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Screening of Biodiesel Production from Waste Tuna Oil (*Thunnus* sp.), Seaweed *Kappaphycus alvarezii* and *Gracilaria* sp.

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Abstract. Biodiesel has several advantages over solar. Compared to solar, biodiesel has more eco-friendly characteristic and produces lower greenhouse gas emissions. Biodiesel that is made from animal fats can be produced from fish oil, while other alternative sources from vegetable oils are seaweed *Kappaphycus alvarezii* and *Gracilaria* sp. Waste tuna oil (*Thunnus* sp.) in Indonesia is commonly a side product of tuna canning industries known as tuna precook oil; on the other hand, seaweed *Gracilaria* sp. and *Kappaphycus alvarezii* are commonly found in Indonesia's seas. Seaweed waste that was used in the present study was 100 kg and in wet condition, and the waste oil was 10 liter. The seaweed was extracted with soxhletation method that used n-hexane as the solvent. To produce biodiesel, trans esterification was performed on the seaweed oil that was obtained from the soxhletation process and waste tuna oil. Biodiesel manufactured from seaweed *K. alvarezii* obtained the best score in flash point, freezing point, and viscosity test. However, according to level of manufacturing efficiency, biodiesel from waste tuna oil is more efficient and relatively easier compared to biodiesel from waste *K. alvarezii* and *Gracilaria* sp.

INTRODUCTION

Petroleum is one of the major energy sources used in many different aspects of our daily life. As the number of motor vehicle increases, so does the consumption of fuel oil [1]. This is followed by the earth's dwindling petroleum supplies as the result of continuous consumption [2]. Over the last decade (2002 - 2012), the petroleum reserves underwent a drop of 92.43 barrel. If there is no effort attempted, this downward trend can deteriorate further, putting the government in difficult situation to fulfil its target production of petroleum at 1.01 million barrel per year [3]. The rising consumption of world's energy and the depletion of fossil fuel reserves as many as 2-3% per year since 2010 can potentially lead to scarcity of fossil fuels in years to come. This energy crisis occurs due to dependence on only one kind of energy resources. The biggest consumption of petroleum lies in diesel or solar [1] with total consumption of 173.134 thousand barrel in 2009, and it continued to increase to 174.669 thousand barrel in 2010 [3]. The need for solar is highly likely to rise in conjunction with the population growth and economic activity volume. To fulfil the increasing need, it is necessary to improve national refinery capacity. The attempt to boost refinery capacity is not an easy thing to do within relatively short time span because refinery is a capital-intensive investment [4].

Indonesia is one of the fossil fuel producers in the world, yet the country is still importing petroleum to meet the need of petroleum in transportation and energy sectors. Indonesia has changed its status from exporter to net importer of petroleum in 2005 with deficit standing at approximately 100 million liter [5]. In the long run, the

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petroleum import will dominate the supply of national energy if no government's policy is enacted to diversify energy sources by taking advantage of renewable sources and others. Using renewable resource is an alternative to fossil fuels that uses biodiesel to replace solar [6].

Biodiesel has several advantages compared to solar; it is more eco-friendly and releases less greenhouse-gas emissions. Biodiesel has several following characteristics: reduction of carbon dioxide emissions by 100%, reduction of carbon monoxide by 10-50%, reduction of hydrocarbon emissions by 10-50%, reduction of polycyclic aromatic hydrocarbons (PAH) especially the hazardous ones [5]. Biodiesel can be manufactured from vegetable oils and animal fats [7], which are renewable resources. Biodiesel from animal fats can be produced from fish oil. Fish oil in Indonesia is commonly a side product of tuna canning industries known as tuna precook oil. Tuna precook oil is usually sold with cheap price to paint industries, wood coating industries, animal feed industries; it is even left unused. Another resource widely found in Indonesia's seas is macro algae *Gracilaria* sp. It is a species of seaweed that can live in either estuary or fish farm with its original habitat in sea [8]. According to [9], the distribution area of seaweed *Gracilaria* in Indonesia reaches 255 km^2. *Gracilaria* is a kind of seaweed that can be cultivated easily with harvesting time around 2-2.5 months and with net production of 1500-2000 kg dried seaweed/acre [10]. According to [11], *Gracilaria* is cultivated by many farmers because of its cheap cost of fish seeds, availability, and easy rearing. *Kappaphycus alvarezii* is a seaweed that is cultivated by many people and widely distributed in almost all Indonesia's seas. Many biodiesel research studies have investigated biodiesel production from microalgae and, accordingly, there is a paucity of biodiesel studies on macro algae or seaweed as raw material for biodiesel production [2

