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The Effect of Seaweed Combination on the Extract of Robusta Coffee (*Coffea robusta*) Waste Extract in Producing Facial Mask Products

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ABSTRACT

Aceh is one of the biggest coffee producers in Indonesia's province. The coffee farmers separate the coffee beans with the flesh of the fruit for processing the seeds, while the coffee fruit is considered as waste used as animal feed. Based on existing research, the coffee flesh produced contains polyphenol compounds as antibacterial. A very promising sea product from Aceh is seaweed. Brown seaweed (*Sargassum sp.*) is very common in the west coast of Aceh province. One of the derivatives is alginate. In this study, a combination of polyphenols extracts from coffee flesh and alginates from seaweed Sargassum sp. used as a basic ingredient in making face masks. Tests on organoleptic observations, pH testing, testing time of dried preparations, and testing of bacterial activity have been carried out in this study. In addition, variations in the mixture preparation based on the particle size of alginate powder have also been carried out in this study. The results obtained indicate that the yield produced from coffee fruit extracts is 5.86%, the highest yield of alginate is by potassium carbonate of 40.77%, the acidity of the combination is 4.5 - 6.5. For preparations, drying time has an average estimated time of approximately 6 minutes. Bacterial activity test showed that K_2CO_3 extracting agent concentration of 2% was strong against bacterial growth with a clean area of 14 mm. With these results, it can be seen the combination is able to inhibit the rate of bacterial growth, so it is recommended for alternative ingredients in the manufacture of cosmetics pharmaceutical industry masks.

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INTRODUCTION

Aceh Province is a province located in the western part of Indonesia. One of the commodities from Aceh province is the coffee producer. Famous coffee from Aceh is Robusta (Coffea robusta) and Arabica (Coffea Arabic) types (Badan Pusat Statistik, 2010). In line with the habits of the Acehnese people who love to drink coffee both in the morning and at night, so the production of coffee plants continues to increase in Aceh. In the other coffee-producing regions applying coffee, cultivation has made a trend as a beverage that is able to accommodate local commodities both in the types of variants and their serving techniques (Ridwansyah, 2003).

Research shows that the skin of coffee fruits and minerals can increase the growth of coffee seeds effectively and increase plant growth and increase the effectiveness of inorganic fertilizers when combined (Pujiyanto, 2007). And the other research shows that coffee can protect against skin cancer in women. The results obtained showed that more than 67,000 women enrolled in the study carried out routine activities by drinking more than four cups of coffee per day, which was associated with a 25 percent reduced risk of endometrial cancer. Women who drink two to three cups per day reduce their risk by 7 percent (Harmandini, 2009; Edward, 2011).

Coffee bean extract containing antioxidants contained caffeine compounds and polyphenols (Harahap, 2017). Polyphenols are an aromatic phenolic group that serves as a barrier to free radicals in the human body while caffeine is a secondary metabolite compound that acts as a stimulant for the human body by providing a calming effect for people who consume them routinely. (Esquivel, 2010; Ferrazzano, 2011; Widyotomo, 2007). Polyphenols in coffee extracts also function as inhibitors of bacterial growth and proliferation, which tend to have negative effects on humans such as E.coli (Fardiaz, 1995; Widyotomo, 2006).

The Province of Aceh also has extraordinary marine potential. One such potential is the seaweed *Sargassum sp* (Harahap, 2018). Green seaweed is one of the agar-producing seaweed or *agarophytes* (Anton, 2017). Sargassum sp. contributed the most compared to other types and the fastest to reproduce (Suparmi & Sahri, 2009). The main function of agar is as a stabilizing agent, stabilizer, emulsifier, filler, purifier, gel maker, and others. Some industries also make use of the nature of the gel's ability of agar as the food, pharmaceutical, cosmetics, skin, photography and as a medium for bacterial propagation (Rahmadi, Pangestuti, & Salim, 2009).

