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Synthesis and Characterization of Catalysts from Rice Husk and Egg Shell for Production of Biokerosene

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Abstract: *The* increasing demand for sustainable and low-cost catalysts for biofuel upgrading has stimulated research into the utilization of agricultural wastes such as rice husk and eggshell for catalyst synthesis. This paer discusses the preparation, characterization, and catalytic potential of $NiCo/SiO_2$and CaO/Al_2O_3. Catalysts synthesized from rice husk and eggshell, respectively. Rice husk serves as an abundant renewable source of silica, while eggshell is a rich source of calcium carbonate that can be converted into calcium oxide through calcination. The synthesized catalysts were characterized using X-ray diffraction (XRD), X-ray fluorescence (XRF), Brunauer–Emmett–Teller (BET) surface area analysis, and thermogravimetric analysis (TGA) to evaluate their structural, thermal, and surface properties. Results revealed that the catalysts possess high surface area, mesoporous structures, and good thermal stability suitable for heterogeneous catalytic applications. $NiCo/SiO_2$ catalyst exhibited enhanced surface area and porosity due to silica derived from rice husk, while The CaO/Al_2O_3 catalyst showed excellent basic characteristics. The study highlights the importance of converting agricultural waste into valuable catalytic materials for biofuel production and environmental sustainability. The utilization of waste-derived catalysts offers economic, environmental, and industrial benefits by reducing waste disposal problems and promoting sustainable energy development.

Keywords*: Synthesis, Characterization, Catalyst, Rice husk, Eggshell and Biokerosene.*

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Introduction

Renewable energy from biomass had been recognized by scientists as a promising source of energy with the potential to play a significant role in complementing the depleting fossil fuel resources in the production of fuels and chemicals (Özçimen and Karaosmanoğlu, 2004; Ambursa *et al*., 2016).

Bio-kerosene (jet fuel) is a form of biofuel that can serve as a direct substitute for aviation kerosene from fossil fuels, and can be produced by hydro-treating bio-oils from plants, animal fats, or other forms of biomass feed stocks (Ekpenkhio, 2024). Bio kerosene possess comparable fuel properties to the conventional kerosene but offers a number of ecofriendly benefits, including lower emission of oxides of carbon, nitrogen, and sulfur (COx, NOx and SOx), thereby, minimizing emissions of greenhouse gases (Kumar *et al*., 2012; Selvam etb al., 2025).

Over the past years, catalytic upgrading of bio-fuels has gain prominence among researchers due to its potential to improve the quality, and yield of the bio-oil production process. Catalyst plays an important role in the different bio-fuel upgrading processes such as hydro treating, cracking and trans-esterification (Ko *et al*., 2012 ; Zhao *et al.*, 2017 ; Kundu *et al.*, 2024).

The most widely used heterogeneous catalysts in bio-oil upgrading are metals and their oxides due to their high stability, activity, selectivity and recyclable nature. Notable examples of heterogeneous catalysts active metals and support reportedly used for bio-fuel upgrading includes Ni, Pd, Pt, Biochar, carbon nanotubes etc (Wang *et al.*, 2012 ; Zhang *et al.*, 2013 ; Nguyen *et al.*, 2016 ;Cheng *et al.*, 2018 ; Carrasco *et al.*, 2024). These catalysts have been reported to improve the yield of biodiesel from triglycerides, improve quality of bio-oil, and increase fuel stability.

Several researches have explored the use of different heterogeneous catalysts for bio-oil upgrading, and these include supported metals, metal oxides and zeolites (Wang *et al.*, 2012; Sihombing *et al*., 2023).

