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Synthesis of Magnetic Nanocatalyst CaO/CoFe3O4 for Biodiesel Production using Pongamia pinnata oil Seed

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Research Article

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Synthesis of Magnetic Nanocatalyst $CaO/CoFe_3O_4$ for Biodiesel Production using *Pongamia pinnata* oil Seed

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Abstract

Biodiesel has emerged as prominent alternative source for commercial fuels. *Pongamia pinnata* (*P. pinnata*) has been recognised as the potential source for biodiesel, prepared from its seeds. The present study is to develop a novel heterogenous magnetic nano catalyst $CaO/CoFe_3O_4$ for the preparation of biodiesel from P. pinnata through transesterification reaction and improve the catalyst reusability. Magnetic core Fe_3O_4 and $CoFe_3O_4$ were prepared by co-precipitation method. Crab shell was calcinated at 600°C for 4 h for the preparation of calcium oxide (CaO). $CaO/CoFe_3O_4$ was synthesized by doping of CaO and $CoFe_3O_4$. Characterization of the synthesised catalyst was done using Fourier Transform Infrared Spectroscopy, Scanning Electron Microscopy, X-ray Diffraction and Particle Size Analyzer to analyses the functional group, morphology, crystalline structure and particle size respectively. The optimized parameter for biodiesel production by transesterification are 12:1 molar ratio of methanol to oil, 2.5wt% catalyst concentration, 120 min reaction time, 65°C reaction temperature and 500 rpm stirring rate to give a maximum of 98.56 wt% yield. The reusability study conferred that the biodiesel yield of above 92 wt% were obtained when regenerating and reusing the catalyst up to six cycles, further the yield drops below 65 wt% at ninth cycle of catalyst reusability. After transesterification, the fuel properties were analyzed using ASTM D6751 standard and it was found within prescribe limit.

Keywords*:* Biodiesel; *Pongamia pinnata*; Magnetic nanocatalyst; Transesterification; Doping; Co-precipitation.

1. Introduction

Conventional fuels such as petrol, diesel, coal, etc., are the significant energy source which needs thousands of years to generate with limited non-renewable resources. The demand for such fuels has been rising daily and the utilization of these conventional fuels may lead to the discharge of more gaseous pollutants. In past two decades, the yearly utilization of petro-diesel fuels has been increasing at a rate of 1.5%, resulting in the increase of the price of the fuels (Quaye, 1996). This uncertainty of petroleum fuel availability has created a need for the production of alternative fuels. Hence, scientists are on the research on biodiesel as an alternate of diesel (Madhu et al., 2016).

Biodiesel ie., mono-alkyl ester is a renewable, carbon neutral, non-toxic, biodegradable fuel which is an alternative to the conventional petroleum derived diesel fuel. It is obtained by the reaction of fatty acids with an alcohol in presence of catalyst. It is an environment friendly fuel and produces fewer pollutants (Knothe, 2009). Biodiesel has higher oxygen content than petro-diesel, and have less sulphur and nitrogen content. On combustion, it releases less CO_2, CO, and unburned hydrocarbon (Arunachalam Sivagurulingam et al., 2022). Owing to the high cost of production, biodiesel has not been expanded worldwide. Also, the regeneration, post treatment and the disposal of catalysts has been creating more crises. Thus, to overcome this, the heterogeneous catalysts can be used (Yadav et al., 2017).

The feedstocks used to produce biodiesel can be of four generations. Soya beans, Canola, Rape seed, Coconut, Wheat, etc., are edible feedstocks and Jatropa oil, Karanja seeds, Castor oil, Palm oil, Neem oil, etc., are non-edible feedstocks (Naik & Katpatal, 2013). Moreover, waste cooking oils and non-edible crude vegetable oils are the potential source for the production of biodiesel due to their lower market price (Baskar & Soumiya, 2016).

The non-edible feedstocks contain the free fatty acid (FFA) content of about 4%, and it also reduces the dependency on food crops as compared to edible feedstock. Also, these oils are cheaper than edible one and it grown in any wastelands and there is no need for agricultural land, water and fertilizer for their growth (Roick et al., 2021). Among the source of non-edible oils, P. pinnata (Pongana or Punga in Tamil) is found in almost every part of India especially in Western Ghats and it is grown in any soil types. It is a Actinorhisal plant which has the ability to fix the nitrogen in the soil. The temperature required is slightly less than 0℃ to above 50℃ with annual rainfall between 500 to 2500 mm. Notably, P. pinnata seed contains 30-40% oil content, making it a potential feedstock for the

synthesis of biodiesel. Additionally, its seeds comprise of 17-31% proteins, 6-7% starch, 5-7% crude fiber, 15-20% moisture and 2-3% ash (Rengasamy et al., 2014).