The present study was carried out to understand the efficiency comparison of biodiesel from different materials: biodiesel from vegetable waste (seaweed *Gracilaria* sp. and *K. alvarezii*), biodiesel from fish waste (waste tuna oil), biosolar and combination of biodiesel from seaweed or from waste tuna fish with biosolar. Biodiesel efficiency can be known by conducting flash point, freezing point, and viscosity test.

MATERIALS AND METHODS

The equipment used in the present study was grinder machine and blender to grind seaweed, soxhlet, flat bottom to do extraction with the methods of soxhletation, beaker glass to take and measure solvent that would be used, funnel to put liquid n-hexane into soxhlet, magnetic stirer to perform homogeneity when esterification process took place, heater to heat during the trans esterification process as well as to vaporize n-hexane, pipette to separate ester and glycerol that would be mixed, thermometer to measure temperature on reaction, volumetric pipette to take biodiesel and bio solar that would be mixed, pH meter to measure the last pH of ester, scale to weigh the used materials, firelighter, timer, wick, bunsen, freezer, test tube and test tube shelf to perform biodiesel efficiency test. The materials used in the present study were waste seaweed *Gracilaria* and *Kappaphycus*, waste tuna oil, n-hexane, KOH, methanol, filter paper, thread, and pH universal.

Method of study is used to solve a problem that can be done by collecting data from observation, survey, or test [12]. The method used in the present study was experimental method. The present study was done by observing and comparing biodiesel efficiency with flash point, freezing point, and biodiesel viscosity as the tested parameters.

The experimental design used in the present study was completely randomized design. It consists of 13 groups of treatment and three replications. The detail explanation of treatment that was used in the present study is as follows: P0 : biosolar 100%; P1 : biodiesel waste seaweed *Gracilaria* 100 %; P2 : biodiesel waste seaweed *Gracilaria* 75% and biosolar 25%; P3 : biodiesel waste seaweed *Gracilaria* 50% and biosolar 50%; P4 : biodiesel waste seaweed *Gracilaria* 25% and biosolar 75%; P5 : biodiesel waste seaweed *Kappaphycus* 100%; P6 : biodiesel waste seaweed *Kappaphycus* 75% and biosolar 25%; P7 : biodiesel waste seaweed *Kappaphycus* 50% and biosolar 50%; P8 : biodiesel waste seaweed *Kappaphycus* 25% and biosolar 75%; P9 : biodiesel waste tuna oil 100%; P10 : biodiesel waste tuna oil 75% and biosolar 25%; P11 : biodiesel waste tuna oil 50% and biosolar 50%; P12 : biodiesel waste tuna oil 25% and biosolar 75%;

As many as 100 kg waste seaweed was washed in fresh water to remove dirt and sands attached to seaweed until it was free from unwanted materials. The clean seaweed was dried in the sun for about 10 days. Then, the waste seaweed was ground with grinder machine and blender until it became flakes in order to extract the oil. Waste tuna oil was obtained from side product of tuna canning industry known as tuna precook oil. The oil extraction process was done with soxhletation method that used n-hexane solvent. Seaweed waste, that would be extracted, was weighted up to 45 gr dried seaweed for each tube. After that, the dried seaweed was wrapped with filter paper tightened with thread. The seaweed in filter paper was transferred to soxhlet tube. The soxhlet process of seaweed to extract the oil required 250 ml of n-hexane as the solvent, and it was placed in the flat bottom. Then, soxhlet machine was turned on, and the machine temperature was adjusted to the used solvent temperature. For n-hexane solvent, the temperature used was 82°C. During the extraction process, the liquid's colour of n-hexane would turn into green as the result of extraction. Extraction process was marked with the liquid of n-hexane that was dripping in the soxhlet tube until its colour changed into transparent. Afterwards, tha n-hexane solvent was vaporized on the heater so that the oil, the result of waste seaweed extraction, was obtained. The same procedure was also applied to the waste tuna oil (*Thunnus* sp.), where the obtained waste tuna oil was filtered, and continued to the soxhletation process with n-hexane solvent. The obtained oil from waste seaweed and tuna was put in the beaker glass and heated with heater. In the transesterification process, the produced oil liquid was added with 40 % methanol liquid, which had been mixed with KOH 2% as the catalyst. During the heating, the temperature was maintained at around 55-60°C for 60 min. Consequently, the main product ester on the upper side and the side product glycerol on the bottom part of ester appeared. The side product was, then, separated from ester so that the main product for biodiesel was taken.

