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Utilization of Waste Tobacco (Nicotiana Tabacum) Post-Harvest as an Alternative Biodiesel

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ABSTRACT

The aims of this research is process into biodiesel. Biodiesel is produced using transesterification reactions with variations of tempetature. Tobacco seed oil is extracted to obtain edible oils as biodiesel. The content of free fatty acid (FFA) in edible oils should be minimized to obtain a high yield of transesterification. The catalyst used in converting edible oils into biodiesel is homogeneous alkaline catalyst. Biodiesel with the higest yield from transesterification reactions with a temperature of 70 oC. Biodiesel was characterized the functional group and phsycal properties including density and viscosity

INTRODUCTION

Jember Regency is a tobacco producing area. Tobacco cultivation in Jember district is covered by PTPN X. One of the types of tobacco cultivated is Besuki Na-Oosgst tobacco. This type of tobacco is planted and harvested in the dry season (Anastasia et al., 2014). Tobacco that has been harvested still has many parts left behind, such as seeds, leaves, shoots, and stems. Many of the parts that are left are not utilized, so the idea of using tobacco waste in the seeds arises to be used as alternative energy. Biodiesel is an alternative fuel that can be produced from a chemical reaction between vegetable oils or animal fats with short chain alcohols. The chemical reaction in question is a reaction for the formation of esters from oil or fat so that the reaction used is an esterification-transesterification reaction (Ma et al, 1999).

Based on the report of Mohamad et al (2014), that tobacco seeds contain vegetable oil that can be used as raw material for biodiesel. Biodiesel is composed of fatty acids with carbon chain lengths ranging from C-10 to C-24 (Moser, 2013). The manufacture of biodiesel in this study focused on the transesterification reaction following the method of Usta et al., (2011).

The basis for choosing this method is obtained through the statement of Issariyakul et al, (2014), which states that the transesterification reaction using methanol and a base catalyst can save energy and reaction time so that it looks easy and economical. This research focuses on the effect of temperature which is not far from the boiling point of the type of alcohol used, so that four temperature variations appear. The biodiesel produced will be identified its functional group to ensure that this biodiesel has a methyl group and an ester group. Biodiesel with the highest yield will be used as a reference to represent the fatty acid composition of its constituents as well as its physical characteristics which include density and viscosity.

RESEARCH METHOD

A. Material

The materials used in this study were: Tobacco seeds (Nicotiana tabacum) taken from PTPN X Jember, 98% methanol, distilled water, technical n-hexane, PP indicator, Merck NaOH, 96% H2SO4, Merck H2C2O4 .2H2O.

B. Equipment

The equipment used in the manufacture of biodiesel from tobacco seed oil (Nicotiana tabacum) is glassware that is often used in laboratories such as; Mohr pipette 5 ml, 10 ml, dropper, measuring flask 10 ml, 50 ml, three neck flask 250 ml, round bottom flask 150 ml, reflux condenser, thermometer 200 oC, beaker 100 ml, 250 ml, spatula, funnel, separating funnel, 50 ml burette, mortar and pestle, soxhlet tool set. Non-glass tools such as spray bottles, aluminum foil, tissue, label paper, stands and clamps, aquarium pumps, silicone hoses, aluminum vessels, and stirrer tillers. The instruments used were Analythical Plus analytical balance, oven, evaporator, heating mantle, hot plate and magnetic stirrer Lab Companion HP-3000, FTIR spectrophotometry Bruker Alpha Sample ATR eco Ge and GC-MS (gas chromatography) Zhimadzu QP2010S.

