

Article

Environmentally Safe Magnetic Nanocatalyst for the Production of Biodiesel from *Pongamia pinnata* Oil

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Abstract: Biodiesel is an alternative fuel in many developing and developed countries worldwide. Biodiesel has significant and numerous economic, environmental, and social benefits. However, the problem with conventional biodiesel production is the high industrial production cost, mainly contributed by the raw materials. Therefore, catalysts and feedstock are essential in increasing total biodiesel production rates and minimizing production costs. Magnetic nano-catalysts play a crucial role in heterogeneous catalysis due to their easy recovery, recyclability, excellent selectivity, and fast reaction rates, owing to their larger surface area. This research activity used heterogeneous magnetic nano-catalysts of ICdO, ISnO, and their modified form, to produce biodiesel. The synthesized nano-catalysts were made through co-precipitation and found quite efficient for transesterifying *Pongamia pinnata* oil. The effect of various parameters on biodiesel yield in the presence of prepared magnetic nano-catalysts has been studied. In the transesterification supported by ISnO, high yield, i.e., 99%, was achieved after 2 h of reaction time at 60 °C. The nano-catalysts were magnetically recovered and reused 4–5 times without any change in their activity. All the synthesized magnetic nano-catalysts performed SEM analysis. Each fraction of the produced biodiesel was assessed for different quality parameters, and the results were per ASTM standards. The components present in biodiesel produced from *Pongamia pinnata* oil were determined by GCMS.

Keywords: magnetic nano-catalysts; heterogenous catalysis; *Pongamia pinnata* oil; biodiesel



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1. Introduction

Primary energy demand is predicted to increase worldwide by 1.6% annually through the next 10 years [1]. Fossil fuels are the primary energy resources used today, such as coal (29%), crude oil (35%), and natural gas (24%). Other resources used for global energy consumption account for renewable energy 5%, and nuclear energy 7%. Therefore, fossil fuels are the single largest energy source and account for 88% of global energy consumption. The combustion of fossil fuels at large scale causes air pollution; thus, shifting towards alternative, more environmentally solutions is of great interest. An alternative to conventional petroleum diesel should be environmentally benign, technically feasible, adequately available, and economically competitive at an affordable cost. Biodiesel, vegetable oils, biogas, and bio-alcohols, are substitute fuel options. Biodiesel is considered a supplementary fuel for diesel engines among these substitute fuels [1].

Biodiesel is made from plant seed oil during the transesterification of triglycerides to fatty acid alkyl esters by using short-chain alcohols such as methanol or ethanol. Biodiesel

has unique properties such as biodegradability, high flammability, low sulfur content, low toxicity, and high cetane number [2,3].

Biodiesel may be used in compression diesel engines with little to no modification due to its adjustable chemical and physical qualities. Compared to petroleum-based diesel fuel, biodiesel has a suitable combustion emission profile. It produces much less sulfur dioxide, carbon monoxide, and unburned hydrocarbons. It can be used as an alternate fuel supply to overcome the current energy demand. Biodiesel may be used in compression ignition (CI) engines with various proportions in combination or in pure form with diesel to provide a substitute solution for fuel. The drawbacks of biodiesel in comparison to diesel are high viscosity, less volatility, increased nitrogen oxides emission, high pour and cloud points, poor spray properties, and lower energy content. All biodiesel fuel problems have been resolved by changing feedstocks types, engine modifications, and additives [4].

The many sources of feedstocks, such as vegetable oils, waste oils, algal oils, microbial oils, and animal fats, can be used to produce biodiesel [5]. Total biodiesel production investment comprises 75% of feedstocks and catalyst selection. The selection of feedstocks depends on the country's economic aspect and biodiesel production availability. Biodiesel produced from edible oil competes with cooking oil. Hence, the focus has shifted to non-edible oils as feedstocks for biodiesel production [6,7].

Pongamia pinnata (local name: Sukh Chain) is resistant to drought and is a nitrogen-fixing and semi-deciduous leguminous tree. It is evergreen and grows with an average size of 15–20 m in height. It grows in Asia's tropical and temperate regions, including the Punjab and Sindh provinces of Pakistan, Sri Lanka, India, Bangladesh, Vietnam, Australia, Philippines, Thailand, Malaysia, Fiji, Florida, and Japan [8,9]. The single almond-sized seed is contained inside the thick-walled elliptical pod shell. Seeds can grow for 4–6 years and produce between 9–90 kg seeds. The yield potential is between 900–9000 kg/hectare. According to statistics, only 6% of the potential annual production (135,000 million tons) of *Pongamia pinnata* oil is being used [10]. A single seed produces 40% oil, and about 50% of this oil consists of C18:1, which is considered appropriate for making biodiesel. One tree produces 8–24 kg of kernels containing 30–40% oil. The air-dried kernels contain oil 27.5%, protein 17.4%, moisture 19%, crude fiber 7.3%, ash 2.3%, and starch 6.6% [9]. In addition, *Pongamia pinnata* trees produce oil in the range of 2–4 Mg/ha/year. Due to its rich availability and higher energy content (34–38.5 MJ/kg), *Pongamia pinnata* oil is appropriate for mass fuel production [11].

Different methods are used for biodiesel production, such as transesterification, pyrolysis, dilution, and micro-emulsification [12]. The transesterification process is the most adaptive and economical process for commercial biodiesel production with high yield. The production of biodiesel by transesterification involves two approaches. One is catalytic transesterification, and the other is the non-catalytic transesterification using supercritical methanol. In catalytic transesterification, either biological or chemical catalysts are used to activate the process. The chemical catalyst comprises heterogeneous agents (solid alkali or solid acid) and homogeneous agents (acid or alkali).

