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Ervatamia Coronaria Seed Oil for Biodiesel Production: An Investigative Approach Via Fatty Acid Chemistry

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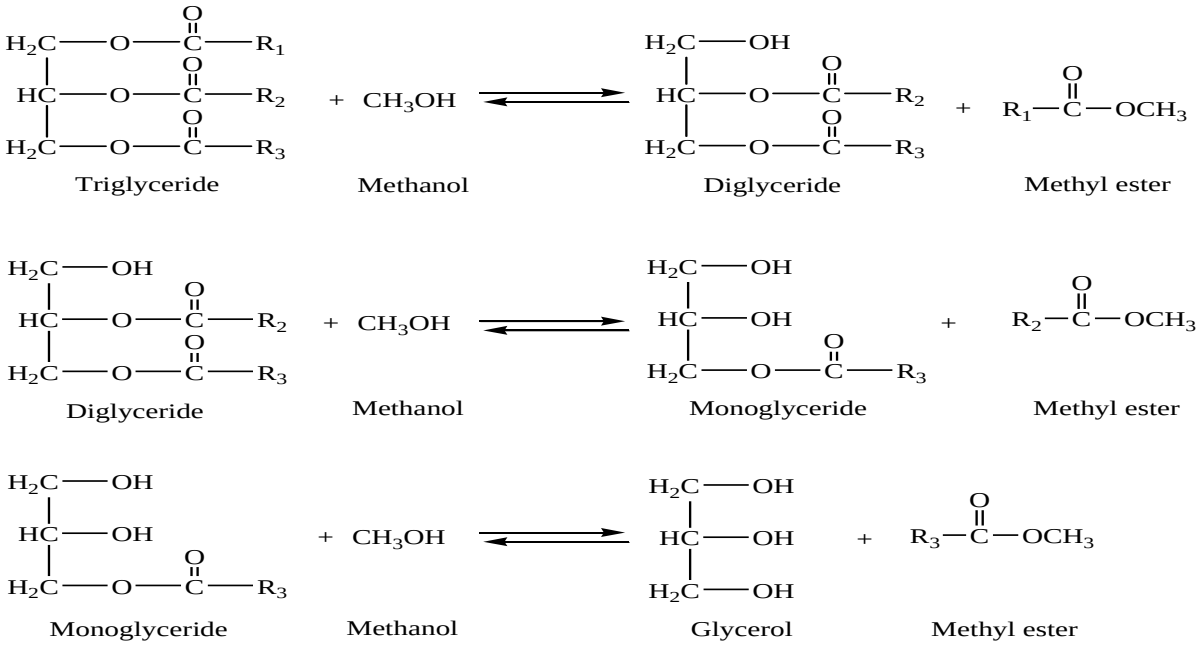
Abstract— The use of non-edible seed oils for biodiesel production is gaining much attention due to the great demand of edible oils in food industries. The present research work deals with the optimization of transesterification of *Ervatamia coronaria* seed oil (ECSO) with varying methanol to oil molar ratio in the range from 4:1 to 8:1, using various alkaline catalysts such as NaOH, KOH, $NaOCH_3$, $KOCH_3$ in the concentration range of 0.50-1.50% by weight, reaction temperature from 50–70°C and stirring rate from 350-800 rpm keeping fixed reaction time of 100 min. The optimum set of transesterification reaction conditions of 0.90% $NaOCH_3$ as catalyst, methanol to oil molar ratio of 6:1, reaction temperature of 65°C and stirring rate of 650 rpm yielded 98.3% fatty acid methyl esters (FAMEs) of *Ervatamia coronaria* seed oil (ECME). The biodiesel properties satisfy ASTM and EN standards.

Keywords- Fatty acid methyl ester, Ervatamia coronaria, non-edible seed oil, biodiesel, transesterification.

1. Introduction

Around the year 1900 the mechanical engineer Rudolf Diesel proposed an idea of using vegetable oil as a substitute for diesel fuel. Vegetable oils have good heating power and the exhaust gases produced during their combustion are almost free from sulphur and aromatic polycyclic compounds. But the challenging point is that, seed oil as diesel fuel has limitation of unfavourably high viscosity [1]. In order to reduce the viscosity of oil and use it without any modification of the diesel engine, different methods are employed. Blending, micro emulsions, pyrolysis and transesterification are the different methods used to reduce the viscosity of oil [2, 3]. Transesterification is the most common method employed for the production of biodiesel. In a transesterification reaction, one mole of triglyceride reacts with three moles of alcohol to form three moles of corresponding fatty acid alkyl esters and one mole of glycerol. The process includes three reversible reactions, in which the triglyceride molecule is converted step by step into diglyceride, monoglyceride and glycerol [4]. The schematic representation and mechanism of biodiesel synthesis through transesterification using methanol is presented in Fig. 1.



