Novel Approach of Biodiesel Production Waste Utilization to Support Circular Economy in Biodiesel Industry

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Abstract. Current development of industry nowadays is more towards sustainability and greener production that minimize the production of waste, for example the production of biodiesel energy from Refined, Bleached, and Deodorized Palm Oil (RBDPO). However, biodiesel production is still could not eliminate the hazardous waste that is glycerin pitch. Glycerin pitch is a waste found in the glycerin purification unit which is a unit used to produce the high-grade glycerin, a side product of biodiesel production. Up until now, the existing glycerin pitch is still not well managed and remains as a hazardous waste. Glycerin pitch contains a sodium salt that can be recovered to produce biodiesel transesterification catalyst, sodium methoxide. Applying a concept of circular economy, a catalyst recovery building block is attached to integrated biodiesel process with glycerol purification. The process consists of three units, solid-liquid extraction to separate inorganic salt contains in the pitch, Chlor-Alkali process to convert inorganic sodium salt to sodium hydroxide, and catalyst making reactor to produce a sodium methoxide. Using a simulation process, the amount of catalyst recovered in the process is 43.78%.

Keywords: circular economy, glycerin pitch, novel approach, process recovery, sodium methoxide

1. Introduction

Indonesia has a rapid growth of crude palm oil production in the last five years, with the number of 47 million tons in 2019 [1]. Domestic consumption only takes 32.3% of its total cumulative production (crude palm oil and palm kernel oil), with the highest of utilization after food industry is for biodiesel making [1]. Biodiesel is an alternative energy that has similar chemical and physical properties to petroleum diesel so that it can be used directly in diesel engines or mixed with petroleum diesel [2]. In Indonesia, biodiesel is mixed with petroleum diesel in certain composition and widely used as a transportation fuel. As an alternative fuel, biodiesel has many advantages: it is derived from a renewable, domestic resource, it is biodegradable and non-toxic, also by utilizing biodiesel, we slowly relieving reliance on petroleum fuel imports [3]. Compared to petroleum fuel, biodiesel has more preferable combustion emission profile, such as low emission of carbon monoxide, particulate matter, and
unburned hydrocarbons [4]. Carbon dioxide produced by combustion of biodiesel can be recycled by photosynthesis, thereby minimizing the greenhouse effect [5].

With B-30 policy implemented in January 2020, which requires 30% biodiesel composition in fuel, the need for biodiesel in Indonesia is increasing [6]. Biodiesel is produced using crude palm oil as raw material through transesterification reaction with methanol catalyzed with acid or base catalyst [7]. Crude glycerol, the by-product in biodiesel production, has percentage about 10% wt of the biodiesel product and still contains about 20% of impurity in it [8]. Using distillation, crude glycerol can be purified to produce glycerin, which commonly has more than 95% purity [9]. Process of glycerol purification generate waste called glycine pitch, which is found at the bottom product in distillation unit [10]. By mid of 2019, it is found that glycine production capacity is 0.88 million tons in Indonesia [11], with the current production of 150,000 tons/year. Anticipating the B-30 policy, glycine production can be increased as high as 500,000 tons/year, thus resulting much higher waste.

Glycerin pitch is a viscous alkaline gel that has brown to dark brown appearance containing non-glycerol organic compound and has a high ash/mineral content [12]. Those compounds exist in various length of carbon chains, from short chain such as leftover methanol until a long fatty acid chain. Soaps and residual oil also found in glycine pitch. Metal catalyst during the biofuel making also being carried over through the whole process of glycine purification and is contained in the pitch.

Government regulation in Indonesia categorized this waste as a hazardous waste that requires special treatment to remove. Industrial practice in dealing glycine pitch is by sending it to a third party for treatment, which requires fee to pay this service. Considering the possibility of increasing glycine production, the amount of fund required to treat glycine pitch waste will be much higher. A better alternative is needed as a solution to this matter. As stated in Government Regulation no. 101 of 2014, the first act of hazardous waste management is to reduce the waste by using material substitution, process modification, or the application of environmentally friendly technology. The next step is to reuse and recycle the waste, which in the case of hazardous waste can be tricky and needs a more pre-treatment process.

