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# Improving biodiesel yield of animal waste fats by combination of a pre-treatment technique and microwave technology

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ABSTRACT: Recently, due to its low cost there has been increased attention on Animal Waste Fats (AWFs) as a feedstock for biodiesel production. Advanced microwave technology has also been reported by many researchers to enhance the transesterification in biodiesel production. However, esterification of free fatty acids in the feedstock reported here has not attracted so much attention. AWFs come with its challenges namely, high free fatty acid (FFA) content and high water content. This study utilizes AWFs (tallow) containing very large amount of FFA; (25wt.%, 18 wt.%, and 9.4 wt.% FFA/AWFs) as feedstock for fatty acid methyl ester (FAME) production. A simple thermal pre-treatment technique followed microwave assisted esterification with methanol (MeOH) was conducted in a batch process to reduce the FFA content to as low as 1wt.% FFA, which is then suitable for the alkaline transesterification process. The pre-treatment of AWFs at 88°C to first reduce water and decrease viscosity, followed by an operating microwave power of 70W producing a power density 1.147mW/m$^3$, achieved a 15% increase in reduction of FFA over 30W microwave power and conventional thermal method. Under optimum conditions, using 2.0 wt.wt% sulphuric acid catalyst/AWFs and 1:6 molar ratio AWF/MeOH, the FFA conversion of 93wt. % was achieved. The results indicated that the pre-treatment and microwave application provided a faster route to high FFA reduction of AWFs during esterification process. The proposed technology is promising for the potential scale up industrial application.

*Keywords: Microwave, Free fatty acid, fatty acid methyl ester, animal waste fats, feedstock*

## 1. INTRODUCTION

The availability and sustainability of adequate supplies of less expensive feedstocks will, to a large extent, fast track the delivery of competitive biodiesel for commercial purposes. Waste fat, oil and grease (FOG) from industrial origin and sewers, and category 1 tallow are valuable alternatives feedstocks, replacing vegetable oils, though requiring some pretreatment for excellent biodiesel yield. These FOGs and tallow are generally difficult to handle because they are solid at room temperature, highly degraded and particularly have a high free fatty acid and water content which requires pretreatment for commercially acceptable conversion efficiency. Many studies have reported various feedstocks for biodiesel production, especially from low value plant oils, but there have been far fewer studies on animal fat. Amongst these Canakci and Van Gerpen [1] developed a technique to reduce the FFA level of high acid feed stock to less than 1.0 wt.% FFA with an acid catalysed pre-treatment using 20 wt.% palmitic and 40 wt.% palmitic acids creating acid values of 41.33 and 91.73 mg.KOH/g respectively. Canoira et al. [2], Ramadhas et al. [3], Encinar et al. [4] also reported acid esterification using mixtures of animal fat and plant oil.

The production of biodiesel involves the transesterification of a triglyceride, (TG) stock (vegetable or animal oil or fat) with excess methanol (MeOH) in the presence of an alkali catalyst such as sodium hydroxide (NaOH) to yield mixed FAME. Reaction rates and the yield of FAME has been extensively studied and the effect of variables such as MeOH: TG ratio, concentration of catalyst, temperature, etc. have been exhaustively examined and reviewed [5]. It is generally accepted that the presence of FFA in the feedstock adversely affects the reaction and particularly that soap formation renders efficient settling difficult if not impossible. The acid value must be reduced below 2 mg.KOH/g of oil for effective processing of the transesterification reaction [1, 6]. The presence of free fatty acid leads to soap formation in the presence of an alkali catalyst during transesterification reaction. Hence the importance of acid esterification process. To avoid this problem high FFA feedstock, usually waste oils and fats, are pre-treated with MeOH in the presence of an acid catalyst such as $H_2SO_4$ (sulphuric acid) thereby esterifying the FFA to FAME. Again, the reaction has been studied, though by no means as exhaustively as the biodiesel transesterification, though the effect of variables resembles that of that reaction as for example reported by

Gnanaprakasam. et al. [5], Chai et al. [7] , Mohammed and Bhargavi [8].

The decision to either progress with (a) one step- alkaline transesterification or two step –acid pre-esterification to reduce the FFA followed by a base-catlysed transesterification depends significantly on the initial quantity of FFA in the oil/fat substrate for biodiesel production. Extensive work has been carried out on the yield of biodiesel with respect to FFA content and it was found that transesterification would not proceed if FFA content in the oil were > 2 wt. % [3, 9].

The effect of microwaves in accelerating the rate of chemical reactions is well known. There have been a wide range of studies, though almost all small scale, on the effect of microwave heating on the transesterification reaction, generally indicating a positive correlation between microwave heating on rate and yield [10, 11]. Similarly, of the application of microwaves to enhance the rate or yield of the esterification is now attracting more attention [12-16]. Most studies have been at laboratory scale using a modified domestic microwave as energy source. It is difficult to compare these laboratory studies, as often there is insufficient detail to estimate parameters such as power density. However, more recently studies on the scale-up of the transesterification have started to appear to report that in a continuous flow reactor a high FAME content of 99.4 wt. % can be obtained in a short residence time of 1.75 min [17]. This process required an energy consumption of about a half of the conventional process and all properties of obtained biodiesel were in the range of EN/ASTM standard limit [18]. It seems reasonable to expect some advantages for the esterification reaction using similar conditions [13, 19]. This study aims to improve conversion efficiency and increase reaction rates of mixture of high FFA substrates in acid esterification process for biodiesel production from animal fat (tallow) using bespoke microwave methods.

