https://doi.org/10.3311/PPch.42974

Periodica Polytechnica Chemical Engineering, 70(2), pp. 260–274, 2025

Microwave-Assisted Transesterification of *Argemone mexicana* Oil: Parametric and Kinetics Study

Milap G. Nayak[1]*, Amish P. Vyas[2]

[1] Chemical Engineering Department, L.D. College of Engineering, Gujarat Technological University, 382424 Ahmedabad, Gujarat, India
[2] School of Engineering, Indrashil University, At. & Po Rajpur Taluka Kadi, Rajpur, Gujarat 384440, India
* Corresponding author, e-mail: milapnayak0510@gmail.com

Received: 14 December 2025, Accepted: 10 March 2026, Published online: 04 June 2026

Abstract

Argemone mexicana is one of the high non-edible oil plants easily grown in barren land. The current study employs microwave-assisted transesterification of *Argemone mexicana* oil (AO) into value-added biodiesel. A systematic experimental approach was utilized to investigate the effect of methanol-to-oil molar ratio, catalyst amount, reaction temperature and time on free fatty acid conversion and *Argemone mexicana* oil methyl ester (AOME) at constant stirring using microwave power. Response trends suggested that the biodiesel yield was maximized to 96% with increasing time, molar ratio, catalyst amount, and temperature up to an optimum value of 3 min, 9:1 methanol-to-oil molar ratio, 1% NaOH, and 60 °C, respectively. Beyond these conditions equilibrium limitations and possible reverse reactions led to reduced yield. FTIR was employed to do a qualitative investigation of AO and AOME. The emergence of a new signal at 1435 cm^{-1} confirmed the development of AOME. The ^1H-NMR research found that 94.91% of the oil was converted into biodiesel. The kinetics of AO transesterification were first-order, with a rate constant 0.726 min^{-1} at 60 °C and an activation energy 27.49 kJ mol^{-1}. The non-thermal impact of the microwave was also revealed by the high frequency factor 14871 min^{-1}. At 60 °C, the changes in Gibbs free energy, enthalpy and entropy were 94.09 kJ mol^{-1}, 24.734 kJ mol^{-1} and −0.2083 kJ mol^{-1} K^{-1}, respectively. The thermodynamic study revealed that it was an endergonic and non-spontaneous process.

Keywords

non-edible oil, transesterification, parametric study, kinetic and thermodynamic study, physicochemical property

1 Introduction

At present, oil, natural gas and coal are the major raw materials governing energy demand worldwide [1]. However, with the pace of urbanization and industrialization, these constrained fuels are estimated to be depleted within the 21st century [2]. However, the most severe issue confronting the globe in the utilization of finite fossil fuel supplies is climate change, which is caused by rising global energy consumption and the buildup of atmospheric CO_2 during the burning of fossil fuels [3–5]. Furthermore, increased public awareness and severe regulations imposed by governments to reduce pollution boost the development and use of renewable and sustainable energy sources [6]. Biodiesel has recently gained significant importance due to its renewable and eco-friendly nature. It has a carbon-neutral feature, which prevents nature from being contaminated by CO_2 [7]. It is obtained by transesterification of various edible and non-edible oil-bearing feedstock [8, 9]. The major cost of biodiesel production and its commercialization depend on the availability of feedstock. The edible feedstocks are produced in large amounts, but are criticized for their use as a fuel due to the severe food crisis [10]. Several sources, such as waste oil or fats, contain high free fatty acids and water. These are adversely affecting the production and purity of biodiesel. Moreover, pre-treatment of oil, separation and purification are necessary for its use as a feedstock [11]. In contrast, non-edible oil crops can easily grow in less fertile or barren land and do not compete with the land used for the edible oil crops cultivation [12].

Argemone mexicana is a non-edible weed, 80 to 150 cm tall. It grows in agricultural and wasteland. It is mostly associated with edible plants such as mustard seed and potato in India [13]. It causes health hazards to human beings and animals due to the presence of toxic

Cite this article as: Nayak, M. G., Vyas, A. P. "Microwave-Assisted Transesterification of *Argemone mexicana* Oil: Parametric and Kinetics Study", Periodica Polytechnica Chemical Engineering, 70(2), pp. 260–274, 2026. https://doi.org/10.3311/PPch.42974

sanguinarine and dihydro sanguinarine alkaloids [14]. The adulteration of edible oil with *Argemone mexicana* oil (AO)causes glaucoma and epidemic dropsy, a disease resulting in neuro paralysis and death of several people [15]. The sanguinarine interferes with the oxidation of pyruvic acid. Pyruvic acid accumulation causes dilation of capillaries and small arterioles [16]. *Argemone mexicana* weed, despite its unsuitability for cattle feed, containing higher oil yield. It requires less care for cultivation. It can be explored as a feedstock for biodiesel synthesis. The *Argemone mexicana* seed is brownish-black, nearly spherical and resembles a mustard seed. A single plant produces 18,000 to 36,000 seeds, which, on self-pollination, rapidly propagate *Argemone mexicana* [17]. It can be harvested in a largely barren land. Plants grown at 40 cm apart from each other in one-hectare barren land can accommodate $4·10^8/(40·40)$, which is equal to 2,50,000 plants. The average seeds obtained from one plant per year are 0.7 to 1 kg. Hence, the annual feedstock productivity is 1,75,000 to 2,50,000 kg ha^{-1}. The seed contains 35 to 40% yellowish-brown oil [18]. Thus, the approximate annual oil production is 61–87 t ha^{-1}.

The majority of works reported in the literature are on homogeneous and heterogeneous transesterification using conventional heat sources. Since heating takes place from outside to inside, conventional heating requires more time and energy due to the loss of energy in conduction and convection [19, 20]. Microwave treatment, due to heating at the molecular level and the inverse temperature gradient, is more effective for chemical synthesis. It is an electromagnetic wave, which influences the polar molecule, such as methanol [21]. Microwave-assisted trans-esterification reaction required 10 times less energy and reduced reaction time by 13 times than conventional heating for a similar yield under the given operating conditions [22].

In the literature, biodiesel production from *Argemone mexicana* using CaO [23] and NaOH [24] catalysts in a conventional heating medium were reported. However, microwave-assisted transesterification of AO is not reported in the literature. The objectives of the present study are: (i) to explore microwave-assisted transesterification of AO for biodiesel production (ii) to investigate the effect various parameters, such as molar ratio of methanol-to-oil, temperature, catalyst amount and time on the yield of *Argemone mexicana* oil methyl ester (AOME), and (iii) a kinetic and thermodynamic study of alkali-catalyzed AOME formation.

2 Material and methods

Argemone mexicana seeds were collected from the barren land in Gandhinagar, Gujarat. The seeds were mechanically crushed to obtain pale yellow AO. The solid impurity in AO was removed by filter paper. All chemicals, such as NaOH, KOH, methanol and ethanol (Loba Chemie, Mumbai, India) were of analytical grade.

2.1 Physico-chemical property

Gas chromatography-mass spectrometry (GC-MS) (Fig. S1 and Table S1 in the Supplement) of AO was carried out to identify the major fatty acids present in it. Table 1 shows the major components are linoleic, palmitic, oleic and stearic acid. Based on fatty acid composition, the degree of unsaturation of AO was 83.47%. The molecular mass of *Argemone mexicana* oil MW_{AO}, based on fatty acid composition presented in Table 1, is determined by Eq. (1):

$$\begin{aligned} MW_{\mathrm{AO}} &= 3 \cdot MW_{\text{fatty acid}} + MW_{\text{glycerol}} - 3 \cdot MW_{\text{water}} \\ &= 3 \cdot 284 + 92 - 3 \cdot 18 = 890 \ \mathrm{g\ mol^{-1}} \end{aligned} \tag{1}$$

where MW stands for the molar mass of the component given in the subscript.

AO was analyzed to find its physicochemical properties as per standard methods and listed in Table 2. The acid value of AO was determined to find the initial free fatty acid (FFA) present in the oil. The observed acid value and FFA content of AO were 1.23 and 2.46%, respectively. It has low FFA content (<3%), so pre-treatment, i.e., esterification process, was not required, and homogenous alkali catalyst NaOH was used directly for the trans-esterification reaction [25]. The measured saponification value is 191 mg g^{-1}. Saponification value suggests the amount of alkali catalyst required to saponify the oil. It is the measure of its chain length.

