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Valorization of Biomass for Catalyst Production in the Pyrolytic Recycling of Waste Pet Bottles

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Abstract

Plastic pollution poses a severe global threat, with only a fraction effectively recycled, and Nigeria ranks among the top contributors. This study aims at addressing this environmental challenge by developing a sustainable, low-cost catalyst from waste eggshell and evaluating its performance in the pyrolysis of Polyethylene terephthalate (PET) plastic into fuel oil. The catalyst was prepared and calcined at 900°C for 5 hours, and its properties were characterized. Pyrolysis of PET with and without the catalyst was conducted at temperatures between 200–400°C, and the resulting oil was characterized using GC-MS and a bomb calorimeter. The results showed that the catalyst had a high surface area of 86.10 m^2/g, strong crystalline Calcium Oxide (CaO) structure, and functional groups that enhanced its catalytic performance. Compared to non-catalyzed pyrolysis, the catalyst significantly improved oil yield from 20% to 1200% across the heated temperatures, with its peak oil extraction at 254°C. At 400°C, the recovery was 147 ml with the catalyst, compared to 123 ml without the catalyst, showing a 19.5% boost with faster degradation and reduced reaction temperatures. The quality of the catalyzed oil showed improved oil quality when compared with the American Society for Testing and Materials (ASTM D975) standards for diesel fuel oils, as it had a flash point of 78°C, viscosity of 2.80 mm^2/s, and specific gravity of 0.8601, which are within the standard range. This shows that the calcined eggshells are effective, eco-friendly alternatives in the pyrolysis of PET, offering a dual benefit of waste valorization and energy recovery

1. Introduction

Globally, over 300 million tons of plastic are produced annually, with at least 8 million tons entering the oceans each year [1], [2]. This is a major global concern, and according to Geyer *et al* [3], if nothing is done, by 2050, 12,000 million tons of plastic garbage will accumulate in landfills and the environment. Also, Nigeria is ranked ninth in the world for plastic pollution, producing an estimated 2.5 million tons of plastic waste yearly, with less than 12% of that amount being recycled [4].

Polyethylene terephthalate (PET), being one of the most widely used plastics, especially in packaging, is highly resistant to degradation [5], poses several threats to the environment, leading to soil degradation, marine ecosystem disruption, and potential human health risks due to

microplastics entering the food chain [6]. This growing environmental burden underscores the urgent need for efficient plastic waste management and valorization strategies. Also, the rising energy demand has further compounded the waste and environmental pollution crisis, most especially in countries like Nigeria, which relies heavily on fossil fuels [7]. In response to these challenges, innovative, simple, and direct conversion technologies such as pyrolysis are being explored to transform these plastic wastes into valuable energy resources [8].

Pyrolysis, which is a thermochemical process carried out at 300–900°C in the absence of oxygen, has gained global attention over the years as a waste-to-energy strategy for converting plastic waste into fuel oil, gas, and char [9]. However, the efficiency of pyrolysis is largely dependent on the type of catalyst used. Common catalysts like ZSM-5, zeolites, and FCC improve reaction rates and product yields but are often expensive, complex to regenerate, and limited in feedstock compatibility [10]. These limitations have sparked growing interest in using biomass-derived catalysts, which are low-cost, renewable, and environmentally benign [11].

In addition to this waste and environmental challenges, eggshell waste, generated in large quantities by the food sector, comprises over 95% calcium carbonate and is produced at an estimated 250,000 tons per year globally [12], [13]. In Nigeria, where waste management systems are underdeveloped, discarded eggshells pose environmental and public health hazards due to improper disposal [14]. Repurposing these eggshells into catalytic materials for plastic pyrolysis presents a dual benefit that aims to reduce both plastic and agricultural waste while enhancing the sustainability of fuel production.

Against this backdrop, this present study aims to develop and characterize a catalyst derived from waste eggshells, assess its efficiency in the pyrolysis of waste PET plastics, and evaluate the resulting pyrolysis oil in terms of quality and yield. This will help curb these environmental pollution challenges, foster the generation of wealth from waste by supporting industrial development, create employment opportunities, and maximize both economic gains and environmental benefits.

2. Materials And Methods

2.1. Sample Collection

Waste eggshells were collected from food courts, restaurants, and pastries within the University of Benin, Benin City, Edo State, Nigeria, school environment. Waste Polyethylene terephthalate (PET) plastic bottles, such as water bottles and juice bottles, were collected from the premises of the University of Benin, Benin City, Edo State, Nigeria.

