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CHARACTERIZATION OF ALUMINA SUPPORTED CHICKEN EGG SHELLS AND POTATO PEELS FOR BIODIESEL CATALYST

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DECLARATION

I declare that this thesis entitled "Characterization of Alumina Supported Chicken Eggshells and Potato Peels for Biodiesel Catalyst" is the results of my own research except as cited in the references.

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CHARACTERIZATION OF ALUMINA SUPPORTED CHICKEN EGG SHELLS AND POTATO PEELS FOR BIODIESEL CATALYST

ABSTRACT

The growing demand for renewable energy sources has prompted the development of effective biodiesel catalysts. This study looks into the characterization of catalysts made from chicken eggshells and potato peels and supported by alumina in order to contribute to environmentally friendly catalytic solutions. The catalyst's composition, structure, and thermal stability were thoroughly investigated using Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), Brunauer-Emmett-Teller (BET) analysis, and Thermogravimetric Analysis (TGA). Key findings highlight the successful integration of alumina support and the predominant presence of calcium oxide derived from chicken eggshells. Furthermore, the roles of potassium and aluminium oxide in the catalyst synthesis process were investigated. The catalyst had a significant BET surface area of 2.507 m^2/g, which is crucial for catalytic efficiency. Pore structure changes were observed as a result of the alumina incorporation and calcination processes, indicating potential increases in catalytic activity. Practically, these findings hold promise for large-scale biodiesel synthesis, providing a sustainable alternative with a lower environmental footprint. This study helps to use renewable resources and develop eco-friendly energy solutions by using agricultural byproducts like chicken eggshells and potato peels. Finally, alumina-supported catalysts derived from natural sources provide a viable pathway to sustainable biodiesel production, emphasizing their importance in addressing the pressing global energy challenge.

Keywords: Biodiesel, Alumina, Chicken eggshells, Potato peels, Sustainable energy

PENCIRIAN ALUMINA YANG DISOKONG KULIT TELUR AYAM DAN KULIT UBI KENTANG UNTUK PEMANGKIN BIODIESEL

ABSTRAK

Permintaan yang semakin meningkat untuk sumber tenaga boleh diperbaharui telah mendorong pembangunan pemangkin biodiesel yang berkesan. Kajian ini melihat ke dalam pencirian pemangkin yang diperbuat daripada kulit telur ayam dan kulit kentang dan disokong oleh alumina untuk menyumbang kepada penyelesaian pemangkin mesra alam. Komposisi pemangkin, struktur dan kestabilan terma telah disiasat dengan teliti menggunakan Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Difraction (XRD), analisis Brunauer-Emmett-Teller (BET), dan Thermogravimetric Analysis (TGA). Penemuan utama menyerlahkan kejayaan penyepaduan sokongan alumina dan kehadiran utama kalsium oksida yang diperoleh daripada kulit telur ayam. Tambahan pula, peranan kalium dan aluminium oksida dalam proses sintesis mangkin telah disiasat. Pemangkin mempunyai luas permukaan BET yang ketara 2.507 m^2/g, yang penting untuk kecekapan pemangkin. Perubahan struktur liang diperhatikan hasil daripada penggabungan alumina dan proses pengkalsinan, menunjukkan potensi peningkatan dalam aktiviti pemangkin. Secara praktikal, penemuan ini menjanjikan sintesis biodiesel berskala besar, menyediakan alternatif yang mampan dengan jejak alam sekitar yang lebih rendah. Kajian ini membantu menggunakan sumber boleh diperbaharui dan membangunkan penyelesaian tenaga mesra alam dengan menggunakan produk sampingan pertanian seperti kulit telur ayam dan kulit kentang. Akhir sekali, pemangkin yang disokong alumina yang diperoleh daripada sumber semula jadi menyediakan laluan yang berdaya maju kepada pengeluaran biodiesel yang mampan, menekankan kepentingannya dalam menangani cabaran tenaga global yang mendesak.

Kata Kunci: Biodiesel, Alumina, Kulit telur ayam, Kulit kentang, Tenaga lestari

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LIST OF ABBREVIATIONS

CES	Chicken Eggshells
PP	Potato Peels
FT-IR	Fourier Transform Infrared
XRD	X-ray Diffraction
BET	Brunauer - Emmett - Teller
WCO	Waste Cooking Oil
Al_2O_3	Aluminium Oxide
K	Potassium
HC	Hydrocarbons
CO	Carbon Monoxide
CaO	Calcium Oxide
$CaCO_3$	Calcium Carbonate
EPA	Egg + Potato + Alumina
TGA	Thermogravimetric analysis

LIST OF SYMBOLS

ml	Millilitre
°C	Celsius
%	Percent
OH^-	Hydroxyl

CHAPTER 1

INTRODUCTION

1.1Background of Study

In recent decades, research on and interest in biodiesel, one of the renewable energy substitutes for fossil diesel in diesel engines, has increased significantly. Fuel made from biodiesel is renewable, biodegradable, sustainable, and favorable to the environment. The benefits of using biodiesel include improved combustion efficiency due to its fuel-bound oxygen and fewer emissions of carbon monoxide (CO) and unburned hydrocarbons (HC) (Raj Bukkarapu and Krishnasamy et al., 2023). Due to its renewable nature and favourable environmental impacts, biodiesel is a possible and more enticing alternative fuel. Making biodiesel from low-quality feedstock, such as animal fats or non-edible oils, is thought to be a profitable process. Transesterification is the method used most frequently to create biodiesel. It produces gasoline quickly, cheaply, and efficiently (Pydimalla et al., 2023).

Every year, approximately 8 million tonnes of egg shell waste are generated worldwide. Meanwhile, various industries in Malaysia generate 70,686 tonnes of eggshell waste. Eggshell waste is classified as hazardous waste under European Union regulations, which raises disposal management costs. Due to its high protein and nutritional content, poultry eggs are a low-cost food source. The egg from a chicken or hen is now a global mass-production industry. Popular choices include quail, duck, goose, turkey, and ostrich eggs. Both white and yellow yolks contain aqua, protein, glucose, fat, and ash, but the proportions of these components vary by egg breed. Eggs also contain essential lipids, proteins, minerals, and low-calorie sources. Egg products are used in a variety of food industrial applications, including thickening, binding, leavening, glazing, and garnishing. An eggshell cuticle, spongeous, and lamellar layers comprise a three-layered structure. The cuticle layer is the outermost layer and is primarily composed of proteins. Protein fibers linked to calcium carbonate ($CaCO_3$) crystals form a spongey and lamellar matrix. $CaCO_3$ makes up 94% of eggshell, with the rest made up of calcium phosphate (1%), magnesium carbonate (1%), and organic matter (4%). The eggshell and albumen membranes are two thin eggshell membranes composed primarily of collagen, alkynes, alkanes, amines, protein amides, and carboxylic acids (Razali et al., 2022).