2.2 Experimental procedure

Fig. 1 shows the experimental setup for microwave-assisted transesterification of AO. Raga's scientific microwave synthesizer (SS batch reactor, 700 W max. power supply, Raga Tech, Pune Maharstra state, India) was used for the trans-esterification of oil. The experiments were carried out at 2.45 GHz, maximum 700 W microwave power, and at constant magnetic stirring. The experimental setup consists of a 100 mL glass flask attached to a water-cooled condenser at the top. It was charged with 5 mL (4.49 g) oil, different volumes of anhydrous methanol (molar ratio 3:1 to 18:1), and varying amounts of catalyst (0.25 to 2 wt%). A Raytec

Table 1 Fatty acid composition of *Argemone mexicana* oil

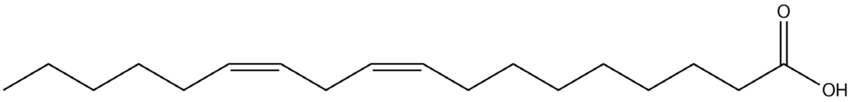
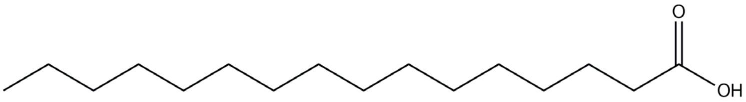
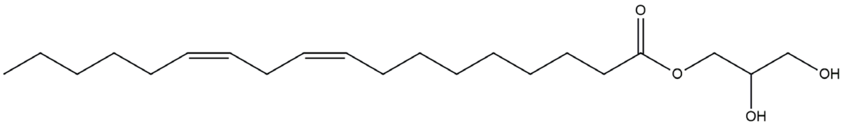
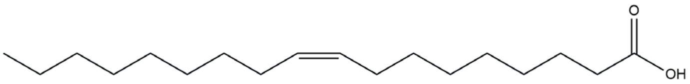
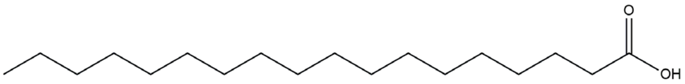
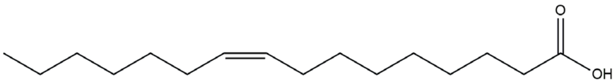
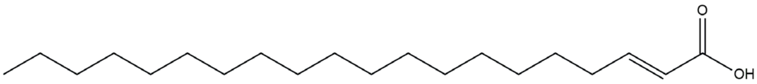
Structure	Name	Amount
	Linoleic acid C18:02	57.74%
	Palmitic acid C16:00	17.60%
	Glyceryl linoleate C19:02	10.04%
	Oleic acid C18:01	9.61%
	Stearic acid C18:00	4.35%
	Palmitoleic acid C16:01	0.32%
	Eicosenoic acid C20:01	0.34%

Table 2 Physicochemical properties of *Argemone mexicana* oil

Properties (unit)	AO
Specific gravity*	0.898
Viscosity at 40 °C (mm^2 s^{-1})	35.61
Cloud point (°C)	−0.08
Saponification number (mg g^{-1})	191
Iodine number	76
Free fatty acid %	2.46
Acid number (mg KOH g^{-1})	1.23
Ester value of oil	189.7
Heating value (calorific value) (MJ kg^{-1})	39
Cetane number	57.53

* Specific gravity is density of oil related to the density of water at 25 °C.

infrared temperature sensor (Raytek, Maharsatra, India) with a controller was used to monitor and control the reaction temperature. Reaction temperature can be set at a constant value using a proportional-integral-derivative (PID) controller. Experiments with parametric and kinetic study were carried out using precise temperature control. The experiments were performed in a microwave reactor by changing different parameters, like molar ratio of methanol-to-oil, reaction time, catalyst amount and temperature.

At the end of the reaction, the mixture was cooled and allowed to settle into two layers as presented in Fig. 2. Biodiesel and oil phase, due to lower density, separated at the top, while the glycerol and methanol phase settled at the bottom. The top layer was isolated and heated at 70 °C for several minutes to remove traces of methanol. The 3:27 test was used to check top-layer containing biodiesel purity [26]. The yield of AOME and free fatty acid (FFA) conversion of AOME was determined as per Eqs. (2) and (3) for every experimental run.

Fig. 1 Experimental setup for microwave synthesis of AOME

Fig. 2 Separation of AOME from glycerol

$$\text{Yield} = \frac{\text{mass of AOME produced}}{\text{Theoretical maximum AOME produced}} \cdot 100 \quad (2)$$

$$\text{FFA conversion} = \frac{AV_{\text{initial}} - AV_{\text{final}}}{AV_{\text{initial}}} \cdot 100 \quad (3)$$

AV is the acid value of the sample. The acid value of biodiesel is defined as the mass in milligrams of potassium hydroxide (KOH) or sodium hydroxide (NaOH) required to neutralize the free fatty acids (FFAs) present in 1 g of the biodiesel sample.

2.3 Kinetics of transesterification

In a transesterification reaction, one mole of triglyceride reacts with three moles of methanol and gives three moles of fatty acid methyl esters (FAMEs) and one mole of glycerol (Fig. 3). The overall reaction takes place in three steps. They are (i) conversion of triglycerides (TG) into di-glycerides (DG), (ii) DG into mono-glycerides (MG) and FAME, (iii) finally MG into monoalkyl esters and glycerol. Fig. 3 presents the overall transesterification reaction.

The kinetics study of the microwave-assisted transesterification of AO into AOME was performed to determine the order of reaction and the rate constant. The experiments were performed at 50 °C, 55 °C, 60 °C, 65 °C and 70 °C and different time interval. The optimized molar ratio and catalyst amount were used during the study. The Arrhenius equation was used to determine the activation energy and pre-exponential factor [27]. The Eyring–Polanyi equation was used to estimate the thermodynamic properties [28].

3 Results and discussion

3.1 Effect of variables on *Argemone mexicana* oil methyl ester yield

3.1.1 Methanol-to-oil molar ratio

The transesterification reaction requires theoretically three moles of methanol per one mol of triglyceride. On the other hand, esterification reaction requires one mol of methanol per one mol of free fatty acid. The effect of

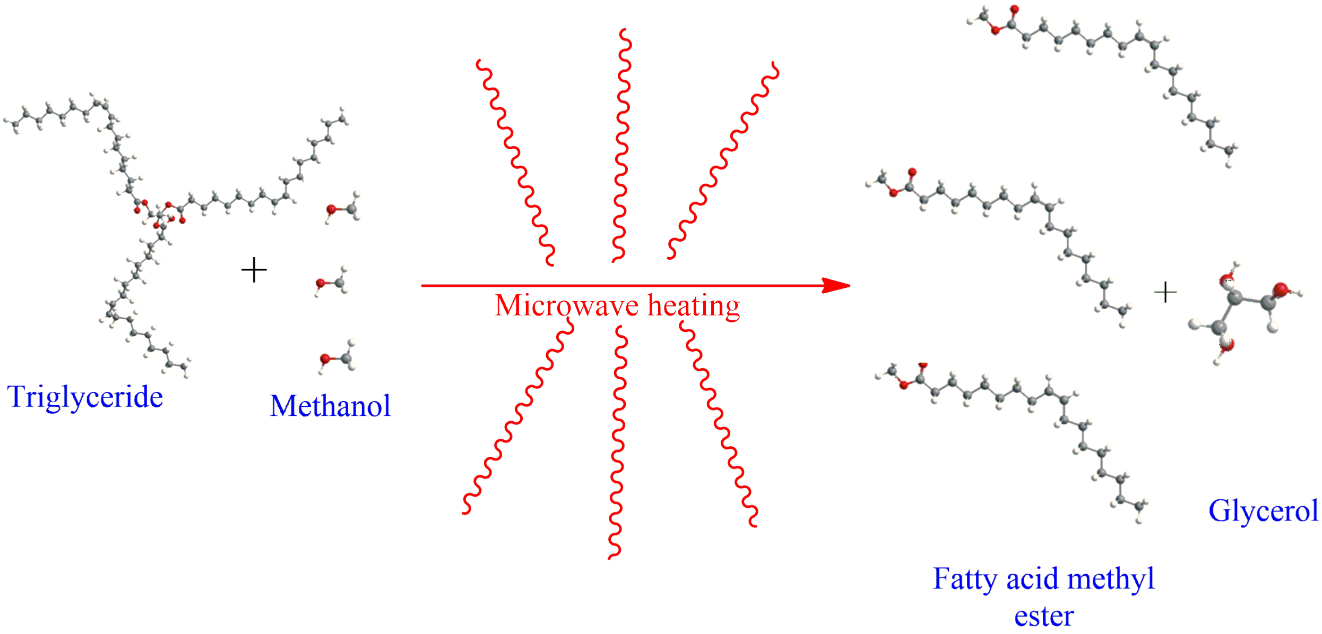


Fig. 3 Microwave-assisted transesterification of triglyceride into fatty acid methyl ester

methanol to methanol-to-oil molar ratio on AOME yield and FFA conversion was first determined by keeping other process parameters at 60 °C, 1 wt% NaOH and 3 min reaction time. Fig. 4 presents the FFA conversion, AOME yield and density of biodiesel at varying molar ratios from 3:1 to 18:1. With the increase in molar ratio from 3:1 to 9:1, both free fatty acid conversion and AOME yield were increased from 15% to 70% and 45% to 96%, respectively. The rise in methanol amount initiates the reaction in the forward direction with the successive conversion of triglyceride into di-glycerides, di-glyceride to mono-glycerides and finally mono-glycerides into FAME [29–31].

Supeno et al. [32] in the oleic acid esterification concluded that an excess volume of methanol reduced the viscosity of the reactants and enhanced the mass transfer rate for esterification and transesterification of oil. Yamin et al. [33] in another study, involving trans-esterification of waste cooking oil with methanol, concluced a similar finding at 9.4:1 molar ratio. Zhou et al. [34] demonstrated that the initial heterogeneous oil and methanol phase gradually converted into a homogeneous phase with the formation of FAME. The excess availability of methanol helped to dissolve the finished product from the unreacted oil phase and gave a higher yield and free fatty acid conversion. The decrease in density of the finished biodiesel phase from 898 kg m^{-3} to 883 kg m^{-3} suggests progress of conversion. However, AOME yield and FFA conversion reduced from 96% to 89% and 70% to 68%, with a further increase in the molar ratio from 9:1 to 18:1. At a higher molar ratio, the relative dilution of catalyst reduced the AOME yield [35]. Ulukardesler [36] conducted waste cooking oil (WCO) transesterification with methanol in presence of KOH catalyst and concluded that increasing the total volume of methanol reduces the catalyst concentration and decreases the biodiesel yield. Chauhan et al. [37] in transesterification of WCO with heterogeneous catalyst CaO-MgO-ZnO-TiO_2 observed maximum 95% biodiesel yield at a 15.5:1 methanol-to-oil ratio, 1.75 wt% catalyst, 65.5 °C, 3 h. This study revealed that excess methanol can dilute reactants, complicate separation biodiesel and glycerol phase separation. Agarwal et al. [38] reported that excess methanol increased the solubility of glycerol in biodiesel phase and hindered the separation process. It led to a decrease the yield of biodiesel [38]. Fadhil et al. [39] concluded that the presence of by-product glycerol drove the equilibrium in the backward direction and formed monoglyceride. The experiment with molar ratio suggests that 9:1 molar ratio gives maximum biodiesel yield and conversion of free fatty acid.

