Improvement of Biodiesel Transesterification Efficiency by Activated Carbon and Microwave Power*

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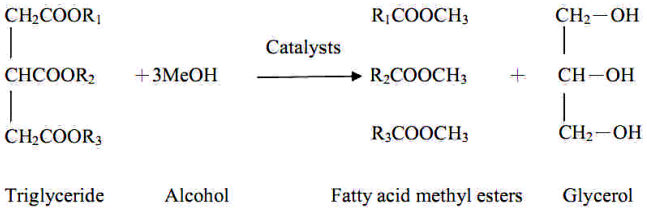
Abstract - ***In this research, the transesterification process to produce biodiesel from waste cooking oil was enhanced by microwave. The positive effect of microwave was even reinforced by the presence of a suitable added amount of activated carbon which helped to increase the microwave absorptivity of the whole reaction system. NaOH was added as the alkaline catalyst for the reaction. The optimum reaction conditions were found as the following: methanol to oil molar ratio 7:1, added NaOH concentration 1.0wt%, added active carbon amount 1.0wt%, microwave power 200W, reaction temperature 65^{o}C and reaction time 120s. The conversion rate of waste cooking oil into biodiesel in these conditions was up to 97.8% which was higher than the Taiwan CNS 15072 standard value of 96.5%.***

Keywords: ***Microwave irradiation, Transesterification, Activated carbon, Biodiesel***

I. INTRODUCTION

The amount of global available fossil fuels is reducing and human being is gradually running out of such fuel types. Therefore, the development of renewable energy resources is of high necessity. Biodiesel, one of those resources, which is defined as the fatty acid methyl esters (FAMEs), is made by transesterification of cooked vegetable oil or waste cooking oil. As compared to conventional petroleum diesel, the green-fuel biodiesel possesses the following many advantages: 1) be an alternative fuel for petroleum diesel; 2) possibly be locally produced; 3) be biodegradable and non-toxic; 4) decompose with almost no emissions of sulfur and aromatic compounds, thereby helping reduce environmentally harmful emissions, especially SOx, greenhouse gas emissions (carbon dioxide); 5) help secure a market share for motor biofuels of the total motor fuel consumption; 6) fallow agricultural land can be effectively utilized to plant the crops for biodiesel production and improve the agricultural economies, as agreed in the European Agricultural Policy regulations; 7) enhance the lubricity of diesel engines, extending engine life; and 8) improve combustion due to its high cetane number and ignition [1-2]. In Scheme 1, triglyceride is the main component of vegetable oil. When triglycerides react with an alcohol, the three fatty acid chains which are liberated from the glycerol skeleton combine with the alcohol to yield FAMEs and glycerol is produced as a by-product [3].

Different processes can be used to synthesize biodiesel, such as transesterification, blending, micro emulsions, and pyrolysis. Out of these, transesterification is the most commonly used method which refers to a catalyzed (base-catalyzed or acid-catalyzed) chemical reaction involving a triglyceride and an alcohol to produce biodiesel (i.e, FAMEs) and glycerol. During the alkaline process, the high purity and high yield of biodiesel can be obtained within 30~60 minutes [2,4,5] and the by-product glycerol is also formed [6,7]. In the current commercial biodiesel manufacturing process, the most common solution for reactions is the use of alkaline catalysts. However, executing such process requires high energy consumption and more water for washing stage, thereby causing difficulties in the recovery of glycerol. In addition, when the content of free fatty acids (FFAs) is higher than 0.5%, which cannot be fully converted into biodiesel, it will cause saponification phenomenon. In such that case, it is suggested that acidic catalysts be used [8,9]. In the acid–catalyzed mixture, the esterification process and the transesterification reaction may take place at the same time. Therefore, the raw materials with the presence of high FFAs (such as waste cooking oil and animal fats), which are much cheaper than refined oil, can be directly used with acid catalysts [10]. The acids catalyze FFAs esterification which produces FAMEs and increases the biodiesel yield. However, the reaction time is very long (48–96 h) even at reflux of methanol and a high methanol to oil molar ratio (30– 150:1 by mol) and high acid catalyst concentration are required [11–13]. In order to significantly shorten the long reaction time, such method of acid-base two-stage catalytic process could be used [14].