The potato (*Solanum tuberosum L.*), one of the most frequently grown cash crops in the world, is consumed by millions of people every day in a variety of ways. The world's top potato producer in 2019 was China, which produced 91.8 million tonnes, with India coming in second with 50.2 million tonnes. The processed food industry currently generates more potato peel waste as a result of the shift in consumer preferences from fresh to more processed commodities like potato chips, french fries, puree, and others. However, potato peels may be a useful bio resource for the commercial production of bio-derived chemicals and fuels. Because of this, potato peels have been used to make bio based fertilizer, bioethanol, chemicals like lactic acid and phenol, as well as bio-oil, biochar, cosmetics, and medicines. Due to its complex combinations of phenol, esters, carboxylic acid, aldehydes, alcohols, ketones, and sugars, bio-oil production is especially intriguing since it may be used as a bio-crude or bio source for the production of both biofuels and specialty chemicals (Daimary et al., 2022)

1.2 Problem Statement

Biodiesel is a promising renewable fuel alternative to fossil fuels due to its environmental benefits and potential to reduce dependence on petroleum. Heterogeneous catalysts, particularly those based on readily available and sustainable materials, are crucial for cost-effective biodiesel production. Chicken eggshells and potato peels are abundant agricultural wastes rich in calcium carbonate and potassium, respectively, which can be used as precursors for biodiesel catalysts. However, the effectiveness of these wastes as catalysts needs to be investigated and optimized for efficient biodiesel production.

Limited information exists on the characterization and performance of alumina-supported chicken eggshells and potato peels as biodiesel catalysts. The effectiveness of these catalysts depends on their surface properties, such as porosity, surface area, and active site distribution, which are largely unknown. The optimal conditions for catalyst preparation, including calcination temperature and metal loading, need to be determined for maximizing catalytic activity and stability.

The calcium carbonate content of chicken eggshells makes them a valuable catalyst for transesterification processes. Egg shells are a cost- and environmentally-friendly solution due to their abundance as a by-product and their porous nature, which offers a vast surface area for catalytic activity. Another agricultural by-product is potato peels, which have a high starch and potassium content and can be processed into sugar for use in the transesterification process. When supplemented by alumina, potato skins also show encouraging catalytic qualities. In addition to offering a long-term solution for waste management, using potato skins as a catalyst can maximize biomass conversion into biofuel.

The waste chicken eggshells and potato peels become more stable and catalytically active when alumina, a frequently utilized support material is added. Alumina support's synergistic action on various agricultural by-products is essential to producing a catalyst that is more efficient and recyclable. Gaining an understanding of how alumina interacts with potato peels or egg shells is crucial to maximizing the catalyst's effectiveness.

1.3 Objective

i. To synthesis bifunctional catalyst with 70% Chicken eggshells, 17% potato peel and 13% alumina.

ii. To characterize chicken eggshells and potato peels to analyze the functional group, investigate the crystalline structure, used to determine the specific surface area and study the thermal stability.

1.4 Scope of Study

This study aims to produce biodiesel catalyst supported chicken eggshells and potato peels. Used chicken eggshells and potato peels has potential to make biodiesel catalyst.

Since the production of leftover potato peel in the processed food sectors has expanded, it has been used as a biodiesel catalyst. The production of bio-oil is particularly significant due to its complex mixtures of phenol, esters, carboxylic acid, aldehydes, alcohols, ketones, and sugars since it may be utilized as a bio-crude or bio source for the production of both biofuels and fine chemicals. Additionally, potato peel can be affordable, environmentally safe, recyclable, and renewable when utilized as a biodiesel catalyst. Potato peel is a feasible and fairly priced source of catalyst material since it is a plentiful and sustainable byproduct of the potato processing industry. In potato peel, there are lots of functional groups such carboxylic acids, phenolic compounds, and amine groups that can act as active sites for the transesterification reaction required for the production of biodiesel. Potato peel is also biodegradable and non-toxic.

Waste chicken eggshells are primarily made of calcium carbonate. Calcium is an essential part of chicken eggshells and can act as a catalyst to improve the generation of biodiesel. In the transesterification step required to produce biodiesel, calcium ions can function as catalysts, accelerating the transformation of triglycerides into methyl esters. Chicken eggshells are a possible supply of calcium ions for biodiesel catalysis due to their high calcium carbonate concentration. The porous nature of chicken eggshells also gives the catalyst a high surface area, improving interaction between the reactants and the catalyst surface and enhancing catalytic activity

1.5 Significance of Study

Firstly, the study investigates the utilization of agricultural waste materials as potential sources of catalysts for the generation of biodiesel, such as chicken eggshells and potato peels. The sustainable use of agricultural waste and the reduction of the dependency on non-renewable fossil fuels are both significantly impacted by this.

The study also focuses on the catalyst’s characterization, which is important for comprehending their properties and potential applications in the synthesis of biodiesel. Finding the catalyst's physical and chemical properties, such as its surface area, pore size, and acidity, is known as characterization. This information is crucial for improving the catalytic activity of a material and selecting the optimum catalyst for a given reaction.

Lastly, the study also has potential in the creation of fresh catalysts for the manufacturing of biodiesel. Agricultural waste can be used as a source of catalysts as an alternative to traditional catalysts, which are frequently expensive and damaging to the environment. The study can aid in the creation of more efficient and economical processes for producing biodiesel by analyzing and improving the catalysts properties.

CHAPTER 2

LITERATURE REVIEW

2.1 Biodiesel

As a renewable energy source, biodiesel provides a number of noteworthy benefits and is crucial to the search for long-term energy solutions. A clean-burning, renewable alternative to petroleum diesel is biodiesel. Locally produced, it offers a number of environmental advantages. The handling, storage, and transportation of biodiesel are safer than those of petroleum because it is less combustible. This implies that it also harms the environment less and is simpler to control in the case of an oil spill. Because they burn cleaner than petrol and are completely biodegradable, biofuels emit fewer greenhouse gases than conventional fuels. Compared to traditional fossil fuels, biodiesel has the potential to considerably cut greenhouse gas emissions. Because it recycles carbon dioxide (CO^2) through the growth of the biomass raw materials used in its manufacturing, biodiesel emits less CO^2 during combustion. These qualities make biodiesel a useful tool for combating environmental issues and reducing climate change. Additionally, the carbon dioxide absorbed from growing soybeans or other feedstock used to make fuel balances the CO^2 released during the combustion of biodiesel. This characterizes it as a renewable and sustainable energy source (Viondra et al., 2022).