Homogeneous catalysts are associated with corrosion problems, a large amount of wastewater production, expensive raw materials, and environmental pollution. Additionally, filtration cannot recover these catalysts directly, thus increasing the production cost [13]. Heterogeneous catalysts are more convenient and effective catalysts for the production of biodiesel at a commercial scale [14]. A lesser amount of heterogeneous base catalyst is required during transesterification than other catalysts. Heterogeneous acid catalysts are more efficient for simultaneous transesterification and esterification processes [15]. However, heterogeneous catalysts also require centrifugation and filtration for recovery, which increases the cost of production, operating time, and energy consumption [13]. In this context, magnetic nanoparticles are attractive additives to the supports used for heterogeneous catalysts. The magnetic separation process increases reusability and avoids the loss of catalyst compared to centrifugation and filtration processes [16]. Due to these magnetic nanoparticles, the catalyst could be separated from the reaction mixture

more efficiently by using an external magnetic field, thereby eliminating the conventional filtration and centrifugation steps [17]. Magnetically separable nano-catalysts have been constructed by using magnetic nanoparticles as solid support. Magnetic nanoparticles have a high surface area to volume ratio; thus, they can carry active catalysts in large amounts on their surface [18].

The advantages of magnetic nano-catalysts include their insolubility in organic solvents, high reusability, less waste production, high stability, and appropriate for large-scale production. Additionally, the synthesis process of magnetic nano-catalysts is simple [13]. Because of these tremendous advantages, magnetic nano-catalysts can be chosen as a critical option for industrial biodiesel production, effectively reducing process costs and saving the environment. Before using them in industry, it is necessary to make sure that magnetic catalysts have a strong magnetic field. Magnetic nano-catalysts will be used soon in mass production and will be economically viable in the industry [19,20]. In earlier studies, numerous magnetic nano-catalysts have been utilized for biodiesel production, including: ZnO/BiFeO₃ [21], CaO/Fe₃O₄ [22], MgFe₂O₄@CaO [23], CaO/CuFe₂O₄ [24], Li/ZnO-Fe₃O₄ and Li/Fe₃O₄ [25], KF/CaO-Fe₃O₄ [26], MgO/MgFe₂O₄ [27], SO₄/Fe-Al-TiO₂ [28], Cs/Al/Fe₃O₄ [29], and Fe₃O₄/SiO₂ [30].

The novelty of this research work is the effective biodiesel production from *Pongamia pinnata* seeds as a cheap feedstock by using different reusable magnetic nano-catalysts of ICdO, ISnO, and their modified forms. An analysis has been done to check the morphology of the prepared magnetic nano-catalyst and the quality analysis of produced biodiesel through different quality parameters.

2. Results and Discussion

In the present research, two magnetic nano-catalysts, ICdO, and ISnO, were prepared. The development of magnetic catalysts used a modified co-precipitation method for the synthesis process. The prepared magnetic ICdO and ISnO nano-catalysts were changed into acidic and basic forms using HCl and NaOH, respectively. An equal quantity of prepared ICdO and ISnO (ICdO + ISnO) nano-catalysts was also mixed. These magnetic nano-catalysts were used to check the yield of biodiesel produced through a transesterification reaction.

2.1. Effect of Catalysts Concentration on Biodiesel Yield

The effect of different catalysts concentration on the biodiesel yield is represented in Figure 1. With increasing catalyst concentration, the yield of magnetically catalyzed biodiesel also increased to a limited extent. Magnetic nano-catalysts such as ICdO (Pure), ISnO (Pure), ICdO + ISnO, ICdO (Acidic), ICdO (Basic), ISnO (Acidic), and ISnO (Basic), were used in different concentrations i.e., 0.5, 1.0, 1.5, 2.0, and 2.5 g.

Conversion efficiency increases with the increase in catalysts amounting to up to 1 g of catalyst concentration. The maximum yield was obtained for 1g of the catalyst with the conversion efficiency of 90%, 99%, 85%, 94%, 75%, 90%, and 81% yield, for catalysts of ICdO (pure), ISnO (pure), ICdO + ISnO, ICdO (Acidic), ICdO (basic), ISnO (acidic), and ISnO (basic), respectively. The maximum conversion efficiency for base-catalyzed transesterification was achieved at 1g of ICdO (basic) and ISnO (basic) catalysts (Figure 1). During tests, a further increase in basic catalyst concentration results in the production of an emulsion. This emulsion increases the viscosity and prevents the glycerol separation, thus decreasing the methyl ester yield [31]. The quantity of acid catalysts used in the transesterification reaction also affects the reduction in acid value [32]. Biodiesel yield also increases with an increase in catalyst concentration up to 1 g of the ICdO (acidic) and ISnO (acidic) catalyst (Figure 1). There is no considerable improvement in biodiesel yield or further increase in the amounts of ICdO (acidic) and ISnO (acidic) catalysts.

