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ORIGINAL ARTICLE

Optimisation on pretreatment of kapok seed (*Ceiba pentandra*) oil via esterification reaction in an ultrasonic cavitation reactor

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Abstract Pretreatment of the kapok seed oil via esterification reaction has been investigated by using an ultrasonic cavitation reactor. Reaction parameters have been optimised by using response surface methodology and found as methanol to oil ratio of 16.23:1, catalyst amount of 9.94 wt% and reaction time of 20.08 min at amplitude of 32.04 %. The significance of the reaction parameters toward free fatty acid reduction was in the order of amplitude > reaction time > molar ration of methanol to oil > catalyst amount. The reaction time and esterified efficiency of ultrasonic cavitation was 4.5-fold shorter and 10.5-fold higher than mechanical stirring. The reactant used per produced biodiesel in ultrasonic cavitation process was about 2.6 lower compared to mechanical stirring process.

Keywords Pretreatment · Kapok seed oil · Ultrasonic cavitation · Optimisation

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Abbreviations

KSO	Kapok seed oil
MS	Mechanical stirring
UC	Ultrasonic cavitation
FFA	Free fatty acid
KOH	Potassium hydroxide
RSM	Response surface methodology
ANOVA	Analysis of variance
CCD	Central composite design

1 Introduction

The depleting of fossil fuel reserves, increasing of environmental issues, fluctuating prices and uncertain supply of fuel due to political disturbances have inspired researchers to look for renewable and sustainable fuel. Biodiesel as sustainable fuel produced from renewable source is a very attractive option to replace diesel fuel based petroleum [1]. It can be used directly in diesel engine without any modifications due to its properties are almost similar to diesel fuel [2]. Biodiesel contributes to environmental protection, which is biodegradation, renewable, non-toxic and reduces greenhouse gas emissions [3]. Biodiesel is composed of mono alkyl ester of long-chain fatty acid, which derives from various sources, viz. edible oil, non-edible oil [4], animal fat and algae [5]. About 95 % of world biodiesel production is derived from edible oil. Consumption of edible oil in biodiesel production has led to the price of biodiesel to increase to 1.5 - to twofold higher than diesel fuel. In order to make a viable biodiesel at a commercial scale, it is important to look at the benefit of using low-cost non-edible oil, which can reduce biodiesel production cost.

Non-edible oil is considered to be promising alternative feedstock, which could avoid the food controversy. The local non-edible plant oils, e.g. nyamplung, jatropha, castor, rubber, kapok and karanja have drawn the attention of the Malaysia government and researchers to consider them as biodiesel feedstock, which can be supplied at a viable quantity based on the large scale of cropped lands [6]. These non-edible plants could bring benefits, e.g. utilise low fertility land, restore degraded land and contribute towards eliminating the food crisis [7]. The non-edible kapok (*Ceiba pentandra*) seed oil (KSO) can be a very promising alternative feedstock, which could reduce the production cost and global emission management cost as a lower pollutant emission. However, based on diesel fuel demand and the available amount of non-edible kapok oil in the world, it still may not be able to replace diesel fuel completely, but could valuably contribute to the reduction of dependency on diesel fuel based petroleum. Kapok tree is grown in India, Malaysia and other parts of Asia. Kapok pod contains 17 % of fibre that is mainly utilised in making pillows and mattresses, whereas the seeds are traditionally waste [8]. Kapok seeds make up about 25–28 wt% of each pod with a potential oil yield average of 1280 kg/ha (hectare) annually [9]. Most common extraction of kapok oil from kapok seeds is using mechanical expeller [10].

Numerous of non-edible oils have been utilised for biodiesel production via mechanical stirring (MS), supercritical, microwave, ultrasonic cavitation (UC) and hydrodynamic cavitation [11], but only a limited number of works have reported on biodiesel production derived from KSO since 2013. Yunus Khan et al. [9] studied the fuel properties on feedstock blends between *Ceiba pentandra* and *Nigella sativa* via MS and Sivakumar et al. [12] deal with optimisation studied of molar ratio of methanol to oil, temperature, time and catalyst amount via MS for *Ceiba pentandra* methyl ester production. Vedharaj et al. [10] claimed that *Ceiba pentandra* methyl ester emitted higher nitrogen oxides compared to diesel fuel. They attempted to reduce the nitrogen oxides by using urea based selective non-catalytic reduction system, which was retrofitted in the exhaust pipe [13]. Another previous work conducted by Bokhari et al. [14], which studied the acid esterification followed by alkaline transesterification of KSO via microwave-assisted.

