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Utilization of zinc doped biochar catalyst for biodiesel production from waste cooking oil: process optimization and characterization

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ABSTRACT

Biodiesel, an eco-friendly and sustainable alternative to diesel, has gained increasing interest over the past few years. The reuse of waste cooking oil as a feedstock for biodiesel production is affordable and environmentally safe. This study aimed to understand the understudied benefits of using zinc-doped biochar as a catalyst for biodiesel production. The zinc-doped biochar catalyst was characterized using techniques such as scanning electron microscope with energy-dispersive X-ray (SEM-EDAX) analysis and Fourier transform infrared (FTIR) analysis, which revealed the morphological and functional characteristics of the catalyst. The optimum process conditions were found to be catalyst concentration of 3.92% (w/w), methanol-to-oil molar ratio of 16:1, reaction temperature of 65 °C, and reaction duration of 66.5 min; these conditions yielded 94.1% biodiesel using a response surface optimization technique. The produced biodiesel was analyzed using gas chromatography-mass spectrometry, and the presence of methyl esters was confirmed. The use of waste cooking oil and biochar catalyst for biodiesel production is an economical and environmentally sustainable process.

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KEYWORDS

Biodiesel; waste cooking oil; biochar; heterogeneous catalyst; process optimization; response surface methodology

Introduction

Petroleum and fossil fuels, commonly used fuel sources, are non-renewable and are rapidly being depleted. Also, they have been mainly responsible for environmental issues such as the emission of greenhouse gases, increasing global warming and ocean acidification. The US consumed 79% of its primary energy through fossil fuels in 2021 [1]. The transport sector is a major consumer of fossil fuels, and its crucial fossil fuel is crude oil, which when processed becomes gasoline, diesel fuel, heating oil, etc. [2]. This must change in the coming years as fossil fuels are rapidly declining and also causing irreparable environmental damage. Carbon dioxide, nitrous oxides, and hydrofluorocarbons are a few examples of greenhouse gases emitted. In 2021, fossil fuel consumption accounted for 37.12 billion tonnes of global carbon dioxide emissions [3].

Due to the above-discussed disadvantages, there is a global need for a rapid shift from fossil fuels to cleaner and greener technologies. Biofuels derived from biomass/natural feedstock have gained interest as alternatives to fuel sources. Biofuels are available in gaseous form (hydrogen and propane) as well as liquid form (ethanol, methanol, butanol, biodiesel, vegetable oils and waste oils). They are environmentally safe, derived from natural sources, and economical in comparison to the ever-rising transportation fuel rates. Among these biofuels, ethanol and biodiesel are the most commonly produced.

Biodiesel is biodegradable and renewable and does not harm the environment due to its low carbon footprint and low greenhouse gas emissions [4], making it a promising alternative fuel [5,6]. The physicochemical properties of

biodiesel such as high flash point, low sulfur level, biodegradability, and good intrinsic lubrication make it a desirable alternative to diesel [7]. Chemically, biodiesel is mono alkyl fatty acid ester or methyl ethyl ester, and it is produced through a process known as transesterification which uses chemical or enzyme catalysts for the reaction [8].

Biodiesel can be extracted from different feedstocks [4]. Four generations of feedstocks are proposed for biodiesel production. The first generation of feedstocks is edible crops, the use of which creates competition with the food industry and makes biodiesel production less profitable [9]. The second-generation feedstock consists of waste and non-edible sources. Some examples are biomass of non-edible plants and their oil, waste cooking oil (WCO) and waste animal fats [10]. This feedstock does not affect the food industry and is also more cost-effective but biodiesel produced from non-edible crops has been reported to have poor flow properties [4]. The fourth generation of feedstock involves genetically modified algae, electro-fuels, and photobiological solar fuels, and these are considered emerging research areas that require further investigation [11]. Biofuel feedstocks are heavily scrutinized due to their ability to either compete with food sources or take over large arable areas for non-edible fuel crop production, eventually impacting food prices [9].

The above-discussed drawbacks can be overcome by using WCO as a biodiesel feedstock. WCO does not impact the food economy and does not overrun food croplands as it uses edible oil which is considered a waste product, contributing to a circular economy [12]. It can be collected

from households and restaurants, making it a cost-effective feedstock. Reusing WCO prevents the dumping of oil into the environment and thus reduces pollution [10]. Biodiesel from WCO also has a higher oxygen content and lower emissions of carbon monoxide (CO), nitrogen oxides (NO), hydrocarbon (HC), and particulate matter (PM), and smoke compared to diesel fuels thus making it a preferable and cleaner fuel alternative. Biodiesel or biodiesel blends with diesel fuel produced from WCO can be used in compression ignition (CI) engines [13].

