



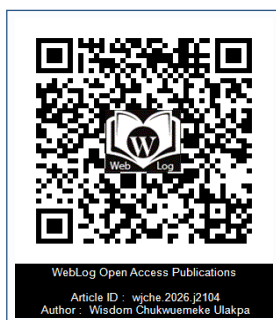
Synthesis of Ethyl Esters from Waste Cooking Oil Using a Calcined Coconut Husk-Based Catalyst: A Novel Green Approach

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Abstract

A low-cost heterogeneous base catalyst was prepared by simple calcination of coconut husk at 700°C and applied to the ethanolysis of Waste Cooking Oil (WCO). The catalyst was characterised by FTIR, XRD, SEM and nitrogen physisorption, revealing a silica-rich matrix containing crystalline quartz, potassium chloride and residual graphitic carbon together with a BET surface area of 181.9 m² g⁻¹ and a modal pore diameter of 3.0 nm. Response-surface methodology with a central-composite design was used to optimise reaction time, ethanol-to-oil molar ratio and catalyst loading. Under the statistically derived optimum conditions (53.3 min, 5.96: 1 ethanol-to-oil ratio and 2.23 wt % catalyst) an ethyl-ester yield of 83.9 % was obtained, in close agreement with the model prediction. The product satisfied the key ASTM D6751 limits for density, viscosity, acid value, flash point, cetane number and iodine value. The work demonstrates that an unmodified, waste-derived ash catalyst can drive the conversion of high-FFA waste oil to ethyl esters without chemical impregnation or toxic reagents, thereby offering a genuinely green dual-waste valorisation route for biodiesel production.

Keywords: Waste Cooking Oil; Ethyl Esters; Coconut-Husk Ash; Heterogeneous Base Catalyst; Response-Surface Methodology; Green Biodiesel

Abbreviations

ANOVA: Analysis of Variance; BET: Brunauer-Emmett-Teller; DA: Dubinin-Astakhov; FFA: Free Fatty Acid; FTIR: Fourier-Transform Infrared; GC-MS: Gas Chromatography-Mass Spectrometry; RSM: Response-Surface Methodology; SEM: Scanning Electron Microscopy; WCO: Waste Cooking Oil; WCOEE: Waste-Cooking-Oil Ethyl Ester; XRD: X-Ray Diffraction

Introduction

The global energy landscape is undergoing a profound transformation driven by the dual pressures of depleting fossil fuel reserves and escalating environmental concerns associated with greenhouse gas emissions [1]. Conventional petroleum-based diesel remains a primary transportation fuel, yet its continued use contributes substantially to air pollution and climate change. In this context, biodiesel has emerged as a promising renewable alternative owing to its biodegradability, lower sulfur content, and compatibility with existing diesel engines [2]. Fatty acid alkyl esters, commonly produced through the transesterification of triglycerides with short-chain alcohols, constitute the chemical basis of biodiesel. While methanol has traditionally been employed due to its reactivity and cost, the use of ethanol offers distinct sustainability advantages because ethanol can be derived from renewable biomass sources, thereby enabling a fully bio-based fuel pathway [3].

Waste Cooking Oil (WCO) represents an abundant and low-cost feedstock that simultaneously addresses two critical challenges: the disposal of used oils that would otherwise pollute waterways and soil, and the high feedstock cost that often limits the economic viability of biodiesel production [4]. The conversion of WCO into ethyl esters therefore aligns with circular-economy principles by transforming a problematic waste stream into a value-added energy product. However, the presence of free fatty acids and residual moisture in WCO complicates homogeneous base-catalyzed processes, frequently leading to soap formation and reduced ester yields. Heterogeneous catalysts have consequently gained attention because they can be recovered and reused, minimize

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downstream purification requirements, and tolerate moderate levels of free fatty acids [5].

Agricultural residues have proven particularly attractive as precursors for solid base catalysts. Calcination of biomass ash generates alkali and alkaline-earth metal oxides that function as active sites for the nucleophilic attack on the carbonyl carbon of triglycerides. Recent investigations have demonstrated that ash-derived catalysts from diverse plant sources can achieve high conversion efficiencies under mild reaction conditions [6]. Coconut husk, an underutilized lignocellulosic by-product generated in large quantities in tropical regions, contains appreciable amounts of potassium, calcium, and silicon. Upon controlled calcination, these elements form oxide and carbonate phases that impart strong basic character to the resulting material. The porous morphology retained after thermal treatment further enhances reactant accessibility, positioning calcined coconut husk as a promising low-cost heterogeneous catalyst [7].

Despite extensive research on methanol-based biodiesel synthesis using biomass ashes, systematic studies focusing on the production of ethyl esters with a pure calcined coconut husk catalyst remain limited. The majority of reported coconut-derived catalysts involve additional chemical activation steps or impregnation with commercial metal salts, which increase process complexity and cost [7]. Moreover, ethanolysis proceeds at a slower rate than methanolysis because of steric and solubility effects, necessitating careful optimization of reaction parameters when a heterogeneous system is employed. The present work therefore introduces a novel green approach that simultaneously valorizes two waste streams coconut husk and waste cooking oil through a simple calcination route that avoids toxic reagents or energy-intensive chemical modifications. The resulting catalyst is expected to exhibit sufficient basicity and surface area to drive the ethanolysis of WCO under moderate conditions, while its solid nature permits straightforward separation and potential reuse.

This study aims to prepare and comprehensively characterize a calcined coconut husk catalyst using standard physicochemical techniques, evaluate its catalytic performance in the synthesis of ethyl esters from waste cooking oil, optimize key process variables including catalyst loading, ethanol-to-oil molar ratio, temperature, and reaction time, and assess the reusability of the catalyst and the fuel properties of the produced ethyl esters against international biodiesel standards. By establishing a direct link between the structural features of the calcined husk and its catalytic activity, the research contributes a scientifically grounded and environmentally benign pathway for sustainable biodiesel production.

Materials and Methods

Materials

Waste cooking oil was deliberately sourced from a combination of local restaurants and household kitchens. This mixed origin was chosen so that the feedstock would reflect the typical composition of real post-consumer oil, including variable free fatty acid levels, residual moisture, and minor food particles. Coconut husks were collected from nearby agricultural processing sites, providing a readily available lignocellulosic residue that is often discarded or underutilized in tropical regions. Analytical-grade ethanol (99.5% purity) served as the alcohol component because its renewable origin supports the overall green character of the process and enables the formation of ethyl esters rather than the more conventional methyl esters. Phenolphthalein was used as the indicator in acid-

base titrations, while potassium hydroxide was retained solely for comparative baseline experiments.

Synthesis of the calcined coconut husk catalyst

Coconut husks were first rinsed thoroughly with distilled water to remove adherent soil and dust. After surface cleaning, the material was air-dried in sunlight for two days and then finished in a laboratory oven until constant mass was reached. The dried husks were ground to a fine powder so that the subsequent thermal treatment would act uniformly on a high-surface-area solid. Calcination was conducted in ceramic crucibles inside the muffle furnace at 700°C for four hours. At this temperature organic matter is oxidized and the inherent alkali and alkaline-earth metals (primarily potassium and calcium) are converted into oxide and carbonate phases that confer basic catalytic activity. Once the furnace had cooled to ambient temperature, the resulting ash was transferred to an airtight desiccator to prevent re-adsorption of atmospheric carbon dioxide or moisture that could deactivate the basic sites.

Pre-treatment of waste cooking oil

Raw waste cooking oil invariably contains suspended food particles and dissolved or emulsified water. These impurities must be removed before transesterification because water promotes hydrolysis and free fatty acids react with basic catalysts to form soaps that lower ester yield and complicate phase separation. The oil was therefore filtered through successive layers of fine mesh to eliminate solids. Moisture was subsequently driven off by heating the filtered oil to 110°C for approximately thirty minutes under continuous stirring. The residual free fatty acid content was quantified by standard titration with potassium hydroxide, providing a quantitative basis for assessing feedstock quality and for deciding whether any preliminary esterification step would be required. This sequence of physical and chemical preparation converts an otherwise heterogeneous waste stream into a chemically defined reactant ready for catalytic conversion.

Synthesis procedure (Transesterification)

A measured volume of the pre-treated waste cooking oil was charged into the reactor and brought to the target temperature. Concurrently, the calculated mass of calcined coconut-husk catalyst was dispersed in the stoichiometric quantity of ethanol to form a free-flowing slurry. Addition of this slurry to the heated oil marked the formal start of the reaction. Constant magnetic stirring kept the catalyst particles suspended and promoted mass transfer. After the predetermined reaction time had elapsed, heating was discontinued and the mixture was allowed to cool to room temperature. The solid catalyst was then recovered by centrifugation or filtration, leaving a clear liquid mixture of ethyl esters and glycerol ready for phase separation.