The use of agricultural wastes such as rice husk and egg shell offer a cheaper and renewable and environmentally friendly alternative source of heterogeneous catalysts for bio-oil upgrading due to their low cost, availability, and biodegradable nature. Rice husk is considered a waste with high resistance to decomposition and has a low nutritional value to animals and also being difficult to digest. Rice husks also have low calorific value (3585 kcal kg^{-1}) and high ash content when burnt (Ameri *et al*., 2019; Tsai *et al*., 2025). The ash contains nearly 95% silica and is an important renewable source of silica. In addition, the ash contains potassium, sodium, magnesium, calcium, iron, phosphorus and much smaller quantities of other elements (Bakar *et al.*, 2016; Hamidu *et al*., 2025). Egg shells are useful in the preparation of heterogeneous catalysts. Eggshell CaO is a promising green catalyst in biofuel production and has many bioactive compounds exhibiting high economic benefits. Internationally, about 250,000 tons of eggshell waste is released annually (Faridi and Arabhosseini, 2018). Egg shells are agricultural bio-waste materials that are biodegradable, recyclable and biocompatible (Pedavoah, 2018). It is mainly composed of calcium carbonate ($CaCO_3$), which accounts for about 93.6% by weight, followed by calcium triphosphate (0.8%), which accounts for about 10% of an egg. This research aim to synthesis and characterize catalysts from rice husk and egg suitable for bio kerosene production.

Material and Methods

Materials

Information on the instruments, chemicals and methods used in this research are presented in the following subsections.

- ***Instruments***

The instruments used in this research work are listed in **Table 2.1**. The instruments were used in accordance to manufacturers' operation guidance.

Table 2.1: List of Instruments

Instruments	Model	Manufacturer
Analytical Balance	AW320	Shimadzu, Japan
FT-IR spectrometer	Cary 630 FTIR	Agilent technologies, USA
X-ray diffraction	Miniflex 600-C	Rigaku Corporation, Japan
BET analyzer	-	-
X-ray Fluorescence analyzer	-	-
Furnace	AAF 1100	AAF 1100

- ***Reagents***

The reagents used in this research work are listed in **Table2.2**. All the chemicals were of analytical grade and were used as purchased without prior purification.

Table 2.2: List of Reagents Used

Sodium hydroxide	NaOH	≥97.0%	Sigma Aldrich, USA

- ***Source of samples***

The samples used in this research are rice husk and egg shell. The rice husk and egg shell were collected from a local rice mill factory located at Gidan Dare, Sokoto North and Local tea sellers (Mai Shayi), usmanu Danfodiyo University minimart, Sokoto respectively. They were thoroughly washed and sun dried.

Methods

- ***Catalyst preparation***

a) Synthesis of silica form rice husk

The rice husk ash was prepared by open air burning to get the rice husk ash. To prepare the catalyst, silica from rice husk ash was treated by impregnation and calcination processes. The silica was mixed with 1M NaOH solutions with ratio 1:10. The mixture was heated at 100 °C for 1 h at a stirring speed of 350 rpm, filtered and dried at 110 °C. The silica was then calcined at 500 °C for 2 hours (Tsai *et al*., 2025).

b) Synthesis of CaO from eggshell

CaO catalyst was prepared through the process of calcining the egg shell. The egg shell was washed thoroughly cleaned using tap water followed by rinsing with distilled water. The cleaned b) egg shell was dried in a hot oven at 105°C for 24 hours. Once dried, the egg shell was grinded into small particles and calcined in a muffle furnace with under air at 900°C for 2.5 hours (Mudi *et al.*, 2023). Both calcined rice husk and egg shell were stored in an air tight container for later use.

c) Synthesis of $NiCo/SiO_2$ and CaO/Al_2O_3

$NiCo/SiO_2$ catalyst was prepared using wet impregnation method by dissolving 11.22g of Nickel nitrate and 5.36g of cobalt nitrate and in 50 mL of deionize water to form an aqueous solution, and 20g of Silica powder was gradually added to the aqueous solution while stirring at 200 r.p.m. The mixture was then stirred at 400 r. p.m. and 70 ^{O}C using a hot plate magnetic stirrer to evaporate all the water, leaving behind a wet solid catalyst that was dried overnight at 80^{o}C in the oven (Mudi *et al.*, 2023). The resulting $NiCo/SiO_2$ catalyst was calcined at 500^{o}C for 5 hours. Similar procedure was used to prepare CaO/Al_2O_3 catalysts.

