnace (600°C) for 3 hours, cooled in a desiccator, and weighed (BSN, 1995).

$$\textbf{Ash Content} = \frac{\mathbf{G}}{\mathbf{H}} \times \mathbf{100\%}$$

E = Initial weight of activated charcoal before heating (g)
F = Final weight of activated charcoal after heating (g)

Pure activated carbon content

The pure activated carbon content was calculated by subtracting the total percentage with the percentages of activated charcoal's volatile matter, moisture, and ash content (BSN, 1995).

$$\textit{Pure Activated Carbon Content} = \mathbf{100\ \% - I - J - K}$$

I = Volatile content (%)
J = Water content (%)
K = Ash content (%)

Iodine absorption

Iodine absorption was measured by weighing activated carbon (0.5 g) and placing it in an Erlenmeyer. The solution was shaken for 15 minutes before being filtered. The filtrate was diluted to 10 mL in an Erlenmeyer using a dropper pipette. After that, the filtrate was titrated with a 0.1 N sodium thiosulfate solution (a starch indicator was added if the yellow color of the solution was almost sallow) and the iodine absorption was calculated (BSN, 1995).

$$\textbf{Iodine Absorption} = \frac{(10 - \frac{N \times V}{0,1})}{S} \times \mathbf{126,9}$$

N = Normality of sodium thiosulfate solution
V = Required sodium thiosulfate solution (mL)
S = Charcoal weight (g)

Formulation of Liquid Bath Soap

The liquid bath soap with activated charcoal from SACG was formulated with various concentrations of CAPB and SLS as a surfactant and was divided into 5 formulas (as shown in Table 1) in order to obtain a good liquid soap with stable and abundant foam.

Evaluation of Liquid Bath Soap Formulation

An evaluation of the liquid bath soap formulation was performed to determine whether the liquid soap formulation met the requirements of a good formulation. An analysis consisting of the tests of organoleptic, pH, viscosity, specific gravity, high foam stability, and irritation were part of the evaluation (DSN, 1996).

Table 1. Formulation of liquid bath soap with activated charcoal from SACG

Ingredients	Concentration (%)				
	F1	F2	F3	F4	F5
Active charcoal	2	2	2	2	2
Olive oil	25	25	25	25	25
Potassium hydroxide (KOH)	15	15	15	15	15
Sodium carboxymethil cellulose (CMC Na)	1	1	1	1	1
Stearic acid	0.25	0.25	0.25	0.25	0.25
Sodium lauryl sulphate (SLS)	5	10	0	0	5
Cocamidopropyl betaine (CAPB)	0	0	5	10	5
Butylated hydroxytoluene (BHT)	0.01	0.01	0.01	0.01	0.01
Glycerin	15	15	15	15	15
Methyl paraben	0.3	0.3	0.3	0.3	0.3
Propyl paraben	0.6	0.6	0.6	0.6	0.6
Fresh mint	qs	qs	qs	qs	qs
Aquadest	Ad 100	Ad 100	Ad 100	Ad 100	Ad 100

Organoleptic Test

The scent, color, and texture of the liquid bath soap were observed during organoleptic testing (Prayadnya et al., 2017).

pH value

The test was performed with a pH meter. The formulation of liquid soap (1 g) was diluted with distilled water to 10 mL. Then, the pH meter electrode was dipped into the solution, and the pH value was recorded (Laksana et al., 2017).

Viscosity test

A viscosity test was performed on a Lamy Rheology viscometer type CP-4000 with spindles no. 20 and 40 at 300 rpm for 30 seconds. Then, the results were recorded.

Specific gravity test

The current experiment involved weighing an empty pycnometer before filling it with distilled water and reweighing it. Following that, the liquid soap formulation was put to the same pycnometer and weighed. The density of the formulation was calculated (Sari & Ferdinan, 2017).

$$\textbf{Specific Gravity Test} = \frac{\textbf{sample weight}}{\textbf{Distilled water weight}}$$

Foam stability test

The sample was weighed (1 g) and placed in a volumetric glass for the current test. Then, 10 mL of distilled water was added and shaken by turning the volumetric glass upside down; the foam height was immediately measured. The foam height was remeasured after 5 minutes (Sari & Ferdinan, 2017).

$$\textbf{Foam Stability} = \frac{\textbf{Final Foam height}}{\textbf{Initial Foam Height}} \times \textbf{100\%}$$

Irritation test

The test was performed on 30 respondents using an open patch test. The formulation was applied to the inner forearm at the attachment site (2.5 × 2.5 cm), then left open for 48 hours and observed. The present test was performed twice a day (morning and evening) for three days in a row. In addition, the respondents would complete a questionnaire based on the presence or absence of an irritation reaction. A positive irritation reaction was indicated by the presence of redness, itching, or swelling on the skin of the forearm being treated (Untari & Robianto, 2018).