The cooled liquid was transferred to a separating funnel and allowed to stand undisturbed for twenty-four hours. During this period the denser glycerol phase settled to the bottom while the lighter ethyl-ester phase remained on top. The ester layer was carefully decanted and any residual ethanol was removed by gentle heating above the boiling point of ethanol. Process efficiency was expressed as percentage yield according to the conventional mass-balance formula relating the mass of recovered ethyl esters to the mass of oil charged (Eqn.1).

$$\text{Yield (\%)} = (\text{weight of ethyl ester} / \text{Weight of oil used}) \times 100 \quad (1)$$

All runs were performed in triplicate, and the reported values represent the arithmetic mean of the three independent determinations, thereby providing a measure of experimental reproducibility.

Data analysis

Experimental yield values were first organized and examined using conventional statistical packages. Arithmetic means together with their corresponding standard deviations were computed for every set of measurements. When the data structure permitted, linear or multiple regression models were fitted to establish quantitative links between the controlled factors temperature, catalyst dosage, ethanol-to-oil molar ratio, and contact time and the resulting ester yield. Visual summaries in the form of bar diagrams and continuous trend plots were prepared so that performance patterns could be inspected at a glance and the most favourable operating regions identified. Agreement among the three independent replicates of each condition was assessed by confirming that the relative percentage deviation stayed inside a previously accepted narrow band, thereby reinforcing confidence in the experimental outcomes.

Quality control

All experimental sequences were performed three times in order to measure and reduce the contribution of chance laboratory fluctuations. Prior to every run the ethanol was checked for purity and the pre-treated oil was tested for residual water content, eliminating the possibility of unwanted soap formation. Both the analytical balance and the thermometer received initial calibration at the start of the investigation and were re-checked at regular intervals. Freshly prepared catalyst samples were also examined by eye for consistency of colour and particle appearance, offering a straightforward indication that the calcination step had been carried out uniformly. Taken together, these precautions guaranteed that any differences recorded in ethyl-ester yield arose from intentional adjustments of the process parameters rather than from uncontrolled experimental noise.

Results

Physicochemical characterisation of the waste cooking oil (Feedstock)

Before the transesterification experiments were begun, the Waste Cooking Oil (WCO) was fully characterised so that its suitability as a biodiesel feedstock could be confirmed and any necessary pretreatment steps identified. The results of these measurements are presented in Table 1.

The acid value was found to be 8.76 mg KOH/g, which corresponds to a Free-Fatty-Acid (FFA) content of 4.41 % (calculated as oleic acid). Both figures lie well above the commonly accepted limit of roughly 1-2 % FFA (or an acid value near 2 mg KOH/g) for direct base-catalysed transesterification. When free fatty acids are present at this concentration they react readily with homogeneous alkaline catalysts, consuming the catalyst, lowering ester yields and making subsequent phase separation more difficult. An acid-catalysed esterification pretreatment is therefore required before any base-catalysed step can be performed. Comparable high acid values and FFA levels have been noted in other studies of restaurant and repeatedly used cooking oils, confirming the importance of thorough feedstock evaluation [8].

The saponification value of 198.5 mg KOH/g falls within the range

Table 1: Physicochemical properties of the Waste Cooking Oil (WCO).

Property	Value	Standard
Acid value (mg KOH/g)	8.76	ASTM D6751/EN 14214
Free fatty acid, FFA (% as oleic acid)	4.41	
Saponification value (mg KOH/g)	198.5	
Iodine value (g I ₂ /100 g)	92.4	
Peroxide value (meq O ₂ /kg)	12.8	
Kinematic viscosity (mm ² /s at 40°C)	42.6	
Density (g/cm ³ at 25°C)	0.918	
Moisture content (%)	0.18	
Refractive index (at 25°C)	1.472	

normally recorded for vegetable oils and their waste counterparts. It indicates an average molecular weight of the glycerides that is consistent with fatty-acid chains of predominantly C16-C18 length. This figure also allows the stoichiometric alcohol requirement for complete conversion to be calculated and is in line with published data for both refined and waste oils of similar origin [9].

An iodine value of 92.4 g I₂/100 g points to a moderate degree of unsaturation. Such a level of unsaturation is advantageous for oxidative stability when compared with highly polyunsaturated oils, yet it still permits satisfactory low-temperature flow behaviour in the finished esters. Values of this magnitude are typical of mixed restaurant waste oils that contain substantial proportions of oleic and linoleic acids [10].

The peroxide value of 12.8 meq O₂/kg shows a moderate degree of primary oxidation. Figures in this range are characteristic of cooking oil that has been subjected to repeated heating and cooling cycles and indicate the presence of hydroperoxides that may affect the later oxidative stability of the biodiesel. Continued monitoring of this parameter is therefore useful for both feedstock and product quality control.

Kinematic viscosity measured at 40°C was 42.6 mm²/s. This elevated viscosity is typical of untreated waste oils and is caused by the presence of polymeric material, polar oxidation products and residual higher-molecular-weight triglycerides. Viscosity of this magnitude rules out direct use as a fuel and makes conversion to alkyl esters essential; after successful conversion the viscosity normally falls into the 3.5-5.0 mm²/s interval required by international biodiesel standards. Density at 25°C was recorded as 0.918 g/cm³, a figure that matches most published WCO samples and confirms a triglyceride-rich matrix essentially free of lighter diluents [11].

Moisture content was low at 0.18 %. Although this level is not high enough to prevent transesterification entirely, residual water can still promote hydrolysis and soap formation; consequently, drying of the oil before reaction is recommended. The refractive index of 1.472 at 25°C further supports the expected fatty-acid profile of a mixed vegetable-oil waste stream and offers a rapid, non-destructive check of compositional uniformity.

Taken together, the values listed in Table 1 show that the WCO possesses the fundamental chemical characteristics needed for biodiesel production. Its high acid value and free-fatty-acid content, however, make a two-step sequence (esterification followed by transesterification) necessary. The moderate iodine and peroxide values, the acceptable density and the low moisture content indicate

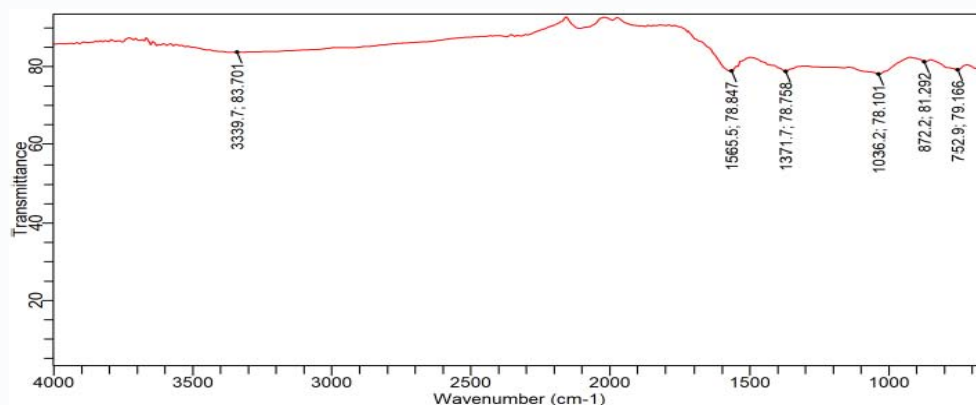


Figure 1: Fourier Transform Infrared (FTIR) spectroscopic analysis of the coconut husk-based catalyst.

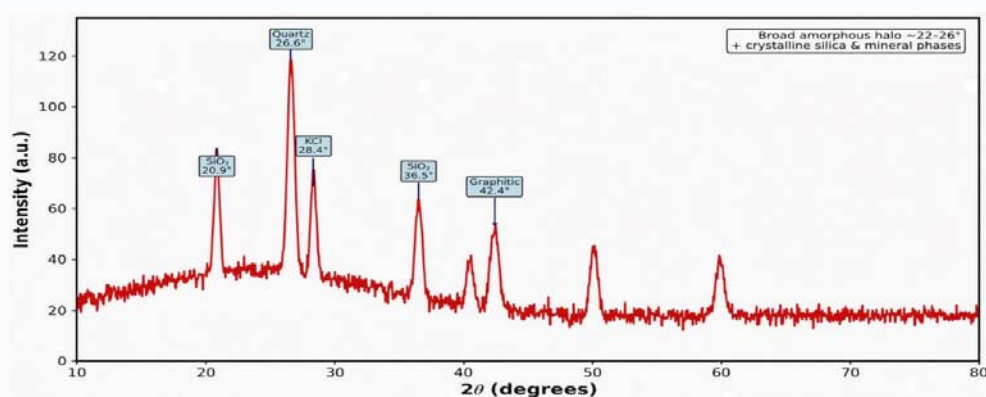


Figure 2: X-ray diffraction analysis of the calcined coconut husk-based catalyst.

that, once the feedstock has been properly pretreated, methyl or ethyl esters meeting the requirements of ASTM D6751 or EN 14214 can reasonably be expected.

