Utilization of Virgin Coconut Oil that has been Extracted in Phenolic Compounds as Resource of Diethanolamide Surfactants

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Abstract One of the vegetable oils that can be used as a surfactant is extracts of phenolic compounds from virgin coconut oil (VCO) or VCO residue. The purpose of this study was to make surfactants from VCO residue and to obtain the correct ratio of reactants and the appropriate concentration of NaOH catalyst to produce diethanolamide surfactant. The materials used are methyl ester from VCO residue and diethanolamine with NaOH catalyst. The amidation process uses a mole ratio of diethanolamine and methyl ester (1:1, 1.25:1, 1.5:1, 1.75:1 and 2:1) at a temperature of 160 °C for 3 hours, and a NaOH catalyst with a concentration of 0.25, 0.5, and 0.75%. The results showed that the treatment of the ratio of methyl ester and diethanolamine and the concentration of NaOH catalyst produced DEA surfactant with the same consistency, namely a semi-solid viscous liquid with a clear yellow color. The higher the concentration of catalyst, the yield tends to increase. All DEA surfactant products have a pH of 10, with viscosities ranging from 252.47 - 509.89 cP, soaping numbers ranging from 35.22 - 52.95 mg KOH / g, and glycerol levels from 0.08 to 29%.

1. Introduction
A surfactant is a molecule that has both a hydrophilic group and a lipophilic group so that it can unite a mixture consisting of water and oil. Surfactant molecule has a polar part that likes water (hydrophilic) and non-polar parts like the oil (lipophilic) [1]. Surfactants are an ingredient needed in the manufacture of cleaning products such as soap and shampoo, as well as cosmetic and food products. Along with the increase in the population of Indonesia, there is also an increase in products that use surfactants. The surfactant industry in Indonesia is still limited, even though surfactants are needed in large quantities as a wetting agent, emulsifying agent and solubilizing agent in personal care products such as soap and shampoo, cosmetic products, and food products. The use of surfactants as an emulsifier aims to increase the stability of the emulsion by reducing the interface stress between the oil phase and the water phase. Surfactants are used either in the form of an oil-in-water emulsion or in the form of a water-in-oil emulsion. The potential to develop surfactants is still very large, so it is necessary to carry out research on surfactants more broadly. Surfactant production from vegetable raw materials has bright prospects in
Indonesia. Currently, from 95,000 tons of Indonesia's surfactant needs per year, about 45,000 tons are still imported [2].

Surfactants are generally synthesized from petroleum (petrochemicals). While the need for surfactants is increasing, the procurement of surfactants made from vegetable oil has been developed. Compared to surfactants made from petrochemical raw materials, surfactants made from vegetable oil raw materials are biodegradable so they are more environmentally friendly. In addition, the sustainability of its procurement is guaranteed because vegetable oil is a renewable natural resource.

One of the vegetable oils used as a surfactant is coconut oil. With the increasing awareness of good health and environment, the demand for biodegradable surfactants based on plants is also increasing. Therefore, a study is needed to obtain surfactants that have these two criteria, namely obtained from raw materials that can be renewed (renewable) and are degradative in nature so that they are ecologically acceptable. One of the surfactants that meet these two criteria is diethanolamide surfactant.

Coconut oil contains up to 48% lauric acid [3]. Meanwhile, virgin coconut oil (VCO) contains ± 90% saturated fatty acids which are dominated by lauric fatty acids around 47-53% and are believed to be bioactive components [4]. The high lauric acid content in virgin coconut oil has the potential to be made as a surfactant because of the nature of lauric acid, namely as a thickener, softener and moisturizer so that it can be used in industrial cleaning products such as soaps, shampoos and others [5]. In addition, because of its good antimicrobial properties, lauric acid is also used in the pharmaceutical industry. The use of coconut oil as a surfactant has several advantages, including being renewable (renewable resources), cleaner (cleaner) and purer than using petrochemical-based raw materials. Research on vegetable oil into surfactants has been carried out, especially palm oil and coconut oil, but research on virgin coconut oil that has been extracted by minor compounds or known as VCO residue has never been carried out. VCO is currently developing the extraction process of minor phenolic compounds contained therein and used as pharmaceutical and cosmetic preparations. In the extraction process, the amount of VCO taken (extracted) is around 20-25%, while the remaining 80-90% has not been utilized. One of the uses of the VCO residue is processing it into diethanolamide surfactant.