## 2. METHOD

### 2.1 Material

Methanol, Optima LC/MS Grade of purity 99.9%, sulphuric acid A.R. Grade of purity 97.5%, ethanol absolute of purity 99%, p-cymene of purity 99+% and potassium hydroxide All reagents used were purchased from Fisher Chemical and Sigma Aldrich. Tallow samples containing 9.4 wt. %, 18 wt.%, 25 wt.% FFA/AWFs and pre-treated waste oils containing 17.9 wt.% FFA/AWFs were provided by Argent Energy UK Ltd. Water in oil test kit was purchased from Hach Company to determine the percentage water in the oil sample.

## 2.2 Experimental Procedures

The tallow was pre-heated in 250ml bottle on a water-bath at 50° C using a Fisher Scientific advanced hotplate stirrer. In typical experiments 50.0g of pre-heated tallow was placed in a round bottom flask and first methanol/AWF (10, 20 wt. %) and then $H_2SO_4$/AWFs (0.8, 1.0 and 2.0 wt. %) were added to the tallow, shaking vigorously but gently to avoid spill.

**T**he CEM Discover SP Microwave system with Explorer 12 Hybrid Auto sampler with reflux set up (open vessel) was prepared for the esterification reaction and all parameters were set as desired for the microwave methods, as shown in Figure 1. The conventional method employed a water bath with reflux set up (open vessel) for the esterification reaction, Figure 2.

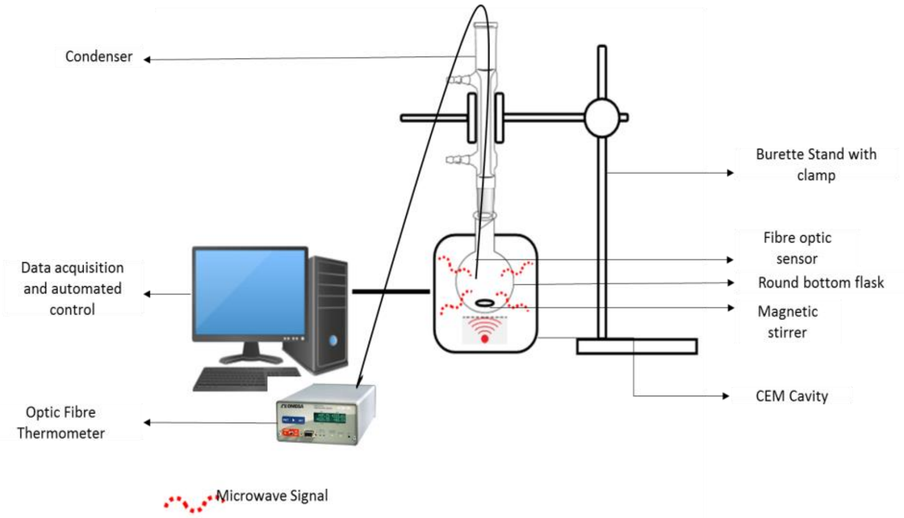

*Figure 1: Schematics diagram for CEM method*

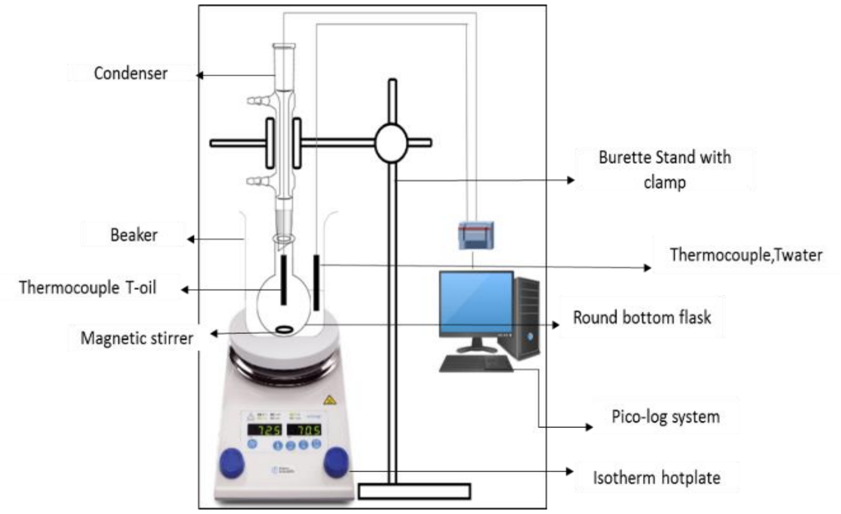

*Figure 2: Schematics diagram for Conventional method*

### *2.2.1. Non-pre-treatment (NPT)*

*(a) CEM method*

The flask was transferred to the CEM Discover SP microwave system with the following settings; temperature;73°C stirring power; high (300rpm) microwave power varied ; (30W, 70W). The Initial temperature of the sample was 38°C and microwave power assisted in ramping temperature from 38 °C to 73°C in 4-6 minutes. Once the desired operating temperature was attained, the microwave power reduces to as low as 0W.