Biodiesel is generally produced by transesterification reaction in the presence of alkali catalysts if the free fatty acid (FFA) content in feedstock oil less than 2 wt. %. Higher FFA content is a major obstruction of direct conversion of triglycerides to alkyl esters by transesterification reaction due to FFA can react with alkali catalyst and results in saponification [15]. Esterification is a reversible reaction adopted for FFA reduction in the presence of acid catalyst (e.g. sulphuric acid) and alcohol. It converts FFA content in the feedstock to alkyl esters. MS is used during esterification process, but it is costly as excessive energy is needed and it also consumes longer reaction time. Process intensification technologies have been proven to be sustainable for pretreatment and alkyl esters synthesis due to its potential to eliminate the mass transfer resistance between the immiscible reactant of oil and alcohol in shorter period and lower energy consumption compared to MS, which is commonly used in biodiesel industrial [16]. The drawbacks of the MS can be overcome by UC. A performed study of the existing literature indicates that there are very little works related to the use of UC for pretreatment of high FFA content in KSO. Consequently, it is crucial to develop sustainable process intensification. This paper targets a development of a novel approach: UC for pretreatment of high FFA content in KSO, which in turn reduces the cost of processing. UC is an efficient technology used to boost the pretreatment process in terms of shorter reaction time and higher esterified efficiency.

The presented work deals with the intensification of esterification reaction in UC reactor, which is based on the generation of cavitation events due to the pressure fluctuations induced by the incident ultrasound waves [17]. The cavities grow (expansion) and collapse (compression) due to pressure recovery releasing large amount of energy locally, in the form of shock waves, and increasing the local temperature and pressure in a few μs [18]. The design of experiment and optimisation of free fatty acid reduction from KSO via UC was conducted by incorporating four reaction parameters: methanol to oil molar ratio, catalyst amount, reaction time and amplitude using response surface methodology (RSM) and four-way analysis of variance (ANOVA). The presented paper has been also applied the MS in the same reactor to treat high FFA of KSO in order to make a comparison with UC in terms of FFA reduction efficiency, reaction time and esterified efficiency.

2 Materials and methods

2.1 Materials

KSO was procured from the East Jawa Province, Indonesia. A standard mixture of 37 fatty acid methyl ester was purchased from Sigma Aldrich, Malaysia < www.sigmaaldrich.com/malaysia.html > [6]. The chemicals used for this work were anhydrous methanol, sulphuric acid, anhydrous sodium sulphate, toluene, 2-propanol, phenolphthalein, acetic acid (glacial), cyclo hexane, Wijs solution, potassium iodide, sodium thiosulphate pentahydrate, starch, chloroform, ethanol, hydrochloric acid and acetone. The physiochemical properties of KSO had been reported in previous paper [19]. Acid value of KSO was calculated by AOCS Cd 3d-63 method [6] and found to be 11.80 mg KOH/g (5.9 % FFA). The saponification, iodine, density (at 20 °C), viscosity and flash point values of KSO were 194 mg KOH/g, 102 g I_2/100 g, 0.91 g/cm^3, 36.21 mm^2/s and 210 °C. The fatty acid composition of KSO was 25.75 wt% (palmitic acid), 5.43 wt% (stearic acid), 24.32 wt% (oleic acid), 42.46 wt% (linoleic acid) and 2.

04 wt% (arachidic acid). All chemicals were analytical grade and procured from Merck, Malaysia <www.merck.com.my> [6].

2.2 Ultrasonic cavitation reactor configuration and experimental procedure

The ultrasonic processor used was Cole-Parmer® 500 W and 20 kHz, in combination with a standard probe in 15-mm Titanium alloy Ti-6Al-4 V. The pretreatment UC reactor with a 100-mL capacity is made up by double jacket reactor. A schematic diagram of UC system is given in Fig. 1. The acid esterification reaction of KSO was carried out at 40 °C in an UC system in a presence of methanol and sulfuric acid as catalyst. Desired amount of methanol, catalyst, reaction time and amplitude were employed on each run, which designed by central composite design (CCD). A 50 g of KSO was heated up to 40 °C in UC reactor. The methanol-catalyst solution at specific amount was added in UC reactor. The completion of esterification reaction mixture was settled under gravity by using separating funnel for 2 h. The by-product and catalyst were discharged, and the products were washed with deionised warm water at 40 °C to remove impurities in the product. The remaining methanol and water in the product were evaporated using a rotary evaporator under vacuum. A 5 g of anhydrous sodium sulphate was added to product sample to ensure complete removal of water and filtered by using filter paper (541 Whatman). Acid value content of the product was analysed by following AOCS Cd 3d-63 method. All the experiments have been performed for three replicates runs to ensure data reproducibility.