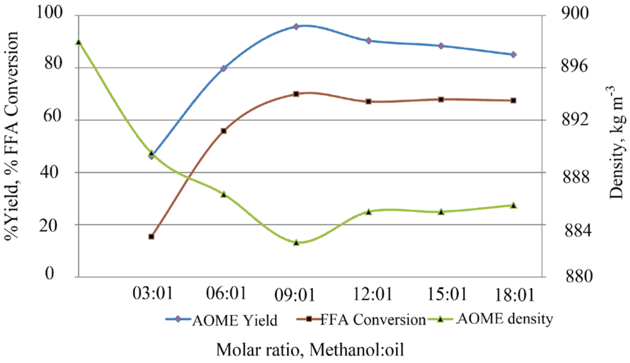


Fig. 4 Effect of molar ratio on AOME yield and FFA conversion at 60 °C, 1 wt% NaOH and 3 min reaction time

3.1.2 Catalyst amount

Effect of catalyst amount on AOME yield and FFA conversion was determined at 9:1 (optimized), 60 °C and 3 min reaction time. Fig. 5 reveals the increase in AOME yield and FFA conversion from 38% to 96% and 19% to 76% when the catalyst amount increased from 0.25 to 1 wt% of the oil.

It was supported by the fact that the catalyst first formed methoxy ions from methanol. The nucleophilic attack of methoxy ions onto the ester bond of triglyceride/fatty acid formed an active intermediate, which was further converted into FAME [40–42]. Thus, the presence of a catalyst initiates the formation of methoxy radicals from methanol, which leads to an increase in yield. Buchori et al. [43] concluded similar findings in the transesterification of WCO using zeolite catalyst. It was reported that the equivalent molar concentration of homogeneous catalyst linearly increased the trans-esterification as long as the catalyst concentration did not surpass that of alcohol [42].

When the catalyst amount increased from 1 wt% to 2 wt%, the AOME yield and FFA conversion decreased from 96% to less than 80% and 78% to 60%, respectively.

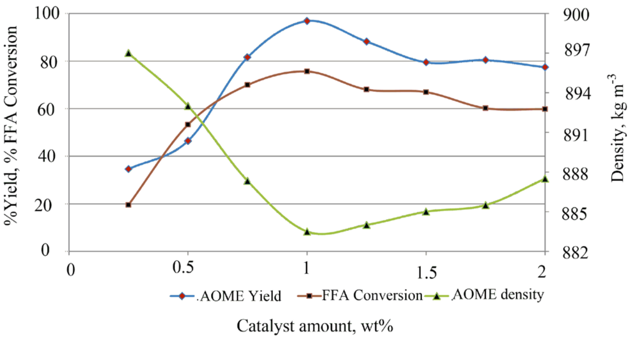


Fig. 5 Effect of catalyst amount on AOME yield and FFA conversion at 9:1 methanol-to-oil molar ratio, 60 °C and 3 min reaction time

Gel formation at the interface between two phases indicates higher selectivity towards undesired saponification reaction in preference to trans-esterification or esterification reaction. It was supported by Alhassan et al. [44] and Priambodo et al. [45] in their research. Miyuranga et al. [46] concluded that the catalyst increased total conversion and yield, but excess concentration hindered transfer of mass due to the formation of viscous phase. Berchmans and Hirata [47] and Cai et al. [48] reported that excess alkali catalyst formed a high viscosity emulsion with oil, which trapped methyl ester, and there was difficulty in biodiesel and glycerol separation. Also, soap formation during the reaction offered additional resistance for reactants, and the rate was controlled by the diffusion of methanol into oil [49]. Experimental investigations suggest 1 wt% NaOH is optimum for a higher yield of AOME.

3.1.3 Reaction temperature

The reaction temperature plays an important role in the transesterification and esterification of oil and free fatty acids, respectively. The effect of temperature on the yield of AOME and conversion of FFA was carried out by varying the temperature from 45 °C to 70 °C with an increment of 5 °C as shown in Fig. 6. Experiments were carried out for 3 min, at 60 °C and a 9:1 molar ratio of methanol to oil. With the increase in temperature from 45 °C to 60 °C, the yield of AOME and conversion of FFA increased from 86% to 96% and 52% to 74%, respectively. The increase in reaction temperature produces a localized hotspot near the methanol phase and provides the energy required to overcome the activation energy for transesterification. In addition, a reduction in viscosity of the mixture improves mobility and reduces mass-transfer resistance. Buchori et al. [43] concluded that a rise in the temperature pushed the reaction toward complete conversion for transesterification of WCO. A similar finding was suggested by Liu et al. [50]. The endothermic nature of the transesterification reaction favors a rise in temperature of the reaction mixture [51]. However, the temperature rise above 60 °C adversely affects the AOME yield. It decreased from 96% to 72% when the temperature of the reaction rose from 60 °C to 70 °C. The bubble formation observed in the reaction mixture and the vaporization of methanol from the reaction mixture reduced the yield. Similar findings were suggested in previous studies [51–54]. Also, undesired saponification of triglyceride by the alkaline catalyst predominated over the trans-esterification reaction, which reduces the AOME yield [55]. The increase in FFA conversion of oil from 74% to 80% with an increment of temperature is mainly due to the conversion of FFA into its salt. Thus, a reduction in methanol amount, the formation of voids, and undesired saponification reduce the yield and desired conversion above 60 °C. Hence, in this experiment, 60 °C is selected as an optimum temperature.

3.1.4 Reaction time

The microwave-assisted transesterification reaction is rapid and effective compared to the conventional heating system due to inside-out heating. The heating takes place at the molecular level as compared to conventional heating with no loss of microwave energy [4]. Fig. 7 presents the effect of reaction time on microwave-assisted transesterification of AO. The process parameters were 700 W microwave power, 9:1 methanol-to-oil molar ratio, 1 wt% catalyst and 60 °C temperature.

It was observed that within 4 min, conversion of FFA and yield of AOME increased up to 75% and 97%, respectively. The drastic reduction in reaction time, as well as higher yield, suggests rapid heating of the reaction mixture by microwave heating. The thermal and nonthermal effects associated with microwave-assisted transesterification provide energy to promote a high reaction rate along with higher pre-exponential factors [56, 57].

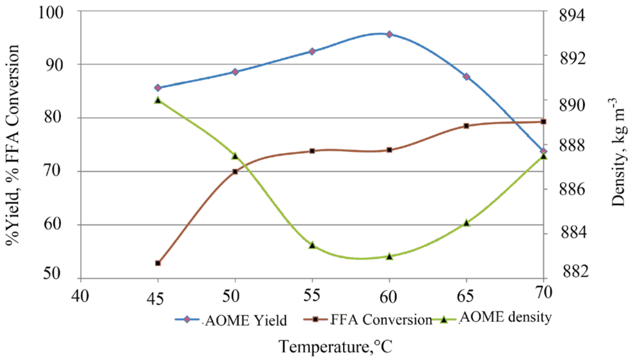


Fig. 6 Effect of temperature on AOME yield and FFA conversion at 9:1 methanol-to-oil molar ratio, 1 wt% catalyst and 3 min reaction time

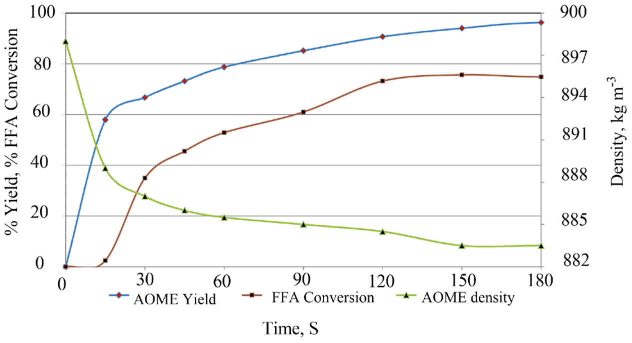


Fig. 7 Effect of time on AOME yield and FFA conversion at 9:1 methanol-to-oil molar ratio, 1 wt% catalyst and 60 °C

Subramaniam et al. [58] in the transesterification of olive oil concluded that microwave heating caused a high collision rate with reduced reaction time of 1 min for 98.2% biodiesel production. Also, with the generation of intermediate diglycerides and monoglycerides, the initial heterogeneous oil-methanol system converted into a homogeneous emulsification system, so the reaction rate increased [30]. However, the further rise in time beyond the optimum time decreases the yield mainly due to degradation of biodiesel into by-products such as monoglycerides [59], hydrolysis of esters into soap [45]. Tarigan et al. [60] concluded that there was no improvement in the yield after equilibrium, but a reduction in effectiveness due to reversible transesterification reaction of oil. El Sherbiny et al. [61] reported that the excess time favored the equilibrium in the reverse direction.