*(b) Conventional method*

The following settings were applied; temperature 73°C, stirring power varied (300rpm). The initial temperature of the sample was 38°C and temperature gradually increases to 73°C after 30-40 minutes. The heating was adjusted to maintain reflux, once the desired operating temperature was attained.

### *2.2.2 Pre-treatment Option (PTO)*

*(a) CEM method*

The sample was first preheated at 88°C using microwave power with stirring power high (300rpm) to achieve a less viscous substrate and reduced water content.

Microwave power was varied (30W, 70W) and temperature ramped to 90°C in 4-6 minutes. Temperature was reduced to 73°C prior to addition of methanol and sulphuric acid solution.

*(b) Conventional method*

The sample was transferred to the conventional water bath system with the following parameters settings; temperature; 88°C and stirring power 300rpm. Initial temperature of the sample was 38$^{o}$C and temperature gradually increased to 88$^{o}$C in 40 minutes. The Isotherm advanced hotplate features a thermostat control system, which was used for resetting temperature to 73°C prior to the addition of methanol and sulphuric acid.

A summary of design of experiment for the study is presented in Table 1 and 2.

***Table 1: Matrix for design of experiment NPT method***

| Method | T (°C) | Molar ratio AWF : MEOH | Microwave Power (W) | Stirrer rpm | Reaction time | Acid catalyst (wt.wt%) |
|---|---|---|---|---|---|---|
| CEM | 73 | 1:3, 1:6 | 30,70 | 300 | 120, 60 | 0.8, 1.0, 2.0 |
| Conventional | 73 | 1:3 | 0 | 300-1000 | 150 | 0.8, 1.0 |

***Table 2: matrix for design of experiment PTO method***

| Method | Pre-treatment T (°C) | Operating T (°C) | Molar ratio AWF : MEOH | Microwave power (W) | Stirrer rpm | Reaction time | Acid catalyst (wt.wt%) |
|---|---|---|---|---|---|---|---|
| CEM | 88 | 73 | 1:6 | 70 | 300 | 120, 60 | 2.0 |
| Conventional | 88 | 73 | 1:6 | 0 | 300-1000 | 150 | 2.0 |

The magnetic stirrer employed for both methods described was 6 x 15mm size oval shape. The reaction lasted for 120 minutes, aliquot were taken every 30 minutes (0, 30, 60, 90 and 120) to determine the reduced FFA.

## 2.3. Analytical methods

### *2.3.1. Washing sample*

An aliquot was collected via the condenser using a narrow diameter tube attached to a syringe and the sample was released in 500ml bottle containing hot water. The mixture was shaken vigorously to remove $H_2SO_4$. The sample was then left to separate in to oil/fat and water layers. The oil/fat (5ml) was pipetted in to 15ml centrifuge tube then a further aqueous suspension was made by adding water (10ml) to the pipetted sample before centrifuging using the benchtop Centrifuge Sigma 3-16PK for 5 minutes. The result is a well-separated into distinct oil and water fractions.

#### *2.3.2. Acid value*

The acid value of the reaction was determined by acid -base titration technique ASTM D 664 (ASTM, 2003). Measured sample was pipetted into a 100ml beaker and then prepared for titration by dissolving the sample in 3:1 ethanol and p-cymene solution and titrated against a standard solution of 1M potassium hydroxide solution using a Metrohm. 848/877 Titrino This study has limited the FFA analysis to acid value which is an acceptable method for FFA analysis in the biodiesel industry.

#### *2.3.3. Water content*

Water content was determined using the Hach water in oil test kit. Model (W0-1) and the procedure provided.

## 3. RESULTS and DISCUSSION

### 3.1. Raw material Content

The raw material parameters are presented in Table 3.

*Table 3: Parameters of samples A, B and C*

| Sample and other parameters | A | B | C |
|---|---|---|---|
| Acid value mgKOH/g | 18.3 | 37.2 | 51.5 |
| Initial Water content, % | 0.60 | 0.62% | 0.60% |
| FFA content, % | 9.4% | 18% | 25% |

### 3.2 Pre-treatment option to reduce FFA

#### *3.2.1 Pre-treatment of AWF*

In order to obtain a high ester yield by pre-esterification with sulphuric acid as catalyst, initial water content was reduced to 0.55% by preheating samples at 88°C using conventional heating method and microwave method respectively as shown in Table 4. Water content was measured after 120 minutes using the Hach Lange Water in Oil test kit. Both conventional and CEM methods recorded similar results as depicted in Table 4.

*Table 4: measurement of water content in oil for after pretreatment option using conventional and microwave heating*

| Sample ID | Initial H20 Content @0hr | Conventional method Final H20 content @2hrs | CEM method Final H20 content @2hrs |
|---|---|---|---|
| 9.4% FFA | 0.60% | 0.55% | 0.55% |
| 18% FFA | 0.62% | 0.55% | 0.55% |
| 25% FFA | 0.60% | 0.55% | 0.55% |

Conversion of fatty acid to biodiesel could be affected if the water content is high [4, 6]. Chung et al. suggested that water and FFA be kept at 0.06wt. % and 0.5wt.% respectively [20]. In acid catalysed method, FAME conversion could be affected by as little as 0.1% water content of the fats [21, 22]. In these experiments the water content of the AWF was found to have values in the range 0.60-0.62 wt. %. These values were further reduced to 0.55 wt.% by pre heating. Due to the peculiarity of AWFs the emulsified water is difficult to break free from the emulsion as observed in this study. One of the reasons other workers found treatment of this type of waste difficult is that reduction of FFA seem to respond to thermal history during heat treatment rather than reduction of FFA simply being a function of temperature and time.