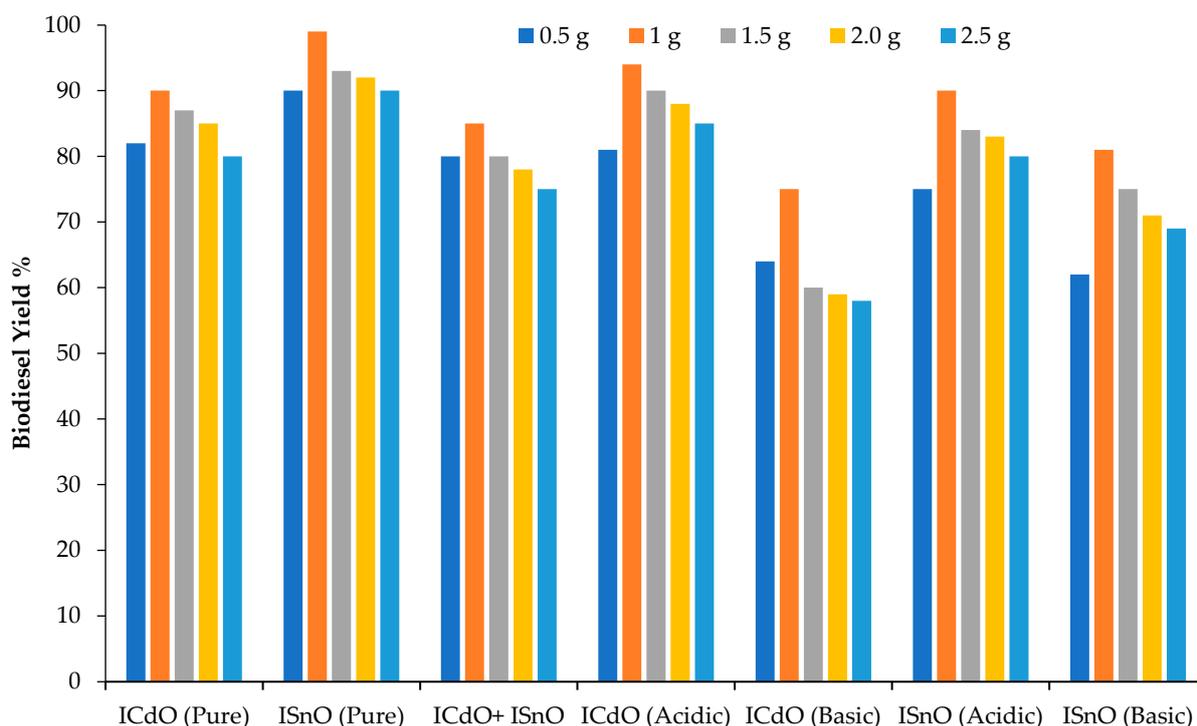


Figure 1. Effect of catalyst concentration on biodiesel yield (oil to methanol molar ratio, 1:10; temperature, 60 °C; time, 120 min).

2.2. Effect of Oil to Methanol Ratio

The ratio of oil-to-methanol is very critical in determining the extent of ester formation. The oil-to-methanol ratio is varied in order of 1:01, 1:05, 1:10, 1:15, and 1:20, to check the effect of its concentration on the percentage of produced biodiesel [33]. As shown in Figure 2, the yield of biodiesel increased as the methanol-to-oil ratio increased from 1:01 to 1:10. The maximum yield obtained at oil-to-methanol ratios of 1:10 with 91%, 99%, 82%, 91%, 80%, 84%, and 97%, for ICdO (pure), ISnO (pure), ICdO + ISnO, ICdO (acidic), ICdO (basic), ISnO (acidic), and ISnO (basic), respectively. However, a further increase in the molar ratio from 1:15 to 1:20 decreased biodiesel yield. When the oil-to-methanol ratio increases, the glycerol separation is difficult as the excess methanol delays the decantation by gravity, causing a decrease in biodiesel yield. The glycerol is still present in the biodiesel phase. According to Harreh et al. [31], the residual glycerol in the solution aids in shifting the equilibrium back to the left and lowers the biodiesel yield.

2.3. Effect of Reaction Time

The reaction mixture was stirred for a specific time and particular temperature to complete the transesterification reaction. It was observed that the yield of biodiesel increases with reaction time and gives maximum yield at 120 min with 91%, 97%, 90%, 91%, 80%, 85%, and 97%, for ICdO (pure), ISnO (pure), ICdO + ISnO, ICdO (acidic), ICdO (basic), ISnO (acidic), and ISnO (basic), respectively (Figure 3). The conversion of fatty acids into esters increases with reaction time (Figure 3). In the beginning, the reaction is slow due to the mixing and dispersion of the oil in alcohol, but afterward, the reaction takes place very quickly. After 2 h of reaction time, the yield remains constant. It indicated that 2 h is the optimum reaction time for the transesterification reaction [34].

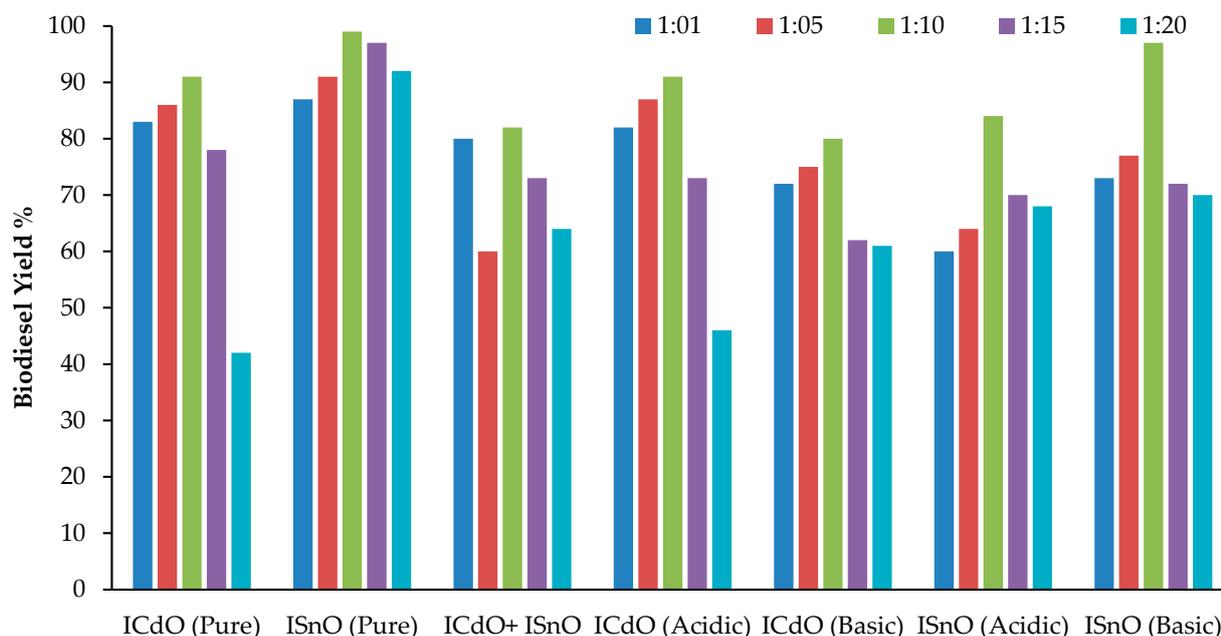


Figure 2. Effect of methanol concentration on biodiesel yield (catalysts concentration, 1 g; temperature, 60 °C; time, 120 min).

