

“Physicochemical Quality Assessment of Biodiesel Synthesized Using Novel Zeolite Catalysts from Waste Cooking Oils: Compliance with EN 14214 and ASTM D6751”

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Abstract

This research explores the transesterification of waste cooking oils (WCOs) over two new heterogeneous catalysts: Sn-RH-Zeolite and K₂CO₃-CaO-Zeolite. The catalysts were rice husk ash-derived chemically modified zeolite and screened for catalytic performance, structural stability by FTIR, XRD, and TGA methods. Both the catalysts yielded over 93% biodiesel under optimized transesterification conditions. Sn-RH-Zeolite was a better catalyst due to its dual Brønsted and Lewis acid nature, successfully catalyzing higher oils containing free fatty acids in. In addition, after five cycles of reuse, it maintained over 70% activity. Initially very active, K₂CO₃-CaO-Zeolite quickly deactivated as a result of structural breakdown and potassium and calcium species leaching. Viscosity, acid value, iodine value, flash point, and FFA content are some of the key physicochemical factors that were evaluated in biodiesel produced from leftover sunflower and mustard oil. Every value was discovered to be within the parameters, vis-a-vis fuel quality and engine compatibility. The results show the potential of modified zeolite catalysts for future sustainable production of biodiesel from degraded feedstocks. Future research should be focused on regeneration of damaged catalysts, scale-up, and integration with waste valorization methods for enhancing circular bio-economy and cleaner energy processes.

Keywords

Biodiesel, WCO, SRZ, KCZ, Transesterification, Catalyst Reusability, Physicochemical Properties, ASTM D6751, EN 14214, FFA.

1 Introduction

The growing global demand for renewable fuel, coupled with environmental issues and dwindling fossil fuel resources, has promoted increasing interest in the application of biodiesel as an alternative. There are certain benefits to biodiesel, which is created by employing a catalyst to transesterify oils or fatty substances with alcohol (mostly methanol) have some advantages of biodegradability, low sulfur content, enhanced lubricity, and reduced greenhouse gas emissions. Waste cooking oils (WCOs), generated in large quantities by households and commercial units, are low-cost and eco-friendly. WCOs are generally high in free fatty acids (FFAs), water, and degradation products due to frequent heating and therefore are a difficult substrate for transesterification. High FFAs lead to soap formation with alkaline catalysts and reduced biodiesel yield and purity. To overcome these, heterogeneous catalysts, especially zeolite-based ones, are chosen because of the ease of catalyst separation, recyclability, and low environmental footprints. Zeolites are excellent for esterification and transesterification because of their large surface area, regulated acidity, and heat stability. This paper explores two novel zeolite catalysts: Sn-RH-Zeolite (rice husk ash and tin derived) for dual acidic functionality, and K₂CO₃-CaO-Zeolite (post-treated natural zeolite) for basic catalysis. These were screened using a series of WCOs sunflower, mustard, soybean, groundnut, palm, and sesame oil over various frying cycles to represent real-time degradation. Biodiesel quality was tested against EN 14214 and ASTM D6751 standards, taking into account significant parameters such as acid value, viscosity, iodine value, flash point, and FFA content. Apart from yield, focus was on ensuring the final fuel met international performance and

safety standards. Additionally, performance of the catalyst through several cycles was evaluated using FTIR and XRD to screen for structural integrity and stability. The research also explores the effects of oil type and degradation degree on the quality of biodiesel and the implications of choosing the right catalyst-feedstock pair. In using waste feedstocks and agro-residues as feedstocks for the synthesis of catalysts, the research supports green energy generation and circular economy principles. The results allow decentralized biodiesel systems that are adequate for resource-poor communities, facilitating environmental sustainability and energy security.

2.1 Materials

Various types of waste cooking oils (WCOs) collected from typical Indian residential kitchen settings. The oils were collected after being utilized for frying typical deep-fried foods such as puris (fried wheat bread) and pakoras (vegetable fritters). For the purposes of realistically mimicking home frying operations and for various degrees of degradation, oils were collected after various degrees of reuse:

First frying stage (fresh oil for one-time frying session)

Second fry process (reusing oil once after first fry)

Third stage of frying (oil used twice or more)

Every subsequent frying process brings it one step nearer to thermal degradation, leading to measurable changes in the makeup of the oil. In particular, the oil oxidizes, hydrolyzes, and polymerizes, with increased free fatty acid (FFA) content, viscosity, and degradation products such as peroxides and carbonaceous residues.