In the aforementioned nations, palm oil is predominantly used as the primary feedstock for the manufacturing of biodiesel in Indonesia, Thailand, Malaysia, and China. Indonesia is the largest producer of palm oil in these nations, which have sizable palm oil industries. A method known as transesterification can turn the flexible feedstock palm oil into biodiesel. These nations produce 834 million liters of biodiesel from palm oil in China and 5,600 million liters in Indonesia each year. The Philippines, on the other hand, uses used cooking oil (WCO) as a feedstock for making biodiesel. The Philippines shows the potential of utilizing waste resources to make biodiesel with a production capacity of 220 million liters. China is notable for having the highest volume of petro diesel consumption at 174,999 million liters. This is explained by the country's substantial population and quick industrialization. Petro diesel use considerably outpaces biodiesel generation in China, demonstrating the country's heavy reliance on fossil fuels. Indonesia has a large demand for conventional diesel fuel with a petro diesel consumption of 32,196 million liters. With 23,602 million liters and 11,624 million liters, respectively, of petro diesel use, Thailand and Malaysia also show substantial usage. The usage of petroleum diesel in the Philippines, at 10,158 million liters, is considerably less.

Table 1: Status of biodiesel and petro diesel in Asian countries (Mahayuddin et al., 2022)

Country	Main Feedstock of Biodiesel	Production of Biodiesel (ml)	Consumption of Petro diesel (ml)
Indonesia (main Palm Oil Producer)	Palm oil	5,600	32,196
Thailand	Palm oil	1,567	23,602
Malaysia (second larger palm oil producer)	Palm oil	1,245	11,624
China	WCO	834	174,999
Philippines	Coconut oil	220	10,158
India	WCO, non-edible industrial oil, animal fats	185	102,079
Japan	WCO	17	60,573

2.2 Heterogeneous Catalyst

The production of biodiesel uses a heterogeneous catalyst, which provides a surface for the reactants to interact on without being consumed or significantly changed. In the production of biodiesel, common examples of heterogeneous catalysts include metal oxides (such as alumina, titania, or zirconia), zeolites, activated carbon, and other supported catalysts. Over the past ten years, heterogeneous catalyst that is simple to remove from the biodiesel has been used. Therefore, creating a highly selective heterogeneous catalyst will aid in the environmentally friendly generation of biodiesel. A heterogeneous catalyst makes separation easier, increases reusability, and is more suited for non-edible oils (Vasu et al., 2023).

2.3 Potato peel

The phrase "potato peel as a biodiesel catalyst" refers to the potato peel's outer layer, which can be used in the transesterification process to produce biodiesel. Although the potato peel doesn't have any catalytic qualities on its own, it can act as a support material for other catalysts that help turn vegetable oils or animal fats into biodiesel, including alkaline catalysts or enzymes. In addition, potato peel has advantages beyond its use as a biodiesel catalyst. Additionally, it can be used to make bio-based fertilizer, bioethanol, chemicals like lactic acid and phenol, as well as bio-oil, biochar, cosmetics, and pharmaceuticals. Due to its complex combinations of phenol, esters, carboxylic acid, aldehydes, alcohols, ketones, and sugars, bio-oil production is especially intriguing since it may be used as a bio-crude or bio-source for the production of both biofuels and fine chemicals (Daimary et al., 2022).

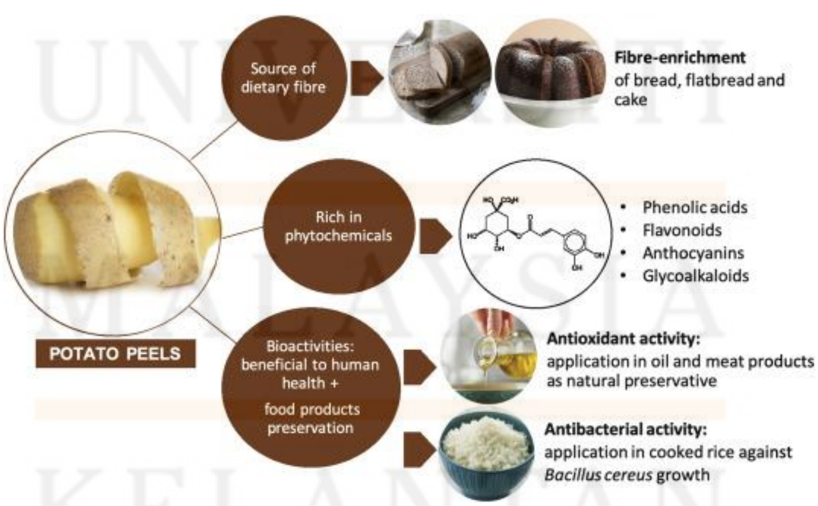

Figure 1: Potato Peels (Sampaio et al., 2020)

2.4 Chicken Eggshells

Chicken eggs are commonly consumed in several Asian countries, such as Singapore, Malaysia, Thailand, and the Philippines. Many nutrients are found in eggs, but the main one is protein, which is found in higher concentrations than in most other meals. Because eggs are so essential to a person's diet, the amount of chicken eggshell (CES) waste produced has skyrocketed. Approximately 10% of a chicken's egg is made up of eggshells, which are useless. The heterogeneous catalyst eggshells are ideal for biodiesel production since they contain 49.98% CaO (Gaide et al., 2022).

One of the CES's primary functions is to serve as a growing chick's incubator, but it also serves as a creative container for essential food. Most commercial layers of premium CES contain about 2.2 g of calcium in the form of calcium carbonate. Chicken faces with a high calcium content and a chemical makeup akin to limestone are known as CES. Given their chemical composition, it is possible that eggshells could function as a low-cost heterogeneous catalyst for the production of biodiesel. CaO is an essential component of eggshells. Global egg production increased by more than 14% during the previous ten years, reaching 76.7 million tonnes in 2018. 10% of a chicken's weight is made up of eggshells (Gaide et al., 2022).

In the sample of avian CES waste, calcium oxide (CaO) remained the most common component, according to the chemical analysis of CES. The high percentage of calcium oxide is linked to the presence of calcium carbonate, which accounts for most avian CES. The remaining 1% is made up of calcium phosphate, 1% of magnesium carbonate, and 4% is composed of organic stuff. Calcium carbonate makes up 94% of egg shells. Eggs are among the foods consumed most frequently worldwide, and their shells are highly calcium-rich. Wasted shells are evaluated to enhance reaction quality using a calcium oxide (CaO) catalyst produced by calcining egg shells. Eggshell is the perfect material to make active heterogeneous catalysts because of its porous structure and high $CaCO_3$ concentration (Nath et al., 2021).

Figure 2: Chicken Eggshells

(Grolms, 2019)

2.5 Bifunctional Catalyst

To provide new reactivity and selectivity in the process of interest, bifunctional catalysis uses low molecular weight, structurally specified molecules with two distinct functional groups. Pronucleophiles and electrophiles generally participate in the reaction, which is typically a polar addition reaction. Ideally, a bifunctional catalytic system transforms low-value, simple starting materials into high-value, stereo chemically specified products in this reaction (Dixon et al., 2016). Furthermore, due to their high surface area and capacity to convert free fatty acids and triglycerides into the synthesis of biodiesel, bifunctional catalysts have become effective catalysts (Changmai et al., 2020).