3.2 Kinetic and thermodynamic parameter estimation

3.2.1 Rate constant and order of reaction

The transesterification reaction kinetics is presented by Eq. (4).

$$\frac{-d[\mathrm{Ca}]}{dt} = k\cdot[\mathrm{Ca}]^{n}\cdot[\mathrm{MeOH}]^{m} \tag{4}$$

The k is the rate constant, [Ca] is the concentration of oil, t is the time, n and m are exponents of the oil and methanol concentrations, respectively.

The kinetics of AO transesterification was followed at a constant 9:1 methanol-to-oil molar ratio and 1 wt% catalyst. The methanol was used in excess. Hence, the rate is zero order with respect to methanol concentration. Equation (5) presents the modified rate expression.

$$\frac{-d[\mathrm{Ca}]}{dt} = k\cdot[\mathrm{Ca}]^{n} \tag{5}$$

The initial concentration of AO was 0.000737 mol mL^{-1}. The AO is converted into AOME with the progress of the reaction. The yield of AOME obtained in each experimental set is used to determine the AOME concentration. Fig. 8 presents the concentration of AOME with time at 50 °C, 55 °C, 60 °C, 65 °C and 70 °C.

The AOME concentration gradually increased with reaction time, while the AO concentration decreased. The differential rate expression is used to determine the rate constant and order of reaction (Tables S2–S6 in the Supplement).

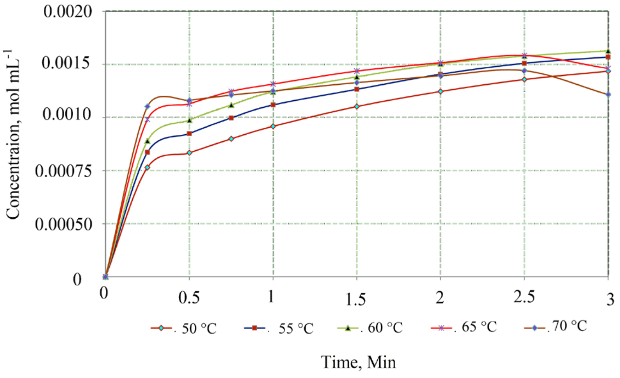


Fig. 8 AOME concentration with time at different temperatures

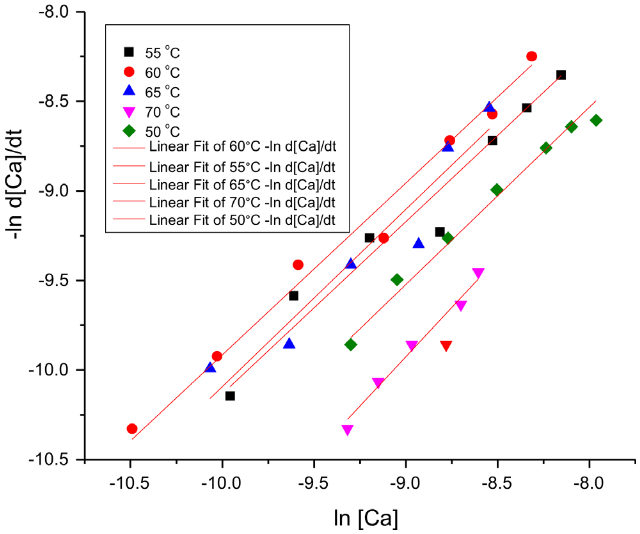


Fig. 9 Plot of $-\ln d[\mathrm{Ca}]/dt$ *versus* ln[Ca] for 50 °C, 55 °C, 60 °C, 65 °C and 70 °C for determination of rate constant and order of reaction at 9:1 methanol-to-oil molar ratio, 1 wt% catalyst amount

Fig. 9 shows the differential plot of $-\ln d[\mathrm{Ca}]/[dt]$ *versus* ln[Ca] for 50 °C, 55 °C, 60 °C, 65 °C and 70 °C. The plot of $-\ln d[\mathrm{Ca}]/dt$ *versus* ln[Ca] for different temperature ranges is best fitted by a straight line. The value of the coefficient of determination R^2 above 0.95 indicates good agreement with experimental data. The order of reaction and the rate constant are determined using the slope and the intercept on the *Y*-axis. The ANOVA of the linear fit with a high *F*-value and low *P*-value suggests close agreement with the experimental data [62]. *F*-value definition: *F* is a calculated test statistic that represents the ratio of explained variance (signal) to unexplained variance (noise). A higher *F*-value indicates stronger evidence that group means are not equal. If the null hypothesis is true, the *F*-ratio should be close to 1. *P*-value means the probability (ranging from 0 to 1) that measures how likely it is to observe the calculated *F*-value (or more extreme) by chance.

The finding suggests that microwave-assisted transesterification of *Argemone mexicana* oil followed pseudo-first-order kinetics and the rate of the reaction varies linearly with a yield of methyl esters. The order of reaction and rate constants are summarized in Table 3.

Table 3 Statistical analysis of the rate constant and the order of reaction

Temp., °C	Order, x	ln$k$	k, min^{-1}	SS	R^2	R^2_{adj}	F-value	P-value
50	0.98	−0.61	0.541	579.5	0.99	0.99	159973	1.6431E−14
55	0.96	−0.5	0.606	584.4	0.99	0.99	35410	1.5195E−12
60	0.96	−0.32	0.726	597.0	0.99	0.99	60486	3.049E−13
65	0.98	−0.19	0.823	521.5	0.99	0.99	18484	4.0834E−10
70	1.10	−0.02	0.983	487.3	0.99	0.99	254302	9.2776E−11

SS: sum of squares, R^2_{adj}: R^2 adjusted, Adjusted R^2 is modified R^2 which takes in account the number of the variables and the number of the observations. R^2_{adj} increases only when new variables truely improves the model. $R^2_{adj} = 1 - ((1 - R^2)(x - 1)/(x - p - 1))$ where x = number of observations, p = number of independent variables. R^2 always increases when more variables are added. Adjusted R^2 increases only if the new variable truly improves the model, by considering the number of predictors in the model. It penalizes unnecessary variables.

3.2.2 Activation energy determination

The activation energy is the minimum energy required for the reaction to proceed. The Arrhenius equation is used to find the activation energy.

$$k = A \cdot e^{-E_a/\mathrm{R}T} \tag{6}$$

The k is the rate constant and A is the Arrhenius parameter, which stands for frequency factor or pre-exponential coefficient (min^{-1}) and E_a is the activation energy (J mol^{-1}). R is the molar universal gas constant (8.314 J mol^{-1} K^{-1}) and T is the absolute temperature (K).

Linearization of the Arrhenius equation is expressed by Eq. (7).

$$\ln k = \ln A - E_a/\mathrm{R}T \tag{7}$$

The scattered plot of rate constant k *versus* temperature T, in Fig. 10, suggests a linear fit, and it follows first-order kinetics.

The plot of ln$k$ *versus* $1/T$ (Fig. 11) gives the activation energy and frequency factor. The activation energy and frequency factor were found to be 27.49 kJ mol^{-1} and 14871 min^{-1}, respectively.

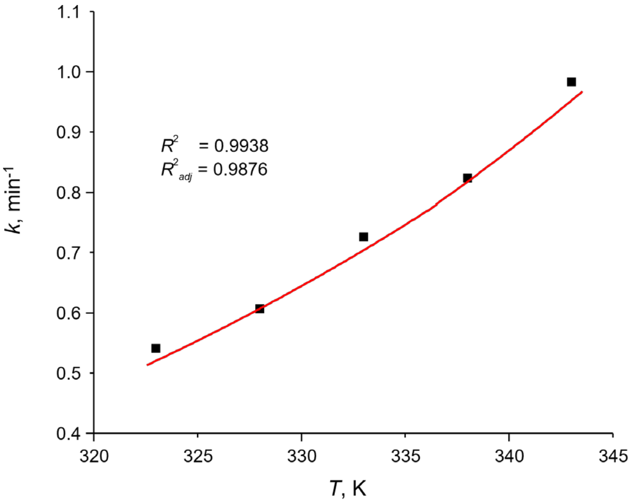


Fig. 10 Plot of rate constant *versus* temperature (see footnote of Table 3 for R^2_{adj})

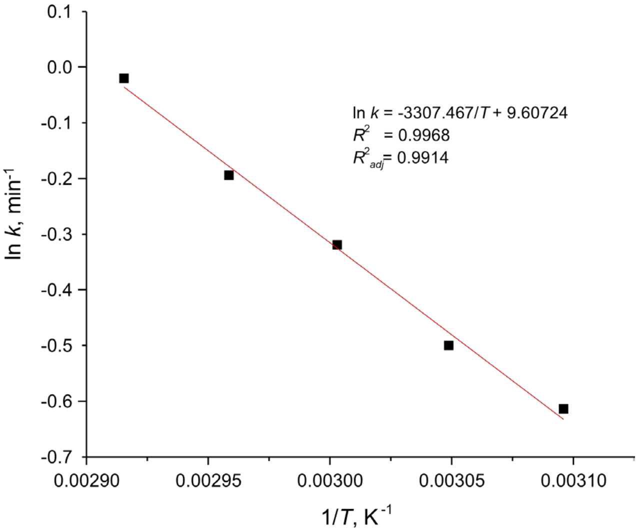


Fig. 11 Plot of rate constant *versus* temperature (see footnote of Table 3 for R^2_{adj})

The nature of oil, such as the degree of unsaturation, also affects the activation energy required for transesterification. Oil with higher unsaturation lowers the reaction rate, hence it has higher activation energy. Types of catalyst, temperature and the source of heating also affect the activation energy. Barekati-Goudarzi et al. [63] reported that the non-thermal effect of a microwave formed the active intermediate complex with a lower state of energy. Also, the higher value of the pre-exponential factor in the microwave-assisted transesterification of AO enhanced the probability of molecular collision due to the dipolar rotation and ionic movement [64].