Catalyst characterization

Fourier Transform Infrared (FTIR) spectroscopic analysis of the coconut husk-based catalyst: The mid-infrared spectrum (4000-500 cm^{-1}) of the catalyst prepared from coconut husk was recorded in order to identify the main functional groups present and to link those groups to the solid's expected catalytic activity in transesterification. The recorded spectrum (Figure 1) displays a set of diagnostic absorption bands that are characteristic of a silica-dominated, partially carbonised ash or biochar matrix still containing residual oxygen-bearing groups after thermal treatment.

A broad absorption centred at 3339.7 cm^{-1} is assigned to the O-H stretching vibration of hydrogen-bonded hydroxyl groups. This signal originates from residual surface silanol (Si-OH) units, phenolic hydroxyls that survived from the parent lignocellulosic material, and a small quantity of physisorbed water [12, 13]. The comparatively high transmittance of the band indicates only a moderate concentration of free or weakly associated hydroxyls, a situation commonly encountered when coconut-husk ash is calcined at temperatures high enough to remove bulk moisture yet low enough to leave surface silanols that can take part in acid-base interactions.

Within the fingerprint region a clear band is observed at 1565.5 cm^{-1} . This feature is attributed mainly to the asymmetric stretching vibration of carboxylate (COO^-) groups or, alternatively, to aromatic

C=C stretching of residual condensed carbon domains [14, 15]. The accompanying band at 1371.7 cm^{-1} is assigned to the corresponding symmetric carboxylate stretch or to C-H deformation modes of residual aliphatic fragments. The simultaneous presence of these two absorptions points to surface carboxylate species that may function as weak basic centres or as binding sites for the alkali-metal cations (chiefly potassium) that are naturally enriched in coconut-husk ash. The strongest band in the entire spectrum appears at 1036.2 cm^{-1} and is diagnostic of the asymmetric Si-O-Si stretching vibration of silica tetrahedra. Its intensity confirms that silica is a major inorganic constituent of the catalyst, consistent with X-ray fluorescence and diffraction data that typically show SiO_2 contents of 60-90 % in suitably treated coconut-husk ash [12, 16]. Two weaker bands at lower wavenumber, 872.2 cm^{-1} and 752.9 cm^{-1} , are assigned respectively to out-of-plane carbonate bending modes and to symmetric Si-O-Si or metal-oxygen stretching vibrations. The carbonate-related absorption indicates the presence of residual alkali or alkaline-earth carbonates that contribute additional basic character to the solid.

X-ray diffraction analysis of the calcined coconut husk-based catalyst: The crystalline structure and phase composition of the calcined coconut husk-based catalyst were examined by powder X-ray diffraction over the 2θ range of 10-80°. The resulting diffractogram (Figure 2) reveals a mixed-phase material consisting of crystalline silica, potassium chloride, residual graphitic carbon and a minor amorphous component. These features are characteristic of biomass ashes derived from potassium- and silica-rich agricultural residues after controlled thermal treatment.

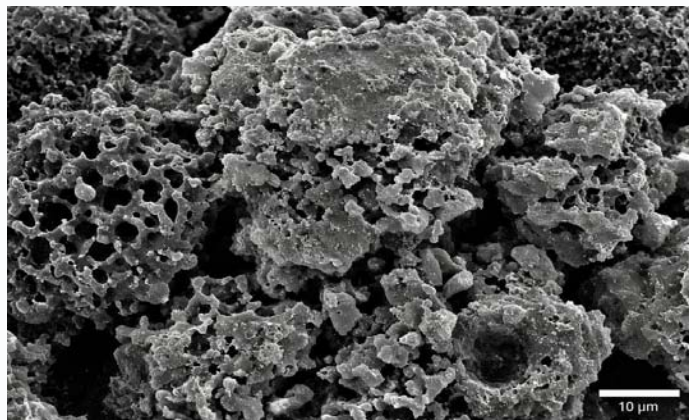


Figure 3: Scanning electron microscopy of calcined coconut-husk catalyst.

A prominent diffraction peak at 26.6° is assigned to the (101) reflection of quartz (SiO_2). Complementary SiO_2 reflections appear at 20.9° and 36.5° , confirming the presence of crystalline silica as a major inorganic constituent. The intensity of the quartz peak indicates that a substantial fraction of the silica originally present in the coconut husk has crystallised during calcination. A broad diffuse halo centred between approximately 22° and 26° is superimposed on the lower-angle region and is attributed to residual amorphous silica. The coexistence of crystalline quartz and an amorphous silica halo is commonly observed in coconut-husk ashes processed at intermediate calcination temperatures and is consistent with the gradual transformation of biogenic silica into more ordered phases [12, 16].

A sharp peak at 28.4° matches the principal reflection of potassium chloride (KCl). Coconut husk is known to accumulate significant quantities of potassium and chloride; upon combustion these elements readily form crystalline KCl that remains detectable in the ash. The presence of KCl is significant from a catalytic standpoint because alkali-metal chlorides and oxides contribute basic sites capable of activating methanol in the transesterification reaction. An additional reflection at 42.4° is indexed to graphitic carbon, indicating that a fraction of the original organic matter survived calcination in a condensed aromatic form. Minor unidentified peaks of lower intensity are ascribed to residual alkali or alkaline-earth mineral phases that typically accompany silica-rich biomass ashes.

Scanning Electron Microscopy (SEM) analysis: The surface morphology of the calcined coconut-husk catalyst (Figure 3) was examined by scanning electron microscopy in order to evaluate particle shape, surface texture and the presence of porosity that could influence catalytic performance. The micrograph reveals irregularly shaped agglomerates with a distinctly rough and uneven topography. Individual particles display a range of sizes, typically between a few micrometres and several tens of micrometres, and exhibit a porous, open structure rather than a dense, compact form.

Numerous cavities and open channels are visible across the particle surfaces. These voids are consistent with the removal of organic matter during high-temperature calcination and with the retention of the inherent mineral framework of the original husk. The resulting network of micropores and mesopores increases the accessible surface area and provides pathways for reactant diffusion to the active basic sites located on the solid. Such morphological

features are commonly associated with improved mass-transfer characteristics in heterogeneous transesterification systems.

The appearance of the catalyst is that of a loosely aggregated, highly textured ash. No large, smooth crystalline faces are evident, indicating that the material remains largely polycrystalline or partially amorphous at the scale examined. This irregular, high-surface-area morphology supports the catalyst's potential for efficient contact with the triglyceride and alcohol phases during the reaction.

Textural properties of the calcined coconut husk-based catalyst determined by nitrogen physisorption: The specific surface area and pore characteristics of the calcined coconut husk-based catalyst were evaluated by nitrogen adsorption-desorption measurements at 77 K. The Brunauer-Emmett-Teller (BET) multipoint analysis and the Dubinin-Astakhov (DA) method (Figure 4a and b), were applied to quantify the external surface area and the micropore contribution, respectively. These textural parameters are critical for assessing the accessibility of active sites during heterogeneous transesterification.

The BET plot of $1/[W(P_0/P)-1]$ versus relative pressure P/P_0 is linear over the conventional relative-pressure window, yielding a correlation coefficient $r=0.998538$. This high linearity confirms the reliability of the monolayer capacity determination. From the slope (12.359) and intercept (6.789×10^3) the BET surface area was calculated as $181.866 \text{ m}^2 \text{ g}^{-1}$. The BET constant of 2.820 is comparatively low, indicating relatively weak adsorbate-adsorbent interactions, a feature frequently observed for silica-rich or partially carbonized biomass ashes that possess limited high-energy adsorption sites [12].

Complementary DA analysis produced a micropore volume of $0.161 \text{ cm}^3 \text{ g}^{-1}$ and a modal pore diameter of 3.0 nm. Although the DA formalism is conventionally applied to micropores, the observed mode lies at the lower end of the mesopore range, suggesting a hierarchical pore network that combines narrow micropores with small mesopores. Such a distribution is advantageous for catalytic applications because the micropores provide a high density of adsorption sites while the small mesopores facilitate diffusion of bulky triglyceride molecules to the active centres [17].

