*International Conference on Agricultural and Food Engineering for Life (Cafei2012)*
*26-28 November 2012*

**CAFEi2012-161**

# PARAMETRIC STUDIES ON *in situ* TRANSESTERIFICATION OF PALM FRUIT AND WASTE MESOCARP FOR BIODIESEL PRODUCTION.

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## ABSTRACT

Transesterification of vegetable oil is the most common method used for biodiesel production. However, a new technique known as "reactive extraction" or "in situ transesterification" can simplify the process and has the potential to reduce the production cost. The potential of biodiesel production from fresh palm fruit and waste mesocarp using this technique has been investigated. Parametric studies were conducted to evaluate the effects of solvent to oil molar ratio, catalyst concentration and temperature on the yield and the reaction rate. Sodium hydroxide was used in this study. A high yield of ester (> 25.99% and >13.99%) was achieved at high solvent to oil molar ratios (400:1 and 500:1) for mesocarp fruit and waste mesocarp respectively. High ester yield can be obtained by using high catalyst concentration (0.15 molal) for both palm fruit and waste mesocarp (34.2% and 13%). Temperature plays an important role in this process since higher temperature give higher ester yield

***Keywords*:** In situ transesterification, reactive extraction, biodiesel, palm fruit, and waste mesocarp,

## INTRODUCTION

Biodiesel is known as an alternative diesel fuel, made from renewable biological resources such as vegetable oils and animal fats. This fuel is biodegradable, non-toxic and produces low emission as compared to petroleum diesel. The usage of biodiesel will allow a balance to be striked between agriculture, economic development and the environment [2]. In conventional biodiesel production, the triglyceride needs to be extracted from the biomass source and transesterified into ester. The transesterification proceeds by the reaction of triglyceride with an alcohol in the presence of a catalyst, producing fatty acid alkyls ester and glycerol as a by-product [1]. In the transesterification reaction, the catalyst can be both acidic and basic catalysts. Alkaline catalyst such as metal hydroxides (i.e. KOH and NaOH) is commonly used as the basic catalyst. The use of alkaline metal hydroxide can produce a certain amount of water even if a water-free vegetable oil and alcohol are used due to the reaction of hydroxide with the alcohol. The presence of water will lead to soap formation from the hydrolysis of esters. This will reduce the biodiesel yield, as well as cause difficulty in the product separation of ester and glycerol [12, 17].

Usually, edible oil such as sunflower, soybean and palm oil are used for biofuel production. To expand the biofuel production, non-edible oil can also be used as the source of biofuel using a technique called *in situ* transesterification [3, 16]. In situ transesterification differs from conventional transesterification whereby the oil bearing material is directly reacted with the alcohol by an acid or base catalyst, hence the extraction and transesterification take place simultaneously [4]. This reaction route eliminates the expense related to solvent extraction and oil clean up, thus simplifies the steps in ester production. Consequently, this method helps to decrease the cost of the production [5]. A study of in situ alkaline-catalysed transesterification of sunflower seeds by using methanol and ethanol as solvent and NaOH as catalyst reported the ester yield as high as 96% using methanol and 90% using ethanol [4].

In Malaysia, there are various types of oil-bearing biomass that can be used to produce biodiesel. The most important source of biomass would be palm fruit. The major fatty acid composition of palm oil is shown at Table 1. Method of producing biodiesel directly from palm fruit without prior extraction of the oil is not yet explored. Waste mesocarp, on the other hand, usually contains 3-4 % of oil and is usually burned in boiler to produce electricity [24]. In situ transesterification method can allow the recovery of oil from the waste mesocarp for biodiesel production in a singlestep operation. As the mesocarp has a relatively low oil content, a singlestep process can reduce wastage compared to a two-step extraction and reaction process. Also, if the process can be designed to be operated on–site, it will eliminate the transport of raw bulk solid materials to a

centralised plant, which is often costly and cumbersome. This paper discussed biodiesel production from palm fruit and waste mesocarp by ethanol as the solvent and NaOH as the catalyst. The effect of ethanol to oil molar ratio, catalyst concentration and temperature was analysed. Ethanol is used in this study due to it being a more environmentally friendly fuel and, in contrast to methanol, is more biodegradable and less toxic. Besides, ethanol can be derived from agricultural products and is renewable [4].

