## EXPERIMENTAL INVESTIGATION OF DIFFERENT CHARACTERISTICS OF BIODIESEL DERIVED FROM WASTE COOKING AND PURE SUNFLOWER OIL

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Abstract: In this investigation, waste cooking and pure sunflower oil were selected as feedstock for biodiesel synthesis under microwave irradiation and conventional heating systems. Experiments were conducted in order to evaluate the effects of reaction variables, such as catalysts amount (0.5-1.5wt %), oil: methanol volumetric ratio (4:1-8:1) and time (1-6 min) in microwave method and time (30-60 min) in conventional method. The best yield of biodiesel (97%) was obtained by the condition of 1 wt% NaOH, 4:1 oil to methanol volumetric ratio, and 4 min using microwave heating system. The important properties of biodiesel were analyzed and found to be within the limits of biodiesel standards. Compared with the conventional heating method the results show that microwave heating can be applied effectively to obtain high yield and reducing the reaction time from 1 h to 4 minutes. The use of microwave will help in providing an energy efficient and economical route for biodiesel production. Results also showed that the biodiesel obtained under optimum conditions from pure sunflower oil (PSO) and waste cooking sunflower oil (WCSO) exhibited no considerable difference but the cost of producing biodiesel from WCSO was lower than PSO. This research demonstrated that biodiesel obtained under optimum condition from PSO and WCSO was of good quality and could be used as a diesel fuel which is considered as renewable energy and environmental recycling process from waste oil after frying.

Keywords: Biodiesel, transesterification, pure sunflower oil, waste cooking sunflower oil, microwave and conventional method.

#### Introduction

Due to the increasing demand for energy especially petrochemical fuels, the need for alternative liquid fuel is growing very fast. Therefore, taking into account the rapid depletion and negative aspects of utilization of nonrenewable energy resources, the energy consumption in the country is shifting towards sustainable and renewable energy. Biodiesel has been considered as one of the most energy efficient and environmentally sustainable alternative fuels for diesel engines over the last few years because of its almost similar properties to diesel fuel and low particulate and gas emissions. (Fjerbaek *et al.*, 2009; Ma & Hanna, 1999)

Among all the biodiesel production processes, transesterification is the key and most important processes to produce fuel from

vegetable oils and animal fats (Vicente et al., 2007; Angelo et al., 2005; Dorado et al., 2000). The last few decades, several researchers reported commercial biodiesel preparation from the oils of palm (Benjumea et al., 2008), rapeseed (Komers et al., 2001), jojoba seeds (Mazloom et al., 2014), soybean (Candeia et al., 2009), rocket seed (Tariq et al., 2011), sesame (Tariq et al., 2013) etc. Primary and secondary alcohols are usually used for the transesterification process. Considering low cost, methanol is more preferred over ethanol and frequently used in biodiesel production (Fukuda et al., 2001). Presently, more than 95% of commercial biodiesel is produced through transesterification of different edible oils (Fan et al., 2011; Wang et al., 2011). Sunflower seeds have a high place in international agricultural market that contains about 42-50% high quality edible oil (Bakhsh et al., 1999; Rashid et al., 2006).

Transesterification reaction be can catalyzed with alkali, acidic or enzymatic catalysts. Among them, alkali process yields high quantity and high purity biodiesel in shorter reaction time as compared to the enzyme catalyst process (Balat et al., 2011; Fukuda et al., 2001); however, this process is not suitable for feedstock with high free fatty acid (FFA) content. An alternative method, namely the microwave-assisted catalytic transesterification, which is an energy-efficient and a quick process to produce biodiesel from different feed stocks has been developed that gives high biodiesel yield and purity. Molecular motions such as ion migration or dipole rotations and rapid heating can be brought about by microwaves without altering the molecular structure, as the mixture of vegetable oil, methanol, and sodium hydroxide contains both polar and ionic components. Microwave heating compares very significantly with conventional methods, where heating may be relatively slow and inefficient since transferring energy into a sample depends broadly upon convection currents and the thermal conductivity of the reaction mixture (Koopmans et al., 2006). Microwave-assisted trans-esterification of different feed stocks such as rapeseed oil, cotton seed oil and waste cooking oils has been reported by several researchers (Demirbas, 2007; Alnuami et al., 2014).

The aim of this study is to prepare and characterize biodiesel from pure and waste cooking sunflower oil. We have also studied the optimum conditions for highest biodiesel production by conventional and microwave methods.