Hedonic test

Thirty respondents were subjected to a hedonic or predilection test. As parameters, we used form, color, scent, and foam height. Respondents would complete a questionnaire with a numerical scale rating of 1–3, with dislike represented by one, like by two, and adore represented by three (Laksana et al., 2017).

Stability test

The sample was laid in a container and transferred to an oven (45±2°C) for 24 hours before being displaced to a freezer at 5±2°C for 24 hours (one cycle). The test was repeated six times, or for 12 days. Furthermore, organoleptic was evaluated and analysis of pH, syneresis, and viscosity were performed (National Health Surveillance Agency, 2005).

Figure 1. Activated charcoal from SACG

Table 2. Characterization of activated charcoal from SACG

No	Characteristics	Results	Indonesian National Standard (SNI)
1	Yield	86.3%	–
2	Volatile substance level	10.67%	Max. 25%
3	Water content	5.66%	Max. 15%
4	Ash content	1.7%	Max. 10%
5	Pure activated carbon content	81.96%	Min. 65%
6	Iodine absorption	1522.8 mg/g	Min. 650 mg/g

RESULTS AND DISCUSSION

Production and Characterization of Activated Charcoal from SACG

The activated charcoal was produced by dehydrating, carbonizing, and activating spent arabica coffee grounds (SACG). Firstly, the SACG was dehydrated or dried to remove water molecules. The dehydration process can aid the burning of raw materials into charcoal. The following procedure, carbonization, involves burning raw materials at high temperatures to convert SACG into charcoal. Afterwards, activating carbonized-charcoal with HCl as an activating agent was to open the pores of the activated charcoal and attracts impurity particles that still clog the surface of the charcoal pores, such as hydrocarbon compounds, nitrogen, tar, and others. Thus, these contributed to causing the charcoal pores to become more open and the absorption higher.

Because of the corrosive nature of HCl, the remaining HCl solution in activated charcoal should be removed by washing with distilled water to a neutral pH of 7, making activated charcoal safer to use as an active ingredient in pharmaceutical preparations. A re-dehydration process removes the remaining water molecules left over from the washing process, resulting in activated charcoal that is free of water molecules, as shown in Figure 1, and can be used as an active ingredient.

The results of activated charcoal production from SACG were an odorless black fine powder. As shown in Table 2, the characterization of activated charcoal was performed by an analysis of the volatile matter, water content, ash content, iodine absorption, and activated carbon purity, then was compared to the SNI or Indonesian National Standard 06-3730-1995 for technical activated charcoal (BSN, 1995).

The characterization results exhibited that all parameters met the standard values. The greater the yield value, the greater the activated charcoal value. The yield of activated charcoal is highly dependent on raw materials and activation treatment factors, such as carbonization, as well as temperature and time of activation (Lempang, 2014). High levels of volatile substances indicate the number of carbon pores that are still covered by a substance, which also reduces the absorption of activated carbon. The appropriate carbonization temperature and time will also affect the volatile matter content. The levels of volatile substances will be lower at high temperatures, and the activation time will be longer because the levels of volatile substances will no longer stick to the pores, leaving the carbon pores free of volatile molecules (Kusdarini et al., 2017).

The lower water content obtained in activated charcoal revealed that the activated charcoal produced had a higher

Figure 2. Liquid bath soap with activated charcoal from SACG

Table 3. Evaluation of liquid bath soap with activated charcoal from SACG

Parameter	Formula					
	F1	F2	F3	F4	F5	Fc
Form	Lq	Lq	Lq	Lq	Lq	Lq
Odor	FM	FM	FM	FM	FM	Pf
Colour	DG	DG	DG	DG	DG	G
pH	8.01	8.02	8.3	8.01	8.02	9.02
Viscosity (cPS)	1025.4	1181.2	1342.3	1345	1370.7	870.0
Specific gravity	1.039	1.041	1.042	1.044	1.049	1.031
Foam height	66.7%	66.7%	66.7%	85.7%	93.3%	75%
Irritation	–	–	–	–	–	–

F1: SLS of 5%, F2: SLS of 10%, F3: CAPB of 5%, F4: CAPB of 10%, F5: combination of 5% CAPB and 5% SLS, Fc: commercial bath soap product, Lq: liquid, FM: fresh mint, Pf: perfume, DG: dark grey, G: grey

quality. This also indicates that fewer carbon pores are covered by water molecules (Irmanto & Suyata, 2010). The high ash content of activated charcoal reduces the quality and absorption of activated carbon, and this condition also indicates that the activated carbon pores are still mostly covered by inorganic substances and metal oxides. The lower ash content obtained in activated charcoal showed that the activated charcoal produced had a higher quality (Mopoung et al., 2015).