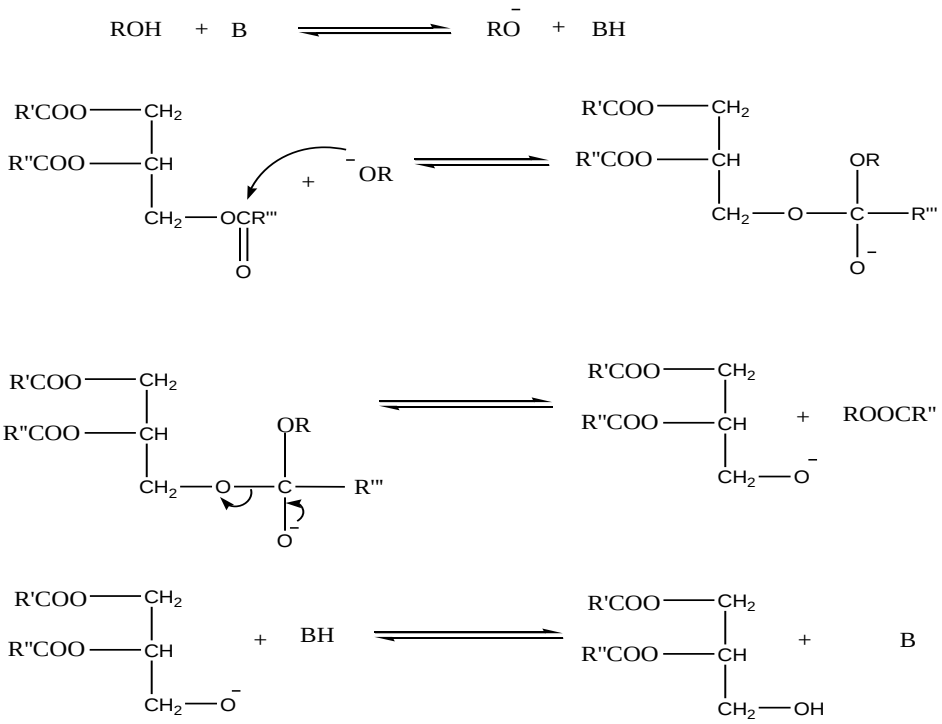
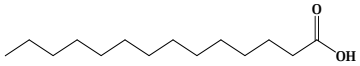
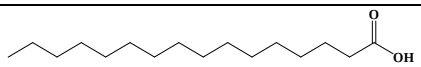
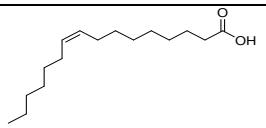
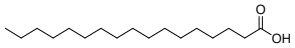


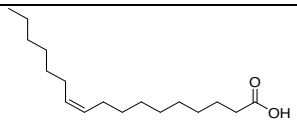
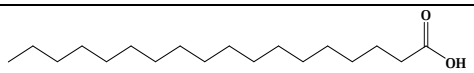
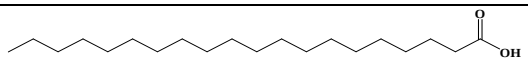
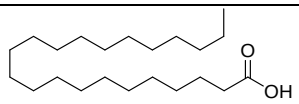
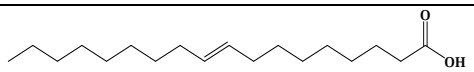
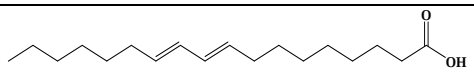
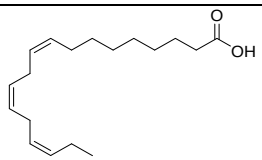
Fig. 1 The schematic representation and mechanism of transesterification

The fossil sources like coal, petroleum and natural gas provide energy required by the world in various segments, but as these sources are limited and are depleting due to modern life style, use of alternative energy resource is a must. Biodiesel appears to be the most promising alternative against the diminishing fossil fuels [5]. Biodiesel fuels have many advantages over petro-diesel. Biodiesel has higher cetane number, produces less smoke and minimum particulates, lower carbon monoxide (COx) and hydrocarbon (HC) emissions, it is non-toxic, biodegradable and renewable [6]. Contrarily, poor cold flow properties and low oxidative stability are technical issues related to biodiesel [7]. Some countries are exploiting edible fatty oils derived from oil seeds like *soybean* seeds*, palm* seeds*, sunflower* seeds*, coconut, linseed* seeds, etc. for biodiesel production wherein the edible seed oil production is excessive than market demand. Fatty acids present in *Ervatamia coronaria* seed oil are represented in Table 1.

TABLE I

MOLECULAR AND STRUCTURAL FORMULA OF FATTY ACIDS PRESENT IN ECSO

Component fatty acids	**N_C:N_{db}***	**Molecular formula**	**Structure**
Myristic acid	14:0	$C_{14}H_{28}O_2$	
Palmitic acid	16:0	$C_{16}H_{32}O_2$	
Palmitoleic acid	16:1	$C_{16}H_{32}O_2$	
Heptadecanoic acid (Margaric acid)	17:0	$C_{17}H_{32}O_2$	

Cis-10-Heptadecanoic acid (Margaric acid)	17:1	$C_{17}H_{32}O_2$	
Stearic acid	18:0	$C_{18}H_{36}O_2$	
Arachidic acid	20:0	$C_{20}H_{40}O_2$	
Behenic acid	22:0	$C_{22}H_{44}O_2$	
Oleic acid	18:1	$C_{18}H_{14}O_2$	
Linoleic acid	18:2	$C_{18}H_{32}O_2$	
Linolenic acid	18:3	$C_{18}H_{32}O_2$	

Note: * N_C and N_{db} are number of carbon atoms and number of C-C double bonds in the FA respectively.