### 3.2.2 Acid esterification condition

It has been reported by many researchers that acid esterification reaction is influenced by variables such as: alcohol to fat ratio, amount of catalyst, effect of temperature, and stirring power [3, 11, 23-25]. The present study also investigated the influenced of these variables; alcohol to fat molar ratio, amount of catalyst, effect of temperature and stirring power. The results from the investigation are presented in graphical and tabular representation.

### *3.2.2.1. Effect of methanol to oil ratio*

*Table 5 microwave assisted FAME production at constant temperature 73 °C in comparison with conventional method(0)*

| Entry | Method | Molar ratio (AWFs:MeOH) | Reaction time(seconds) | Catalyst wt.wt% | Microwave power(W) | Conversion (%)MW method | Conversion (%) Conventional method |
|---|---|---|---|---|---|---|---|
| 1. | II | 1:6 | 600 | 2.0 | 70 | 34 | 5 |
| 2. | II | 1:6 | 1200 | 2.0 | 70 | 50 | 10 |
| 3. | II | 1:6 | 1800 | 2.0 | 70 | 77 | 41 |
| 4. | II | 1:6 | 3600 | 2.0 | 70 | 88 | 71 |
| | | | | | | | |
| 5. | I | 1:6 | 1800 | 2.0 | 70 | 28 | 12 |
| 6. | I | 1:6 | 3600 | 2.0 | 70 | 85 | 64 |
| 7. | I | 1:6 | 7200 | 2.0 | 70 | 93 | 95 |
| | | | | | | | |
| 8. | I | 1:6 | 1800 | 1.0 | 30 | 46 | 34 |
| 9. | I | 1:6 | 3600 | 1.0 | 30 | 55 | 55 |
| | | | | | | | |
| 10 | I | 1:6 | 1800 | 0.8 | 30 | 48 | 22 |
| | | | | | | | |
| 11 | I | 1:3 | 3600 | 0.8 | 30 | 55 | 45 |
| | | | | | | | |
| 12 | I | 1:3 | 1800 | 1.0 | 70 | 61 | 34 |
| 13 | I | 1:3 | 3600 | 1.0 | 70 | 66 | 62 |

A few studies have reported acid catalysed esterification using animal fat feedstock [4, 26-28]. Many studies reported that acid catalyst requires excess alcohol for good fatty acid methyl ester (FAME) yield [3, 6, 29]. Bhatti et al. [27], Canakci and Van Gerpen [1] advocated the use of large excess quantities of alcohol. The conversion efficiency of the acid esterification of different feedstock in relation to molar ratio obtained in the present study is presented in Table 5. The FAME yield increased with increasing molar ratio from 66% FFA conversion at 1:3 molar ratio to 88% FFA conversion at 1:6 molar ration in 3600sec reaction time for the microwave method. A similar trend was observed with the conventional method with FAME yield increased from 62% to 71% in 3600sec reaction time. One of the objectives of the study was to optimize material usage with particular emphasis on methanol reduction. Some studies reported the use of ≥25% v/v of methanol/AWF in the acid esterification process [28-30]. Ghadge and Raheman [30] reported 50 wt.% reduction in acid value at low methanol quantities and over 80% acid value reduction at high- methanol quantities. A few papers reported the use of 10% vol of methanol and suggested that the methanol water fraction was collected at the top layer while the oil layer settles at the bottom [6, 30]. The present study, found that with lower percentage volume or weight of methanol used in acid esterification, the water and acid catalyst fraction migrates to the bottom layer with little methanol fraction at the top layer. However, the inconsistency in reporting might be due to the different feedstock and the wt.% acid catalyst used in the acid esterification reaction.

The use of lower percentage of methanol is preferred in the industry for many reasons. The flammability property of methanol, reduced cost of production and lastly, ease of phase separation are some clear reasons to reduce methanol usage in the biodiesel production.

#### *3.2.2.2. Effect of acid catalyst amount*

Three different concentrations of $H_2SO_4$ acid catalyst; 0.8.wt%. 1.0 wt%. and 2.0 wt. % $H_2SO_4$/AWF were tested to assess the percentage conversion of FFA for substrates with large amount of FFA contents. Figure 3 shows the percentage conversion FFA for substrate sample with initial FFA content of 37.2 mg.KOH/g. The percentage conversion FFA increased with an increase in acid catalyst for both microwave and conventional methods. A 2 wt. % acid catalyst recorded 93 wt. % and 95 wt. % conversion FFA for microwave and conventional methods respectively. However, there seems to be a trend with acid catalyst achieving better reduction in the first 60 minutes with microwave method compared to the conventional method as depicted in Figure 3 and Table 5.