The production of biodiesel from any feedstock needs a suitable catalyst. There are many types of catalysts: homogeneous (acid/base), heterogeneous (acid/base), enzymatic, biomass-based, and bifunctional catalysts [14]. The disadvantages of using base homogeneous catalysts are the high cost and their tendency to produce a soap with the free fatty acids (FFAs) present during the process. Acid catalysts are corrosive and difficult to store, making them unsuitable for industrial use [15]. Heterogeneous catalysts form visible physical phases with reactants that can be easily separated, unlike homogeneous catalyst that results in a uniform undifferentiated mixture [16]. However, the reaction rate with a heterogeneous catalyst is much lower compared to using a homogeneous catalyst. Heterogeneous catalysts are reusable, non-corrosive, and less susceptible to the saponification process [6,17]. Despite being advantageous, heterogeneous catalysts can cause environmental damage. Enzyme catalysts can be used only around 35 to 40 °C and have disadvantages such as deactivation, high cost and slower reaction rates, making them unsuitable for the large-scale production of biodiesel [18].

These disadvantages can be overcome by using a sustainable carbon-based catalyst such as biochar, which also has a higher surface area and hence yields more biodiesel [19,20]. Biochar is a stable carbon product produced from biomass feedstock. The production of biochar from biomass is done through pyrolysis, a thermochemical process that works by decomposing biomass in an oxygen-free or oxygen-restricted environment [21]. Biochar product as the solid component is formed by slow pyrolysis at a low heating rate such as 1–100 °C/min and for a long duration [22]. Biochar parameters such as surface area, porosity, and active catalytic sites determine the quality and usage of biochar as a catalyst [23,24]. Researchers have been studying modifications to biomass types, pyrolysis parameters, and additional treatments to engineer highly beneficial biochar [25]. Biochar catalysts can be produced from different precursors and are treated with different parameters for the varying precursors. The properties of biochar also vary depending on the source of catalyst precursors. Chicken manure used as a catalyst for biodiesel production from WCO gave a yield of 95% compared to biochar synthesized from oat hull which yielded only 75%. Pre-/post-treatment has been shown to improve the properties of biochar. For example, one of the earliest studies, by Dehkhoda et al. [26], demonstrated that biochar acquired high acid density and surface area due to pretreatment with sulfuric acid for an elongated period. This resulted in a higher yield of biodiesel [26]. Reusability is also a preferable characteristic of biochar; therefore, biochar catalysts that can be separated from the biodiesel produced and have high reactivity for longer cycles are preferred [27]. Activating biochar with

potassium hydroxide increases its surface area and is also a preferred treatment method. Researchers achieved 84% reusability of biochar on the 10th cycle and 98.2% biodiesel yield using potassium hydroxide-treated biochar from pig meat and bone meal catalyst precursor [17].

Recently biochar-based catalysts were used for biodiesel production and produced effective results. The novelty of this study was to develop a zinc-doped biochar catalyst for efficient biodiesel production, and to optimize the process parameters using response surface methodology (RSM). This study will help to develop various combinations of biochar catalysts in biodiesel production. This study examines the understudied usage of zinc-doped biochar as a catalyst for biodiesel synthesis from WCO. The synthesized catalyst and biodiesel were characterized. The experimental process parameters of biodiesel were also investigated using RSM and then characterized using gas chromatography-mass spectroscopy (GC-MS).

Materials and methods

Sample collection and preparation

WCO was collected from cafeterias and cafes in Chennai, Tamil Nadu, India. The WCO was allowed to settle for days and was filtered to remove any food/inorganic substances. The oil was pretreated at 90 °C for 1 h for water removal. This study used pure chemicals such as ammonia, H₂O₂, zinc nitrate, and methanol purchased from Chemi Laboratory Pvt. Ltd., Mumbai. The study was aided by using 99% pure chemicals.

Catalyst synthesis and characterization

Biochar synthesis

The wood waste was collected and chopped into small pieces, washed with water to remove the impurities, and dehydrated in an oven at 60 °C. The dried chips of wood were heated at 300 °C to produce biochar. Solutions A and B were prepared by dissolving 0.5 g of biochar and 7.14 g of zinc nitrate in 50 mL of water. Solutions A and B were mixed in a stirrer followed by the addition of 8% ammonia solution (v/v). Using a 0.4 M addition of Na₂CO₃, the above solution was precipitated after two hours of continuous stirring. The precipitated mixtures were divided and dried at 80 °C for 3 h, followed by calcination at 600 °C [28].