Procedure

Raw Material Preparation

Tobacco seed waste is dried and crushed in preparation for extraction. The tobacco seeds were weighed and extracted using the Soxhlet method using n-hexane with a ratio (g/v) (1:5). Tobacco seed oil is obtained by evaporating the solvent using a rotary evaporator and weighed to determine the extraction yield (Stanisavljevic et al., 2007)

Oil Esterification

Tobacco seed oil will be esterified to minimize the free fatty acid content. The oil esterification step begins with preparing a solution including loading a solution of sodium hydroxide and hydrated oxalic acid. The preparation of this solution follows the AOCS Cd 3a-63 method, in which an acid-base titration is carried out to calculate the fatty acid content with the existing equation constants. It is hoped that the results of this esterification will reduce the fatty acid content of the oil to below two percent by weight of the oil. The esterification process was carried out using methanol with a ratio (v/v) to oil of 6:1 and assisted with sulfuric acid

catalyst Pa as much as one percent by weight of oil. The result of esterification is the triglycerides that make up the oil with a lower free fatty acid content than the extracted tobacco seed oil (Srinivas et al., 2013).

Transesterification of Oil into Biodiesel

Tobacco seed oil that has been esterified with low fatty acid content can be transesterified immediately. The transesterification reaction process includes the preparation of a catalyst. The catalyst in question is a homogeneous base catalyst of sodium methoxide obtained from the reaction between sodium hydroxide and methanol (Usta et al., 2011). The calculation of the molar ratio of a homogeneous base catalyst is 6 moles of methanol and one percent sodium hydroxide which refers to the moles and weight of the oil used in the reaction. Molar ratio coefficient of homogeneous base catalyst: oil (n/n) used in the reaction is 6:1 (Veljkovic et al., 2006). Transesterification reactions were varied at temperatures (50, 60, 70, 80)oC. The result of transesterification is placed in a separating funnel where the top is glycerol and the bottom is biodiesel (methyl ester). Biodiesel was washed with distilled water three times to remove the remaining catalyst and then heated above 100oC.

Biodiesel Identification and Characterization

The biodiesel produced was then analyzed using a FITR spectrophotometer to ensure that there were methyl groups and ester groups present in the biodiesel. Biodiesel with the highest yield will be analyzed for its fatty acid composition and also its physical properties which include density and viscosity (Khasanah et al., 2009).

RESULTS AND DISCUSSION

Tobacco Seed Extract

Tobacco seed extraction is the initial stage to obtain vegetable oil as raw material for biodiesel production. Tobacco seeds were weighed as much as 40 g and put into filter paper whose shape was adjusted to the Soxhlet flask. This adjustment is because the extraction method used is the Soxhlet method. The solvent used in this extraction process is n-hexane with the use of 200 mL for each extraction. The extraction process was carried out as many times as needed and had an extraction yield of 16.41% (120 g from 736.59 g of tobacco seeds). The yield produced is relatively small because more oil should be produced if the ratio of the volume of the extraction solvent volume to the extracted sample is increased.

Oil Esterification

The extracted oil that has been produced is analyzed for the amount of fatty acid content. The purpose of the analysis of the fatty acid content is to determine whether this oil can be directly converted into biodiesel or not. The result of the analysis of the extracted oil content was 7.1% which caused the need for esterification using an acid catalyst to reduce the free fatty acid content. Calculation of free fatty acid levels using the method of AOCS Cd 3a-63. Reduction of fatty acid levels involves a reaction between methanol and triglycerides catalyzed by concentrated sulfuric acid. The results of this esterification then produce two phases which are separated by a separating funnel. The esterified oil that has been separated is then heated to remove the remaining methanol and the free fatty acid content is recalculated using the same method. The results of the calculation obtained the results of free fatty acid levels of 0.67%. The esterified oil was also analyzed using FTIR to prevent the presence of compounds that could interfere with the transesterification reaction such as water groups and others.