Scheme 1. *The transesterification of triglyceride with alcohol.*

Microwave irradiation activates the smallest degree of variance of polar molecules and ions in the mixture of vegetable oil, potassium hydroxide and methanol. The polar and ionic components interact with molecular dipoles and charged ions, making the molecules and ions rotate rapidly. Heat is generated due to molecular friction. Microwave irradiation thus enhances the chemical reaction and increases yield faster. When the reaction is carried out under microwave irradiation, the reaction time of transesterification decreases and high-purity biodiesel is obtained with reduced energy consumption [15-17]. According to Hsiao et.al [17], the combination of ultrasonic mixing (within the first 1 min) and closed microwave irradiation (within the successive 2 min) can considerably accelerate the transesterification. Under this condition, the conversion rate is 97.7%, the

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amount of catalyst used is 1.0 wt%, the reaction temperature is 65°C, and the methanol/oil molar ratio is 6:1. Hence, microwave irradiation accelerates the chemical reaction, resulting in a high yield within a short time [18,19].

The materials which interact with microwaves to produce heat are called microwave absorbers. The ability of a material to be heated in the presence of a microwave field is defined by its dielectric loss tangent ($\tan\delta=\varepsilon''/\varepsilon'$). The dielectric loss tangent is composed of two parameters, the dielectric constant (or real permittivity), ε', and the dielectric loss factor (or imaginary permittivity), ε'', as shown in Eq. (1):

$$\varepsilon = \varepsilon' - i\varepsilon'' \quad (1)$$

where ε is the complex permittivity. The dielectric constant (ε') determines how much of the incident energy is reflected and how much is absorbed, while the dielectric loss factor (ε'') measures the dissipation of electric energy in form of heat within the material [20]. For optimum microwave energy coupling, a moderate value of ε' should be combined with high values of ε'' (and thus, high values of $\tan\delta$), to convert microwave energy into thermal energy. Therefore, while some materials do not possess a sufficiently high loss factor to allow dielectric heating and are considered transparent to microwaves, other materials, e.g. some inorganic oxides and most carbon materials, are excellent microwave absorbers. On the other hand, electrical conductor materials reflect microwaves. For example, graphite and highly graphitized carbons may reflect a considerable fraction of microwave radiation [21]. The aim of this study is to examine the effect of activated carbon on the transesterification of waste cooking oil assisted by microwave irradiation. Several previous studies [5,7,22–28] have proved that the following factors: methanol/oil molar ratio [5,7,22–26,28], amount of catalyst [5,7,22,23,25,27,28], reaction temperature [5,7,22,23,25,27,28] and reaction time [5,7,28] were all important factors influencing the transesterification process. Accordingly, this study also considers these factors in the analyzing process.

II. METHOD AND EXPERIMENT SETUP

A. Materials

Acetic acid, Methyl laurate and hexane, Active carbon (20~60 mesh) were purchased from Fluka Co. Ltd. Sample of waste cooking oil was provided from local restaurant. Acid value (AV) [KOH mg/g] and saponification value (SV) [KOH mg/g] of the waste cooking oil were first determined by the standard titration method (AOCS: American Oil Chemists' Society), then they in turn were used to calculate the molecular weight (MW) of the oil. The experimental results of AV, SV and MW were 0.67, 208.91 and 805.75, respectively. The reaction process was catalyzed by Sodium hydroxide (NaOH) (99% purity) and methanol (99.8% purity) purchased from Shimakyu Co. Ltd. (Japan).

B. Equipment

A commercial closed type microwave apparatus (Milestone ETHOS 900), which consists of a continuous 2.45GHz microwave power delivery system with power output from 0 to 900W and a temperature and time setting component, was used to assist fully all the experimental reactions. A sample analyzer Perkin Elmer GC Clarus 600 machine including a capillary column (SPBTM-WAX, 30m × 0.75m × 1.0μm) and a flame ionization detector (FID) was used to analyze the experiment samples.