A homogeneous alkali and acid catalysts catalyst are commonly used for the biodiesel production. The alkali catalysts like sodium hydroxide and potassium hydroxide widely used in the industrial production of biodiesel in particular wt% with respect to the amount of feedstock used. Even though, when the oil with high FFA is used, the alkali catalyst reacts with FFA and produce soap. The catalyst gets deactivated and supress the production of biodiesel. These drawbacks of alkali catalyst led to the use of acid catalyst such HCl, H_3PO_4, and H_2SO_4. But, those catalysts are not effective for the large scale production due to its lower reaction rate (Tamjidi et al., 2021).

Heterogeneous catalysts have emerged as a viable option for biodiesel production, offering numerous benefits over traditional homogeneous catalysts (J. Zhang et al., 2010). These catalysts are easily separable from products, non-corrosive, eco-friendly, and can be regenerated for reuse. Also, over all heterogeneous catalysts, calcium-based catalysts were employed as potential catalyst for the production of biodiesel (Xie & Zhao, 2013). However, the integration of nanotechnology in production of biodiesel has provided way to develop the nanocatalysts, offering a paradigm shift in catalytic efficiency. Recent advancements in nanotechnology have provided way for the preparation of magnetic nano catalysts, which have shown great promise in biodiesel production from inexpensive feedstocks. These catalysts offer easy separation and reusability due to their magnetic properties, eliminating the need for centrifugal techniques and ultrafiltration. Research has demonstrated that magnetic nanomaterials maintain their catalytic activity over multiple cycles (Miceli et al., 2021).

Magnetic nano catalysts were undissolved during the reaction this may leads to easy separation from the product via magnetic field. Magnetic nano catalyst has the reusable and recovery results in cost reduction and environmental impacts (Salimi & Hosseini, 2019) Advantages of the magnetic nano catalysts includes the high reusing ability, non-soluble nature in organic solvents, production of minimum waste and high stability (Sami et al., 2022a).

The four major techniques to produce biodiesel are dilution, transesterification, thermal cracking (pyrolysis) and microemulsion. Due to the high viscous nature of the oil, transesterification is widely used to prepare biodiesel (Uddin et al., 2017). Catalysts play a vital role in the transesterification process, thereby enhancing both the rate of reaction and overall yield of biodiesel production. (Ejikeme et al., 2010).

Biodiesel production using transesterification reaction involves transforming triglycerides into fatty acid methyl ester (FAME). The primary component produced was biodiesel and the by-product obtained was glycerol. It is the most commonly used process for biodiesel production due to its high yield, relatively low cost and ability to produce biodiesel that meets international fuel standards. It is also a versatile process that uses various feedstocks, but it is sensitive because of the occurrence of FFA and water. With proper feedstock preparation and control of reaction conditions, transesterification remains the cornerstone of industrial biodiesel production (J. Zhang et al., 2010). However, the biodiesel obtained should satisfy the physico-chemical properties as prescribed in the ASTM D6751 standards. So, it is mandatory to determine the physio-chemical property of the biodiesel. (Sami et al., 2022b).

This study is aimed to synthesis biodiesel from the oil of P. pinnata seed using a first-of-its-kind heterogeneous nano magnetic $CaO/CoFe_3O_4$ catalyst. FTIR, SEM, XRD, and PSA studies were used to characterize the synthesized catalyst. The oil was extracted from P. pinnata seed and then exposed to a variety of reaction circumstances, including concentration of catalyst, molar ratio of methanol to oil, stirring rate, time of reaction, and temperature of reaction to optimize the transesterification process which led to the production of biodiesel. Moreover, a study on catalyst reusability was conducted to investigate the biodiesel yield when the catalysts were reused several times. The properties of the fuel were then ascertained using ASTM procedures and it was contrasted with the standard of ASTM D6751.

2. Materials and Methods

2.1 Materials

Crab shells were collected from Ukkadam fish market, Coimbatore. Hexane and methanol was purchased from Hi-media Laboratories private ltd., Mumbai. The experiments were performed by utilizing analytical grade chemical without any purification, and the deionized water was employed throughout the experiments.

2.2 Collection, pre-treatment of oil seed and extraction of oil

P. pinnata seed were collected from a farm land in Pongalur, Tirupur. To remove the moisture content, the seeds were dried for 2 h at 110℃. The skin was peeled, milled into powder and stored in desiccator for further use (Bobade SN & Khyade VB, 2012). Oil extraction was carried out using soxhlet apparatus at 70℃ for 8 h. The powdered seed and n-hexane was taken in the ratio of 1:6 (w/v) %. Then, the oil was separated by simple distillation

and the solvent was recovered for reuse (Hasnah et al., 2020). The yield of the extracted oil is calculated using the given Eq. (1).

$$\text{Oil yield (wt \%)} = \frac{Weight\ of\ oil\ extracted\ (g)}{Weight\ of\ seed\ taken(g)} \text{ x } 100 \quad (1)$$