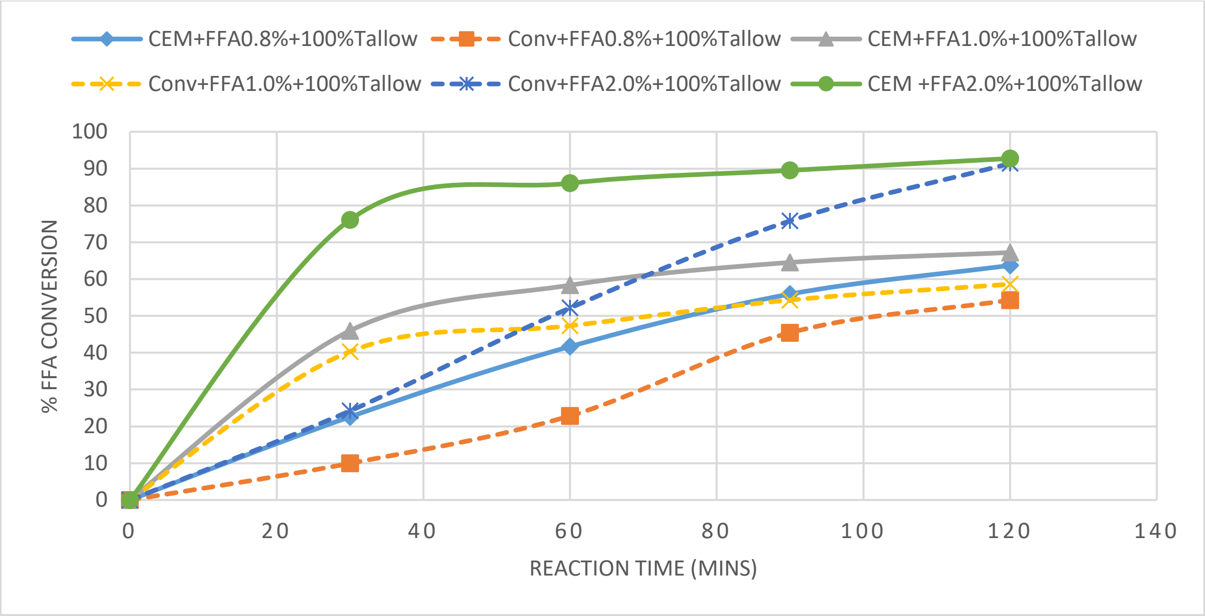

***Figure3: Effect of perecent amount of sulphuric acid on FFA reduction***

Improving the conversion efficiency and reaction time of biodiesel production is a clear objective of this study and the concentration of acid catalyst is one of the important variables for this conversion. Khan et al. demonstrated that 0.4% sulphuric acid produces maximum conversion efficiency with high FFA waste cooking oils [31]. In addition, few researchers reported 0.5 wt. % $H_2SO_4$ as the optimum condition for fats with high FFA content [3, 4, 13]. Some researchers reported a range of sulphuric acid amount of ≥1.0 wt. % as optimal condition for maximal acquisition of FAME yield [25, 30]. Canakci and Van Gerpen worked with a range of acid catalyst values (0, 5, 15 and 25 wt. %) and reported that even at 25 wt.% $H_2SO_4$/oil, the acid value did not reduce to 2mgKOH/g after 1 hour reaction time [1]. Gole and Gogate reported catalyst concentration over a range 2 to 4 wt. % of $H_2SO_4$/oil using microwave, ultrasound and the sequential of both methods recorded 2wt.% as the optimal concentration for conversion efficiency [32]. There seems to be a variation in the amount of acid catalyst required for optimal conversion, which might necessitate further investigation.

In this study a range of concentrations of sulphuric acid were investigated with the various FFA feedstocks. Concentrations of FFA decrease rapidly initially but as the reaction progresses the rate falls following a pseudo first order reaction kinetics. This behaviour is consistent with previous studies [33-36]. The slow decrease of FFA later in the reaction may also be due to accumulation of water. In addition, the sulphuric acid tends to migrate into the water phase from methanol phase when the stirring is poor and becomes unavailable for the FAME production.

#### ***3.2.2.3 The effect of temperature***

The conversion efficiency is low and reaction rate is very slow at room temperature even after 2 hours stirring for some feedstocks. With an increase temperature, the conversion takes place at a faster rate. The optimum temperature for the acid esterification reaction on a large scale is 73°C, which applies in the industry. Many researchers suggest that esterification reactions can be performed at lower temperatures when using microwave heating, compared to the standard heating process, which was the basis for investigating lower temperatures [4, 6, 26, 28, 29]. Jeong et al. suggested that the optimum reaction temperature depended on catalyst amount and was between 60-70°C [25]. As expected the reaction temperature also exerted significant influence on synthesis rate and high reaction temperatures tended to induce methanol evaporation. Temperatures below the boiling point of methanol reduced reflux and the methanol/AWF interface. This can be explained due to the fact that increase temperature favours the acceleration of the forward direction reaction as the reaction is endothermic under a kinetically controlled regime, which has been also demonstrated in earlier research [16]. From Le Châtelier’s principle, , the equilibrium shifts to the right for endothermic reactions as the temperature increases [37]. The present study investigated the effect of a range of temperatures using conventional heating and microwave technology on FFA reduction for feedstock with large amount of FFA shown in Figure 4. Increased reaction temperature from 55°C to 80°C showed positive effect on the conversion of FFA. Interestingly, at 30 minutes reaction time, the microwave method (CEM 30 min) converted 44% FFA to FAME in comparison with the conventional method (Conventional 30min) which produced 30% FFA conversion as depicted in Figure 4. Conversely, at 120 minutes reaction time, both microwave and conventional method converted 70% and 68% FFA to FAME. The results might suggest that microwave method is effective and showed increased percentage conversion in the first 30-60 minutes of the esterification reaction in comparison with conventional method under similar conditions.

