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Effect of Roasting Type on the Physicochemical Characteristics of Natural Coffee Soap Produced by Cold Process Technology.

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Abstract. This study investigated the physicochemical characteristics of natural coffee soap produced using the cold process method with different coffee roasting types—light, medium, and dark. The analysis focused on moisture content, pH, free fatty acids, caffeine, and free alkali levels. Results showed that moisture content ranged from 13.47% to 15.16%, pH from 10.16 to 10.67, free fatty acids from 0.79% to 1.02%, caffeine from 0.16% to 0.20%, and free alkali from 0.30% to 0.43%. Statistical analysis revealed that roasting type significantly affected moisture content, pH, free fatty acids, and caffeine levels but not free alkali content. Dark roast soap exhibited the lowest moisture content and the highest pH, indicating a firmer and more stable texture, while medium roast soap had the highest caffeine concentration. The decrease in free fatty acids with increasing roast intensity suggests enhanced lipid degradation during roasting. These findings indicate that roasting type plays a crucial role in determining the physicochemical quality of coffee-based soap. Dark-roasted coffee produced a more balanced formulation, though further optimization is needed to minimize residual alkali for safe cosmetic application.

Keywords: *Coffee soap; roasting type; cold process; physicochemical characteristics*

1. Introduction

Coffee is one of natural ingredients to make health soap. Robusta coffee beans are known to contain alkaloids, tannins, saponins, and polyphenols [1]. Coffee also provides many health benefits because coffee beans contain various chemical compounds such as carbohydrates, proteins, minerals, caffeine, chlorogenic acid, fats, glycosides, and volatile components [2]. One of the main benefits of coffee for the skin is its ability to act as a natural exfoliant. Fine coffee granules can be used as a scrub to remove dead skin cells and make skin smoother. Caffeine is an alkaloid chemical compound that is beneficial for skin health, such as repairing damaged skin, neutralizing irritated skin, providing nutrition to the skin, as well as eliminating body odor [3]. Chlorogenic acid most abundant phenolic compound in coffee after caffeine which has antioxidant, anti-cancer, neuro-protective,anti-inflammatory, anti-viral properties [4]. The antioxidant activity of coffee is mainly attributed to phenolic compounds due to their ability to scavenge free radicals in the biological system [5].

There are several levels of coffee roasting, light, medium, and dark. The physical, chemical, and taste properties of coffee are influenced by temperature and roasting time [6]. Roasting conditions affect several physicochemical characteristics of coffee beans, such as color, browning index,caffeine, phenolic acids, and antioxidant capacity [7]. Roasting activities are related to temperature and roasting time, so the types of roasting levels also vary. Based on research conducted by [8] stated that temperature treatment and roasting time significantly affect the yield of roasted Arabica coffee beans and the acidity of the brew. [9] showed that there is an effect of temperature and roasting time on Robusta coffee beans on the moisture content of coffee beans before roasting is 12%. Roasting at a temperature of 200˚C for 10 minutes produces well-roasted coffee beans. The texture of coffee beans during roasting tends to be more brittle as seen from the fracture stress value. The relationship between the effect of temperature and roasting time on changes in the density of Robusta coffee beans greatly influences the physical changes that occur in coffee beans[10].

The methods commonly used to make solid soap are the cold process and the hot process. The fundamental difference between these two methods lies in the temperature used in the soap-making process [11]. The quality of soap can be influenced by several factors, such as The stirring rotation speed, the volume of distilled water, and the mass of salt will cause different soap textures [12]. The reaction rate, operating temperature, and reaction time affect the saponification value, moisture content, free alkali content, and pH [13]. The different Moisture content of each soap can be influenced by the method used in the soap making process. Each soap manufacturer adds water differently depending on each manufacturer's soap formulation [14]. The formulation and use of ingredients can also affect the quality of soap, such as organoleptic quality, moisture content, and pH [15].Thus, the aim of this research is to analyze the physicochemical characteristics of natural coffee soap produced using the cold process method with different coffee roasting types.