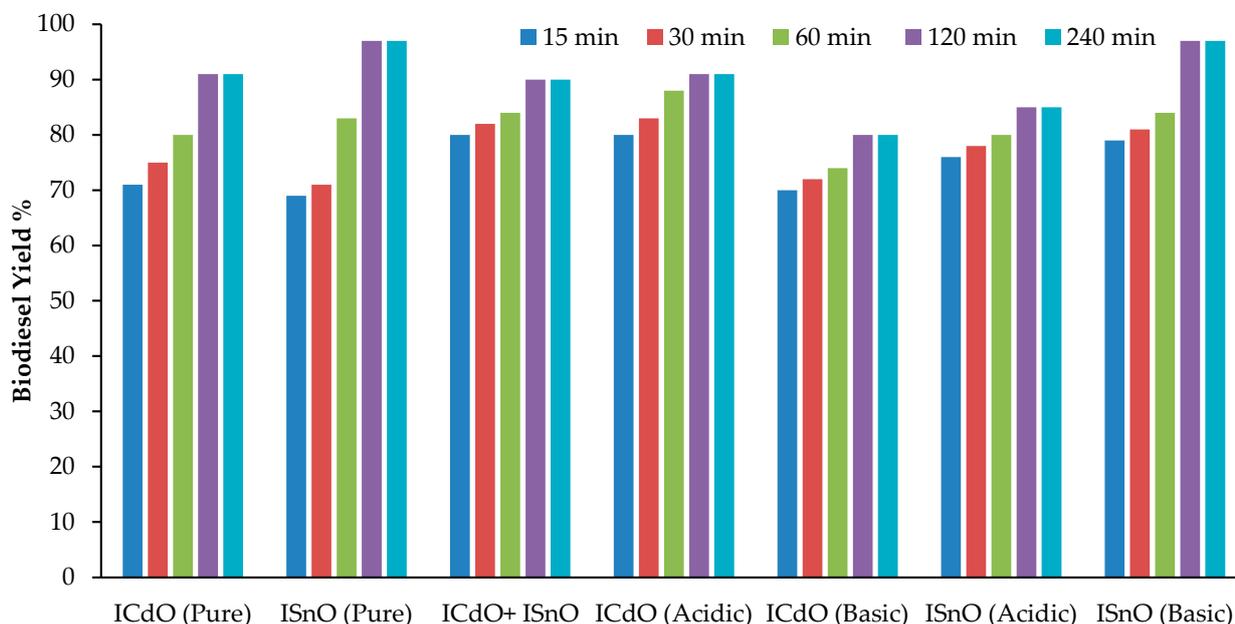


Figure 3. Effect of reaction time on biodiesel yield (catalysts concentration, 1 g; temperature, 60 °C; oil to methanol molar ratio, 1:10).

2.4. Effect of Reaction Temperature

An optimum temperature is required to obtain an optimum yield of biodiesel. Reaction temperatures of 30 °C, 40 °C, 50 °C, 60 °C, and 70 °C, were chosen to find an optimum temperature for biodiesel production. Several biodiesel samples were prepared at different temperatures, and the produced biodiesel yield was recorded. A maximum biodiesel yield was obtained at 60 °C (near the evaporating temperature of methanol) with 91%, 98%, 90%, 91%, 81%, 86%, and 90% yield, of ICdO (pure), ISnO (pure), ICdO + ISnO, ICdO (acidic), ICdO (basic), ISnO (acidic), and ISnO (basic), respectively (Figure 4). The reaction

rate increases with an increase in reaction temperature to a limited extent. According to Bello et al. [35], the optimum temperature (60 °C) for biodiesel production was near the evaporating temperature of methanol (64 °C). A further increase in temperature decreases the biodiesel yield due to the evaporation of methanol. The reaction temperature influences biodiesel yield and reaction rate. According to research, a further rise in temperature will negatively affect the conversion process [36].

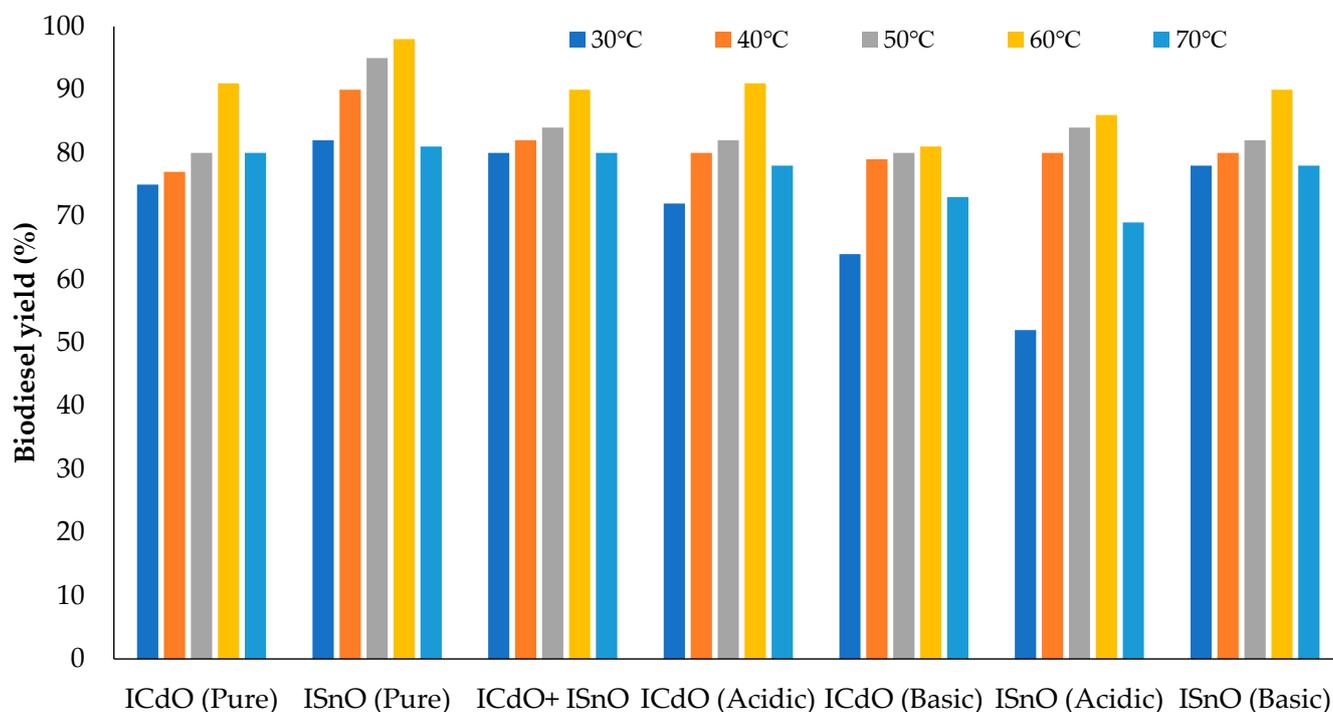


Figure 4. Effect of reaction temperature on biodiesel yield (catalysts concentration, 1 g; oil to methanol molar ratio, 1:10; time, 120 min).