2.2. Preparation of The Catalyst from The Waste Eggshells

The discarded eggshells underwent a cleaning process with tap water to eliminate impurities on their surfaces, and the eggshells were left to air-dry for a period of three days. Following drying, the eggshells were finely pulverized into powder using a wooden mortar and pestle. The resulting particles were subjected to calcination in a muffle furnace, maintained at 900°C for a duration of 5 hours [15]. Before the muffle furnace could cool to room temperature, the hot, calcined samples were taken out and immediately placed inside a desiccator to cool. To avoid a reaction with moisture, the samples were taken out of the desiccator and maintained in an airtight container. This catalyst preparation was done following the same procedure outlined by Akhabue *et al* [15].

2.3 Reactor Set-Up

Figure 1 shows the setup for the SA-5-12 Electric Murfle Furnace Pyrolysis Reactor used in the pyrolysis of the PET plastic waste, and this was carried out in LUCO CHEMICAL LABORATORY LTD, in Edo State, Nigeria.

Figure 1: Sa-5-12 Electric Murfle Furnace Pyrolysis Reactor

2.4. Experimental Procedures

1000g of chopped PET waste (<1 cm^2) and 25 g of CaO_2 were introduced into the Sa-5-12 electric muffle furnace at temperatures between 200 to 400 °C with a 15 °C/min heating rate at 30 30-minute intervals. Considering the temperatures, the gas vapors were condensed into liquid fuel (waste plastic oil), and product yields were evaluated by weight to assess the impact of the eggshell catalyst on oil quality [16], [17].

2.5.Characterization of the Catalyst and Pyrolysis Oil

The eggshell-derived catalyst was characterized using Brunauer–Emmett–Teller (BET) Analysis, X-ray Diffraction (XRD), X-ray Fluorescence (XRF), Fourier Transform Infrared Spectroscopy (FTIR), and Thermogravimetric Analysis (TGA) to determine its crystal structure, elemental composition, functional groups, and thermal stability. The pyrolysis oil was analyzed using Gas Chromatography–Mass Spectrometry (GC-MS) for chemical composition, and its heating value was measured using a Bomb Calorimeter (ASTM D240), while gas yield was estimated via mass balance.

3. Results and Discussion

3.1. Catalyst Characterization

Table 1 shows the physical properties of the calcined chicken eggshell catalyst as compared to previously supported studies, determined through BET analysis, which has a high surface area (86.10 m^2/g), moderate bulk density (1.285 g/cm^3), fine particle size (<100 μm), and appreciable porosity (48%) as compared and supported by previous studies [18], [19]. According to Badnore *et al*., the surface area of nano-CaO is 97.63 $m^2\ g^{-1}$, whereas commercial CaO has a somewhat lower

surface area of 74.64 m^2 g^{-1}, confirming that the calcined eggshell catalyst exhibits favorable physical characteristics. Also, according to the findings by Ahmad *et al.* [20], pore structures <100 µm contribute significantly to the efficacy of the calcined eggshell catalyst in various catalytic processes, most especially in biodiesel production [18]. These properties are conducive to enhanced catalytic performance, as they facilitate improved reactant diffusion and provide more active sites for reactions, as reported by [18].

Table 1: Physical Properties of Calcined Eggshell Catalyst Compared to Standard Ranges from Previous Studies

Property	Calcined Eggshell Catalyst	Calcined Eggshell Catalysts from previous studies and references
Surface Area	86.10 m²/g	50–150 m²/g [20],[21]
Bulk Density	1.285 g/cm³	1.0–2.5 g/cm³ [22]
Particle Size	<100 µm	<200 µm [17]
Porosity	48%	30–70% [17]

3.1.1. XRF-EDS analysis

The XRF-EDS analysis revealed the elemental composition as shown in Figure 2, where calcium (Ca) was the predominant element, and other elements, including silicon (Si) and aluminum (Al), phosphorus (P), and sulfur (S). Trace amounts of iron (Fe), zinc (Zn), Nickel (Ni), and copper (Cu) were also present, each contributing to the total composition [23]. This is in confirmation with the analysis done by Fahad *et al*., who reported a high amount of Ca and the presence of other elements in lower quantities. This high CaO content boosts conversion by enhancing catalytic activity, increasing fuel yield, improving thermal stability, and supporting environmentally sustainable and cost-effective processes for PET conversion into fuel oil [23].