3.2.3 Estimation of thermodynamic parameters

The Eyring–Polanyi equation presented in Eq. (8) relates the Gibbs free energy to the rate constant:

$$k = \frac{k_b T}{h} \cdot \exp\left(\frac{-\Delta G}{\mathrm{R}T}\right), \tag{8}$$

where k_b is the Boltzmann constant ($1.38 \cdot 10^{-23}$ J K^{-1}) and h is the Planck constant ($6.63 \cdot 10^{-34}$ J s)

Taking the natural logarithm of Eq. (8) and substituting $\Delta G = \Delta H - T\Delta S$, where ΔH and ΔS are the enthalpy and entropy of activation, respectively. Equation (9) presents the linear form of Eq. (8).

$$\ln \frac{k}{T} = \frac{-\Delta H}{\mathrm{R}T} + \frac{\Delta S}{\mathrm{R}} + \ln \frac{k_b}{h} \tag{9}$$

Fig. 12 presents the plot of $\ln \frac{k}{T}$ *versus* $\frac{1}{T}$ for microwave-assisted transesterification of AO. The slope $\frac{-\Delta H}{\mathrm{R}}$ and intercept $\frac{\Delta S}{\mathrm{R}} + \ln \frac{k_b}{h}$ of the linear fit are used to calculate thermodynamic properties. The values of ΔH and ΔS were found to be 24.73 kJ mol^{-1} and -0.2083 kJ mol^{-1} K^{-1}, respectively. Using the values of ΔH and ΔS, at 333 K, ΔG is found to be 94.09 kJ mol^{-1}. A positive value of ΔH shows that heat input is required to bring the reactants to the transition state and to form the products.

A negative value of ΔS indicates that the degree of ordered geometry/alignment of the transition state is better than compared of the reactants in the ground state. A positive value of ΔG and ΔH indicates a non-spontaneous and endergonic reaction. Table 4 presents the summary of kinetic and thermodynamic properties of the present work with reported work in the literature [2, 27, 63, 65–70]. The kinetic and thermodynamic properties in the microwave-assisted transesterification of AO differ from soybean oil [65], rapeseed oil [66] and *Schleichera trijuga* oil [2]. The variation in kinetic and thermodynamic parameters is due to the difference in the quality of oil and the catalyst system adopted for biodiesel production.

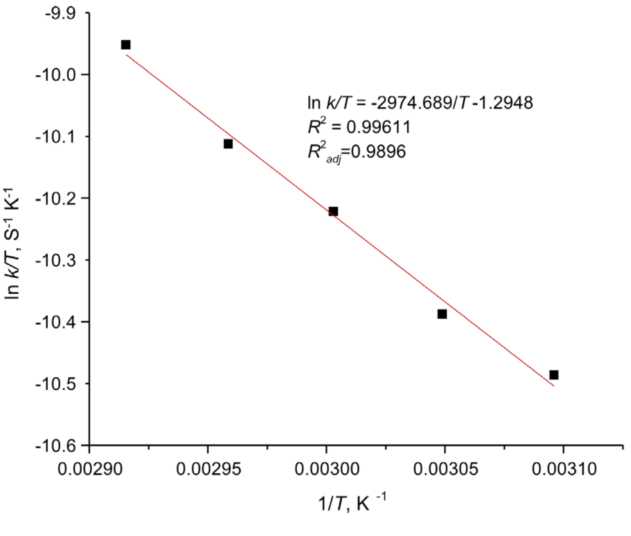


Fig. 12 Estimation of the thermodynamic properties (see footnote of Table 3 for R^2_{adj})

3.3 Analysis of *Argemone mexicana* oil methyl ester

3.3.1 Physicochemical properties

Physicochemical properties of microwave-assisted AOME, such as specific gravity, flash point, viscosity, cloud point, free fatty acid content, heating value and cetane number were determined and summarized in Table 5. The properties were compared with the ASTM standard, and the results show that all the compared properties of the produced biodiesel are close to ASTM D6751-12 [71].

3.3.2 Fourier transform indfrared spectroscopy analysis of microwave-assisted *Argemone mexicana* oil methyl ester

Fig. 13 presents the Fourier transform infrared (FTIR) spectra of AO and AOME. IR spectra were recorded on Bruker Alpha-E FTIR (Zn-Se optics) with an ATR module at 4 cm^{-1} resolution. Peaks observed at $V_{\max}$ = 2853 cm^{-1} (where $V_{\max}$ is the wavenumber of the maximum absorption peak) in AO and AOME show the symmetrical stretching vibration of CH_2–, CH_3– in R_1–C(OR)=O, where R_1 represents long chains of hydrocarbons. All groups present in CH_2–O reduced to CH_3–O due to methanolysis gave a new signal at 1436 cm^{-1} is an indication of transesterification. The next visible transformation in the ester control signal area is approximately at 1200 cm^{-1}. The strong, broad signal at 1162 cm^{-1} in *Argemone mexicana* oil FTIR spectrum separates into two distinct signals at 1170 and 1196 cm^{-1} in AOME. It represents the averaging of the energy over the triple ester group of the triglycerides is gone [72].

3.3.3 ^{1}H nuclear magnetic resonance analysis

The structure and chemical properties of biodiesel were determined by nuclear magnetis resonance (NMR) spectroscopy. FT-NMR-Bruker, Proton NMR (500 MHz) was recorded in $CDCl_3$ solvent. Fig. 14 presents the ^{1}H-NMR spectra of microwave assisted AOME. A strong singlet at δ = 3.66 ppm indicates the protons of the methyl group of *Argemone mexicana* biodiesel, which is absent in the oil. Another multiplet resonance observed at δ = 0.86–0.88 ppm indicates terminal methyl protons (C–CH_3). Multiplet resonance detected at δ = 5.3 ppm signifies the proton attached to the olefinic carbon (one double bond).

Table 4 Summary of comparison of kinetic parameters (order of reaction, rate constant, pre-exponential factor and activation energy) and thermodynamic properties of AOME (ΔG, ΔH and ΔS) according to literature

Triglyceride	Heating source	Reaction conditions	n	k, min^{-1}	A, min^{-1}	E_a, kJ mol^{-1}	ΔG, kJ mol^{-1}	ΔH, kJ mol^{-1}	ΔS, $\text{kJ mol}^{-1}\ \text{K}^{-1}$	Reference
Argemone mexicana oil 1.23 AV Un: 83%	M, Batch	NaOH 1 wt%, 9:1 MeOH:oil (molar), 60 °C	1	0.7267	14871	27.49	94.09	24.73	−0.208	Present work
Chinese tallow seed	M, Continuous	NaOH 4 wt%, 47.5:39.3:20 MeOH:hexane:oil w/w, 60 °C	1	0.0846	5.5	20.57	85.57	−0.57	−0.267	[63]
Soybean oil 0.5 AV Un: 84.5%	C	NaOH 0.77 wt%, 7.47:1 MeOH:oil, 1.76% bentonite, 57.8 °C	1	0.1648	12456	31.03	83.30–87.69	28.33	−0.18	[65]
Rapeseed oil 2.29 AV Un: 94.9%	C	KOH 0.7 wt%, 9:1 MeOH:oil (molar) 60 °C	1	0.1689	461.22	21.88	75.26–79.06	19.59	−0.19	[66]
Schleichera trijuga oil 0.84 AV	U	$Ba(OH)_2$ 3 wt%, 9:1 MeOH:oil (molar)	2	0.2678	$6.1 \cdot 10^7\ \text{L mol}^{-1}\ \text{min}^{-1}$	53.26	82.44–85.55	50.62	−0.11	[67]
Spirulina platensis algae biomass Un: 41%	C	H_2SO_4 catalyst: 60 wt% concentration, Biomass to Methanol ratio wt:vol:1:4, 55 °C, 90 min	1	0.001	2.21	14.51	92.71	16.35	−0.23	[2]
Leather tanning waste Un: 71%	S	Non catalytic 250–325 °C, 12 MPa, 40:1 MeOH:oil molar ratio	1	0.32–0.96	1176	36.01	153.64–171.16	31.37	−0.233	[68]
Waste cooking oil	M	150 °C, 2 h, 28:1 molar ratio of MeOH:oil, 10 wt% deep eutectic solvent	1	–	610001	51.24	66.8–70.1	47.3	−0.165	[69]
Kusum oil	M	65 °C, 13 min, silicotungstic acid	1	0.224	219000	38.82	98.89	36.06	−0.185	[27]
Waste cooking oil	M	115 °C, 2 h, 24:1 molar ratio of methanol to oil MeOH:oil, 10% catalyst	1	0.026	–	13.67	99.7–109.8	10.6	−0.252	[70]

Un = Un-saturation, M = microwave, C = Conventional, U = Ultrasound, S = supercritical, n = order of reaction, k = rate constant, A = pre-exponential factor, E_a = activation energy, AV = acid value

Table 5 Physicochemical properties and characteristics of *Argemone mexicana* oil methyl ester

Properties (unit)	AOME	ASTM D6751-12 [71]
Specific gravity	0.88	0.86–0.9
Flash point (°C)	138	>130
Viscosity at 40 °C (mm^2 s^{-1})	3.12	1.9–6
Molecular mass (g/mol)	276	–
Cloud point (°C)	4	5
Free fatty acids %	<0.40	<1.60
Acid number (mg KOH g^{-1})	<0.20	<0.80
Heating value (MJ kg^{-1})	39.74	–
Cetane number	53.2	47