C. Experimental procedure

Triglycerides were firstly converted into esters with methanol using a NaOH. The reaction of the reagents was assisted by microwave irradiation. The spherical glass reactors had volumes of 250ml that were modified by introducing a water condenser for conducting atmospheric pressure experiments. With ice water running through the condensation pipe of the microwave accelerated system, 100g waste cooking oil sample was added into the special 250ml three-neck reaction flask, and mixed with catalyst, activated carbon and methanol of the relevant ratio. The speed of the electromagnetic stirrer was 350rpm. The mixture was automatically irradiated with microwaves in a pre-setting procedure (the average power output of microwaves is 600W). According to the experimental design, the transesterification process was tested at five levels of reaction time (30, 60, 90, 120 and 150 sec), five levels of methanol/ oil molar ratio (5, 6, 7, 8, and 9), five levels of used amount of catalyst (0.5, 1, 1.5, 2 and 2.5 w/w% of oil), five levels of amount of used activated carbon (0.5, 1, 1.5, 2 and 2.5 w/w% of oil), eight levels of microwave power used (50, 100, 200, 300, 400, 500, 600 and 700W) and five levels of reaction temperature (50, 55, 60, 65 and 70 °C).

D. Analytical methods

The top layer of the mixture, which was obtained after the reactions finished, contained mainly FAMEs. The FAMEs were removed in a separating funnel after settling for half an hour and then were washed at least three times with deionized water before being dried in an oven at 105 ± 3°C. Sample analysis was done by using Perkin Elmer GC Clarus 600 machine. The sample weight was 0.5g including 0.05g methyl laurate (acting as an internal standard) and 10ml of hexane. Reagents in the sample were fully mixed and then 1μl of the mixture was injected into the gas chromatography analyzer under the following conditions: carrier gas, nitrogen, with flow rate of 45ml/min; air flow rate of 450ml/min; split ratio, 1:20; injection temperature, 280°C; detector temperature, 300°C. The oven at first kept the temperature at 210°C within 4 minutes, then increased the temperature gradually at the rate of 4°C/min and finally held the temperature at 240°C within 8 minutes. The conversion rate of crude biodiesel was calculated according to the area of FAME as expressed in the following Eq. (2):

$$\text{Conversion rate}(\%) = \frac{(\text{area of FAME/area of reference}) \times \text{weight of reference}}{\text{weight of crude biodiesel}} \times 100\% \quad (2)$$

III. RESULTS AND DISCUSSION

Fig.1 shows the effect of methanol/oil molar ratio, which ranged from 5:1 to 9:1, on the biodiesel conversion rate. Reaction conditions were: added amount of catalyst 1.0wt%; added amount of activated carbon 1.0wt%; microwave power 600W; reaction temperature 65°C and reaction time 120s. It could be seen that at alcohol/oil molar ratio lower than 7:1, after 120s, the conversion rate variation was proportional to the increment of the molar ratio. It reached to the maximum value of 88.93% when the methanol to oil molar ratio was at 7:1. Meanwhile, with the molar ratio higher than 7:1, the conversion rate dropped down from the maximum value instead of rising more since the excess of methanol diluted the concentration of the catalyst in the reaction system, resulting in the less opportunity of contact between the catalyst and oil. Therefore, the most suitable alcohol/oil molar ratio in the scope of the experimental study was 7:1.

The effect of different added amount of activated carbon on the conversion rate of biodiesel can be revealed in Fig.2. Reaction conditions were: methanol/oil molar ratio 7:1; used amount of catalyst 1.0wt%; microwave power 600W; reaction temperature 65°C and reaction time 120s. The figure shows that the increment from 0.5wt% to 1.0wt% of activated carbon addition did help enhance the conversion rate as the activated carbon could assist the absorption of the microwaves. As a result, the reactants could be quickly and uniformly heated to 65°C. However, when the added amount of activated carbon was increased to 1.5 wt% up, the conversion rate began to decline because too much added activated carbon would lower down the degree of mixing of oil with alcohol, and thus the desired reactions were impeded. Therefore, the best added amount of activated carbon should be 1.0wt%.

