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# Effects of catalyst types and concentrations on biodiesel production from waste soybean oil biomass as renewable energy and environmental recycling process

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**Abstract**

Biomass and agricultural derived materials have been suggested as alternative energy sources and the use of biodiesel as fuel presently a promising potential that grows rapidly due to its great contribution to the environment and to its role as a strategically source of renewable energy in substitution to diesel oil and other petroleum-based fuels. It is non-toxic, biodegradable and contributes a minimal amount of net greenhouse gases. A study was conducted to produce biodiesel from waste oils to reduce the waste and pollutions. Several important variables such as volumetric ratio, catalyst types and concentration were selected to obtain a high quality biodiesel fuel with the specification of American Standard for Biodiesel Testing Materials (ASTM D 6751) and European Norm (EN 14214). The highest biodiesel yield was obtained (68.5% ) under conditions of 3:1 oil-to-methanol molar ratio, 0.5% NaOH catalyst at 55º C reaction temperature and 250 rpm stirring speed. The results showed that biodiesel production from different oil to methanol molar ratio, catalyst types and concentrations exhibited considerable differences. Biodiesel yield was higher in NaOH than in KOH while used 0.5% as catalyst and the highest yield was obtained having 1% NaOH compared to 0.5 and 1.5% NaOH.. There was little difference in viscosity, acid value and chemical elements (Fe, Mg, Ca, Na, P etc.) at different parameters. The research investigated that biodiesel could be obtained under optimum conditions and catalyst concentrations from completely waste oil which considered as recycled of waste cooking oil.

**Keyword:** waste soybean oil, biodiesel, viscosity, acid value, element content.

**Abbreviations:** ASTM_American Standard for Biodiesel Testing Material; EN_European Norm; NaOH_Sodium hydroxide; KOH_ Potassium hydroxide

## Introduction

The occurrence of oil depletion, global warming and the greenhouse effect has become an alarming condition where it is needed to search for an alternative energy. Biodiesel is a good alternative energy which is one of the most promising energy sources (Demirbas, 2005). There is an increasing interest in alternative energy sources since the major part of all energies consumed worldwide comes from petroleum, charcoal and natural gas. These sources were limited. Biomass and agricultural derived materials have been used as alternative energy sources (Al-Widyan and Al-Shyoukh, 2002; Mushrush et al., 2001; Harten, 2003). Sources of renewable energy are substitution to diesel oil and other petroleum-based fuels (Cardone et al., 2002; Bagley et al., 1998; Monyem et al., 2001). Biodiesel is typically produced by a reaction of a vegetable oil or animal fat with an alcohol such as methanol or ethanol in the presence of a catalyst to yield mono-alkyl esters and glycerin, which is removed. Biodiesel consists of long-chain fatty acid esters (Haas et al., 2001; Abreu et al., 2004) produced by transesterification reaction of vegetable oils with short chain alcohols (Noureddini et al., 1998; Encinar et al., 2002). It is compatible with conventional diesel fuel and already comprises a commercial fuel in Europe (Knothe et al., 2003; Dorado et al., 2003; Serdari et al., 1999). Biodiesel production from soybean oil is very popular. Researchers have focused on different catalyst systems, different solvents, and different acetyl acceptors. Soybean oil has five fatty acids: approximately equal amounts of palmitic acid, oleic acid, and linolenic acid (about 13% each), linoleic acid (approximately 55%), and stearic acid (approxmately 4%). A useful industrial application of soybean oil is in biodiesel blends. According to Kinney and Clemente (2004), soybean oil-derived biodiesel possess enhanced biodegradation, increased flashpoint, reduced toxicity, lower emissions, and increased lubricity. The source for biodiesel production is chosen according to the availability in each region or country. Any fatty acid source may be used to prepare biodiesel, but in the scientific articles reviewed, transesterification reactions have been studied for many vegetable oils such as soybean, palm (Chen, et al., 2009, Hossain et al., 2009a) and fish oil. Since the prices of edible vegetable oils, e.g. soybean oil, are higher than that of diesel fuel, waste vegetable like palm, sunflower and algal oils (Encinar, et al., 1999, Hossain et al., 2008, Hossain et al., 2009b) and non-edible crude vegetable oils have been intensively investigated as potential low priced biodiesel sources. Biodiesel made from this feedstock was predicted to be more economical than the biodiesel produced from refined vegetable oil. Because of its primary feed stock is vegetable oil, biodiesel is generally considered to be renewable. The waste oil product can be of advantage over