Biodiesel efficiency test could be proven with flash point, freezing point, and viscosity test. The initial testing was conducted with the help of nine panellists, followed by laboratory test. The panellist flash point test was done by placing 5 ml of sample in each petri dish with wick. Every wick in Bunsen was lighted with firelighter, and the time of which wick that ignited first was counted. Freezing point test was carried out by inserting 3 ml of sample into test tube and storing it in the freezer. Then, an observation was made on how long it took to reach freezing point at temperature (-18°C). Viscosity test was performed by counting the flow time from each treatment put in the titration instrument. Laboratory analysis was also done in the laboratory of fuel and combustion owned by mechanical engineering department, Faculty of Industrial Technology, Institute of Technology Sepuluh November, Surabaya.

RESULTS AND DISCUSION

The test parameters used to discover the efficiency of biodiesel used are flash point, freezing point, and viscosity. From the test result, it is known that the one which had the highest flash point from *Gracilaria* sp. is P5, biodiesel seaweed *K. alvarezii* 100 % was in accordance with the panelist test result score of 5.00. P5 produced blue flame with longer combustion time compared to other treatments. The statistic test result with Friedman test showed lower significant score from 0.05 ($P<0.05$) with ranking average score among treatments. Interestingly, P0 with biosolar 100% indicated a result with the lowest flash point at 77.4°C and along with the panelist score of 3.00. According to the panelist test result, the one that had the highest freezing point is P0 biosolar 100%, and the one with the lowest point is P5 biodiesel *K. alvarezii* 100 %. This indicates that biodiesel *K. alvarezii* 100 % can freeze easily at the temperature -2.6°C in storage place condition with low temperature. From the panelist test result, it is noticed that the one with the highest viscosity from biodiesel *Gracilaria* is P5, is biodiesel *K. alvarezii* 100 % with laboratory rest result of 3.75 cSt, while the lowest viscosity belongs to P0 Biosolar 100% with the score of 3,45 cSt. According to Indonesian National Standard/*Standart Nasional Indonesia* (SNI), the minimum flash point for biodiesel is 100°C and 60°C for biosolar, while the viscosity score must be in the range of 2.3-6.0 cSt for biodiesel and 2.0-5.0 cSt for biosolar. Referring to the laboratory test result on flash point, freezing point, and viscosity, biodiesel *K. alvarezii* 100 %, biodiesel *K. alvarezii* 75% and biosolar 25%, biodiesel *Gracilaria* sp. 100% as well as biodiesel waste tuna oil 100% fulfilled the SNI requirement for flash point > 100°C. Interestingly, all treatments carried out in the present study fulfilled the flash point standard as biosolar and viscosity value of biodiesel and biosolar. The same result was also discovered in pH measurement result where normal or neutral score of pH 7 was obtained in all treatments of biodiesel.

Flash point is a measure that indicates the lowest temperature at which the surface of fuel will flash upon application of ignition source [13]. In the combination treatment of biosolar, each treatment has flash point that has fulfilled the biosolar standard issued by PT. Pertamina (Persero) in 2007, an Indonesian state-owned oil and natural gas corporation, stating that the minimum flash point of biosolar fuel is 60°C [4]. The result of panelist and laboratory tests showed that the volatility level is lower and flash point temperature is higher when the time needed for combustion is longer. Flash point indicates volatility level and biodiesel's ability to combust because of a fuel [14]. Low volatility is property of biodiesel that can reduce combustion speed, resulting in high flash point [15]. Flash point is also affected by respective fuel viscosity. Low viscosity will lead to poor atomization [16]. Extremely high flash point of fuel can cause ignition delay, while extremely low flash point can result in small explosion before the fuel goes to combustion room [17].