Table 1: Major fatty acid composition of palm oil [6]

| Fatty acid | Composition (%) |
|---|---|
| Linoleate acid | 10.3 |
| Palmitate acid | 43.7 |
| Oleate acid | 39.9 |
| Streate acid | 4.4 |

## MATERIALS AND METHODS

### Materials

Palm fruit and waste mesocarp were obtained from Ulu Langat, Dengkil, Palm Oil Mill. The fruit and waste mesocarp werefreshly taken at the mill in the morning and dried under sun for about 30 minutes and 1 day respectively to remove any moisture adhered to the fruit and waste mesocarp. After that the fruit was properly packed and stored in a refrigerator at 5$^{o}$C, while the waste mesocarp was packed in a plastic container and stored at room temperature. The fruit sample was prepared by cleaneing the fruit first and then it was cut into a small size of 1cm thick and 2-3cm long. For the sample preparation of waste mesocarp, after the waste mesocarp was dried, it was grinded into 1mm to 2 mm particle size using a fiber grinder. After grinded, the waste mesocarp in particle size was stored properly in a air-tight plastic at room temperature. Hexane (99%), methanol (99%), and ethanol (99.6%) were purchased from Fisher Scientific. The standards of methyl heptadecanoate (MHDN) and ethyl esters, (ethyl streate (99%), ethyl palmitate (99%), ethyl oleate (99%) and ethyl linoleate (99%)) were purchased from Sigma Aldrich. Sodium hydroxide of analytical reagent grade was purchased from Evergreen.

### Fatty acid ethyl ester (FAEE) analysis

A gas chromatography was used to analyse individual ester and total ester content. MHDN was used as the internal standard. The stock solution from the internal standard was prepared by dissolving 5 g of MHDN to 322.6g of ethanol and stored in the refrigerator at 5$^{o}$C. The sample was prepared by adding 1g of sample to 1.25g stock solutions in a sample vial. The ester yield was quantified using a gas chromatography equipped with Flame Ionized Detector (FID) and Agilent column (0.53mm x 25m length and 15um coating thickness). The carrier gaswas helium at 2 mL/min flow. Temperature of 220 $^{o}$C was maintained at the column, injector and detector. The injection volume used was 0.2uL. The weight percentage of ester in bulk ethanol phase (C) was calculated using the equation below:

$$C = \frac{\frac{100 - Area\ MHDN\%}{Ara\ MHDn\&}\ x\ solvent\ ratio\ x\ mass\ of\ standard\ used\ (g)}{Mass\ of\ sample\ used\ (g)}$$

$$Ester\ yield\ (g) = C * mass\ of\ total\ filtered\ solution\ from\ reactive\ extraction$$

$$Ester\ yield\ (wt\%) = \frac{mass\ of\ ester\ phase * 0.995 * 100}{mass\ of\ triglyceride\ in\ the\ seed * 100}$$

### Moisture content

Palm fruit and waste mesocarp were weighed at 10 g and kept overnight in the oven at a temperature of 108$^{o}$C until a constant weight was reached. The moisture content was calculated in term of percentage as:

$$\text{Moisture content (\%)} = \frac{\text{Initial weight – final weight}}{\text{Initial weigh}} \times 100$$

**Oil Extraction**

Determination of oil content from palm fruit and waste mesocarp was carried out using a Soxhlet extraction apparatus with ethanol and hexane as the solvent. 10 g of sample was weighed and 230 ml of solvent was used for the extraction process. The reaction was carried out for 2, 4, 6, and 8 hours. After the extraction process, the solvent and oil was separated with a rotary evaporator and dried in an oven at 80$^{o}$C. The oil yield was expressed in term of mass percentage (%) as:

$$yield\ (\%) = \frac{Mass\ of\ oil\ extracted\ (g)}{Mass\ of\ sample(g)}\ x\ 100$$

Free fatty acid analysis

The free fatty acid was determined based on ACOS Official method Ca.5a-40. 7 g of extracted palm fruit is weighed into 200mL Erlenmeyer flask. 75 mL of 95% ethanol is heated to 40$^{o}$C and 2mL of phenoftalein was added to the oil. The oil ethanol mixture was titrated by 0.25N sodium hydroxide solution. This oil mixture was vigorously shaken until the first appearance of a permanent pink colour. The procedure is repeated with waste mesocarp. The percentage of free fatty acid was calculated as below:

$$Free\ fatty\ acid\ (\%) = \frac{ml\ of\ alkali\ x\ alkali\ molarity\ x\ 28.2}{Mass\ (g) of\ oil\ sample}$$