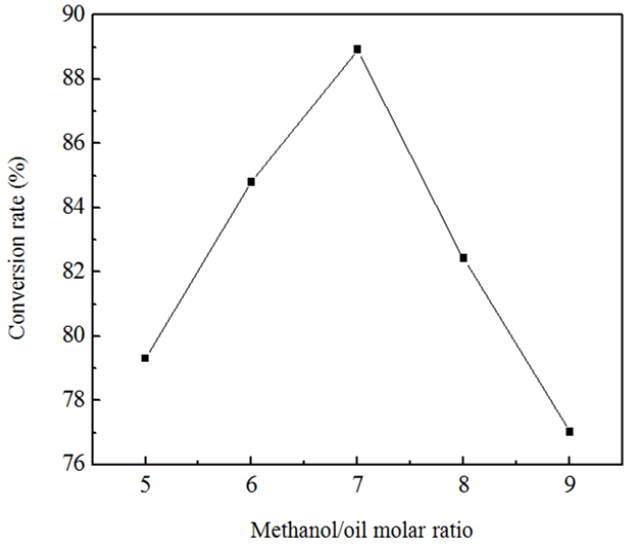


Figure 1. *Effect of the methanol/oil molar ratio on the conversion rate of waste cooking oil assisted by microwave irradiation and catalyzed by NaOH*

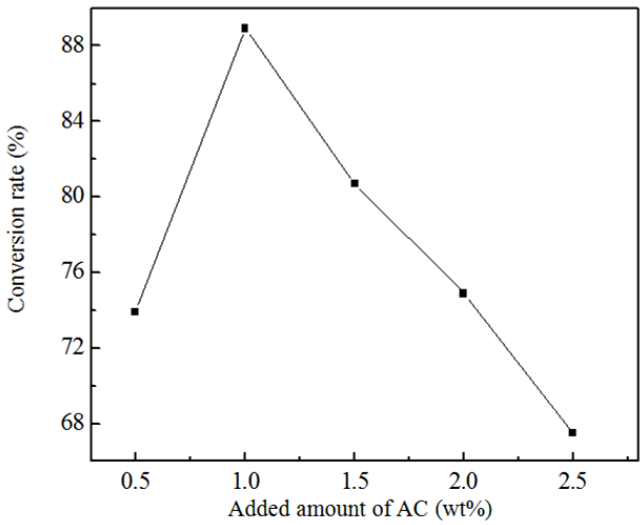


Figure 2. *Effect of the amount of activated carbon used on the conversion rate of waste cooking oil assisted by microwave irradiation and catalyzed by NaOH*

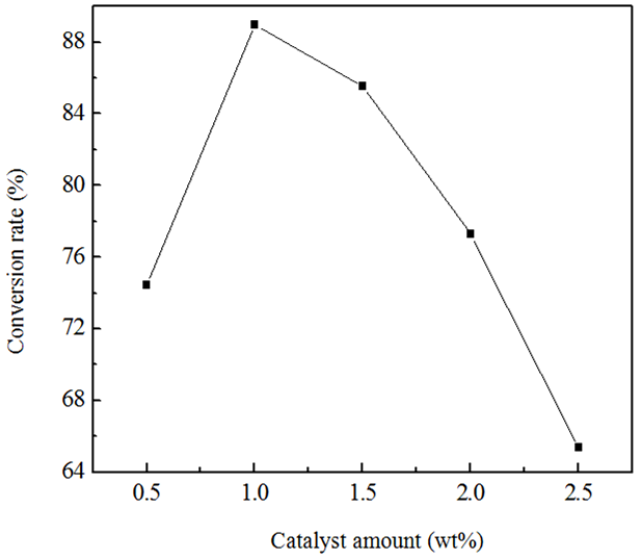


Figure 3. *Effect of the amount of catalyst used on the conversion rate of waste cooking oil assisted by microwave irradiation and catalyzed by NaOH*