**Table 1**. Variable and fixed parameters used in this study

| Variable parameters | Fixed parameters |
|---|---|
| Alcohol to oil molar ratio<br>• 1 : 1<br>• 3 : 1 | • Types of alcohol: Methanol<br>• Types of catalyst: NaOH<br>• Amounts of catalyst: 0.5%<br>• Reaction time: 2 hours<br>• Mixing intensity: 250rpm |
| Types of catalyst<br>• KOH<br>• NaOH | • Alcohol to oil molar ratio: 1:1<br>• Types of alcohol: Methanol<br>• Amounts of catalyst: 0.5%<br>• Reaction time: 2 hours<br>• Mixing intensity: 250rpm |
| Amounts of catalyst<br>• 0.5%<br>• 1.0%<br>• 1.5% | • Alcohol to oil molar ratio: 1:1<br>• Types of alcohol: Methanol<br>• Types of catalyst: NaOH<br>• Reaction time : 2 hours<br>• Mixing intensity: 250rpm |

the neat vegetable oils as they have a higher proportion of saturated fatty acids (Sarin et al., 2007). Waste cooking oil is a good feedstock to produce biodiesel for waste management and recycling process (Hossain et al., 2009a, BFC, 2008). Biodiesel produced from waste biomass does not compete with the edible materials which is a challenge in human life. The objectives of this study were to investigate the optimum conditions of biodiesel production (fatty acid methyl ester) using different catalysts and concentrations and potential use of waste soybean cooking oil as waste management and recycling process. Besides, to investigate the standard quality (ASTM standard) like viscosity, acid number and element content of biodiesel whether it would be useful for diesel engine

## Materials and methods

### *Materials*

Soya cooking oil was bought from a hypermarket and then it was used at the University Cafeteria and collected after frying (for 2 hours with the same source) and used in this study. The chemicals and reagents were ordered and obtained from the store of the Institute of Biological Sciences department, Faculty of Science, University of Malaya. The chemicals were purchased from Chemo-lab, Kuala Lumpur, Malaysia. The chemicals used include methanol ($CH_3OH$), ethanol ($C_2H_5OH$), 1-butanol ($C_4H_{10}O$), sodium hydroxide (NaOH) and potassium hydroxide (KOH) in pellet form, and sodium sulfate in powder form. Other materials used include Double Ring 102 filter paper and aluminum foil.

### *Preparation of waste cooking oil*

The Sunbeam soya cooking oil which purchased from hypermarket was used to fry for the preparation of waste cooking oil. The waste cooking oil was then transferred into a clean plastic bottle. Later, the waste cooking oil was filtered by filter paper to remove bits of food residues. The filtered clean cooking oil was then collected in a clean conical flask and used for experiments.

### *Preparation of potassium and sodium alcoxide*

An appropriate volume of alcohol was measured and poured into a 500ml conical flask. The catalyst in pellet form was weight and mixed with alcohol. The mixture was then shaken for about 1 hour until all the catalyst dissolved. Since alcohols would evaporate easily, the flask was covered with aluminium foil during shaking to reduce the loss of alcohol by evaporation. This covering can also avoid the alcoxide from absorbing water from the air.