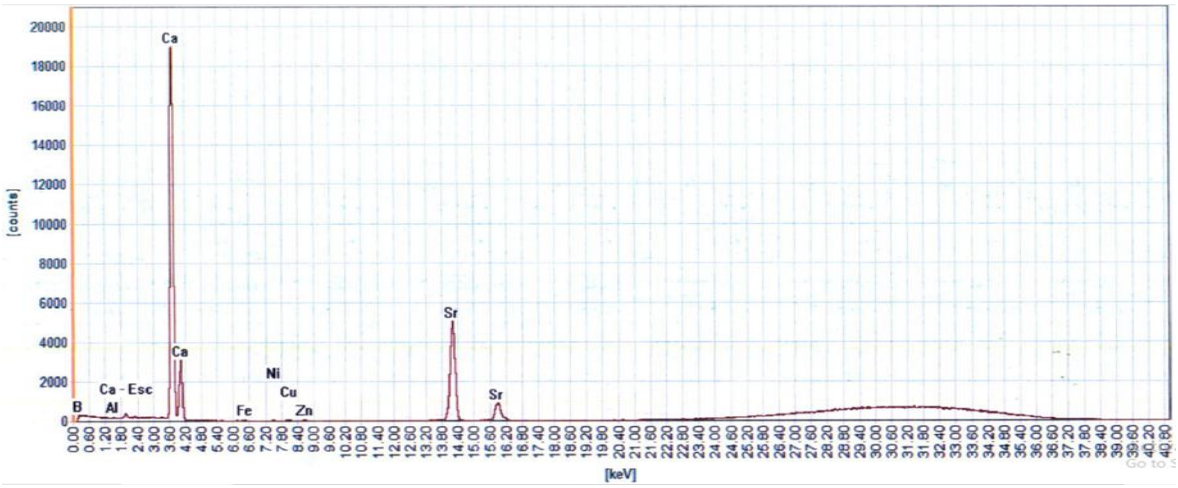

Figure 2: EDXRF Spectrum of Calcined Egg Shell Catalyst

3.1.2. X-ray diffraction (XRD) analysis

Figure 3 shows the X-ray diffraction (XRD) analysis, which revealed the crystallinity of the catalyst. It confirmed the presence of cubic CaO (lime) and hexagonal $Ca(OH)_2$ (portlandite) crystalline phases, which showed a strong similarity to the standard CaO file from ICDD (The International Centre for Diffraction Data) (JCPDS 37-1497) [20]. Characteristic CaO peaks were observed at 2θ = 32.5°, 35.0°, 37.5°, 54.0°, 65.0°, and 67.5°, while $Ca(OH)_2$ peaks appeared at 2θ = 18.0°, 34.0°,

51.0°, and 65.0° as Badnore *et al*. also documented a distinct XRD peak at 2θ = 37.42°, and the respective calculated densities were 3.34 g/cm³ and 2.20 g/cm³, with cell volumes of 111.35 × 10^6 pm³ and 54.46 × 10^6 pm³. These intense and sharp peaks indicate a well-defined crystalline structure dominated by calcium-based compounds [20].