2.5. SEM Analysis of Catalysts

SEM analysis was used to evaluate the synthesized magnetic nano-catalysts morphology. The SEM images of ISnO catalyst and ICdO catalysts are shown in Figures 5 and 6, respectively. SEM images of ISnO catalysts and ICdO catalysts were used to differentiate their morphological and surface properties. From Figure 5, it can be seen that different SEM images of ISnO were taken at different magnifications. The morphology of ISnO catalysts observed was rod-like shape along with some irregular shapes. The surface was observed to be rough. However, the degree of the surface roughness of ISnO catalysts was increased after treatment with the acid (Figure 5b); in contrast the treatment of ISnO with base, the surface became very smooth (Figure 5c).

From Figure 6, it can be seen that different SEM images of ICdO were taken at different magnifications. The morphology of ICdO (pure) catalysts observed was the rod-like structure along with soft macroscopic separations. However, in treatment with acid and base, the morphology of these catalysts was changed to a round or irregular shape. However, Figure 7 represented the SEM images of magnetic nano by mixing equal quantities of ICdO and ISnO at 200 nm and 5 µm. All these prepared catalysts showed different catalytic activities toward biodiesel production.

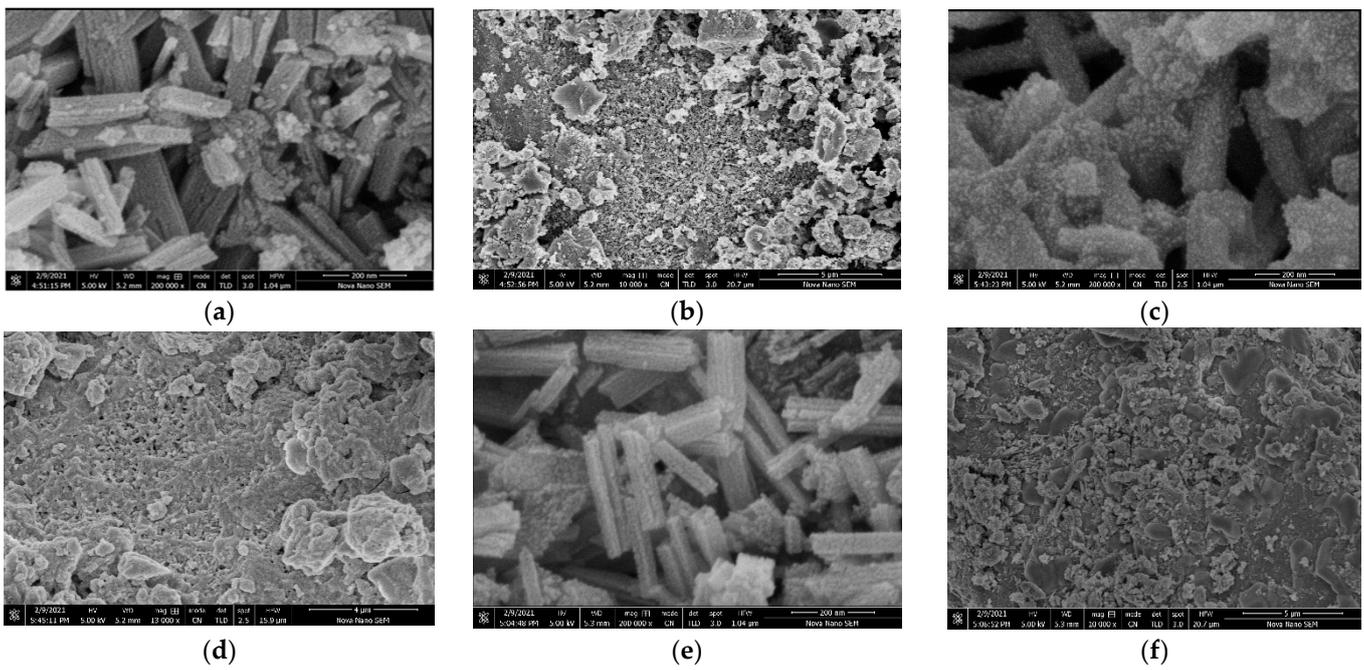


Figure 5. SEM images of prepared nano-catalysts of ISnO at different magnification. (a) SEM image of pure ISnO taken at 200 nm, (b) SEM image pure ISnO taken at 5 μm, (c) SEM image taken acidic ISnO at 200 nm, (d) SEM image of acidic ISnO taken at 5 μm, (e) SEM image of basic ISnO taken at 200 nm and (f) SEM image of basic ISnO taken at 5 μm.

