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Synthesis of a Biolubricant from Waste Cooking Oil Using Calcium Oxide from Animal Bones as a Heterogeneous Catalyst

[1]Aliu S.O., *[2]Ekebafe, L.O., [1]Mahmud, H., and [1]Anetokhe, M.A.
[1]Department of Chemical Engineering Technology, Auchi Polytechnic, Auchi, Nigeria.
[2]Department of Chemistry, University of Lagos, Akoka Campus, Lagos, Nigeria
*Corresponding Author: lawekebafe@gmail.com

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ABSTRACT

In this work, calcium oxide (CaO) obtained from animal bones was used as a heterogeneous catalyst to prepare a biolubricant from spent cooking oil. The cooking oil was transesterified with methanol with CaO as a catalyst. The biolubricant was obtained by further esterifying the resultant methyl esters with glycerol. FTIR, GC-MS, NMR, and TGA were used to characterize the biolubricant, which demonstrated good specific gravity (0.8943), density (0.8943), and flash point (0.8943). The results revealed that the biolubricant is suitable for use in a variety of industrial applications due to its cloud point (-8), pour point (-12), refractive index (1.45), total acid number (12.57), total base number (24.08), saponification value (165.70), iodine value (83.63), peroxide value (7.06), cetane number (79.24), calorific value (42.68), kinematic viscosity (9.75), and high viscosity index (75.80). It was discovered that using calcium oxide produced from animal bones as a heterogeneous catalyst was very successful in producing a high conversion rate, good product yield, and a viable strategy for the environmentally friendly and sustainable production of lubricants.

Keywords*:* Biolubricant, calcium oxide, waste cooking oil, catalyst.

INTRODUCTION

A bio-lubricant is a type of lubricant that is made without the use of petroleum. It is made from renewable resources like vegetables or animal fats. Compared to petroleum-based lubricants, biolubricants are usually less toxic and biodegradable, making them more environmentally friendly. They are employed in different applications, such as industrial machinery, hydraulic systems, and automobile engines. Because more sectors are calling for lubricants that are less harmful to the environment, the market for biolubricants is predicted to expand. Businesses can lower their carbon footprint and adhere to sustainability laws by using biolubricants. The creation of novel biolubricant formulations with enhanced performance attributes is also anticipated to propel market expansion. Bio-based lubricants are growing in popularity as they are more environmentally friendly and perform better than conventional lubricants [1].

Because it is inexpensive and readily available, waste cooking oil (WCO) is a viable feedstock for the creation of bio-based lubricants. Calcium oxide in animal bone is frequently employed as a heterogeneous catalyst in the synthesis of several biofuels and chemicals [2].

Using calcium oxide from animal bones as a heterogeneous catalyst, waste cooking oil can be converted into a biolubricant by esterifying free fatty acids (FFAs) and transesterifying triglycerides into fatty acid methyl esters (FAMEs) [3-5]. There are various benefits to using CaO as a catalyst, characteristics including affordability, accessibility, and quick response. Moreover, animal dung might serve as a possible supply of calcium oxide and can be obtained by a straightforward calcination procedure [6].

Animal bones contain calcium oxide which can be used as a catalyst to produce chemicals and biofuels, according to a number of studies. For instance, a study demonstrated the manufacture of biodiesel from waste cooking oil utilizing calcium oxide generated from chicken bones as the catalyst [7]. The study discovered that, with a 97.32% conversion rate, CaO acted as a useful catalyst for the synthesis of biodiesel. A different study described employing calcium oxide in pig bone as a catalyst to produce bio-based lubricants from soybean oil [8-9]. The results of the investigation showed that CaO, which has a viscosity index of 142 and a pour point of -30°C, was a useful catalyst for the synthesis of bio-based lubricants. A biolubricant made from spent cooking oil, using as calcium oxide a heterogeneous catalyst is a viable method for producing environmentally friendly lubricants [10]. One of the many benefits of using CaO as a catalyst is its easy availability and inexpensive cost. CaO from animal bones has been successfully used in a number of experiments to produce biofuels and chemicals, suggesting that it may also be used to produce bio-based lubricants [11-13].

In spite of significant progress in the conversion of waste cooking oil into biodiesel and biolubricants, the application of animal bone derived calcium oxide as a heterogeneous catalyst remains underexplored, particularly for biolubricant production. This limitation constrains the development of low cost, sustainable systems that integrate waste valorization with renewable energy materials. In this context, the present study proposes a novel approach utilizing animal bone derived calcium oxide for the synthesis of biolubricant from waste cooking oil, thereby contributing to circular resource utilization and sustainable energy development.

