

The Quality of Transparent Soap from Farmer's Crude *Calophyllum* Seed Oil

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Abstract—*Calophyllum inophyllum* plants is one of the potential non-edible vegetable oil in Indonesia. Currently, the utilization of *C. inophyllum* seed oils directed for biofuels such as biodiesel feedstock. The oleochemical product diversification of *C. inophyllum* seed oil into transparent soap products is prospective to be developed, considering its utilization that does not compete with edible oil. The purpose of this research is to obtain the best conditions for the production process of *C. inophyllum* seed oil transparent soap. The research process is conducted in two phases. The first is the determination of the basic formula of transparent soap from *C. inophyllum* seed oil, and the second is the improvement of the organoleptic quality of the *Calophyllum* transparent soap. The research design used is the completely randomized factorial design with two treatments, i.e. the addition of olive oil and ethanol. The determination of the best formula for *Calophyllum* transparent soap is based on the weighting method on a number of physicochemical and organoleptic criteria. The results of the research show that the addition of olive oil and ethanol has a significant effect on the amount of *Calophyllum* transparent soap suds ($P < 0,05$). The quality improvement of *Calophyllum* transparent soap is conducted with the addition of honey, fragrance, and coloring agent. The addition of honey has a significant effect on the transparency and color of *Calophyllum* transparent soap ($P < 0,05$). The addition of fragrance types has a significant effect on transparency, color and aroma ($P < 0,05$), but does not have a significant effect on the hedonic test on texture and impression after use ($P > 0,06$). Based on the composite weighting method, the most preferred formula for *Calophyllum* transparent soap according to the panelists is the use of olive oil 5%, ethanol 15%, honey 0,3%, and floral fragrance 1%. This best condition for *Calophyllum* transparent soap are able to meet the standards of the Saudi Arabian Standard Organization (SASO) for the water content and evaporating substances, as well as free alkali.

Keywords— *Calophyllum inophyllum* seed oil, olive oil, ethanol, transparent soap.

I. INTRODUCTION

Tamanu (*Calophyllum inophyllum* L) is classified as a forest plant that is potential as a source or producer of non edible vegetable oil. The oil content in the core or kernel of the tamanu seed is very high. Dweek and Meadows reported the tamanu seed oil content of 75% [1]. According to Greshoff in Heyne, it was 55.5% in the fresh core and 70.5% in the dry core with a 3.3% of water content [2]. Soerawidjaja reported a range of 40%-73% [3] and the finding of Sahirman was 17.5% in the pressing process of wet seeds or 48.6% of the dry core weight [4]. However, using hexan solvents with the soxhlet method, the yeild reached 61.20%.

The utilization of tamanu seed oil is primarily directed to biofuels such as biodiesel. One of the derived product

diversifications from the tamanu seed oil compound, namely soap products, has a good prospect to be developed, moreover because its utilization does not compete with food interests. The main raw material for soap commonly uses edible oil such as palm oil and coconut oil. The development of soap products with its various kinds has had its own market, particularly for personal care, health, beauty and skin care products.

Calophyllum inophyllum L (tamanu) seed oil is indicated to have properties as antimicrobial and cytotoxic agents. Xanthone antibiotic contents in tamanu oil can inhibit *Staphylococcus aureus*, *Pseudomonas aeruginosa*, *Bacillus subtilis*, *Salmonella typhimurium*, and *Klebsiella pneumoniae* [1]. Hasibuan *et al.* reported that tamanu seed oil positively contains flavonoid, saponin, steroid and triterpenoid compounds [5]. Tamanu seed oil can inhibit the

growth of *S. aureus* with the biggest resistor average diameter, i.e. 6.75 mm on the antibacterial activity test.

The utilization of active substances in tamanu oil is expected to be an added value for derivative products of *Calophyllum* transparent soap. Thus, the diversification of the utilization of tamanu seed oil will develop, besides as a biofuel and to encourage the development of down stream industries of oleochemical products based on *Calophyllum* seed oil or tamanu seed oil. Transparent soap is classified as solid soap that has a luxurious appearance with its transparency that gives a natural and attractive impression [6]. This soap is generally aimed at the upper-middle market segment as a beauty soap or for treatment.