## Materials and methodology of biodiesel production

## Samples and biodiesel preparation procedure

In this study, conventional and microwaveassisted transesterification methods were investigated for biodiesel production from both Waste cooking sunflower oil (WCSO) and pure sunflower oil (PSO). In the case of conventional method, WCSO was purified through filtration and the sample was placed in 250ml bottom round flask with a reflux condenser. The flask was placed on an electric heater with a temperature controller and magnetic stirrer as shown in Figure 1. Sodium hydroxide pellets (wt% of oil) has dissolved in required amount of methanol. The transesterification reaction was performed at different volumetric ratio of oil to methanol, varying from 4:1, 5:1, 6:1, and 8:1. The reaction time was kept constant at 1 hour for all experiments. The transesterification reaction for PSO was also performed at the same volumetric ratio of oil to methanol as WCSO. The catalysts NaOH and KOH have been used (% wt. of oil). The reaction was carried out at  $60 \,^{\circ}\text{C} \sim 65 \,^{\circ}\text{C}$  for 30, 40, 50 and 60 minutes with vigorous stirring. Then, biodiesel and the mixture of glycerin, residual methanol and catalyst were separated from the reaction product.



Figure 1: Conventional transesterification process

On the contrary, in the case of microwave irradiation method, satisfactory transesterification of both WCSO and PSO was achieved in a short time with the same oil to alcohol volumetric ratio. The microwave-assisted transesterification was performed using a modified domestic microwave oven (with an output power of 800 W) as illustrated in Figure

2. The microwave oven was modified and fitted with a water-cooled reflux condenser. The reaction was carried out at different power such as low, medium low, medium high, high for 1 to 6 minutes. Then the product was separated in a process which is the same as conventional method.



Figure 2: Modified domestic microwave assisted transesterification

#### Determination of different properties of oil

**Results and Discussion** 

A wide range of standard methods were used to determine the various properties of biodiesel. The specific gravity, viscosity, flash point, pour point, cetane number and ash content of the biodiesel were measured by the standard method IP 160/57, ASTM D 445, ASTM D 93, ASTM D 97, ASTM D 613 and ASTM D 482, respectively. The moisture content is the amount of water present in the oil calculated by-Moisture content (%) =  $(W_1-W_2)/W_1 \times 100\%$ 

Where,  $W_1$  and  $W_2$  are the weights of oil before and after drying. The calorific value of the oil was determined by bomb calorimeter.

# Effects of different parameters on biodiesel yield

#### Effect of volumetric ratio of oil to methanol

The experimental investigation was carried out at different volumetric ratios of 4:1, 5:1, 6:1 and 8:1, using 1% wt of NaOH catalyst at a constant temperature of 65  $^{\circ}$ C. The biodiesel yield decreases with increasing volumetric ratio as depicted in Figure 3.



Figure 3: Effect of volumetric ratio of sunflower oil to methanol on biodiesel yield

With the increase in volumetric ratio, the volume of oil increases; however methanol decreases as percentage of above oil to methanol ratio. As a result, biodiesel yield decreases at higher volumetric ratios. In this experiment, the best biodiesel yield was observed in a volumetric ratio of 4:1. In a comparison between the microwave assisted and conventional method, it was found that the microwave method produced higher percentage of biodiesel than the conventional method. In contrast, WCSO yielded highger amounts of biodiesel compared to PSO.

### Effect of Different catalysts

In this research, NaOH and KOH were used as catalyst in order to investigate the influence of

catalysts on biodiesel yield. The experiment was carried out in the best volumetric ratio of oil to methanol 4:1, temperature 65 °C and reaction time 4 minutes. The use of NaOH showed the highest percentage of biodiesel yield than KOH for both microwave-assisted and conventional method as illustrated in Figure 4. The yield percentage of biodiesel in case NaOH from PSO in the both methods was exactly the same while from WCSO the amount slightly decreases in conventional methods. On the other hand, in conventional method for KOH catalyst the biodiesel production from both WCSO and PSO decreases than the microwave-assisted method.



Figure 4: Effect of catalysts on biodiesel yield

Effect of different concentrations of catalyst

In figure 5, it is clearly show that the biodiesel yield due to the use of KOH catalyst continuously rises from about 85 to 94% when the catalyst concentration increases from 0.5% wt to 1.5% wt. However, the yield production is not as significant as NaOH because in case of NaOH it could be possible to obtain up to 98% of biodiesel. Based on the results of this investigation, it was found that 1.0% wt of NaOH or KOH contributed for the best yield to biodiesel production among 0.5%, 1.0% and

1.5%. Normally, when the amount of catalyst increases, it helps to increase the speed of the reaction and gives better yield. However, every reaction has its optimum catalyst concentration value. Beyond that value, excessive catalyst for example NaOH or KOH takes part in saponification which reacts with triglyceride to form soap and water and to reduce the biodiesel yield. In this case, the fatty ester yield increased with the increase of concentration of catalyst until1.0%. Clear of that value, the fatty ester yield started to drop above the 1.0% of catalyst concentration.