2.2 Waste Cooking Oil Pre-treatment

To ensure quality and removal of interfering impurities before the transesterification reaction, the WCOs were pretreated in three steps as follows:

Filtration Process

WCOs are typically accompanied by particulate matter such as food particle residue, charred residue, and carbonaceous deposits. These solids have the potential to deactivate the catalyst and clog the reactor system. The oils were therefore first left to stand in beakers or containers to allow coarse particles to settle. The supernatant oil was further filtered through multiple layers:

A coarse filter or a muslin to trap big impurities

Fine grade filter paper (Whatman No. 1 or equivalent) for additional purification

This step improved oil clarity, reduced catalyst fouling, and offered smooth flow throughout the reaction.

Moisture Evaporation (Heating)

Water in WCOs can induce hydrolysis of triglycerides, which subsequently produces FFAs and soap, particularly when basic catalysts are employed. Hot-plate or heating mantle and stirring were employed to heat filtered oil to 110°C for 30 minutes for removal of residual water. The temperature was ample enough to drive off water without affecting the molecular structure of the oil. Cool-down and room temperature and storage for subsequent use were employed after the oil was cooled.

Storage Procedure

Proper pretreated WCO storage is required to avoid re-contamination and oxidation. Dried and filtered oils were stored in glass, stainless steel, or HDPE tight, light-tight containers. For short-term storage, to avoid oxidative degradation, the containers were kept in a dark, cool, and dry atmosphere. For longer storage, oils were kept at 4–10°C under refrigeration. This process ensured oil stability, reduced peroxide formation, and ensured consistent feedstock quality for the biodiesel conversion process.

2.3 Catalyst Synthesis and Characterization

Two heterogeneous zeolite-based catalysts, Sn-RH-Zeolite and K₂CO₃-CaO-Zeolite, were synthesized and characterized based on previously reported methods [Mina and Jayshree, 2025]. The Sn-RH-Zeolite catalyst was synthesized from rice husk ash-silica, followed by tin impregnation to create bidentate acid sites, well-suited for transesterification and esterification reactions simultaneously. By contrast, the K₂CO₃-CaO-Zeolite

catalyst was synthesized using sequential acid-washed natural zeolite, followed by impregnation with potassium carbonate and calcium oxide to form basic active sites well-suited for low-FFA feedstocks.

Both the catalysts were distinguished by: FTIR, XRD, TGA -Detailed synthesis protocols and full characterization data are presented in the above study:

2.4 Transesterification Reaction Setup

A 250 mL round-bottom flask with a single neck was used as a batch reactor for conducting the transesterification reaction. Uniform blending and constant temperature were ensured by a magnetic stirrer with a heating plate. A reflux condenser was attached to the flask so that the evaporation of methanol during the reaction was prevented. 50 mL of preheated waste cooking oil was used in each experiment. Methanol was used in a molar ratio of 9:1- 15:1 (methanol to oil). The catalyst was kept constant at 3-4 wt.% of the weight of oil. The mixture was agitated at 650 rpm and heated at 60 ± 5 °C for 180 minutes

2.5 Post-Reaction Processing

On reaction, the resulting mixture was allowed to set before poured into a separatory funnel. The funnel was allowed to remain undisturbed for 3-4 hours to enable clean separation into two different layers.

The top layer was biodiesel (fatty acid methyl esters, FAMES)

The bottom layer had glycerol, excess methanol, leftover catalyst, and soaps.

The biodiesel phase was then slowly harvested and washed multiple times with warm distilled water (45–50 °C) until the wash water was neutral ($\text{pH} \approx 7$). The biodiesel was then dried in an oven (110 °C) for 4 hours to eliminate any residual water and methanol. The final biodiesel samples were then stored in clean, amber bottles for subsequent physicochemical analysis

2.6 Determination of Fuel Properties

The following major fuel parameters of the produced biodiesel ensured fuel-grade compatibility (ASTM International, 2020; European Committee for Standardization, 2012).

2.5.1 Kinematic Viscosity (ASTM D445)

Viscosity at 40 °C was determined using a Redwood Viscometer. The time taken by a pre-fixed volume of biodiesel to flow through a calibrated orifice was determined and converted into kinematic viscosity (mm^2/s).