### *Transesterification*

Transesterification also called alcoholysis which is the displacement of alcohol from an ester by another alcohol in a process similar to hydrolysis except that an alcohol is used instead of water (Murugesan et al., 2009). This has been widely used to reduce the viscosity of the triglycerides. The transesterification is represented as

$$\underset{\text{Ester}}{RCOOR'} + \underset{\text{Alcohol}}{R'OH} \rightleftharpoons \underset{\text{Ester}}{R'COOR'} + \underset{\text{Alcohol}}{ROH}$$

The methanol was used in this process, then it was called methanolysis.

### *Biodiesel preparation*

The filtered oil was heated up to a temperature of 50°C in water bath to melt coagulated oil. It is important not to make overheat the oil above 65°C, because at that temperature alcohol would boil away easily. The heated oil of 100mL was measured and transfer into a conical flask containing catalyst-alcohol solution. The reaction considered starting at this moment, since heated oil assists the reaction to occur. The reaction mixture was then shaken by using incubator shaker at a fixed (250 rpm) speed having 50°C.

### *Separation of biodiesel from by-products*

The product of reaction was exposed to open air to evaporate excess methanol for 30 minutes. The product was then allowed to settle down overnight. Two distinct liquid phases: crude ester phase at the top and glycerol phase at the bottom would be produced in a successful transesterification reaction. There are a few methods to separate these 2 layers, including using the separating funnel and removing the biodiesel using pipette. It was used later in this experiment.

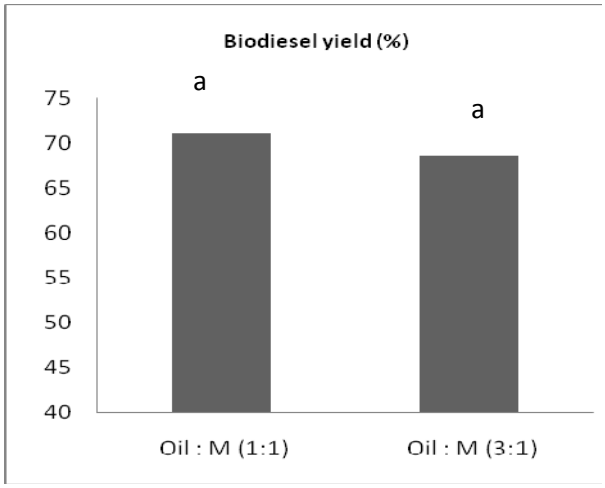


**Fig 1**. The effect of oil to methanol molar ratio on biodiesel production. Same letters (a, a) are not significantly difference at 5% by LSD.

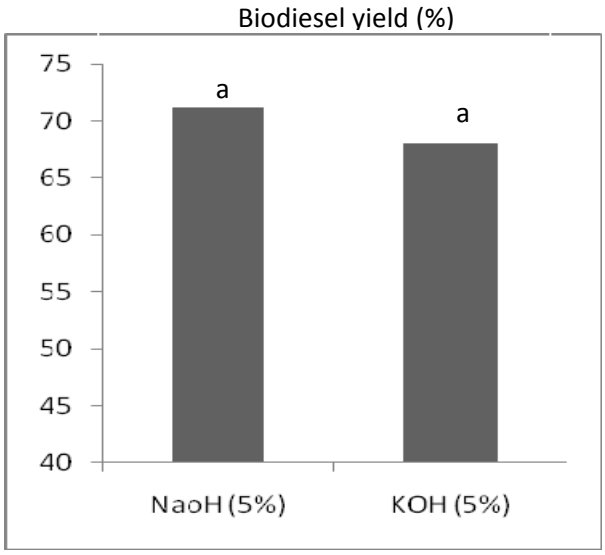


**Fig 2.** The effect of catalyst types on biodiesel production. Same letters (a, a) are not significantly difference at 5% by LSD.