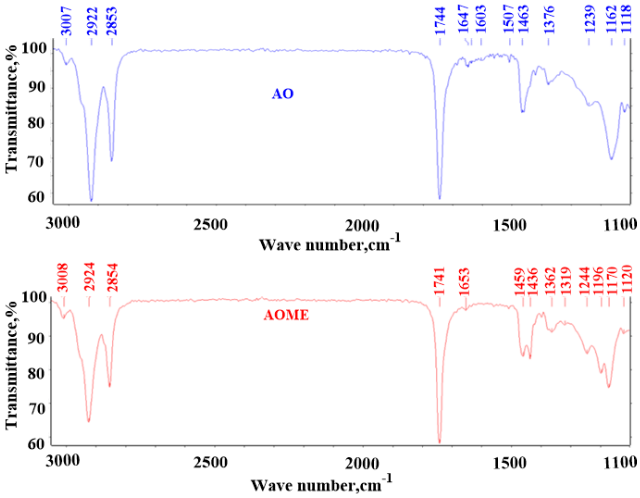


Fig. 13 FTIR of (a) *Argemone mexicana* oil and (b) microwave-assisted AOME at 9:1 methanol-to-oil molar ratio, 1 wt% NaOH, 60 °C, and 3 min

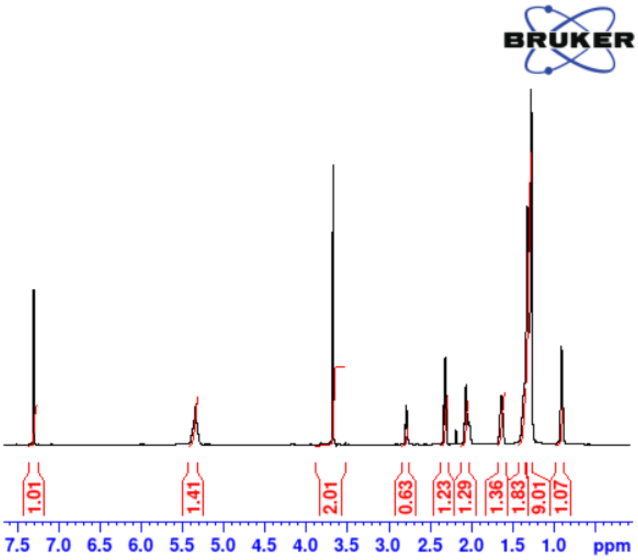


Fig. 14 ^{1}H-NMR of AOME sample at 9:1 methanol-to-oil molar ratio, 1 wt% NaOH, 60 °C and 3 min

The multiplet at $\delta = 1.61$ ppm indicates β-methylene protons to ester bond (CH_2–C–COOMe), and a strong resonance at $\delta = 1.30$ ppm is due to the protons of backbone methylene protons of the aliphatic fatty acid chain. The absence the peaks for glyceride protons at $\delta = 4.2–4.3$ ppm and the presence of methyl resonance at $\delta = 3.66$ ppm confirmed the conversion of oil into biodiesel. Using ^{1}H-NMR, the protons of the methylene group adjacent to the ester moiety in the triglycerides and the protons in the alcohol moiety of the product methyl esters are used to monitor the yield by Eq. (10).

$$C = 100 \cdot \left(\frac{2 \cdot A_{\mathrm{ME}}}{3 \cdot A\alpha\text{-CH}_2} \right) = 100 \cdot \left(\frac{2 \cdot 2.0058}{3 \cdot 1.4089} \right) = 94.91\% \quad (10)$$

The C is the conversion of AO to the AOME, A_{ME} is the integration value of the protons of the methyl esters (the strong singlet peak), $A\alpha$-CH_2 is the integration value of the methylene protons.

The factors 2 and 3 are used to indicate methylene carbon possesses two protons and the alcohol (methanol-derived) carbon has three attached protons.

4 Conclusion

Microwave-assisted transesterification of *Argemone mexicana* oil was carried out to find the effect of temperature, catalyst concentration, methanol-to-oil molar ratio and time on the yield of biodiesel. Temperature up to 60 °C increases the molecular collision frequency and fluidity of the mixture, resulting in an improvement of the AOME yield. Beyond 60 °C, methanol vaporization and the reduction in the effective concentration reduce the yield. The molar ratio of methanol-to-oil up to 9:1 improves AOME yield due to a shift of equilibrium to the product. However, above molar ratio 9:1 the dilution of the reactants and the catalyst as well as an increase in the solubility of glycerol in the oil phase affect the yield. NaOH catalyst up to 1% improves the yield as more active sites are available for the transesterification of oil. However, above 1 wt% NaOH side reactions increase the solution viscosity and the mass transfer resistance reduce the yield. Microwave irradiation enables rapid heating and efficient energy transfer, achieving high AOME yield in short reaction time. The result suggested that in each effect, biodiesel yield passes through maxima. 96% AOME yield was obtained at 3-min reaction time, 1% catalyst concentration, 9:1 methanol-to-oil ratio and 60 °C reaction temperature. The FTIR spectra of biodiesel confirmed the formation of biodiesel. NMR study confirms that 94.91% conversion of *Argemone mexicana* oil into biodiesel takes place in the above conditions. The physicochemical properties confirmed the feasibility of *Argemone mexicana* oil as one of the feedstocks for biodiesel production, with a promising potential to be used as an alternative to conventional diesel. The rate constant, frequency factor and the activation energy for microwave-assisted transesterification of AO were found to be 0.7267 min^{-1} (60 °C), 14871 min^{-1} and 27.49 kJ mol^{-1} respectively. A higher value of pre-exponential confirmed the presence of the non-thermal effect of the microwave. The values of thermodynamic parameters ΔG, ΔH and ΔS at 60 °C were found to be 94.09 kJ mol^{-1}, 24.73 kJ mol^{-1} and −0.2083 kJ mol^{-1} K^{-1}, respectively, which show that the reaction is non-spontaneous and endergonic in nature.

Acknowledgement

We confirm that this research work is not funded by any organization.

References

[1] Nayak, M. G. "Review and comparison of the methodology adopted for biodiesel production", Carbon Resources Conversion, 9(1), 100343, 2026. https://doi.org/10.1016/j.crcon.2025.100343

[2] Nautiyal, P., Subramanian, K. A., Dastidar, M. G. "Kinetic and thermodynamic studies on biodiesel production from *Spirulina platensis* algae biomass using single stage extraction–transesterification process", Fuel, 135, pp. 228–234, 2014. https://doi.org/10.1016/j.fuel.2014.06.063

[3] Salim, S. M., Izriq, R., Almaky, M. M., Al-Abbassi, A. A. "Synthesis and characterization of ZnO nanoparticles for the production of biodiesel by transesterification: Kinetic and thermodynamic studies", Fuel, 321, 124135, 2022. https://doi.org/10.1016/j.fuel.2022.124135

[4] Rokni, K., Mostafaei, M., Soufi, M. D., Kahrizi, D. "Microwave-assisted intensification of transesterification reaction for biodiesel production from camelina oil: Optimization by Box-Behnken Design", Bioresource Technology Reports, 17, 100928, 2022. https://doi.org/10.1016/j.biteb.2021.100928

[5] Nayak, M. G., Gamit, R. D. "Parametric study and optimisation of supercritical extraction of Chlorella Vulgaris microalgae using Response surface methodology", Chemical Physics Impact, 11, 100923, 2025. https://doi.org/10.1016/j.chphi.2025.100923

[6] Nayak, S. N., Nayak, M. G., Bhasin, C. P. "Microwave-mediated conversion of Kusum seed oil into biodiesel and optimization using RSM-central composite design", Results in Chemistry, 20, 102970, 2026. https://doi.org/10.1016/j.rechem.2025.102970

[7] Sahani, S., Roy, T., Chandra Sharma, Y. "Clean and efficient production of biodiesel using barium cerate as a heterogeneous catalyst for the biodiesel production; kinetics and thermodynamic study", Journal of Cleaner Production, 237, 117699, 2019. https://doi.org/10.1016/j.jclepro.2019.117699

[8] Daud, N. M., Sheikh Abdullah, S. R., Abu Hasan, H., Yaakob, Z. "Production of biodiesel and its wastewater treatment technologies: A review", Process Safety and Environmental Protection, 94, pp. 487–508, 2015. https://doi.org/10.1016/j.psep.2014.10.009

[9] Lin, J.-J., Chen, Y.-W. "Production of biodiesel by transesterification of Jatropha oil with microwave heating", Journal of the Taiwan Institute of Chemical Engineers, 75, pp. 43–50, 2017. https://doi.org/10.1016/j.jtice.2017.03.034

[10] Tomei, J., Helliwell, R. "Food versus fuel? Going beyond biofuels", Land Use Policy, 56, pp. 320–326, 2016. https://doi.org/10.1016/j.landusepol.2015.11.015

[11] Ambat, I., Srivastava, V., Sillanpää, M. "Recent advancement in biodiesel production methodologies using various feedstock: A review", Renewable and Sustainable Energy Reviews, 90, pp. 356–369, 2018. https://doi.org/10.1016/j.rser.2018.03.069

[12] Gebremariam, S. N., Marchetti, J. M. "Economics of biodiesel production: Review", Energy Conversion and Management, 168, pp. 74–84, 2018. https://doi.org/10.1016/j.enconman.2018.05.002

[13] Lusweti, A., Wabuyele, E., Ssegawa, P., Mauremootoo, J. "Argemone mexicana (Mexican Prickly Poppy)", [online] Available at: https://keys.lucidcentral.org/keys/v3/eafrinet/weeds/key/weeds/Media/Html/Argemone_mexicana_(Mexican_Prickly_Poppy).htm [Accessed: 17 July 2018]