Alginate is the main component in the class of brown algae (*Phaeophyceae*) and is an important compound in the cell walls of these plant species. When viewed from the chemical aspect, alginate is a pure polymer of uronic acid arranged in the form of a straight line based on its structure. Alginate has mannopyranosil uronic monomers and gulopyranosyl uronic acid. Heteropolymer compounds that combine from these monomers will form alginate compounds. Alginate is used as an additive, emulsion and stabilizer in the food, medicine and cosmetics industries. This compound is better known as the salt of alginic acid which is most often found in the form of sodium alginate (Prasetyaningrum, 2002).

Based on these data, researchers are interested in conducting research by utilizing waste coffee fruit into cosmetics face masks halal products by doing a combination of Robusta coffee extract (*Coffea robusta*) with seaweed *Sargassum sp* from Aceh.

MATERIALS AND METHODS

Materials

The materials used in this study were a glass device (Pyrex®), pH meter, universal pH indicator, mesh size sieve and FTIR Frontier. At the same time, the materials used such as aquades, HCl p.a., NaOH pellets, methanol, HPMC, Na_2CO_3, K_2CO_3, cultures of *Escherichia coli* and *Staphylococcus aureus* bacteria.

Extraction and separation of polyphenols from coffee bean skins

Coffee bean skin waste consisting of pulp (mesocarp), skin (exocarp), mucilage and parchment (endocarp). The waste is then dried and then mashed to obtain a fine particle size. Eight hundred grams of finely ground coffee bean skin is extracted with methanol for approximately 3 days. The extract was then concentrated with a rotary evaporator so that a concentrated extract was obtained.

Brown seaweed (*Sargassum sp*) preparation as a filler

Brown seaweed (*Sargassum sp*) is dried and cleaned then cut into small sizes. After being small size then dried in the sun until completely dry. Pieces of brown seaweed (*Sargassum sp*) are then extracted into alginate. The dried alginate is then ground into a fine powder and then sieved with a size of 20 mesh, 30 mesh and 40 mesh according to the preparation of the mask according to Indonesian National Standard No. 16-6070-1999.

A combination of coffee fruit extract with brown seaweed (*Sargassum sp*) powder

A total of 10 ml of Robusta coffee fruit extract 3% mixed with 1 gram of hydroxypropyl methylcellulose (HPMC) was stirred until homogeneous. After that, 10 grams of brown seaweed (*Sargassum sp*) was added the same thing is done for each particle size of brown seaweed powder (*Sargassum sp*) according to the Table 1.

Table 1. Variations in the combination of ingredients for making face masks

No	Ingredient	F1 (%)	F2 (%)	F3 (%)
1	Robusta Coffee Fruit Extract 3%	10	10	10
2	HPMC	1	1	1
3	Brown seaweed (*Sargassum sp*) powder 20 mesh	10	-	-
4	Brown seaweed (*Sargassum sp*) powder 30 mesh	-	10	-
5	Brown seaweed (*Sargassum sp*) powder 40 mesh	-	-	10

FTIR test

The extract combination that has been obtained will be tested using the FTIR instrument to determine the active functional groups before mixing and after mixing plays a role in inhibiting bacteria.

Evaluation of Mask Preparations

Organoleptic Observations

Organoleptic testing of the sample is done by observing changes in the shape, odor and color of the preparation done visually after making the base material. Preparations are usually clear with solid consistency.

PH testing

PH testing is done using universal pH sticks dipped in samples that have been dissolved in distilled water. The color changes in the universal pH are then matched with the range of universal pH indicators. The appropriate pH requirements are 4.5-6.5.

Testing the time of preparation to dry

Testing the time to dry, is done by applying a mask to the skin of the hand and observing the time needed for the preparation to dry, counting the time it starts when it is applied until a dry layer is completely formed. The requirements for drying time are 15-30 minutes. Then this time is compared with the dry time mask of innovator products on the market.