- ***Catalyst characterization***

a) X-ray diffraction (XRD) and X-ray fluorescence (XRF) analyses

The powdered X-ray Diffraction (PXRD) analysis was carried out using the Rigaku MiniFlex 6G equipped with the Rigaku SmartLab Software Situated at Central Advance Science Laboratory Complex (CASLAC), Usmanu Danfodiyo University, Sokoto. The measurement was done with Cu-Kα radiation (40Kv, 15mA) using a Sc-70 detector recorded over 3 – 90º 2 range at a speed of 10.00º/min. The basal spacing (d001) values were also determined from XRD patterns. The X-ray fluorescence (XRF) analyses was carried out using EDXRF analyzer at Advance Science Central Laboratory, Umaru Musa Yar'Adua University, Katsina.

b) Thermo-gravimetric analysis (TGA)

Thermo-gravimetric analysis is an analytical technique in which weight loss of the sample is measured with respect to time or temperature. In this study, it was used to examine the thermal stability of the rice husk and egg shell catalysts. A PerkinElmer TGA 800 equipment was used to carry out thermo-gravimetric analyses of the samples. About 17.5 mg of the catalyst was placed on the zeroed sample pan, nitrogen flow switched on and the sample loaded onto the furnace.

Thermo-gravimetric analysis was performed on the catalysts under nitrogen atmosphere (purity 99.9%) and the temperature ramped from 25°C to 800°C at a rate of 10°C per minute. During TGA analyses, sample weight changes over the temperature range are continuously recorded as the gaseous products are removed.

c) Surface area and pore volume determination

The surface area, pore width and pore volume analyses of the prepared catalysts were determined using N_2 adsorption isotherms at 77.35K using Quantachrome Instrument (Version 11.03). The samples were degassed for 3 hours at 250ºC in a vacuum. The surface area was was determined using multiple point Brunauer-Emmet-Teller (BET) method. The pore volume and pore size were estimated using Barrett-Joyner-Halenda (BJH) method (Brame and Griggs *et al*., 2016).

Results and Discussion

Catalyst Characterization

Catalyst characterization is a crucial aspect of catalyst development. There is a strong connection between the structural features of heterogeneous catalysts and their catalytic performance. This stage of catalyst development uses physical and chemical methods to evaluate important catalytic properties of the catalysts and also provide a clear understanding of the relationship between catalyst structure and its performance.

The obtained calcined rice husk and egg shell were analyzed using Brunauer-Emmett-Teller (BET) surface area, Thermo-gravimetric (TGA), X-ray Fluorescence Spectroscopy (XRF), Fourier Transform-infrared (FT-IR) spectroscopy and X-ray diffraction (XRD) analyses.

➢ ***X-ray Diffraction Spectroscopy (XRD) analyses***

X-ray diffraction (XRD) is a rapid technique primarily used to examine the degree of crystallization, which are the size, composition and crystal structure of pure samples. **Figure 3.4** shows the XRD diffractogram of the calcined egg shell and rice husk.

