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Effect of the Duration Time of Cooking Frequency on Chemical and Physical Properties of Waste Cooking Oil Used for Biodiesel Production



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Abstract

The energy problems and global warming has led to intense research into the existence of alternative fuels with high efficiency and high calorific value. Oily plants have a high calorific value when using the resulting oil, especially palm oil. The search for alternative fuels has always conflicted with the use of edible oils.

This paper aims to study the effect of cooking times on biodiesel production from oil. The study was applied to different samples of used waste cooking oil (WCO) for several times, the samples were fresh oil, oil cooked for several times: 3, 6, 9 and more than 9 times (local WCO).

The best yield of biodiesel is about 97% for local WCO. The biodiesel color is clear as the Lovibond No. dwindles down which means less FFA% and good quality of biodiesel. This method can be using to decrease density and viscosity of biodiesel and oil to be like diesel. The best preheated temperatures are 60 °C for biodiesel and about 100°C for oil. Cetane number increased with increasing number of using oil . The local WCO is the best Cetane number. But Calorific value is best with fresh oil and local WCO. The local WCO is the optimum selection condition.

Keywords: WCO; biodiesel; fuel characterization; waste cooking oil

1. Introduction

Currently there is a steady global increase in energy. There are many sources of energy, for example natural gas, followed by oil and coal in terms of pollutants. They are all non-renewable fuels, so they are all constantly decreasing daily [1]. The available energy resources, together with the unequal distribution of fossil fuels, have accelerated the undesirable energy-related problems, thereby triggering energy security problems in various sectors such as power generation, transport, the chemical industry, and others [2-4]. Biofuel has gained considerable interest in numerous countries around the world recently due to its renewability, reduced gas efficiency, and biodegradability. Huge use of fossil worldwide eventually resources caused the accumulation of carbon in our environment [5,6]. The additional carbon inputs from the large use of fossil

fuels far outweigh the maximum capacity of the earth to assimilate it through the natural carbon cycle [7]. The use of fossil energy directly affects the global warming problem because it increases carbon dioxide concentration in the atmosphere [8]. Governments and environmentalists also agree that biofuels are the most promising sustainable alternatives required to achieve the objectives of reducing overall reliance on fossil fuels and reducing CO₂ emissions, as this will help local farming and grow economies [9,10], there is also no need to change the engine to use biodiesel as a fuel [11]. Biodiesel is pure, safe and non-hazardous, as opposed to traditional petroleum diesel because of its biodegradability, sustainable and carbon-neutral existence [12,13] .Biodiesel is becoming more competitive between different types of biofuels in terms of its properties and chemical composition, which can be used as a mixture with diesel fuel [14].

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Biodiesel can be made from a broad variety of feedstocks, including virgin oils (e.g. palm oil, rapeseed oil, soybean oil, etc. (e.g. yellow grease), used cooking oils, microalgae oil, and animal fats [15-22] . Biodiesel production from waste cooking oil has economic benefits, as its cost is the lowest among both renewable and conventional fuels [23]. Biodiesel is more desirable in terms of energy density compared with bioethanol, which is possibly due to a relatively long carbon chain attributed to fatty acids. In addition, the relatively easy conversion process for biodiesel compared with that for bioethanol can be a driving force for increasing its use and marketing in a relatively short timeframe [24]. Since plants and vegetable oils and animal fats are renewable sources of biomass, biodiesel represents a mostly closed cycle of carbon dioxide (about 78%) as it is derived from renewable sources of biomass [25]. Most of the currently made biodiesel uses soybean oil, methanol, and an alkaline catalyst. The high value of soybean oil as a foodstuff allows a very cost-effective fuel production [26]. Edible vegetable oils make up over 95% of world biodiesel production [27]. While [28,29] stated that the use of edible oils in biodiesel production could lead to some negative impacts in developing countries, such as malnutrition and higher food prices. Where the prices of refined virgin oils are generally very high, commercial production of biodiesel fuel is therefore impracticable [30]. Where the biggest issue in biodiesel production is the cost of the raw material which could account for up to 75 percent of the total cost of production [31]. These results in prices of biodiesel 1.5 times higher than those of diesel petroleum [32]. So, stated that the use of HORECA (hotels, restaurants and catering) WCO as raw material is one way to reduce costs, since these oils are 2-3 times cheaper than vegetable oils from crops or trees [33]. In addition, the use of waste cooking oil often decreases the costs of handling food. There is no legislation in many parts of the world which prohibits the disposal of waste cooking oil into the drainage system. Disposing waste cooking oil into drains causes many issues with service and maintenance. Thus, the disposed oil can block drainage systems and pollute wastewater and waterways [34]. The use of virgin oil as a feedstock for the production of biodiesel contributes to competition between food and energy sources [35]. Biodiesel benefits include safe, reliable, displaces diesel fuel, reduces global warming, and reduces gas pollution like air toxics. Biodiesel drawbacks include high cost relative to gasoline, a