**In situ transesterification**

In this process, the samples were extracted and transesterified using sodium hydroxide as the catalyst at different operating parameters. Alkaline solvent was prepared by mixing the desired amount of sodium hydroxide in ethanol. 5 g of sample was placed in a 100 ml screw capped bottle and mixed with the desired amount alkaline solvent. The bottle is placed in orbital shaker at 200rpm at desired time and temperature. After operation at desired time, a known amount of glacial acetic acid was placed into the liquid to stop the reaction. Vacuum filtrationwas used to separate the sample and the solution. The solid residue was washed repeatedly by ethanol to recover any product that still adhered to the seed. The excess of ethanol was removed by a rotary evaporator. The mass of liquid that was formed after the reaction was recorded and the ester content was examined using a gas chromatograph.

**Effect of ethanol to oil molar ratio**

Different ethanol to oil molar ratios was used as different weight of alkaline solvent. 100 to 500 and 400 to 600 ethanol to oil molar ratio were used for in situ reaction of palm fruit and waste mesocarp respectively. The reaction time and reaction temperature was maintained at 1 hour and 60$^{o}$C respectively for each experiment.

**Effect of catalyst concentration**

Four different catalyst concentrations were used for in situ reactions which are 0.03, 0.05, 0.10 and 0.15 molal. The parameters for this experiment were at 60$^{o}$C , at 1 hour and used 476:1 ethanol to oil molar ratio.

**Effect of temperature**

Four different levels of temperature of 30, 40, 50 and 60$^{o}$C were tested for each sample. 476:1 ethanol to oil molar ratio was used for this reaction while the reaction time and catalyst concentration was maintained at 1 hour and 0.1 molal respectively.

## RESULTS AND DISCUSSIONS

**Oil Analysis:**

**Extraction of palm fruit and waste mesocarp**

For both raw materials, the highest yield of oil can be obtained after 8 hours of extraction. Two types of solvent were used to extract the oil, which are hexane and ethanol. Being a polar solvent, the use of ethanol may increase the extraction yield as it is able to extract other polar materials as well [20]. The extraction of 10 g palm fruit yields 47.42% oil by hexane and 47.76% oil by ethanol as shown in Table 2. The yield is in the range found in the literature which is about 45% to 50% [15, 24 39]. 10 g of waste mesocarp extraction shows that there is 9.98% and 9.82% oil as extracted by hexane and ethanol respectively. Hence, similar amount of oil was extracted using ethanol and hexane. Since there is about 10% of oil still left in the waste mesocarp after passing through the screw pressed process, further recovery of this oil as biodiesel can help to utilise this waste into value added product and contribute to a diversified energy source.

Table 2: Extraction of palm fruit and palm fiber by using ethanol and hexane as solvent

| | Hexane (%) | Ethanol (%) |
|---|---|---|
| Palm fruit | 47.42 | 47.76 |
| Palm fiber | 9.98 | 9.825 |

**Free fatty acid content**

Table 3 shows the free fatty acid content of the oil extracted from waste mesocarp is in the range of 62 to 65 %, while the fruit free fatty acid content is below 19%. The free fatty acid of palm fruit is slightly higher than that reported in the literature of about 13% [18]. The high free fatty acid content in the palm fruit may be due to the hydrolysis of triglycerides in the presence of a lipase enzyme. Prolong storage of the palm fruit may cause the increase in free fatty acid content compared to fresh fruit. On the other hand, improper handling after cutting can also cause the reaction of lipase enzyme since the cell structure of the fruit has been adversely affected [21]. Contrary to fruit, waste mesocarp free fatty acid content reaches to about 62%, which is considered very high. The high free fatty acid content in waste mesocarp may be the result of the fruit going through the oil extraction process [24].