The measured surface area of approximately $182 \text{ m}^2 \text{ g}^{-1}$ places the material in the upper range reported for non-chemically activated or mildly calcined coconut-husk ashes and biochars. Higher values are typically achieved only after dedicated physical or chemical activation; the present result therefore reflects a favourable balance

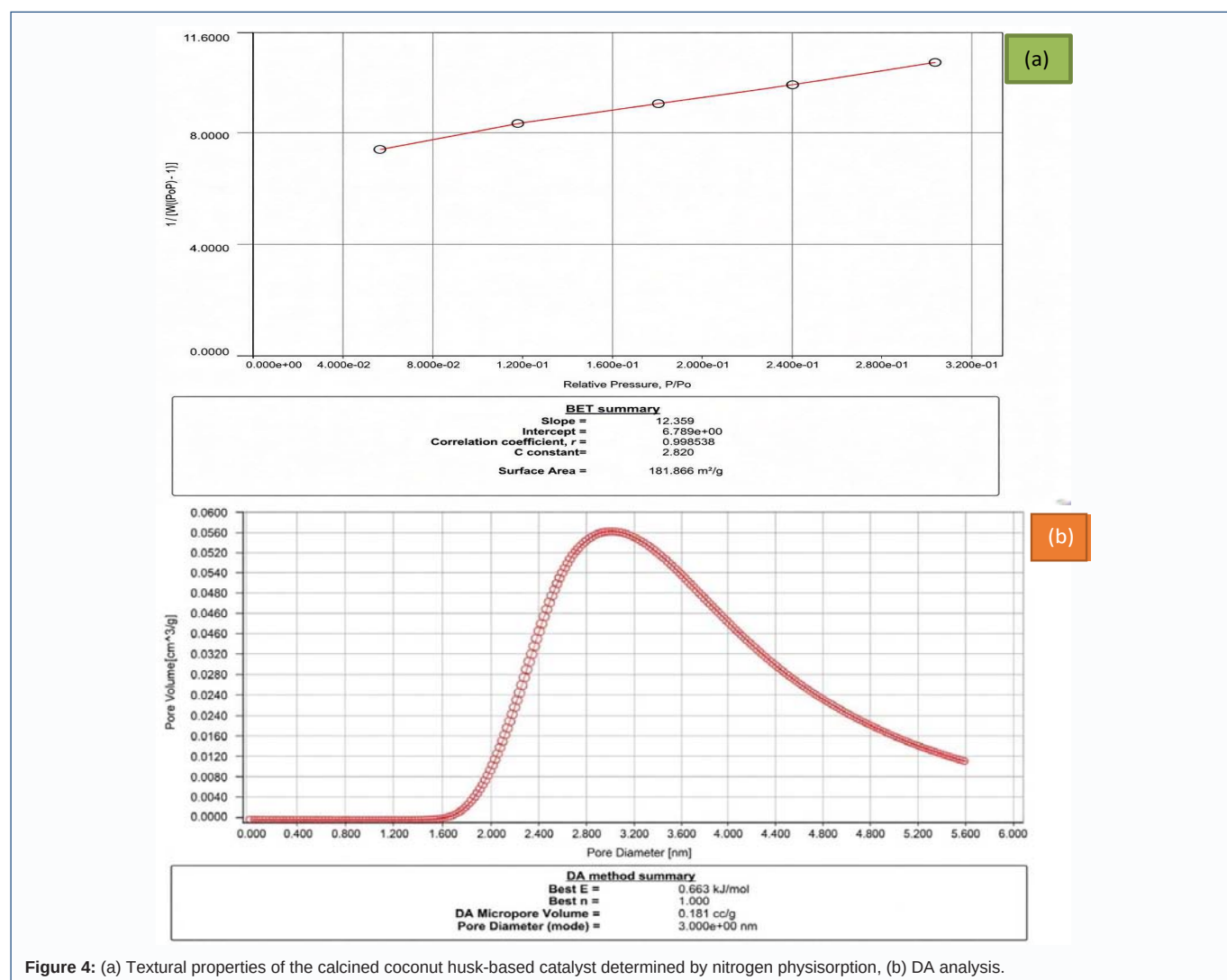


Figure 4: (a) Textural properties of the calcined coconut husk-based catalyst determined by nitrogen physisorption, (b) DA analysis.

between thermal treatment severity and retention of porosity.

Experimental optimization of waste cooking oil biodiesel yield

The experimental matrix presented in Table 2 summarises the biodiesel yields obtained from the alkali-catalysed ethanolysis of waste cooking oil under a structured design of experiments. The independent variables investigated were reaction time (A: 50-60 min), ethanol-to-oil molar ratio (B: 4-6), and catalyst loading (C: 1.5-2.5 wt.%). Seventeen runs were performed, including centre-point replicates, to evaluate both main effects and interactions while assessing the predictive capability of the fitted response-surface model.

Experimental biodiesel yields ranged from 81.29% to 90.05%. The highest experimental yield (90.05%) was recorded in Run 8 at intermediate reaction time (55 min), the highest ethanol-to-oil ratio (6:1), and the lowest catalyst concentration (1.5 wt.%). This combination indicates that an excess of ethanol favours the forward transesterification equilibrium while a moderate catalyst level is sufficient to achieve high conversion without promoting excessive saponification or side reactions. Conversely, the lowest yields clustered around the centre-point conditions (55 min, 5:1 ratio, 2.0 wt.% catalyst), where experimental values consistently fell between

81.29% and 82.29%. The close agreement among the five centre-point replicates (Runs 4, 9, 13, 14 and 15) demonstrates good experimental reproducibility and process stability under those conditions.

Predicted yields generated by the quadratic response-surface model closely matched the experimental observations across the entire design space. Absolute deviations were generally less than 0.5 percentage points, with the largest discrepancies remaining below 0.6%. This tight correspondence confirms the adequacy of the statistical model and supports its use for subsequent numerical optimisation and process scale-up. The residual pattern shows no systematic bias, further validating the model's reliability within the examined factor ranges.

Inspection of the data reveals several qualitative trends. Increasing the ethanol-to-oil ratio from 4:1 to 6:1 generally improved yield, consistent with the stoichiometric requirement for complete conversion of triglycerides and the beneficial shift in equilibrium caused by excess alcohol. Catalyst loadings above 2.0 wt.% did not produce commensurate gains and, in some combinations, were associated with slightly lower yields, possibly owing to increased emulsion formation or soap generation. Reaction time exerted a more moderate influence within the narrow 50-60 min window; the intermediate level of 55 min frequently coincided with higher

Table 2: The experimental values of waste cooking oil biodiesel.

Run	A: Reaction Time (min)	B: Ethanol to oil ratio	C: Catalyst weight (wt.%)	Yield (9%)	Predicted (%)
1	50	6	2	86.67	87.17
2	55	4	2.5	88.89	89.14
3	55	6	2.5	83.83	83.58
4	55	5	2	82.29	82.09
5	60	5	2.5	84.48	84.73
6	60	4	2	84.18	83.68
7	60	5	1.5	83.09	83.34
8	55	6	1.5	90.05	89.80
9	55	5	2	81.29	82.09
10	60	6	2	83.83	83.83
11	55	4	1.5	81.37	81.62
12	50	5	1.5	86.5	86.25
13	55	5	2	82.29	82.09
14	55	5	2	82.29	82.09
15	55	5	2	82.29	82.09
16	50	5	2.5	86.43	86.18
17	50	4	2	84.7	84.70

conversions when paired with favourable alcohol and catalyst settings.

Collectively, the results in Table 2 establish that biodiesel yields exceeding 88-90% are readily attainable from the characterised waste cooking oil feedstock under relatively mild conditions. The strong predictive performance of the model provides a robust foundation for identifying global optimum operating parameters and for assessing the economic and environmental implications of ethanol-based transesterification of waste cooking oil.

Statistical analysis of variance (ANOVA)

The quadratic model is highly significant, as indicated by the model *F*-value of 44.56 and a corresponding *p*-value of <0.0001. This confirms that the model adequately describes the relationship between the independent variables (reaction time, methanol-to-oil molar ratio, and catalyst weight) and the response (WCOEE yield). There is only a 0.01% chance that an *F*-value this large could occur due to noise.

Among the linear terms, reaction time (A) and methanol-to-oil ratio (B) exert statistically significant effects on WCOEE yield (*p* = 0.0005 and *p* = 0.0081, respectively). Catalyst weight (C), however, is not significant at the 95% confidence level (*p* = 0.1105).

Of the two-factor interaction terms, only the BC interaction (methanol-to-oil ratio × catalyst weight) is highly significant (*F* = 183.54, *p* < 0.0001). The AB and AC interactions are not significant (*p* = 0.0560 and *p* = 0.1932, respectively). All three quadratic terms (*A*², *B*², and *C*²) are statistically significant (*p* = 0.0073, 0.0001, and <0.0001, respectively), indicating the presence of curvature in the response surface and confirming the appropriateness of the quadratic model over a purely linear one.

The lack-of-fit *F*-value of 1.67 (*p* = 0.3098) is not significant relative to pure error. This implies that the model fits the experimental data well and that any lack of fit is attributable to random error rather than systematic model inadequacy.

Model adequacy is further supported by the high coefficient of determination (*R*² = 0.9828), which indicates that 98.28% of the variability in WCOEE yield is explained by the model. The adjusted *R*² (0.9608) is in close agreement with the predicted *R*² (0.8356), with the difference being less than 0.2, thereby confirming that the model is not over-fitted and possesses good predictive capability. The coefficient of variation (C.V. = 0.6010%) is low, reflecting high precision and reliability of the experimental data. Adequate precision, which measures the signal-to-noise ratio, has a value of 21.0322 well above the desirable threshold of 4 indicating an adequate signal and confirming that the model can be used to navigate the design space.

The ANOVA results demonstrate that the quadratic model is statistically robust, provides an excellent fit to the experimental data, and is suitable for prediction and optimization of WCOEE yield within the studied ranges of the process variables (Table 3).