2.3 Synthesis of heterogenous magnetic nano catalyst ($CaO/CoFe_3O_4$)

Iron oxide (Fe_3O_4) was co-precipitated by dissolving ferric chloride ($FeCl_3$) and ferrous sulphate heptahydrate ($Fe_2SO_4.7H_2O$) and titrated against 4N NaOH solution (Wei et al., 2012). The crab shell were collected, cleaned, dried at room temperature and heated at 600°C for 4 h in a muffle furnace to obtain the powdered CaO, which is a catalytic core (Madhu et al., 2016). The magnetic core was synthesised by reacting cobaltous sulphate heptahydrate ($CoSO_4.7H_2O$) and iron oxide (Fe_3O_4) using 4N NaOH solution as precipitating agent. The magnetic core ($CoFe_3O_4$) and the catalytic core (CaO) was co-precipitated to form a nanocatalyst ($CaO/CoFe_3O_4$), which has the magnetic property as well as catalytic property (P. Zhang et al., 2014a). The preparation of $CaO/CoFe_3O_4$ is given by the chemical reaction as follows (Eq. 2)

$$CaO + CoFe_3O_4 \xrightarrow{NaOH} CaO/\ CoFe_3O_4 \quad (2)$$

2.4 Catalyst characterisation

The spectrum of the $CaO/CoFe_3O_4$ catalyst was recorded between the wavelengths ranging from 400-4000 cm^{-1} at a resolution of 4 cm^{-1} using FT/IR–4600 type A spectrometer to study the functional group exists on the catalyst surface. The structure of the catalyst were analysed by means of Empyrean X-ray diffractometer (Ali et al., 2017). Surface topography of the prepared catalyst was analysed using Carl ZEISS EVO 18-Germany. Mastersizer 3000 laser scattering particle size analyzer is used determine the size and distribution of particles size of the synthesized catalyst (Booramurthy et al., 2020).

2.5 Biodiesel reaction setup

The experiment was performed by placing the 250 ml two neck round bottom flask in the magnetic stirrer with hot plate. A known quantity of oil was introduced initially into the reaction setup. Subsequently, to the oil catalyst dispersed in methanol was added. The reaction parameters were optimized by varying the methanol to oil molar ratio (3:1-15:1), catalyst concentration (0.5-3 wt %), reaction time (30-150 min), reaction temperature (35-75°C) and the stirring speed (100-600rpm) (Rezaei et al., 2013). For the optimization process one factor at a time

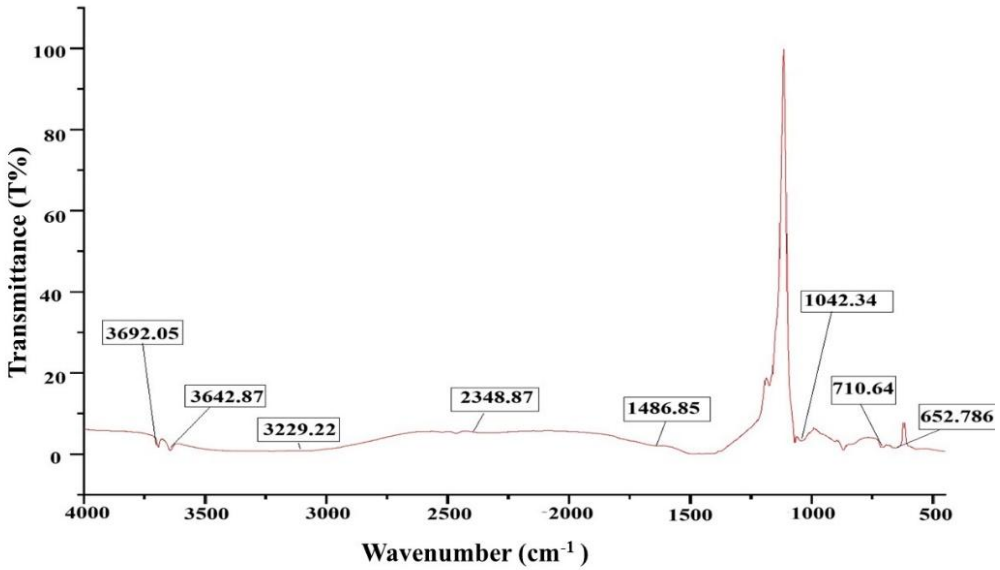
157 method was employed, in which one parameter was varied while other variables remained constant. After the
158 reaction was completed, the catalyst was recovered using the permanent magnet and the content in the flask was
159 poured in the separating funnel and allowed overnight for the phase separation. The upper layer was biodiesel and
160 the lower layer is crude glycerol (Senthilkumar et al., 2019). The biodiesel yield was then calculated gravimetrically
161 using the equation given below. (Eq. 3) (Ramasamy et al., 2023).