Table 3: Free fatty acid content of palm fruit and waste mesocarp

| | Waste mesocarp | Fruit |
|---|---|---|
| Hexane | 65.03 | 18.34 |
| Ethanol | 62.41 | 17.05 |

**Moisture content of palm fruit and waste mesocarp**

From table 4, the moisture content of palm fruit is about 24.83% while waste mesocarp is 43.67%. This value is similar to the value reported in literature of 24% and 42% for palm fruit and waste mesocarp respectively [24].

Table 4: Moisture content of palm fruit and waste mesocarp

| Sample | Moisture (%) |
|---|---|
| Fruit | 24.83 |
| Waste Mesocarp | 43.67 |

**In situ transesterification:**

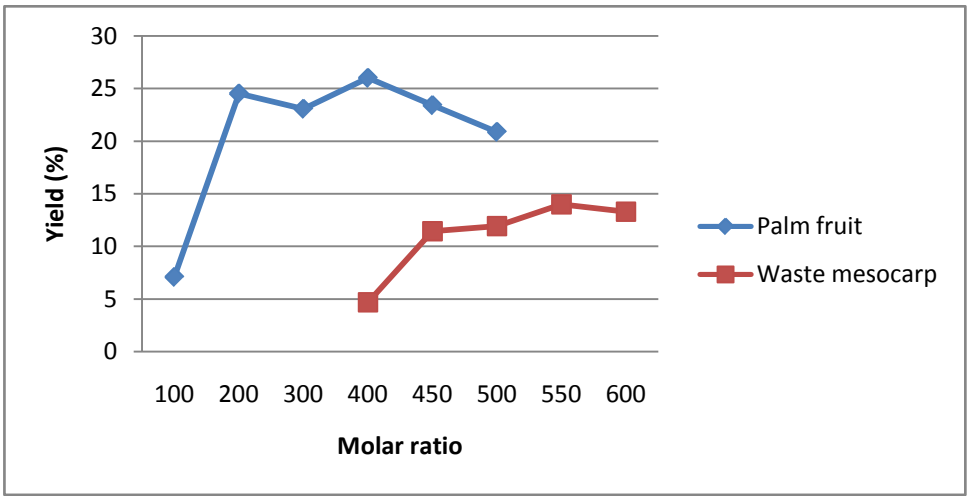
**Effect of ethanol to oil molar ratio**

Figure 1(a) shows the yield of reactive extraction of palm oil fruit and waste mesocarp with sodium hydroxide as the catalyst after reaction for 1 hour. For palm fruit, the result shows that the ester yield increased

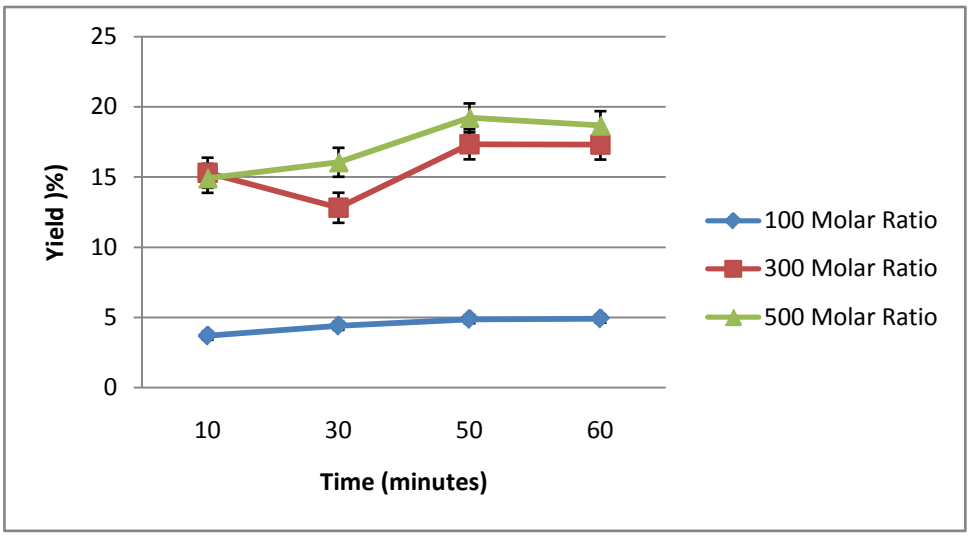
significantly from 6% to 24% when the solvent to oil molar ratio was increased from 100:1 to 200:1. Further increasing the molar ratio from 200:1 to 500:1 does not increase the yield to any significant extent. The maximum ester yield that can be obtained is about 26% at 400:1 ethanol to oil molar ratio. On the other hand, the ester yield for waste mesocarp sharply increases when the solvent ratio was increased from 400:1 to 450:1 but further increase in solvent ratio only increases the yield slightly. The maximum yield of ester (13 %) is achieved at 550:1 ethanol to oil molar ratio. A higher solvent ratio is needed for waste mesocarp compared to fresh fruit. This may be due to the physical characteristic of the waste mesocarp itself. Since most of the oil in the waste mesocarp has been extracted, the fiber contains a higher pore volume causing a high amount of solvent to be absorbed into the fiber. Hence, a high amount of solvent is needed to ensure adequate amount of solvent is presence in the bulk solvent phase, which is important to drive the extraction.