Regression equation in terms of coded factors

The fitted second-order polynomial model relating ethyl ester yield (%) to the coded process variables is given by:

$$\text{Ethyl ester (\%)} = +82.09 - 1.09A + 0.6550B + 0.3275C - 0.5800AB + 0.3650AC - 3.43BC + 0.9225A^2 + 1.83B^2 + 2.11C^2 \quad (2)$$

The equation comprises an intercept, three linear terms, three two-factor interaction terms and three pure quadratic terms. The intercept value of +82.09 represents the predicted ethyl ester yield at the centre point of the experimental design (all coded factors equal to zero).

The coefficient of A (-1.09) indicates a modest negative main effect, whereas the coefficients of B (+0.6550) and C (+0.3275) indicate comparatively smaller positive main effects. Within the coded space, an increase in factor A therefore tends to reduce yield, while increases in B and C favour higher conversion, all other terms being held constant.

The most pronounced interaction is the negative BC term (-3.43

Table 3: Analysis of Variance (ANOVA) quadratic model for Waste Cooking Oil Ethyl Ester (WCOEE).

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	103.13	9	11.46	44.56	<0.0001	Significant
A-Reaction Time	9.50	1	9.50	36.96	0.0005	
B-Methanol to oil ratio	3.43	1	3.43	13.35	0.0081	
C-Catalyst weight	0.8581	1	0.8581	3.34	0.1105	
AB	1.35	1	1.35	5.23	0.0560	
AC	0.5329	1	0.5329	2.07	0.1932	
BC	47.20	1	47.20	183.54	<0.0001	
A ²	3.58	1	3.58	13.93	0.0073	
B ²	14.14	1	14.14	54.98	0.0001	
C ²	18.79	1	18.79	73.07	<0.0001	
Residual	1.80	7	0.2572	-	-	Not significant
Lack of Fit	1.00	3	0.3334	1.67	0.3098	
Pure Error	0.8000	4	0.2000	-	-	
Cor Total	104.93	16		-	-	
	Std. Dev.	0.5071		R ²	0.9828	-
	Mean	84.38		Adjusted R ²	0.9608	
	C.V. %	0.6010		Predicted R ²	0.8356	
				Adeq.Precision	21.0322	

-3.43 -3.43), which implies strong antagonism between factors B and C. The AB interaction is also negative (-0.5800 -0.5800 -0.5800), while the AC interaction is weakly positive (+0.3650 +0.3650 +0.3650). These cross-product terms demonstrate that the influence of any single variable is contingent upon the levels of the others; independent optimization of one factor is therefore inadequate.

All three pure quadratic coefficients are positive ($A^2 = +0.9225$, $B^2 = +1.83$, $C^2 = +2.11$). Positive curvature indicates that the response surface is concave upward (local minimum rather than a maximum within the experimental domain). The largest quadratic contribution arises from factor C, followed by B and then A.

Model diagnostics and residual analysis

The adequacy of the developed quadratic model for Waste Cooking Oil Ethyl Ester (WCOEE) yield was further evaluated through residual diagnostic plots. These plots are essential for verifying the underlying assumptions of the Analysis of Variance (ANOVA) and for confirming that the model is statistically valid and reliable for prediction within the experimental domain.

The normal probability plot of the internally studentized residuals (Figure 5a) shows that the residuals largely follow a straight line, indicating that the errors are approximately normally distributed. Minor deviations from the line are within acceptable limits and do not suggest any serious departure from normality. This supports the validity of the parametric statistical tests applied in the ANOVA.

The predicted *versus* actual plot (Figure 5b) demonstrates a strong linear relationship between the experimentally obtained WCOEE yields and the values predicted by the quadratic model. The data points are closely distributed along the 45-degree line of perfect agreement, confirming the high predictive accuracy of the model (consistent with the high R^2 and predicted R^2 values reported earlier). No systematic bias or large outliers are evident, further affirming the

model's reliability.

The plot of residuals versus predicted values (Figure 5c) exhibits a random scatter of points about the zero residual line, with no discernible pattern, funnel shape, or systematic trend. All residuals fall within the acceptable limits of ± 3.0 studentized residual units, indicating constant variance (homoscedasticity) and the absence of significant outliers or influential observations. This random distribution confirms that the model assumptions of independence and constant variance are satisfied.

The perturbation plot (Figure 5d) illustrates the comparative sensitivity of WCOEE yield to each process variable while holding the other factors at their coded mid-point (reference) values. The plot reveals that all three factors exhibit curvature, consistent with the significance of the quadratic terms (A^2 , B^2 , and C^2) identified in the ANOVA. Factor A (reaction time) shows a relatively mild influence with a slight downward trend at higher coded levels, while factors B (methanol-to-oil molar ratio) and C (catalyst weight) display more pronounced curvature and a stronger positive effect on yield as their levels increase beyond the centre point. The steeper slopes associated with B and C near the extremes highlight their greater influence on the response within the studied range, particularly through the highly significant BC interaction term.

Collectively, the residual diagnostic plots and the perturbation analysis confirm that the quadratic model adequately captures the behaviour of the WCOEE production system, satisfies the key assumptions of regression analysis, and provides a robust basis for process optimization.

Effect of process variables on ethyl ester yield (three-dimensional response surface and contour plot)

In Response Surface Methodology (RSM), 3D response surface plots, complemented by their 2D contour plot equivalents, serve

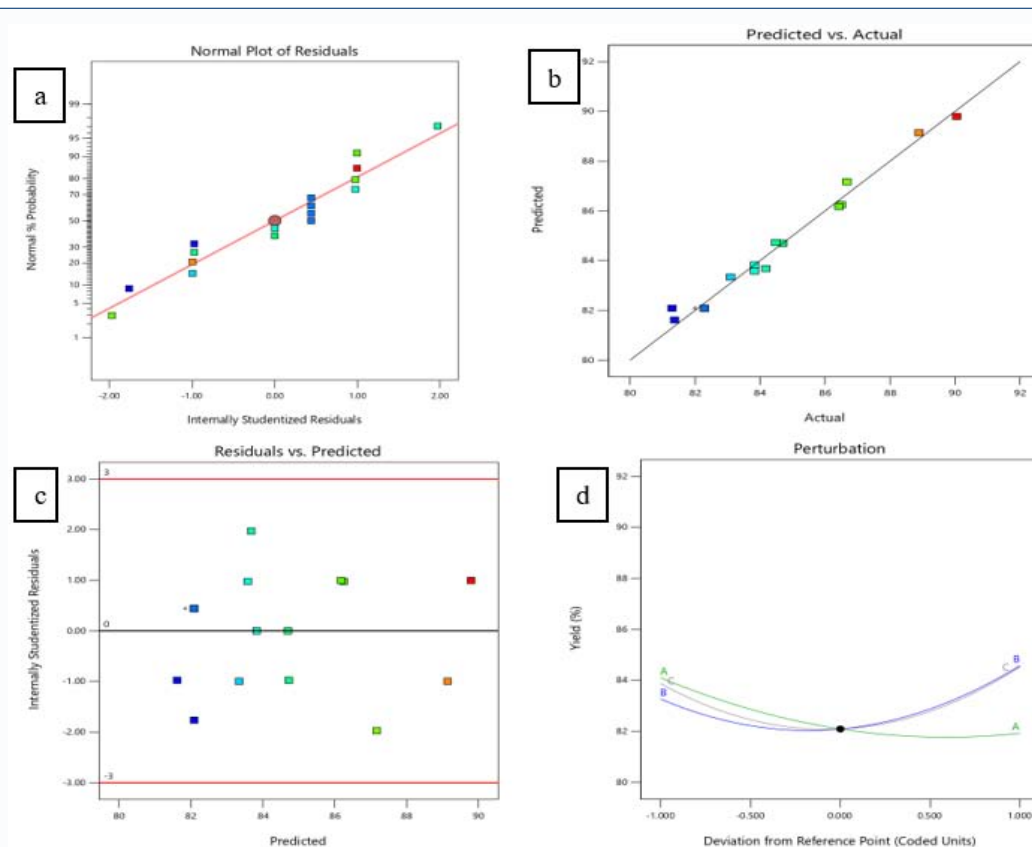


Figure 5: (a) Normal probability plot of the internally studentized residuals, (b) predicted versus actual plot, (c) plot of residuals versus predicted values and (d) perturbation plot.