The aim of this study is to synthesize and characterize a biolubricant from waste cooking oil using animal bone derived calcium oxide as a heterogeneous catalyst. Specifically, the study seeks to: (i) pre-treat the waste cooking oil feedstock; (ii) synthesize and characterize the CaO catalyst; (iii) perform transesterification and esterification reactions; and (iv) evaluate the physicochemical, thermal, and spectroscopic properties of the resulting biolubricant to assess its suitability for renewable and industrial applications.

MATERIALS AND METHODS

Collection and Pre-treatment of Waste Cooking Oil

Waste cooking oil was collected from a local restaurant and was pre-treated to remove impurities by heating it, then cooling and filtering it through a filter paper (Plate 1).

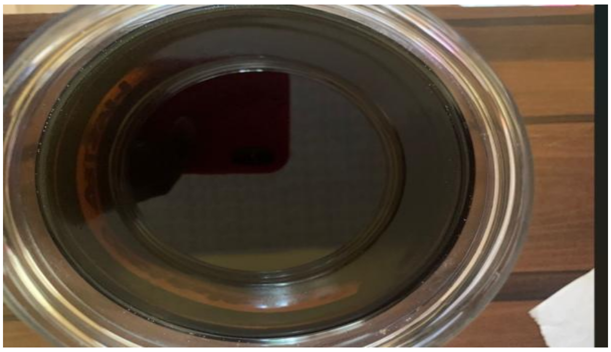

Plate 1: Pre-treated Waste Cooking Oil

Preparation of Calcium Oxide Catalyst

The bones of the animals were taken from a nearby butcher and carefully prepared to eliminate any soft tissues. To achieve a successful calcination process, animal bones are modified beforehand by cleaning and preparing the bones. The actions taken are:

Cleaning: This is the process of getting rid of any leftover meat, fat, or other biological matter from the bones. Using a combination of scraping and soaking in a soapy solution, the bones were boiled in water.

Degreasing: Natural oils found in bones may obstruct the calcination process. The bones were immersed in a degreasing solution—a concoction of dish soap and water—for a few days in order to get rid of these oils. The solution was frequently altered.

Drying: To cleaned bones were dried in an oven for 24 hours at 100°C. Throughout this procedure, it was ensured that the bones were not burned or overheated (Plate 2).

Plate 2: Dried Sized Animal Reduced Bones

Size Reduction: Smaller bits of the dried bones were crushed with a hammer.

Calcination: The bones were put in a furnace and progressively heated up to 900°C. For three hours, the bones were maintained at this temperature to guarantee full calcination.

Cooling: In order to prevent damage or cracking, the bones were cooled gradually over several.

Grinding: A mortar and pestle were used to grind the calcined bones.

Sieving: The ground bones were sieved using a mesh size of 0.063 micrometers. After that, the particles became agglomerated, which were taken out and placed in a desiccator to prevent a reaction with moisture or air and preserved for additional examination in an airtight container.

Characterization of Calcium Oxide Produced from Animal Bones

The oxide compositions of the catalyst were ascertained using an X-Ray fluorescence (XRF) spectrophotometer in order to characterize the calcium oxide generated from animal bones. Also, they were examined using a scanning electron microscope (SEM) to determine the surface morphology. Using an X-Ray diffractometer (XRD) fitted with a Cu anode, the crystalline components in the catalyst were identified. Fourier transform infrared spectroscopy was used to assess the functional groups in the samples, and the acquired wavelength area of the samples' spectra was noticed. Using the Bruneian-Emmett-Teller method, the catalyst's surface area was calculated.

Determination of Percentage Loss on Ignition

A sample weighing (1.0 g) (W1) was heated to a temperature of between 900 and 1,000°C, cooled, and then weighed in a platinum crucible at 25°C. A second heating at the same temperature for five minutes was used to verify the weight decrease, and the content was weighed again. This process was repeated until a constant weight was reached and the result was recorded as W2. The decrease in weight after ignition was attributed to weight loss and the percentage loss on ignition was determined as follows:

$W_3 = W_1 - W_2$,

% Loss of ignition = $\frac{(w_3 \text{ x } 100)}{w1}$

Where:

W1 = weight of sample taken.

W_3 = Loss in weight.

Transesterification Reaction

Using the procedure of Lani et al [14] a batch reactor was used to assess the heterogeneous catalyst's performance for esterification and transesterification. The waste cooking oil underwent esterification and transesterification in a 250 ml conical flask set up on a magnetic stirrer with a constant temperature. Generally, 4 g of CaO catalyst was added to 100 g of pre-treated waste cooking oil in a 500 ml conical flask. Then, 25 ml of methanol and 5 ml of H_2SO_4 were added to a beaker and constantly swirled until the H_2SO_4 completely dissolved in the methanol. The mixture was added to the flask, which was set on a magnetic stirrer with temperature control, and the contents heated to 65 ^oC while being stirred for two hours. To avoid losing methanol during the reaction, the stirrer speed was kept constant, and the conical flask was appropriately lag-timed. After the reaction, the reaction mixture was cooled to room temperature, and a cotton filter was used to filter the catalyst out of the mixture [15]. A separator funnel was used to divide the mixture's upper layer into distinct phases.