***Purification of biodiesel by washing***

The top ester phase (biodiesel) was separated from the bottom glycerol phase by transferring to a clean 250mL conical flask. The biodiesel was then purified by washing with distilled water to remove all the residual by-products like excess alcohol, excess catalysts, soap and glycerine. The volume of distilled water added was approximately 30% of the biodiesel volume. The flask was shaken gently for 1 minute and placed on the table to allow separation of biodiesel and water layers. After separation, the biodiesel was transferred to a clean conical flask. The washing process was repeated for several times until the washed water became clear. The clean biodiesel was dried in incubator for 48 hours, followed by using sodium sulphate. Then biodiesel was stored for analysis in room temperature ($28^0$C). Variable and fixed parameters used in this study are shown in Table 1.

**Results**

***The effect of different oil to methanol molar ratio on biodiesel production***

In this study, different oil to methanol molar ratios like 1:1 and 3:1 were used. The reactions were carried out using 0.5% sodium hydroxide for 2 hours at room temperature. Figure 1 showed the yield of biodiesel from waste soybean oil by using different types of molar ratio of oil to methanol. The results showed that increasing of methanol to oil molar ratio increased the yield of biodiesel production. Oil to methanol molar ratio of 1:1 gave the higher yield (71.2%) of biodiesel than 3:1 oil to methanol molar ratio.

***The effect of different catalyst types on biodiesel production***

Yield of biodiesel can be influenced by types of catalyst used in the reactions. In this experiment, only 2 types of catalyst were used, they were NaOH and KOH. The reactions were carried out by using 0.5% of catalysts, 1:1 oil to methanol molar ratio for 2 hours mixing time at room temperature. Figure 2 shows the yield percentage of biodiesel using NaOH and KOH as a catalyst. The results showed that NaOH gave the better yield, compared with KOH. NaOH catalysts gave 71.2% of biodiesel yield whereas only 68.9% of biodiesel yield obtained using KOH as catalyst.

***The effect of catalyst concentrations on biodiesel production***

Biodiesel production can be affected by the amount of catalyst used in the reactions. In this experiment, different concentrations of NaOH like 0.5, 1.0 and 1.5% were used. The reactions were carried out by using methanol with 1:1 oil to alcohol molar ratio for 2 h of reaction time at room temperature. Figure 3 shows biodiesel yield using different concentrations of NaOH as a catalyst. From the results, the optimum yield of biodiesel can be obtained at 1.0% of NaOH concentration. It reached 72.7% of the biodiesel yield.

***Viscosity measurement of biodiesel***

One of the main purposes of the transesterification reaction was to reduce the viscosity of cooking oil in order to achieve the properties that is more suitable for its function as fuel. The standard range of viscosity for biodiesel in ASTM D6751 is 1.9-6.0 $mm^2$/sec at 40°C. Figure 4 shows the viscosity of biodiesel produced from waste soybean oil in several of treatments. The viscosity was maintained the ASTM D6751 standard. The lowest viscosity was found in 1:1 oil to methanol ratio following 6 hours shaking time.

***Determination of the total acid number (TAN) value***

Total acid number (TAN) indicates the level of free fatty acids (FFAs) present in biodiesel. TAN value lower than 0.5 mg KOH/g is ideal as fuel for vehicle. A high TAN value can have a strong solvency effect on rubber seals and hoses in the engine, thereby causing premature failure. It may also be left deposits, which can clog the fuel filter or drop fuel pressure. Figure 5 showed the result of TAN value of biodiesels produced using different reaction conditions. The lower TAN value of biodiesel was the range of 1-1.5 mg KOH/g using ethanol followed by the ratio of 2:1 with 6 hours shaking time

**Table 2.** Element contents (ppm) in biodiesels produced from different alcohol to oil molar ratios. Same letters (a ,a; b, b; c, c, d, d) are not significantly difference at 5% by LSD.