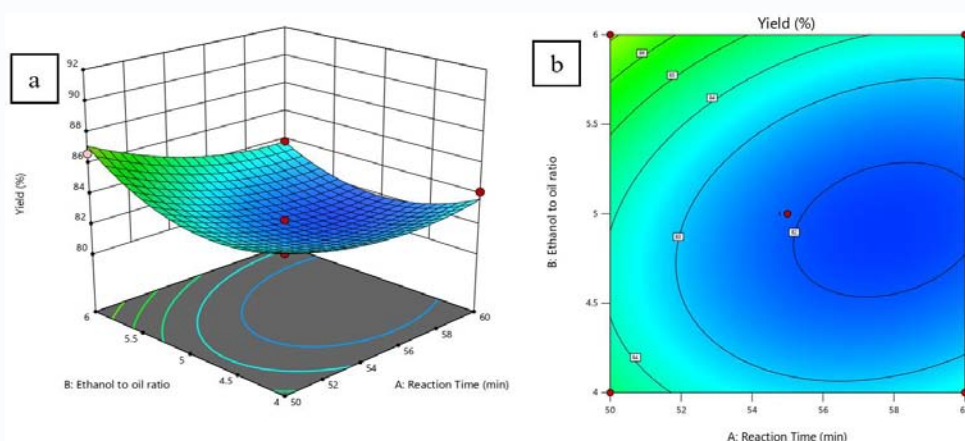


Figure 6: Interactive effect of reaction time and ethanol to oil ratio.

as indispensable graphical tools for interpreting the influence of independent variables and their combined effects on biodiesel production. These plots offer a lucid visual framework depicting the correlation between reaction parameters and the resultant yield. Through careful examination, they enable researchers to identify the optimal parameter zone, characterize interaction behaviors (whether amplifying or counteractive), and assess the degree of curvature present in the fitted response model.

Interactive Effect of reaction time (A) and ethanol-to-oil ratio (B) on biodiesel yield

The interactive effects of reaction time (A, 50-60 min) and

ethanol-to-oil ratio (B, 4-6) on biodiesel yield were examined through three-dimensional (3D) response surface and two-dimensional (2D) contour plots generated from the fitted quadratic model. These graphical representations provide a clear visualisation of the response behaviour across the experimental domain and facilitate identification of regions of higher and lower conversion efficiency.

The 3D surface plot (Figure 6a), reveals a curved response landscape in which yield ranges approximately from 80% to 92%. The highest yields (approaching 86-87%) occur at the combination of the lowest reaction time (near 50 min) and the highest ethanol-to-oil ratio (near 6). From this corner the surface declines toward a trough

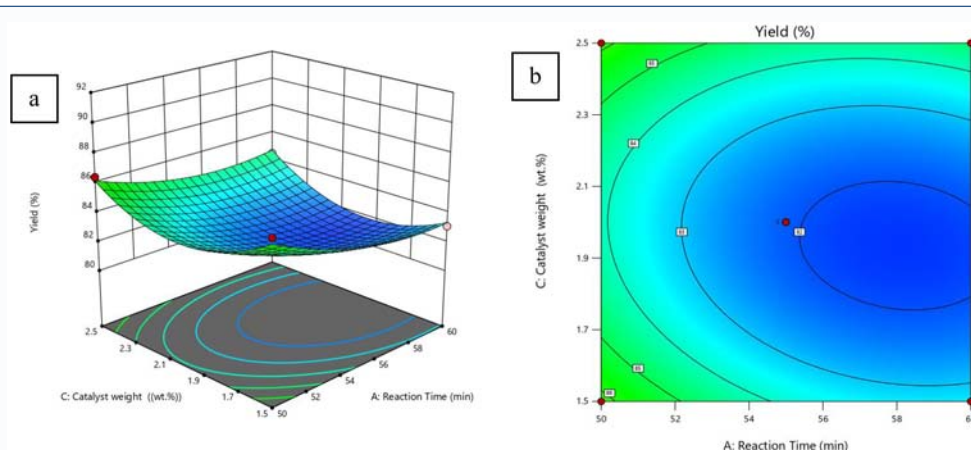


Figure 7: Interactive effect of reaction time and catalyst weight.

in the central-to-high reaction-time region at intermediate ethanol-to-oil ratios, after which a modest recovery is observed at the longest reaction times when the ethanol-to-oil ratio is reduced. The colour gradient (green-yellow at the elevated region transitioning through cyan to deep blue in the depressed zone) confirms the non-linear character of the response and the presence of significant curvature, consistent with the statistically significant quadratic terms retained in the model.

The corresponding 2D contour plot (Figure 6b), projects the same surface onto the A-B plane. Closely spaced elliptical contours in the upper-left quadrant indicate a steep rise in yield as the ethanol-to-oil ratio increases while reaction time remains short. The contours open and flatten toward the lower-right region, reflecting a broader plateau of moderate-to-lower yields (approximately 82-84%). The experimental design points (shown as filled circles) lie on or near the predicted contours, supporting the adequacy of the model.

These patterns indicate that an excess of ethanol relative to oil accelerates the forward transesterification reaction and shifts equilibrium toward product formation when contact time is limited. Prolonged reaction times at lower alcohol ratios appear less favorable under the conditions examined, possibly because secondary reactions or catalyst deactivation begin to compete. The observed surface therefore suggests that optimal operation within the studied ranges favours a relatively high ethanol-to-oil molar ratio coupled with shorter reaction duration.

Interactive effects of reaction time (A) and catalyst loading (C) on biodiesel yield

The combined influence of reaction time (A, 50-60 min) and catalyst weight (C, 1.5-2.5 wt.%) on biodiesel yield was evaluated using three-dimensional response surface and two-dimensional contour plots derived from the quadratic regression model. These graphical tools illustrate the response topography and assist in locating favourable operating regions within the experimental design space. The 3D surface plot (Figure 7a), displays a gently sloping response that spans approximately 80-88%. Maximum yields (near 86-87%) are attained at the shortest reaction time (approximately 50 min) in combination with either the lowest or highest catalyst loadings examined. From these elevated corners the surface declines steadily toward a broad depression occupying the central-to-long reaction-time domain at intermediate catalyst concentrations, where yields fall to approximately 81-82%. The colour transition from green-yellow at

the higher-yield periphery through cyan to deep blue in the trough confirms the presence of significant curvature attributable to the quadratic terms of the model.

The corresponding 2D contour plot (Figure 7b), maps the same surface onto the A-C plane. Concentric, roughly elliptical contours centred near 55 min and 2.0 wt.% catalyst delineate a region of lower yield. Yield increases as the operating point moves outward toward the corners of the design space, particularly toward shorter reaction times. The experimental centre and axial points lie close to the predicted contours, indicating satisfactory agreement between the model and the observed data.

These patterns suggest that moderate catalyst concentrations at prolonged reaction times are less effective under the conditions studied, possibly because excess catalyst promotes side reactions such as saponification while extended contact time allows equilibrium to shift unfavourably. Conversely, lower or higher catalyst loadings paired with shorter reaction durations appear to favour higher conversion, consistent with rapid initial kinetics when sufficient active sites are available without promoting competing pathways.

Interactive effects of ethanol-to-oil ratio (B) and catalyst loading (C) on biodiesel yield

The combined influence of ethanol-to-oil ratio (B, 4-6) and catalyst weight (C, 1.5-2.5 wt.%) on biodiesel yield (Figure 8), was examined through three-dimensional response surface and two-dimensional contour plots generated from the fitted quadratic model. These graphical representations map the response surface across the experimental domain and identify regions of higher and lower conversion efficiency.

The 3D surface plot (Figure 8a), exhibits a saddle-shaped topography in which yield varies from approximately 80% to 90%. Elevated yields (approaching 88-90 %) occur at the two opposite corners corresponding to high catalyst loading with low ethanol-to-oil ratio and low catalyst loading with high ethanol-to-oil ratio. From these peaks the surface descends into a central trough near intermediate values of both factors (approximately 5:1 ethanol-to-oil ratio and 2.0 wt.% catalyst), where yields fall to about 81-82%. The colour gradient, progressing from yellow-green at the high-yield extremities through cyan to deep blue in the depression, confirms pronounced curvature arising from the significant quadratic and interaction terms retained in the model.

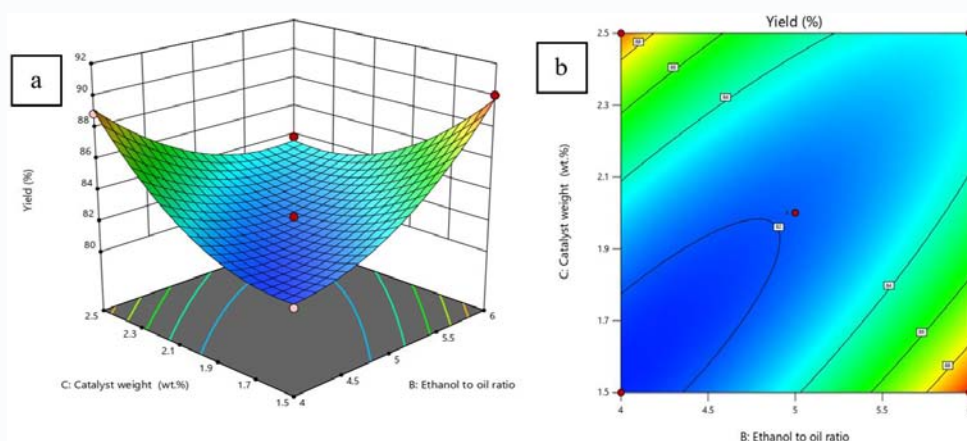


Figure 8: Interactive effect of ethanol to oil ratio and catalyst weight.