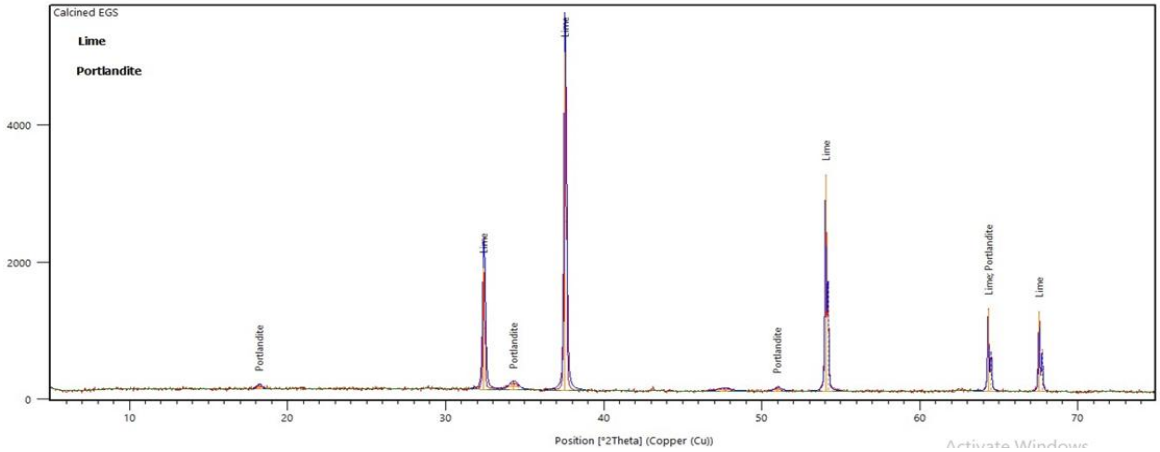

Figure 3: XRD Analysis of Calcined Eggshell Catalyst

3.1.3. FTIR analysis

The FTIR analysis identified key functional groups present in the calcined catalyst as shown in Figure 4. A strong peak at 1416 cm^{-1} corresponded to the carbonate ion (CO_3^{2-}), while a weak peak at 3641 cm^{-1} indicated the presence of hydroxyl (O–H) bonds which is connected to the surface of CaO, indicating a larger proportion of CaO as reported and confirmed by [18], [21]. Another minor peak at 872 cm^{-1} was attributed to epoxy and oxirant (C–O–) groups [24]. These functional groups influence the surface chemistry and adsorption behavior of the catalyst [18].

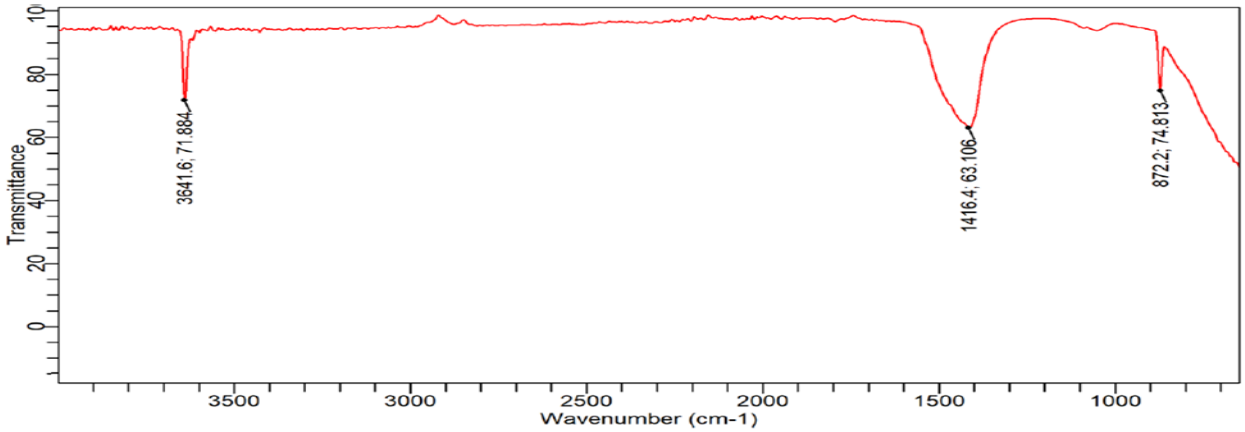

Figure 4: FTIR spectrum analysis of calcined chicken eggshell catalyst

3.1.4. Thermogravimetric (TGA) and Differential Thermal Analysis (DTA)

Figure 5 shows thermogravimetric (TGA) and differential thermal analysis (DTA), which revealed the thermal behavior of the catalyst. The first weight loss (<10%) occurred between 23.19°C and 320°C due to the release of adsorbed moisture and organics. A significant second-phase weight loss

(about 80%) between 320°C and 560°C indicated further dehydration and decomposition of carbonates. Beyond 560°C, a third phase involved thermal degradation, with complete decomposition near 846°C. DTA showed an initial endothermic peak at ~200°C and a sharp endothermic peak at 480°C, aligning with decomposition events in the TGA data, following the result reported by [24].