2.3 Fatty acid methyl ester analysis

The esterification of the samples is further verified and analysed following the EN 14103 standard method using GC-FID—Agilent Technologies, 7890A GC System equipped with automatic injector, split, FID and capillary column—methylpolysiloxane (DB-23) (60 m × 0.25 mm × 0.25 μm). The temperature programme employed starts at 100 °C holding for 2 min, heating at 10 °C/min until 200 °C, heating at 5 °C/min until 240 °C and holding for 7 min. Helium is used as a carrier gas at the flow rate of 4 mL/min. The hydrogen and air are used at the flow rate of 50 and 400 mL/min for flame.

2.4 Esterified efficiency calculations

The esterified efficiency is calculated using the following Eq. 1 [14].

$$\text{Esterified efficiency} = \frac{\frac{\text{Initial acid value} - \text{Final acid value}}{2 \times 100} \times \text{Mass of treated product (g)}}{\text{Power} \times \text{Reaction time (J)}} \qquad (1)$$

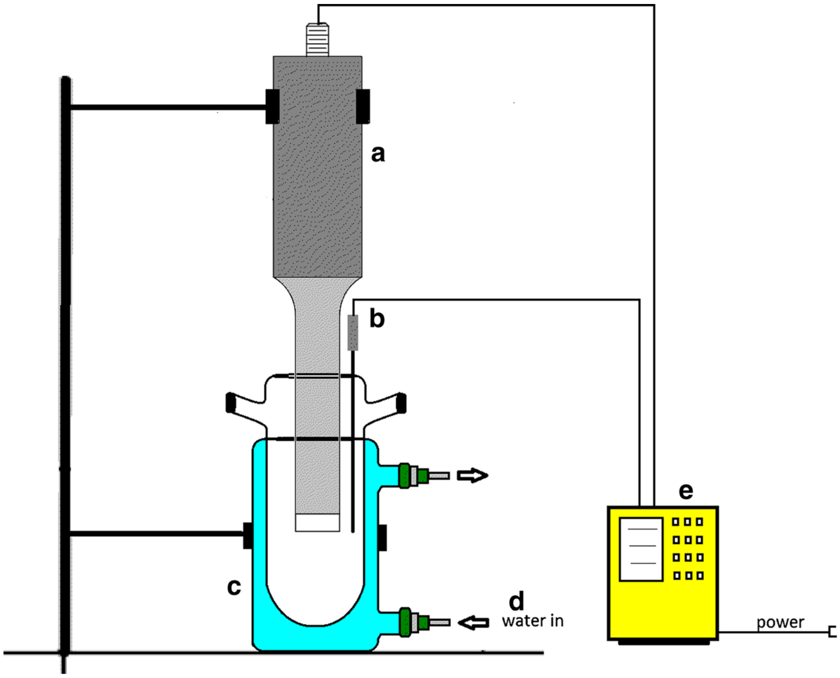


Fig. 1 A schematic diagram of ultrasonic cavitation system. **a** Transducer, **b** thermometer, **c** ultrasonic reactor, **d** cryostat, **e** ultrasonic generator

2.5 Experimental design

Parametric studies on the optimised orifice plate have been done by employed CCD using Design Expert 8.0 software. Further optimisation of operating parameters was analysed by numerical optimisation technique in RSM. The experimental arrays were designed by CCD. The reaction variables and its respective ranges are shown in Table 1. The independent input process variables are primarily classified in terms of low and high levels. The factors are distributed into versatile points called axial, centre and factorial points. The axial points are coded by CCD with $-\alpha$ and $+\alpha$. Low and High level factor points are designated as −1 and +1. Whereas, centre points are coded with 0 and repeated experimental arrays designed on centre points.

3 Results and discussion

3.1 Statistical analysis

RSM was applied for determining the optimum reaction conditions for esterification of KSO via UC that incorporates alcohol to oil molar ratio, catalyst amount, reaction time and amplitude simultaneously. The FFA content in percentage (FFA %) was considered as an output response at a given set of variables. Design Expert 8.0 software [14] designed 21 experiments using CCD. All the experiments were performed according to the designed runs, and each run was operated by following the set process conditions. At each run, FFA % was calculated as the response variable. Table 2 shows the detailed experimental designed run along with its output response variables for KSO. The FFA content in KSO was reduced to 0.49–1.76 %.