Figure 5: Effect of different concentrations of catalyst on biodiesel yield

## Effect of Alcohol

Both methanol and ethanol were used to investigate the consequence on biodiesel production. The results from different volumetric ratios exhibited that the highest percentage (almost 97%) biodiesel yield was obtained by using methanol. The production of biodiesel by using ethanol in alkali-catalyzed transesterification has found to be more difficult than that by using methanol. This was due to the formation of stable emulsion during ethanolysis. In contrast with this, for methanolysis, the emulsions formed would break down easily to form a lower glycerol rich layer and upper methyl ester rich-layer. While in ethanolysis, the emulsions formed were more stable due to the presence of larger non-polar group in ethanol, making the separation and purification of biodiesel more difficult. In this study, the biodiesel yield from ethanolysis was lower than from methanolysis.

## Effects of Reaction Time

The experiments were carried out with 1 wt% of NaOH catalyst, a microwave power of 800

W, oil to methanol volumetric ratio of 4:1, and various reaction times (1-6 min) to examine the effects of reaction time on biodiesel yield. As shown in Figure 6, the yields of sunflower biodiesel were 33, 47, 79, 97, 91 and 85% from WSCO for reaction times of 1, 2, 3, 4, 5, and 6 min, respectively. The Figure also illustrated that the yields of sunflower biodiesel were 32, 47, 78, 95, 90 and 86 % from PSO for the same reaction times of 1, 2, 3, 4, 5, and 6 respectively. An increase in reaction time from 1 to 4 min caused a significant increase in biodiesel yield, which then decreased with a further increase to 6 min. This was due to the incomplete transesterification reaction between methanol and oil and the longer reaction had a higher reaction temperature, resulting in greater solubility of glycerin. In this research, the result showed that microwave heating had a significantly shorter reaction time than conventional heating.



Figure 6: Effects of reaction time (min) on the yield of sunflower biodiesel at a constant medium high power of microwave

#### Effects of Microwave Power

This research was carried out with 1 wt% NaOH catalysts, a reaction time of 4 min, oil to methanol volumetric ratio 4:1 and various microwave powers to investigate the effect of microwave power on biodiesel yield. As revealed in Figure 7, the yields of sunflower biodiesel were 93, 94, 95, 96 and 97% for pure sunflower oil because of Low, medium low, medium high, high, respectively microwave power whereas that values for waste cooking sunflower oil were 91, 93, 94, 95 and 96% for the same microwave power. It was clearl that the yields of biodiesel increased with increasing of microwave power.



Figure 7: Effect of power at reaction time 4 min and oil to methanol volumetric ratio 4:1

# *Effect of Reaction Time and Temperature on FAME*

To investigate the influence of reaction time and temperature on the progress of Fatty acid methyl ester (FAME) conversion process, the reactions were carried out at different temperatures between 27°C to 65°C as displayed in Figure 8. The other conditions for this experiment were oil to methanol volumetric ratio of 4:1, catalyst concentration of 1 (wt% oil) and constant stirring rate. The temperature had detectable effect on the ultimate conversion to ester. However, higher temperature decreased the time necessary to reach maximum conversion.



Figure 8: Effect of reaction time at different temperatures on FAME yield

The optimum temperature for obtaining the maximum amount of FAME was 65°C for 1 h while at a lower temperature of 27 °C, the process was incomplete.

## Relationship between viscosity and time

Figure 9 completely showed that the viscosity of biodiesel for all conditions decreases with increase of reaction time. As illustrated in Figure 9, the viscosity was higher at 1 minute; this is due to the incomplete transesterification reaction between methanol and oil. Conversely, viscosity decreases at reaction time of 4 minutes due to the complete transesterification reaction. The viscosity obtained from conventional method was higher at reaction time of 1 minute while slightly lower at a time of 3 minutes compared to microwave-assisted method.