CHAPTER 3

MATERIAL AND METHODS

3.1 Materials

The waste chicken eggshells were obtained from a local market in Jeli, Kelantan while potato peels were sourced from a market in Tanah merah, Kelantan. The chemical substance like alumina powder were gained by the Sigma-Aldrich (M) Sdn Bhd at Subang Jaya Selangor, Malaysia.

3.2 Chemicals

The chemical substance like alumina powder used in the study were gained by the Sigma-Aldrich (M) Sdn Bhd at Subang Jaya Selangor, Malaysia are outlined in Table 2.

Table 2: The chemical used in the research

No.	Chemical
1	Alumina Powder Al_2O_3

3.3 Apparatus and Equipment

The apparatus and equipment used for chicken eggshells and potato peels for catalyst preparation are summarized Table 3 and Table 4.

Table 3: List of apparatus used in the research

No.	Apparatus and Equipment
1	Beaker (50mL,250ml & 500ml)
2	Measuring cylinder (10ml & 100ml)
3	Zip lock plastic bag
4	Aluminium foil
5	Zip lock plastic bag
6	Aluminium foil
7	Spatula
8	Gloves
9	Mask
10	Sieve (300 µm)
11	Funnel

Table 4: List of equipment used in the research

No	Equipment	Purpose
1	Oven	Drying
2	Heavy Duty Blender Machine	Particle size reduction for reactivity
3	Muffle furnace	Calcination and carbonization
4	Magnetic Stirrer	Measuring light absorbance across during the wet impregnation process
5	Desiccator	Storage

3.4 Methods

3.4.1 Chicken Eggshells and Potato Peels for Catalyst Preparation

The chicken eggshells were first thoroughly washed with hot water to aid the removal of proteins and lipids and then with tap water and dried in hot air oven at 105 °C for 24 h. Using a specialized grinder, the shells were ground into a powder that was then sieved to create 300 µm particles. Similarly, the potato peels were separated from unwanted debris and dirt by hand and then washed with distilled water to eliminate residual dirt. The dried peels were sieved to obtain 300 µm particles. The chicken eggshells were calcined at 900 °C for 3 hours, then allowed to cool overnight in a muffle furnace (Niju et al., 2014). They were stored in an airtight container after being desiccated to dry them out. The potato peels were desiccated, dried, and then carbonized for three hours at 500 °C in a muffle furnace before being put in an airtight container (Amenaghawon et al., 2022). Using the aforementioned wet impregnation technique, aluminum-doped composite catalysts were produced using carbonized potato peels and calcined chicken eggshells. The chicken eggshells, potato peels, and aluminum oxide were combined (70%, 17%, and 13%) in 400 ml of deionized water and aggressively agitated for 2 hours at 80 °C using a magnetic stirrer. The resulting slurry was then dried in the oven for an additional hour before being calcined for four hours at 900 °C. The catalyst was finished, sealed in a container, and allowed to cool in a desiccator before being used once more.

3.4.2 Characterization of the Catalyst

Fourier Transform Infrared (FT-IR) analysis

Fourier Transform Infrared (FT-IR) analysis is a potent analytical method that is frequently employed in materials science and chemistry. It is a crucial instrument for characterizing materials. FT-IR is crucial in clarifying the molecular structure and composition of the materials being studied in relation to alumina-supported chicken eggshells and potato peels as possible biodiesel catalysts. By measuring a sample's absorption of infrared light, FT-IR spectroscopy can provide precise details on the sample's molecular bonds and functional groups. Numerous vibrational modes related to distinct chemical functions are explored in the designated wavenumber region 4000-400 cm^{-1}. The primary absorption bands linked to functional groups like hydroxyl, carbonyl, and aliphatic bonds are included in this spectral region, making it especially pertinent for research on organic compounds like those found in potato skins and eggshells. Furthermore, the FT-IR technique aids in the detection of pollutants, impurities, or undesired byproducts, hence advancing a thorough comprehension of catalyst efficiency and purity. The repetitive measurements made possible by this technique's non-destructive nature allow for the observation of structural or catalytic changes over the course of the catalyst's life cycle.

The X-ray Diffraction (XRD) analysis

X-Ray Diffraction (XRD) analysis using a Bruker Model D2 in the scan range of 10°C to 90°C revealed important information about the crystal structure and phase composition of alumina-supported chicken eggshell and potato peel catalysts. This technique identifies the crystallographic phases in the catalyst material and determines crystal size, lattice parameters, and crystallinity. By analyzing the diffraction patterns produced when X-rays interact with the atomic arrangement of the sample, we gain valuable information about the catalyst's structural properties, which aids in the optimization of its performance for biodiesel production.

Brunauer-Emmett-Teller (BET)

Brunauer-Emmett-Teller (BET) analysis is a valuable characterization method. Alumina-based chicken eggshell and potato peel catalysts for biodiesel production. BET analysis provides useful information about catalyst surface area, pore volume, and pore size distribution, all of which have an impact on catalyst activity and efficiency. BET analysis determines specific surface area and pore characteristics by measuring gas molecule adsorption on the catalyst surface at different pressures. This information aids in understanding catalyst porosity, which influences their accessibility to reactant molecules and product diffusion, thereby affecting catalyst performance. Furthermore, BET analysis can reveal the effectiveness of catalyst support materials, such as alumina, in providing a large surface area for active site deposition. BET analysis is important in optimizing the design and synthesis of catalyst materials for biodiesel production, as it improves catalyst efficiency and sustainability.

Thermogravimetric analysis (TGA)

Thermogravimetric Analysis (TGA) was a useful technique for characterizing materials, such as alumina-supported chicken eggshell and potato peel biodiesel catalysts. This method involved exposing the sample to controlled temperature changes while measuring its weight loss or gain as a function of temperature. TGA revealed information about the catalysts' thermal stability, decomposition behavior, and weight loss kinetics by heating them in a nitrogen atmosphere between 30°C and 900°C. TGA revealed the temperature at which various catalyst components decomposed or volatilized, which helped researchers understand their thermal degradation pathways. Furthermore, TGA data provided information on the catalysts' composition, moisture content, and organic content, which guided optimization strategies for their synthesis and application in biodiesel production.

3.5 Research Flow Chart

The research flow chart will be divided into three stages. Stage 1 is material preparation, stage 2 for catalyst characterization and stage 3 is for analysis, evaluation and comparison of the obtained experimental data as shown below.