The size of the pure activated carbon content produced was highly dependent on the values of volatile substances, water content, and ash content (Sahara et al., 2017). The surface area of activated carbon correlates with its iodine adsorption capability. The greater iodine absorption value revealed that the activated carbon had a greater surface area. This also implies that the activated charcoal produced had a better ability to absorb impurity particles, and it had a higher quality (Rasdiansyah et al., 2014). Activated carbon plays a vital role in pharmaceuticals and chemicals because it removes impurities, which aids in quality control (eliminating toxic chemicals) (Soonmin et al., 2022). The porous nature of activated charcoal is advantageous because of its negative electrical charge. It attracts positively charged gases and toxins. These molecules were then trapped in activated charcoal, preventing the adsorption of poisons into the bloodstream (Ge et al., 2017). Many advances have been made in researching the beneficial effects of carbon over time (Cukierman, 2013; Bomfim et al., 2022).

Formulation and Evaluation of Liquid Bath Soap with Activated Charcoal from SACG

The results of liquid bath soap formulation with activated charcoal from SACG exhibited that the liquid bath soap colour was gray to dark gray (as shown in Figure 2).

In addition, an evaluation of a liquid bath soap formula with activated charcoal and various concentrations of surfactants was shown in Table 3. The commercial bath soap product was used as a comparison.

The evaluation results of liquid bath soap formulations showed that all parameters met the requirements. The pH value met the SNI requirements for a good formulation, namely at a pH range of 8–11. The pH value has a significant impact on the absorption of formulations into

the skin (BSN, 1996). A too low pH value would irritate the skin, while a too high pH value would affect skin humidity (the skin becomes arid). As a consequence, pH balance has become mandatory and must meet established standards for good formulation.

The commercial bath soap product (Fc) had a lower viscosity compared to the other five soap formulas. However, the viscosity values of these five formulations were within the range of viscosity values for liquid bath soap formulations. The viscosity of liquid bath soaps on the market ranges between 400 and 4,000 cPs (Williams & Schmitt, 2002).

The foam height test results exhibited that increasing the CAPB concentration would make the produced foam become more stable. The formulation containing the surfactant combination of CAPB and SLS (F5) demonstrated up to 93.3% foam stability. This condition was due to the fact that the CAPB was compatible with other surfactants, such as SLS. CAPB was composed of anionic (negatively charged) surfactants and cationic (positively charged) amphoteric surfactants. As a consequence, when incorporating CAPB with SLS (which also has an anionic group) in a liquid soap formula, these surfactant combinations would perform synergistically to produce abundant and stable foam. Customers believe that soap with a little foam will leave a slippery impression on their skin, making them feel unclean. As a result, foam is a critical parameter in soap preparation (Tanjung et al., 2020).

Furthermore, the irritation test on 30 panelists revealed that the five formulations (F1, F2, F3, F4, and F5) did not inflict any reaction of redness, itching, or swelling on the skin. According to a previous study (Kabra et al., 2018), activated charcoal plays an essential role in cases of skin-related problems. It shields the skin from potentially harmful chemicals, dust, bacteria, toxins, dirt, and microparticles. Essentially, it acts as a protector for the skin. In general, activated charcoal was suitable for the majority of skin types and could thus be used to treat acne, to relieve itching, to heal damaged skin, and to minimize pores. Charcoal has the ability to absorb dirt and oil from the pore surface, making it cleaner and less oily (Soonmin et al., 2022). This circumstance indicates that there was no risk of skin irritation and the formulation was safe to apply.

CONCLUSION

The formula containing the surfactant combination of 5% SLS and 5% CAPB (F5) had a better evaluation outcome for the formulation of liquid bath soap with activated charcoal from spent arabica coffee grounds (SACG). This finding might emphasize the potency of SACG as a raw material for activated charcoal in liquid bath soap formulas.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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