2. Research Methods

2.1 Tools and materials

This research was conducted at the Laboratory of Pat Petulai University, mathematics and natural science laboratory Universty of Bengkulu and the Laboratory of the Faculty of Agricultural Technology, Gadjah Mada University. The tools used in this research include digital scales, rubber gloves, masks, faces hiled, measuring cup,hand mixer, spatula, spoon, basin, measuring cup, and cloth cover. The ingredients used are Robusta ground coffee with (roasting type light, medium, and dark), olive oil, coconut oil, palm oil, NaOH, and distilled water.

2.2 Sample preparation

The sample used was solid soap with the addition of Robusta ground coffee with a particle size of 80 mesh. This research used a Completely Randomized Design (CRD) with one treatment factor, namely roasting type (light, medium, and dark), each treatment was repeated 3 times (triple).
F1: Roasting Type Light
F2: Roasting Type Medium
F3: Roasting Type Dark

Sample preparation consists of two stages, namely coffee roasting and making soap. Before the roasting process, the roasting machine is first prepared and heated to a tube temperature of 150 °C, then 1 kg of coffee beans (green beans) are weighed. The green beans that have been weighed are put into the roasting machine, and the green beans are roasted.

Roasting produces a light type for 15 minutes, a medium type for 20 minutes, and a dark type for 25 minutes. Coffee roasting is repeated three times for each type of roast.

Making natural coffee soap is done using the cold method. Preparation of tools and materials in the form of 300 g olive oil, 300 g coconut oil, and 300 g palm oil. At the same time, the NaOH solution is made up of as much as 131 grams of NaOH with 342 grams of distilled water until the solution temperature reaches 30 °C± 5 °C. After that, the oil and NaOH solution are mixed and stirred using a hand blender for 3 minutes until the mixture becomes slightly thick. Then add 20 grams of coffee powder and stir for 2 minutes. Put the soap dough into the mold and let it rest for 24 hours until the soap becomes solid. Soap that is already solid is not tested immediately but is waited for 8 weeks for the curing process while being aired in a place with air circulation. So that the saponification process is completed perfectly, the water content is reduced, and the soap becomes harder and has a pH that is safer to use.

2.3 Analysis of Physicochemical Characteristics Natural Coffee Soap

The physicochemical characteristics analyzed consisted of: Moisture content, pH, free fatty acids, free alkali and caffeine. Moisture content, oven test methods AOAC 934.01, Oven the weighing bottle for 24 hours (daily) before use and determine the weight. Weigh the sample in powder form or ground material of 1-2 grams in a clean weighing bottle and know the weight. Dry in the oven at 100–105 °C for 3-5 hours, depending on the material. The greater the moisture content, the longer the oven process will take. Heat again in the oven for 30 minutes, cool in a desiccator, and weigh. Repeat this treatment until a constant weight is achieved. Weight reduction is the amount of water in the material.

pH, Standardization (calibration) of pH meters: Turn on the pH meter and let it stabilize. Rinse the electrode with buffer solution or distilled water, dry it with a tissue, and just pat it; don't rub or press it. Dip the electrode in buffer solution and set the pH measurement. Allow the electrode for a few moments to equilibrate with the buffer solution so that a stable reading is obtained. Adjust the pH meter standardization regulator (calibration button) until a pH value is obtained that corresponds to the pH of the buffer at the measured temperature. For routine standardization, the pH meter is calibrated with two types of buffers, namely pH 4 and pH 7. pH setting, If the sample is in the form of a homogeneous solution that is not too concentrated, then the pH can be determined directly; if it is concentrated, it is necessary to dilute it (each sample must be the same) and then measure the pH. If the sample is a soluble solid, it needs to be dissolved with distilled water (pay attention to the ratio between the weight of the sample and the water added). If the sample is flour (dry and porous), it needs to be extracted first. After sample preparation is complete, turn on the calibrated pH meter. First, rinse the electrode with distilled water and dry it with a tissue. After that, dip the electrode in the sample solution and record the pH of the sample.

Standardization of a 0.05 N NaOH solution Weighing ±400 mg of potassium biphthalate, which had previously been pollinated and dried at 28°C for 2 hours, was put into a 250 mL Erlenmeyer flask. Add 75 mL of distilled water. Add 2 drops of phenolphthalein (PP) indicator. Titrate with NaOH until the color changes to pink without changing again. Determination of free fatty acid levels Weigh a 14-gram sample of cooking oil in a 250-mL Erlenmeyer flask. Add 25 mL of 95% ethanol. Add 2 mL of phenolphthalein (PP) indicator. Titrate with 0.05 NaOH until a pink solution forms and does not disappear for 30 seconds. Record the volume of NaOH used to calculate the free fatty acid content in the sample.