Transesterification of Oil into Biodiesel

Transesterification is the most common method for converting triglycerides to esters. Biodiesel is also called methyl ester. The transesterification process was carried out at several temperatures, namely (50, 60, 70, 80) oC. The effect of transesterification temperature can be studied by analyzing the biodiesel yield from each transesterification temperature. The method used in this study uses the method that has been carried out by Usta et al., (2011). The transesterification was carried out using NaOH and methanol as a base. NaOH is dissolved in methanol in a mole ratio (1:6). The purpose of base dissolution is to form

methoxide ions which greatly affect the formation of esters from triglycerides. The yield of biodiesel produced increased starting from the transesterification temperature of 50-70 oC, then the yield was reduced at a temperature of 80 oC. The highest yield was obtained from transesterification at 70 oC, which had a success rate of 47.6% with biodiesel yield of 1.77% of the total extracted oil. Theoretically, the transesterification reaction that has been carried out has followed the Arrhenius equation. In addition to the reaction kinetics, it can also be influenced by the reaction precursors used, especially methanol which has a boiling point of 64.7 oC, so that at a transesterification temperature of 80 oC the yield decreases. This reduction in yield was possible because more methanol evaporated before reacting with triglycerides. The following table presents the percentage of success of the transesterification reaction for 5 mL of oil used for each reaction:

Table 1. Percentage of transesterification and Biodiesel Yield

Temperature of Trans-esterification (°C)	Biodiesel Volume (mL)			Rendemen Trans-Esterification (%)	Rendemen Biodiesel (%)
	1	2	Average		
50	1,60	1,75	1,68	33,6	1,25
60	1,70	1,90	1,80	36,0	1,34
70	2,35	2,40	2,38	47,6	1,77
80	1,70	1,80	1,75	35,0	1,30

The percentage of transesterification is calculated by dividing the volume of biodiesel by the volume of oil used in one reaction. The following graph shows that temperature affects the transesterification reaction.

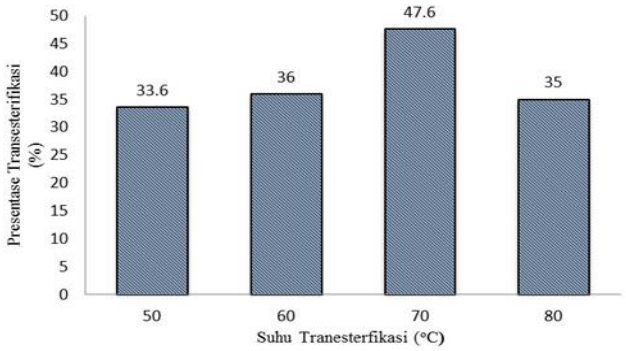

Figure 1. Graph of Transesterification Success Percentage

Biodiesel Identification and Characterization

Biodiesel identification aims to determine the fatty acid composition of the biodiesel produced. Biodiesel which is identified by its fatty acid is biodiesel with the highest yield according to Table 1. This identification uses GC-MS analysis. The results obtained from the GC-MS chromatogram show that there are five fatty acid compounds that make up this biodiesel as shown in Table 2 below:

Table 2. Fatty Acid Methyl Ester Tobacco Seed Oil

Fatty acid		Retention time (min)	Area (%)
Methyl palmitate	C16 :0	35,625	12,18
Methyl hexadenoate	C16 :2	39,150	63,90
Methyl linoleate	C18 :1	39,257	18,72
Methyl stearate	C18 :0	39,617	4,88
Methyl eicosenoate	C20 :0	43,258	0,32

The resulting biodiesel is dominated by methyl hexadenoate followed by methyl linoleate, methyl palmitate, methyl stearate, and methyl eicosenoate. The amount of methyl ester obtained is based on the area of the GC chromatogram.

The biodiesel that has been produced will then be characterized using an FTIR spectrophotometer to identify the functional groups present in biodiesel. The functional groups that can be analyzed can be used to compare the characters and types of functional groups between biodiesel and esterified oil used in the transesterification reaction. The following table 3 presents the functional groups of biodiesel and esterified oil.