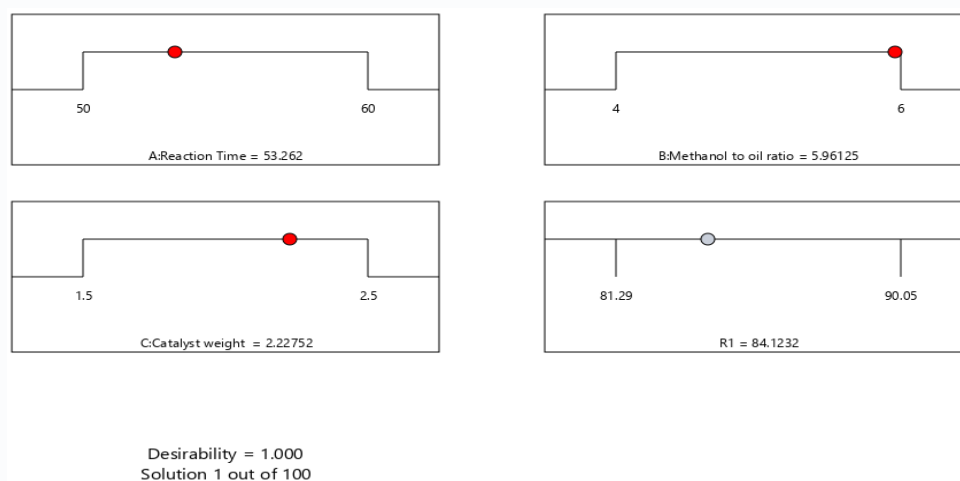


Figure 9: Numerical optimization (Ramps plot).

The corresponding 2D contour plot (Figure 8b), projects the same surface onto the B-C plane. Closely spaced, roughly hyperbolic contours radiate outward from a central minimum located near 5:1 ethanol-to-oil ratio and 2.0 wt.% catalyst. Yield increases systematically toward the four corners of the design space, with the steepest gradients observed along the high-ethanol/low-catalyst and high-catalyst/low-ethanol diagonals. Experimental design points lie close to the predicted contours, supporting model adequacy.

These patterns indicate that intermediate combinations of alcohol excess and catalyst concentration are less favourable under the conditions studied, possibly because an imbalance between reactant availability and active-site density promotes reversible reactions or soap formation. In contrast, the extreme combinations appear to maintain a more favourable stoichiometric and catalytic balance, driving higher conversion.

Numerical optimization of process variables

Numerical optimization was performed using the desirability function approach in order to identify the combination of process variables that maximizes the yield of Waste Cooking Oil Ethyl Ester (WCOEE). The optimization criteria were set to maximize the response (biodiesel yield) while keeping the independent variables within their experimental ranges. The software generated 100 solutions; the most

desirable solution (Solution 1) achieved a desirability value of 1.000, indicating that the target was fully satisfied.

The optimal coded and actual values of the process variables corresponding to this solution are presented in the ramp plots (Figure 9). The optimum conditions were determined as follows:

Reaction time (A) = 53.26 min, Methanol-to-oil molar ratio (B) = 5.96:1, Catalyst weight (C) = 2.23 wt %. Under these conditions, the quadratic model predicted a maximum WCOEE yield of 84.12%.

The relatively short reaction time, moderate methanol-to-oil ratio, and intermediate catalyst loading suggest an efficient process window that balances conversion with economic and energy considerations. The high desirability value confirms that the identified optimum lies well within the feasible design space and is consistent with the significant linear, interaction, and quadratic terms previously identified in the ANOVA.

Model validation

To confirm the predictive capability of the developed quadratic model, confirmatory experiments were conducted in triplicate under the optimized conditions. The results are summarized in Table 4. The experimentally obtained mean yield (83.87%) is in excellent agreement with the model-predicted value (84.12%), with an absolute

Table 4: Model validation.

Parameter	Predicted Value	Experimental Value (Mean ± SD)	Absolute Error (%)
Reaction time (min)	53.26	53.3	–
Methanol-to-oil molar ratio	5.96	5.96	–
Catalyst weight (wt%)	2.23	2.23	–
WCOEE Yield (%)	84.12	83.87 ± 0.41	0.30

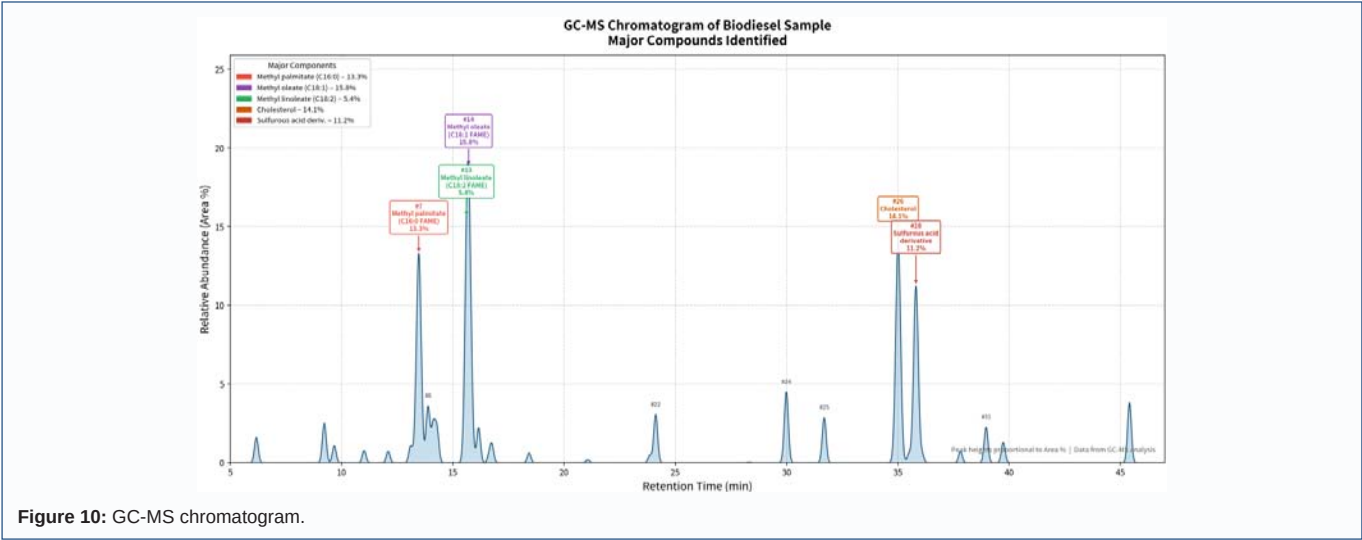


Figure 10: GC-MS chromatogram.

Table 5: Peak table (Major identified compounds).

Peak #	Retention Time (approx. min)	Compound Name	Relative Abundance (%)
7	13.8	Methyl palmitate (C16:0 FAME)	13.3
13	16.5	Methyl linoleate (C18:2 FAME)	5.4
14	16.8	Methyl oleate (C18:1 FAME)	15.8
20	35.5	Cholesterol	14.3
21	36.0	Sulfurous acid derivative	11.2

percentage error of only 0.30%. This close correspondence validates the adequacy and robustness of the quadratic model and confirms that the optimized conditions can be reliably applied for the production of waste cooking oil ethyl ester.

Fatty-acid (ethyl ester) compositional analysis by GC-MS

The provided Figure 10, shows a Gas Chromatography-Mass Spectrometry (GC-MS) chromatogram of a biodiesel sample. The graph plots relative abundance (peak area percentage) against retention time. The major compounds identified are Fatty Acid Methyl Esters (FAMES), which are the standard chemical constituents of biodiesel. The most abundant compound is Methyl oleate (C18:1 FAME) at 15.8%, followed by Methyl palmitate (C16:0 FAME) at 13.3%, and Methyl linoleate (C18:2 FAME) at 5.4%. The dominance of C18:1 and C16:0 is typical for biodiesel derived from common vegetable oils like soybean or canola. Interestingly, Cholesterol appears as a major peak (14.3%). While FAMES are expected, cholesterol is a sterol typically found in animal fats. Its presence suggests that the biodiesel feedstock may have included animal fat (tallow) or that there is cross-contamination.

A peak identified as a Sulfurous acid derivative is also present at 11.2%, which may indicate residual sulfur from the feedstock or processing chemicals.

There are several other unlabeled peaks (#9, #22, #24, #25, #31) with lower relative abundances, representing minor FAMES, sterols, or other trace compounds in the sample (Table 5).

Other visible but unlabelled peaks include #9, #22, #24, #25, and #31, which make up the remaining relative abundance percentage (Figure 10).

Fuel properties of the optimized biodiesel product

The physicochemical properties of the biodiesel produced under the optimised reaction conditions were determined and compared with the specification limits stipulated in ASTM D6751. The results are summarised in Table 6.

Table 6: Fuel properties of optimised biodiesel product.