The reactive extraction rate at different solvent to oil molar ratio for palm fruit is shown in Figure 1(b). For all the molar ratios, the reaction predominantly occurs between 0 to10 minutes, after which the reaction rate starts to reduce. The equilibrium is generally reached after 50 minutes. The equilibrium yield obtained in this study is low compared to the reactive extraction of other reported biomass such as rapeseed and soybeans [11, 19] where as high as 99% yield was achieved. This may be caused by the high amount of free fatty acid in the oil which consumed the alkaline catalyst. Hence, the effect of increasing the catalyst concentration is studied next in section 3.4.2. Furthermore, most studies of in situ transesterification such as sunflower and soybean used methanol as the solvent. Methanol has a shorter carbon chain than ethanol and was able to react with the oil at a lower temperature reaching high ester yield in a shorter time [19]. Contrarily, the longer carbon chain of ethanol may give more complexity to the process and affect the optimum operating parameters [30]. For example, a study of transesterification of ethyl ester from crude palm oil shows that the used of ethanol causes incomplete separation of glycerol resulting in low recovery of ethyl ester [29].

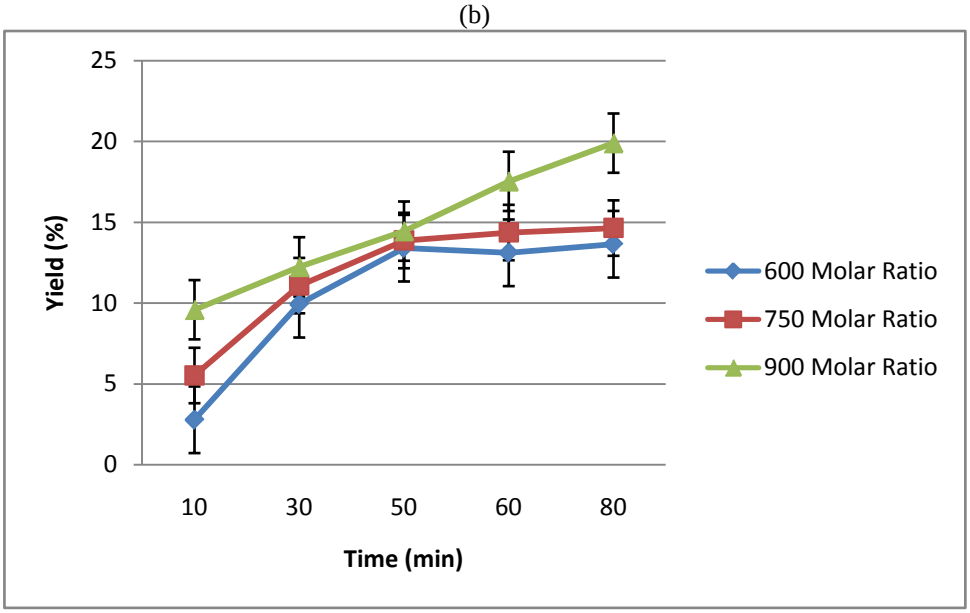
The reaction progress of waste mesocarp is shown in Figure 1(c). In contrast to palm fruit, the reaction rate of waste mesocarp is relatively slower and the equilibrium yield is only achieved after 50 minutes. This again maybe due to the reduction in the catalyst concentration from the neutralization of the higher amount of free fatty acid in waste mesocarp than in palm fruit The graph also shows that the reaction rate for solvent ratio of 900:1 is higher than that at 600:1 and 750:1. The higher amount of solvent used, as well as the higher catalyst amount may have increased either or both the extraction and reaction steps.