162 $$Biodiesel\ yield\ (wt\%) = \frac{Weight\ of\ biodiesel\ (g)}{Weight\ of\ oil\ (g)} \quad (3)$$

163 **3. Results and Discussion**

164 **3.1 Catalyst characterisation**

165 The spectral analysis of FTIR shown in the Fig. 1 indicates the intense absorption takes place at 3692.05
166 cm^{-1} and 3642.87 cm^{-1} which is particular to medium and sharp -OH stretching. This functional group represents the
167 presence of free alcohol group property (Hazmi et al., 2021). Strong absorption band at 2348.87 cm^{-1} corresponds to
168 strong O=C=O stretching and it corresponds to the CO_2 absorption and indicating the strong basic property (Ali et
169 al., 2017). The peaks at 3229.22 and 1486.85 show the existence of moisture that on the catalyst surface
170 (Mohammadi & Barikani, 2014). Peak at 1042 cm^{-1} indicates strong and broad CO-O-CO stretching which
171 represents the occurrence of anhydrides. A broad peak at 710.64 cm^{-1} and 652.786 cm^{-1} shows the existence of
172 medium C=C alkene bending vibration and presence of halo compounds (Gaurav et al., 2021).



173

174 **Fig. 1 FTIR spectrum of $CaO/CoFe_3O_4$ catalyst**

The XRD pattern of magnetic core $CoFe_3O_4$ and solid catalyst $CaO/CoFe_3O_4$ is shown in the Fig. 2. From the graph, it is inferred that the catalyst is crystalline in nature as the peaks are sharp. The peaks at 23.8, 27.7°, 37.8° and 33.5° shows the presence of CaO and magnetite (ICDD 37-1497) (Liu et al., 2016).The smooth peak at 37.8° shows the magnetite's cubic inverse spinal system (Booramurthy et al., 2020). The diffraction with the peak value of 21.9° and 30.4° represents the magnetic core $CoFe_3O_4$ according to the standard ICDD 22-1086 (P. Zhang et al., 2014) ; (Teo et al., 2019).

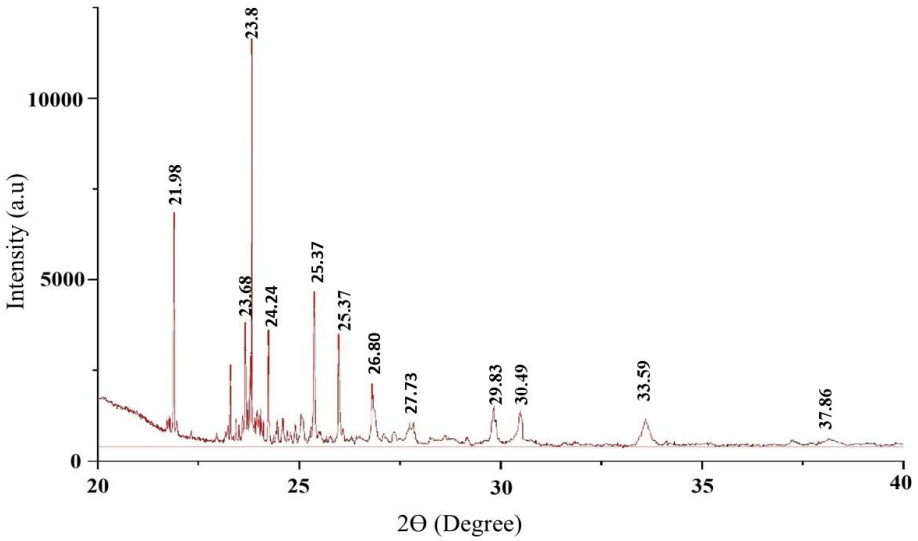


Fig. 2 XRD pattern of $CaO/CoFe_3O_4$ catalyst

SEM analysis was done to confer and understand the topography of the catalyst. The surface topography revealed a distinctive, flower like cluster arrangement as in shown the Fig. 3. The thin petal like features observed in the structure contributes to the catalyst's high surface area (Teo et al., 2019); (Gaurav et al., 2021). The image at high magnification shows that the powder consists of submicron multifaceted particles which reveals uniform distribution of CaO on the surface of Fe_3O_4 (Ali et al., 2017).