Use of edible oils for production of biodiesel is not feasible because of an enormous gap in demand and supply for food and for fuel [8]. Increased pressure to boost the production of edible oil has limited the utilisation of edible oils for production of biodiesel. Therefore, such plants are thought for biodiesel production which produce non-edible oil in considerable amount and grow in massive scale on non-cropped marginal lands and unused lands [9]. In the present study *Ervatamia coronaria* seeds are chosen as the feedstock for biodiesel production.

1.1 *Ervatamia coronaria* feedstock used in the biodiesel production

The *Ervatamia coronaria* (*Tabernaemontana divaricata*) belongs to the family *Apocynaceae*. It is a glabrous, evergreen tree indigenous to India. It is cultivated in gardens for its ornamental quality. The flowers are white in colour and the seeds are brown in colour as shown in Fig. 2 and Fig. 3. The plant material is widely used as a purgative, stimulant to the brain, the spleen and the liver; in the treatment of cancer, wounds and inflammations in Indian traditional system of medicine [10, 11].

Fig. 2 *Ervatamia coronaria* plant with flower

Fig. 3 *Ervatamia coronaria* seeds

2. MATERIALS AND METHODS

A. *Extraction of oil from seeds of Ervatamia coronaria*

The seeds of *Ervatamia coronaria* are collected and dried in the oven at 50°C for about half an hour in order to remove the moisture present in the seeds. Then they are crushed and powdered. The oil is extracted from the powdered seeds using Soxhlet extractor with petroleum ether (B.P. 40-60°C). The seed oil extract is filtered and dried over anhydrous Na_2SO_4. Petroleum ether is recovered by distillation under vacuum using a rotary evaporator. The percentage yield of oil is calculated by the equation,

$$\textit{Percentage yield of oil} = \frac{\text{Weight of oil}}{\text{Weight of seeds}} \times 100$$

B. *Transesterification of seed oil of Ervatamia coronaria*

Several experimental trials are carried out to evaluate the effects of catalyst type, catalyst concentration, and methanol to oil molar ratio, stirring intensity and reaction temperature on the methanolysis of crude ECSO. The reaction time of 100 min is kept constant for the complete investigation.

The transesterification reaction is carried out in a three necked round bottom flask. ECSO is preheated initially to the required temperature. A mixture of methanol and oil in the molar ratio of 6:1 (w/w) is taken in the reaction flask. Calculated amount of catalysts like NaOH, KOH, $NaOCH_3$ and $KOCH_3$ are used in different experimental sets. As the reaction time is kept constant, each trial is conducted for 100 min in order to ensure complete conversion of *Ervatamia coronaria seed* oil to its methyl esters. After the completion of reaction, the contents are allowed to cool and then transferred into the separating funnel to form two phases. The upper organic phase comprised of fatty acid methyl esters with minor impurities like glycerol, residual alcohol and glycerides. The lower phase mainly comprised of industrially important byproduct glycerol along with the traces of catalyst, un-reacted methanol, etc. The aqueous phase is drained and the organic phase containing FAMEs is washed with warm de-ionized water and then treated with Na_2SO_4 to remove any entrapped moisture content..

3. RESULTS AND DISCUSSION

B. *Analysis of ECSO and ECME*

The oil is extracted using Soxhlet apparatus and the yield of oil is 40.5%. The molecular weight of oil is calculated from fatty acid composition and is found to be 726.9 g/mol. The iodine value (IV) and saponification value (SV) are 98.01g I_2/100g and 200.32 mg KOH/g respectively. ECSO has an acid value (AV) of 0.80 mg KOH/g, and indicates that acid catalyzed transesterification step before base-catalyzed transesterification is not necessary. The physicochemical properties evaluated are presented in Table 2.

TABLE II PHYSICOCHEMICAL PROPERTIES OF *ERVATAMIA CORONARIA* SEED OIL

Physicochemical properties	**ECSO**
Yield of oil (%)	40.5
Density of oil (g/cm^3)	0.925
Molecular weight of oil (g/mol)	726.9
Saponification value (mg KOH/g of oil)	200.32
Iodine value (g I_2/100 g of oil)	98.01
Acid value (mg KOH/g of oil)	0.80

C. Spectral analysis

- *Gas chromatogram of ECSO*

ECSO consists of both saturated and unsaturated fatty acids. Literature survey [12] and GC-FID taken in the present work reveal the following fatty acid composition in *E. coronaria* seed oil (Table 3). Herein, 21.65% of total saturated fatty acid (TSFA) and 78.26% of total unsaturated fatty acid is present in *Ervatamia coronaria* seed oil.

TABLE III COMPONENT FATTY ACIDS IN *ERVATAMIA CORONARIA* SEED OIL

Component fatty acids in *E. coronaria* seed oil	**% Fatty acid [12]**	**% Fatty acid (present work)**
Caprylic acid ($C_{8:0}$)	NA	NA
Lauric acid ($C_{12:0}$)	NA	NA
Myristic acid ($C_{14:0}$)	NA	0.06
Palmitic acid ($C_{16:0}$)	24.4	12.45
Palmitoleic acid ($C_{16:1}$)	0.2	0.06
Cis-10-Heptadecanoic acid (Margaric acid) ($C_{17:1}$)	NA	0.14
Heptadecanoic acid (Margaric acid) ($C_{17:1}$)	NA	0.06
Stearic acid ($C_{18:0}$)	7.2	6.8
Arachidic acid ($C_{20:0}$)	0.7	1.3
Behenic acid ($C_{22:0}$)	0.2	1.1
Oleic acid ($C_{18:1}$)	50.5	49.4
Linoleic acid ($C_{18:2}$)	15.8	26.7
Linolenic acid ($C_{18:3}$)	0.6	1.9
Others	0.2	NA
%TSFA	32.5	21.65
%TUSFA	67.3	78.26