Figure 9: Viscosity of biodiesel at different time

# Comparative study of physiochemical and elemental properties

The physiochemical properties of biodiesel include a wide range of parameters like specific gravity, viscosity, calorific value, flash point, pour point, fire point, cetane number etc. It is essential to characterize the biodiesel obtained from both WCSO and PSO and to compare with standard biodiesel or conventional diesel for use it in engine. The properties of biodiesel from WCSO and PSO for both microwave assisted method and conventional method are presented in Table 1. The specific gravity and viscosity was exactly the same as standard biodiesel (Table 1). The flash point and the fire point of biodiesel from both WCSO and PSO in microwaveassisted method was between the range 100~170 for standard biodiesel. On the other hand, this value for both WCSO and PSO in conventional method was below the lower limit of standard biodiesel. The high calorific value (62-65 MJ/ Kg) of the obtained biodiesel specifies its high heat content. Moreover, the most important properties of biodiesel, the cetane number had a high value ranging from 49 to 62 indicating the easiness to start the engine.

	Microwave method		Convention	Stan dand	
Properties	Biodiesel (from PSO)	Biodiesel (from WCSO)	Biodiesel (from PSO)	Biodiesel (from WCSO)	biodiesel
Specific gravity (at 150C)	0.88	0.84	0.86	0.88	0.84~0.88
Viscosity (at 400C)	4.768	3.984	4.608	4.336	2.5~6.0
Flash point (oC)	147	143	42	58	100~170
Fire point (oC)	150	145	47	64	100~170
Moisture content	Nil	Nil	Trace amount	0.01%	0.05% max.
Pour point (oC)	-6	-7	-8	-10	-10 ~ -15
Calorific value (MJ/Kg)	65	62	65	62	-
Cetane number	62	58	49	50	-
Ash content (%)	0.008	0.0059	0.0121	0.018413	-

## Comparative investigation of compositional analysis

## Study of Infrared Spectroscopy

A Fourier transform infrared spectroscopy (FTIR) is a qualitative method that identifies the functional groups and bands that correspond to bending or stretching vibration in the oil and biodiesel samples. Changes of the carbonyl functional group to the metoxy carbonyl group indicate that the transesterification reaction has occurred and the FAME has been formed. In this study, the formation and loss of functional groups between sunflower oil and FAME were identified using FTIR. Figure 10 and Figure 11 revealed the FTIR spectra of the biodiesel obtained from sunflower oil in both microwave and conventional method.

The sunflower oil absorption peak was identified at 1098.7 cm<sup>-1</sup> and this indicated the C-CH<sub>2</sub>-O

vibration. The FAME spectrum showed a peak at 1198.4 cm<sup>-1</sup>, which can be attributed to the O-CH<sub>3</sub> initial methyl group stretch and one peak at 1437.1 cm-1 originated from the -CH, asymmetric bending vibration. Based on the appearance of the FAME peaks, the transesterification of the oil occurred, thereby forming the methyl molecule as a product. The FAME peaks identified at 1198.4 cm<sup>-1</sup> and 1437.1 cm<sup>-1</sup> were very comparable to that of the waste cooking oil. The band that occurred between 3103.5 cm-1 and 3644.6 cm-1 showed the overtone of the ester functional group. The most important peaks appeared at 1198.4 cm-1 displaying the initial formation of the methyl group (O-CH<sub>3</sub>), and at 1437.1 cm<sup>-1</sup> indicated the asymmetric bending vibration of CH<sub>3</sub>. Furthermore, a C-H stretching vibration of cis-double bond (=CH) was identified between 3103.5 cm-1 and 3644.6 cm-1.



Figure 10: Comparison of sunflower oil spectrum and FAME spectrum produced at 1 wt% NaOH, 4:1 oil to methanol volumetric ratio using WCSO in conventional method



Figure 11: FTIR spectrum of biodiesel obtained from WCSO in microwave method

The band in 3008.95, 1244.09 and 721.38 cm<sup>-1</sup> were correspondent to vibrations of elongation and roll out of plane deformation of the functional group -C=C- in the cis position respectively. On the contrary, the FTIR spectra identified the band 1741.72 cm<sup>-1</sup>, 1168.86 cm<sup>-1</sup>, and 1460.11 cm<sup>-1</sup>, corresponding to a stretching vibration of -C=O group of the ester, stretching vibration -CO-group of methyl ester and stretching vibration of CO<sub>2</sub>- group respectively. Besides, the band at 1435.04 cm<sup>-1</sup> was corresponding to deformation vibration of -O-CH<sub>3</sub> group of methyl ester.