Catalyst characterization

The chemical properties, morphological structure, and size of the synthesized catalyst were characterized using various techniques. We examined the surface topography of the Zn-doped biochar using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDAX) analysis. The activated and functionalized sample was analyzed using Fourier transform infrared spectroscopy (FTIR), which displayed the functional groups.

Biodiesel production and process optimization

Transesterification process

The different concentrations of Zn-doped biochar catalyst were combined at different temperatures and times with varied methanol-to-oil (M:O) molar ratios. This mixture was then

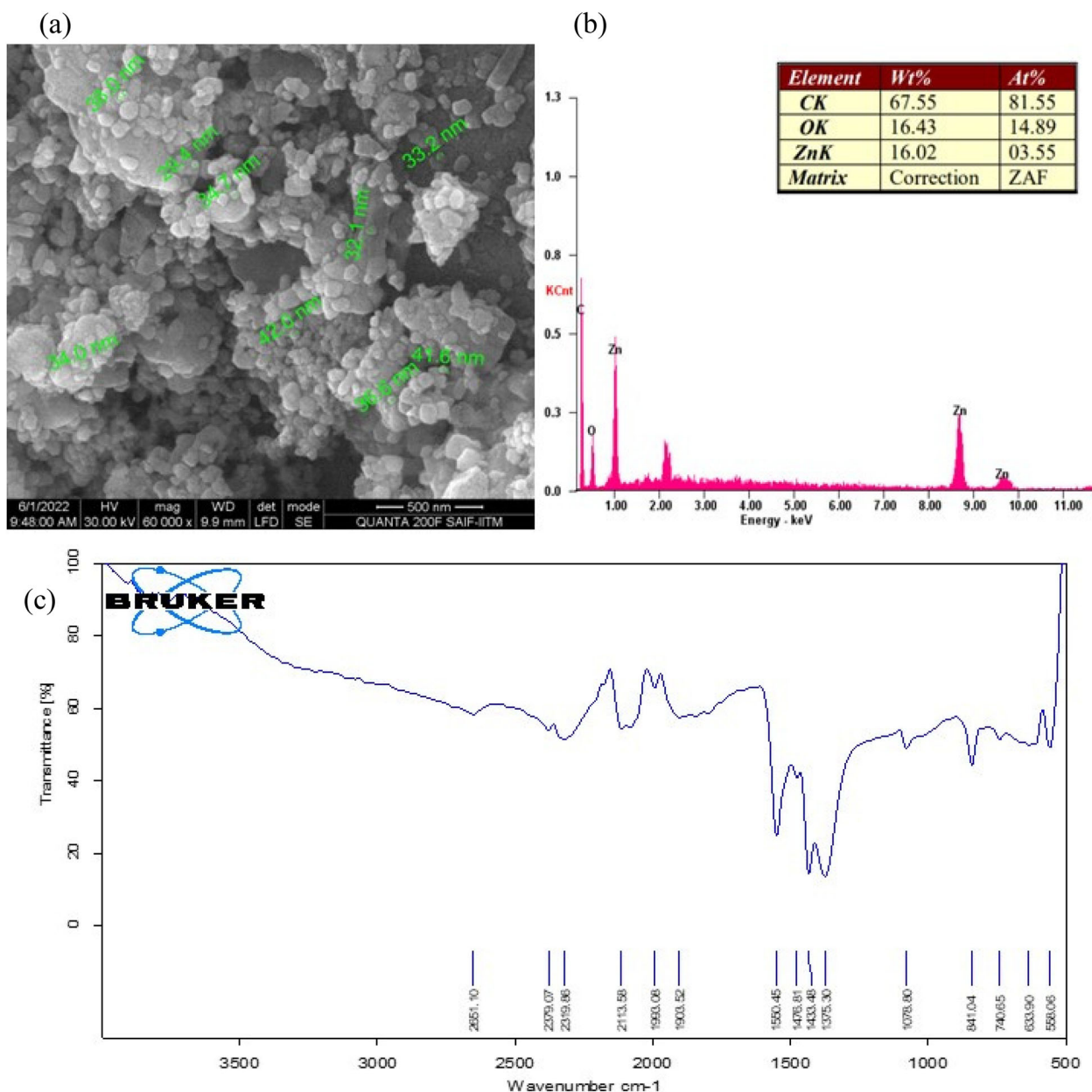


Figure 1. (a) Scanning electron microscope analysis, (b) Energy-dispersive X-ray analysis, and (c) FTIR analysis of zinc-doped biochar catalyst.

subjected to a separation process where biodiesel is separated into the top layer. This supernatant was collected carefully and the denser bottom layer containing the glycerol and catalyst was discarded. Some impurities suspended in the biodiesel were removed in the washing process. The unreacted methanol in the collected biodiesel was removed by heating it at 65 °C, above the boiling point of methanol. The excess water content in the final product can be evaporated by heating it at 110 °C for 10 min.