However, the quantity of FFA were considerably still too high to proceed on to transesterification with method I (NPT). Therefore, improving the conversion of high FFA tallow using the pretreatment method and conditions specified in the matrix shown in Table 2 achieved a least 15% increase over no-pre-treatment method and conventional methods as depicted in Figure 4. Preheating the tallow at 88 $^{o}$C prior to injection of methanol and sulphuric acid solution might have further reduced the water content in the fat and likewise reduce the viscosity of the fat/oil promoting good stir and increasing the methanol/sulphuric/fat contact. The improvement in viscosity is dependent on the chain length and the degree of saturation of the fatty acids, and temperature [38-40].

These properties enhance the forward reaction and further reduces the FFA in the substrate.

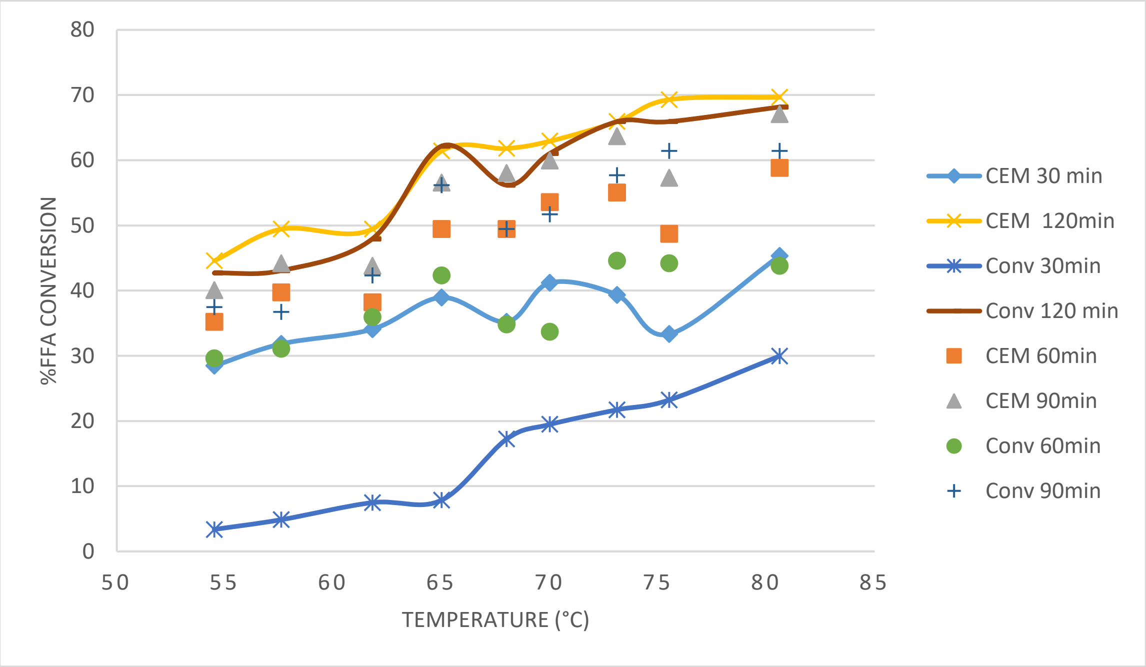

*Figure 4: The effect of temperature on FFA reduction at (30, 60, 90 & 120 minutes) reaction time for microwave and conventional reactions*

### 3.2.2.4 Effect of stirrer sizes and stirring on FFA conversion

The present study used a range of stirring magnets e.g. the pea-shaped magnetic stir, small 2mm, 6mm and 9.5mm magnetic fleas were used. Figure 5 show the difference in the flea sizes and effect and its effect on FFA reduction. The 6mm magnetic stir at

300rpm stirring presented better results when compared to the 2mm and 9.5mm at 300rpm for CEM and 6mm at 300rpm for conventional method as illustrated in Figure 5.