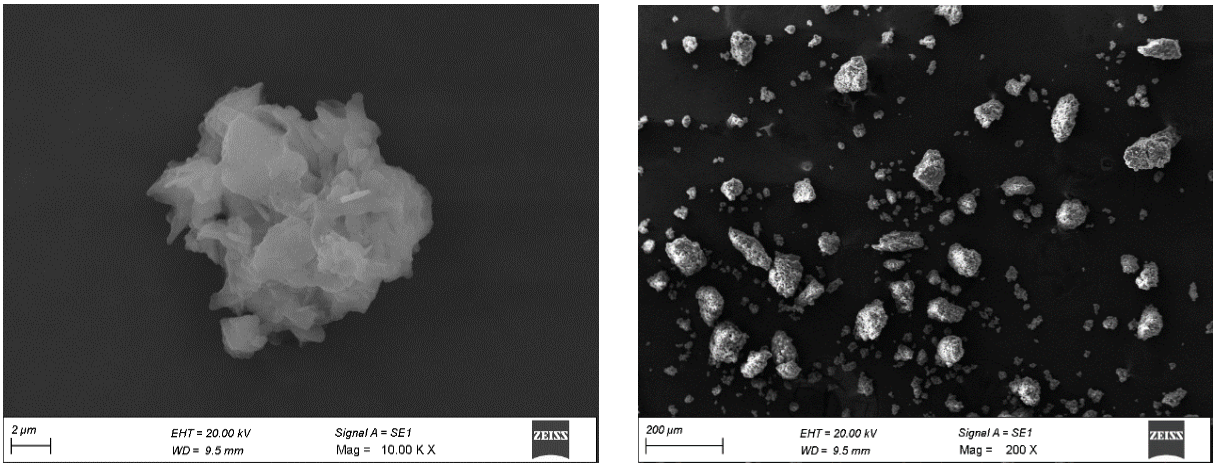


Fig. 3 SEM image of $CaO/CoFe_3O_4$ catalyst

In Particle size analyzer, it seen from the Fig. 4 that the distribution of particles lies between 20 and 100 nm. The peak was observed at 70 nm as shown in the Fig. 4, because most of the particle size distribution nearly 90% takes place between 20 and 70 nm. Hence, it is confirmed that the synthesized catalyst $CaO/CoFe_3O_4$ was in nano size distribution (Booramurthy et al., 2020b). Using the Debye-Scherrer's relation (Eq. 4), the crystalline diameter of the catalyst is calculated as 63 nm. This confers to the result obtained through particle size analyser (Alhassan et al., 2016; Booramurthy et al., 2022).

$$T = \frac{0.94\,\lambda}{\beta Cos\theta} \tag{4}$$

where, T is the average particle size, λ is the X-ray wavelength, β is the full width at half maximum and θ is the diffraction angle (Booramurthy et al., 2022).

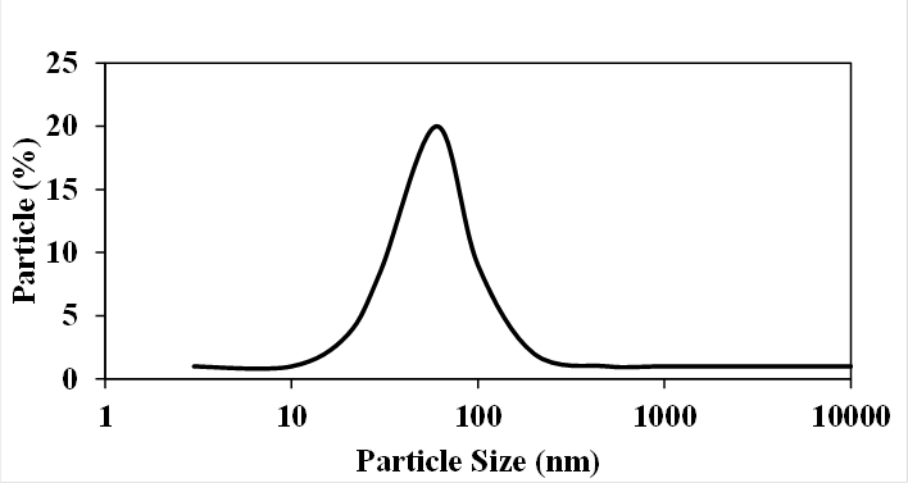


Fig. 4 Particle size distribution of $CaO/CoFe_3O_4$ catalyst

3.2 Characterization of *P. pinnata* oil

The yield of oil extracted from the *P. pinnata* seed was about 57.59 wt%. It was yellowish orange in colour at room temperature. The acid value of the oil was found to be 0.88 %, which is below the threshold value of 1%. This condition favours direct transesterification without undergoing esterification process (Chai et al., 2014). The fatty acid composition of the *P. pinnata* oil was determined and given in the Table 1.

Table 1 Fatty acid composition of *P. pinnata* oil

Fatty acid	Carbon number	Composition (%)
Oleic acid	C18:1	51.54
Palmitic acid	C16:0	13.76
Stearic acid	C18:0	8.50
Linoleic acid	C18:2	16.69
Lignoceric acid	C24:0	2.62
Behenic acid	C22:0	1.35
Eicosanoic acid	C20:0	4.45
Myristic acid	C14:0	1.09

3.3 Optimization of transesterification process

The transesterification is crucial process with multiple variables that affects the yield of biodiesel. The various parameters such as effect of concentration of catalyst, molar ratio of methanol to oil, stirring rate, time of reaction, and temperature of reaction are to be optimized to get maximum yield of biodiesel.