Bacterial activity test

Antibacterial activity test was carried out on two types of bacteria, namely Escherichia coli and Staphylococcus aureus bacteria on two samples, namely alginate powder with variations of extracting agent Na_2CO_3 2% and K_2CO_3 2%. The extracting alginate extract samples were each diluted with various concentrations of 6.25% and 12.5% with 3 repetitions. The solvent used is

aquades. Antibacterial testing was carried out using the disk diffusion method. With the principle of work, test material saturated into paper discs. Disc paper was planted in, in each media so that the sloping agar was mixed with Escherichia coli and Staphylococcus aureus bacteria in each sample. Furthermore, each positive control sample used was amoxicillin while the negative control was aquades. Then incubated at 37 ℃ for 24 hours. After the incubation period is achieved, then the inhibition diameter is formed in each sample and the bacteria produced. (Department of Pharmacology, 2007)

Inhibition zone diameters formed due to the antibacterial power of each extracted product, measured from the left side to the right side using a ruler. Davis and Stout (1971) stated that if the inhibition zone formed is <5 mm, the inhibitory activity is categorized as weak, if the inhibition zone of 5-10 mm is categorized as a medium, the inhibition zone of 10-19 mm is categorized as strong and 20 mm or more categorized very strongly.

RESULT AND DISCUSSION

The separation method used is the maceration method. Coffee bean skin peeling and cleaning. 800 grams of shelled coffee bean extracted with methanol for approximately 72 hours. After the extract is obtained, it is then concentrated with a rotary evaporator so that the extract is concentrated. The extract obtained was brownish yellow as much as 45.5 mL.

The yield extract is calculated. The yield can be seen in Table 2.

$$\% \text{ Yield} = \frac{Extract\ weight}{Sample\ weight} \text{ x } 100\ \% \text{ ..... (1)}$$

Table 2. Results of the extraction of coffee fruit flesh with methanol as a solvent

Sample	Dry Weight (g)	Weight of Methanol Extract (mL)	Yield (%)	Extract Color
Pulp	800	45,5	5,68	Brownish yellow

From the data obtained, it can be seen that the extract yield produced very little. This is because in the flesh content of coffee fruits contain more water so that when extracted water tends to dissolve with ethanol and evaporate when heating the rotary evaporator.

Brown seaweed (*Sargassum sp*) preparation as a filler

Sample preparation is the most important step in analyzing the characteristics of alginate powder in brown seaweed (*Sargassum sp*) with a variety of extracting agents. In analyzing the characteristics of alginate powder in brown seaweed (*Sargassum sp*) with a variety of extracting agents, several steps are needed, namely the refinement of the sample, the preparation of HCl solution with a concentration of 5%, Na_2CO_3 solution with a concentration of 5%.

Sample preparation starts with taking the dried sample, then blended using a blender. This aims to reduce the surface area so that the compounds in the sample are easily attracted. It is soaking the sample using 5% HCl. The immersion aims to eliminate the salt and mineral content in the seaweed. After that, the seaweed is extracted where the solvent used is 5% Na_2CO_3. Each extract was filtered using a filter cloth to separate the filtrate and residue. The filtrate added with NaOCl aims to blanch and eliminate dyes. Next 5% HCl is added to form alginic acid on the surface. The alginic acid is then filtered and washed clean. The next stage added 10% NaOH aims to convert alginic acid into sodium alginate.

FTIR Analysis

The extract was obtained and tested using Fourier Transform Infrared Spectroscopy (FTIR)

to determine the functional groups contained in the sample. From the data obtained to describe the typical form of polyphenol compounds. The resulting area is 3367.31 cm^{-1}; 1631.86 cm^{-1}; 1421.83 cm^{-1}; and 1079.53 cm^{-1} (Figure 1). These results indicate that the OH group at wavelength numbers 3600-2800 cm^{-1} with valleys at 3367.31 cm^{-1} which shows the widening of the band, which is a characteristic of OH groups. At a wavelength of 1631.86 cm^{-1}, there is a movement of the C=O carbonyl group connected to the amide group. For wavelengths of 1421.83 cm^{-1}, the presence of carboxylic groups and alkene groups. A wavelength of 1079.53 cm^{-1} indicates the presence of sulfur contained in amino acids in proteins known as active antioxidant compounds. From the data obtained, it can be concluded that the coffee fruit extract contains phenol compounds, amides, carboxylic acids, alkenes, and amino acids.