2.6.2 Acid Value (ASTM D664)

To ensure full solubilisation, a 0.5 g sample of biodiesel was dissolved in 50 cm^3 of neutralized ethanol and then heated in a water bath. After cooling, phenolphthalein was used as an indicator to titrate the mixture with 0.1 M KOH. The amount of remaining acidic components in the sample is indicated by the volume of KOH utilized. This was used to determine the fuel purity by calculating the AV. .

2.6.3 Iodine Value (EN 14111)

Iodine value was ascertained using the Wijs method, to which iodine monochloride was added to the unsaturated fatty acid chains. The iodine absorbed was ascertained by titration against sodium thiosulfate as an indicator with starch.

2.6.4 Flash Point (ASTM D93)

Flash point was measured with a Pensky–Martens closed-cup test apparatus. It is an important factor in the transportation and safe storage of biodiesel.

2.6.5 Free Fatty Acid (FFA) Content

FFA content was indirectly measured by acid value which assists in determining the esterification effectiveness and the purity of the product. According to Abatyough et al. (2019). This assisted in determining the esterification effectiveness and product purity.

$$\% \text{ FFA} = \frac{\text{Acid Value}}{1.99}$$

2.7 Data Interpretation and Quality Compliance

These values obtained through measurement for all the physicochemical parameters were also verified with standard limits mentioned in biodiesel standards. Samples meeting or even exceeding both standard limits were found to be fit for the use as biodiesel in compression ignition engines.

2.8 Yield Calculation

Biodiesel yield (%) was calculated using the following equation:

$$\text{Yield (\%)} = \frac{\text{Weight of biodiesel produced}}{\text{Weight of oil used}} \times 100$$

3.1 Catalyst Performance Evaluation

The catalytic efficiency of heterogeneous zeolite-based catalysts was critically assessed by evaluating biodiesel yield under varying operational parameters and across multiple reaction cycles. Two catalysts were compared Sn-RH-Zeolite and K_2CO_3 -CaO-Zeolite both synthesized from low-cost and sustainable precursors. This section outlines the performance metrics in terms of yield, influence of reaction conditions, and cycle-wise reusability trends.

3.1 Catalyst Performance Evaluation

Catalytic efficiency of heterogeneous zeolite-based catalysts was evaluated critically by contrasting biodiesel yield under different operating conditions and for multiple reaction cycles. The efficiency of two catalysts prepared from cost-effective and ecofriendly precursors Sn-RH-Zeolite and K_2CO_3 -CaO-Zeolite was compared. The performance attributes in terms of yield, effect of reaction conditions, and cycle-wise reuse trend are highlighted in this section.

3.1.1 Sn-RH-Zeolite and K_2CO_3 -CaO-Zeolite Yield Comparison

Biodiesel yield is a critical measure of catalyst efficiency. Under ideal conditions, both Sn-RH-Zeolite and K_2CO_3 -CaO-Zeolite were active catalysts, with yields of more than 90%. For instance, Sn-RH-Zeolite yielded 93.6% using waste sunflower oil, and K_2CO_3 -CaO-Zeolite yielded 93.2% using waste mustard oil. This difference in performance is reflective of catalyst compatibility with feedstock characteristics. Medium free fatty acid (FFA) and higher iodine value sunflower oil was favored by the twin acidity of Sn-RH-Zeolite, allowing for both esterification and transesterification. Highly basic K_2CO_3 -CaO-Zeolite, by contrast, was compatible with low-FFA mustard oil but would perform less efficiently on more degraded oils due to the risk of soap formation. These results suggest that Sn-RH-Zeolite would be more suited to moderately degraded or unsaturated oils, while K_2CO_3 -CaO-Zeolite would be more suited to cleaner, low-FFA oils.

3.1.2 Effect of Methanol-to-Oil Ratio, Catalyst Loading, and Reaction Time

Transesterification is an equilibrium-controlled reaction, and equilibrium towards methyl ester production can be attained by optimizing the important parameters. Three parameters were controlled in this work, i.e., methanol-to-oil mole ratio, catalyst loading, and reaction time. All reactions were conducted at a constant temperature of 65 °C.