The biodiesel conversion calculation formula (Equation 1) is as follows:

$$\text{Biodiesel Conversion (\%)} = \frac{\text{Volume of converted biodiesel}}{\text{Volume of oil used}} \times 100 \quad (1)$$

Process optimization

For process optimization, major parameters such as catalyst concentration (0.5–4 wt%), M:O ratio (5:1–30:1), time (20–70 min) and temperature (40–90 °C) were selected. Each experiment was repeated three times, and the mean value was used

for further study. Based on the experimental results, the parameters were again investigated using RSM. The process parameters can be optimized using RSM to find more accurate results. Minitab-19 software tools were employed for this purpose. Using central composite design (CCD), the optimization of four factors – catalyst concentration (A), M:O molar ratio (B), time (C) and temperature (D) – was carried out. The CCD model containing 30 runs was increased at the center point to assess pure error. Minitab-19 was utilized to drive the quadratic polynomial model. The quadratic equation is shown in Equation (2). The fitted model is supported by the R^2 value (coefficient of determination).

$$Y = A_0 + AX_1 + BX_2 + CX_3 + DX_4 + EX_1X_2 + EX_1X_3 + EX_1X_4 + EX_2X_3 + EX_2X_4 + EX_3X_4 + GX_1X_2X_3 + GX_1X_2X_4 + GX_1X_3X_4 + GX_2X_3X_4 + IX_1^2 + IX_2^2 + IX_3^2 + IX_4^2 + LX_1 + LX_2 + LX_3 + LX_4 \quad (2)$$

Y = response; A_0 = intercept term; A, B, C, D = linear coefficients; E, F, G, H, I, J = interactive coefficients; and X_1, X_2, X_3, X_4 = code variables [29].