Waste management is a sustainable process which will be beneficial to the environment. However, the acts described above is not enough, since there is also a possibility to utilize the waste. Hence comes the circular economy. Circular economy is a further concept beyond recycling, which is to treat the waste produced as a new resource in a particular process [13]. This concept will drive people forward to create a new innovation that can utilize unwanted waste becomes a new product that is beneficial and has an added value. Circular economy concept should be applied within the whole process, in this case is from biodiesel making to glycine purification. The present study elaborates alternative pathway to utilize the glycine pitch. It is expected that the proposed process pathway may reduce the amount of waste produced and promote the circular economy in biodiesel industries.

2. Biodiesel process

Biodiesel is an alternative energy source that is now widely used to replace fossil diesel. There are currently four generation of biodiesel that are distinguished by raw materials: (a) first generation biodiesel, which use edible oils; (b) second generation biodiesel, produced from non-edible oils; (c) third generation biodiesel which utilize waste oils as raw material; and (d) fourth generation, which is a newest development of biodiesel making that the feedstocks are advance solar oils, including photobiological solar fuels and electro-fuels, using a synthetic biology technologies [14]. Biodiesel that is currently available in the market are the first-generation biodiesel. In Indonesia, biodiesel making commonly needs a Refined, Bleached, Deodorized Palm Oil (RBDPO), which is categorized as an edible oil. RBDPO, which is a product of palm oil product, is more preferrable rather than using raw palm oil, because of its low free fatty acid (FFA) which can produce more biodiesel [15]. Raw palm oil also contains 1% of useful materials that becomes impurity in biodiesel production, such as carotenoids, vitamin E, sterols, phospholipids, glycolipids, thus the process of refining, bleaching, and deodorizing is carried out [16].
The main process in biodiesel making is transesterification, the reaction between fatty acids or fatty oils with alcohol in the presence of catalyst [17]. During the reaction, triglycerides convert into monoglycerides and then diglycerides and finally glycerol and one molecule of ester is produced at each step of the reaction [18]. Based on the reaction, the stoichiometric ratio of alcohol and triglycerides of the reaction is 3:1. However, in industrial practice, this ratio is increased to 6:1 with excess alcohol so that the equilibrium of the reaction is always toward the product. The high content of free fatty acids and water from raw oil material is a problem faced by the transesterification process. Water reacts with catalyst therefore the yield of the reaction will decrease.

Although there are various catalysts in biodiesel making (homogenous, heterogenous, acid, basic, or enzyme catalysts), homogenous basic catalysts are the most prevalent, such as sodium methoxide, potassium methoxide, sodium hydroxide and potassium hydroxide [19]. Between those four, sodium methoxide (CH₃ONa) catalyst is the most active one that can induced a better process separation after the reaction, thus the use of sodium methoxide is more commonly found in industry [20]. About 70% of biodiesel industry in North America used this catalyst. All major technology providers including Lurgi PSI, Desmet Ballestra and Crown Iron Works offer biodiesel process technologies and plant designs that utilize sodium methoxide [21]. This type of catalyst is also widely used in Indonesian biodiesel industries. Another parameter that plays an important role in the yield of biodiesel is temperature of transesterification [15]. A temperature cannot be too low nor too high: should be optimum where the time needed to convert triglycerides is short with a high amount of conversion. Optimal temperature falls in the range of 60-70ºC in the use of homogenous basic catalyst with 6:1 ratio of alcohol and triglycerides [22].