3.3.1 Effect of methanol to oil molar ratio

The molar ratio of methanol to oil was investigated over a range from 3:1 to 15:1 to find the optimal condition. A maximum biodiesel yield of 85.64% was achieved at molar ratio of 12:1 as shown in the Fig. 5. As compared to (Sami et al., 2022b), where the yield is 84% in 15:1 molar ratio. This shows that increase in yield of biodiesel even in lower methanol concentration. Normally the increase in the methanol concentration leads to high yield of biodiesel due to shift in equilibrium. However, the increase in methanol concentration may disrupt the catalyst activity by dissolving it, so the reduction in the yield happens at high methanol concentration (Chingakham et al., 2019).

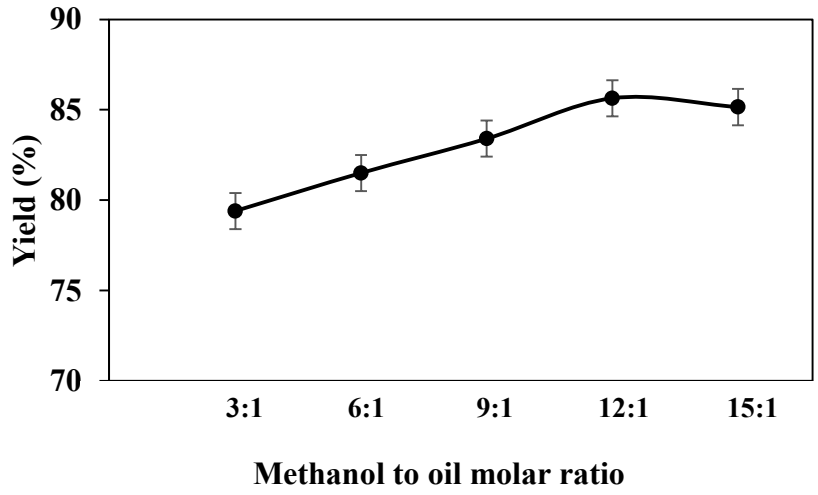


Fig. 5 Effect of methanol to oil molar ratio

3.3.2 Effect of catalyst concentration

The catalyst concentration was investigated over a range of 0.5 to 3 wt%, in which maximum yield of 88.14% was achieved at 2.5 wt% with molar ratio of methanol to oil of 12:1, as illustrated in Fig. 6. From the study conducted by (Hanif et al., 2022), at similar catalyst concentration used the yield obtained was 87%, but in this study maximum yield was achieved at 2.5 wt%. When exceeding this threshold of catalyst concentration, it results in declining the biodiesel yield. The loading of catalyst is optimized on the basis of type and nature of the catalyst, also with respect to the composition of fatty acid present in the oil.

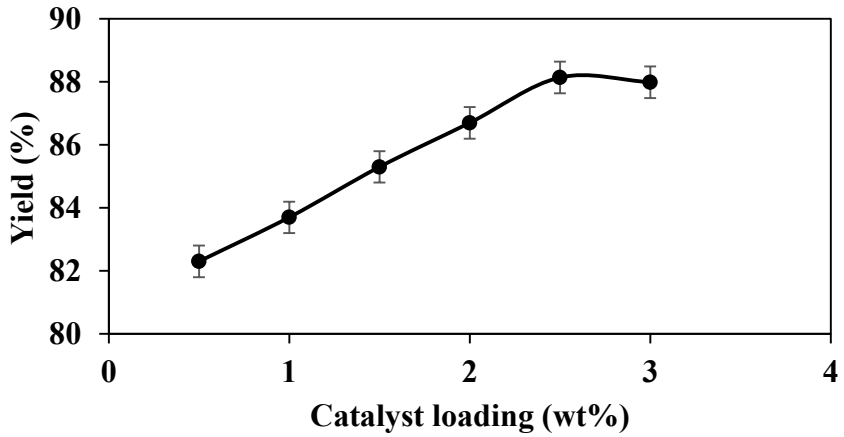


Fig. 6 Effect of catalyst concentration

3.3.3 Effect of reaction time

The reaction is carried out between the 30-150 min. From the Fig. 7 it was observed that the biodiesel yield increases with increase in time and reaches a maximum of 92.43% at 120 min with methanol to oil molar ratio of 1:12 and catalyst concentration of 2.5wt%. Similar yield was obtained at the same reaction time and with higher methanol concentration (Hanif et al., 2022). Prolonged reaction times beyond the optimal point lead to the reduction in biodiesel yield, which is due to the deactivation of the catalyst's active sites (Arunachalam Sivagurulingam et al., 2019).