[14] CABI International "Argemone mexicana (Mexican poppy)", 2022. https://doi.org/10.1079/pwkb.species.6878

[15] Verma, S. K., Dev, G., Tyagi, A. K., Goomber, S., Jain, G. V. "*Argemone mexicana* poisoning: autopsy findings of two cases", Forensic Science International, 115(1–2), pp. 135–141, 2001. https://doi.org/10.1016/S0379-0738(00)00322-4

[16] Husain, S., Narsimha, R., Rao, R. N. "Separation, identification and determination of sanguinarine in argemone and other adulterated edible oils by reversed-phase high-performance liquid chromatography", Journal of Chromatography A, 863(1), pp. 123–126, 1999. https://doi.org/10.1016/S0021-9673(99)00964-4

[17] Bosch, C. H. "*Argemone mexicana* L.", In: Schmelzer, G. H., Gurib-Fakim, A. (eds.) PROTA (Plant Resources of Tropical Africa / Ressources végétales de l'Afrique tropicale), University of Wageningen, Wageningen, Netherlands, 2007. [online] Available at: https://uses.plantnet-project.org/en/Argemone_mexicana_(PROTA) [Accessed: 09 March 2019]

[18] Pramanik, P., Das, P., Kim, P. J. "Preparation of biofuel from argemone seed oil by an alternative cost-effective technique", Fuel, 91(1), pp. 81–86, 2012. https://doi.org/10.1016/j.fuel.2011.07.011

[19] Kumar, S., Singhal, M. K., Sharma, M. P. "Utilization of mixed oils for biodiesel preparation: a review", Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, 47(1), pp. 4811–4844, 2025. https://doi.org/10.1080/15567036.2021.1884771

[20] Oyekunle, D. T., Gendy, E. A., Barasa, M., Oyekunle, D. O., Oni, B., Tiong, S. K. "Review on utilization of rubber seed oil for biodiesel production: Oil extraction, biodiesel conversion, merits, and challenges", Cleaner Engineering and Technology, 21, 100773, 2024. https://doi.org/10.1016/j.clet.2024.100773

[21] Nayak, S. N., Bhasin, C. P., Nayak, M. G. "A review on microwave-assisted transesterification processes using various catalytic and non-catalytic systems", Renewable Energy, 143, pp. 1366–1387, 2019. https://doi.org/10.1016/j.renene.2019.05.056

[22] Lin, Y.-C., Chen, S.-C., Chen, C.-E., Yang, P.-M., Jhang, S.-R. "Rapid Jatropha-biodiesel production assisted by a microwave system and a sodium amide catalyst", Fuel, 135, pp. 435–442, 2014. https://doi.org/10.1016/j.fuel.2014.07.023

[23] Ashine, F., Kiflie, Z., Prabhu, S. V., Tizazu, B. Z., Varadharajan, V., Rajasimman, M., Joo, S.-W., Vasseghian, Y., Jayakumar, M. "Biodiesel production from *Argemone mexicana* oil using chicken eggshell derived CaO catalyst", Fuel, 332, 126166, 2023. https://doi.org/10.1016/j.fuel.2022.126166

[24] Asfaw, M. D., Yadeta, A. T., Yewalie, B. G., Negash, Y. W. "Production and characterization of biodiesel from *Argemone mexicana* seed oil", Results in Chemistry, 13, 102055, 2025. https://doi.org/10.1016/j.rechem.2025.102055

[25] Ong, H. C., Masjuki, H. H., Mahlia, T. M. I., Silitonga, A. S., Chong, W. T., Leong, K. Y. "Optimization of biodiesel production and engine performance from high free fatty acid *Calophyllum inophyllum* oil in CI diesel engine", Energy Conversion and Management, 81, pp. 30–40, 2014. https://doi.org/10.1016/j.enconman.2014.01.065

[26] Alovert, M., Andrew, M. "The 3-27 Conversion Test | Quality Testing", [online] Available at: http://www.make-biodiesel.org/Quality-Testing/the-3-27-conversion-test.html [Accessed: 02 November 2017]

[27] Nayak, S. N., Nayak, M. G., Bhasin, C. P. "Microwave-Assisted Transesterification of Kusum Oil: Parametric, Kinetic and Thermodynamic Studies", Asian Journal of Chemistry, 32(11), pp. 2893–2903, 2020. https://doi.org/10.14233/ajchem.2020.22890

[28] Nayak, M. G., Vyas, A. P. "Optimization of microwave-assisted biodiesel production from Papaya oil using response surface methodology", Renewable Energy, 138, pp. 18–28, 2019. https://doi.org/10.1016/j.renene.2019.01.054

[29] Dai, Y.-M., Wu, J.-S., Chen, C.-C., Chen, K.-T. "Evaluating the optimum operating parameters on transesterification reaction for biodiesel production over a $LiAlO_2$ catalyst", Chemical Engineering Journal, 280, pp. 370–376, 2015. https://doi.org/10.1016/j.cej.2015.06.045

[30] Wu, H., Zhang, J., Wei, Q., Zheng, J., Zhang, J. "Transesterification of soybean oil to biodiesel using zeolite supported CaO as strong base catalysts", Fuel Processing Technology, 109, pp. 13–18, 2013. https://doi.org/10.1016/j.fuproc.2012.09.032

[31] Zabeti, M., Daud, W. M. A. W., Aroua, M. K. "Biodiesel production using alumina-supported calcium oxide: An optimization study", Fuel Processing Technology, 91(2), pp. 243–248, 2010. https://doi.org/10.1016/j.fuproc.2009.10.004

[32] Supeno, M., Sihotang, J. P., Panjaitan, Y. V., Damanik, D. S. Y., Tarigan, J. B., Sitepu, E. K. "Room temperature esterification of high-free fatty acid feedstock into biodiesel", RSC Advances, 13(47), pp. 33107–33113, 2023. https://doi.org/10.1039/D3RA06912E

[33] Yamin, J., Al-Hamamre, Z., Sandouqa, A. "Modelling and optimisation of biodiesel production using waste cooking oil using the response surface methodology", International Journal of Sustainable Energy, 43(1), 2355654, 2024. https://doi.org/10.1080/14786451.2024.2355654

[34] Zhou, H., Lu, H., Liang, B. "Solubility of Multicomponent Systems in the Biodiesel Production by Transesterification of *Jatropha curcas* L. Oil with Methanol", Journal of Chemical & Engineering Data, 51(3), pp. 1130–1135, 2006. https://doi.org/10.1021/je0600294

[35] Sánchez, B. S., Benitez, B., Querini, C. A., Mendow, G. "Transesterification of sunflower oil with ethanol using sodium ethoxide as catalyst. Effect of the reaction conditions", Fuel Processing Technology, 131, pp. 29–35, 2015. https://doi.org/10.1016/j.fuproc.2014.10.043

[36] Ulukardesler, A. H. "Biodiesel Production from Waste Cooking Oil Using Different Types of Catalysts", Processes, 11(7), 2035, 2023. https://doi.org/10.3390/pr11072035

[37] Chauhan, S. J., Devliya, B., Patel, B., Nagapara, J., Mevada, S., Solanki, H., Patel, H. D. "Biodiesel production from waste cooking oil using novel heterogenous CaO-MgO-ZnO-TiO_2 nanoconjugate catalyst (CMZT-Nano Cat): A green approach towards renewable energy", Biomass and Bioenergy, 200, 107985, 2025. https://doi.org/10.1016/j.biombioe.2025.107985

[38] Agarwal, M., Chauhan, G., Chaurasia, S. P., Singh, K. "Study of catalytic behavior of KOH as homogeneous and heterogeneous catalyst for biodiesel production", Journal of the Taiwan Institute of Chemical Engineers, 43(1), pp. 89–94, 2012. https://doi.org/10.1016/j.jtice.2011.06.003

[39] Fadhil, A. B., Ahmed, K. M., Dheyab, M. M. "*Silybum marianum* L. seed oil: A novel feedstock for biodiesel production", Arabian Journal of Chemistry, 10(1_suppl), pp. 683–690, 2017. https://doi.org/10.1016/j.arabjc.2012.11.009

[40] Kafuku, G., Mbarawa, M. "Biodiesel production from *Croton megalocarpus* oil and its process optimization", Fuel, 89(9), pp. 2556–2560, 2010. https://doi.org/10.1016/j.fuel.2010.03.039

[41] Thliveros, P., Uçkun Kiran, E., Webb, C. "Microbial biodiesel production by direct methanolysis of oleaginous biomass", Bioresource Technology, 157, pp. 181–187, 2014. https://doi.org/10.1016/j.biortech.2014.01.111

[42] Likozar, B., Levec, J. "Transesterification of canola, palm, peanut, soybean and sunflower oil with methanol, ethanol, isopropanol, butanol and tert-butanol to biodiesel: Modelling of chemical equilibrium, reaction kinetics and mass transfer based on fatty acid composition", Applied Energy, 123, pp. 108–120, 2014. https://doi.org/10.1016/j.apenergy.2014.02.046

[43] Buchori, L., Widayat, W., Muraza, O., Amali, M. I., Maulida, R. W., Prameswari, J. "Effect of Temperature and Concentration of Zeolite Catalysts from Geothermal Solid Waste in Biodiesel Production from Used Cooking Oil by Esterification–Transesterification Process", Processes, 8(12), 1629, 2020. https://doi.org/10.3390/pr8121629

[44] Alhassan, Y., Kumar, N., Bugaje, I. M., Pali, H. S., Kathkar, P. "Co-solvents transesterification of cotton seed oil into biodiesel: Effects of reaction conditions on quality of fatty acids methyl esters", Energy Conversion and Management, 84, pp. 640–648, 2014. https://doi.org/10.1016/j.enconman.2014.04.080