Table 1 Design parameters for esterification reaction

Variable	$-\alpha$	−1	0	+1	$+\alpha$
Alcohol to oil molar ratio	6.59	10	15	20	23.41
Catalyst amount (wt%)	3.30	5	7.5	10	11.70
Reaction time (min)	14.89	20	27.5	35	40.11
Amplitude (%)	13.18	20	30	40	46.82

ANOVA was employed to statistically analyse the output response. The ANOVA results for the KSO pretreatment are illustrated in Table 3. The model *p* value determined the percentage of error in the model along with individual and combined effects of the input variables. The model *p* value obtained was less than 0.05, which implied that the model was significant for input designed variables with respect to the response. The *F* value describes the reliability of the fitted model with the output response. The highest *F* value for amplitude indicates a highest influence on the output response. Catalyst amount, alcohol to oil molar and reaction time were less significant input variables when compared to amplitude. Most of the combined manipulative variables show the significant behaviour based on their p-values. The output response was fitted to the input process variables through regression analysis (R^2). The regression analysis produced response surface equation for the output response model in terms of actual and coded terms. This equation represented a second order polynomial regression model as shown in Eq. 2.

$$\begin{aligned} \mathrm{FFA}\,(\%) = {} & +0.63 + 0.042\mathrm{A} - 0.025\mathrm{B} + 0.032\mathrm{C} - 0.11\mathrm{D} - 0.28\mathrm{AB} - 0.069\mathrm{AC} - 0.24\mathrm{AD} \\ & + 0.11\mathrm{BC} + 0.23\mathrm{BD} + 0.12\mathrm{CD} \end{aligned} \tag{2}$$

The fitting of the model was assessed by the R^2 for the KSO pretreatment, which showed that the models can be used for predicting the output response with reasonable precision. The value of the evaluated adjusted R^2 is in a good agreement with R^2. A sufficient precision determines the signal to noise ratio; and a ratio greater than 4 is desirable. Indeed, ratio greater than 4 was obtained for KSO model, indicating sufficient signal.

3.2 Predicted versus actual terms

The plotted graph for the predicted value of FFA versus the actual experimental FFA value was shown below in Fig. 2. Result indicates that all the experimental values were well fitted with the predicted value. The points were also closed towards the centre linear line. Only a few experimental points were deviated and not close to the centre line. Most of the points lie between the ranges of 0.49–0.73 % of FFA.

3.3 Parametric effects and optimisation of acid pretreatment process

The profiles of the calculated FFA % with incorporation of two design parameters are represented as 3D plots in Fig. 3(a–f). Figure 3a represents the two parameters

Table 2 Esterification experimental designed and response of free fatty acid content

Run no.	Point type	Alcohol to oil molar ratio	Catalyst amount (wt%)	Reaction time (min)	Amplitude (%)	Response FFA (%)
1	Axial	15.00	7.50	14.89	30.00	0.70
2	Factorial	10.00	10.00	20.00	40.00	1.04
3	Axial	15.00	7.50	27.50	46.82	0.49
4	Centre	15.00	7.50	27.50	30.00	0.65
5	Factorial	20.00	5.00	35.00	40.00	0.59
6	Factorial	20.00	10.00	20.00	20.00	0.70
7	Axial	15.00	7.50	27.50	13.18	0.87
8	Axial	23.41	7.50	27.50	30.00	0.73
9	Axial	15.00	3.30	27.50	30.00	0.70
10	Centre	15.00	7.50	27.50	30.00	0.53
11	Factorial	10.00	5.00	20.00	20.00	0.73
12	Centre	15.00	7.5	27.50	30.00	0.67
13	Axial	15.00	7.50	40.11	30.00	0.67
14	Centre	15.00	7.50	27.50	30.00	0.76
15	Factorial	10.00	10.00	35.00	40.00	1.76
16	Factorial	10.00	5.00	35.00	20.00	0.52
17	Factorial	20.00	10.00	35.00	20.00	0.67
18	Centre	15.00	7.50	27.50	30.00	0.72
19	Axial	15.00	11.70	27.50	30.00	0.62
20	Axial	6.59	7.50	27.50	30.00	0.59
21	Factorial	20.00	5.00	20.00	40.00	0.59

correlation between alcohol to oil molar ratio and catalyst amount against FFA % at constant reaction time and amplitude. This figure shows maximum FFA % was reduced at the methanol to oil ratio of 10:1. Further increase in methanol to oil molar ratio did not significantly reduce FFA % and also caused separation problem. Excess