where, NA = Not available

- *FTIR spectroscopic study* The Fourier Transform Infrared (FTIR) analysis of seed oils and its biodiesel are carried out using KBr cell and FTIR spectrophotometer. The FTIR spectrum is measured in the range of 400–4000 cm^{-1}. The spectral studies of fatty acids and their methyl esters in *Ervatamia coronaria* indicate C=O band stretching around 1747.19 cm^{-1}, $–CH_3$ group around 1362.05 cm^{-1} and $–CH_2$ group around 723.65 cm^{-1}. The absorption band at 3009.34-2854.24 cm^{-1} refers to asymmetric and symmetric $–CH_3$ stretching vibrations ($–CO–O–CH_3–$), 1465.55–1438.76 cm^{-1} indicates alkane group (–C–H), 1246.26–1019.80 cm^{-1} refers to the stretching vibration of (–C–O) ester groups and absorption band at 1743.45 cm^{-1} refers to the ester (C=O) carbonyl group (Fig. 4a). The major distinction observed between the IR spectra of seed oil and the biodiesel is a small displacement of the stretching C=O band and stretching C–H band of biodiesel to lower energy (Fig. 4b).

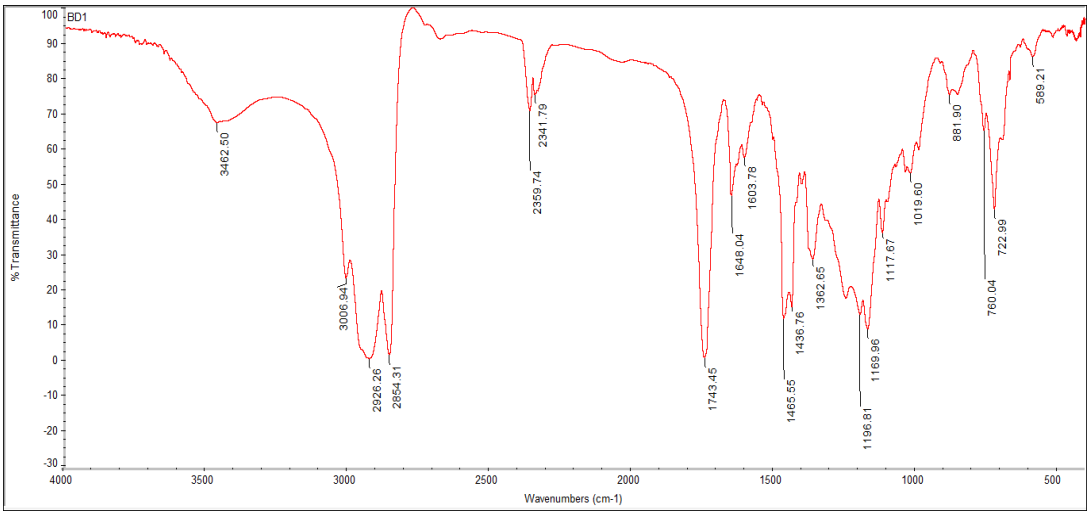


Fig. 4a FTIR of *Ervatamia coronaria* seed oil

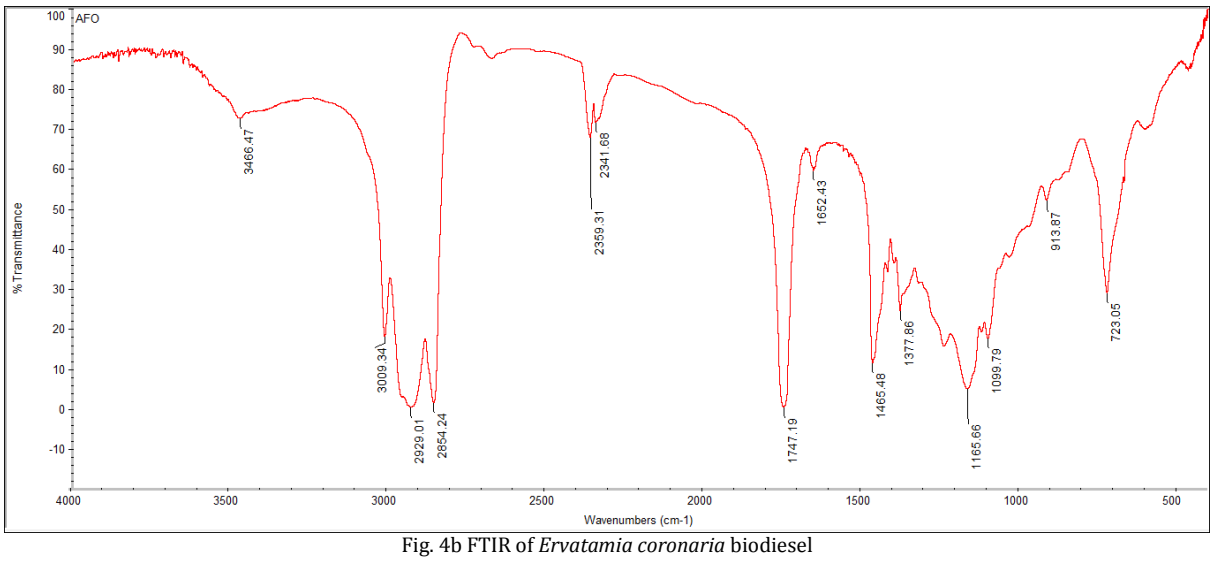


Fig. 4b FTIR of *Ervatamia coronaria* biodiesel

- *^{1}H NMR analysis*

The conversion of ECSO to ECME is analyzed by ^{1}H NMR spectroscopy. At around 4.1-4.3 ppm, resonation of the methylene glyceridic protons in the triglycerides occurs as two doublets while that of the glyceridic methine protons occurs at around 5.25 ppm in ECSO (Fig. 5a). The methylene glyceridic and methyl protons of the FAMEs resonated at around 3.6 ppm and confirm the formation of fatty acid methyl esters from the *Ervatamia coronaria* seed oil (Fig. 5b).