| Ratios | 1:1 | 3:1 | NaOH (0.5%) | KOH (0.5%) | KOH (1%) | KOH (1.5%) |
|---|---|---|---|---|---|---|
| Elements | | | | | | |
| Fe | 1a | 0.5 | 1.5 | 1 | 0.7 | 0.5a |
| Al | 0 | 0 | 0 | 0.5 | 0.5 | 0.5a |
| Cu | 0.5a | 1 | 0.5 | 1 | 1.5 | 2b |
| Pb | 0.5a | 0 | 0.5 | 0.5 | 0.5 | 0.5a |
| Sn | 4d | 2 | 4.5 | 7 | 0 | 0 |
| Ni | 3c | 2.5 | 3 | 3.5 | 2.5 | 2.5b |
| Mn | 0 | 0 | 0 | 0 | 0 | 0 |
| Ag | 6.0e | 6.6 | 8.5 | 7.4 | 7.0 | 9.2d |
| Mo | 2b | 1 | 2 | 3 | 0.5 | 0.5a |
| Zn | 0 | 0 | 0.5 | 0.5 | 0.6 | 0.5a |
| P | 3.5cd | 3.5 | 0 | 3 | 0 | 0 |
| Ca | 2b | 2 | 2.5 | 2 | 6.5 | 6.5c |
| Mg | 1a | 1 | 1 | 1 | 2 | 2b |
| Si | 1.5ab | 5.5 | 3.5 | 4 | 2.0 | 2.5b |
| Na | 4.3d | 6.0 | 4 | 3.0 | 5.4 | 8.4d |

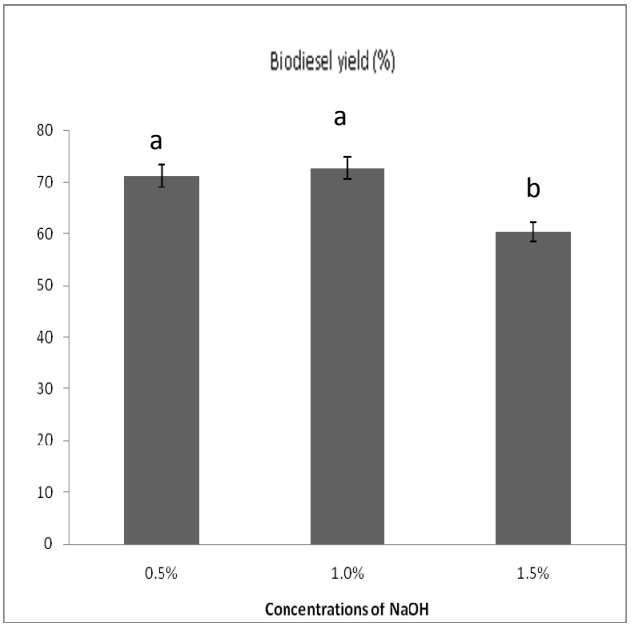


**Fig 3.** Effect of catalyst concentration on biodiesel production. Same letters (a ,a; b, b) are not significantly difference at 5% by LSD.

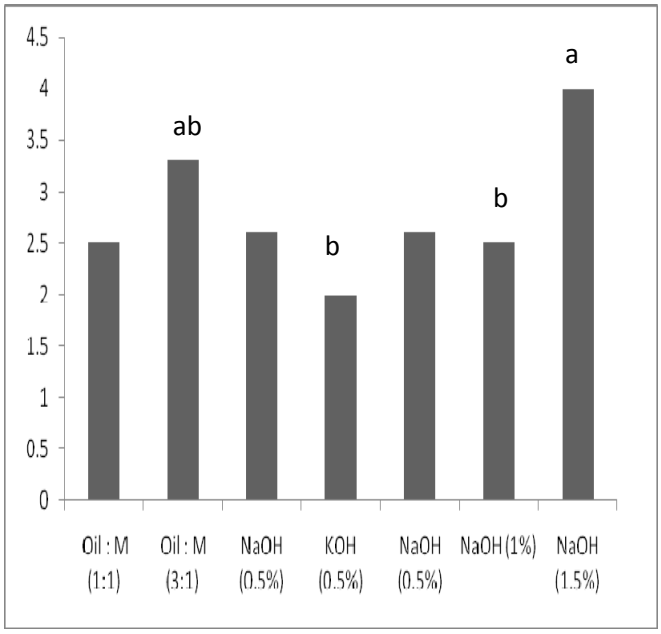


**Fig 4.** Biodiesel viscosity as affected by different types of parameters. Same letters (a, a; b, b) are not significantly difference at 5% by LSD.