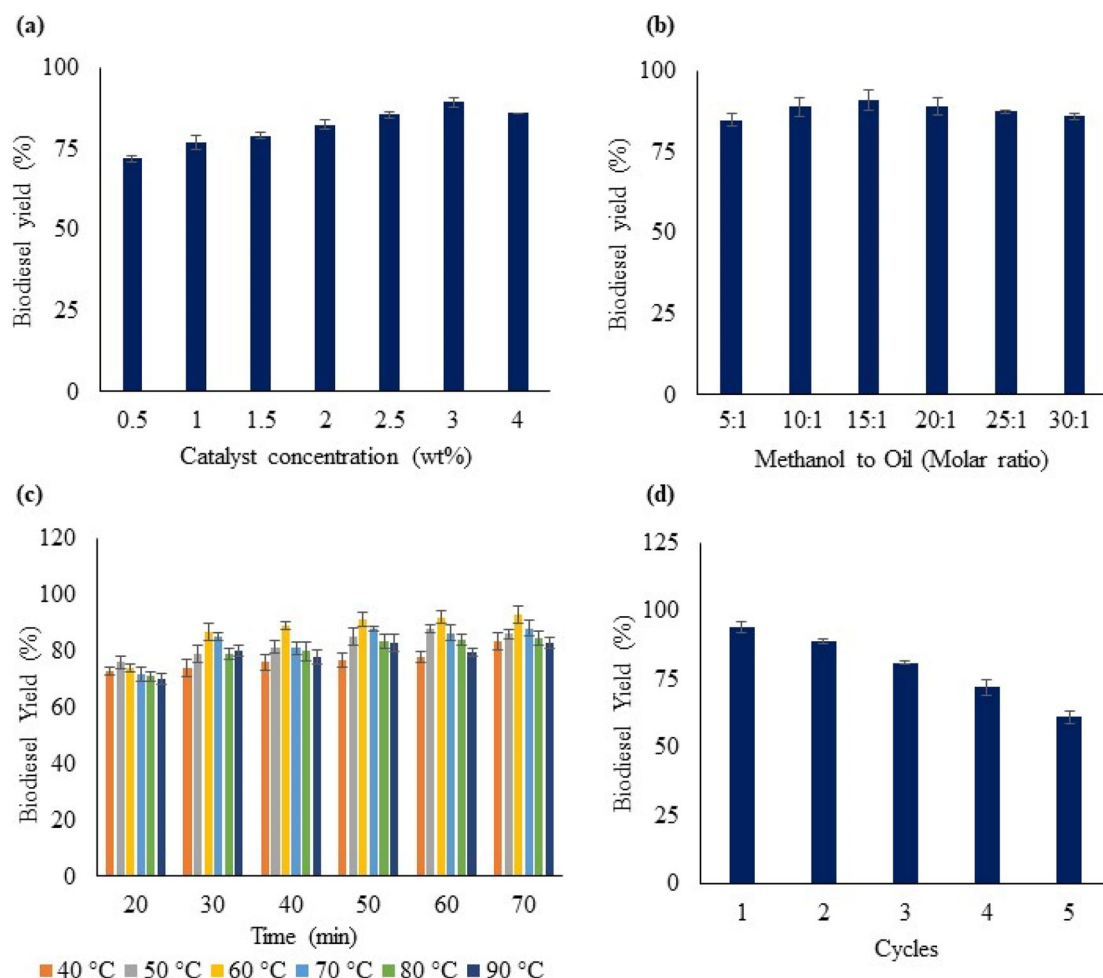


Figure 2. (a) Effect of catalyst concentration; (b) effect of methanol-to-oil molar ratio; (c) effect of temperature and time; (d) catalyst reusability.

Biodiesel characterization

GC-MS was used to analyze fatty acid methyl esters (FAMES) which are the primary molecules found in biodiesel. The quality of biodiesel is affected by various parameters such as the type and quality of the feedstock, production techniques, lipid composition and the purification process. The results from the analysis of the physiochemical properties of the biodiesel produced should fit the American society for testing and materials (ASTM) and European (EN) standards.

Results and discussion

Characterization of Zn-doped CaO catalyst

Characterization of Zn-doped CaO catalyst by SEM-EDAX

The Zn-doped biochar catalyst underwent analysis via SEM-EDAX. The irregular surface of the catalyst was observed in the SEM analysis, as shown in Figure 1(a). The size of the biochar was found to be 29.4–42 nm. The image demonstrated cubic structures with pointed edges and indicates a large surface area of the biochar. The presence of Zn on the surface of the biochar after synthetic doping was verified by the EDAX analysis Figure 1(b).

Characterization of Zn-doped CaO catalyst by FTIR

The FTIR spectrum of the Zn-doped CaO catalyst is shown in Figure 1(c). O-H bending was observed at 1375.3 and 1433.48 cm^{-1} (carboxylic acid) broadband wavelength and

the presence of a strong stretch of N–O (nitro compound) was detected at 1550.45 cm^{-1} . At 1078.8 cm^{-1} , a high absorption band shows a strong stretch of the C–O bond. The 841.04 cm^{-1} showed a strong C–H bond. The FTIR graph shows the major peaks.

Biodiesel process optimization

Effect of catalyst concentration

Parameters such as the catalyst concentration, M:O ratio, temperature and time were varied during the transesterification process using the Zn catalyst-doped biochar. In this study, the concentration of the catalyst varied from 0.5% to 4% (w/w) and the associated biodiesel yield is shown in Figure 2(a). The biodiesel yield increased steadily from 0.5% to 3%, then started to decline. At 3% (w/w) catalyst concentration, the highest biodiesel yield of 89.5% was achieved.

Effect of M:O ratio

The biodiesel yield is significantly influenced by the M:O ratio. The procedure was carried out with a 3% (w/w) catalyst concentration for 60 min at a temperature of 60 °C. The M:O ratio was changed from 5:1 to 30:1 while holding the other variables constant. The biodiesel yield increased with an increasing M:O ratio from 5:1 to 15:1 and decreased gradually as the ratio increased from 15:1 to 30:1. As shown in Figure 2(b), 91% biodiesel yield was achieved at an M:O molar ratio of 15:1.

Table 1. Experimental design for biodiesel production using zinc-doped biochar catalyst.