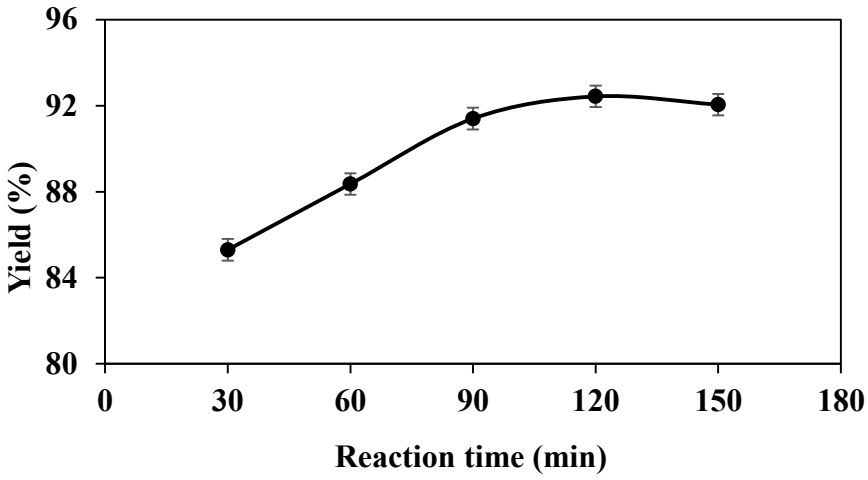


Fig. 7 Effect of reaction time (min)

3.3.4 Effect of reaction temperature

The reaction temperature varies from 35 to 75℃ with methanol to oil molar ratio of 12:1, catalyst concentration of 2.5 wt% and reaction time of 120 min. From the Fig. 8, the optimum temperature was identified as 65℃ to get a maximum yield of 95.12%. The increase in temperature above 65℃ which may result in the reduction in biodiesel production and results in vaporization of methanol because the boiling point of methanol is 65℃ and it illustrate the strong effect of temperature in the transesterification reaction (Deshmane & Adewuyi, 2013).The reaction temperature is not related to the feed stock used for the transesterification (Arunachalam Sivagurulingam et al., 2019).

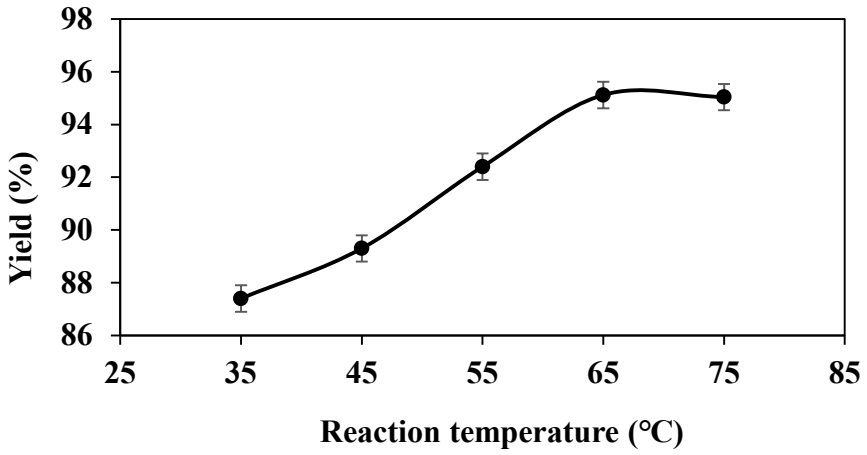


Fig. 8 Effect of reaction temperature

3.3.5 Effect of Stirring rate

In order to improve the biodiesel yield, it is important to mix the oil with methanol. The stirring rate was varying from 100 to 600 with the methanol to oil molar ratio of 12:1, catalyst concentration of 2.5 wt%, the reaction time and temperature at 120 min and 65°C respectively. A maximum yield of 98.56% was achieved at the optimized stirring rate of 500 rpm and further increasing the speed show drop in yield as shown in the Fig. 9.