(a)




(b)




(c)

Fig. 1 (a) : Effect of ethanol to oil molar ratio for palm fruit and waste mesocarp at 1 hour reaction time, 0.1 catalyst concentration and at 60 $^{o}$C. 1 (b): Effect of ethanol to oil molar ratio on equilibrium yield for palm fruit . Reaction conditions: 60 °C, 0.1 m catalyst, 5 g seed. 1 (c): Effect of ethanol to oil molar ratio on equilibrium yield for waste mesocarp. Reaction conditions: 60 °C, 0.1 molal catalyst, 5 g seed.

**Effect of catalyst concentration**

Catalyst concentration is important in reactive extraction; a suitable amount of catalyst will produce a high ester yield while a high catalyst concentration of sodium hydroxide will lead to saponification reaction and soap formation, hence lowering the ester yield [4]. A 476:1 ethanol to oil molar ratio was used since it has been shown earlier that reactive extraction of palm fruit at 400:1 solvent ration has reached the maximum yield. Figure 2(a) shows the effect of catalyst concentration on palm fruit at different catalyst concentration. The reaction rate for all catalyst concentration shows similar trend where the rate is increasing rapidly from 0 to 50 minutes, but after 50 minutes the rate becomes slower. As expected, increasing the catalyst concentration increases the rate of reaction, but most importantly increasing the catalyst concentration also increases the equilibrium yield. Previous study on the influence of various parameters on reactive extraction of Jatropha curcas L shows that increasing sodium hydroxide catalyst will increase the extraction efficiency as well as the ester yield [23].The highest yield of 34,2 % can be obtained at 0.15 molal catalyst concentration and at 80 minutes reaction time. The yield of 0.15 molal catalyst concentrations however is only slightly higher than that at 0.1 molal indicating that increasing the catalyst concentration much further will not improve the yield, but will cause excessive saponification instead.

Similar effect of catalyst concentration was also seen on waste mesocarp as shown in Figure 2(b) A catalyst concentration of 0.15 molal produces the highest ester yield although the yield is slightly higher than that at 0.1molal. Again, this indicates that increasing the catalyst concentration much further is not expected to increase the yield. Although higher yield is obtained at 0.15 molal catalyst concentrations, the ester yield obtained at this catalyst concentration is still low compared to other studies [13, 11, 19].

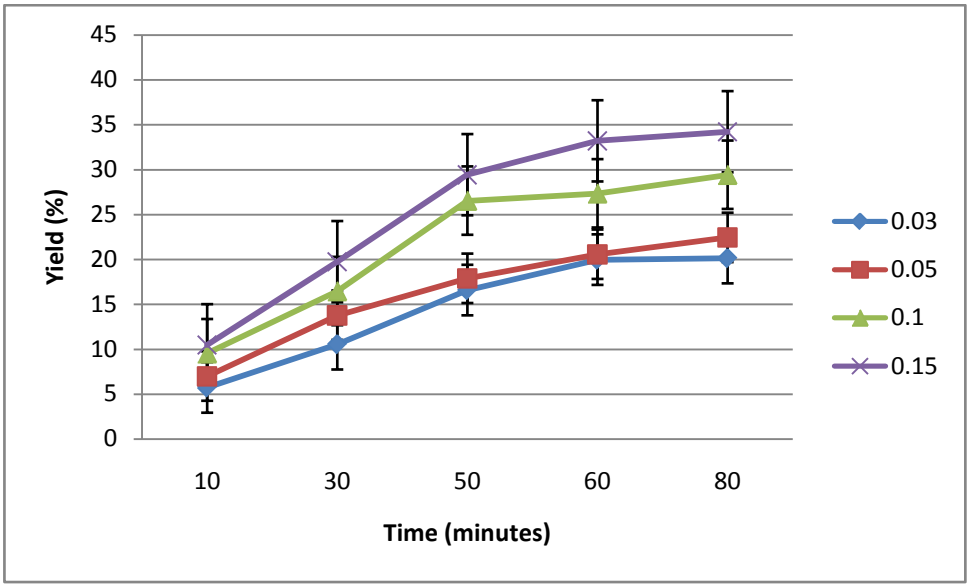



Fig. 2 (a): Effect catalyst concentration for palm fruit at 1 hour reaction time, 476:1 ethanol to oil molar ratio and 60$^{o}$C .