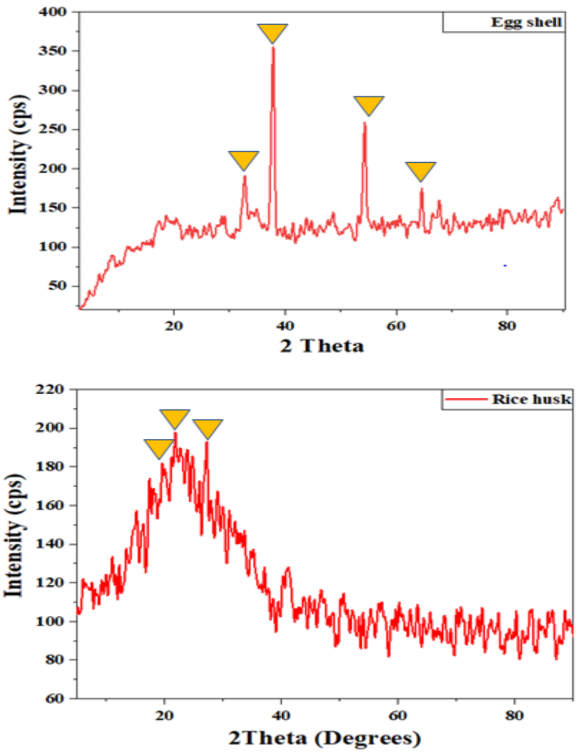


Figure 3.4:X-ray Diffraction Spectroscopy (XRD) analyses

The XRD analyses of egg shell showed a diffraction peak of the crystalline phase which is the main component of egg shell (**Fig 3.4a**). The main peaks were observed at 2θ values of 32.2º (111), 37.4º (200), 53.9º (22064.2º (311) and 67.4º (222), which are the characteristic peaks of calcium oxide obtained from the decomposition of calcium carbonate at high temperature of 700 ºC – 800 ºC (Naemchanthara, *et al*., 2008; Park *et al*., 2007) .

The XRD of the rice husk catalyst **(Figure 3.4b)** revealed a broad peak at 2θ value of 22º for amorphous cellulose, peaks at 2θ =20-25, and 26.6 are for quartz (SiO_2) (Park , 2007; Mijan , 2014). This confirm that the rice husk catalyst mainly contain amorphous SiO_2.

The XRD analyses of the synthesized catalyst showed that the egg shell is made of primarily calcium oxide as the mineral phase while the rice husk catalyst is made of mainly silica as the major component. Both CaO and Silica have proven good candidates for bio-oil upgrading reactions reported in the literature (Yi *et al*., 2019; Zhang et al., 2024; Böhme *et al*., 2025).

➢ ***BET Pore Size Distribution Analysis***

The pore size distribution analysis provides important information regarding the textural properties and accessibility of active sites within the catalyst structure (Kesserwan, 2021). The CaO/Al_2O_3 catalyst showed a relatively uniform pore size distribution with mesoporous characteristics, which favors efficient diffusion of reactants and products during catalytic reactions (Kesserwan, 2021). The mesoporous structure can be attributed to the incorporation of alumina support, which enhances pore development and dispersion of CaO particles obtained from eggshell waste (Kesserwan, 2021).

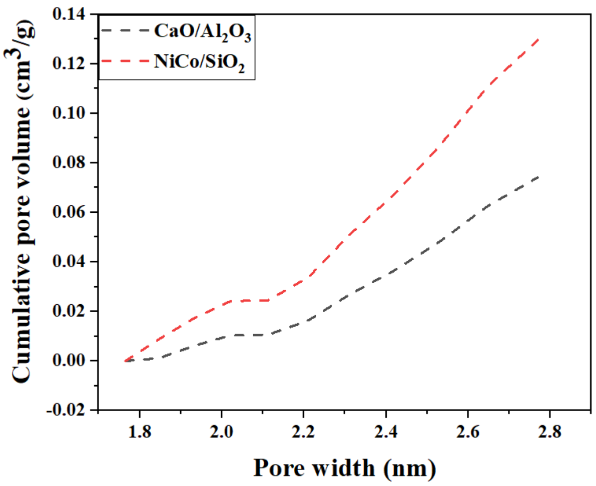


Figure 2: Pore Size Distribution of CaO/Al_2O_3 and $NiCo/SiO_2$ catalysts

The $NiCo/SiO_2$ catalyst also exhibited a well-developed mesoporous structure with narrow pore size distribution (Alshammari *et al*., 2026). The silica synthesized from rice husk contributed significantly to the formation of highly porous channels and improved surface morphology (Alshammari *et al*., 2026). The incorporation of Ni and Co metals onto the silica support may have slightly altered the pore arrangement but maintained sufficient pore accessibility for catalytic activity (Alshammari *et al*., 2026). The mesoporous nature of both catalysts is advantageous because it enhances mass transfer, increases active site accessibility, and improves overall catalytic performance.