Trial	A (Catalyst concentration) (wt%)	B (Methanol molar ratio)	C (Temperature) (°C)	D (Time) (min)	Experimental biodiesel yield (%)	Predicted biodiesel yield (%)
1	4	10	50	50	82.2	81.9605
2	2	20	50	50	84.8	83.8438
3	2	10	70	50	85.9	84.8716
4	4	20	70	50	88.5	88.3883
5	2	10	50	70	89.9	88.8105
6	4	20	50	70	90	89.8272
7	4	10	70	70	90.2	89.9549
8	2	20	70	70	88.9	87.9383
9	3	15	60	60	92.7	92.3009
10	3	15	60	60	93.1	92.3009
11	2	10	50	50	82	82.7938
12	4	20	50	50	86	86.0605
13	4	10	70	50	85.2	85.0883
14	2	20	70	50	84.3	85.1216
15	4	10	50	70	88.3	87.8772
16	2	20	50	70	87.2	87.7105
17	2	10	70	70	89.5	89.8383
18	4	20	70	70	91.5	91.1049
19	3	15	60	60	92.1	92.3009
20	3	15	60	60	91.9	92.3009
21	2	15	60	60	90.8	91.2297
22	4	15	60	60	91.9	92.3964
23	3	10	60	60	88.7	89.563
24	3	20	60	60	90.6	90.663
25	3	15	50	60	90.4	90.7741
26	3	15	70	60	91.9	92.4519
27	3	15	60	50	88.5	88.1297
28	3	15	60	70	91.2	92.4964
29	3	15	60	60	92.1	92.3009
30	3	15	60	60	92.4	92.3009

Table 2. Regression coefficients of the quadratic polynomial model for optimization of biodiesel production using zinc-doped biochar catalyst.

Term	Coeff.	SE coeff.	t value	p value	Significance
A	0.583	0.15	3.9	0.002	
B	0.55	0.15	3.68	0.003	
C	0.839	0.15	5.61	0.001	Significant
D	2.183	0.15	14.6	0.001	Significant
A A	0.488	0.398	1.23	0.242	
B B	2.188	0.398	5.5	0.001	Significant
C C	0.688	0.398	1.73	0.108	
D D	1.988	0.398	4.99	0.001	Significant
A B	0.762	0.159	4.81	0.001	Significant
A C	0.263	0.159	1.66	0.122	
A D	0.025	0.159	0.16	0.877	
B C	0.2	0.159	1.26	0.229	
B D	0.537	0.159	3.39	0.005	
C D	0.263	0.159	1.66	0.122	

Table 3. ANOVA of the quadratic polynomial model for optimization of biodiesel production using zinc-doped biochar catalyst.

Source	df	Adj. SS	Adj. MS	F value	p value	Significance
Model	16	275.736	17.2335	42.83	.001	Significant
Linear	4	110.042	27.5106	68.37	.001	Significant
Square	4	99.088	24.7719	61.56	.001	Significant
2-way interaction	6	16.78	2.7967	6.95	.002	
Error	13	5.231	0.4024			
Lack of fit	10	5.086	0.5086	10.52	.039	
Pure error	3	0.145	0.0483			
Total	29	280.967				

Effect of time and temperature

The previously optimized catalyst concentration of 3% (w/w) and the 15:1 M:O molar ratio were used for biodiesel synthesis. The process was carried out under varying reaction times ranging from 20 to 70 min and the temperature was varied from 40 to 90 °C. As shown in Figure 2(C), the biodiesel yield increases as the temperature increases from 40 to 60 °C and then drops. This drop occurring beyond

60 °C could be the result of alcohol evaporating above this temperature, as it is close to the boiling point of methanol. The maximum biodiesel yield of 92% was obtained at 60 min and 60 °C (Figure 2c).

Effect of reusability on biodiesel production

Biochar catalysts are solid catalysts, which are exploited for their reusability value. Lower production costs are achieved with greater reusability. The biochar catalyst was reused for five cycles. The biodiesel yield was the highest in the first cycle (94%) and declined to 61% by the end of the fifth cycle (Figure 2(d)). External factors such as moisture and CO₂ content of the air negatively affect the active catalytic sites, thus affecting the reusability.

Process optimization using RSM

RSM was used to optimize biodiesel production by optimizing the independent variables catalyst concentration, M:O molar ratio, time, and temperature. To find the ideal conditions for achieving the highest biodiesel yield (%), the four input variables were changed from low to high ranges throughout the course of this study. Table 1 lists the CCD techniques applied to 30 experimental runs. Table 2 includes a summary of regression coefficients and their importance to the procedure. To examine the impact of catalyst concentration on FAME conversion, the concentration of the catalyst was varied between 2%, 3%, and 4% (w/w). To observe the impact of the M:O molar ratio on FAME conversion, M:O molar ratios of 10:1, 15:1, and 20:1 were used.