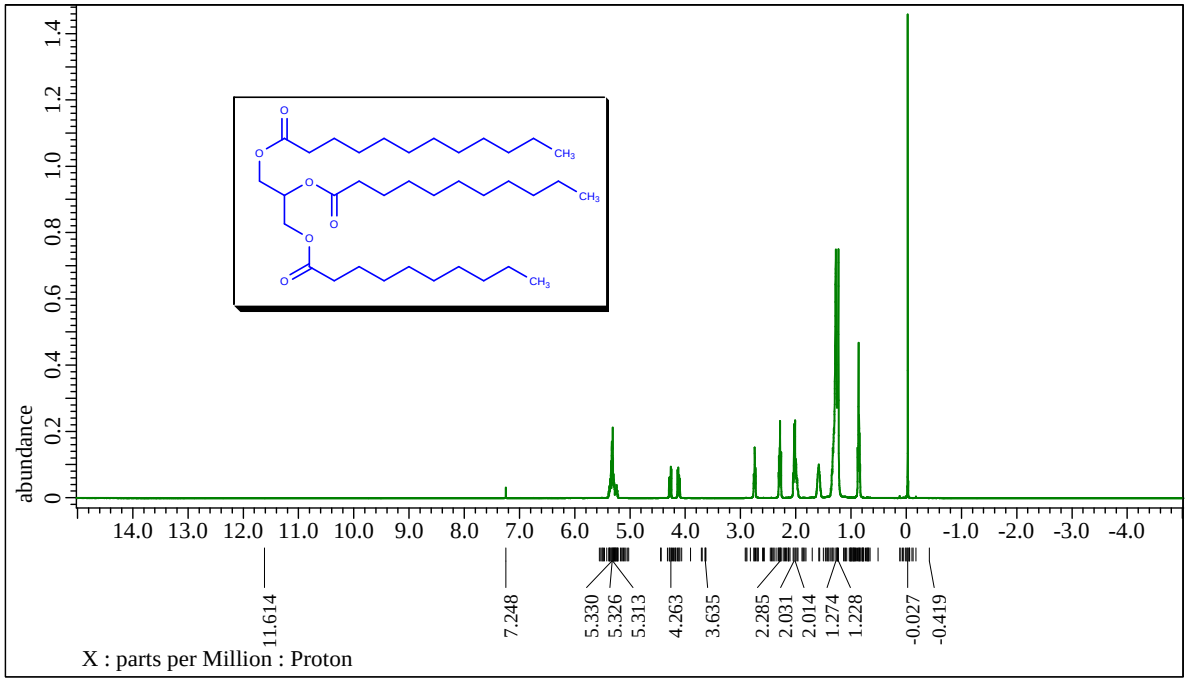


Fig. 5a [1]H-NMR spectra of *Ervatamia coronaria* seed oil

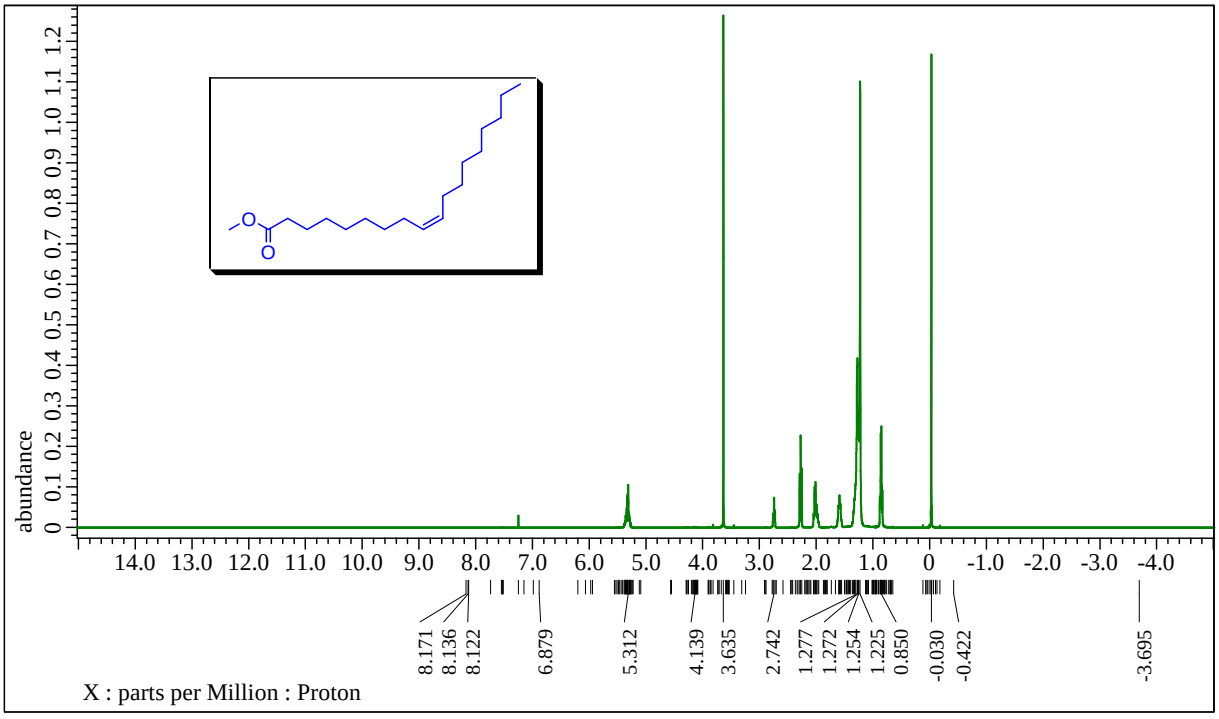


Fig. 5b[1]H-NMR spectra of *Ervatamia coronaria* biodiesel

C. Optimization of Ervatamia coronaria seed oil transesterification

The reaction variables employed during optimization of E. coronaria seed oil transesterification are methanol to oil molar ratio (4:1 to 8:1), catalyst type (NaOH, KOH, $NaOCH_3$, $KOCH_3$) catalyst concentration (0.50-1.50% w/w), reaction temperature (50-70^{0}C) and stirring rate (350-800 rpm). Reaction time of 100 minutes is fixed as a constant parameter throughout the optimization experiments

- *Effect of type of catalyst and catalyst concentration on ECSO transesterification*

  Four different experiments using NaOH, KOH, $NaOCH_3$ and $KOCH_3$ at a concentration of 0.90% w/w are tried in order to determine the effect of catalyst type on the production yield of ECME. In all the experiments, variables such as methanol to oil molar ratio of 6:1, agitation intensity of 650 rpm and reaction temperature of 65^{0}C are kept constant; the optimum yield of ECME is obtained with $NaOCH_3$ catalyst [Fig. 7]. It is found that methoxide is the best catalyst producing greater ester yield than the corresponding hydroxides which indicates that sodium methoxide is better in providing catalytic efficacy toward esterification. In agreement with our present analysis, Vicente, Martinez and Aracil 2004 [13] also reported that the use of sodium methoxide is more appropriate for transesterification of oils.

Along with the type of catalyst used in transesterification, the catalyst concentration also show an important variable that affects the ester yield. As shown in Fig. 8, the increase in the catalyst concentration from 0.70-0.90% increased the methyl ester yield. Further, increase in catalyst concentration decreased the ester yield. This turn down in the ester yield is due to the formation of an emulsion caused due to the addition of excessive amount of alkaline catalyst [14].