Transparent soap has differences from other soaps, both in terms of appearance and the additional materials used. One of the additional materials that has a great effect on the formation of soap transparency is ethanol. To obtain a good level of transparency on *Calophyllum* transparent soap, the addition of an appropriate amount of ethanol is required. The characteristics of the thick, dark green tamanu oil with a pungent aroma may affect consumer preferences on the *Calophyllum* transparent soap produced. Therefore, it is necessary to do research on the best formulation of *Calophyllum* transparent soap and on its organoleptic quality improvement.

The purpose of this study is to determine the best combination in the production of *Calophyllum* transparent soap with the addition of olive oil and ethanol, and to make improvements in the organoleptic quality of *Calophyllum* transparent soap with the addition of honey, fragrance, and coloring agent.

II. RESEARCH METHOD

The research was conducted at the Bioenergy and Environmental Laboratory of Djuanda University Bogor from March 2013 to August 2013. The research process is conducted in two phases. The first is the determination of the basic formula of transparent soap from *C. inophyllum* seed oil and the second is the improvement of the organoleptic quality of the *Calophyllum* transparent soap.

In the early stages, the degumming process is performed on the raw material, i.e. crude *Calophyllum* oil. The degumming result *Calophyllum* oil, using a 20% H_3PO_4 solution with a concentration of 0.2%, is further given a bleaching treatment using activated carbon powder at a temperature of 120°C for 30 minutes. The results of *Calophyllum* oil bleaching with the best clarity level is used as the raw material for *Calophyllum* transparent soap.

The production of *Calophyllum* transparent soap is done by modifying the Cognis formula [7]. The production of *Calophyllum* transparent soap uses basic materials, i.e. tamanu seed oil with olive oil and ethanol addition treatments. The early stages of the *Calophyllum* transparent soap production are reacting oil with stearic acid and NaOH at a temperature of 60-65°C, which is subsequently added with other materials such as water, glycerin, DEA, sugar, salt, citric acid, and ethanol. Aging is done for 7-14 days for further physicochemical and organoleptic testing. Furthermore, the quality improvement of *Calophyllum* transparent soap is done by treatments of adding honey, fragrance, and coloring. The determination of the best

formula for *Calophyllum* transparent soap is done by a weighting (scoring) method based on the interest value of physicochemical and organoleptic parameters.

The experimental design used in the initial study is the factorial Completely Randomized Design (CRD) with two factors, namely the A factor (the effect of olive oil addition) with 3 levels of treatment (0%, 5%, 10%) and the B factor (the effect of ethanol addition) with 3 levels of treatment (15%, 20%, 25%) with two replications. The parameters tested include data on the results of analysis on physicochemical and organoleptic testing. The mathematical model for physicochemical testing can be written as follows [8].

$$Y_{ijk} = \mu + A_i + B_j + (AB)_{ij} + \epsilon_{ijk} \quad (1)$$

where

- Y_{ijk} : the k^{th} observation ($k=1,2$) on the i^{th} treatment ($i=1,2,3$) and the j^{th} treatment ($j=1,2,3$)
- μ : a fixed constant
- A_i : the effect of olive oil index addition
- B_j : the effect of ethanol aq. index addition
- AB_{ij} : the interaction effect of olive oil index addition and ethanol aq. index addition
- ϵ_{ijk} : the error within each of the treatments.

III. RESULT AND DISCUSSION

Physically, transparent soap from degumming result *Calophyllum* oil has a low level of transparency, but its texture and level of hardness is quite good. This is because the low average value of the transmission percentage of degumming result tamanu oil, i.e. 15%. Therefore, to improve its clarity, a bleaching process is necessary before it is used as a raw material for transparent soap. The bleaching process is carried out using activated carbon in powder form in various concentrations (0.5%, 1%, 1.5%, 3%, and 5%). The average value of the transmission percentage of bleaching result *Calophyllum* oil uses activated carbon powder ranging between 46.6% -61.5% (Figure 1). Concentration treatments of activated carbon powder significantly affect the value of the transmission percentage of the oil ($P < 0.05$). The use of activated carbon powder dose of 1% resulted in the highest value of the average transmission percentage, i.e. 61.5%. For the concentration treatment of this 1% activated carbon powder, the bleaching of *Calophyllum* oil for transparent soap is used.

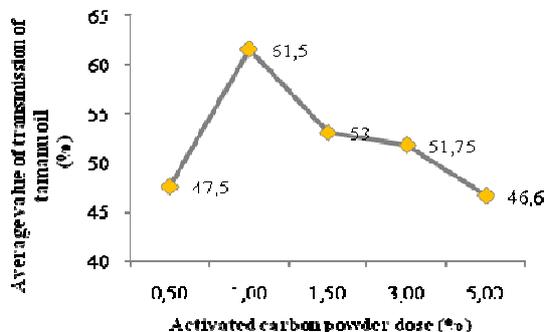


Fig. 1 The effect of activated carbon powder dose on the transmission percentage of *Calophyllum* oil.