The process variables reaction time and temperature were varied from 50 to 70 min and 50 °C to 70 °C, respectively. Analysis of variance (ANOVA) was used to study the process parameters and determine the relevant conditions,

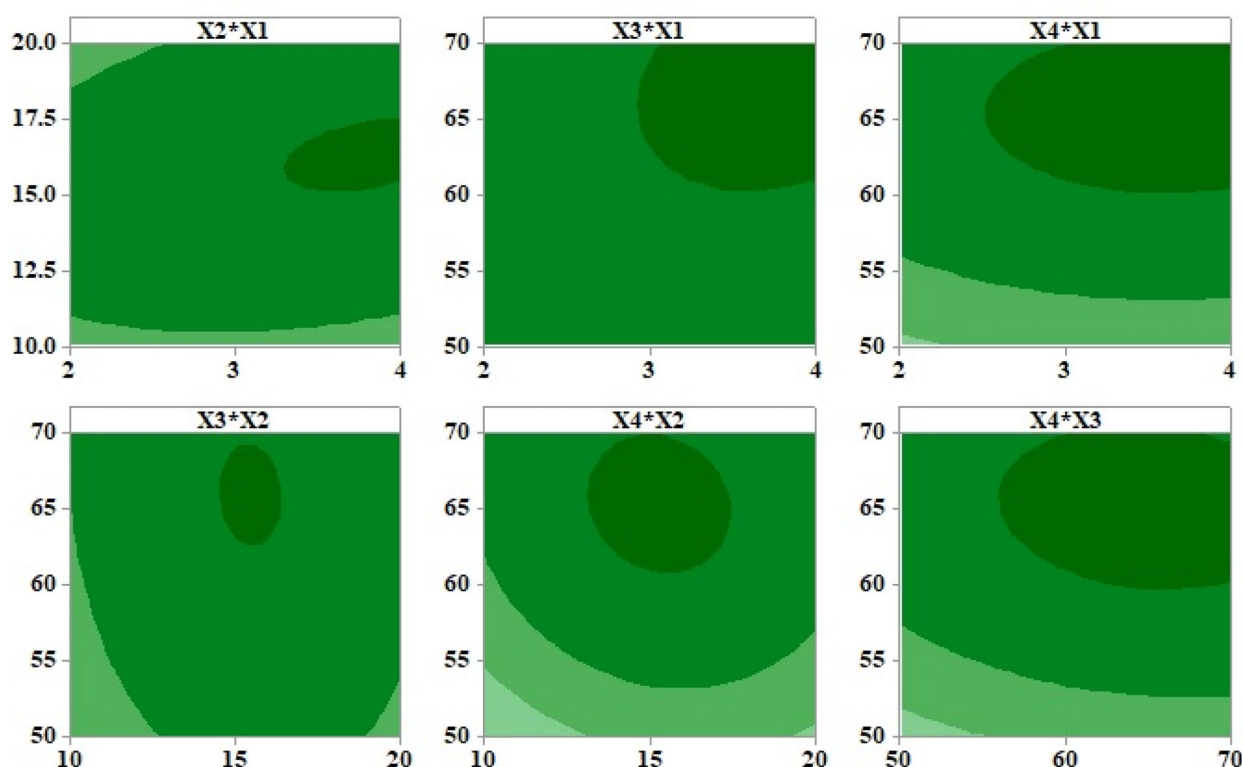


Figure 3. Response contour plots: (a) Methanol:Oil ratio Catalyst concentration; (b) Temperature Catalyst concentration; (c) Time Catalyst concentration; (d) Temperature Methanol:Oil ratio; (e) Time Methanol:Oil ratio; (f) Time Temperature.