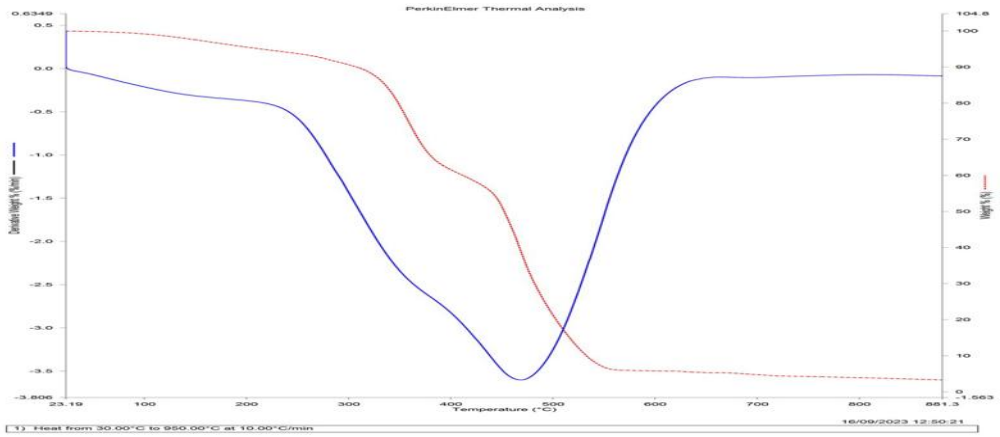

Figure 5: DTA/TGA of Calcined Eggshell Catalyst

3.2.Pyrolysis Performance of the Waste PET Bottles

Figure 6 presents the cumulative oil recovery under both catalytic and non-catalytic conditions and the corresponding visual trend. This result shows that the pyrolysis of the waste PET bottles, carried out at temperatures ranging from 200°C to 400°C, demonstrated an increase in oil recovery when using the calcined eggshell catalyst to the non-catalytic pyrolysis, with yields rising from 0 ml without catalyst to 7 ml with the use of the calcined eggshell catalyst at low temperatures between 200°C and 210°C, and at 254°C, oil recovery increases by 1200 % with the catalyst, from 1 ml to 13 ml, and at 300°C, by 133 % from 12 ml to 28 ml, showing a boost in oil yield by 20 to 1200 percent across this temperature range. As the temperature further increases, the catalyst-enhanced oil recovery increases, reaching the highest range at 147 ml at 400°C compared to 123 ml without a catalyst, representing a 19.5 % improvement. This shows that the introduction of the calcined eggshell catalyst substantially accelerated the thermal breakdown of the PET across the given temperature range, with the highest percentage recovery of 1200 % observed at 254 °C, leading to earlier and higher oil yields. This is also confirmed with the results reported for oxide catalysts by Kosloski-Oh *et al* [20].

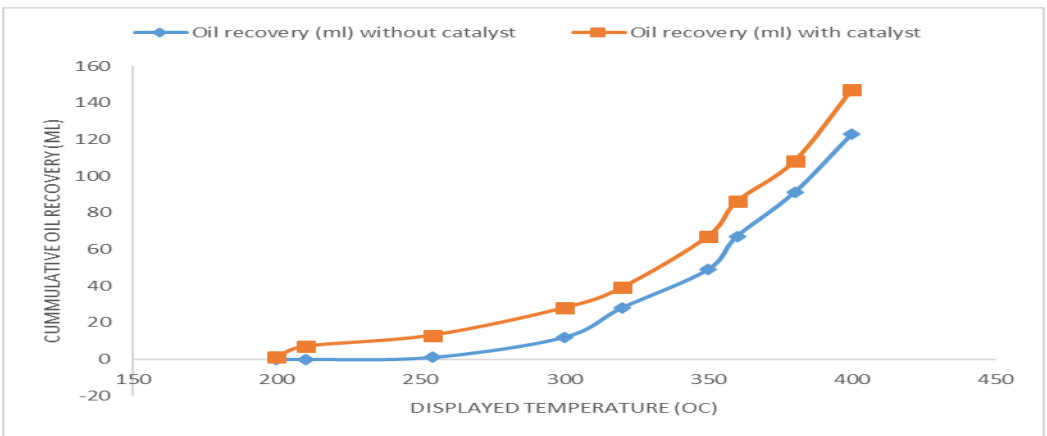

Figure 6: The Cumulative Oil Recovery of Pyrolysis of PET

3.3.Physicochemical and Chemical Characterization of Pyrolysis Oil

The pyrolysis oil obtained from waste PET bottles was subjected to a series of laboratory analyses to determine its physicochemical properties and chemical composition, with the results presented in Table 2. When compared to standard diesel fuel based on ASTM D975, the oil exhibited a lower calorific value (16,420 kcal/kg) but a higher flash point (78°C), comparable viscosity (2.80 mm^2/s), and acceptable specific gravity (0.8601), making it a promising candidate for energy recovery, an alternative fuel, and potential blending with conventional fuels [26].