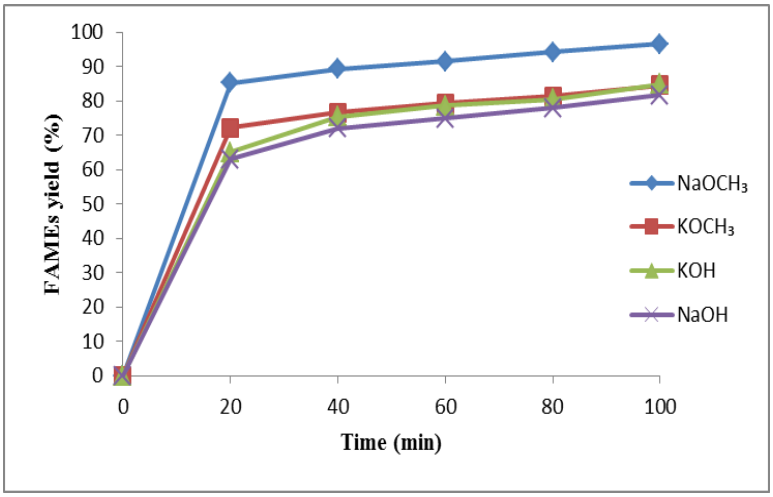


Fig.7 Effect of catalyst type on the yield of ECME

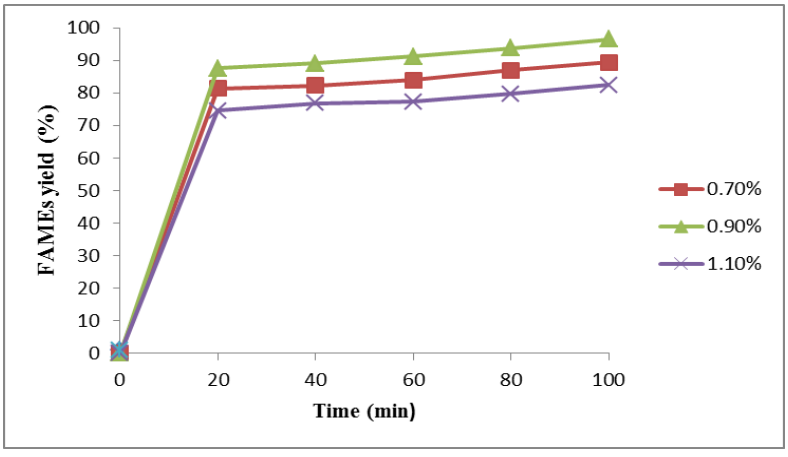


Fig.8 Effect of catalyst ($NaOCH_3$) concentration on the yield of ECME

- *Effects of methanol to oil molar ratio on ECSO transesterification*

The effect of methanol to oil molar ratio as the most important parameter on the yield is appraised by varying the ratio between 4:1 and 8:1, where the reaction temperature, catalyst concentration and agitation rate at 65^{0}C, 0.9% and 700 rpm respectively are kept constant. Methanol to oil molar ratio 6:1 offered the highest biodiesel yield of 98.3% [Fig.9]. When the methanol to oil molar ratio is increased further, to 8:1, there is slight decrease in the ester yield. It was reported by Leung and Guo 2006 [15] that the excess methanol hinders gravity decantation and the yield decreases because a portion of glycerol and other contaminants remain in the organic biodiesel phase.

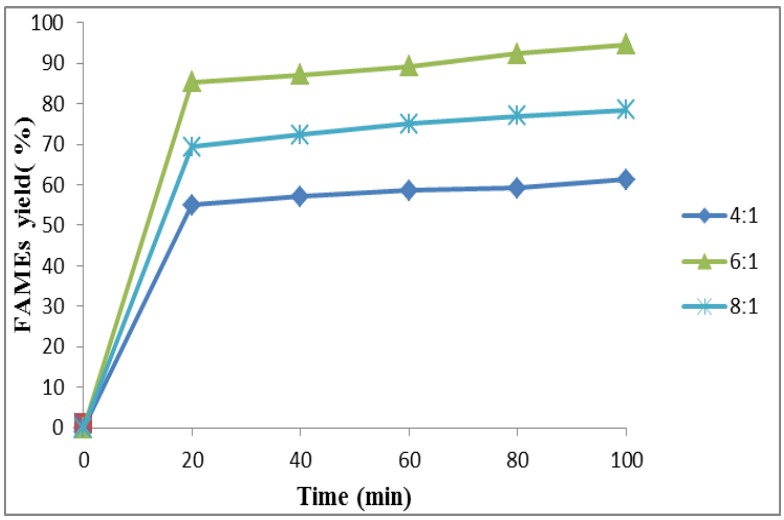


Fig.9 Effect of Methanol to oil molar ratio on the yield of ECME

- *Effect of reaction temperature on ECSO transesterification*