***Multi-element analysis***

Multi-element analyzer was used to measure wear debris and additives depletion. Table 2 shows the concentration of elements in biodiesels that produced from different parameters. The concentrations of most of the elements in biodiesel produced from waste soybean oil were quite low generally. However, the concentration of argentums or silver was surprisingly high. Table 3 shows the standard value of viscosity and acid value.

The elements measured in this study were iron, cromium, aluminium, copper, lead, tin, nickel, manganese, titanium, argentum, molybdenum, zinc, phosphorus, calcium, barium,

**Table 3.** Parameters for the quality of biodiesel (Meher *et al*., 2006)

| Parameters | Austria (ON) | Czech republic (CSN) | France (journal official) | Germany (DIN) | Italy (UNI) | USA (ASTM) |
|---|---|---|---|---|---|---|
| Viscosity at 40°C ($mm^2/s$) | 3.5-5.0 | 3.5-5.0 | 3.5-5.0 | 3.5-5.0 | 3.5-5.0 | 1.9-6.0 |
| Acid number (mg KOH/g) | ≤0.8 | ≤0.5 | ≤0.5 | ≤0.5 | ≤0.5 | ≤0.8 |
| Group I metals ($Na^+$ K) mg/l | - | - | - | - | - | 5.0 max EN 14214 |
| Group II metals ($Ca^+$ Mg) mg/l | - | - | - | - | - | 5.0 max EN 14214 |
| Phosphorus content, mg/l | - | - | - | - | - | 10.0 max EN 14214 |

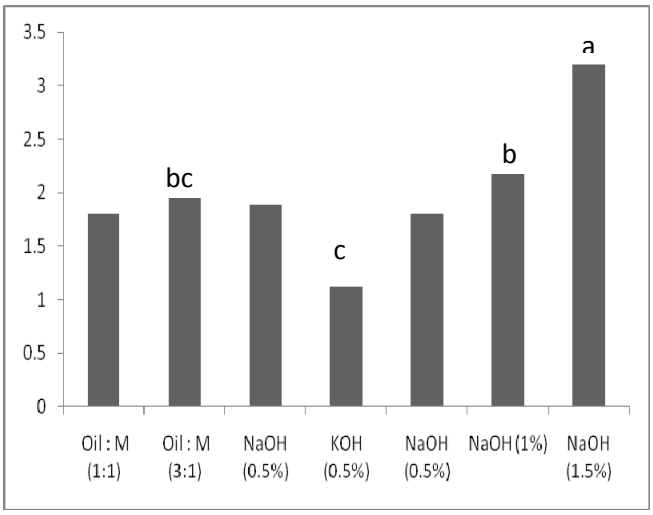


**Fig 5.** Biodiesel TAN value as affected by different types of parameters. Same letters (a ,a; b, b; c, c) are not significantly difference at 5% by LSD.

magnesium, silicon, sodium, boron and vanadium. The result showed that the concentrations of all the elements were lower than 5ppm, except argentum and sodium. However, most of the element content followed standard.

**Discussions**

***The effect of different oil to methanol molar ratio on biodiesel yield***

From the results, it can be explained that 1:1 oil to methanol molar ratio gave the best ester yield. Oil to methanol molar ratio of 3:1 gave the lowest yield of biodiesel. The idea is when increasing the oil to methanol molar ratio, means alcohol amounts is elevated, and the FAME yield is increased. To maximize the fatty ester yield, it is needed to drive the transesterification reaction to shift to the product that is ester and glycerin. Therefore, it is needed to increase the concentration of reactant such as alcohol amount, so that the reactions would be more favorable to generate FAME yield. And there are many researchers found that less alcohol will cause the transesterification to incomplete. Many of them found that the optimum oil to methanol molar ratio was 1:6. With that molar ratio, one can get a higher ester yield if other reaction conditions are optimum also. However, the high molar ratio of alcohol to vegetable oil interferes with separation of glycerin because; there is an increase in solubility. When glycerin remains in solution, it helps drive the equilibrium to back to the left, lowering the yield of esters (Murugesan et al., 2009).