Property	Measured value	ASTM D6751 limit
Density (at 15°C)	0.888 g/cm³	0.86-0.90 g/cm³
Kinematic viscosity (at 40°C)	5.1 mm²/s	1.9-6.0 mm²/s
Acid value	0.42 mg KOH/g	Max. 0.50 mg KOH/g
Flash point	173°C	Min. 93°C
Cetane number	56	Min. 47
Iodine value	112 g I₂/100 g	Max. 120 g I₂/100 g

Table 7: Results of related research work.

Study / Catalyst source	Alcohol	Feedstock	Optimum conditions	Yield (%)	Special feature	Reference
Present study - pure calcined coconut husk	Ethanol	WCO (FFA 4.41 %)	53 min, 6:1, 2.2 wt %	83.9	No chemical activation; dual-waste route	This work
Coconut-shell-pith / eggshell CaO-AC composite	Methanol	WCO	90 min, 10:1, 8 wt %	92.5	Dual-waste composite requiring impregnation	[7]
Citrus limetta peel ash	Methanol	Coconut oil	180 min, 6:1, 1 wt %	100	Food-grade oil feedstock	[6]
Biomass-boiler bottom ash	Methanol	Waste frying oil blend	120 min, 10:1, 8 wt %	95	Industrial ash source	[5]
Sawdust-derived sulfonated acid catalyst	Methanol	WCO (FFA 4.28 %)	480 min, 30:1, 10 wt %	81.2	Acid catalyst; long reaction time	[4]
Corn-cob ash	Methanol	Vegetable oil	120 min, 9:1, 3 wt %	96.8	Methanolysis only	[19]

Density measured at 15°C was 0.888 g/cm³, which falls comfortably within the prescribed range of 0.86-0.90 g/cm³. This value indicates that the fuel possesses appropriate mass-to-volume characteristics for efficient atomisation and combustion in compression-ignition engines.

Kinematic viscosity at 40°C was recorded as 5.1 mm²/s, well inside the allowable window of 1.9-6.0 mm²/s. The viscosity lies toward the upper half of the specification yet remains sufficiently low to ensure adequate fuel flow, pumpability and spray characteristics under typical operating temperatures.

The acid value of 0.42 mg KOH/g is below the maximum limit of 0.50 mg KOH/g. This low acidity confirms effective conversion of free fatty acids during the transesterification process and indicates a reduced risk of corrosion to engine components and fuel-system materials.

Flash point was determined to be 173°C, substantially higher than the minimum requirement of 93°C. The elevated flash point enhances the safety profile of the fuel during storage, handling and transportation by reducing the likelihood of accidental ignition.

Cetane number reached 56, exceeding the minimum threshold of 47. The higher cetane rating reflects favourable ignition quality, which is expected to promote shorter ignition delay, smoother combustion and reduced emissions of unburned hydrocarbons.

Iodine value was measured at 112 g I₂/100 g, remaining below the maximum limit of 120 g I₂/100 g. This result demonstrates an acceptable degree of unsaturation, thereby limiting the propensity for oxidative degradation and polymerisation during long-term storage.

Collectively, all six key properties satisfy the ASTM D6751 requirements, confirming that the optimised biodiesel meets the quality standards necessary for commercial diesel-engine applications. The compliance across density, viscosity, acidity, ignition characteristics and oxidative stability underscores the effectiveness of the selected process conditions in producing a fuel of suitable technical performance [18].

Comparison with related studies

Table 7 summarises the principal features of the present work alongside six recent investigations that also employed biomass-derived heterogeneous catalysts for the conversion of waste or non-edible oils.

The comparison shows that the present catalyst operates with ethanol rather than methanol, requires neither chemical impregnation nor hydrothermal activation, and still delivers a competitive yield from a high-FFA waste feedstock under comparatively mild conditions.

Conclusion/Recommendation

Calcined coconut husk yields a functional heterogeneous base catalyst whose silica-rich, potassium-containing structure and open porosity enable the ethanolysis of waste cooking oil. Under the optimised conditions identified by response-surface methodology an ethyl-ester yield of 83.9% was achieved. The product met all examined ASTM D6751 limits. The process utilises two abundant waste streams, avoids toxic reagents and energy-intensive chemical activation steps, and therefore constitutes a genuinely green route to biodiesel.

Future work should examine the long-term reusability of the catalyst under continuous-flow conditions, quantify metal leaching into the product, and evaluate the economic and life-cycle performance of the dual-waste process at pilot scale. Extension of the same simple calcination protocol to other potassium-rich agricultural residues is also warranted.

Conflict of Interest

The authors declare that there are no conflicts of interest regarding this article.

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References

- Patel K, Singh SK. Sustainable biodiesel from used cooking oil: A comparative life cycle, energy, and uncertainty analysis. *Environ Develop Sustain*. 2025; 27: 14755-14780.
- Yousuf M, Das AP, Masurkar P, Bag MK, Mitra D, Ray A. Recent advances and research in biodiesel as a sustainable, eco-friendly approach toward the green economy. *Environ Sci Eng*. 2025.
- Eremeeva AM, Marinets AR, Oleynik IL, Povarov VG. Recycling of waste cooking oils into a biodiesel fuel: Kinetics and analysis. *Recycling*. 2026; 11(2): 41.
- Lee DG, Yeom SH. Highly regenerable waste sawdust-derived acidic catalysts for multiple-cycle biodiesel production from waste cooking oil. *Biotechnol Bioprocess Eng*. 2026; 31: 63-72.
- Nahuelcura B, González ME, Gutierrez N, Nanculeo J, Romero-García JM. Biodiesel production from waste frying oil (WFO) using a biomass ash-based catalyst. *Catalysts*. 2024; 14(8): 553.
- Kaushik P, Kaur G, Hasan I. Biodiesel synthesis from coconut oil using the ash of Citrus limetta peels as a renewable heterogeneous catalyst. *Catalysts*. 2024; 14(8): 549.
- Rashad MA, Hussain M, Jamil F, Hamayun MH, Ahsan A, Akhter P, et al. Dual-waste valorization of eggshell and coconut shell-pith biomass into CaO/activated carbon catalysts: Synthesis, characterization, and structure-performance correlations in biodiesel transesterification. *Carbon Lett*.

- 2026; 36: 1321-1335.
8. Oparanti SO, Obebe EO, Fofana I, Jafari R. A state-of-the-art review on the potential of waste cooking oil as a sustainable insulating liquid for green transformers. *Appl Sci.* 2025; 15(14): 7631.
 9. Oyedele OA, Jekayinfa SO, Alade AO, Enweremadu CC. Catalytic evaluation of an optimized heterogeneous composite catalyst derived from fusion of tri-biogenic residues. *Biomass.* 2024; 4(4): 1219-1237.
 10. Esipovich AL, Kanakov EA, Charykova TA, Otopkova KV, Smirnov MA, Mityukova YA, et al. A comprehensive study on physicochemical properties of fatty acid esters derived from different vegetable oils and alcohols and their potential application. *Energies.* 2024; 17(24): 6407.
 11. Carvalho SC, Silva MMC, Siqueira AF, Melo MP, Giordani DS, Senra TOS, et al. Olfactory profile and stochastic analysis: An innovative approach for predicting the physicochemical characteristics of recycled waste cooking oils for sustainable biodiesel production. *Sustainability.* 2024; 16(22): 9998.
 12. Anuar MF, Fen YW, Zaid MHM, Matori KA, Khaidir REM. The physical and optical studies of crystalline silica derived from the green synthesis of coconut husk ash. *Appl Sci.* 2020; 10(6): 2128.
 13. Sim SF, Mohamed M, Lu NALMI, Sarman NSP, Samsudin SNS. Computer-assisted analysis of Fourier transform infrared (FTIR) spectra for characterization of various treated and untreated agriculture biomass. *Bioresources.* 2012; 7(4): 5367-5380.
 14. Husin H, Abubakar A, Ramadhani S, Sijabat CFB, Hasfita F. Coconut husk ash as heterogeneous catalyst for biodiesel production from Cerbera manghas seed oil. *MATEC Web Conf.* 2018; 197: 09008.
 15. Chaos-Hernández D, Hernández-Ramírez A, Ruiz-Ruiz E, Reyes-Vallejo O. Coconut-based heterogeneous catalysts for biodiesel production. *Bulg Chem Commun.* 2019; 51: 89-92.
 16. Faiz MSA, Azizan A, Hamzah F. Characterisation of silica extracted from coconut husk ash via acid and alkali treatments. *J Phys.* 2022; 2266(1): 012011.
 17. Thushari I, Babel S. Preparation of solid acid catalysts from waste biomass and their application for microwave-assisted biodiesel production from waste palm oil. *Waste Manag Res.* 2018; 36(9): 847-857.
 18. ASTM International. Standard specification for biodiesel fuel blendstock (B100) for middle distillate fuels (ASTM D6751-23a). 2023.
 19. Aleman-Ramirez J, Villamizar CP, Barragán-Trinidad M, et al. A new green heterogeneous solid catalyst from corn cob agricultural residue for biodiesel production. *Biomass Convers Bioref.* 2026; 16: 113.