Dietanolamide surfactants are nonionic surfactants, namely surfactants that are not charged. The hydrophobic / lipophilic part of dietanolamide is the carbon chain of lauric acid as the most fatty acid component in coconut oil, while the hydrophilic / lipophobic part is a molecule that is uncharged [6]. The synthesis of dietanolamide is carried out in two ways, namely the reaction between methyl ester and dietanolamine with a by-product in the form of methanol or the reaction between fatty acids and dietanolamine with a by-product in the form of water. These two reactions are called the amidation reaction [7]. During the synthetic dietanolamide or the amidation process, the mole ratio of the reactants, temperature and reaction time affect the amount of fatty amides produced. The use of dietanolamine must be excessive, at least the required mole ratio of dietanolamine: methyl ester is 1.1:1. This is because the higher the mole ratio of dietanolamine, the better the process [8]. However, excessive use of dietanolamine allows the presence of dietanolamine which does not react to form dietanolamide.

Based on this, a research was conducted on the manufacture of dietanolamide surfactants from coconut oil (crude coconut oil and VCO which had been extracted the phenolic compounds). The aim of this research is to make VCO surfactant that has been extracted from phenolic compounds and to obtain the correct ratio of reactants (dietanolamine: methyl ester) and concentration of NaOH catalyst to produce diethanolamide surfactant.

2. Methodology

2.1. Materials and Tools.
The main ingredients used are virgin coconut oil / VCO which has extracted its phenolic compounds (VCO residue), 96% methanol, sodium hydroxide catalyst, 10% H2SO4, and anhydrous Na2SO4 for the transesterification process. Meanwhile, the materials used for the amidation process are methyl ester from VCO and diethanolamine with sodium hydroxide as catalyst.

The tools used in this study were Pyrex 250 and 500 ml beaker, filter paper, 1000 ml Pyrex three-neck flask, reverse cooler, 5 cm magnetic stirrer, Biosan MM-1000 overhead stirrer, Memmert waterbath, statif, clamp, alcohol thermometer (0-100 °C) and (0-250 °C), Pyrex Erlenmeyer 250 and 500 ml, Pyrex 1000 ml separating funnel, Pyrex 500 ml measuring cup, pipette, stirrer, Sartorius analytical balance (0.0001 g) type BSA224S-CW, freezer and refrigerator, also Shimadzu Gas Chromatograph Mass Spectrometer with Auto-Injector & FID Detector type GCMS-QP2010 ultra.

2.2. Methyl ester resulting from the transesterification reaction

The composition of the fatty acid methyl ester using Gas Chromatography was tested in the laboratory of the Manado Industrial Research and Standardization Center. The yield calculation, as well as the methyl ester quality test were tested in the SBRC testing laboratory of the Surfactant and Bioenergy Research Center of IPB Bogor. The density of the test method according to ASTM D 4052-11. The viscosity of the test method according to the Brookfield IK-40 Rheometer. Saponation number, acid number, Iod number, total glycerol content according to SNI 7182: 2012 [9].

2.3. Dietanolamide surfactant

The viscosity of the test method according to IK-40 Rheometer Brookfield, saponation number and total glycerol content according to SNI 7182: 2012 and pH, were all tested in the SBRC testing laboratory of the Surfactants and Bioenergy Research Center of IPB Bogor.