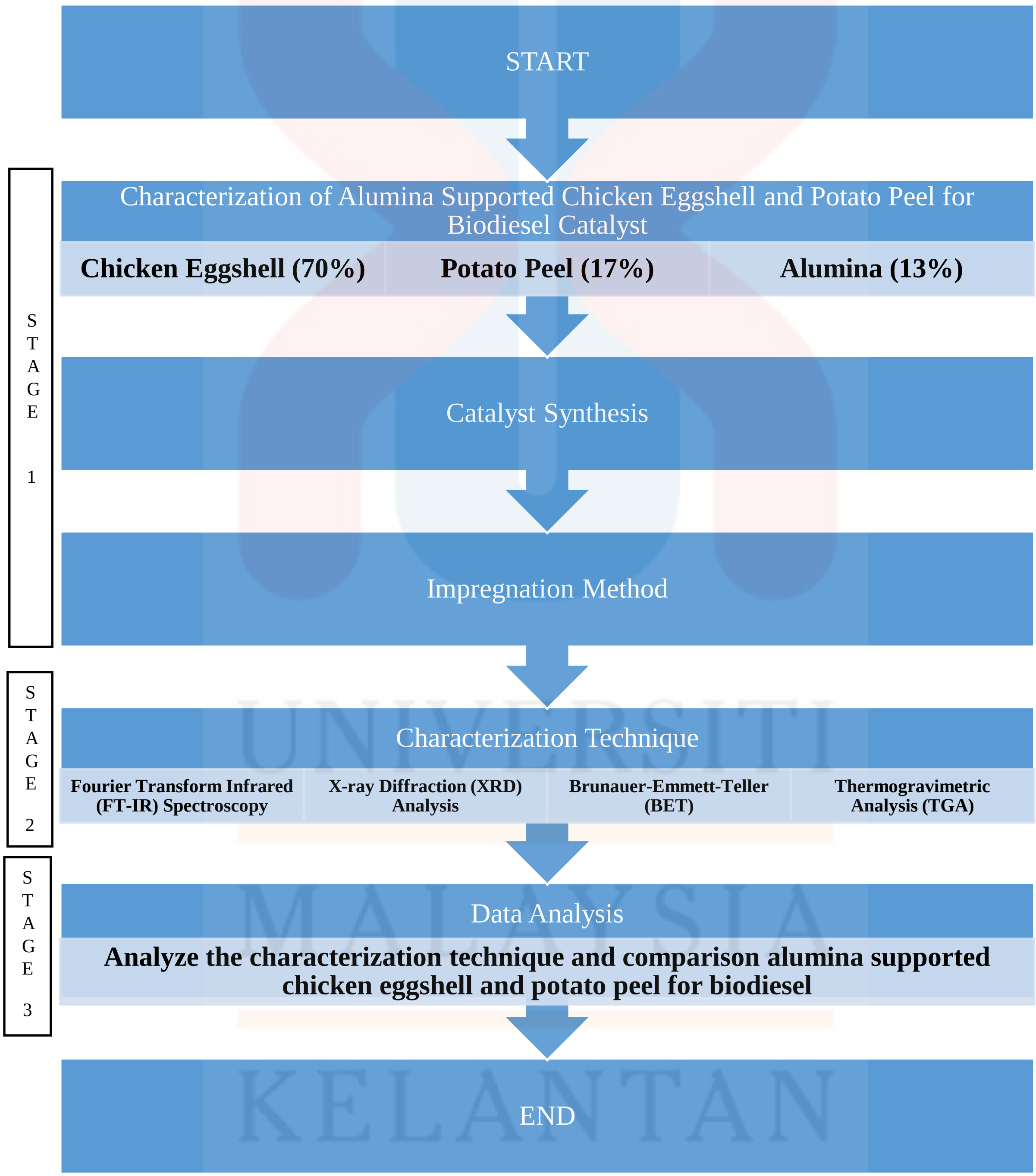

Figure 3.5: Flow Chart for alumina supported chicken eggshell and potato peel for biodiesel

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Fourier Transform Infrared (FT-IR) spectrometer

The prepared sample were analyzed by FTIR at wavenumber region of 4000-400 cm^{-1} to determine the catalyst structure. The FTIR results are shown in figure 6, the major peaks of catalyst were recorded at 3698.28, 2359.70, 1733.67, 1697.53, 1418.47, 687.05 and 501.91 cm^{-1}. The peaks at 3698.28 cm^{-1} and 2359.70 cm^{-1} correspond to stretching vibrations of hydroxyl (OH-) groups. These groups are frequently associated with metal oxides and could indicate the presence of metal-oxygen bonds in the catalyst structure.

The peak at 1733.67 cm^{-1} represents carbonyl (C=O) stretching vibrations. This suggests the presence of carbonyl groups in the catalyst, which could come from a variety of sources such as esters, aldehydes, or ketones. The presence of carbonyl functionalities is essential for catalytic processes, particularly biodiesel production.

Furthermore, peaks at 1697.53 cm^{-1} and 1418.47 cm^{-1} indicate C=C stretching vibrations and CH_2 bending vibrations. The former indicates the presence of unsaturated bonds, which could be associated with organic compounds that contribute to catalytic activity. The latter is characteristic of methylene groups, which are commonly found in aliphatic chains and indicate the presence of hydrocarbons. Notably, the peaks at 687.05 cm^{-1} and 501.91 cm^{-1} indicate metal-oxygen vibrations (Hezam et al., 2017). These could be linked to the alumina support in the catalyst, indicating that the support material was successfully incorporated.

The combined FTIR results indicate a complex catalyst structure with various functional groups derived from both the support material (alumina) and bio-derived sources (chicken eggshells and potato peel). The presence of hydroxyl and carbonyl groups indicates potential catalytic sites for biodiesel synthesis. Furthermore, the observed metal-oxygen vibrations indicate that the catalyst's alumina support is being used effectively.