Effect of reaction temperature on methyl ester yield during transesterification of *E. coronaria* seed oil is investigated by choosing the reaction temperatures of 50^{0}C, 55^{0}C, 60^{0}C, 65^{0}C and 70^{0}C. Methanol to oil molar ratio (6:1) and the catalyst concentration (0.90%) of $NaOCH_3$ by weight of oil are kept constant. The maximum ester yield is achieved at 65^{0}C. The graphical representation in Fig. 10 clearly shows that, the ester yield increases with an increase in temperature at 65^{0}C; on the other hand, when the reaction temperature increases beyond the optimal level, yield of biodiesel product decreases. This is observed because of the higher reaction temperature accelerating the saponification reaction of the triglycerides [15].

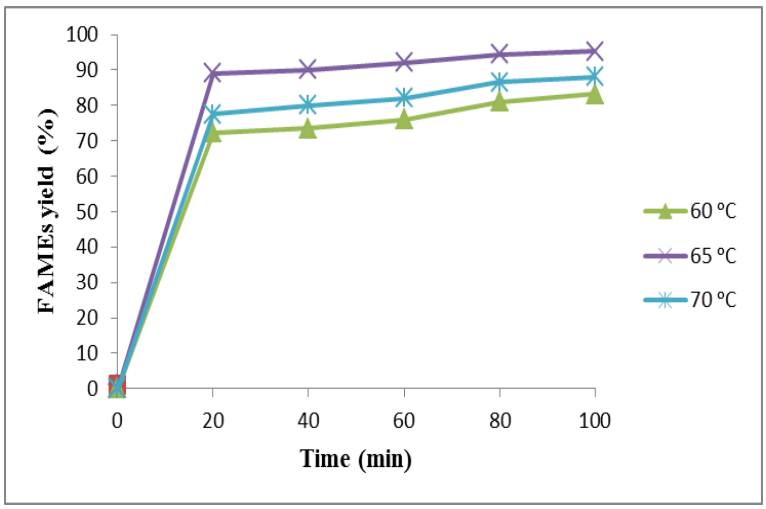


Fig.10 Effect of temperature on the yield of ECME

- *Effect of stirring rate on ECSO transesterification*

The methyl esters yields at different stirring rates during transesterification are presented in Fig. 11. The transesterification reaction conducted at 65^{0}C is optimised with the highest attainable mixing level and the molar ratio of methanol to oil at 6:1 with the best catalyst concentration at 0.90% by weight of *Ervatamia coronaria* oil. The transesterification reaction is not complete at low agitation. But, when the agitation is increased, the ester yield increased appreciably. The ester yield is noted to be high at 650 rpm, later, by increasing stirring intensity the yield appears to decrease slightly.