Table 2: Physicochemical Properties of Pyrolyzed PET Oil vs Diesel (ASTM D975 Standard)

Property	Pyrolyzed PET Oil	Diesel Fuel (ASTM D975)
Calorific Value (kcal/kg)	16,420	42,500 – 45,500
Flash Point (°C)	78	≥52
Viscosity (mm^2/s @ 40°C)	2.80	1.9 – 4.1
Specific Gravity	0.8601	0.820 – 0.870

3.4.GC-MS Analysis of Oil from Pyrolyzed Waste PET Bottles

The GC-MS analysis of the pyrolyzed PET oil revealed a broad range of saturated hydrocarbons (C_9–C_{36}), indicative of a complex mixture of aliphatic compounds, with Pentatriacontane being the most abundant, a significant presence of long-chain alkanes, as shown in Table 3. This composition aligns with findings from similar studies by [27], [28], which reported the presence of long-chain hydrocarbons in pyrolyzed plastic oils. The prevalence of these long-chain alkanes contributes to the fuel-like properties of the pyrolysis oil, enhancing its potential as an alternative energy source [27], [28].

Table 3: Compounds analyzed from the pyrolyzed oil through GC-MS Analysis

Compound	R.T.	QIon	Response	Conc	Units		Dev(Min)
Target Compounds							Qvalue
1) Nonane	3.934	57	9191	0.04	mg/L	#	1
2) Decane	5.610	57	378547	4.18	mg/L	#	43
3) Undecane	6.864	57	866	Below Cal		#	1
4) Dodecane	8.306	57	1465	0.03	mg/L	#	46
5) Tridecane	9.719	57	288	Below Cal		#	1
6) Tetradecane	11.064	57	1092	Below Cal		#	1
7) Pentadecane	12.300	57	1049	Below Cal		#	1
8) Hexadecane	13.473	57	825	0.01	mg/L	#	13
9) Heptadecane	14.628	57	711	Below Cal		#	49
10) Pentadecane, 2,6,10,14...	14.686	57	1613	Below Cal		#	54
11) Octadecane	15.693	57	1616	Below Cal		#	31
12) Hexadecane, 2,6,10,14-...	15.801	57	1194	Below Cal		#	25
13) Nonadecane	16.660	57	270066	3.15	mg/L	#	7
14) Eicosane	17.243	57	1057647	9.54	mg/L	#	49
15) Heneicosane	18.336	57	256136	2.41	mg/L	#	1
16) Docosane	19.086	57	762030	6.95	mg/L	#	42
17) Tricosane	20.299	57	4150	0.11	mg/L	#	68
18) Tetracosane	21.152	57	32034	0.36	mg/L	#	1
19) Pentacosane	22.027	57	4792	0.06	mg/L	#	57
20) Hexacosane	22.765	57	1943	0.07	mg/L	#	39
21) Heptacosane	23.515	57	971	Below Cal		#	42
22) Octacosane	24.219	57	3166	0.01	mg/L	#	16
23) Nonacosane	24.922	57	3486	0.05	mg/L	#	13
24) Triacontane	25.660	57	4976	0.13	mg/L	#	1
25) Hentriacontane	26.221	57	12009	0.43	mg/L	#	6
26) Dotriacontane	26.874	57	5305	0.19	mg/L	#	36
27) Tritriacontane	27.451	57	2777	0.07	mg/L	#	62
28) Tetratriacontane	28.104	57	37414	1.86	mg/L	#	29
29) Pentatriacontane	28.756	57	262684	16.08	mg/L	#	41
30) Hexatriacontane	29.317	57	124737	11.38	mg/L	#	63

4. Conclusion

This study demonstrates the viability of using calcined eggshells as an effective and eco-friendly catalyst for the pyrolysis of waste PET bottles. The catalyst's efficiency was thoroughly assessed and characterized, and the physicochemical properties of the resulting pyrolysis products were analyzed. XRD and FTIR analyses confirmed the presence of cubic CaO (lime) and broad hydroxyl (O–H) bonds attached to the CaO surface, indicating a high CaO content that enhances the conversion of PET into fuel oil. The use of the eggshell catalyst resulted in a 1200% increase in oil yield, rising from 1 mL to 13 mL at a desirable temperature of 254 °C, compared to the non-catalytic reaction and the produced fuel exhibited a high calorific value of 16,420 kcal/kg, flash point of 78 °C, viscosity of 2.80 mm²/s, and specific gravity of 0.8601, demonstrating excellent fuel potential and safe handling characteristics when compared to the standard diesel fuel based on ASTM D975. This research highlights the effectiveness of eggshell biomass-derived catalysts in improving plastic waste valorization. It further supports the advancement of sustainable recycling technologies and promotes the development of low-cost, green solutions for waste-to-energy applications.

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