![Figure 1. Transesterification reaction of triglycerides and alcohol [23].](image)

The process of biodiesel production in industry starts from fresh RBDPO that is rich in triglycerides entering transesterification reactor to conduct the transesterification reaction in the presence of homogenous catalyst and methanol. Once transesterification finished at the specified time, two different liquid phases will form in the reactor, which the oil phase mostly contains fatty acid methyl ester (FAME) and water phase is rich in glycerol and excess methanol. Due to the mixing, those liquid phases will be seen as one phase, thus, separation is needed. This mixture is transferred into a separating funnel. Separation is carried out until two distinct layers of the product is achieved. Glycerol as a by-product is
formed in the bottom layer, mixed with the excess methanol, and is associated with a higher density than fatty acid methyl esters (FAMEs) in the upper layer [24].

Through some stage of purification, FAMEs, methanol, and crude glycerol is separated [25]. To purify the upper layer that contain methyl ester from the separator, it is usually washed by water and then distilled to produce high purity biodiesel. The glycerol produced from the transesterification process is known as crude glycerol, which still contains components other than glycerol such as methanol, fatty acid compounds, soap, and others. So that the refining process need to be carried out to get pure glycerol, which is valuable, that can be used in industry. The purification process of crude glycerol or known as sweet water begin with adding solution of acid into crude glycerol to decompose the soap and form free fatty acids [26]. This process is also called neutralization or acidification because there is an addition of acid to the crude glycerol compound. Methanol that has been purified can be reused in the process, and crude glycerol is distilled to produce a pure glycerin.

Before carrying out the acidification process, the crude glycerol stream introdused into the evaporation column to separate methanol from crude glycerol. After it separated, the output methanol stream was fed back to the feed stream, making use of the recovered methanol. During the glycerol formation process, triglycerides react with alcohol with the help of an alkaline catalyst. So that the glycerol produced as a by-product of this transesterification reaction is alkaline. The acidification process which has been mentioned above is then took place. The purpose of this acidification process is to neutralize the glycerol from the alkaline catalyst that is still contained therein. In this process, acid solution such as hydrochloric acid, carbonic acid or sulfuric acid with certain concentration and amount is added to crude glycerol solution. In this study, hydrochloric acid which acted as neutralizing solution was used. Crude glycerol that has been acidified still contains water, so it is necessary to separate it to obtain pure glycerol. The products obtained after glycerol purification are pharma grade glycerol and glycerin pitch. The glycerin pitch that obtained from biodiesel production contains several components such as water, matter organic non-glycerol (MONG), and salt.

![Block flow diagram of biodiesel production and glycerol purification](image)

**Figure 2.** Block flow diagram of biodiesel production and glycerol purification.
The mass balance calculation of the biodiesel production process is carried out using a simulator, Aspen Plus v.10. The design basis is to produce 1 ton/h biodiesel from the transesterification reaction of palm oil and methanol which is catalyzed by sodium methoxide. Fresh methanol which has been mixed with recycled methanol, fed to the reactor with a molar to oil ratio of 6. The catalyst used in the transesterification reaction is added and mixed with fresh methanol in a mixing tank with a quantity of 1%-wt. from the amount of oil treated [27]. The residual methanol from the reactor fed into the methanol purification tower, then the pure methanol product recycled to be mixed with fresh methanol as feed for the reaction. Based on the simulation and assumption, to produce 1 ton/hour of biodiesel, feed of 1042.5 kg/h RBDPO and 119.97 kg/h fresh methanol is required. In addition, to obtain pure glycerol by-products (107 kg/h), 7.2 kg/h solution of hydrochloric acid fed in neutralization reactor. The waste produced from the whole process is glycerin pitch with a rate of 61.1 kg/h which contained matter organic non-glycerol (MONG) and sodium chloride salt.