***The effect of different catalyst types on biodiesel production***

Transesterification using NaOH as a catalyst gave slightly better fatty ester yield when comparing with KOH. There are no significant different between these two catalyst in term of biodiesel yield. There are also no clearly reasons to explain why NaOH is better catalyst, nor KOH. But, there are many researchers found that types of catalyst performance are strongly dependent on feedstock used. Besides, catalysts performances were also influenced by reaction conditions.

***The effect of different concentrations of catalyst on biodiesel yield***

Based on results that obtained, we found that 1.0% wt of NaOH gave the best yield on biodiesel production among 0.5%, 1.0% and 1.5%. We can observe that fatty ester yield increasing with the concentration of catalyst increased until 1.0%. Beyond that value, the fatty ester yield started to drop. Normally, when we increase the catalyst amount, it will help to fasten the reaction and gave better yield. However, every reaction got it optimum catalyst concentration value. Beyond that value, excessive catalyst for example NaOH will participate in saponification which reacts with triglyceride to form soap and water. Hence, it will reduce the biodiesel yield.

***Analysis of biodiesel viscosity***

From the results, it was obtained that biodiesel viscosities were under ASTM standard. Average of biodiesel viscosities that produced were maximum 4.4 cst at 55°C. However, the ASTM D6751 standard limit is 1.9 – 6.0 $mm^2/s$ or cst/s at 40 °C. Higher viscosity may be due to the long storage time. There are few researchers found that longer the storage time of biodiesel, higher the viscosity value. During storage the viscosity of ethyl esters increases owing to the formation of oxidized polymeric compounds that can lead to the formation of gums and sediments that clog filters (Bouaid et al., 2009). And eventually, it increases the viscosity of biodiesel in stored condition. It was reported that Kinetic viscosity of biodiesel from waste cooking oil was 5.3 and 1.9-4.1 $mm^2/s$ at 313 K commercial biodiesel fuel (Demirbas, 2005)

***Analysis of biodiesel TAN value***

Based on results obtained, the average total acid number from biodiesel samples was higher than ASTM D664 standard. Present results showed higher acid content, this may be caused by the long storage due to the analysis facilities problem. In addition, acids can be formed when traces of water cause hydrolysis of the esters into alcohol and acids (Bouaid et al., 2009). As expected, the acid number increases

with an increase in peroxides because the esters first oxidize to form peroxides which then undergo complex reactions, including a split into more reactive aldehydes which further oxidize into acids.

***Analysis of biodiesel element composition***

Biodiesel from waste soybean cooking oil had considerably lower Fe, Cu, Pb, Ca, Na, P content in 1% KOH than in all catalysts concentrations. Most of the elements (Fe, Cr, Pb, Cu, Ag, Mg, Ca, Na, Zn, Si and P) fulfilled the requirement of the standard method specification as well (ASTM D 6751 & EN 14214 methods). Hossain et al., (2009a) reported the same result in their previous research results. From the results, concentration of various element the biodiesel produced were generally lower as expected. However, only argentums or silver were little bit higher but maintained ASTM standard.

**Conclusion**

The results showed that the highest triglyceride conversion rate of 71.2% was achieved after 2h of reaction at $55^0$C having 1:1 molar ratio of methanol to waste soybean oil and 1.0 % catalyst of sodium hydroxide. The optimal values of these parameters for achieving maximal conversion of oil to esters depended on the chemical and physical properties of these oils. The NaOH having 1% concentration can be recommended to complete conversion of triglycerides into esters based on higher yield. The overall results showed that waste cooking oil could be used to produce biodiesel from waste oil as waste management and recycled process.

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