#### Nuclear Magnetic Resonance

The Proton Nuclear Magnetic Resonance (NMR) spectra for pure sunflower oil and pure biodiesel showed a similar chemical group composition. However, the differences between the spectra indicated the presence of more saturated molecules in the biodiesel when compared to pure sunflower oil. The position of every peak on the ppm scale of the spectrum is an indication of the position of protons in the chemical structure associated with each peak, and the integration values under each peak. The integration provides the relative chemical group composition. If methylium (CH<sub>2</sub>) is regarded as a point of reference, whose peak is represented approximately between 0.6 and 0.8, the integral must be divided by 3 to represent the 3 protons in methyl. The number of carbons was estimated using the proton spectrum by identifying the structure of the compound represented by each peak according to the chemical structure of the free fatty acids that make up sunflower oil. Since the peak between 0.6 and 0.8 ppm represents methylium (CH<sub>2</sub>) for every 3 protons, in which there is 1 carbon in the chain. The peaks between 1.5 and 3 ppm are characteristic of methylene

 $(CH_2)$  therefore, for every 2 protons, there would be 1 carbon in the structure. For the rest of the peaks, carbon content of 1:1 was assumed as carbon hydrogen (CH).

The NMR results gave a reasonable estimation, since each fatty acid present in the sunflower oil contains approximately 18 carbon atoms, thus in a triglyceride, the values obtained for the number of protons and carbons would be multiplied by 3. From the NMR analysis of biodiesel, it was highlighted that the glycerol, initially present in the pure sunflower oil between 4 and 4.5 ppm was no longer present in all the samples of biodiesel. This was an indication of triglyceride break down into smaller chains. From the spectrum, an estimation of the average number of carbon and hydrogen atoms in the test specimen can be established. This was done by standardizing the NMR integration to the methyl ester resonance.

### UV-Vis Study

UV-Vis spectroscopy corroborated the presence of conjugated double bonds present in the respective FAME biodiesel obtained. Figure 12 and Figure 13 showed the corresponding spectra of biodiesel from both WCSO and PSO in microwave method. It was appreciated that the signals 260, 254 and 248 nm were the same in the two products without significant variations correspond to transitions and  $\pi$ - $\pi$ \* between 250-260 nm of the double bonds and n-transitions of the presence of  $\pi^*$  of the carboxylic acids. The most significant variation between both products was shown where two signals were noted at 228 and 224 nm which can be explained by transitions  $\pi$ - $\pi$ \* of carboxylic acids  $\lambda$  and unsaturated.

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Figure 12: UV-Vis spectrum of biodiesel obtained from WCSO in Microwave Method



Figure 13: UV-Vis spectrum of biodiesel obtained from PSO in Microwave Method

## Cost Analysis of the biodiesel production methods

The cost of biodiesel production is one of the most important factors to its commercial application. In this research, biodiesel production in both conventional and microwave methods were analyzed. In this case, the cost variables, i.e., electricity cost, catalyst cost, methanol cost and sunflower cost, were considered for the analysis. It was estimated that approximately 1.12 L of sunflower oil, 0.280 L of methanol and 0.01008 kg of NaOH were required to produce 1 L of biodiesel. All the associated costs were considered in Bangladeshi Taka (BDT) and presented in Table 2. The total cost for biodiesel

production in microwave method was BDT 94.7 and that in conventional method was BDT 120.7, almost 27.46% higher than the microwave method. The microwave method offered a fast and easy route for biodiesel production as it saved considerable amount of reaction time.

Method	Reaction Time (min)	Cost of electricity (BDT)	Cost of catalyst (1.0 wt %) (BDT)	Cost of methanol (BDT)	Cost of sunflower oil (BDT)	Yield (%)	Total cost (BDT)
Microwave system	4	8.64	13.1	35.1	35	97	94.7
Conventional system	60	32.6	13.1	35.1	35	95.8	120.7

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Table 2: The	cost-effectiveness	of synthesis	of sunflower	biodiesel

## Conclusion

The experimental result showed that basecatalyzed transesterification is a promising area of research for the production of biodiesel. The yield of sunflower biodiesel also increased with increasing reaction power, although the microwave output must not be too high as it may damage the organic molecules. An increase of reaction time caused a substantial increase in biodiesel yield up to optimum value and then decreased because of incomplete transesterification reaction between methanol and oil. The best sunflower biodiesel was 97% under the following reaction conditions: 1% wt of NaOH catalysts, oil to methanol volumetric ratio of 4:1, reaction time 4min (medium high) in microwave system. The final properties of biodiesel from pure sunflower and waste cooking sunflower oil by transesterification method (microwave and conventional) were compatible to the standard values. Experimental results indicated that the reaction time reduced significantly and the yield of sunflower biodiesel improved using microwave system. Microwave system can achieve better performances when compared with conventional heating.

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