### Table 1. Fatty acid composition of RBDPO used in simulation [28].

| Fatty acid | Composition (%) |
|-----------|-----------------|
| Saturated |                 |
| Myristic  | 0.92            |
| Palmitic  | 46.3            |
| Stearic   | 3.52            |
| Total     | 50.74           |
| Unsaturated |              |
| Olein     | 39.58           |
| Linoleic  | 9.68            |
| Total     | 49.26           |

### 3. Glycerin Pitch Treatment
Glycerin pitch from the process contains some materials, such as fatty acid, glycerol, methanol, and inorganic salts. Sodium methoxide and sodium hydroxide are feed to the process in the step of transesterification of biodiesel and neutralization of glycerol, respectively. Thus, inorganic matter in glycerin pitch is none other than sodium salt. It is proven in the simulation that glycerin pitch contains 19% of inorganic salt in the form of sodium chloride. Sodium chloride is formed after the sodium from neutralization phase reacted with chlorine atom from the acidification. In the industry, however, the percentage of each component differs based on the process parameters carried out. Typically, inorganic salt contains in glycerin pitch is about 10% or under 5% in crude glycerol after neutralization phase [8, 29]. This value might seem insignificant but anticipating the increase in glycerin production of 500,000 tons/year, the amount of inorganic salts that can be extracted will also as large as 40,000 tons/year.

### Table 2. Glycerin pitch composition.

| Component | Typical range** | Simulation output |
|-----------|-----------------|-------------------|
| Glycerol  | 0-30            | 5                 |
| Water     | < 2             | < 1               |
| Ash       | 20-60           | 19                |
| MONG*     | 40-60           | 75                |

* MONG = matter organic non-glycerol  
** Typical range obtained from Apolin, 2019

Liquid extraction process can be carried out to separate inorganic salt from glycerin pitch. A study proved that liquid-solid extraction can be used to recover 87.59% of inorganic salt with methanol as solvent with solute mass: solvent volume ratio of 1:5 in the operation temperature of 70°C [8]. As the
continuation of the process in Fig 2, an extraction vessel is installed to extract inorganic salts in form on sodium chloride from glycerin pitch using methanol as a solvent. The amount of inorganic salt that can be recovered from the process with a basis of 1 ton/h biodiesel is 9.87 kg/hr.

Table 3. Mass balance of solid-liquid extraction.

| Component          | Glycerin pitch | Solvent | Raffinate | Extract |
|--------------------|----------------|---------|-----------|---------|
| Mass flowrate (kg/hr) | 61.10          | 9787.60 | 9834.1    | 11.28   |
| organic fraction   | 0.815          | 0.005   | 0.995     | 0.876   |
| inorganic fraction | 0.185          |         | 0.124     | 0.124   |

The continuation of solid-liquid extraction is how to convert sodium chloride (NaCl) into sodium hydroxide (NaOH). Sodium hydroxide is the material that is needed to produce catalyst that is used in biodiesel transesterification process. Reaction between sodium hydroxide and methanol will be resulting in sodium methoxide and water. One of the process that can be used to convert sodium chloride into sodium hydroxide is using Chlor-Alkali process [30]. This process has been established and widely used in industry, since caustic soda or sodium hydroxide is one of the main components in FMCG industry. Chlor-alkali process carried out using electrolysis process in either mercury cell, diaphragm cell, or membrane cell [31]. Industry nowadays tend to use the membrane cell because of safety consideration and power consumption [30]. A brine with concentration of 26% wt. will enter the cell and electrolysis process will be carried out. Reaction resulting in sodium hydroxide occurs in the anode side of the reactor, alongside with the production of chlorine gases. Hydrogen gases also formed in the cathode side of the reactor. The product stream, sodium hydroxide, comes out from the reactor having a concentration of 32%. The overall reaction has 50% of conversion [32]. An evaporator is installed after the reactor to eliminate the water content in the NaOH until 50% wt.

Figure 3. Chlor-alkali process for NaOH making.
Anode:
\[ 2 \text{NaCl (aq)} + 2 \text{H}_2\text{O (l)} \rightarrow 2 \text{NaOH (aq)} + \text{Cl}_2 (g) + 2 \text{H}^+ + 2 \text{e}^- \]

Cathode:
\[ 2 \text{H}^+ + 2 \text{e}^- \rightarrow \text{H}_2 (g) \]

Overall reaction:
\[ 2\text{NaCl (aq)} + 2\text{H}_2\text{O (l)} \rightarrow \text{Cl}_2 (g) + \text{H}_2 (g) + 2\text{NaOH (aq)} \]