small rise in emissions of nitrogen oxides, and longterm storage changes in fuel products [36].

The transesterification procedure is the reaction of a triglyceride (fat/oil) with an alcohol to form esters and glycerol in the presence of any catalyst. A triglyceride has an appended glycerin particle as its foundation with three long chains of unsaturated fats. The oil/fat attributes are influenced by the concept of the unsaturated fats linked to the glycerin. Thus, the concept of unsaturated fats will affect the biodiesel attributes. The partition of the ester and glycerol layer after the response time entails a fruitful transesterification response [37,38].

One of WCO key concerns is their alteration of the compound by the responses that occur during searing. Broiling is an unpredictable process in which different reactions assisted by three operators (water, oxygen, and high temperature) cause changes in physical and substance on plant oil. During searing, unsaturated fats, diglycerides, oxidized free monomers, dimers and polymers and some unpredictable mixtures (aldehydes, ketones, hydrocarbons, etc.) are generated [39]. WCO can also be weakened during storage, which can alter cinematic viscosity, density, water content and acid count [31]. The molecular composition influences the physical properties of the fuel, for example, viscosity and volatility as well as the chemical characteristics of the fuel, which oversee the burning responses as far as the beginning and length of ignition and its vitality discharge rates [40]. In this manner biodiesel properties contrarily meddle with the infusion procedure prompting poor fuel atomization, deficient ignition, and unnecessary carbon stores on fuel spouts. The response is generally influenced by various variables including: alcohol to oil molar ratio, reaction time. catalyst nature and quantity, reaction temperature, and composition of the feedstock [33]. the key factors influencing transesterification are alcohol to oil molar ratios, pressure, catalyst, reaction time, response temperature, water contents, and free unsaturated fats levels in oils and fats [41]. Where the issue with preparing these minimal effort oils and fats is that they frequently contain a lot of FFA that can't be changed over to biodiesel utilizing an alkaline catalyst [26]. The aim of this paper is to determine the effect of cooking times frequency on chemical and physical properties of waste cooking oil used for biodiesel production.

2. Materials and Methods

2.1 Material and Instruments Used in Transesterification

WCO samples were collected from a public restaurant in Cairo city- Egypt, this oil was palm oil. Methanol (CH₃OH) 99% was used as a solvent, pure and dried sodium hydroxide (NaOH) was used as catalyst were used. The materials that used in the titration process were consisted of distilled water, isopropyl alcohol (99%), water and the acetic acid were used in the washing process while it was used as an indicator of the phenol solution and naphthalene .Viscometer was used to measure kinematic viscosity, Magnetic Stirrer Hot Plate mixer to agitate and heat up the mixture to a desired temperature and Gas Chromatography (GC) was used to determine the Fatty acid compositions of samples. The solids in the oil are eliminated by a filtration process. The oil is heated to a temperature of 35 ° C. Then the oil is heated again at a concentration of 100 °C [38,39]. Transesterification chemical reaction and transesterification process layout are shown in Fig. 1.



2.2 Titration

In the transesterification process, the titration process is used to obtain the sum (grams) of catalyst (NaOH) per liter of oil which will yield optimum yield. In short, dissolve 4 grams of NaOH in 1 liter of distilled water solution (0.4 per cent NaOH). One ml of oil is dissolved in 10 ml of pure isopropyl alcohol, then stir until clear in the Magnetic Stirrer Hot Plate mixer. To get the end point, two drops of a solution of phenolphthalein are added. Using a burette, 0.4 per cent NaOH solution is added drop by drop to the solution of oil alcohol phenolphthalein and stirred all the time until the solution remains pink for 10 seconds. As shown in Fig 3, the number of ml of 0.4 per cent NaOH solution used will be reported. [40,41].





b Fig. 2 (a) Viscometer (b) Magnetic Stirrer Hot Plate mixer.