Table 3 ANOVA analysis for esterification process

	Sum of squares	df	Mean square	*F* value	*p* value prob > F	
Model	1.31	14	0.09	5.44	0.0234	Significant
A—Methanol to oil molar ratio	0.0098	1	0.0098	0.57	0.4787	
B—Catalyst amount (wt%)	0.003613	1	0.003613	0.21	0.6627	
C—Reaction time (min)	0.014367	1	0.014367	0.84	0.3958	
D—Amplitude (%)	0.072	1	0.072	4.20	0.0863	
AB	0.26	1	0.26	15.37	0.0078	
AC	0.04	1	0.04	2.24	0.1851	
AD	0.20	1	0.20	11.42	0.0149	
BC	0.10	1	0.10	5.96	0.0504	
BD	0.17	1	0.17	10.07	0.0192	
CD	0.11	1	0.11	6.63	0.0420	
Residual	0.10	6	0.017			
Lack of fit	0.07	2	0.036	4.71	0.0887	Not significant
Pure error	0.0307	4	0.007675			
Cor total	1.41	20				
Statistic terms	*R*-squared = 0.926977		Adj *R*-squared = 0.75659	Adeq precision = 11.1923		
	Std. dev. = 0.13		Mean = 0.73	C.V% = 18.04		

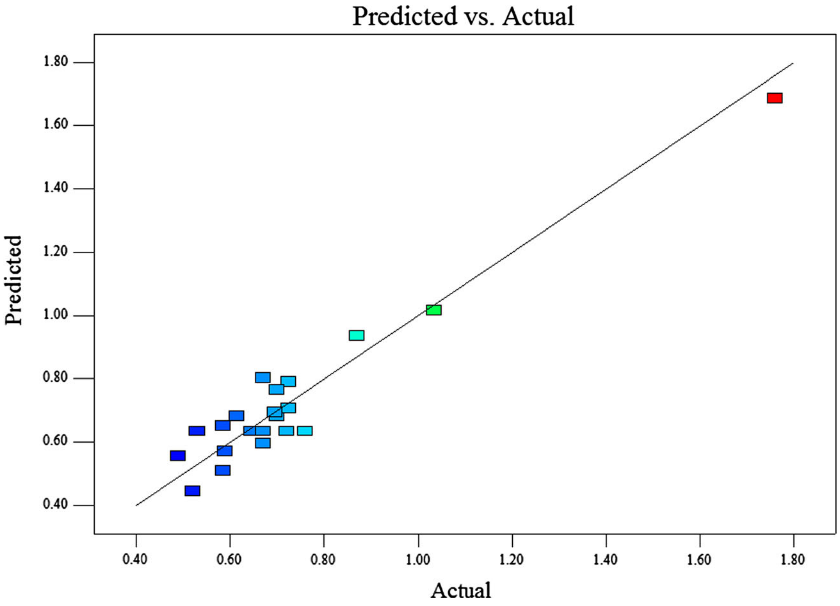


Fig. 2 Predicted versus actual plot for acid esterification of kapok seed oil

methanol is used to transfer reaction equilibrium towards product, and it also helps to dissolve the water during the reaction in order to avoid hydrolysis. In this figure, it can be seen that higher significance level of methanol to oil molar ratio than catalyst amount in maximising the response. Figure 3c depicts the 3D plot of the FFA % for the combined parameters of amplitude and alcohol to oil molar ratio. Results revealed that that FFA % decreased significantly by increasing the amplitude. Higher methanol to oil ratio was reduced the FFA %, but not as significant as amplitude. It could be attributed to the longitudinal vibrations of the ultrasonic probe were transmitted into the liquid as ultrasonic waves consisting of alternate expansions and compressions. The pressure fluctuations give birth to microscopic cavities, which expand during the negative pressure excursions, and implode violently during the positive excursions. As the cavities collapse, millions of shock waves eddy, and extremes in pressure and temperature are generated at the implosion sites. Thus, higher amplitude results in an increase of mass transfer efficiency between the methanol and oil, but up to certain value, the esterification performance decreased resulting in no significant increase in the cavitational effect. The response plot of FFA % incorporating the interaction between reaction time and catalyst amount is given in Fig. 3d. From this figure, it can be observed that marginal decrease in FFA % for further decrease of catalyst amount below 6 wt%, but significantly increased at the catalyst amount from 6 to 10 wt%. Another observation is FFA % tends to significant decreased at reaction time from 20 to 29 min, but beyond this reaction time, FFA reduction was not significantly. Figure 3e depicts the effect of catalyst amount and amplitude on FFA reduction. Based on the results, the amplitude was more influenced than catalyst amount on FFA reduction. Figure 3f illustrates the effect of reaction time and amplitude on FFA %. In terms of FFA reduction, the amplitude has higher impact compared to reaction time. The FFA value decreased gradually with an increase of amplitude from 20 to 40 %. However, there was no significant effect on FFA reduction for the amplitude between 40 and 47 %. Mass transfer between oil and alcohol is enhanced when specified residence time is given for reactants to interact at cavitation zone. Cavitation event is also the driven force to increase the esterification reaction rate.