3. Result and Discussion

3.1 VCO Residue Raw Material

Table 1 shows that the quality of the residual VCO raw material has a moisture content and free fatty acids that meet the quality requirements of SNI 7381: 2008 VCO [10], namely <0.2%. The quality of oil raw materials greatly affects the transesterification process of oil and alcohol into methyl esters. If the free fatty acids are <2%, the conversion of oil to methyl esters is carried out directly using the transesterification process, whereas if ALB > 2%, the esterification process must be carried out before the transesterification process, or there will be lathering. Based on ref [11], high levels of free fatty acid (FFA) oil in the process of making biodiesel or methyl ester cause a saponification reaction which will result in a decrease in FAME (fatty acid methyl ester) levels. One of the main factors determining the success of methyl ester production is the level of FFA. High levels of FFA in the production of methyl ester can trigger a saponification reaction which will result in a decrease in the resulting FAME levels. The FFA levels from oil above 1% will reduce the yield rate produced and increase soap formation, so that the separation process of biodiesel / methyl ester and glycerol becomes difficult. Therefore, oil FFA levels above 1% are not recommended to use alkaline catalysts (transesterification reaction) directly without reducing the FFA levels by using an acid catalyst (esterification reaction). The flowchart diethanolamide surfactant manufacturing process can be seen in Figure 1.
Figure 1. Flowchart diethanolamide Surfactant Manufacturing Process
Table 1. VCO Residue Test Results

| No. | Parameter Test       | Unit | Result (Average) |
|-----|----------------------|------|------------------|
| 1.  | Water content        | %    | 0.16             |
| 2.  | Free Fatty Acids (FFA)| %    | 0.19             |

From Table 2, it can be seen that the VCO residue still contains lauric acid which is quite high with an amount of 37.96% compared to the lauric acid content of VCO which is around 48-53%. Lauric acid is a thickener, softener, and moisturizer so that it can be used in the cleaning products industry such as soap, shampoo and others.

Table 2. Composition of VCO Residue

| Composition of VCO Residue | Amount (%) |
|----------------------------|------------|
| Caprylic Acid (C8)         | 6.02       |
| Capric Acid (C10)          | 6.94       |
| Lauric Acid (C12)          | 37.96      |
| Myristic Acid (C14)        | 21.01      |
| Palmitic Acid (C16-0)      | 12.52      |
| Stearate (C18-0)           | 4.74       |
| Linoleic Acid (C18-2)      | 2.12       |
| Oleic Acid (C18-1)         | 8.69       |

3.2 Methyl Esters

From Table 3 it can be seen that the resulting methyl ester product has a density of 0.86 gr / cm3 which meets the quality requirements of SNI 7182: 2015, which is around 0.85-0.89 gr / cm3. This means that the methyl ester obtained from the transesterification results has been separated from glycerol because the presence of glycerol in biodiesel / methyl ester affects the density of biodiesel / methyl ester because glycerol has a fairly high density (1.26 g / cm3). If glycerol is not properly separated from biodiesel / methyl ester, the density of the methyl ester will increase [12]. The total glycerol content in the methyl ester product is 0.33%, slightly above the requirements of SNI 7182: 2015.

The viscosity of the methyl ester product is 1.39 cP (30°C). Viscosity is one of the factors that affect the speed of separation of glycerol from biodiesel in addition to density. High viscosity causes slow separation of methyl esters and glycerol. In addition, the viscosity of the methyl ester is related to the specific density [13]. The high specific density causes high viscosity, making it difficult for fuel to flow and will slow down the combustion process. The viscosity produced in this study was still below the SNI Biodiesel standard. This viscosity is slightly below biodiesel quality requirements which means it is slightly thinner.