➢ ***BET Surface area, pore sizes and pore volume***

Three important properties of heterogeneous catalysts are the catalyst surface area, pore size and pore volume.

The surface area, pore volume and pore size of the CaO/Al_2O_3 and $NiCo/SiO_2$ catalysts were determined using N_2 adsorption-desorption isotherms as shown in **Table 1.**

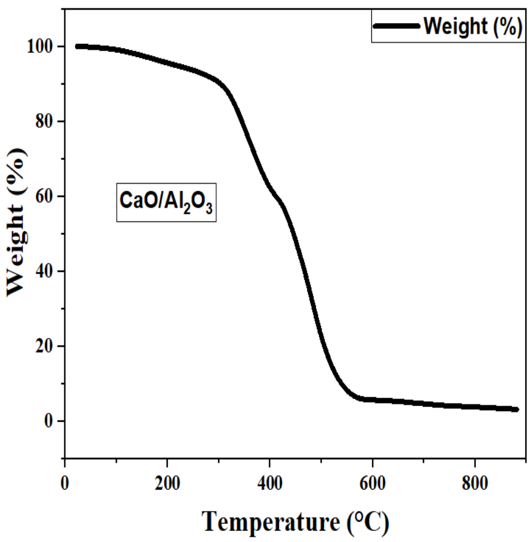
Table 1: BET analysis showing the catalyst surface area, pore diameter and pore volume

Catalyst	**Surface area (m2/g)**	**Pore Diameter (nm)**	**Pore Volume (cc/g)**
CaO/Al_2O_3	266.808	2.960	0.144
$NiCo/SiO_2$	397.840	2.820	0.241

The $NiCo/SiO_2$ catalyst exhibited a significantly higher surface area of 397.840 m^2/g, pore diameter of 2.820 nm, and pore volume of 0.241 cc/g. The high surface area is mainly attributed to the highly porous silica synthesized from rice husk, which promotes better dispersion of Ni and Co active metals (Alshammari *et al*., 2026). The larger pore volume indicates improved porosity and greater adsorption capacity, which are beneficial for catalytic applications requiring efficient reactant diffusion and active site accessibility (Alshammari *et al*., 2026). The BET results therefore suggest that $NiCo/SiO_2$ may exhibit superior catalytic performance due to its higher surface area and pore volume. pore diameter obtained for the synthesized catalysts are in consistent with and in some cases surpasses that of some previously reported catalysts for biokerosene production in literatures (Neuling and Kaltschmitt, 2015 ; Al-Muttaqii *et al*., 2019 ; Achinas *et al*., 2021).

- ***Thermo-gravimetric and Differential Analyses (TGA)***

Thermogravimetric analysis (TGA) was used to evaluate the thermal stability and decomposition behavior of the synthesized CaO/Al_2O_3 and $NiCo/SiO_2$ catalysts. The CaO/Al_2O_3 catalyst synthesized from eggshell-derived CaO exhibited a gradual weight loss with increasing temperature, which can be attributed to the removal of physically adsorbed moisture, volatile organic compounds, and decomposition of residual carbonate species present in the eggshell precursor (Kesserwan, 2021).