Numerical optimisation tool in Design Expert 8.0 software has been used to optimise the operating parameters of acid esterification pretreatment process for UC technology. All the input variables set are in-range with output response set at minimum level. The numerical optimisation tool gives the optimised condition for methanol to oil ratio of 16.23:1, catalyst amount of 9.94 wt%, reaction time of 20.08 min and amplitude at 32.04 % with standard deviation of 0.13 and point predicted FFA of 0.49 %.

3.4 Comparative performance of mechanical stirring and ultrasonic cavitation

The optimised process parameters between UC and MS were compared—see Table 4. It can be observed that UC requires shorter time to reduce FFA compared to MS. This could be attributed to the high intensity of micro level turbulence generated by oscillating cavities with high interfacial area. UC

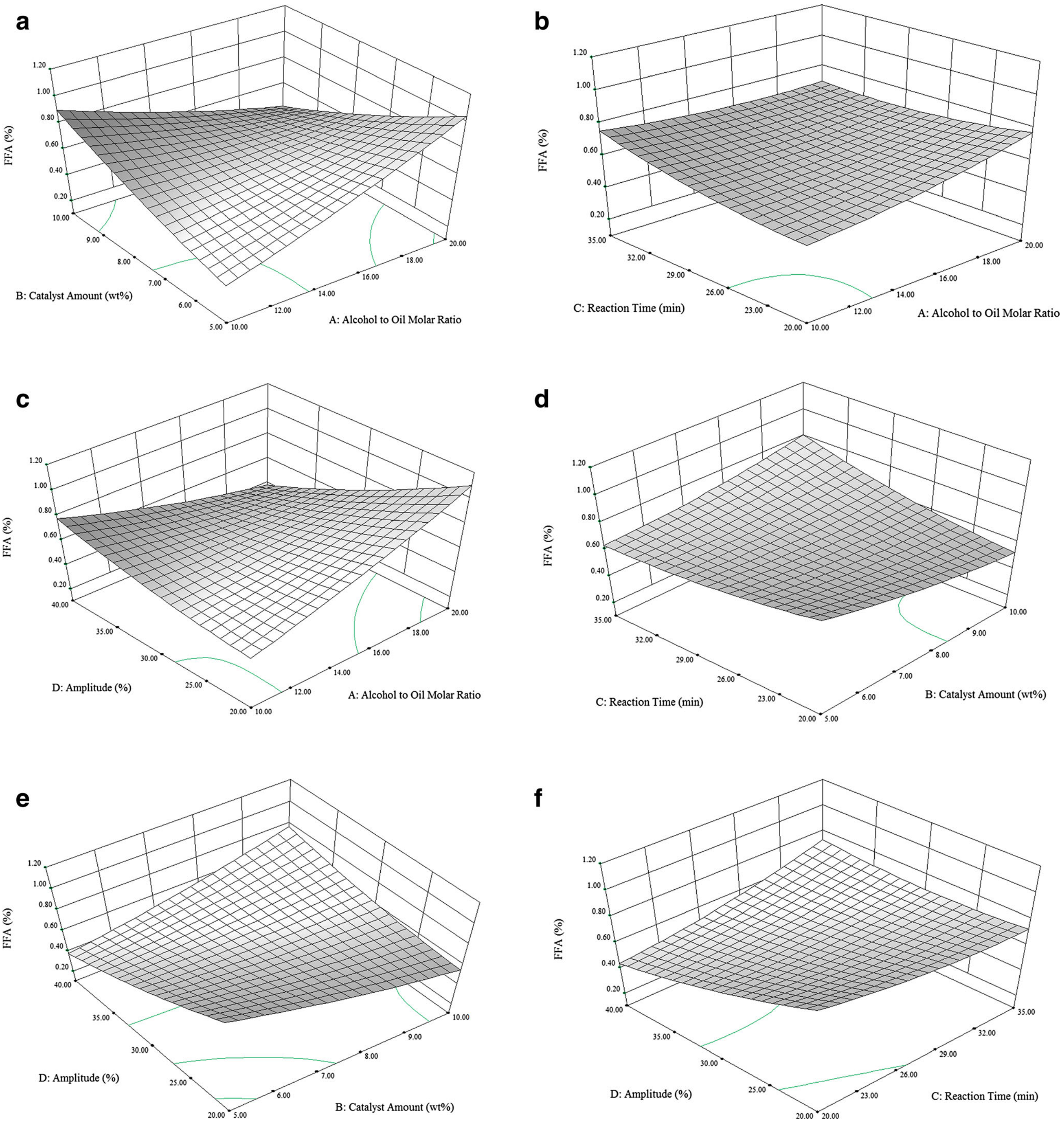


Fig. 3 Parametric analysis of reaction variables

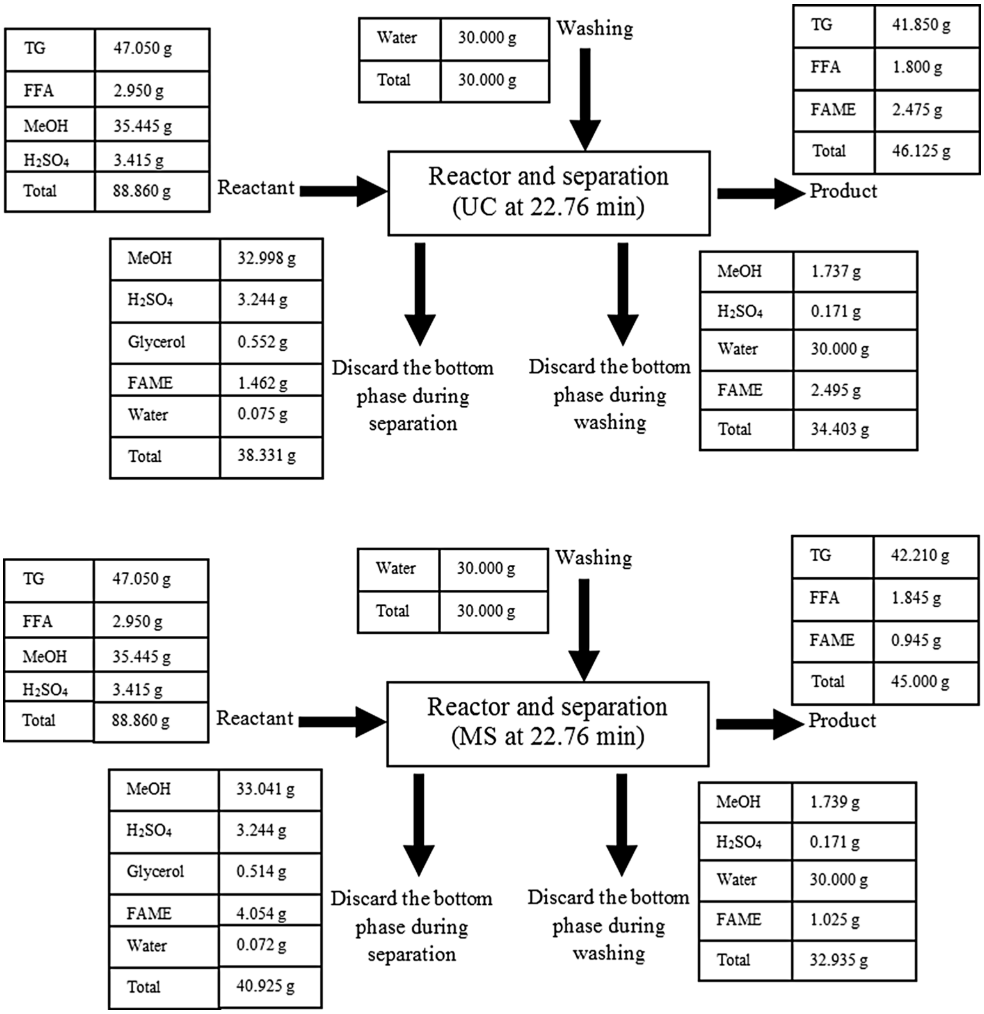
reactor is very effective in eliminating the mass transfer resistance during the reaction and hence higher esterification performance. The oil phase towards the methanol-oil interface was only dependent on the stirrer bar (MS). This could be a limitation and produce poor mass transfer between the reactants resulted in slow esterification rate. The reaction time and esterified efficiency of HC were 4.5-fold shorter and 10.5-fold higher than MS.