Table:1 Comparison of Transesterification Parameters for Sn-RH-Zeolite and K₂CO₃–CaO–Zeolite Catalysts

Parameter	Sn-RH-Zeolite	K ₂ CO ₃ –CaO–Zeolite
Methanol-to-Oil Ratio	9:1	15:1
Rationale	Sufficient for dual-acid catalyst working with moderately impure oils	Excess methanol shifts equilibrium and compensates for FFA sensitivity in base-catalyzed reactions
Catalyst Dosage	3 wt. %	4 wt. %
Implication	Lower amount needed due to better dispersion and surface activity	Higher amount required to ensure adequate active site availability
Reaction Time	180 minutes	120 minutes
Reason	Slower reaction due to combined esterification and transesterification	Faster kinetics typical of basic catalysts

These results indicate that Sn-RH-Zeolite can work effectively at decreased catalyst loading and methanol feed, but at the cost of increased reaction time. Alternatively, the K₂CO₃-CaO-Zeolite provides quicker conversion but at the cost of strict feedstock conditions and increased methanol feed.

3.1.3 Catalyst Reusability and Yield Degradation over Cycles

Reusability of heterogeneous catalysts is an effective consideration for their economic and operational feasibility in the process of biodiesel manufacture. In the current study, Sn-RH-Zeolite and K₂CO₃-CaO-Zeolite were employed in four successive transesterification cycles under the same reaction conditions to assess long-term deactivation of catalytic performance. The catalysts were separated after each cycle, washed thoroughly with ethanol to desorb absorbed glycerol and excess oil, and dried at 100 °C for 4 hours before reuse.

Catalyst Reusability Summary

Sn-RH-Zeolite showed good reusability, and yield of biodiesel declined from 94.2% in Cycle 1 to 76.5% in Cycle 4, i.e., an 17.7% decline. This decline is mainly attributed to Sn species leaching and blockage of pores, but the catalyst also exhibited good activity due to its dual acid sites and structural stability.

K₂CO₃-CaO-Zeolite started at 92.6% yield in Cycle 1 and dropped to 72.9% in Cycle 4 a decline of 19.7%. Spurred degradation was the result of leaching of K and Ca species, reduced surface basicity, and soap formation, especially in the case of very slightly impure oils.

Overall, Sn-RH-Zeolite showed better reusability and stability and was therefore more suitable for treatment of variable-quality WCOs in repeated cycles.

To better show the relative trend in catalyst performance, the following table and the following explanation summarize the biodiesel yield in four cycles:

Cycle No.	SRZ	KCZ Yield (%)
Cycle 1	94.2	92.6
Cycle 2	89.7	87.1
Cycle 3	83.4	80.2
Cycle 4	76.5	72.9

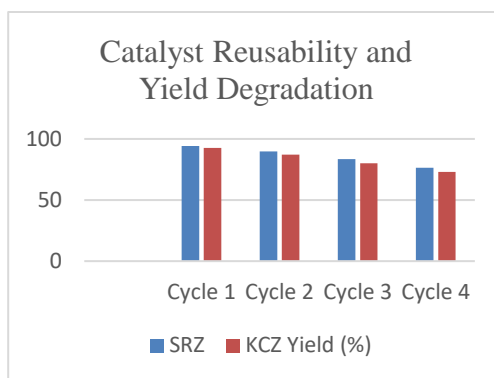


Table 2- Catalyst Reusability

Fig. 1 Graphical Representation

3.2 Physicochemical Properties of Biodiesel

Physicochemical properties of WSO biodiesel and WMO biodiesel are shown in Table 3. These are of utmost significance in the determination of fuel properties within acceptable limits. Viscosity of the samples of the biodiesel was determined at 40 °C by a Redwood Viscometer according to ASTM D445. WSO biodiesel registered 4.3 mm²/s viscosity, whereas WMO biodiesel registered 4.7 mm²/s slightly higher viscosity. Both are within the acceptable range (1.9–6.0 mm²/s) and are an indication of excellent diesel fuel application flow characteristics with no blending or additives. Acid value is directly related to the FFAs left behind, which corrode engine parts. According to ASTM D664, titration analysis showed acid value to be 0.017 mg KOH/g in the case of WSO biodiesel and 0.37 mg KOH/g in the case of WMO biodiesel. Both were below the 0.50 mg KOH/g standard limit, indicating that FFAs were successfully converted to methyl esters by the catalysts. Iodine value is an indication of the state of unsaturation in the biodiesel. Higher iodine values indicate higher unsaturation, which lowers oxidative stability. WSO biodiesel registered around 114 g I₂/100 g of iodine value, whereas WMO biodiesel registered around 107 g I₂/100 g of iodine value. Both were below the 120 g I₂/100 g limit of EN 14214, although WSO contained slightly higher unsaturation due to the inherent linoleic acid content. Flash point is a safety parameter of prime importance in storage and transportation. Pensky-Martens closed-cup tester readings as per ASTM D93 showed flash points at 170 °C for WSO biodiesel and 150 °C for WMO biodiesel. These are significantly higher than the ASTM specification of 93 °C and the EN specification of 101 °C, suggesting complete removal of residual methanol and safe storage. Free fatty acid (FFA) content, not explicitly discussed in ASTM or EN standards, was estimated from acid values (Abatyough et al., 2019). FFA content was found to be ~0.1% for WSO biodiesel and ~0.6% for WMO biodiesel. The higher FFA content in WMO could be due to extended frying cycles, which hydrolyze triglycerides to FFAs. This higher FFA content reduces the transesterification efficiency and calls for increased tolerance of the catalyst. Overall,