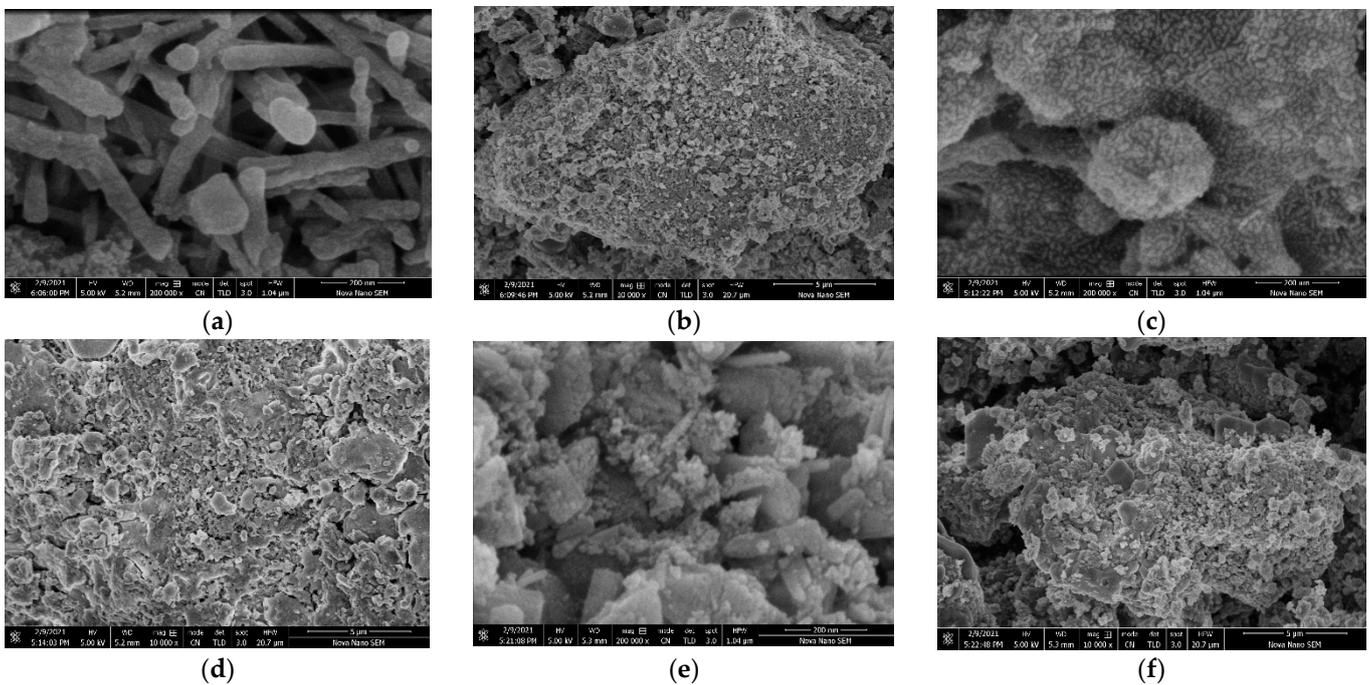


Figure 6. SEM images of prepared nano-catalysts of ICdO at different magnification. (a) SEM image of pure ICdO taken at 200 nm, (b) SEM image pure ICdO taken at 5 μm, (c) SEM image taken acidic ICdO at 200 nm, (d) SEM image of acidic ICdO taken at 5 μm, (e) SEM image of basic ICdO taken at 200 nm and (f) SEM image of basic ICdO taken at 5 μm.

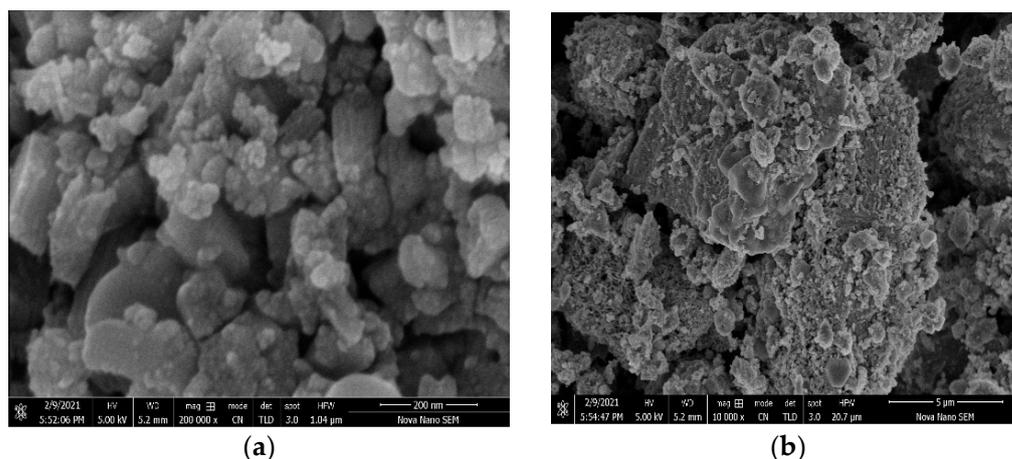


Figure 7. SEM images of prepared nano-catalysts of ICdO + ISnO. (a) SEM image of ICdO + ISnO taken at 200 nm, (b) SEM image ICdO + ISnO taken at 5 μm .

2.6. Recyclability and Leaching of the Catalysts

In order to examine the recyclability of the magnetic nano-catalysts after transesterification, the nano-catalysts were recovered by the use of a magnet. After recovery, nano-catalysts can be reintroduced in the reaction five times without any modification [33]. The leaching of sodium metal (Na^{2+}) into the biodiesel phase at optimized conditions were determined by flame photometry. The ISnO (basic) nano-catalyst sample was reused five times and the leaching test was conducted after each run, and the amount of Na^{2+} ion metal concentration was reduced from 2.05 mg/L to 0.96 mg/L. It was observed that after many reactions cycle of ISnO (basic) nano-catalyst, there was leaching in the biodiesel phase. It is suggested that the catalyst be incorporated with the more stable and inert materials, which can be an ideal way to enhance the stability of the catalyst.

2.7. Gas Chromatographic-Mass Spectrometric (GC-MS) Analysis of *Pongamia pinnata* Seed Oil

GC-MS analysis was performed for the quantification of fatty acids present in the *Pongamia pinnata* seed oil. From Table 1, it was found that *Pongamia pinnata* seed oil consists of major components of oleic acid (54.16%), linoleic acid (12.21%), palmitic acid (11.11%), and eicosanoic acid (8.23%), while behenic acid (3.46%), lignoceric acid (3.22%), linolenic acid (3.14%), and stearic acid (2.66%), were present in smaller amounts. Myristic acid (0.68%), arachidic acid (0.54%), lauric acid (0.23%), capric acid (0.14%), margaric acid (0.09%), ricinoleic acid (0.04%), and erucic acid (0.02%), were present in trace amount. According to the previous study, biodiesel with major fatty acids containing more than 15 carbon atoms is of higher quality. Fatty acids with more than 15 carbon atoms were found in higher quantities in *Pongamia pinnata* seeds oil.