A. Physicochemical Characteristics of *Calophyllum* Transparent Soap

1) *Water content and volatile substance*: The existence of water in transparent soap products highly determines the quality of the transparent soap. The more water contained in the soap, the easier the soap will shrink and become uncomfortable when used [9].

Results of the analysis of variance on the water content show that the treatment of the addition of ethanol very significantly affects the water content and volatile substance of the *Calophyllum* transparent soap ($P < 0.01$), whereas the addition of olive oil and the interaction of both do not significantly affect the water content and volatile substance of *Calophyllum* transparent soap ($P > 0.05$).

The average value of water content and volatile substance of *Calophyllum* transparent soap produces ranges between 27.50%-30.06% (Figure 2). This value is in the range of the standard water content and volatile substance of transparent soap set by the Saudi Arabian Standard Organization (SASO), i.e. minimum 17%. The value of water content and volatile substance of *Calophyllum* transparent soap becomes higher with the increase in the amount of ethanol used. The more addition of ethanol, the higher the level of volatile substance contained in the transparent soap [9]. Results of the LSD post hoc test at the significant level of 0.05 shows the treatment of 15% ethanol addition resulting in the best average value of water content and volatile substance. The content of volatile substance in transparent soap products comes from its volatile constituent materials, or it may also come from the oxidation reaction of fatty acids contained in the transparent soap [10].

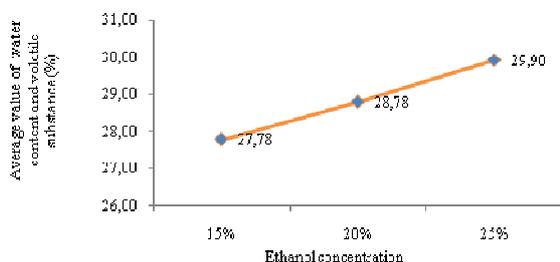


Fig. 2 Average value of water content and volatile substance of *Calophyllum* transparent soap with different levels of ethanol addition.

2) *pH value and free alkali level*: According to ASTM [11], the pH quality requirement of soap ranges between 9-11. The pH value of *Calophyllum* transparent soap products produced a range between 9.00-9.31. The lowest pH value is resulted from the A3B1 treatment (the addition of 10% olive oil and 15% ethanol), while the highest pH is resulted from the A3B3 treatment (the addition of 10% olive oil and 25% ethanol). Results of the analysis of variance on the pH of *Calophyllum* transparent soap show that treatments of olive oil and ethanol addition, as well as their interaction do not affect the pH value of *Calophyllum* transparent soap ($P > 0.05$).

The free alkali level in soaps is due to the presence of alkali that does not react with fatty acids in the saponification process. The saponification process is affected by the molecular weight which is also different,

depending on the number of carbon, oxygen and hydrogen atoms that compose triglycerides [12]. Olive oil is dominated by the same fatty acids as those composing tamanu seed oil, i.e. oleic acid. This causes the addition of neither olive oil nor ethanol to affect the free alkali level in *Calophyllum* transparent soap.

Based on analysis results, the free alkali level in *Calophyllum* transparent soap ranges between 0.17-0.28 percent. The free alkali level in the *Calophyllum* transparent soap meets the standard of that based on SASO [13], i.e. a maximum of 1%. The average value of free alkali level in *Calophyllum* transparent soap is presented in Table 1.

TABLE I
AVERAGE VALUE OF *CALOPHYLLUM* TRANSPARENT SOAP FREE ALKALI LEVEL WITH DIFFERENT LEVELS OF OLIVE OIL AND ETHANOL ADDITION

Ethanol concentration	Olive oil concentration		
	A1 (0%)	A2 (5%)	A3 (10%)
B1 (15%)	0.28±0.07	0.20±0.05	0.20±0.01
B2 (20%)	0.22±0.01	0.22±0.01	0.17±0.02
B3 (25%)	0.23±0.02	0.22±0.01	0.21±0.04

3) *Amount of suds*: The analysis of variance results on the amount of suds indicate that treatments of olive oil and ethanol addition significantly affect the amount of *Calophyllum* transparent soap suds ($P > 0.05$), but their interaction does not have an effect ($P > 0.05$). The average value of the highest amount of suds at 120.01 cm³ comes from the A1B1 treatment, while the average value of the lowest amount of suds at 97.53 cm³ is from the A3B3 treatment.