Independent alkali testing: carefully weigh 0.1 gram of sample, then dilute in a 100-ml volumetric flask. Transfer the solution into a 250-ml Erlenmeyer flask, then add 3 drops of PP indicator. If a pink color occurs, then titrate with H2SO4 0.02 N until the pink color disappears. Note the use of H2SO4 0.02 N, and then calculate the alkalinity level.

Caffeine Analysis Procedure Iodometric Back Titration Method, Methods AOAC 960.25

Way of work: Weigh the sample that has been finely ground and passed a 30 mesh sieve as much as 5 grams and put it in an Erlenmeyer flask. Add 5 grams of MgO and 200 ml of distilled water. Cover with refrigerant, then simmer slowly for 2 hours. Once cool, dilute with distilled water in a measuring flask so that it is exactly 500 ml, then filter. Take 300 ml of the filtrate, put it in a boiling flask, add 10 ml of H2SO4 (1:9), and then boil until the liquid volume remains approximately 100 ml. Put the liquid into the separating funnel. The godok flask was rinsed with a little sulfuric acid (1:99) and shaken several times with chloroform successively using 25 ml, 20 ml, 15 ml, 10 ml, 10 ml, and 10 ml. The rinsing fluid is put into the separating funnel. In a separating funnel, add 5 ml of 1% KOH, then shake and leave for a while until the liquid separates clearly. The bottom liquid is a solution of caffeine in chloroform, removed and collected in an Erlenmeyer flask. In a separating funnel, add another 10 ml of chloroform, shake, and leave until the liquid is clearly separated. Then, the bottom liquid is removed and collected in the same Erlenmeyer flask as in the previous stage. This treatment is repeated once again. The caffeine solution in chloroform is then heated in a water bath until the residue remains, then dried in a 100°C oven until a constant weight is obtained, which is the weight of crude caffeine. Pure caffeine levels can be determined by determining the N content of microkjeldahl or by other means.

2.4 Data analysis

Data analysys with, a one-way ANOVA was carried out to compare means between sample treatments, and Tukey's HSD post-hoc test was used to separate the means. Differences were statistically significant at $p < 0.05$.

3. **Results and Discussion**

The data on the physicochemical characteristics of coffee soap are presented in Table 1 below.

Table 1. Chemical Characteristics Of Coffee Soap

Chemical characteristics	*Light*	*Medium*	*Dark*
Moisture content	15.16 ±0.014^{c}	14.08 ±0.113^{b}	13.47 ±0.141^{a}
pH	10.16±0.15^{a}	10.29±0.20^{a}	10.67±0.18^{b}
Free Fatty Acids	1.02±0.06^{b}	1.01±0.08^{b}	0.79±0.15^{a}
Alkali Free	0.37±0.02	0.36±0.10	0.39±0.07
Caffeine	0.17±0.01^{a}	0.19±0.01^{b}	0.19±0.00^{b}

Note: Different letters behind the average value in the same row indicate a very significant difference ($p<0.05$). Data is the average of analysis results.

The Moisture content of soap will affect the quality of the soap produced. Each soap has a different Moisture content because each manufacturer has a different soap-making formulation.Therefore, testing the Moisture content of soap needs to be carried out. The average results of the soap Moisture content with the coffee roasting type formula were, respectively, 15.16%, 14.,08%, and 13.47%. The observed soap Moisture content still meets SNI 3532:2016 with a maximum of 15% for solid bath soap[16]. The results of the ANOVA show that the type of coffee roasting affects the moisture content of the soap, dark roasting type has the best moisture content. This also shows that the longer the coffee roasting process, the lower the moisture content of the coffee. The color of coffee beans changes to become darker as the coffee roasting level increases, this coffee roasting level is correlated with the length of time the coffee beans are roasted[17]. [18] heat treatment and roasting time provide different values for the moisture content of ground coffee. This is because the longer the roasting time, the slower the temperature will increase and the greater the amount of water released. The higher temperature will cause the moisture content of the coffee grounds to decrease.