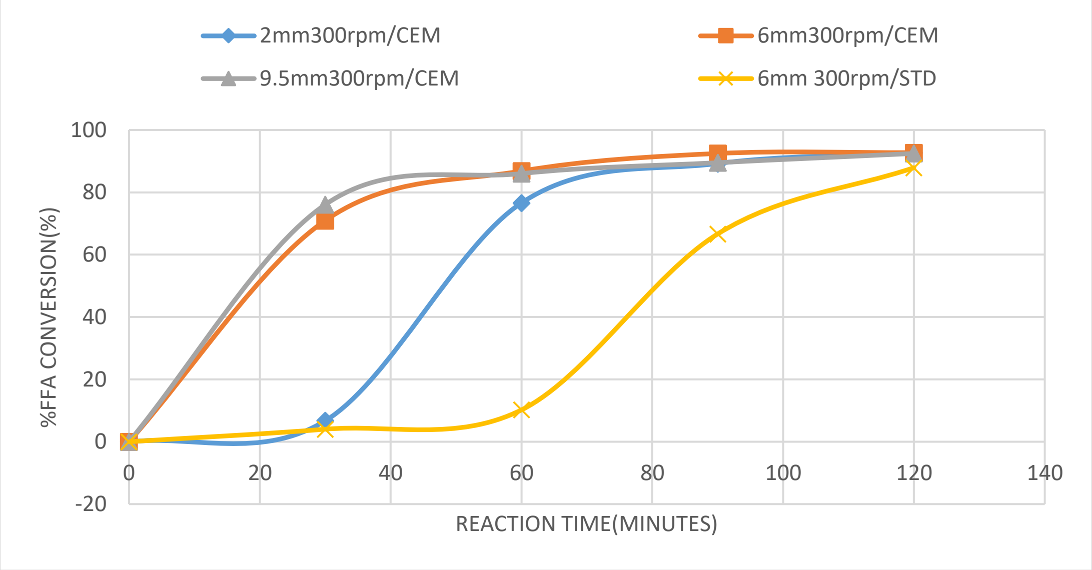

***Figure 5: The effect of stirring frequency and stirrer sizeon %FFA conversion***

Results from experiments shown in Figure 5 suggest that the size and shape of magnetic stirrer bar influences the stirring power and the reactant-catalyst contact. Although, when using the CEM method, it is important to choose appropriate magnetic stir to prevent deflection of the electromagnetic waves from the system. Hence, the 9.5mm was ineffective for the microwave method but produced good reactant-catalyst contact in the conventional method. Stirring during the acid esterification reaction also, plays a role in uniform mixing of reaction mixture (improve surface contact area). A few studies reported the stirring rates range between 150-800 rpm [2, 32].

### 3.2.2.5 The effect of microwave on FFA conversion

Microwave technology has been reported to reduce reaction time in transesterification to a few minutes [41, 42]. In contrast, Suppalakpanya et al. suggested a reaction time of 60 minutes for esterification at microwave power 70W but with no reference to temperature at which reaction occurred [43]. The present study is in agreement with previous report on microwave increasing the rate of reaction as observed in Figure 6.

Increase in microwave power increase the FFA percentage conversion as presented in Figure 6. Esterification at microwave power 70 W and 1: 6 AWF: MeoH molar ratio achieved 93% FFA conversion while conventional method was observed to be slightly better than the microwave power at 30W. The pretreatment method with microwave application presented 15% increase in conversion yield over conventional method and non-pre-treatment option at the same temperatures as depicted in Figure 7.

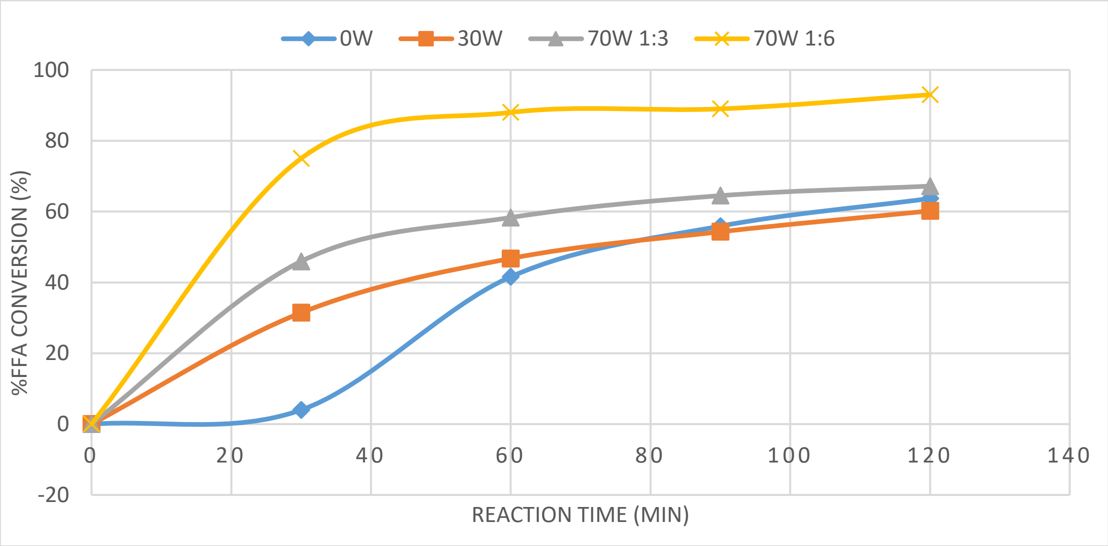

***Figure 6: shows %FFA conversion at 0, 30, &70W in 120 minutes, reaction kept at constant reaction condition, 1:3 AWFs/ methanol molar ratio, 1.0% w/w sulphuric acid and 73°C in comparison with 1:6 AWFs/methanol, 2.0% w/w sulphuric acid***

A comparison of the present study with previous studies is presnted in Figure 7. The present study has attempted to reduce AWF with large amount of FFA( >10 wt.%FFA) via batch process, with pretreatment option using conventional heating and microwave application. In addition, a reduced concentration of methanol and sulphuric acid with moderate microwave power exposure is compared with previous studies [23, 33]. Esterification reaction with moderate microwave power, 70W, and a pretreatment option gave 88% FFA conversion yield in 3600 sec reaction time.