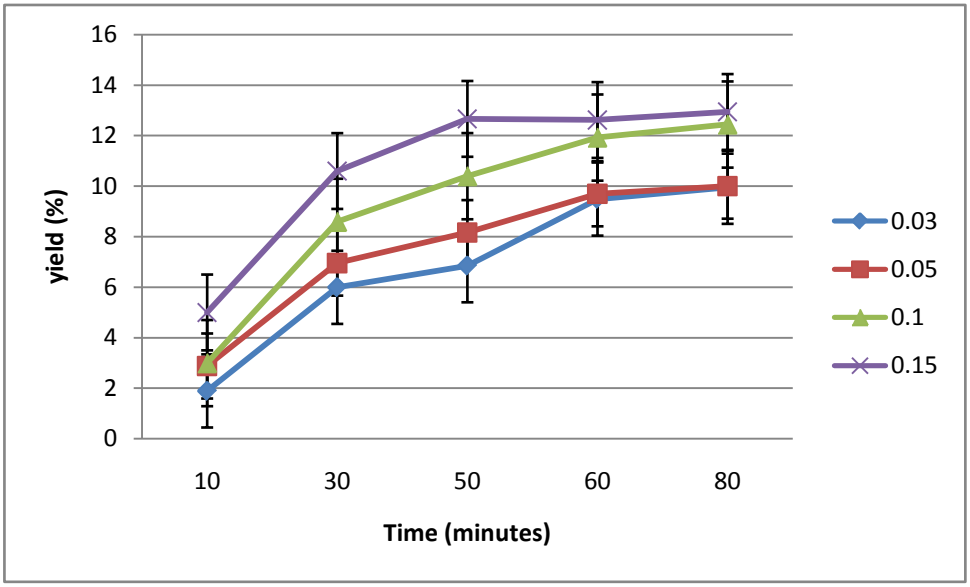



Fig. 2 (b): Effect of catalyst concentration for waste mesocarp at 1 hour reaction time, 576:1 ethanol to oil molar ratio and 60$^{o}$C .

**Effect of temperature**

In this study, temperature plays an important role for reaching a high ester yield. Figure 3(a) shows the effect of temperature on reactive extraction palm fruit. The higher temperature used for the reaction, the higher the ester rate and yield obtained. The final yield at 60$^{o}$C is much higher than that at 30$^{o}$C. In contrast, studies on reactive extraction of sunflower seed shows that reaction temperature does not have a significant effect on the final yield and yield as high as 98% can be achieved at 20$^{o}$C. The dissimilarities found in this study may be due to the fatty acid composition of palm fruit. As indicated in Table 1, palm fruit fatty acid composition contains high palmitic acid which is a saturated type of fatty acid. The palmitic acid content of palm fruit is 43.7% which is relatively high compared to other oilseed [6, 34]. Palmitic acid is solid at room temperature, and it has a high

melting. At low temperature, the fatty acid of palm oil may be very viscous causing a reduced extraction and reaction rate. The presence of a high amount of free fatty acid also may cause an overall increase in viscosity and hence a lower extraction rate. A similar trend was observed for waste mesocarp as shown in Figure 3(b) due to the similar fatty acid profile of the oil.