The results of data analysis using ANOVA showed that the type of coffee roasting had an effect on the characteristics of coffee soap with $P<0.002$.The results of the analysis obtained the average pH for natural coffee soap with light (10.16), medium (10.29), and dark (10.67) roasting. shows that the light and medium roasting types are not significantly different, but the dark roasting type is significantly different from the light and medium roasting types. The data shows that the darker the roast level, the higher the pH. Coffee with a darker roast level has a higher pH than light roast coffee, corresponding to a decrease in organic acid content [19]. During the roasting process, high temperatures (around 200–240°C) cause the thermal degradation of organic acids such as chlorogenic acid, citric acid, malic acid, and acetic acid. As a result, the total acidity decreases and the pH value increases. The longer or darker the roast, the less acid remains in the coffee beans. More severe roasting conditions cause degradation of organic acids, decrease total acidity, and increase the pH of the coffee brew [20].

Average results of natural soap-free fatty acids coffee with light (1.02), medium (1.01), and dark (0.79) roasting. The results of the ANOVA show that the type of coffee roasting affects the free fatty acids of the soap. The longer or darker the roasting level (dark roast), the lower the free fatty acids in the coffee soap will be. In line with the statement of [21] that the longer or darker the roasting level (dark roast), the more free fatty acids are broken down, so that the FFA content in coffee beans decreases. This is in line with the fact that roasting at high temperatures breaks down the lipid structure (triglycerides, phospholipids) and triggers oxidation reactions and the formation of volatile products (aldehydes, ketones, etc.). These reactions change or decompose free fatty acids so that the FFA content measured after dark roasting is often lower than after lighter roasting [22]. According to BSN (2016), the maximum amount of fatty acids in bath soap is 2.5%. This shows that coffee soap contains free fatty acids that are still within the standard.

Average results of natural soap-free alkaline coffee with light (0.37), medium (0.36), and dark (0.39) roasting. There was no effect of the coffee roasting level on the free alkali content of coffee soap, because the amount of NaOH used was in accordance with the saponification reaction requirements in all treatments. [23]**,** The free alkali content depends primarily on the balance between the amount of base and oil, not on the type or degree of oil processing as long as the raw material is still suitable for saponification. So the level of coffee roasting does not have a significant effect on the free alkali content of coffee soap, because the soap formulation uses a balanced amount of base and oil, so that the saponification reaction takes place perfectly in all treatments.

The results of the analysis show that there is an influence of the type of coffee roasting on the caffeine content of natural coffee soap (Table 1). The results of this study show that the caffeine content of coffee soap is between 0.17% and 0.19%. As with measuring the caffeine content of ground coffee, different types of roasting affect the caffeine content. Temperature variations have a very significant effect on the caffeine content of coffee; the higher the temperature, the darker the color of the roasted coffee [24] . The highest caffeine levels are found in medium-maturity coffee, and the lowest caffeine levels are found in coffee with a young maturity level [25] . Based on the statement of [26] caffeine content is directly proportional to the temperature and roasting time; the higher the temperature and the longer the roasting time, the higher the percentage of caffeine content obtained, in line with the results of this research. The caffeine content of natural coffee soap is higher along with the medium and dark roasting types, where this type of roasting process uses a longer temperature and time compared to the light roasting type. Caffeine is relatively heat stable during coffee roasting, although the caffeine content tends to be lower in dark roasted coffee compared to light roasted coffee due to loss of sublimation at high roasting temperatures [27]. This can be seen from the caffeine content of natural coffee soap being lower after heating when compared to the caffeine content of ground coffee.

4. Conclusions

This study demonstrated that the type of coffee roasting significantly influenced the physicochemical characteristics of natural coffee soap produced using the cold process method. Variations in roasting type (light, medium, and dark) affected moisture content, pH, free fatty acids, and caffeine levels, while no significant effect was observed on free alkali content. The dark roast soap exhibited the lowest moisture content (13.47%) and the highest pH (10.67), indicating a firmer texture and greater chemical stability. Free fatty acid levels decreased with increasing roast intensity, suggesting that prolonged heating promoted lipid degradation. The highest caffeine concentration (0.19%) was found in the medium roast treatment, whereas free alkali levels remained relatively constant across all treatments (0.36–0.39%). Overall, dark-roasted coffee produced the most balanced and stable soap formulation.

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