A similar variation of conversion rate happened to the relationship between the conversion rate and the amount of added catalyst in the reaction system (see Fig.3). Reaction conditions were: methanol/oil molar ratio 7:1; used amount of activated carbon 1.0wt%; microwave power 600W; reaction temperature 65°C and reaction time 120s. Under these conditions, the best conversion rate was obtained when the added amount of NaOH was at 1.0wt%. The rate reduced on both trends of decreasing and increasing the catalyst amount. It was because the decrease of NaOH amount to lower than 1.0wt% led to the reduction of the contact area between the catalyst and reactants and in this case the desired reactions required a longer reaction time. On the other hand, with further addition of catalyst (to more than 1.0wt%), a slight downward trend of the conversion rate of

biodiesel occurred mainly because the excessive alkali catalyst brought about saponification reaction, making a gelatinous substance or even eventually a colloid. The appearance of this colloidal state caused the viscosity of the reactants to increase and hindered the reaction from taking place. Another bad side effect of this phenomenon was that the separation of the final product was more difficult and required larger amount of water for the washing step after the reaction. Accordingly, 1.0wt% added NaOH amount should be considered as the best choice.

In order to tell how much microwave power should be applied, experiments in the following conditions were conducted: methanol/oil molar ratio 7:1; amount of catalyst used 1.0wt%; amount of activated carbon used 1.0wt%; reaction temperature 65°C and reaction time 120s. Fig.4 shows that a increment of microwave power from 50W to 200 W did make the biodiesel conversion rate increase very fast up to 97.80%. However, the microwave power more than 200W seemed to be unlike expectation as the biodiesel conversion rate dropped reversely proportional to the increment of the consumed power (e.g. to 95.25 percent at 300W and only 86.33% at 700W). The reason was that at the low power levels the reactant temperature relatively slowly reached to 65ºC whilst at the high power levels, the temperature rose to 65ºC so quickly. Recalling that the microwave oven was set to stop walking as long as the reaction system temperature reached to 65ºC, the too short or too long period of maintaining microwave effect on the reaction system both led to lower usefulness of the used power. Therefore, 200W could be considered as the optimal microwave power level under the above mentioned reaction conditions.

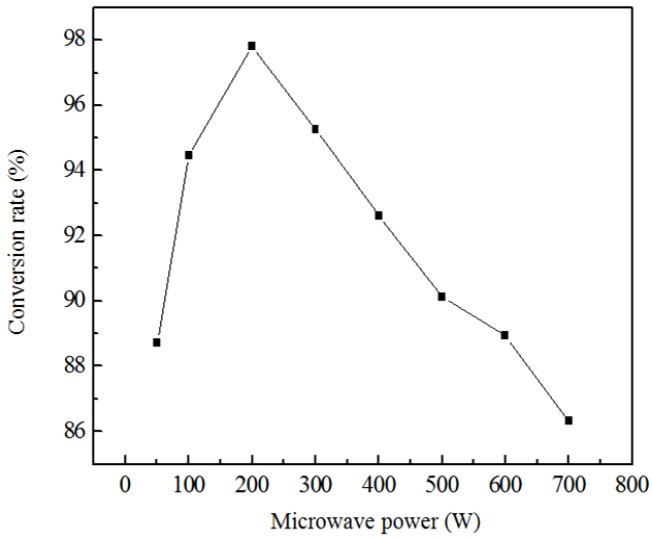


Figure 4. *Effect of the power of microwave used on the conversion rate of waste cooking oil assisted by microwave irradiation and catalyzed by NaOH*

The reaction time of the transesterification process was maintained to different periods in another test under the following reaction conditions: methanol/oil molar ratio 7:1; amount of catalyst used 1.0wt%; amount of activated carbon used 1 wt%; microwave power 200W and reaction temperature 65°C. With the help of microwave, only after about 30s did the biodiesel yield get to 89.57%. The yield kept increasing, although nonlinearly, and reached to the maximum value 97.80% within 120s reaction time. Nevertheless, a longer reaction time than 120s would not favor more the conversion rate but reduce it, e.g. 93.45% at 150s. The reason may be because the transesterification reaction was a reversible reaction, thus when the reaction time was too long, the conversion rate may become lower. As a result, the most suitable reaction time with the given experimental conditions was 120s.