[45] Priambodo, R., Chen, T.-C., Lu, M.-C., Gedanken, A., Liao, J.-D., Huang, Y.-H. "Novel Technology for Bio-diesel Production from Cooking and Waste Cooking Oil by Microwave Irradiation", Energy Procedia, 75, pp. 84–91, 2015. https://doi.org/10.1016/j.egypro.2015.07.143

[46] Miyuranga, K. A. V., Arachchige, U. S. P. R., Marso, T. M. M., Samarakoon, G. "Biodiesel Production through the Transesterification of Waste Cooking Oil over Typical Heterogeneous Base or Acid Catalysts", Catalysts, 13(3), 546, 2023. https://doi.org/10.3390/catal13030546

[47] Berchmans, H. J., Hirata, S. "Biodiesel production from crude *Jatropha curcas* L. seed oil with a high content of free fatty acids", Bioresource Technology, 99(6), pp. 1716–1721, 2008. https://doi.org/10.1016/j.biortech.2007.03.051

[48] Cai, Z.-Z., Wang, Y., Teng, Y.-L., Chong, K.-M., Wang, J.-W., Zhang, J.-W., Yang, D.-P. "A two-step biodiesel production process from waste cooking oil via recycling crude glycerol esterification catalyzed by alkali catalyst", Fuel Processing Technology, 137, pp. 186–193, 2015. https://doi.org/10.1016/j.fuproc.2015.04.017

[49] Encinar, J. M., González, J. F., Rodríguez, J. J., Tejedor, A. "Biodiesel Fuels from Vegetable Oils: Transesterification of *Cynara cardunculus* L. Oils with Ethanol", Energy & Fuels, 16(2), pp. 443–450, 2002. https://doi.org/10.1021/ef010174h

[50] Liu, W., Yin, P., Liu, X., Chen, W., Chen, H., Liu, C., Qu, R., Xu, Q. "Microwave assisted esterification of free fatty acid over a heterogeneous catalyst for biodiesel production", Energy Conversion and Management, 76, pp. 1009–1014, 2013. https://doi.org/10.1016/j.enconman.2013.08.051

[51] Singh, D., Sharma, P. K., Pawar, A., Godara, S. S., Sharma, D., Goyal, R. "Parametric analysis of biodiesel synthesis from palm oil using homogenous base catalyst: experimental and numerical investigation", Scientific Reports, 15(1), 41781, 2025. https://doi.org/10.1038/s41598-025-25746-2

[52] Ulakpa, W. C., Ulakpa, R. O. E., Egwunyenga, M. C., Egbosiuba, T. C. "Transesterification of non-edible oil and effects of process parameters on biodiesel yield", Cleaner Waste Systems, 3, 100047, 2022. https://doi.org/10.1016/j.clwas.2022.100047

[53] Rachmaditasari, R., Darojat, M. I., Mahfud, M. "Production of biodiesel (isopropyl ester) from coconut oil by microwave assisted transesterification: parametric study and optimization", International Journal of Renewable Energy Development, 13(4), pp. 662–672, 2024. https://doi.org/10.61435/ijred.2024.60174

[54] Aseibichin, C., Ulakpa, W. C., Omenogor, I., Doyah, E., Olaseinde, A. A., Anakpoha, O. C., Keke, M., Karuppannan, S. "Modeling and optimization of transesterification of Jatropha oil to fatty acid methyl ester: application of response surface methodology (CCD) and Taguchi orthogonal method", RSC Advances, 14(17), pp. 11784–11796, 2024. https://doi.org/10.1039/D4RA01149J

[55] Amani, H., Ahmad, Z., Asif, M., Hameed, B. H. "Transesterification of waste cooking palm oil by MnZr with supported alumina as a potential heterogeneous catalyst", Journal of Industrial and Engineering Chemistry, 20(6), pp. 4437–4442, 2014. https://doi.org/10.1016/j.jiec.2014.02.012

[56] Likozar, B., Levec, J. "Effect of process conditions on equilibrium, reaction kinetics and mass transfer for triglyceride transesterification to biodiesel: Experimental and modeling based on fatty acid composition", Fuel Processing Technology, 122, pp. 30–41, 2014. https://doi.org/10.1016/j.fuproc.2014.01.017

[57] Silva, G. F., Camargo, F. L., Ferreira, A. L. O. "Application of response surface methodology for optimization of biodiesel production by transesterification of soybean oil with ethanol", Fuel Processing Technology, 92(3), pp. 407–413, 2011. https://doi.org/10.1016/j.fuproc.2010.10.002

[58] Subramaniam, K., Wong, K. Y., Wong, K. H., Chong, C. T., Chiong, M. C., Ng, J.-H. "A synergic approach to intensify biodiesel transesterification through microfluidics and microwave irradiation", Energy, 313, 134056, 2024. https://doi.org/10.1016/j.energy.2024.134056

[59] Chee Loong, T., Idris, A. "Rapid alkali catalyzed transesterification of microalgae lipids to biodiesel using simultaneous cooling and microwave heating and its optimization", Bioresource Technology, 174, pp. 311–315, 2014. https://doi.org/10.1016/j.biortech.2014.10.015

[60] Tarigan, J. B., Barus, A. F., Simamora, N. T., Tarigan, R. S., Perangin-angin, S., Ginting, J., Sitepu, E. K., Taufiq-Yap, Y. H. "Microwave-intensified esterification of high-free fatty acid feedstock into biodiesel using waste chicken eggshells as a heterogeneous catalyst", Case Studies in Chemical and Environmental Engineering, 11, 101107, 2025. https://doi.org/10.1016/j.cscee.2025.101107

[61] El Sherbiny, S. A., Refaat, A. A., El Sheltawy, S. T. "Production of biodiesel using the microwave technique", Journal of Advanced Research, 1(4), pp. 309–314, 2010. https://doi.org/10.1016/j.jare.2010.07.003

[62] Nayak, M. G., Vyas, A. P. "Kinetic and Thermodynamic Studies of Microwave-Assisted Transesterification of Papaya Oil", Asian Journal of Chemistry, 31(8), pp. 1688–1692, 2019. https://doi.org/10.14233/ajchem.2019.21943

[63] Barekati-Goudarzi, M., Muley, P. D., Clarens, A., Nde, D. B., Boldor, D. "Continuous microwave-assisted *in-situ* transesterification of lipids in seeds of invasive Chinese tallow trees (*Triadica sebifera* L.): Kinetic and thermodynamic studies", Biomass and Bioenergy, 107, pp. 353–360, 2017. https://doi.org/10.1016/j.biombioe.2017.09.006

[64] Lewis, D. A., Summers, J. D., Ward, T. C., McGrath, J. E. "Accelerated imidization reactions using microwave radiation", Journal of Polymer Science Part A: Polymer Chemistry, 30(8), pp. 1647–1653, 1992. https://doi.org/10.1002/pola.1992.080300817

[65] Wu, L., Wei, T., Lin, Z., Zou, Y., Tong, Z., Sun, J. "Bentonite-enhanced biodiesel production by NaOH-catalyzed transesterification: Process optimization and kinetics and thermodynamic analysis", Fuel, 182, pp. 920–927, 2016. https://doi.org/10.1016/j.fuel.2016.05.065

[66] Encinar, J. M., Pardal, A., Sánchez, N. "An improvement to the transesterification process by the use of co-solvents to produce biodiesel", Fuel, 166, pp. 51–58, 2016. https://doi.org/10.1016/j.fuel.2015.10.110

[67] Sarve, A. N., Varma, M. N., Sonawane, S. S. "Ultrasound assisted two-stage biodiesel synthesis from non-edible *Schleichera triguga* oil using heterogeneous catalyst: Kinetics and thermodynamic analysis", Ultrasonics Sonochemistry, 29, pp. 288–298, 2016. https://doi.org/10.1016/j.ultsonch.2015.09.016

[68] Ong, L. K., Kurniawan, A., Suwandi, A. C., Lin, C. X., Zhao, X. S., Ismadji, S. "Transesterification of leather tanning waste to biodiesel at supercritical condition: Kinetics and thermodynamics studies", The Journal of Supercritical Fluids, 75, pp. 11–20, 2013. https://doi.org/10.1016/j.supflu.2012.12.018

[69] Demir, E., Çalhan, S. D., Sönmez, Ö. "Microwave-assisted biodiesel production from waste cooking oil using deep eutectic solvent catalyst: Kinetic and thermodynamic insights", Biomass and Bioenergy, 201, 108148, 2025. https://doi.org/10.1016/j.biombioe.2025.108148

[70] Ibrahim, A. S. I., Boyaci, H., Gözmen, B., Sönmez, Ö. "Microwave-assisted biodiesel production from waste cooking oil using pyrrolidonium ionic liquid catalyst: optimization, kinetic and thermodynamic studies", Biomass and Bioenergy, 206, 108630, 2026. https://doi.org/10.1016/j.biombioe.2025.108630

[71] ASTM "ASTM D6751-12 Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels", ASTM International, West Conshohocken, PA, USA, 2012. https://doi.org/10.1520/D6751-12

[72] Ullah, K., Ahmad, M., Sultana, S., Teong, L. K., Sharma, V. K., Abdullah, A. Z., Zafar, M., Ullah, Z. "Experimental analysis of di-functional magnetic oxide catalyst and its performance in the hemp plant biodiesel production", Applied Energy, 113, pp. 660–669, 2014. https://doi.org/10.1016/j.apenergy.2013.08.023