Based on quality, compared to biosolar, biodiesel has higher flash point which causes it to combust longer when ignited. Nevertheless, biodiesel *K. alvarezii* 100 %, biodiesel *K. alvarezii* 75% and biosolar 25%, biodiesel *Gracilaria* sp. 100% as well as biodiesel waste tuna oil 100% have better flame quality and more lasting flame than those of biosolar when ignited. It is also noticeable that biosolar has red flame mixed with black smoke, indicating significant pollution caused by biosolar combustion. This converges with the statement of [6] saying that biodiesel is more eco-friendly because it contains no Sox emissions, has low toxicity, low CO and hydrocarbon emissions [16]. Flash point is required for the safety purpose in handling oil that is prone to fire risks [13]. After seeing the flash point result of biodiesel biodiesel *K. alvarezii* 100 %, biodiesel *K. alvarezii* 75% and biosolar 25%, biodiesel *Gracilaria* sp. 100% as well as biodiesel waste tuna oil 100% which has the highest flash point, it can be said that both biodiesel are in safe limits towards fire risk during storage, handling, and transportation. Freezing point is a temperature characteristic where biodiesel turn into solid, not liquid anymore [18]. The more biosolar composition a treatment has, the lower freezing point it has. Biodiesel *K. alvarezii*, biodiesel *Gracilaria* sp. and biodiesel waste tuna oil in the present study have good freezing point characteristic because the freezing point is below 0°C, so they can be used in areas with low or cold temperature like Europe and are still safe to be used in tropical areas. In general, cloud and pour point of biodiesel is higher than solar, so is the freezing point. This can cause a problem in the use of biodiesel especially in countries that have winter. To overcome this problem, certain additives are usually added to biodiesel in order to prevent agglomeration. Crystals can appear in biodiesel in low temperature. In addition to additives, mixing biodiesel and solar is another possible alternative [19]. Viscosity is a number that indicates fluid's resistance to flow [13] or the required time to flow within certain distance [5]. The viscosity test in the present study revealed that the biodiesel in the present study fulfils the biodiesel quality requirement based on SNI-04-7182-2006, stating that viscosity of biodiesel fuel is 2,3-6,0 cSt and biosolar fuel viscocity is 2,0-5,0 cSt. The high or low score of kinematic viscosity is influenced by the thickness of the respective fuel. On thickness, catalyst type and excessive concentration in transesterification triggered soap reaction, resulting in varied biodiesel density values. With the increasing level of triglyceride conversion into methyl ester, the biodiesel density will decline because the methyl ester density is lower than triglyceride density [17]. The higher the viscosity is, the thicker and more difficult a fluid will flow [13]. The viscosity data explain the role and purpose of oil-fat conversion to ester (biodiesel), which is to reduce the viscosity so that it fulfills the diesel fuel requirements. The mixture of biodiesel fuel and biodiesel seaweed, waste tuna oil, and biosolar in various compositions induces dwindling thickness value towards biodiesel viscosity fuel. The varied biosolar composition in combination generates lower viscosity, so the fuel turns out to be more watery, enabling smoother flow during pumping/supplying process of fuels from tank to combustion room. Biodiesel viscosity is affected by the unresponsive triglyceride content towards some chemical compounds. These chemical compounds are methanol, fatty acid methyl ester compositions of biodiesel, intermediate substances such as monoglyceride and diglyceride that have quite high polarity and molecular weight [20]. Some other contributing factors are position and number of double bonds (unsaturation degree) in biodiesel as well as type of alcohol used in esterification process. The correlation between the three test parameters (flash point, freezing point, and viscosity on biodiesel) revealed that higher flash and freezing point caused higher viscosity. This is in line with [16], who stated that low fuel flow leads to poor fuel atomization. Several types of biodiesel have relatively higher viscosity and need combustion heat in order to stimulate atomization in combustion room. Higher viscosity results in poor atomization; hence, the flash point of the fuel becomes higher. According to [16], biodiesel is an ester that tends to swell polymeric materials; hence, if the 100% polymeric materials are used, they have to be replaced with other materials that are resistance to ester. Therefore, as an alternative of use, a mixing between biodiesel and solar is done, considering that many otomotives used polymeric materials as fuel pipes or leak sealer. Another biodiesel's disadvantage lies in its storage period. According to [21], biodiesel is less stable than diesel fuel, so it will break down for long term