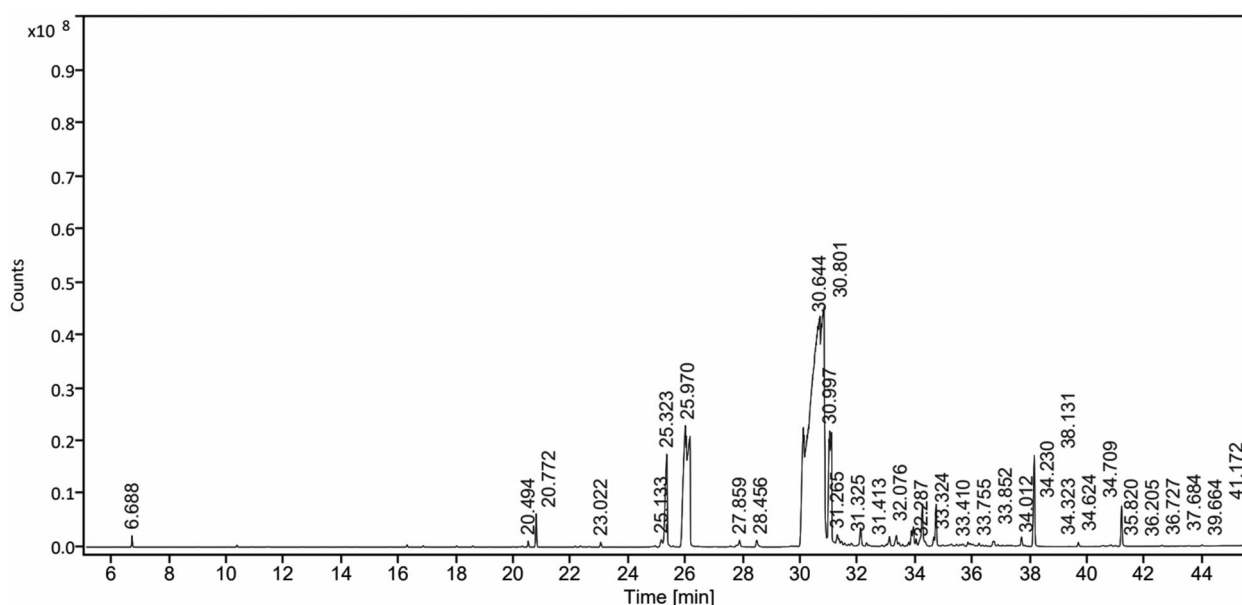


Figure 4. Gas Chromatography Mass Spectrometry analysis of the produced biodiesel.

as shown in Table 3. The significant and quadratic operational parameter models were confirmed to be accurate after a set of models was evaluated.

$$\begin{aligned} \text{Yield} = & 92.301 + 0.583 A + 0.550 B + 0.839C + 2.183D \\ & 0.488A A + 2.188 B B + 0.688C C + 1.988D D \\ & + 0.762A B + 0.263A C + 0.025A D \\ & 0.200 B C + 0.537 B D + 0.263C D \end{aligned}$$

(3)

The significant influence of the factors with higher F values and lower p values is demonstrated using ANOVA in Table

3. Each model's relevance was established by a low p value (.001), and a variable with a p value greater than .001 is considered to be non-significant. The depletion of FAME production is positively correlated with the temperature coefficient as shown in the table. The biodiesel production was primarily influenced by reaction temperature compared to other parameters. The biodiesel conversion response contour plots are depicted in Figure 3.

The optimum conditions of the parameters – catalyst concentration of 3.92% (w/w), M:O molar ratio of 16:1, reaction temperature of 65 C, and reaction duration of 66.5 min – yielded the maximum product (FAME% conversion) of 94.1% biodiesel. The experimental validation

supported this result. In contrast to the 91% biodiesel produced in a previous study from *Sapindus trifolius* oil at a catalyst concentration of 4% (w/w), 58 °C and 73 min using Mg-CaO catalyst, we obtained the greatest yield of 93% biodiesel with a lower catalyst concentration of 3% (w/w) at 60 °C and 70 min [30]. In another study, a 90.2% biodiesel yield was obtained from WCO using magnetic biochar derived from waste palm kernel shell under the following optimum conditions: 13:1 M:O ratio, catalyst concentration 3.66 wt%, temperature 65 °C and time 102 min. Comparatively, the present study showed increased biodiesel yield with minimum reaction time [31].

Biodiesel characterization

GC-MS was used to analyze several compounds present in the synthesized biodiesel. The peaks from GC-MS are shown in Figure 4. A total of 33 compounds were found in the biodiesel. Octanoic acid, methyl ester, methyl myristoleate, methyl tetradecanoate, pentadecanoic acid, and methyl ester were present. Methyl stearate was also detected in the biodiesel. The results confirmed the presence of methyl esters in the synthesized biodiesel.

Conclusions

Biodiesel production from WCO using a Zn-doped biochar catalyst had a substantial influence on the FAME content. The quality of this biodiesel complied with ASTM requirements, suggesting WCO and Zn-doped biochar catalysts may be a good alternative to other expensive and less environmentally safe feedstocks and catalysts. This study produced the highest yield of 94.1% under ideal processing conditions. Future studies involving WCO and Zn-doped biochar are needed to evaluate the commercialization of the produced biodiesel.

Disclosure statement

The authors declare no competing interests.

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