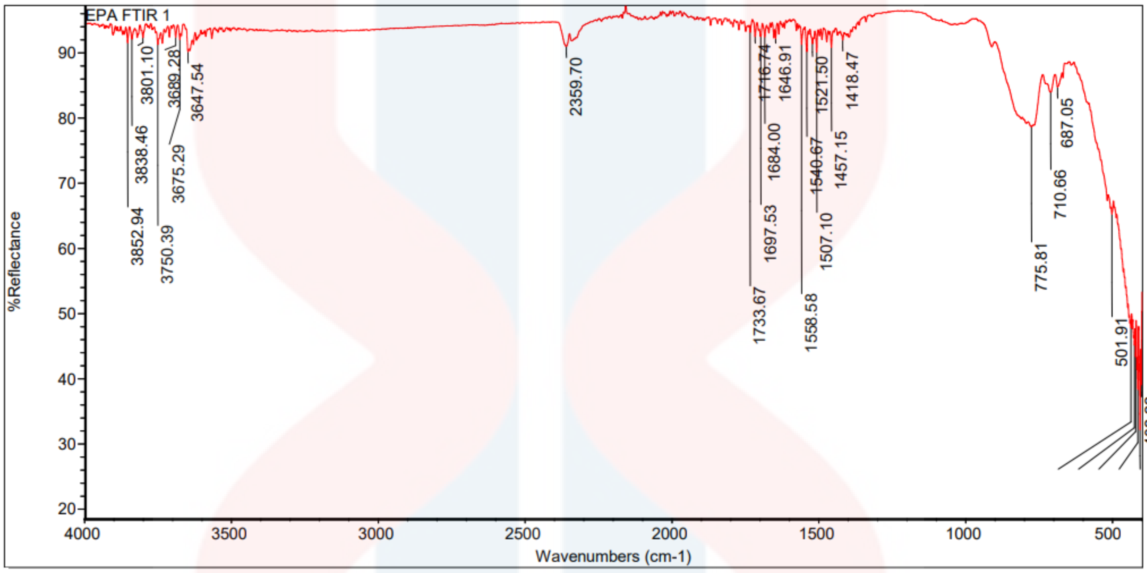

Figure 4.1: Fourier Transform Infrared (FT-IR) of the EPA catalyst

4.2 X-ray Diffraction (XRD) Analysis

Based on the figure 4.2, the X-ray Diffraction (XRD) analysis presented in this study reveals a high concentration of calcium oxide (CaO) in the material derived from chicken eggshells. The major peaks at 32.194°, 37.345°, 53.843°, 64.134°, and 67.357° correspond to CaO-specific reflections, confirming the compound's dominance in the analyzed sample. The origin of CaO is attributed to the use of chicken eggshells as a precursor material, providing valuable insights into the successful transformation of calcium carbonate into calcium oxide via the chosen synthetic route.

In comparison to other studies that used crab shells as a precursor material for CaO synthesis, the observed XRD peaks differed significantly. Previous studies found that the main CaO peaks were at 20°, 22°, 26°, 28°, 30°, 43°, and 45°. This peak's position differs from that observed in this study, indicating a possible difference in crystal structure or synthesis conditions between the two precursor materials (Amenaghawon et al., 2022).

Furthermore, the XRD analysis shows the presence of potassium (K) in the sample, with significant peaks at 30.459°, 43.616°, 54.129°, 63.387°, and 71.944°. The detection of potassium is noteworthy, and the corresponding peaks correspond to the expected positions for K in the XRD pattern. The data collection pressure of 1.13 kPa is an important parameter to consider, as it influences the crystalline behavior and peak positions of the identified phases. This information helps to provide a comprehensive understanding of the sample composition and the conditions under which the XRD data were collected.

A comparison of the results to a previous study that used plantain peel as a precursor material for K synthesis revealed significant differences in the observed XRD peaks. The previous study identified major K peaks at 38°, 40°, and 50°. The differences in peak positions between plantain peel and potato peel-derived K indicate that the two precursor materials may have different crystalline structures or synthesis conditions (Amenaghawon et al., 2022). Variations in precursor material composition, morphology, or synthesis methodology could explain observed differences in peak positions. Variations in experimental parameters during the XRD analysis may also contribute to these differences. In particular, the characterization of K obtained from potato skins in our study sheds light on the material's unique properties and potential catalytic applications.

The analysis also reveals the presence of aluminium oxide (Al_2O_3) in the material, as indicated by major peaks at 14.511°, 19.813°, 21.201°, 21.316°, and 23.434°. The presence of Al_2O_3 suggests that alumina oxide was used in the synthesis process or as a component of the starting materials. The distinct peaks associated with Al_2O_3 help to characterize the sample and provide valuable information about the material's crystalline phases.

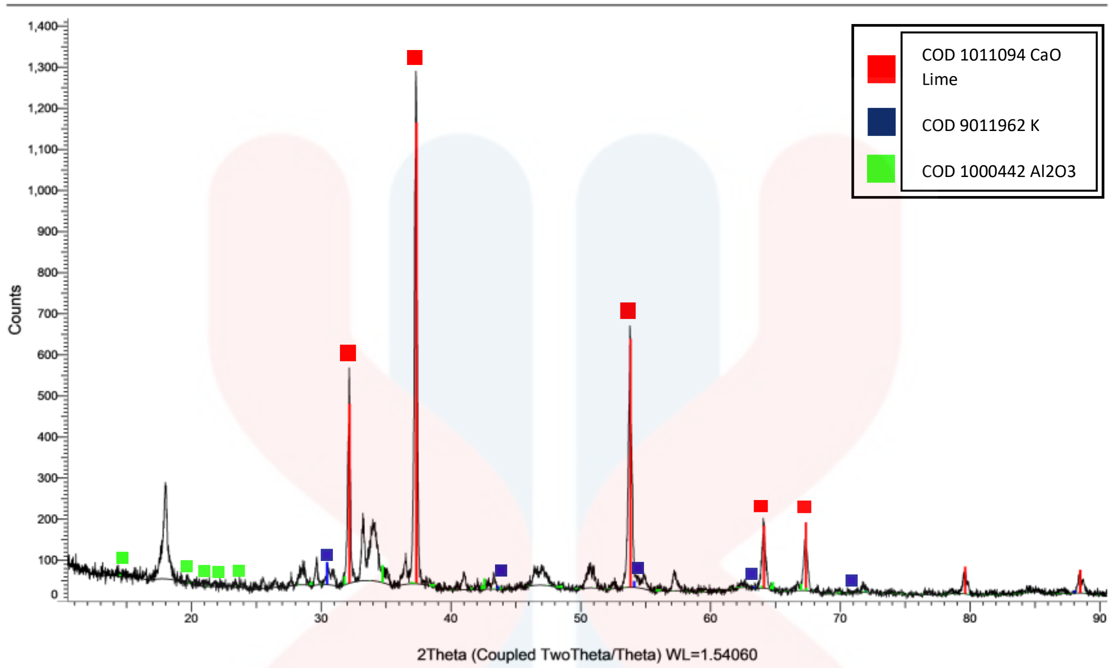

Figure 4.2: XRD analysis of the EPA catalyst

4.3 Brunauer-Emmett-Teller (BET)

The catalyst of EPA has a BET surface area of 2.507 m²/g, indicating its suitability for catalytic reactions. While this surface area is lower than that reported in previous studies for chicken eggshell and potato peel alone, the combination of these materials with alumina creates a synergistic effect that contributes to catalytic activity. The presence of pores in the catalyst structure, with a measured pore volume of 1.272 cc/g and an average pore diameter of 7.92965 nm, is critical for mass transport and catalytic activity. Although the pore volume is lower than those reported for potato peel alone, the average pore diameter is significantly larger than that of chicken eggshell alone. This implies that the pore structure has changed because of the alumina incorporation and calcination processes.

The structural properties of the precursor materials, particularly chicken eggshell and potato peel, have a significant influence on the catalyst's surface area and pore characteristics. Both materials have inherent porosity due to their natural structure, which is enhanced during the calcination process. The decomposition of organic matter in these precursors during calcination creates void spaces and pores in the material, increasing the catalyst's overall surface area and pore volume.

In comparison to other research for raw materials, chicken eggshell has a surface area of 48.6 m^2/g, a pore diameter of 4.03 nm, and a pore volume of 0.209 cc/g (M. Rashid et al., 2017). Potato peel has a surface area of 147 m^2/g, a pore diameter of 5.3 nm, and a pore volume of 0.65 cc/g (Kyzas et al., 2016). These values highlight both materials' inherent porosity and serve as benchmarks for understanding each component's contribution to the final catalyst structure.