Based on Figure 3, it can be seen that a decline occurred in the amount of suds along with the increase in the amount of olive oil addition. Results of the LSD post hoc test show that the treatment of olive oil addition at the level of 0 percent (A1) and 5 percent (A2) are not significantly different from each other, but both are significantly different from the treatment of olive oil addition at the level of 10 percent (A3).

Olive oil contains non-glyceride small components, among others n-paraffin compounds, which is a small component of hydrocarbon fraction [14]. The main hydrocarbon component contained in olive oil is *triterpenic squalen* which ranges between 150-700 mg per 100 g of oil [15]. The content of non-glyceride components contained in olive oil belongs to non saponified fraction which affects its ability in forming suds when made into soap [16]. Besides, olive oil also contains methanol and ethanol which are esters of fatty acid as volatile compounds [17]. Volatile compounds that are found in soaps may give anti suds effects [18-20].

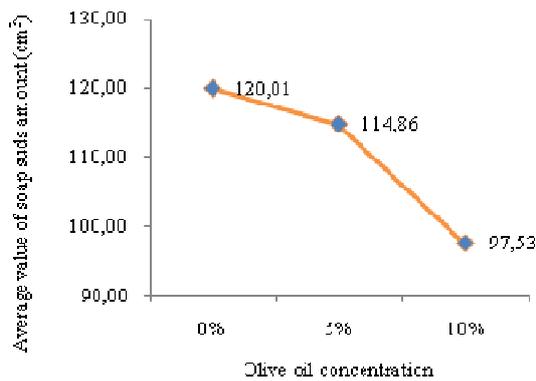


Fig. 3 Effect of olive oil addition on average value of *Calophyllum* transparent soap suds amount

Based on Figure 4, it can be seen that a decline occurs in the value of the suds amount, along with the increase of the ethanol amount added. Results of the LSD post hoc test show that the treatment of ethanol addition at the level of 15% (B1) results in the best average value of suds amount. Besides acting as a solvent and transparent agent in *Calophyllum* transparent soap, ethanol can also give an anti suds effect [18], so that the more addition of ethanol, the lower the suds produced.

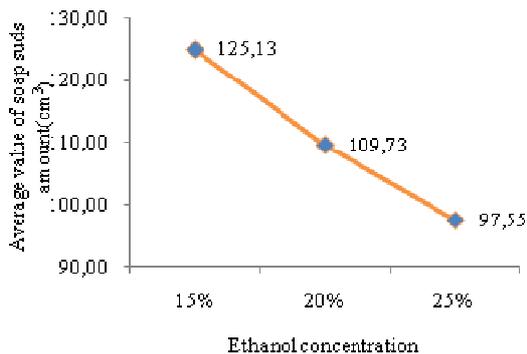


Fig. 4 Effect of ethanol addition on average value of *Calophyllum* transparent soap suds amount

B. Organoleptic Quality of *Calophyllum* Transparent Soap

Organoleptic quality of *Calophyllum* transparent soap: The organoleptic test performed is the hedonic test. The hedonic test is conducted to determine the panelists' level of acceptance on the products of *Calophyllum* transparent soap produced. This organoleptic test includes tests on the trained panelists' preference on transparency, texture, aroma, color and coarse impression on the skin after the use of transparent soap. The rating scale used is 1 to 5 (1=strongly dislike; 2=dislike; 3=average; 4=somewhat like; 5=extremely like).

1) **Transparency and texture:** The level of transparency in *Calophyllum* transparent soap is one of the important parameters that distinguish the appearance of transparent soap from other solid soaps. The evaluation on preference level on the soap's transparency is done visually by looking at the samples of *Calophyllum* transparent soap tested. Most of the panelists give the impression from average to

somewhat like (score 3.1-3.7) on the transparency of the *Calophyllum* transparent soap tested. When compared to a commercial transparent soap, the preference average value on the transparency of this soap product is still lower (score 4.1-4.2).