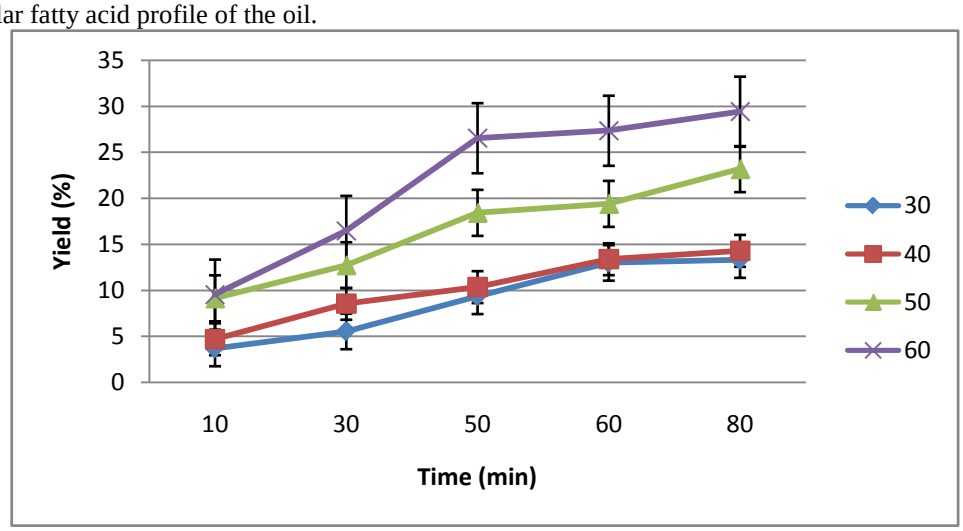



Fig. 3 (a): Effect temperature for palm fruit at 1 hour reaction time, 476:1 ethanol to oil molar ratio and 0.1 mol catalyst concentration

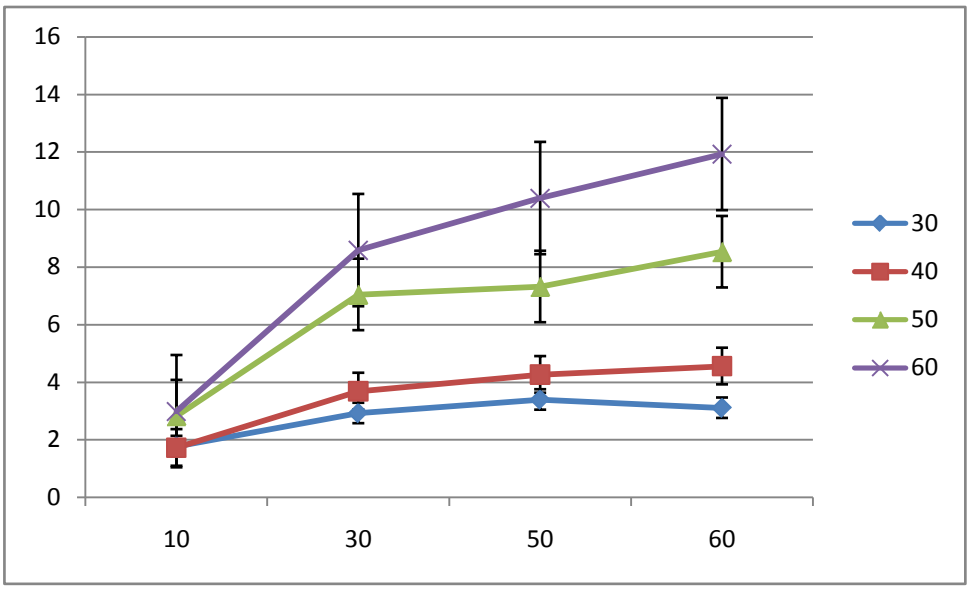



Fig. 3 (b): Effect temperature for waste mesocarp at 1 hour reaction time, 576:1 ethanol to oil molar ratio and 0.1 mol catalyst concentration

## CONCLUSIONS

From the result of the parametric study, it can be concluded that a high yield of ester from both palm fruit and waste mesocarp can be achieved by increasing the solvent molar ratio, catalyst concentration and temperature. However, the final ester yield from in situ transesterification of palm fruit and waste mesocarp is lower than the other biomass feedstock such as sunflower which reaches 99% ester conversion [4]. The lower ester yield may be due to the high free fatty acid content of the palm fruit and waste mesocarp which consume and neutralise the catalyst preventing further transesterification to take place. In order to overcome the weakness of this reaction process, further study of reactive extraction by acid or lipase catalyst is needed in order to achieve high ester yield even though the process is generally much slower than alkaline catalyst. Further studies in process design and optimization are still required to optimize the use of this biomass feedstock.

e-ISBN 978-967-960-322-4

## ACKNOWLEDGEMENT

This work was supported by the Research University Grant Scheme (RUGS) and the author gratefully acknowledges the financial support from Graduate Research Fellowship (GRF)

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