both the samples of biodiesel complied with international standards, with WSO biodiesel showing marginally superior quality parameters.

Table 3. Physicochemical Properties of Biodiesel Samples

Property	EN 14214 Standard	ASTM D6751 Standard	WSO Biodiesel	WMO Biodiesel
Viscosity (40 °C)	3.5–5.0 mm ² /s	1.9–6.0 mm ² /s	4.3 mm ² /s	4.7 mm ² /s
Acid Value	≤ 0.50 mg KOH/g	≤ 0.50 mg KOH/g	0.017 mg KOH/g	0.37 mg KOH/g
Iodine Value	≤ 120 g I ₂ /100 g	Not Specified	114 g I ₂ /100 g	107 g I ₂ /100 g
Flash Point	≥ 101 °C	≥ 93 °C	170 °C	150 °C
FFA Content (%)	Not Specified	Not Specified	~0.1%	~0.6%

3.2.6 Analysis of Variation

Variations in biodiesel qualities can be explained by the nature of the feedstock and the level of degradation that happens during frying. WSO, having lower FFA and improved oxidative stability, had slightly better acid and iodine values for the biodiesel. WMO, due to its possible degradation through cooking, had slightly higher acid and FFA values but within specifications. Such variation emphasizes the need to use the appropriate catalysts and pretreatment processes to handle varying qualities of waste oil.

3.3 FT-IR Analysis of WMO Biodiesel

FTIR spectrum of waste mustard oil (WMO) biodiesel was recorded from the wave number range of 400–4000 cm⁻¹ to detect the various functional groups present in biodiesel sample. FTIR spectrum of WMO

biodiesel is shown in Fig.2, which shows the presence of methylene stretching vibrations at 2924.45 cm^{-1} that agrees with the presence of aliphatic CH_2 chains typical of FAME. The presence of a sharp peak within the range of $1737.92\text{--}1742.46\text{ cm}^{-1}$ shows carbonyl (C=O) group typical of ester linkages, and hence the successful transesterification of triglycerides to methyl esters. In addition, other bands at 2854.10 cm^{-1} and 1455.30 cm^{-1} show symmetric CH_2 stretching and bending vibrations, respectively. Moreover, some weak bands were noted between 700 and 500 cm^{-1} at 551.99 cm^{-1} and 472.25 cm^{-1} , which are typical of skeletal vibrations typical of long-chain hydrocarbons. The absence of a broad O-H peak at 3400 cm^{-1} indicates that excess H_2O and unreacted FFA were removed successfully. The observation is corroborative of low FFA content and greater purity of biodiesel product, as evidenced by the reported physicochemical values.