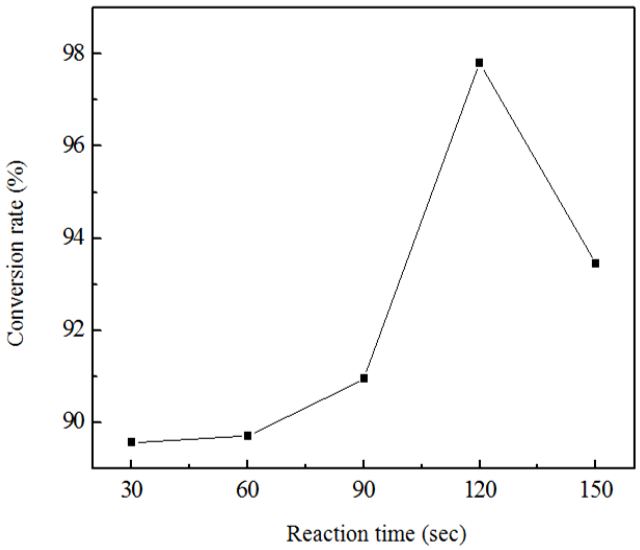


Figure 5. *Effect of reaction time on the conversion rate of waste cooking oil assisted by microwave irradiation and catalyzed by NaOH*

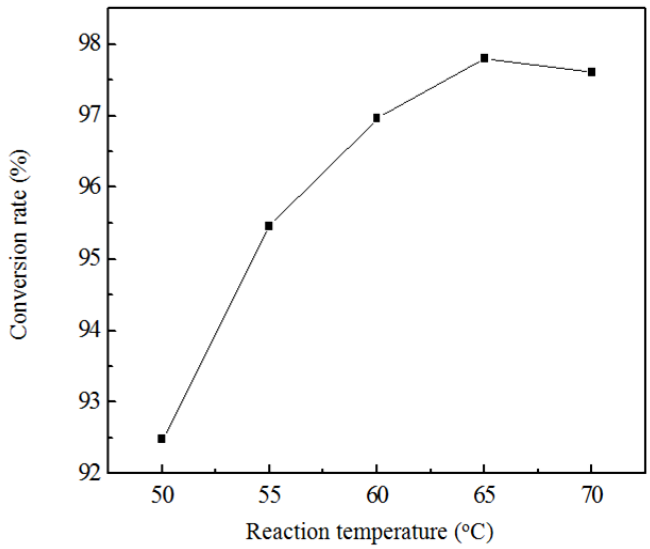


Figure 6. *Effect of reaction temperature on the conversion rate of waste cooking oil assisted by microwave irradiation and catalyzed by NaOH*

It was predictable that reaction temperature could affect on the final product yield. Fig.6 shows the variation of the biodiesel conversion rate according to the reaction temperature in the following preset reaction conditions:

methanol/oil molar ratio 7:1; amount of catalyst used 1.0wt%; amount of activated carbon used 1.0wt%; microwave power 200W and reaction time 120s. This figure indicates a critical value of reaction temperature 65°C at which the yield was the most (97.8%). In fact this was also a very close value to the boiling temperature of methanol (64.5°C). At the range lower than 65°C, the higher the temperature was, the higher the conversion rate was since the reaction system was more active due to higher internal disturbance. Meanwhile, at the range higher than 65°C, the evaporation of methanol from the reaction system caused the reduction of methanol amount present in the system, thus the conversion rate was down. This result was quite similar to the case that the methanol/oil was preset less than 7:1 (Fig.1). Basing on this result, the chosen reaction temperature was 65°C.