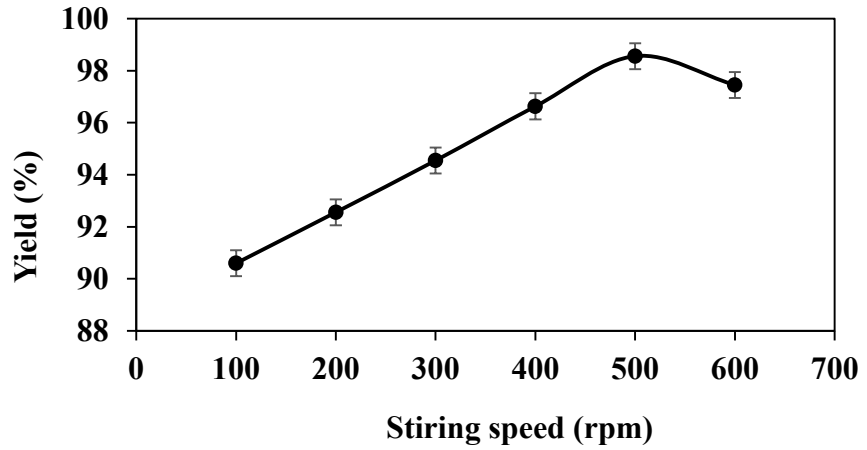


Fig. 9 Effect of stirring rate

3.4 Catalyst Reusability

The catalyst reusability is crucial step for reducing the biodiesel production costs, as poor reusability can lead to decreased catalyst performance over time. To assess this, the catalyst was recovered and regenerated after each cycle by washing with hexane and methanol to remove the residues and triglycerides (Booramurthy et al.,

2020b). The reduction in biodiesel yield was observed from 98.56 to 92.6 wt% and it declines below 92 wt% beyond the sixth cycle and it is shown in the Fig. 10. Moreover, the use of same catalyst leads to a drastic reduction in yield to 64 wt% after sixth cycle and this reduction due to the leaching of active site and partial blockage of the pores of the catalyst (Kaur et al., 2018).

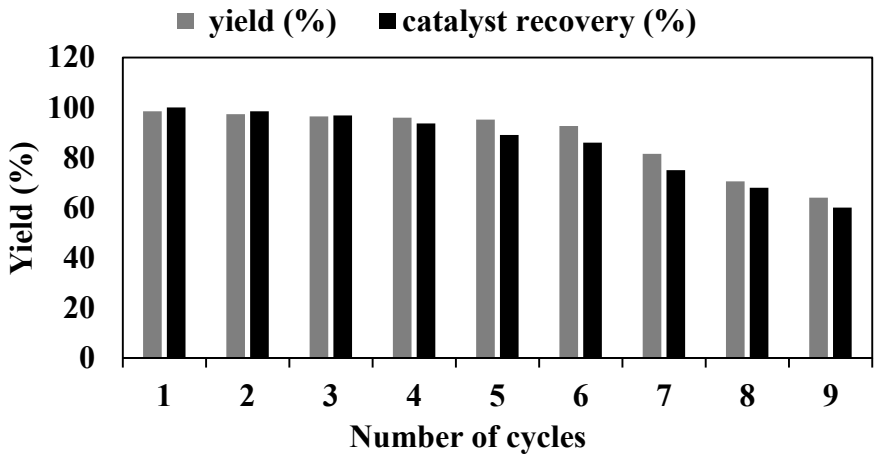


Fig. 10 Catalyst ($CaO/CoFe_3O_4$) reusability

3.5 Fuel properties of *Pongamia pinnata* oil seed

ASTM D6751 is the standard specification for biodiesel and to ensuring about the quality and safety for use in diesel engine. As per the ASTM standard the physiochemical properties of *P. pinnata* was determined and given in the Table 2.

Table 2 Fuel properties of produced biodiesel from *P. pinnata* oil seed

Properties	ASTM D6751 Standard	*P. pinnata* biodiesel
Density at 15℃ (kg/m^3)	-	883
Viscosity at 40℃ (mm s^{-1})	1.9- 6.0	4.6
Flash point (℃)	130 min	165
Sulphated ash (% mass)	0.02 max	0.012
Oxidation stability (h)	3.0 min	5.76
Cetane number	47 min	53
Water & sediments (Vol %)	0.05 max	0.031
Acid value (mg KOH g^{-1})	0.05 max	0.032
Copper strip corrosion	No. 3 max.	1a
Carbon residue (wt %)	0.05 max.	0.038

4. Conclusion

The heterogeneous magnetic nano catalyst ($CaO/CoFe_3O_4$) was used to catalyse the transesterification reaction to produce biodiesel from *P. pinnata* oil. The maximum yield of 98.56% obtained by maintain the reaction with optimized parameter such as 12:1 methanol to oil molar ratio, 2.5 wt% catalyst concentration, reaction time of 120 min at the temperature of 65°C and stirring rate of 500 rpm. The catalyst was recovered and reused up to six cycles with the considerable yield above 92 wt% and further the yield drops below 65 wt% at ninth cycle of catalyst reusability. The physico-chemical properties of the biodiesel were determined as per ASTM D6571 standard and it lies within the prescribed limit.

Declaration

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This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

296 **Conflict of interest**

297 The authors declare that they have no competing interests

298 **Ethics approval and consent to participate**

299 There is no data involving about human being or animal or tissue in this manuscript.

300 **Consent for publication**

301 The manuscript does not contain any individual person's data.

302 **Data availability**

303 Data will be made available on request.

304 **Materials availability**

305 Not Applicable

306 **Code availability**

307 Not Applicable

308 **Author contribution**

309 Equal Contribution from all authors

310 **References**

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