2.8. Assessment of Fuel Quality Parameters

The structural features of any fatty esters present in biodiesel determine its physical properties. The biodiesel should have physical properties in the range of prescribed values by EN (14214) and ASTM (D6751); that is why it is necessary to identify the contents present in biodiesel in order to obtain the overall properties of produced fuel [37]. The current research found that all the fuel quality parameters of produced biodiesel are per the ASTM standards, as shown in Table 2. Acid values determined for *Pongamia pinnata* biodiesel were less than 0.8 mg KOH/g. The lower acid value exhibited the presence of less quantity of free fatty acids in the oil. The saponification value of *Pongamia pinnata* seeds biodiesel produced from different magnetic nano-catalysts ranged from 185 to 195 mg/KOH/g. The saponification value represented the short chain of alkyl groups in fatty acids in biodiesel. The higher the cetane number, the better the fuel's ignition quality. Cetane's number of produced biodiesels is greater than 45. The degree of unsaturation present in the alkyl

group attached to the methyl esters is exhibited by the iodine value. Iodine values for *Pongamia pinnata* seed biodiesel were greater than 86.5.

Table 1. Fatty acid profile of *Pongamia pinnata* seeds oil.

Sr. No.	Fatty Acid	Molecular Formula	Percentage Composition
1	Oleic acid	C ₁₈ H ₃₄ O ₂	54.16
2	Linoleic acid	C ₁₈ H ₃₂ O ₂	12.21
3	Palmitic acid	C ₁₆ H ₃₂ O ₂	11.11
4	Eicosanoic acid	C ₂₀ H ₄₀ O ₂	8.23
5	Behenic acid	C ₂₂ H ₄₄ O ₂	3.46
6	Lignoceric acid	C ₂₄ H ₄₈ O ₂	3.22
7	Linolenic acid	C ₁₈ H ₃₀ O ₂	3.14
8	Stearic acid	C ₁₈ H ₃₆ O ₂	2.66
9	Myristic acid	C ₁₄ H ₂₈ O ₂	0.68
10	Arachidic acid	C ₂₀ H ₄₀ O ₂	0.54
11	Lauric acid	C ₁₂ H ₂₄ O ₂	0.23
12	Capric acid	C ₁₀ H ₂₀ O ₂	0.14
13	Margaric acid	C ₁₇ H ₃₄ O ₂	0.09
14	Ricinoleic acid	C ₁₈ H ₃₄ O ₃	0.04
15	Erucic acid	C ₂₂ H ₄₂ O ₂	0.02

Table 2. Quality parameters of biodiesel samples at varied reaction conditions.

Type of Catalyst	Catalysts Conc. (Grams)	Oil:Methanol	Temperature (°C)	Time (h)	Shaking Speed (Rpm)	Saponification Value (mgKOH g ⁻¹)	Cetane Number	Iodine Value	Acid Value (mg KOH g ⁻¹)
ICdO (Pure)	0.25	1:15	30	2	100	189.2	48	109.5	0.23
ISnO (Pure)	0.25	1:10	30	2	100	193.4	47	111.8	0.20
ICdO + ISnO	0.25	1:01	30	2	100	187.6	49	118.9	0.31
ICdO (Acidic)	0.25	1:15	30	2	100	187.4	49	107.6	0.17
ISnO (Acidic)	0.25	1:01	30	2	100	187.8	47	115.9	0.19
ICdO (Basic)	0.25	1:01	30	2	100	182.3	49	117.8	0.41
ISnO (Basic)	0.25	1:15	30	2	100	182.9	46	119.8	0.29

3. Materials and Methods

3.1. Materials

Non-edible seeds of *Pongamia pinnata* were collected from different locations in Faisalabad, Pakistan. Cadmium chloride (CdCl₂), stannic chloride (SnCl₂), ferric chloride (FeCl₃), nitric acid (HNO₃), deionized water, methyl orange, phenolphthalein indicator, HCl, NaOH, methanol, sodium sulfate (anhydrous), and Wijs solution, were used in the present study and purchased from Merck, Pakistan.

3.2. Extraction of Oil

The selected healthy *Pongamia pinnata* seeds were dried under the sun, crushed, and ground to a uniform particle size of 1 mm by a heavy-duty grinder available in the general physical laboratory of the department [38]. Oil was extracted from the grounded seeds using a screw press machine [39]. Later, oil was filtered through vacuum filtration to save time under the pressure of 700 mmHg.

3.3. Preparation of Cadmium and Tin Based Magnetic Nano-Catalysts

In the present research, two magnetic nano-catalysts, ICdO, and ISnO, were prepared. The catalysts were synthesized through a modified method of co-precipitation using aqueous solutions of SnCl₂ and CdCl₂ with FeCl₃ in an alkaline medium as described in the literature [40]. A solution consists of a metal ratio of 2:1 v/v of FeCl₃ (0.5 mol L⁻¹) with SnCl₂ (0.5 mol L⁻¹) and CdCl₂ (0.5 mol L⁻¹) was prepared. Then, this solution was quickly added to a 1 L aqueous solution of NaOH (2 mol L⁻¹) at 100 °C temperature, under high

stirring conditions, and kept at this condition for two 2 h. Then, the dark precipitates of nano-catalysts were obtained. The dark precipitates were washed with distilled water to separate the excess ions. The sediments were treated with HNO_3 (1 mol L^{-1}) and then washed with distilled water. The dark precipitates were filtered using Whatman filter paper No. 1. The filter paper trapped the solid residues, and the liquid was drawn through the filter paper into the flask below. The sample was then dried in an oven at $100 \text{ }^\circ\text{C}$ for 2 h to eliminate the excess moisture from the residues. The solids nano-particles were then activated at $300 \text{ }^\circ\text{C}$ for 4 h [33]. The prepared nano-catalysts were ground to uniform particle size using a pestle and mortar available in the analytical laboratory of the department.

3.4. Modification of Nano-Catalysts

The prepared magnetic ICdO and ISnO nano-catalysts were modified into acidic and basic forms using HCl and NaOH. Acids-modified catalysts exhibit higher tolerance to water and free fatty acids in oils. Acids-modified catalysts can catalyze both the esterification and transesterification reactions at the same time. Base-modified catalysts speed up the chemical reaction, and the reaction can be performed at room temperature. An equal quantity of prepared ICdO and ISnO nano-catalysts were also mixed.