The acid number of the transesterified methyl ester is higher than the quality requirements of SNI Biodiesel (Methyl Ester) because it ranges from 1.20-2.53 mgKOH / gr, while the requirement is a maximum of 0.5%. This happens because in the purification of the transesterified methyl ester, 10% H2SO4 is used to deactivate the NaOH catalyst. In the next washing process, warm water is used until the washing water has a neutral pH, but it turns out that there is still sulfuric acid bound to the methyl ester product [14].
Table 3. Results of Methyl Ester Quality Testing from VCO Residue

| Parameter               | Results | Test method                        | SNI 7182:2015       |
|-------------------------|---------|------------------------------------|---------------------|
| Density (gr / cm³)      | 0.86    | ASTM D4052-11                      | 0.85-0.89           |
| Viscosity (cP, 30 °C)   | 1.39    | IK-40 Rheometer Brookfield         | 2.3-6.0 mm /s       |
| Acid Number (mg KOH / gr)| 2.53    | SNI 7182:2012 part 9.13            | 0.5 max             |
| Saponification (mg KOH / g) | 285.00 | SNI 7182:2012 part 9.15            | -                   |
| Iod number (g-I₂ / 100 gr)| 2.53    | SNI 7182:2012 part 9.16            | 115 g-I₂/100 gr max |
| Total Glycerol (%)      | 0.33    | SNI 7182:2012 part 9.14            | 0.24 max            |
| Yield (%)               | 74.69   | Calculation                        | -                   |

The saponation number is a measure for seeing the reaction between KOH and methyl ester. It is also a measure of the average relative molecular mass (chain length) of all the fatty acids present in biodiesel / methyl esters. The soaping number is usually influenced by the molecular weight of the material. The saponification number of the methyl ester resulting from the transesterification process tends to increase with increasing transesterification time, but the increase is only slightly. This means that the transesterification time has very little effect on the formation of soap from the methyl ester product.

The iodine number represents the degree of saturation. The greater the iodine number, the higher the degree of unsaturation. High iodine numbers are an unfavorable property for fuels. The iodine number of methyl ester obtained meets the quality requirements of biodiesel (a maximum of 115 g-I₂ / 100 gr) because it ranges from 1.03-5.79 g-I₂ / 100 gr.

Furthermore, laboratory testing of the methyl ester content was carried out to see the level of purity of the purified methyl ester, where the laboratory test results mean that the methyl ester content of the sample was 97.46%. Furthermore, the methyl ester composition of the purified samples was tested using GC-MS. The test results can be seen in Table 4 below.

Table 4. Composition of Methyl Ester from VCO Residue

| Composition of Methyl Ester | Amount (%) |
|----------------------------|------------|
| Caprylic Acid (C8)         | 5.01       |
| Capric Acid (C10)          | 6.57       |
| Lauric Acid (C12)          | 34.49      |
| Myristic Acid (C14)        | 20.97      |
| Palmitic Acid (C16-0)      | 13.67      |
| Stearate (C18-0)           | 5.73       |
| Linoleic Acid (C18-2)      | 3.16       |
| Oleic Acid (C18-1)         | 10.41      |

From Table 4, it can be seen that the results of the transesterification of the fatty acid methyl ester composition test using GC-MS produce 8 (eight) compounds with the main compound being lauric acid methyl ester. This is in line with the results of research which explains that the composition of the transesterified fatty acid methyl ester from VCO produces 8 (eight) compounds with the main compound is lauric acid [15].
3. 3 Diethanolamide Surfactant

The yield of diethanolamide surfactant obtained from the amidation process is by reacting methyl ester (fixed amount) and diethanolamine with several ratios (1: 1,25; 1: 1,5; 1: 1,75 and 1: 2) using a NaOH catalyst 0.25, 0.5, 0.75% can be seen in Table 5.

From Table 5, it can be seen that the yield of DEA surfactant increases with increasing concentration of NaOH catalyst. The average yields of DEA surfactants using 0.25%, 0.5% and 0.75% NaOH catalyst were 93.6%, 94.52% and 94.60%. NaOH catalyst is a homogeneous catalyst, which has the same phase as the reactants. The function of this catalyst is to break the bonds in the methyl ester to make the reaction easier. If the concentration is too low, the possibility of the reaction progressing also slower.

Treatment ratio of methyl ester and diethanolamine were 1: 1,25; 1: 1,5; 1: 1,75 and 1: 2, and yielded yields: 93.51%, 94.70%, 94.18% and 94.58%. This means that the highest yield was obtained in the ratio of methyl ester and diethanolamine 1: 1.5.