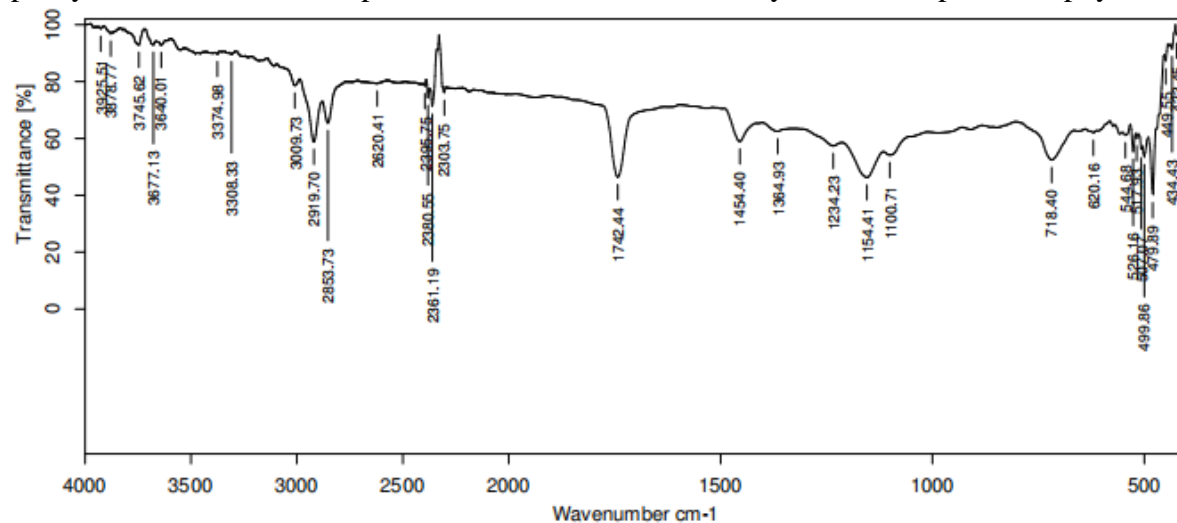


Fig. 2 FTIR of WMO

3.3 Catalyst Reusability and Stability

The reusability of heterogeneous catalysts is essential for cost-effective and sustainable biodiesel production. Both Sn-RH-Zeolite and $\text{K}_2\text{CO}_3\text{-CaO-Zeolite}$ catalysts were evaluated over five reuse cycles to assess stability and performance.

3.3.1 Sn-RH-Zeolite

FTIR Analysis showed retention of characteristic peaks (-OH at $\sim 3440\text{ cm}^{-1}$, Si-O-Si at $1000\text{--}1100\text{ cm}^{-1}$, Sn-O-Si at $\sim 960\text{ cm}^{-1}$). Slight reduction in hydroxyl bands was observed, with no signs of carbon deposition shown by fig. 3.

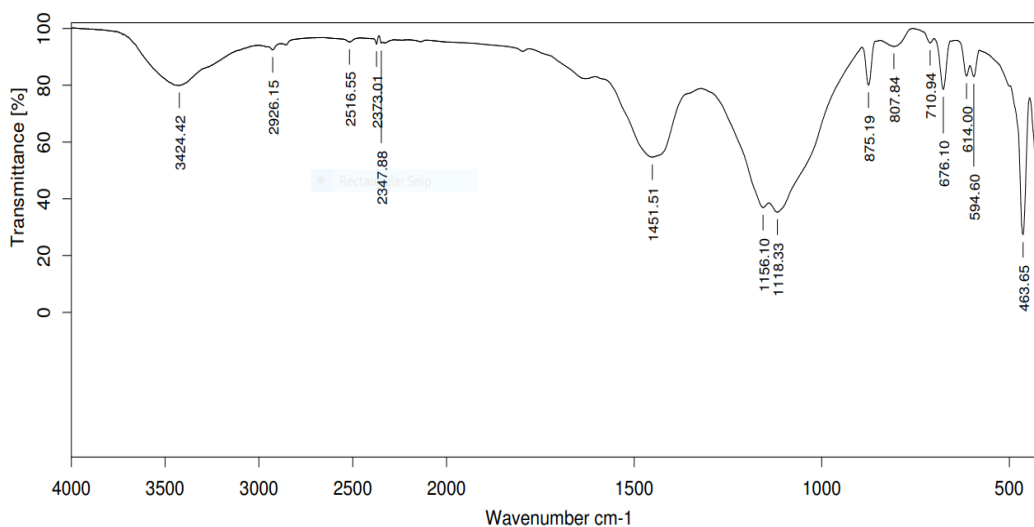


Fig.3- FTIR of SRH Catalyst

XRD Analysis confirmed that major diffraction peaks ($2\theta = 8.73^\circ, 25.43^\circ$) remained unchanged, with minor peak broadening indicating slight crystallite degradation. No impurity phases like SnO_2 were detected.