Fig. 3 Titration process.

2.3 Preparing of Sodium Methoxide (Catalyst)

Based on the molar ratio of oil to methanol which is 6:1, the volume of methanol used is 20 per cent of the oil by mass. They measure this using their molar mass. Twenty grams of methanol is combined in a separate vessel with 0.8 g of NaOH and poured into a round bottom flask while constantly stirring until all of the NaOH dissolves in the methanol, producing exothermic Sodium Methoxide [42].

2.4 Heating and Mixing

In the oven, the oil is heated to 65 ° C and stirred at intervals to permit a uniform heat distribution and to avoid temperature gradients. The oil is poured into a flat bottom flask and put on an electric mixer and stirred with a magnetic stirrer. Too much noise should be avoided as it can cause splashing, bubbles by vertexing and decreases the efficiency of mixing. The sodium methoxide is injected into the oil and mixed for 1.5 hr. The transesterification process removes the glycerol from the methyl esters. The methanol caps CH3O off the ester chains, and the NaOH OH stabilizes the glycerin as shown in Fig. 4 [43].



Fig.4 Heating and Mixing process.

2.5 Settling and Separation of Biodiesel

The solution is poured into a separate funnel and can separate for up to 12 hours. The methyl ester (biodiesel) will float at the top while the denser glycerin will be at the bottom of the funnel. The bottom layer is composed of glycerin, excess alcohol, catalyst, impurities, and unreacted oil traces. The funnel tap is carefully open for separating the glycerin from the biodiesel. If the glycerin congeals in the pipe, the glycerin is reheated just enough to liquefy again. The minimum volume of glycerin should be around 20 per cent of the original volume of oil. The phase of glycerin is much denser than the phase of biodiesel and Glycerol looks very dark compared to the yellow biodiesel as shown in Fig. 5 [38,40].

2.6 Alcohol Removal

If the phases of glycerin and biodiesel have been separated, the excess alcohol is extracted via a flash evaporation process or by distillation at each step. The alcohol is separated in other processes, and the mixture neutralized before separating the glycerin and esters. In this case, the alcohol is extracted and re-used using distillation equipment. Care should be taken to ensure that no water accumulates in the alcohol stream recovered [43,44].



Fig. 5 Settling and Separation of Biodiesel.

2.7 Glycerin Neutralization

The glycerin byproduct includes unused catalysts and soaps, which are neutralized with an acid and sent as crude glycerin for storage. In certain cases, the salt produced during this process is collected as fertilizer for use. The salt is mostly left in the glycerin. Water and alcohol are separated to manufacture 80-88 percent pure glycerin ready for sale as crude glycerin. Glycerin is purified to 99 percent or higher purity in more advanced operations and marketed in the cosmetics and pharmaceutical markets [38].

2.8 Washing and Drying of Biodiesel

Biodiesel was combined, washed with warm distilled water to extract the alcohol, oil and catalyst that had not reacted. It can settle for 24 hours, under gravity. When you first wash the biodiesel, a small amount of dilute acetic acid is applied when adding the bath. This is to get the solution's PH closer to zero, since it neutralizes and lowers any NaOH that is suspended in biodiesel. Two layers were formed, biodiesel was the upper layer and water and impurities was the lower layer. This method was repeated until the lower phase had a pH value close to that of distilled water, meaning that only water was present, and the catalyst is finally eliminated in the washing process. It was found that some ester was lost due to emulsion formation during washing. Separated biodiesel for characterization is taken as shown in Fig. 6 [45-49].



Fig. 6 Washing and Drying of Biodiesel.