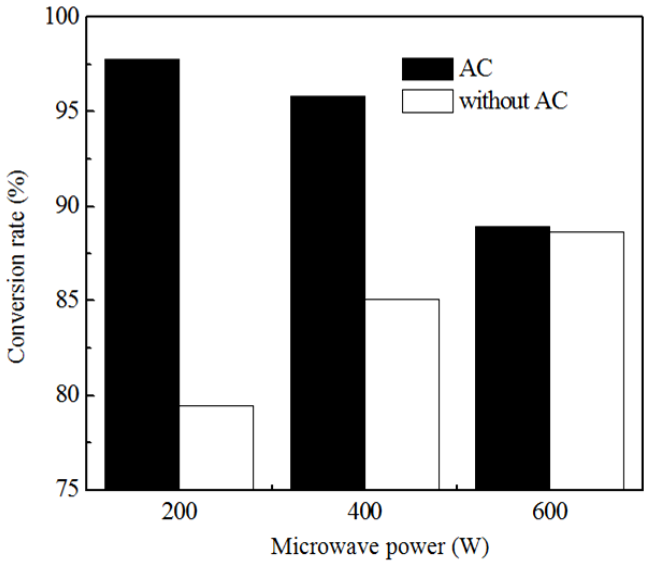


Figure 7. *Effect of the used amount of activated carbon and microwave power consumption on the conversion rate of waste cooking oil catalyzed by NaOH*

There is no doubt for the useful meaning of the presence of active carbon in this reaction. And the use of this material did not only help increase significantly the conversion rate but also reduce largely the required energy consumption of the microwave equipment. All these could be seen clearly in Fig.7, which reveals the change of the biodiesel yield according to the added amount of the activated charcoal under the following reaction conditions: methanol/oil molar ratio 7:1; amount of catalyst used 1.0wt%; amount of activated carbon used 1.0wt%; microwave power 200, 400 and 600W; reaction temperature 65°C and reaction time 120s. The first interesting result occurred at the low microwave power level (200W). The attendance of the activated carbon helped increase the conversion rate up to 97.80%, which was 18.33% higher than when the activated carbon was not used. This revealed that the activated carbon indeed could promote the absorptivity of the microwave power of the reaction system. The second interesting result was that at the 3-time higher microwave power level (600W), the conversion rate was almost the same (88.93% with activated carbon and 88.63% with no activated carbon) no matter the reaction system was with or without the activated carbon. The reason for these two results could be explained as follows. When the low power (200W) was applied, the heating rate was low, thus addition of activated carbon in the experimental system could significantly accelerated the reaction temperature. However, at high power level, the heating rate was significantly higher and the reaction system temperature reached to 65°C in almost the same short time with or without added active carbon. Thus the conversion rate was almost the same in these two cases. Finally, comparison of the results in Fig.7 showed that the highest conversion rate was achieved at the lowest microwave power level. This was obviously made by the activated carbon.

IV. CONCLUSION

According to the experimental result analyses, the main finding of this study is the ability of gaining higher biodiesel yield at low microwave power level by means of adding activated carbon into the experimental reaction system. Meanwhile, the addition of the activated carbon become unnecessary if the preset microwave power level is high enough. In this way, in terms of energy saving, the attendance of the activated carbon will not require high power consumption for the microwave equipment. Recalling that microwave irradiation assisted transesterification is more efficient than the conventional bath biodiesel production catalyzed by NaOH, the efficiency is even higher with this new method. An optimal set of reaction conditions has also been found as the following: methanol/oil molar ratio 7:1, amount of catalyst 1.0wt%, amount of activated carbon 1.0wt%, microwave power 200W, reaction temperature 65°C and reaction time 120s. The maximum conversion rate is 97.8% which is well higher than the limits prescribed by the EN-14214 standard.

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