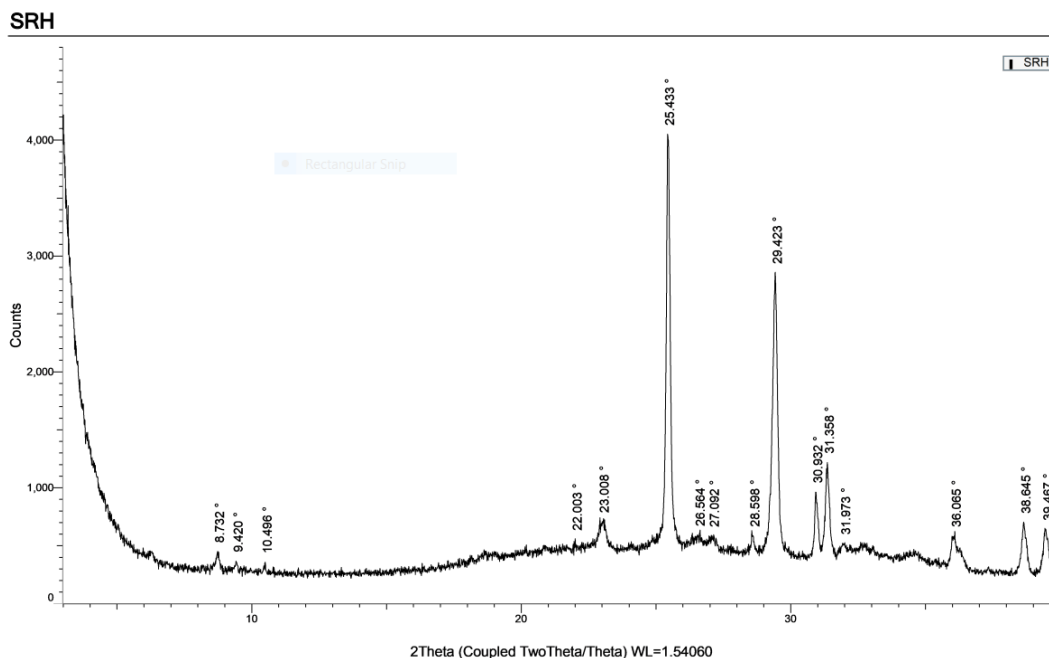


Fig.4-XRD of SRH Catalyst

Yield Trend fell from 94.2% (Cycle 1) to 70.2% (Cycle 5) primarily as a result of Sn leaching and pore blocking, but retained high catalytic activity.

Implication: Exhibited good reusability and structural stability, showing that it can be used with high-FFA waste oils and continuous use systems.

3.3.2 K_2CO_3 -CaO-Zeolite (KCZ)

Decrease in Yield Performance over cycles:

Palm oil: 96% \rightarrow 82.4% (Cycle 3)

Soybean oil: 76% \rightarrow 59.3% (Cycle 5)

Groundnut oil: 86.4% \rightarrow 72.5% (Cycle 3)

Reduction was associated with leaching of Ca^{2+} and K^+ , surface fouling, and physical deterioration. FTIR Analysis showed recurring -OH peaks ($\sim 3440\text{ cm}^{-1}$) with decreasing carbonate bands ($1460, 875, 712\text{ cm}^{-1}$), indicating partial loss of K_2CO_3 .