3.5. Transesterification

The oil extracted from *Pongamia pinnata* seeds was transformed into biodiesel and glycerol using methanol through catalytic transesterification. The glycerol is produced in a transesterification reaction as a by-product [41]. Magnetic iron cadmium oxide, iron tin oxide nano-catalysts, and modified form of these catalysts, were used for biodiesel production. Furthermore, reaction conditions such as the methanol-to-oil ratio, temperature, catalyst concentration, and reaction time, were optimized [38]. After biodiesel production, the catalysts used were separated using an external magnet. The prepared biodiesel underwent washing with hot water, and the glycerol layer was removed [42]. The sodium sulfate was added to the final product (biodiesel) to remove the moisture.

3.6. Surface Morphology of Catalysts

The surface morphology, shape, and behavior, of the synthesized magnetic nano-catalysts, ICdO (pure), ISnO (pure), ICdO (acidic), ISnO (acidic), ICdO (basic), ISnO (basic), and the mixture of equal quantity of ICdO and ISnO, were evaluated by using SEM (scanning electron microscopy) (Nova NanoSEM). However, the seed oil composition was determined by GCMS analysis.

3.7. Measurement of Physiochemical Properties of Biodiesel

3.7.1. Determination of Iodine Value

The iodine value was determined by taking a mixture of the sample (0.1 g), CCl_4 (20 mL), and Wijs solution (25 mL) in a glass stoppered iodine flask having a capacity of 250 mL. The reaction mixture was shaken vigorously continuously period and kept in the dark for about 0.5 h. After that, distilled water (100 mL) and 15% KI (20 mL) were added to this solution and potentially titrated against $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ having normality of 0.1 using an effective starch indicator until the yellow color of the reaction vanished. The entire procedure was repeated in the same manner for a blank [43]. The iodine value was calculated by using the following equation:

$$\text{Iodine Value} = \frac{(\text{BT} - \text{ST}) \times \text{normality of } \text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O} \times 12.69}{\text{weight of sample in grams}}$$

where BT—value of blank, ST—value of sample titration

3.7.2. Determination of Saponification Value

A solution of 20 mL KOH (alcoholic) and oil (0.5 g) was mixed in a round bottom flask for saponification value determination. The whole mixture was refluxed until it became

clear. The appearance of a clear solution indicated the endpoint of the saponification reaction. After adding a few drops of phenolphthalein, the components of the mixture were cooled to room temperature. The mixture was titrated against 0.5 N HCl until the pink color vanished. The same procedure was used for blank. The saponification value (mg/sample in grams) was calculated by the following equation [44]:

$$\text{Saponification Value} = \frac{(B - S) \times N \times 56.1}{W}$$

where B—volume (mL) of titrant used for blank, S—volume (mL) of titrant used for sample, N—HCl normality of (mmol/mL), and 56.1—molecular wt. of KOH (mg/mmol), W—sample wt. (g).

3.7.3. Determination of Free Fatty Acid Contents and Acid Value

The acid value of selected samples of biodiesel was calculated by taking 0.5 g oil and 10 mL ethanol in a conical flask having a capacity of 250 mL. After adding of 1–2 drops of phenolphthalein indicator, this reaction mixture was titrated against a standard of sodium hydroxide with normality of 0.1 [45]. The emergence of light pink color is representative of the endpoint after the reaction. Free fatty acid percentage is shown as oleic acid was preferably determined by using following form the equation [30]:

$$\% \text{FFA} = \frac{V \times N \times 28.2}{w} \quad (1)$$

where N—NaOH Normality (mol/1000 mL), V—NaOH volume (mL), W—sample mass (g), 282—oleic acid molecular weight (g/mol).

3.7.4. Determination of Cetene Number

The cetane number of biodiesels measures the ignition value of diesel in a complex mixture of liquid methylnaphthalene and represents the percentage of cetane by volume. The cetane number of all prepared biodiesel samples was calculated using the use of the following formula to find the ignition value as shown in equation [46]:

$$\text{CN} = \frac{46.3 + 5458}{SV} - 0.225 \times IV \quad (2)$$

where CN—cetane number, SV—saponification value, IV—iodine value.

3.8. Stability and Durability Test

The ISnO (basic) nano-catalyst was examined repeatedly under the optimized reaction conditions of 1 g of catalyst loading, 60 °C reaction temperature, 1:10 oil-to-methanol ratio and 2 h reaction time. The Na²⁺ leaching of catalyst was assessed from the final product (biodiesel) by flame photometer.

4. Conclusions

Being an agricultural waste, *Pongamia pinnata* seeds are a good source of biodiesel in the twenty-first century, when the depletion of petroleum-based fuel and environmental issues are major threats to the advancement and maintenance of human living standards. In the separation process, magnetic catalysts are more effective than non-magnetic catalysts. Different reusable magnetic nano-catalysts of ICdO, ISnO, and their modified forms, were synthesized through the coprecipitation method. These catalysts were used for biodiesel production through the transesterification process. The nano-catalysts were magnetically recovered and reused 4–5 times without any change in their activity. The magnetic separation process increases reusability and avoids the loss of catalyst compared to centrifugation and filtration processes. The effect of different process parameters was studied. Maximum yield was obtained for ISnO (pure) catalyst with the conversion efficiency of 99% after 2 h

of reaction time at 60 °C. SEM analysis was performed to check the morphology of different prepared magnetic nano-catalysts. *Pongamia pinnata* seeds oil contains low-chain fatty acids making it ideal for biodiesel production. The fatty acid (%) detected in *Pongamia pinnata* seeds oil biodiesel was oleic acid (54.16%), linoleic acid (12.21%), palmitic acid (11.11%), and eicosanoic acid (8.23%). The properties of biodiesel, such as saponification value, cetane number, iodine value, and acid value, were measured and compared as per the ASTM specification. The information provided by this study clarifies the use of *Pongamia pinnata* seeds as a novel, less expensive feedstock to produce biodiesel with better fuel properties.

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