The physical properties of the surfactants can be seen in Table 6. It can be seen that the DEA surfactants produced by comparison treatment of methyl ester and diethanolamine produce DEA surfactants with the same consistency, namely semi-solid viscous liquid with clear yellow color. Likewise for the treatment of using 0.25, 0.5 and 0.75 NaOH catalysts produced DEA surfactants with the same consistency, namely semi-solid viscous liquid with a clear yellow color.

| Ratio of Methyl Ester and Diethanolamine (A) | Concentration of NaOH (B) | Yield (%) |
|---------------------------------------------|---------------------------|-----------|
| (1) 1:1.25                                  | (1) 0.25 %                | 92.80     |
| (2) 1:1.5                                   | 95.20                     |
| (3) 1:1.75                                  | 93.07                     |
| (4) 1:2                                     | 93.33                     |
| Average B (1)                               |                           | 93.60     |
| (1) 1:1.25                                  | (2) 0.5 %                 | 94.40     |
| (2) 1:1.5                                   | 93.70                     |
| (3) 1:1.75                                  | 94.80                     |
| (4) 1:2                                     | 95.20                     |
| Average B (2)                               |                           | 94.52     |
| (1) 1:1.25                                  | (3) 0.75 %                | 93.33     |
| (2) 1:1.5                                   | 95.20                     |
| (3) 1:1.75                                  | 94.67                     |
| (4) 1:2                                     | 95.20                     |
| Average B (3)                               |                           | 94.60     |
| Average A (1)                               |                           | 93.51     |
| Average A (2)                               |                           | 94.7      |
| Average A (3)                               |                           | 94.18     |
| Average A (4)                               |                           | 94.58     |
Table 6. Observation Results of the Consistency and Appearance of DEA Surfactants on Several Comparisons of ME and Diethanolamine Treatment

| Comparison of ME and Diethanolamine | Consistency                  | Color / appearance   |
|-------------------------------------|------------------------------|----------------------|
| 1:1.25                              | Semi solid / viscous liquid | Clear yellow         |
| 1:5                                 | Semi solid / viscous liquid | Clear yellow         |
| 1:1.75                              | Semi solid / viscous liquid | Clear yellow         |
| 1:2                                 | Semi solid / viscous liquid | Clear yellow         |

Use of a NaOH catalyst (%)

| 0.25 | Semi solid / viscous liquid | Clear yellow         |
| 0.5  | Semi solid / viscous liquid | Clear yellow         |
| 0.75 | Semi solid / viscous liquid | Clear yellow         |
| 1.0  | Solid                       | A little light brown |

The use of 1.0% NaOH catalyst produces DEA surfactant with a solid consistency and a slightly light brown color. This means that the use of 1.0% NaOH catalyst is not recommended because it produces DEA surfactants with a consistency and color that does not match the appearance standards (consistency and color) of DEA surfactants on the market.

The results of chemical testing of DEA surfactants and the average test results with catalyst concentration treatment can be seen in Table 7. It can be seen that the average viscosity of DEA surfactants decreases with the increase of NaOH catalyst from 0.25% -0.75%. namely 411.38 cP, 400.92 cP and 367.34, respectively. The average viscosity of DEA surfactants decreased with increasing amounts of dietanolamine, namely as follows. At a ratio of 1: 1.25 produces a viscosity of 440.46 cP, 1: 1.5 produces a viscosity of 423.27 cP, 1: 1.75 produces a viscosity of 358.91 cP and 1: 2 viscosity of 354.86 cP.

The degree of acidity (pH) is one of the characteristics of a surfactant. Each type of surfactant has a different pH, for example, the pH of the surfactant dietanolamide ranges between 9 and 10. In using surfactants, pH needs to be considered because it will affect the activity of the surfactants even though there are some types of surfactants that are not affected by changes in pH. All surfactant products resulting from the oxidation reaction in all treatments have a pH of 10.