The evaluation of preference level on the texture is done by touching and feeling the texture of the *Calophyllum* transparent soap tested. Most of the panelists give the impression from *average to somewhat like* (score 3.3-3.6) on the texture of the *Calophyllum* transparent soap. The results of evaluation on the texture of the *Calophyllum* transparent soap is slightly lower when compared to that of a commercial transparent soap (score 4.1-4.2).

2) **Color and aroma:** The *Calophyllum* transparent soap produced has a natural yellow color in accordance with the color of the tamanu oil raw material used. Most of the panelists give the impression of average (scale 3.0) on the color of the *Calophyllum* transparent soap produced. The *Calophyllum* transparent soap formula with the addition of 5% olive oil and 15% ethanol gives a transparent soap color that is more acceptable to the panelists compared to the other soap treatments.

Tamanu seed oil has a specific and strong smell. The panelists give the impression of *average* (scale 3) on the aroma of the *Calophyllum* transparent soap produced. This value is under the evaluation results of the commercial transparent soap, i.e. at scale 4 (*somewhat like*) and 4.5 (*like*).

3) **Impression of use:** The comfort test is a test to evaluate the impression of comfort perceived by the skin after using the *Calophyllum* transparent soap. Most of the panelists give the impression from average (scale 3) to *somewhat like* (scale 4) after using *Calophyllum* transparent soap. The highest average value as results of the panelists' evaluation on the impression after use is obtained from the transparent soap with the A1B3 and A2B1 treatments, i.e. 3.6 (*somewhat like*). The value of both of these treatments is almost close to the value given by panelists on that of the commercial transparent soap (score 4.1-4.2).

C. Improvement on organoleptic acceptance of *Calophyllum* transparent soap

Based on the analysis on the organoleptik test results on the *Calophyllum* transparent soap, it is discovered that the panelists' acceptance on the aroma and color of the *Calophyllum* transparent soap is still low when compared to that of the control transparent soap. Therefore, the modification of feedstock composition, addition of fragrance and coloring to increase the panelists' organoleptic acceptance on the *Calophyllum* transparent soap produced is carried out.

Based on the analysis of variance results, it is discovered that the addition of fragrance affects the panelists' acceptance on the aroma of the *Calophyllum* transparent soap. The addition of fragrance is able to improve the organoleptic quality for the aroma of the *Calophyllum* transparent soap equal to the control transparent soap [21].

TABLE II
THE BEST FORMULA OF *CALOPHYLLUM* TRANSPARENT SOAP

Parameter	Value	Note
Water content and volatile substance (%)	27,7	min. 17% (SASO)
Free alkali level (%)	0,2	max. 1% (SASO)
Soap suds stability (%)	93,6	-
Soap suds amount (cm ³)	121,3	-
Preference on transparency	4,6	Like
Preference on texture	4,1	Somewhat like
Preference on color	4,3	Somewhat like
Preference on aroma	4,2	Somewhat like
Coarse impression on the skin after the use of transparent soap	4,5	Like

The highest average value on the hedonic test for color is produced by the addition of coloring 113 (blue floral) at 4.3 and coloring 238 (pink floral) at 4.1. The average value of preference on the color of the *Calophyllum* transparent soap product is still lower than that of the commercial transparent soap (score 4.1-4.4), except for the *Calophyllum* transparent soap product which is added coloring 113 (score 4.3) and coloring 238 (score 4.1). Floral pink (438), is the coloring much preferred by consumers or panelists based on the determination of the best coloring.

IV. CONCLUSIONS

Calophyllum oil has a good potential to be processed into transparent soap. Based on the chemical test results, the products of *Calophyllum* transparent soap produced is able to meet the transparent soap standards of the Saudi Arabian Standard Organization (SASO) for the parameters of water content and volatile substances, as well as free alkali. The physicochemical characteristics of *Calophyllum* transparent soap with the addition of olive oil has a range of water content and volatile substances of 27.50–30.06%, a pH of 9.00-9.31, a free alkali level of 0.17-0.28%, suds stability of 88.25-96.29%, suds amount of 85,75-121,5 cm³. Organoleptic test results show that the panelists' acceptance on the organoleptic quality of the *Calophyllum* transparent soap is still lower than that of the commercial transparent soap.

The modification of feedstock composition, addition of fragrance and coloring is able to improve the organoleptic acceptance of *Calophyllum* transparent soap equal to the commercial transparent soap. The types of fragrance in *Calophyllum* transparent soap preferred by panelists are floral, apple and orange fragrances; while the best color of *Calophyllum* transparent soap is obtained from the use of pink and blue colors.

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