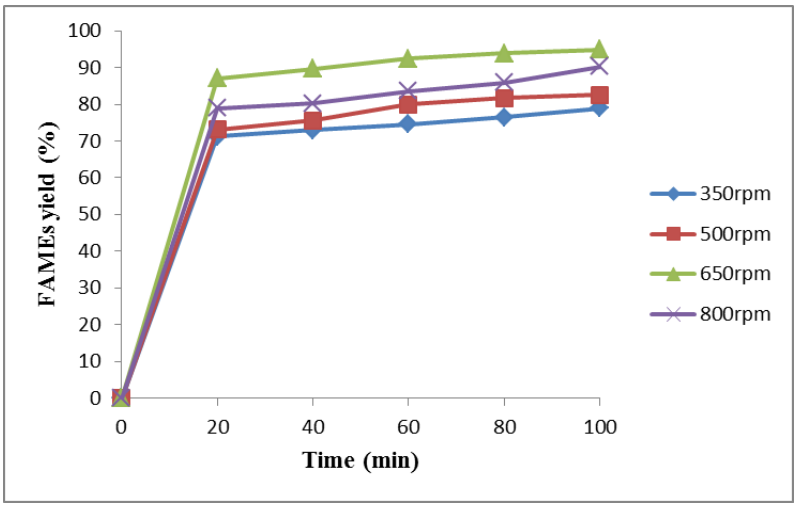


Fig.11 Effect of rate of agitation on the yield of ECME

TABLE IV COMPARISON OF FUEL PROPERTIES OF ECME WITH EXISTING BIODIESEL AND ASTM D6751 AND EN14214 STANDARDS [16, 17]

Fuel property	ECME	JPME	ASTM D6751	EN14214	Test Method
Cetane number	55.72	57	>47	51	D 613
HHV ($MJkg^{-1}$)	38.6	38.6	NA	NA	ASTM D 5865
Viscosity at 40^0C ($mm^2 s^{-1}$)	3.650	4.84	1.9-6.0	3.5-5.0	D 445
Density at 15^0C (gcm^{-3})	0.890	0.880	NA	0.860-0.900	D 1298
Flash point (^0C)	190	192	>93	>101	D 93
Cloud point (^0C)	– 4.7	8	– 3 to 12	NA	D 2500
Pour point (^0C)	– 11.9	6	– 15 to 16	NA	D 97
Cold flow plugging point	– 7.6	4.2	NA	NA	D 6371
Water and sediment (vol %)	0 .02	0.025	0.05	0.05	EN ISO 12937
Carbon residue (wt %)	0.010	0.020	0.05	0.3	EN ISO 10370
Sulfated ash (mass %)	0.010	0.012	0.020	0.02	EN ISO 20846
Oxidation stability (h)	4.10	4.6	>3.0	6.0	EN 14111

NA- not applicable

- *Fuel properties of Ervatamia coronaria methyl esters*

The ASTM standard and EN standard have set the cetane number limitation as minimum 47 and 51 respectively. The cetane number of *Ervatamia coronaria* methyl esters is calculated using the equation $CN_{mix} = \sum A_C \times CN_C$ [18] and the value obtained is 55.72 which satisfy the biodiesel standards of ASTM and EN and is comparable with the existing Jatropha biodiesel. Ramdhas A S [19] reported that the higher heating value (HHV) of petro-diesel is 46.0 MJ kg^{-1}. In the present work, HHV of FAMEs of *Ervatamia coronaria* is determined to be 38.6 MJ kg^{-1} which is comparable to the existing biodiesel but lower than that of petro-diesel. The higher heating value of petro-diesel is because of presence of higher carbon and hydrogen content in petro-diesel. However, biodiesel which is a mixture of fatty acid esters (oxygenates) envisages reduced carbon content per unit mass. Nonetheless, the oxygen present in biodiesel molecules promotes relatively better combustion efficiency lowering HHV of ECME [20]. Kinematic viscosity of ECME obtained at 40^0C is 3.650 $mm^2 s^{-1}$ which satisfies the ASTM standard and is comparable with Jatropha biodiesel. EN 14214 specification for density of biodiesel is 0.860-0.900 g cm^{-3} and density obtained for ECME is 0.890 g cm^{-3}. Flash point is determined according to ASTM D93 standards and is found to be 190^oC (Table 4). The obtained value is comparable with the existing Jatropha biodiesel and also satisfies the ASTM and EN conditions.

Even the water and sediment content is low when compared with the standards. The carbon residue is found to be 0.01%. The oxidation stability is found to be 4.10 hr. The observed CP, PP, CFPP values are -4.7^0C, -11.9^0C and -7.6^0C respectively, and the values obtained are acceptable by ASTM and EN standards

4. Conclusion

The present study examined the optimum reaction conditions for transesterification of *Ervatamia coronaria* seed oil to be 0.90% $NaOCH_3$ as catalyst, 6:1 as methanol to oil molar ratio, 65^0C as the reaction temperature and 650 rpm as the stirring rate offering 98.3% biodiesel yield. From the present investigation, it can be concluded that under an optimized set of transesterification conditions, non-edible *Ervatamia coronaria* seed oil can be used as a feedstock for biodiesel production with a very good yield

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