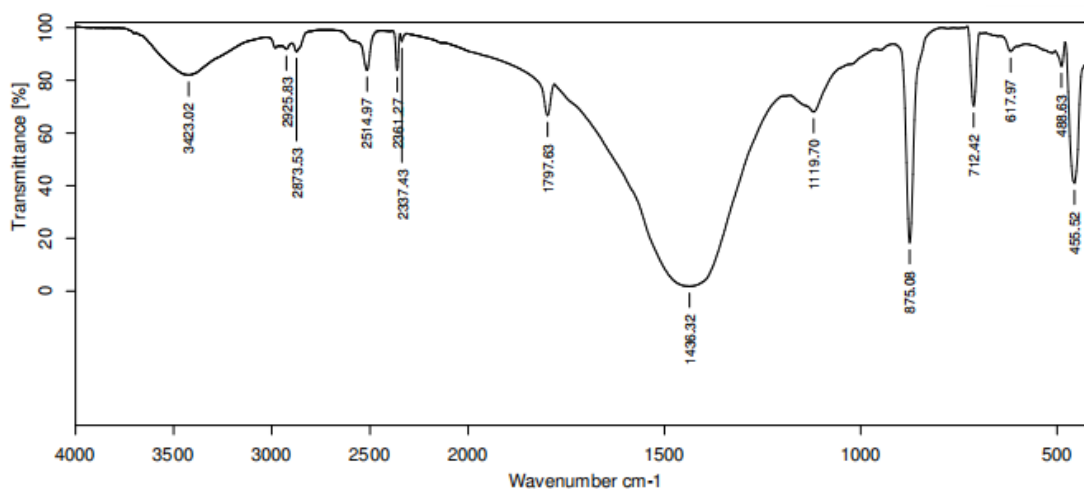


Fig.5-FTIR of KCZ Catalyst

XRD Patterns maintained major reflections ($29.39^\circ, 36.01^\circ, 39.45^\circ$), although with decreased intensities (10–15%), indicating slow loss of crystallinity. Zeolite support was structurally stable (22° – 28°).

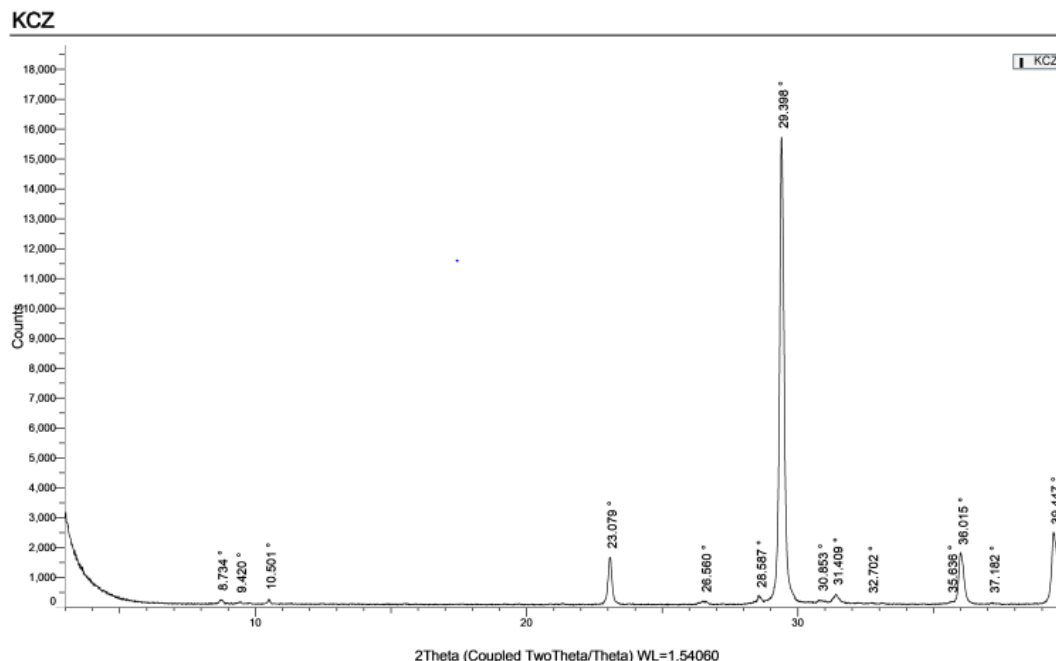


Fig. 6 - XRD of KCZ Catalyst

Conclusion and Future Scope

This research effectively proved the feasibility of two modified heterogeneous catalysts Sn-RH-Zeolite and K_2CO_3 -CaO-Zeolite for the effective production of biodiesel from WCO. Both catalysts produced biodiesel yields of over 93% under the optimal conditions. Sn-RH-Zeolite was more resistant to FFAs and more reusable with structural stability established through FTIR and XRD. K_2CO_3 -CaO-Zeolite exhibited higher performance loss on account of leaching and surface modification. The resulting biodiesel was in accordance internationally accepted biodiesel specifications for viscosity, acid value, iodine value, flash point, and FFA content, and it was suitable for diesel engine fuel. Variations in fuel quality reflected the nature and extent of degradation of the feedstock, highlighting the importance of proper pretreatment and catalyst selection. Future research could entail catalyst regeneration, process scale up, and investigation of varied feedstocks. Kinetic modelling, life cycle assessment, and glycerol valorization could further increase the environmental as well as economic sustainability of the process. Overall, these catalysts offer a sustainable route for biodiesel production and make a significant contribution to circular bio-economy strategies.

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