**Figure 4.** Reactions take place in electrolysis reactor.

| stream/ component | NaCl (aq, conc) | used salt solution | NaOH (aq) 32% | Cl\(_2\) | H\(_2\) | NaOH (aq) 50% |
|-------------------|-----------------|--------------------|---------------|---------|---------|---------------|
| Mass flow (kg/hr) | 37.99           | 36.99              | 13.93         | 2.99    | 0.08    | 6.75          |
| NaCl (% wt.)      | 26.0            | 24.0               | 32.0          |         |         |               |
| H\(_2\)O (% wt.)  | 70.3            | 72.2               | 68.0          |         |         | 50            |
| NaOH (% wt.)      |                 |                    |               |         |         | 50            |
| Cl\(_2\) (% wt.)  |                 |                    |               | 100     |         |               |
| H\(_2\) (% wt.)   |                 |                    |               |         | 100     |               |
| impurities        | 3.7             | 3.8                |               |         |         |               |

**Table 4.** Mass balance of Chlor-Alkali process.

The last process that is conducted is the catalyst making. The reaction takes place in a reactor with a process temperature of 160°C and pressure 3 bar with 98.3% reaction conversion [33]. Feed used are methanol (CH\(_3\)OH) and sodium hydroxide obtained from the previous process. The aim of this process is to produce a 20% wt. sodium methoxide catalyst in methanol that used in the biodiesel transesterification reaction. The simplified process is shown in the Figure 5 while the mass balance is in Table 5. Later on, a purification process can be conducted to separate unconverted NaOH and water.

**Figure 5.** Transesterification catalyst making process.

| stream/ component | NaOH | Methanol | Catalyst |
|-------------------|------|----------|----------|
| Mass flow (kg/hr) | 6.75 | 22.41    | 27.64    |
| NaOH (% wt.)      | 50.00| 10.62    | 12.62    |
| CH\(_3\)OH (% wt.)| 95.00| 61.58    |          |
| CH\(_3\)ONa (% wt.)| 50.00| 12.42    |          |
| H\(_2\)O (% wt.)  | 5.00 | 15.38    |          |

**Table 5.** Mass balance of catalyst making.
4. Circular Economy Concept
Circular economy is a further concept beyond recycling, which in to treat the waste produced in the process as a new resource in particular process [14]. This concept will drive people forward to create an innovation that can utilize unwanted waste becomes a new product that is beneficial and has an added value. In biodiesel making, the process consists of two different main blocks, that are methyl-ester production from RBD Palm Oil and glycerol purification to obtain a high purity glycerin. This process generates a glycerin pitch waste that contains a sodium salt, component of transesterification catalyst. Attaching a recovery process of sodium salt, a new and fresh sodium methoxide can be produced. Some of the methanol that recovered from biodiesel production also can be used as a feed to the downstream process. Sodium methoxide recovered can be re-used in the transesterification thus reducing the needs of fresh sodium methoxide.

However, sodium catalyst recovery process can only recover about 43.78% of the catalyst. This low recovery is mainly because the Chlor-Alkali process that only has 50% conversion. Another thing that is become concern is the amount of methanol used in this system is quite high, 9813 kg/h. This due to the fact that solid-liquid extraction process requires a high ratio of methanol as solvent vs solute. Another thing to consider is that the process of Chlor-Alkali requires a high amount of energy, 3 MWh per ton caustic produced [34]. Comparing the cost of production in catalyst recovery to the case if there is no catalyst recovery in Fig. 2, the latter one has a higher profit. Thus, to apply a circular economy concept in biodiesel production, a better approach should be proposed rather than recycling a sodium-based salt that is abundant in the market with an affordable price.

![Image of biodiesel production process]

**Figure 6.** Circular economy concept.

5. Conclusion
Using the concept of circular economy, a catalyst recovery process can be attached to biodiesel and glycerin purification process to recycle glycerin pitch to recover the sodium salt. However, this concept has some concerns that need to take into an account before integrating in the real life.
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