The soaping number is expressed as the number (milligrams) of KOH needed to lather one gram of fat or oil. This figure represents the crude molecular weight of fats and oils. Oil which is composed of short carbon chain fatty acids means that it has a relatively small molecular weight and will have a large number of saplings. Conversely, if the oil has a large molecular weight, the saponification number is relatively small [16]. The DEA surfactants produced in this study have saponification numbers between 35.22-52.95 mg KOH / g. The higher the amount of dietanolamine used in comparison with the methyl ester results in a decreasing number of saponification levels.

Total glycerol content indicates the total amount of glycerol both in the bound state (mono-, di- and triglycerides) and in the free state in the DEA surfactant product. The total amount of glycerol present in the DEA surfactant product ranges from 0.08% -0.29%. This is because the methyl ester raw material from the transesterification process used in the amidation process contains glycerol 0.33% (see Table 7).
### Table 7. The results of the viscosity test, pH, saponification number and glycerol content of the surfactant diethanolamide

| Treatment | ME and Diethanolamine ratio (A) | Concentration of NaOH catalyst (B) | Viscosity (cP) | pH | Saponification numbers (mg KOH/g) | Glycerol levels (%) |
|-----------|---------------------------------|-----------------------------------|----------------|----|-----------------------------------|--------------------|
| (1)       | 1:1.25                          |                                   | 374.66         | 10 | 51.79                             | 0.15               |
| (2)       | 1:1.5                           | (1) 0.25 %                        | 392.92         | 10 | 50.36                             | 0.12               |
| (3)       | 1:75                            |                                   | 455.24         | 10 | 43.37                             | 0.10               |
| (4)       | 1:2                             |                                   | 422.71         | 10 | 41.76                             | 0.10               |
| Average B (1) |                                |                                   | 411.38         | 10 | 46.82                             | 0.12               |
| (1)       | 1:1.25                          |                                   | 436.83         | 10 | 46.82                             | 0.08               |
| (2)       | 1:5                             | (2) 0.5 %                         | 422.91         | 10 | 41.16                             | 0.12               |
| (3)       | 1:75                            |                                   | 354.55         | 10 | 37.25                             | 0.29               |
| (4)       | 1:2                             |                                   | 389.40         | 10 | 35.22                             | 0.19               |
| Average B (2) |                                |                                   | 400.92         | 10 | 40.11                             | 0.17               |
| (1)       | 1:1.25                          |                                   | 509.89         | 10 | 52.95                             | 0.20               |
| (2)       | 1:1.5                           | (3) 0.75 %                        | 440.06         | 10 | 38.89                             | 0.18               |
| (3)       | 1:1.75                          |                                   | 266.94         | 10 | 36.63                             | 0.22               |
| (4)       | 1:2                             |                                   | 252.47         | 10 | 35.80                             | 0.18               |
| Average B (3) |                                |                                   | 367.34         | 10 | 41.68                             | 0.20               |
| Average A (1) |                                |                                   | 440.46         | 10 | 50.52                             | 0.14               |
| Average A (2) |                                |                                   | 423.27         | 10 | 43.47                             | 0.14               |
| Average A (3) |                                |                                   | 358.91         | 10 | 39.08                             | 0.20               |
| Average A (4) |                                |                                   | 354.86         | 10 | 37.59                             | 0.16               |

### 4. Conclusion

The phenolic compounds extracted VCO produced DEA surfactant and glycerol by-products. The optimal esterification time in making methyl ester (purified) with the highest yield is esterification time of 1.5 hours using 1.25% NaOH catalyst, with an average yield of 57.92%, and 0.5 hours of esterification time using KOH catalyst 1% with an average yield of 74.69%. The appropriate ratio of reactants to produce surfactant DEA is the ratio of methyl ester and diethanolamine 1: 1.5 with yield about 94.58%, viscosity about 423.27 cP, pH about 10, Saponification number 43.47 mg KOH / g and total glycerol content 0.14%.

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