The following table shows (Table 4) the results of the test characteristics of alginate powder from brown seaweed (*Sargassum sp*) origin of the Lampoh Sibrek Lhoknga Aceh Besar coast. Based on Table 5, the spectra data of FTIR sodium alginate powder spectrum patterns in the wavelength region 3425.58 cm^{-1} indicate the presence of a hydroxyl group (- OH). Wavelength 1610.56 cm^{-1} indicates the presence of a carbonyl group (C = O). Wavelength 1402.25 cm^{-1} indicates the presence of Na in the alginate isomer. Wavelength 1047.35 cm^{-1} shows the carboxyl group (C - O). Wavelength 935.47 cm^{-1} indicates the presence of groups C - H. Wavelength 891.11 cm^{-1} indicates the presence of guluronate fingerprints and at wavelength 815.88 cm^{-1} indicates the presence of mannuronate fingerprints (Figure 2).

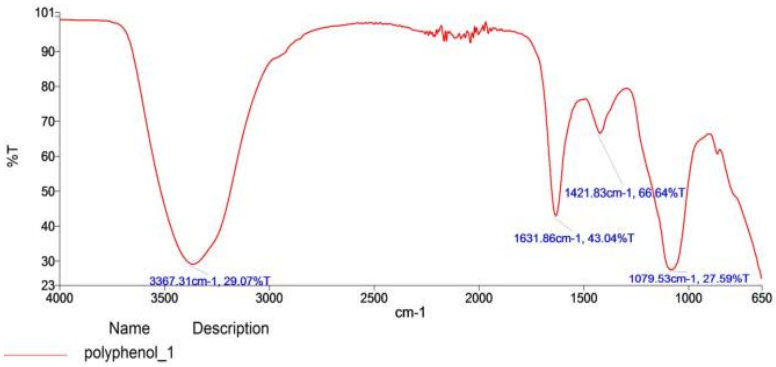

Figure 1. FTIR spectrum graph of Robusta coffee

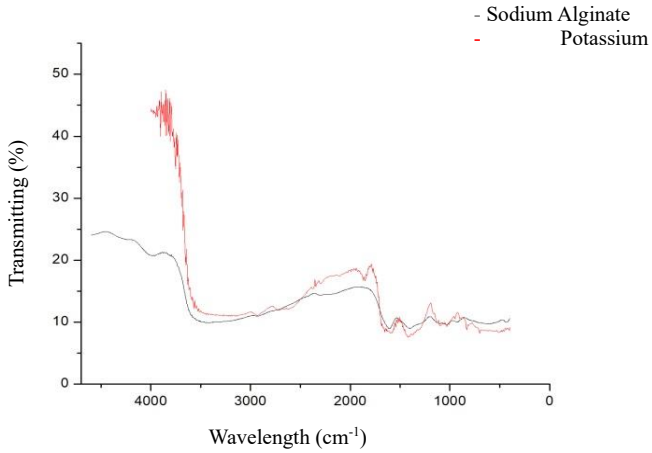

Figure 2. IR Spectrum Extracted Results

Table 4. Data Analysis of IR Spectrum Sodium Alginate Extraction

Wavelength (cm^{-1})	Functional Groups	Vibration
3425,58	Hydroxyl group (O – H)	*Stretching*
1610,56	Carbonyl group (C = O)	*Stretching*
1402,25	Sodium in alginate isomers	*Bending*
1047,35	Carboxyl group (C – O)	*Bending*
935,47	C – H	*Bending*
891,11	Guluronate (Fingerprint)	*Bending*
815,88	Mannuronate (Fingerprint)	*Bending*