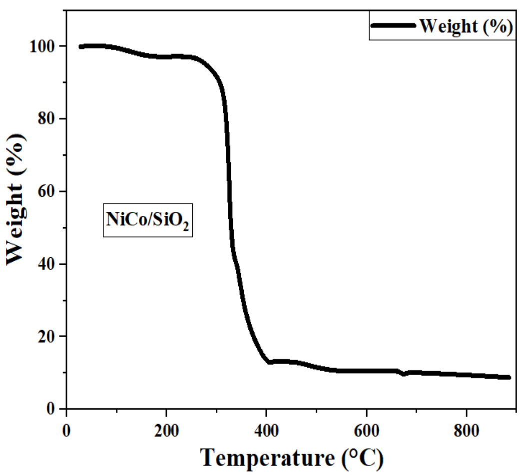


Figure1: Thermogravimetric analyses of a) CaO/Al_2O_3 and b) $NiCo/SiO_2$

The initial weight loss observed at lower temperatures is mainly associated with dehydration processes, while the minor weight reduction at intermediate temperatures may result from decomposition of calcium carbonate residues into CaO and CO_2 (Kesserwan, 2021). At higher temperatures, the catalyst displayed improved thermal stability, confirming successful calcination and formation of stable CaO/Al_2O_3 phases suitable for catalytic applications (Kesserwan, 2021).

Similar catalyst stability was observed for some reported catalysts used for biokerosene production (de Sausa *et al*., 2021; de Medeiros *et al*., 2022).

- ***X-Ray Fluorescence Spectroscopy***

The mineral content and their percentage in the egg shell and rice husk catalysts were assessed using X-Ray Fluorescence Spectroscopy as shown in the **Table 3.1**

Table 2: XRF analysis of CaO/Al_2O_3 and $NiCo/SiO_2$ catalysts

Catalyst	**Compound**	**Mole %**
1) CaO/Al_2O_3	CaO	51.77
	Al_2O_3	35.51
	SiO_2	8.295
2) $NiCo/SiO_2$	NiO	17.164
	SiO_2	65.9
	Al_2O_3	2.866
	CaO	8.664

For the $NiCo/SiO_2$ catalyst, the XRF analysis revealed the presence of SiO_2 as the dominant component with 65.9 mole %, confirming successful synthesis of silica from rice husk ash. NiO and Co-containing species were also detected, indicating successful incorporation of active metals onto the silica support (Alshammari *et al*., 2026). The high silica content contributes to improved thermal stability, porosity, and surface area of the catalyst (Alshammari *et al*., 2026). The presence of CaO and Al_2O_3 in smaller quantities may be associated with residual impurities or support modification during synthesis (Alshammari *et al*., 2026). The XRF results confirm successful preparation of both catalysts from sustainable waste-derived materials suitable for heterogeneous catalytic applications (Yi *et al*., 2019; Yi *et al*., 2020; Kotsur, 2022).

Conclusion

The conversion of agricultural waste such as rice husk and egg shell into catalyst for bio-kerosene production is a green strategy. The present work was designed to synthesize and characterize CaO and SiO_2 based heterogeneous catalyst from egg shell and rice husk. The characterizations conducted on the two catalysts reveal the feasibility of transforming agricultural wastes into valuable resources for green energy applications. Both rice husk and egg shell exhibited favourable chemical and structural properties vital for catalytic application in bio-kerosene production. The CaO catalyst derived from egg-shell showed higher surface area, larger pore sizes, and better thermal stability, making it very suitable for hydrodeoxygenation and trans-esterification reactions. On the other hand, SiO_2 catalyst derived from rice husk, have a moderate surface area, amorphous structure, and offers potential as both catalyst and support material for bio-kerosene production. XRD, XRF, and FT-IR analyses confirmed the successful synthesis of calcium oxide (CaO) and Silica (SiO_2) phases, while TGA confirmed the catalysts stability at high temperatures, indicating durability during catalytic reactions.

These research findings support the use of low-cost renewable, agricultural wastes as alternate source for active heterogeneous catalyst development, contributing to green and sustainable bio-fuel production and proper waste management. The study offers a solid foundation for future research on optimization of catalyst synthesis parameters (impregnation method and calcination temperature) to enhance catalyst performance. This work is also in line with the on-going global fight against climate change, promoting circular economy by reducing the over-dependence on fossil fuel and the use of expensive catalysts for bio-fuel production.

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