As the FFA content in raw KSO was 5.9 %, so the triglycerides content in KSO was 94.1 %. In UC approach, the obtained FFA reduction was 93.2 % and methyl ester conversion was 6.6 %. Therefore, only FFA 5.5 %

Table 4 Comparison of optimised conditions in different intensification approaches

Optimum conditions	This study	
	Ultrasonic cavitation	Mechanical stirring
Methanol to oil ratio	16.23:1	16.23:1
Catalyst amount (wt. %)	9.94	9.94
Amplitude (%)	32.04	-
Revolutions per minute (rpm)	-	600
Temperature (°C)	40	60
Reaction time (min)	20.08	100
Esterified efficiency, 10^{-7} (g/J)	18.12	1.72
FFA (%)	0.4	0.5

(5.9 − 0.4 = 5.5 %) in KSO were converted to methyl ester, and 1.1 % (94.1 − 93.0 = 1.1 %) of triglycerides in raw KSO were converted to fatty acid methyl ester. Consequently, only 0.4 % of unreacted FFA and 93.0 % (94.1 − 1.1 = 93.0 %) of triglycerides is going to be converted into methyl ester in the following alkali catalysed transesterification stage. In MS approach, the obtained FFA reduction was 89.8 %, and methyl ester conversion was 6.1 %. Consequently, only FFA of 5.3 % (5.9 − 0.6 = 5.3 %) in KSO were converted to methyl ester, and 0.8 % (94.1 − 93.3 = 0.8 %) of triglycerides in raw KSO were converted to fatty acid methyl ester. As a result only 0.6 % of unreacted FFA and 93.3 % (94.1 − 0.8 = 93.3 %) of triglycerides is going to be converted into methyl ester in the following alkali catalysed transesterification stage. The FFA has been reduced to 0.5 % for MS approach and 0.4 % for HC approach. Mass balance results of UC and MS approaches in pretreatment esterification process are depicted in Fig. 4. It was found that the reactant used per produced biodiesel in UC process was 1.192 g g^{-1} compared to MS process with 3.122 g g^{-1}. This makes the UC process more environmental friendly.



TG	47.050 g
FFA	2.950 g
MeOH	35.445 g
H_2SO_4	3.415 g
Total	88.860 g

Water	30.000 g
Total	30.000 g

TG	41.850 g
FFA	1.800 g
FAME	2.475 g
Total	46.125 g

MeOH	32.998 g
H_2SO_4	3.244 g
Glycerol	0.552 g
FAME	1.462 g
Water	0.075 g
Total	38.331 g

MeOH	1.737 g
H_2SO_4	0.171 g
Water	30.000 g
FAME	2.495 g
Total	34.403 g

TG	47.050 g
FFA	2.950 g
MeOH	35.445 g
H_2SO_4	3.415 g
Total	88.860 g

Water	30.000 g
Total	30.000 g

TG	42.210 g
FFA	1.845 g
FAME	0.945 g
Total	45.000 g

MeOH	33.041 g
H_2SO_4	3.244 g
Glycerol	0.514 g
FAME	4.054 g
Water	0.072 g
Total	40.925 g

MeOH	1.739 g
H_2SO_4	0.171 g
Water	30.000 g
FAME	1.025 g
Total	32.935 g

Fig. 4 Mass balance of pretreatment esterification process via ultrasonic cavitation and mechanical stirring

4 Conclusions

An esterification reaction has been carried out for KSO pretreatment via UC. RSM and four-way ANOVA have been well applied for designing and optimising the experiments with respect to methanol to oil molar ratio, catalyst amount, reaction time and amplitude. The optimum conditions from RSM were 16.23:1 molar ratio of methanol to oil in the present of 9.94 wt% catalyst with 32.04 % amplitude for 20.08-min reaction time at 40 °C. About 10.5-fold higher esterified efficiency and 4.5-fold shorter reaction time using UC compared to MS.

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