2.9 Biodiesel Yield

After the reaction, the biodiesel yield was measured from the upper layer of the decanter, and the removal of methanol. The weight of biodiesel produced relative to the initially used oil has been taken as the yield of biodiesel. However, two potential side reactions can occur in the transesterification process, neutralization of the oil's free fatty acids and/or triglyceride saponification. Both manufacture sodium soaps, and thus reduce the yield of biodiesel. The free fatty acids could be neutralized because the acid value of the oil used was around 1 mg KOH / g of oil. Triglyceride saponification may be of minimal benefit as the water content of the oil used was zero, but the presence of the hydroxide group may induce saponification of triglycerides. Biodiesel yields were determined using the following equation [40,41]:

Yield of biodiesel (%) = $\left(\frac{\text{Total weight of methyl esters}}{\text{Total weight of oil in the sample}}\right) x100$

2.10 Glycerol Purification

The coating of glycerol was gathered from the base of the decanter. This layer contained the unreacted methanol glycerol component, formulated soaps, and the catalyst. These free fatty acids may be used in some industries, such as soap production or may be subjected to further purification for pharmaceutical use [40,50].

2.11 Gas Chromatography analysis for oil

This is a technique used to calculate the concentration of fatty acids in the oil sample which allows mixtures to be separated based on their boiling point. Gas Chromatography (GC) oil sample was prepared by method of methylation. Methanolic Sodium Hydroxide was prepared in the methylation process by dissolving 2 g of sodium hydroxide (NaOH) in a 100 ml methanol solution. The mixture was mixed for 2 minutes until it had formed a simple solution. Mixed 0.2 g oil sample with 6 ml of Methanolic Sodium Hydroxide Solution. The mixture was then refluxed for 10 minutes. Then 10 ml of a mixture solution, consisting of 30 ml of concentrated HCL and 20 ml of methanol, was applied for another 10 minutes and refluxed. Then 10 ml of hexane was added, and 2 minutes refluxed and allowed to cool. 10 ml of distilled water was eventually applied, and the mixture poured into a separate funnel. Calcium chloride was extracted and applied to the upper layer. That was then used as a GC research sample [51,52]. Oil with greater unsaturated fatty acid content has a relatively short induction duration. The peak values shown in Fig.9 indicate areas that contribute time to relative abundance as shown in Table 3.

A- Preparation of Methanolic Sodium Hydroxide

In short, 2 g of sodium hydroxide (NaOH) sample was weighed and dissolved in 100 ml of methanol solution. The mixture was stirred for 2 minutes until a transparent solution was obtained containing the hydroxide solution Methanolic Sodium.

B- Methylation of the Oil Sample

In short, 0.2 g of the oil was weighed into a quick fit conical flask and 6 ml of Methanolic Sodium Hydroxide solution was applied. It had refluxed into a steam bath for 10 minutes. 10 ml of a solution composed of 30 ml of concentrated HCL and 20 ml of methanol was then applied to the steam bath and refluxed for another 10 minutes. 10ml of hexane was then applied to the steam bath and refluxed for 2 minutes and allowed to cool. 10 ml of distilled water was eventually applied, and the mixture poured into a separate funnel. Calcium chloride was precipitated and applied to the organic layer (upper layer). That was then used as a GC research sample [53–55]. 3. Results and Discussions

3.1. Effect of cooking frequency on Free Fatty Acids

3.1.1 Cooking frequency affecting on Free Fatty Acid percentage

The Free Fatty Acid percentage affected by number of oil using. Table (2) gives the proportion of fatty acids influenced by the number of uses of oil. Titration is used to measure the concentration of FFA in the oil. These formulae estimate the estimation of the FFA percentage from a titration value [46,53].

FFA% = (28.2 t n) / w (1) Where

t = Amount in ml of the titration solution, n = NaOH solution standard (n=0.025), and w = Oil sample weight (1ml = 0.92 g) in grams. Thus, FFA% = 0.766 t (2)

3.1.2 Cooking frequency affecting on Free fatty acid composition

The chemical and physical properties are affected by number of time oil using. oil with greater unsaturated fatty acid content has relatively short induction duration. The peak values, as shown in Fig. (7), indicate areas that contribute time to relative abundance as shown in Table (1). Data shows that 9-Octadecenoic acid or oleic acid (C18:1) and palmitic (C16:0) were the abundant two fatty acids and their content was varied slightly due to cooking frequency. Local oil presented 69% of oleic acid with 31% of palmitic acid. The high content of oleic acid meeting the high food quality and seems to be more resistance against heat decomposition. This finding was in harmony to those mentioned by [54] who found that stearic (C18:0) 11.495% acids, oleic (C18:1) 43.138% and linoleic (C18:2) 10.184% acid were found and palmitic acid contents was more or less similar between two compared oil sources. Lower (3times) and moderate (6 timed) cooking frequency slightly increased oleic acid content parallel with the decrease in palmitic acid content and the opposite pattern was observed with the higher cooking time frequency (9 times). Temperature and heating time effect on palm oil was studied by [55]. They summarized that fatty acid composition (FAC) of oil extracted showed a reduction in unsaturated fatty acids as the heating duration increased. After 3 months of storage, 60 min

of heating time via wet heating process was recorded the lowest increment of FFA (1.32%) and low reduction of DOBI value (0.41). However, carotene content gave the highest loss with 113.54 ppm towards the end of the storage.