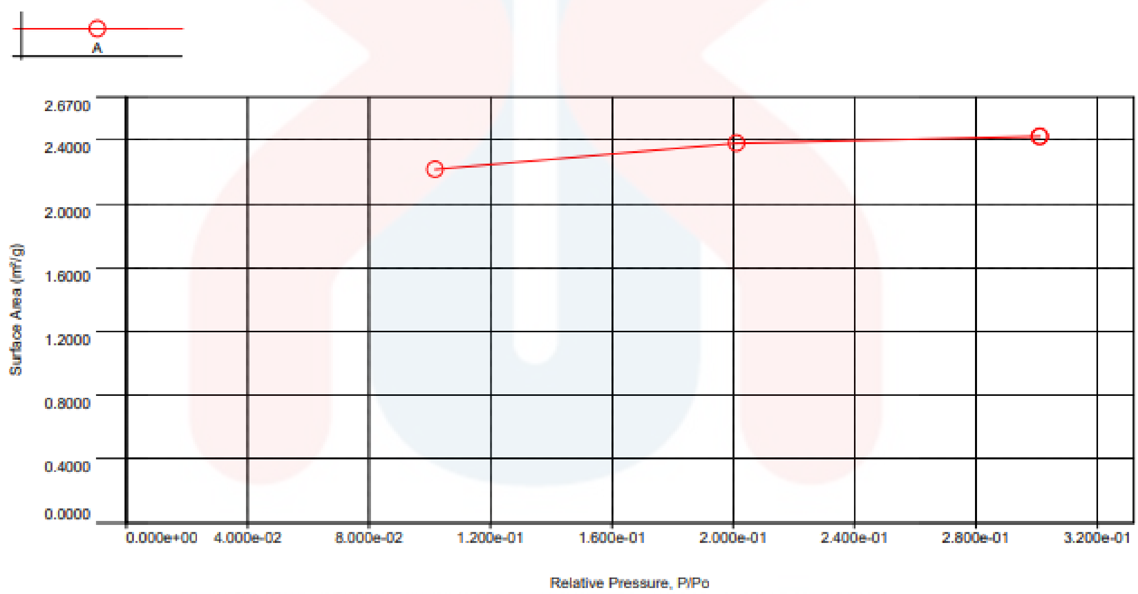

Figure 4.3: Single point surface area of the EPA catalyst

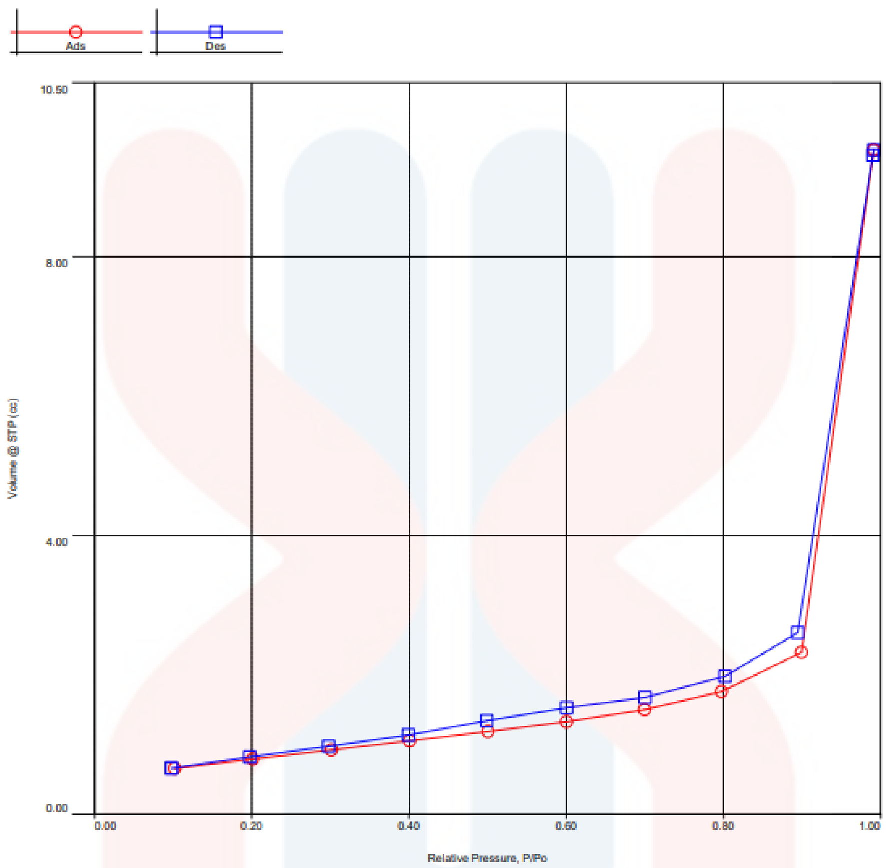

Figure 4.4: Raw linear of the EPA catalyst

4.4 Thermogravimetric Analysis (TGA)

In order to understand the thermal degrading behavior of the EPA catalyst made of potato peels and chicken eggshells supported by alumina, thermogravimetric analysis, or TGA, was used as a characterization approach in this study. TGA is an important method for characterizing catalysts because it makes it possible to examine how a material's weight varies in response to temperature in a controlled setting, usually a nitrogen atmosphere. We conducted an analysis and observation of the thermal breakdown or volatilization of different catalyst components by exposing the catalyst to a temperature range of 30°C to 900°C.

Based on figure 4.5, two reaction steps occurred in this experiment. The temperature ranges between -17.1557% and 0.4632 mg, with temperature limits ranging from 39.79°C to 496.70°C, the first substantial weight loss event was noted. For this stage, the midpoint of the mode sample temperature was determined to be 409.55°C. Then there was another weight loss event that ranged from -20.2974% to -0.5480 mg and left a residue of 1.6885 mg to 62.5408%. This step's temperature range was 496.83°C to 896.55°C, with a sample temperature midpoint of 626.90°C for the mode. Notably, the temperature ranges for the first and second weight drops were 400°C to 450°C and 550°C to 680°C, respectively.

By using TGA analysis, the thermal behavior of the EPA catalyst was better understood, which improved knowledge of its composition and potential applications in the production of biodiesel. The essential criteria for further catalyst synthesis process optimization and refinement were found to be the temperature intervals at which weight loss events occurred.