Table 5. Test Results for the Characteristics of Alginate Powder

Parameter	Sodium Alginate	Potassium Alginate	Alginate Quality Standards (***Food Chemical Codex, 1981)***
Yield	26,6 %	40,77 %	> 18 %
Ash	1,23 %	6,1 %	18 – 27 %
Water content	9,30 %	2,9 %	< 15 %
Viscosity	10 cP	11,64 cP	10 – 5000 cP
pH	7,40	8	3,5 – 10

Evaluate mask preparations

Evaluation of mask preparations that have been combined is carried out observation and testing as follows Tabel 6.

Table 6. Comparison of each mask preparation

Sample	Organoleptic Observations			pH	Time to dry
	Texture	Odor	Color		
F1	Smoot (+)	Odorless	Dark brown	6,0	6 minutes 56 seconds
F2	Smoot (+)	Odorless	Dark brown	6,0	6 minutes 15 seconds
F3	Smoot (++)	Odorless	Dark brown	6,0	5 minutes 24 seconds

Bacterial activity test

Samples with a variation of Na_2CO_3 2% extracting agent and K_2CO_3 2% extract, each made an initial stock of 25% extract. Each extracted sample was made with a concentration variation of 6.25% and 12.5% to determine the inhibitory activity of bacterial growth at that concentration. The dilution can be calculated using the following formula:

Initial concentration (%).V_1 = concentration (%) .V_2 …….(2)

The results of testing the antibacterial activity of alginate extracts with concentrations of 6.25 and 12.5% respectively for *Escherichia coli* and *Staphylococcus aureus* bacteria can be seen in Tables 7 and 8.

Table 7. The diameter of inhibition zones of alginate extract with variations of extracting agent Na_2CO_3 2% at concentrations of 6.25% and 12.5% against *Escherichia coli* and *Staphylococcus aureus* bacteria.

Repetitions	Concentration (%)	Inhibition zones (mm)	
		Escherichia coli	*Staphylococcus aureus*
I	6,25	7,1	7,6
II		7,2	7,8
III		7,1	8
I	12,5	11,6	9,4
II		11,9	8
III		11,5	9,6
Control (+)		27,7	20,6
Control (-)		-	-

Note: I, II, and III were repetitions of the bacterial activity test treatment. Control (+) is a disk that has been soaked with amoxicillin while the control (-) is soaked with distilled water.

Tabel 8. The diameter of inhibition zones of alginate extract with variations of extracting agent K_2CO_3 2% at concentrations of 6.25% and 12.5% against *Escherichia coli* and *Staphylococcus aureus* bacteria

Repetitions	Concentration (%)	Inhibition zones (mm)	
		Escherichia coli	*Staphylococcus aureus*
I	6,25	10,7	8,7
II		10,4	10
III		10	9,8
I	12,5	14	11
II		14,5	10,9
III		14	11,1
Control (+)		30,2	21
Control (-)		-	-

Note: I, II, and III were repetitions of the bacterial activity test treatment. Control (+) is a disk that has been soaked with amoxicillin while the control (-) is soaked with distilled water.

CONCLUSION

Based on the results obtained showed that the yield produced from coffee fruit extracts was 5.86%, the highest yield of alginate was produced by potassium carbonate of 40.77%, the acidity of the combination was 4.5 - 6.5. Preparations drying time has an average estimated time of approximately 6 minutes. Bacterial activity test showed that K_2CO_3 extracting agent concentration of 2% was strong against bacterial growth with a clean area of 14 mm. With these data, it can be seen that the combination is able to inhibit the rate of bacterial growth, so it is recommended for alternative ingredients in the manufacture of cosmetics pharmaceutical industry masks.

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