storage (six months). The arising problem of storing biodiesel for extended periods of time is hydrolytic and oxidative degradation. Biodiesel tends to have high flash point and viscosity, causing poor combustion and machine endurance [16]. Moreover, if it is used in large percentage, biodiesel has solvency characteristics. Usually sediments and crusts are formed in storage tank as the result of the solar use in diesel machine. The most common encountered problem with biodiesel's solvency is that it can dissolve these sediments and crusts. Consequently, this can clog the fuel system and the fuel filters. The solvency effect of fuels that have biodiesel mixture will plunge as the biodiesel level in fuels declines [5]. As for price comparison, the efficiency comparison of using biodiesel from seaweed and waste fish oil is very profitable. Similarly, the availability of both materials is quite plentiful, so there will not be any problem in continuous biodiesel production. Based on those several aspects, the most efficient material to be used as an alternative source in biodiesel manufacturing is seaweed *K. alvarezii* 100%. In order to suppress the production cost with seaweed material of *K. alvarezii* and to prevent competition with people's consumption needs, it is better to use seaweed *K. alvarezii* of low quality or the sorting result from processing factories of seaweed *K. alvarezii*. The flash point parameter demonstrated that the amount of exhaust emissions in the form of smock density experienced a decrease, proving that biodiesel seaweed waste is more eco-friendly than biosolar. Generally, diesel exhaust gasses contain particulates due to unclean fuel factor [22]. The components of those hazardous gasses are black smokes, unburned hydrocarbon (UHC), carbon monoxide (CO), nitrogen oxide (NO) and NO_2 commonly indicated with NO_x [23]. The high flash point, which was caused by high level of heat to ignite fire, engendered ignition and combustion delay of fuels so that fire risk is smaller. This is in accordance with the statement of [24], who explained high flash point eases managing and storing fuels because fuels need not to be stored at low temperature. Conversely, if the flash point is too low, it is more dangerous because it has higher risk during ignition; thus, it must be stored at low temperature. It was mentioned by [25] that high flash point causes fuels take longer time to combust. All previous explanations emphasize that seaweed *K. alvarezii* can become alternative fuel replacement for biosolar. The other parameter to determine biodiesel quality is viscosity. Viscosity is important to be tested because excessively high viscosity can disrupt the performance of vehicle injection apparatus. [2] also stated that viscosity plays an important role in lubrication of frictions between the moving parts and wear. The result of biodiesel production from *K. alvarezii* revealed the highest viscosity score at temperature of 40^0C is 3.75 cSt, making biodiesel *K. alvarezii* the best fuel alternative to use in diesel machine. [13] explained that the higher the viscosity is, the thicker and the more difficult a fluid flow. Furthermore, excessively high viscocity hampers carburetion process [25]. Likewise, high freezing point will cause machine not to operate at low temperature. The result of the biodiesel production from *K. alvarezii* showed the freezing point is at temperature -2.6^0C. This highlighted that biodiesel *K. alvarezii* can be considered the best biodiesel compared to *Gracilaria* and waste tuna oil in tropical regions. Freezing point is not an important indicator to determine fuel standard in tropical areas like Indonesia, but freezing point can play a significant role when this fuel is used in countries with four seasons because fuels with high freezing point need heater to maintain oil remain liquid to be easily transferred to combustion chamber. However, according to efficiency of manufacturing procedure, biodiesel waste tuna oil is more efficient than biodiesel *K. alvarezii* because waste tuna oil can be found easily in markets or tuna canning factories. It is not the same as biodiesel *K. alvarezii* where extracting the oil requires soxhletation method and takes long time in order to produce biodiesel concentration. Based on the result of biodiesel efficiency comparison, it can be concluded that biodiesel seaweed *K. alvarezii*, *Gracilaria* sp. and waste tuna oil can become biodiesel production materials. Biodiesel seaweed *K. alvarezii* scored the best in flash point, freezing point, and viscosity test. However, if both are compared in terms of efficiency level, biodiesel waste tuna oil is more efficient than biodiesel *K. alvarezii* because biodiesel manufacturing process is more obtainable from waste tuna than seaweed *K. alvarezii*. On the other hand, it is necessary to discover extraction technology that enables to manufacture biodiesel efficiently with seaweed materials.

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