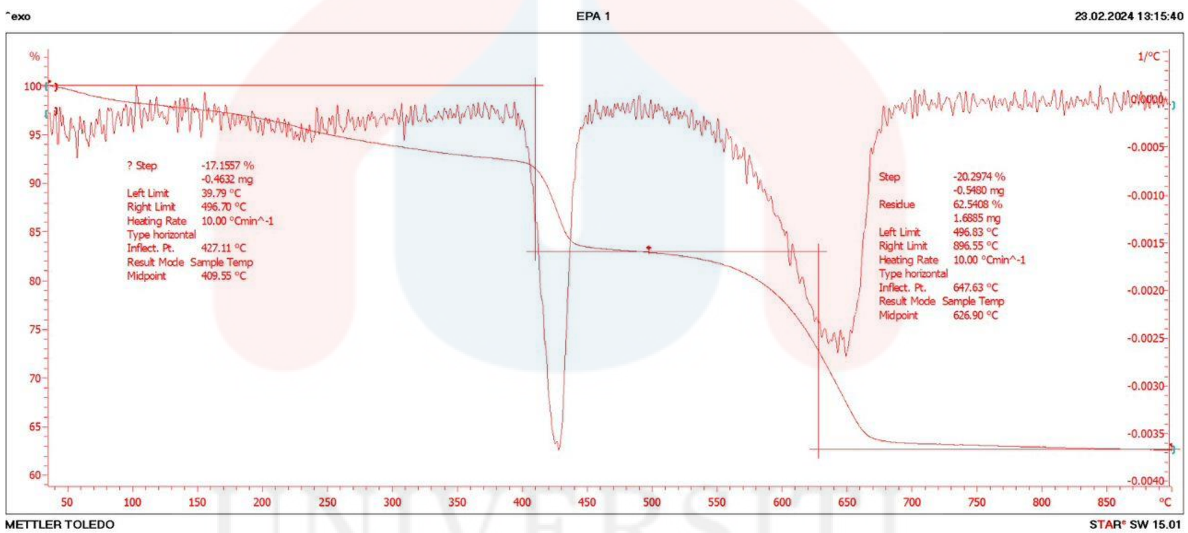

Figure 4.5: TGA analysis of the EPA catalyst

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In conclusion, conducted a thorough investigation into the characterization of alumina-supported catalysts derived from chicken eggshells and potato peels for potential biodiesel production. Careful examination using various techniques, such as wet impregnation, to reveal the structural and compositional properties of the developed catalyst. The first step in this thesis process is to carefully prepare raw materials, specifically chicken eggshells and potato skins. The process of washing, drying, grinding, and filtering removes impurities and produces fine powder particles. Calcination and carbonization at high temperatures are critical steps that cause transformations in the material and lay the groundwork for subsequent catalytic activity.

This thesis investigated the characterization of alumina-supported catalysts derived from chicken eggshells and potato peels in order to develop sustainable and effective catalysts for biodiesel production. A thorough understanding of the catalyst's composition, structure, and potential catalytic activity was achieved using a variety of sophisticated techniques such as Fourier Transform Infrared (FT-IR) spectroscopy, X-ray Diffraction (XRD) and Brunauer-Emmett-Teller (BET) surface area analysis.

The decision to used alumina as a support material was based on its well-known catalytic properties and stability. The wet impregnation technique improved the uniform distribution of chicken eggshells, potato peels, and aluminium oxide in the catalyst composition. The chosen proportions (70% chicken eggshells, 17% potato peels, and 13% aluminium oxide) were optimized for catalytic efficiency while taking into account the synergistic effects of each component.

Finally, combining various characterization techniques has resulted in a comprehensive understanding of alumina-supported catalysts derived from chicken eggshells and potato peels. Successful and comprehensive characterization pave the way for potential applications in biodiesel production. This work not only advances the field of catalysis, but it also aligns with sustainable practices by using agricultural waste as a catalyst component. The findings of this study pave the way for further research, ensuring the ongoing evolution of environmentally friendly and efficient catalysts for renewable energy applications.

5.2 Recommendations

Characterization of alumina supported chicken eggshells and potato peels for biodiesel catalyst is critical to reiterate the recommendation to delve deeper into catalyst synthesis process optimization. This requires a thorough investigation of parameters such as calcination temperature, impregnation methods, and support-to-waste ratios. By systematically varying these parameters, we can gain a comprehensive understanding of their effects on catalyst activity and selectivity, resulting in the development of highly efficient biodiesel catalysts.

Furthermore, mechanistic studies should be prioritized to better understand the catalytic processes involved in biodiesel production. Understanding reaction pathways, selectivity, and kinetics is critical for accurately measuring transesterification reactions. Mechanistic studies can provide valuable insights for the design of catalysts with improved performance and stability.

The importance of assessing the long-term stability of alumina-supported catalysts cannot be overstated. Exploring potential deactivation mechanisms and developing catalyst regeneration strategies are critical steps towards ensuring long-term catalytic activity. Addressing these issues will significantly improve the practical viability of developed catalysts for industrial applications.

Scale-up studies are critical in bridging the gap between laboratory-scale experiments and real-world applications. The evaluation of scalability and performance under industrial conditions is critical. Furthermore, economic analyses should be carried out to compare the cost-effectiveness of the developed catalysts to traditional methods, providing useful insights for industrial decision-making.

To broaden the scope of the investigation, researchers should look into alternative waste materials as catalyst precursors. Various agricultural and food waste sources present promising opportunities for catalyst synthesis. By broadening the range of precursor materials, novel catalysts with improved properties can be developed, helping to ensure the sustainability of biodiesel production processes. Collaboration with biodiesel production industries is critical for ensuring the relevance and applicability of research results. Industry partnerships make it easier to integrate innovative catalysts into industrial processes, accelerating technological advancements and promoting sustainable biodiesel production practices.

Finally, the proposed recommendations aim to advance the field of catalysis and promote sustainable biodiesel production. This research has the potential to make significant contributions to the field by addressing optimization parameters, conducting mechanistic studies, assessing long-term stability, conducting scale-up studies, diversifying catalyst precursors, and encouraging collaboration with industries.

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APPENDIX A

Potato peel (PP)

Chicken eggshell (CES)

Drying CES and PP in the oven

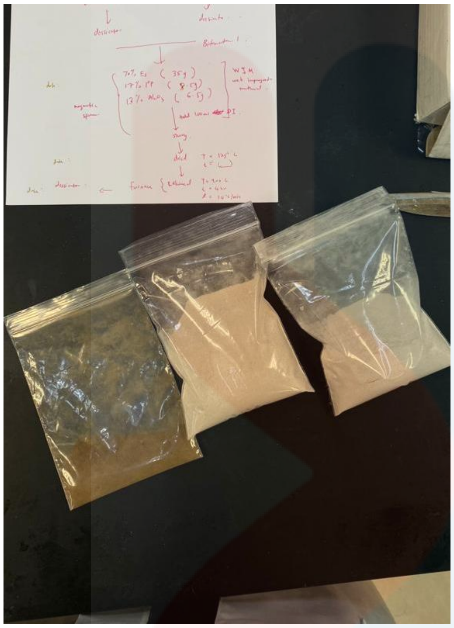
Dried using the oven

Grinding CES

Grinding PP

Sieve CES (300 µm)

Sieve PP (300 µm)



The samples are stored in a zipper bag

Furnace process

CES after being in the furnace

PP after being in the furnace

CES and PP

Wet impregnation method

The sample is dried in the oven

Samples that have been fully prepared