Cooking frequency	Palmitic acid,	9-Octadecenoic acid (Oleic acid)		
Palm fresh oil	30.62	69.38		
WCO cooked 3 times	29.25	70.75		
WCO cooked 6 times	28.77	71.23		
WCO cooked 9 times	33.16	66.84		
Local WCO (>9)	31.00	69.00		

 Table 1. Palmitic and oleic acid content (%) as affected by cooking frequency.

3.2 Cooking frequency affecting on oil color

The number of using oil changes the oil color and hence the oil quality. Oil used color is represented by Lovibond scale as indicated in Fig. (8)[56]. Oil colors at different number of uses in Table 2. The usage number influences both the chemical and physical properties. According to use the oil is hydrolyzed and oxidized. Higher using has darker color oil. The darker color of oil is caused by using oil several times.

3.3 Temperature effect on oil and biodiesel-density

The variations in densities for different temperatures of number of using oil are shown in Fig. (10). Oil densities decrease with temperature rise and the density is inversely proportional to the oil temperature as obtained by the calculated results which possessed a linear relation (Table 3).

Number of using oil changes the oil color and quality as show in Fig.9. Biodiesel color is represented by using Lovibond scale as a comparative method [50,51,52,56,57]. The biodiesel color is lighter as the Lovibond No. dwindles down which means less FFA% and better oil quality.

3.4 Temperature affecting on oil and biodieselviscosity

The variations in viscosities at different temperatures of oil at the different number of used are indicated in Fig.11. However, the viscosity decreases with increase in oil temperature. One of the solutions to solve the problems associated with the higher oil viscosity in diesel engines is preheating of bio oil fuel pipes. The viscosity for the various samples can be compared and fitted as shown in Table 4. Depending on the increase in oil temperature the viscosity decreases as a power function.





# Value	Color
2	
3	
4	
6	
7	
10	
13	
17	
22	
24	
30	
38+	

Fig. 8. Lovibond scale for used oil color.

Table 2. Free fatty acids percentage and Lovibond number of fresh and cooked-oil.

No. of	NaOH,	FFA,	Lovibond
Cooking	ml	%	No.
Palm fresh oil	1.00	0.766	3
WCO cooked 3 times	1.70	1.302	4
WCO cooked 6 times	2.60	1.992	5
WCO cooked 9 times	2.80	2.145	б
Local WCO (>9)	6.00	4.596	10

 Zero
 3
 6
 9
 Local

Fig. 9. Biodiesel color for different samples.

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Table 5. Temperature effect on oil and biodiesel-density.						
Temp. (°C)	Fresh oil	3 times	6 times	9 times	Local WCO	Diesel at 40 °C
Oil density (Kg.m ⁻³)						
20	909	907	904	907	916	
40	896	896	895	895	900	
60	883	885	884	882	889	828.58
80	870	873	876	872	877	
100	864	863	863	864	866	
Biodiesel density (Kg.m ⁻³)						
30	861.21	862.62	855.97	856.58	861.01	
40	859.20	857.79	851.14	853.76	859.60	
60	854.36	841.07	841.07	842.48	846.91	828.58
80	824.76	827.17	831.20	834.63	833.82	
100	811.67	812.47	821.53	817.30	809.85	

Table 3. Temperature effect on oil and biodiesel-density

Table 4. Temperature effect on oil and biodiesel-viscosity.