Table 3. Biodiesel Functional Groups

Sample	Wavelength (cm^{-1})	Functional groups
Biodesel 50 °C	1738,34	C=O aliphatic
	1172,19	C–O aliphatic ester
	2855,94- – 2926,96	C–H aliphatic (methyl)
Biodesel 60 °C	1740,95	C=O aliphatic
	1171,44	C–O aliphatic ester
	2855,05- – 3008,05	C–H aliphatic (methyl)

Biodesel 70 °C	1742,74	C=O aliphatic
	1170,74	C−O aliphatic ester
	2854,76- – 3009,07	C−H aliphatic (methyl)
Biodesel 80 °C	1742,06	C=O aliphatic
	1170,78	C-O ester aliphatic
	2854,75- – 3008,93	C-H aliphatic (methyl)
Vegetable oil	1744,60	C=O aliphatic
	1164,44	C-O ester aliphatic
	2854,33- – 2925,06	C-H aliphatic (methyl)

The wave number that characterizes the ester group is the peak at the wave number 1750 – 1730 cm-1 which indicates the C=O group (aliphatic carbonyl). The second characteristic is the wave number of 1300 – 1100 cm-1 bonds of aliphatic C-O groups (Stuart, 2004).

There is a peak difference between transesterified biodiesel and tobacco seed oil which is found at the peak of wave numbers 1500-1400 cm-1 and 1250-1100 cm-1. The resulting peak (Figure 2) of tobacco seed oil in the range of these two wave numbers only appears one peak. This one peak indicates that the oil component is still in the form of triglycerides. The biodiesel spectrogram in the two wavenumber ranges in Figure 2 produces two adjacent peaks. The two wave crests in the range of wave numbers are 1458 cm-1 and 1435 cm-1 which are the wave numbers of (CO)-O-CH3. Two wave crests that appear in the range 1250-1100 cm-1 in biodiesel are wave numbers of the C-O group. The presence of two peaks in the two wave number ranges indicates that the triglycerides from oil have become monoglycerides of ester monoglycerides in biodiesel as shown in Figure 2 (Shimadzu GmbH Europa, 2016).

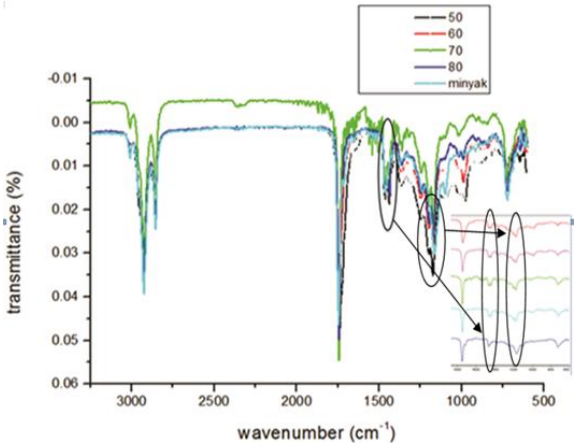

Figure 2. Biodiesel and Oil FTIR Spectrogram

The last identification is identification for its physical properties in the form of density and viscosity. The reference for calculating these physical properties is based on the SNI 04-7182-2006 Agency which states that the density of biodiesel must have a value of (0.85-0.89 g/cm3).

The kinematic viscosity should be between 2.3-6.0 cSt. The following table shows the results of the calculation of the two physical properties that meet SNI:

Table 4. Density and Kinematic Viscosity of Biodiesel

Test result	Biodiesel
Density(g/cm^3)	0,855
Kinematic viscosity (cSt)	3,12

CONCLUSION

The conclusion of the research that has been done is that the transesterification reaction temperature affects the yield of biodiesel as indicated by the difference in yield value and the optimal temperature at 70oC with the success of biodiesel transesterification reaching 47.6%. The results of the identification of the functional groups of each biodiesel yield have similar frequencies in the ester group as shown in the wave number C=O (1750-1730 cm-1) and C-O bonds (1300-1100 cm-1). The physical characteristics of the density and viscosity of the highest yield biodiesel complied with SNI.

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