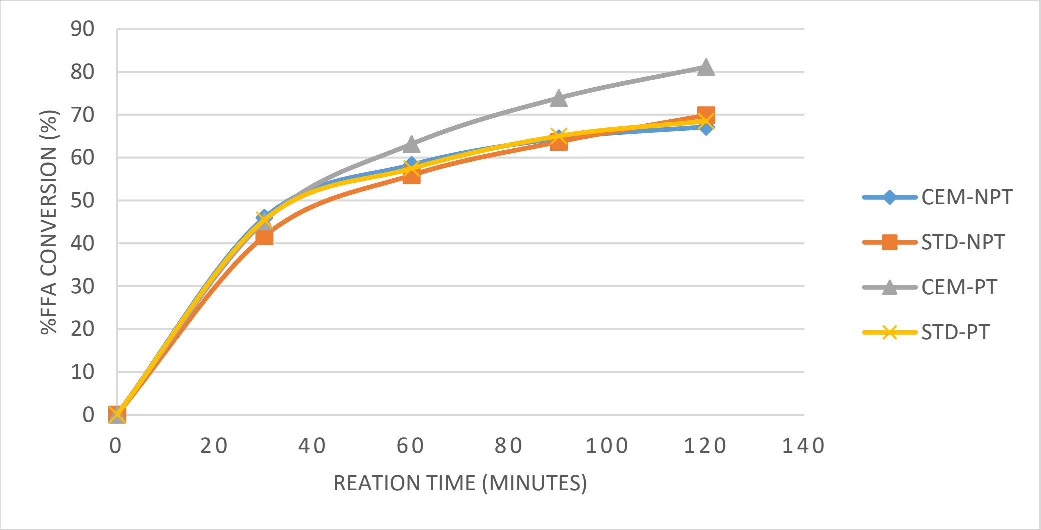

***Figure 7 A comparison between pre-treatment and non pre-treatment option and its effect on %FFA conversion with microwave method and conventional method (1:3 AWFs/methanol molar ratio, 1.0 sulphuric acid at 73°C)***

***Table 6 : A matrix of experimental conditions and % FFA conversion in comparison with other studies***

| **AUTHORS** | **METHOD** | **AMOUNT OF ACID CATALYSED** | **MOLAR RATIO** | **MICROWAVE POWER** | **REACTION TIME** | **INITIAL FFA in oil/fat** | **% FFA CONVERSION** |
|---|---|---|---|---|---|---|---|
| Kamath et al 2011 | 2.45GHz Domestic mw Open vessel | 3.73%w/w | 1:10 | 180, 300W | 190s | 8.8% | 89-91% |
| Suwannapa and Tippayawong [33] | 2.45GHz Domestic mw Open vessel | 1.5%w/w<br>1.0%w/w<br>2.0%w/w<br>1.5%w/w<br>1.0%w/w | 1:3<br>1:6<br>1.6<br>1:9<br>1:12 | 340W | 1800s<br>1200s<br>2400s<br>1800s<br>1200s | 6.18-6.80% | 95%<br>94.5%<br>95.6%<br>98.3%<br>97.6% |
| Present study | 2.45GHz CEM Open vessel (73°C) | 0.8%w/w<br>1.0%w/w<br>1.0%w/w<br>2.0%w/w<br>2.0%w/w | 1:3<br>1:3<br>1:3<br>1:6<br>1:6 | 30W<br>30W<br>70W<br>70W<br>70W | 3600<br>3600<br>3600<br>1800<br>3600 | 18%-25% | 47%<br>55%<br>64%<br>75%<br>88% |

## 4. CONCLUSION

The shift towards animal waste fats as feedstock for biodiesel production on a large scale would be welcome but could be challenging. AWF such as tallow are typically very low grade with high FFA content (>20 wt. %) making them difficult to use for the conventional biodiesel process without the preliminary reduction of the FFA. Esterification, better described as pre-esterification is normally carried out with an acid catalyst and allows such low-grade raw material to be effectively and economically converted to biodiesel. However, the esterification reaction adds an additional step to the overall process and demands either higher reactor volumes or reduced throughput unless reaction times can be reduced. Therefore, anything that increases yield or decreases processing time would be extremely welcome. One potential avenue explored here is the use of microwave heating to accelerate the reaction. This is well known in laboratory scale studies of the biodiesel trans-esterification, though not employed significantly commercially. A similar use of microwave heating for esterification, especially of high FFA fats has not been widely reported and is certainly not used on an industrial scale. This laboratory-scale study on AWF with FFA content >20 wt. % has shown that the application of microwave either replacing the conventional heating or complementing it, could essentially improve the process by reducing the reaction time for FFA reduction and also increasing FAME yield, thus making this type of low cost feedstock available for the biodiesel industry. Further studies are underway to transfer this process to an industrial scale.

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## CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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