T (°C)	Palm Fresh oil	3 times	6 times	9 times	Local WCO	Diesel at 40 °C
Oil Viscosity, CP						
20	34.5	7.8	7.2	7.7	17.1	
40	2.9	3.4	4.2	4.0	4.0	
60	2.5	2.3	2.5	2.3	2.8	1.188
80	2.0	1.9	2.0	2.1	2.2	
100	1.5	1.7	1.6	1.8	1.7	
Biodiesel Viscosity, CP						
30	1.586	1.473	1.543	1.473	1.568	
40	1.421	1.402	1.374	1.441	1.509	
60	1.117	1.160	1.133	1.185	1.239	1.188
80	1.089	1.013	0.991	1.019	1.109	
100	0.900	0.890	0.926	0.922	1.004	



Fig. 10. Temperature effect on a) oil and b) biodieseldensity.

3.5.2 Glycerol Purification

The coating of glycerol was gathered from the base of the decanter. This layer included glycerol, part of the unreacted methanol, soaps formed, and catalyst formed. Glycerol may be used in some industries such as soap making, and may be subjected to further purification for pharmaceutical purposes [40]. Yield of glycerol for samples shown in Table 5. The yield of Glycerol was calculated using the following equation [40]:

Yield of glycerol (%) =
$$\left(\frac{\text{Total weight of glycerol}}{\text{Total weight of oil in the sample}}\right)$$

x100

Table 5. Yield of glycerol and biodiesel in the samples.

Cooking frequency	Glycerol	Biodiesel
Fresh oil	22.09	87.43
Cooked 3 times	21.60	83.27
Cooked 6 times	18.73	90.31
Cooked 9 times	18.57	94.47
Local WCO (>9)	10.80	97.14



Fig. 11. Temperature effect on a) oil and b) biodieselviscosity.

3.5 Biodiesel and Glycerol Purification 3.5.1 Biodiesel Purification

The Biodiesel layer was collected from the top of the decanter. This layer contained the biodiesel in our research, the yield of biodiesel is about 97% (Table 5). The yield of biodiesel was calculated using the following equation [40,41]:

Yield of biodiesel (%) = $(\frac{\text{Total weight of methyl esters}}{\text{Total weight of oil in the sample}} \times 100$

No.	Cooking frequency	Cetane number	Calorific value, kJ.kg ⁻¹
		ASIM D-976	ASIM D-240
1	Biodiesel from fresh Palm oil	63.79	43456
2	Biodiesel from Palm oil used 3 times	62.54	42920
3	Biodiesel from Palm oil used 6 times	64.09	41986
4	Biodiesel from Palm oil used 9 times	64.41	35196
5	Biodiesel from local WCO (used >9)	65.19	42948

Table 6. Cetan number and calorific value of biodiesel for different using times.

3.6 Effect of times of cooking on biodiesel properties

The measured calorific values of biodiesel produced from fresh palm oil, palm oil used 3 times, palm oil used 6 time, palm oil used 9 times and local WCO (used >9) were 43456, 42920, 41986, 35196 and 42948 kJ/kg respectively, as presented in Table (6). The fuel's calorific value determines the amount of heat required to produce the engine output. Hence calorific values are significant in choosing an alternative fuel for higher engine efficiency for diesel engines. Measured Cetane number of biodiesel produced from fresh palm oil, palm oil used 3 times, palm oil used 6 times ,palm oil used 9 times and local WCO (used >9) are 63.79, 62.54, 64.09, 64.41 and 65.19, respectively. Cetane number is an indicator of a fuel's combustion efficiency in diesel engines and is related to the fuel volatility and time delay for ignition. Ignition quality and number of Cetane affect engine output, starting cold, warming up and roughness of the engine combustion. Higher Cetane number leads to shorter delay in ignition and a reduction in engine output.

4 Conclusions

- 1. The yield of biodiesel is about 97% for local WCO (>9 times using). The yield was increased with increase times of cooking. The density is inversely proportional to the oil temperature as a linear relationship. It was decrease with increasing oil temperature. The viscosity decreases with increasing oil temperature.
- 2. Number of using oil changes the oil color and hence the oil quality. The biodiesel color is lighter as the Lovibond No. dwindles down which means less FFA% and hence a better oil quality.
- 3. The biodiesel and oil density and viscosity affected by preheating temperature. This method can be using to decrease density and viscosity of biodiesel and oil to be like diesel. The best preheated temperatures are 60 °C for biodiesel and about 100°C for oil.

- 4. Cetane number increased with increase of number of using . The local WCO is the best Cetane number.
- 5. Calorific value is best for fresh oil the local WCO is the optimum selection condition.

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