To get rid of any remaining catalyst and contaminants, the separate layer was cleaned using distilled water and a 10% NaOH solution. The washing procedure was repeated until the cleaned water seemed clear. After cleaning, the layer was filtered through a Buchner funnel and dried over 1 g of $MgSO_4$ for 30 minutes at 105 ^oC.

Plate 3: Transesterification Process of Biolubricant after Washing

Esterification Reaction

For the transesterification procedure, 10 milliliters of glycerol were added to the liquid after it had cooled (Plate 3). For an hour, the mixture was stirred at 80 °C. After that, the mixture was moved to a separatory funnel so that it could be divided into several phases. Two strata were distinguished and segregated. The biolubricant-containing upper layer and the glycerol-containing bottom layer are the two phases. After all, the biolubricant was produced (Plate 4).

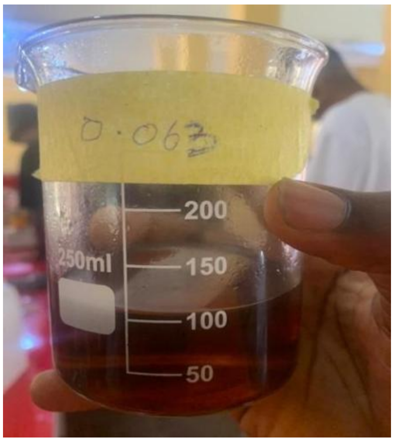

Plate 4: Biolubricant prepared from this study

Characterization of Biolubricant

The synthesized biolubricant was characterized through a variety of analytical techniques, including nuclear magnetic resonance spectroscopy (NMR) to confirm the structure of the biolubricant and monitor the reaction progress, thermo gravimetric analysis (TGA) to determine the thermal properties of fatty esters, and gas chromatography-mass spectrometry

(GC-MS) to analyze the composition of the bio lubricant, identify and quantify the various components, and determine the molecular weight and structure of the compounds present. The biolubricant's physio-chemical characteristics will also be assessed.

RESULTS AND DISCUSSIONS

The physiochemical characteristics (Table 1) show that the prepared biolubricant satisfies the international standard specification when compared to the ASTM and EN biolubricant specifications. Additionally, it was noted that the observed flash point of 120^{0}C exceeded the standard specification. This indicates that biodiesel has a lower energy density and is therefore safer to handle than biolubricants with low flash points, which are too dangerous due to their easy ignition and propensity to settle oxides on metal surfaces.

Table 1: Physiochemical properties of the biolubricant prepared from this study

Parameters	Biolubricant
Physical State	Liquid
Specific gravity (40^{o}C)	0.8943
Kinematic viscosity (40^{o}C) (cP)	9.75
Viscosity Index	75.80
Density (40^{o}C) (g/cm^3)	0.8943
Saponification value (mgKOH/g)	165.70
Iodine value (gI_2/100g)	83.62
Peroxide value (mg/g)	7.06
Refractive index	1.45
Total acid number (mgKOH/g)	12.57
Total base number (mgKOH/g)	24.08
Flash point (^{o}C)	128
Pour point (^{o}C)	-12
Cloud point (^{o}C)	-8
Cetane number	79.24
Calorific value (MJ/kg)	42.68

Fourier transform infrared spectrometry

The result of FTIR analysis of the biolubricant is shown in Figure 1 and Table 2. It revealed that the O-H bond stretch resulted in an early broad 2998.85 cm^{-1} (Table 2).

Table 2: FTIR Analysis

Run #	Peak Wavelength(cm^{-1})	Transmittance (%)	Assignment	Functional group
1	2998.85	28.41	O-H stretching vibrations as a result of some impurities.	Hydroxyl group
2	2901.08	31.26	C-H stretching vibrations in the hydrocarbon component of the bio-lubricant	Alkanes
3	2798.26	76.90	C=O stretching vibrations	Esters
4	1507.32	47.16	C═O stretching vibration	Carbonyl
5	1405.18	56.86	C–H scissoring and bending for methylene group	Methylene group
6	1398.75	76.53	C-H bending vibration	Alkanes
7	1146.05	78.02	C–O stretching vibration	Ester
8	789.96	69.54	C–H group vibration	Aliphatic

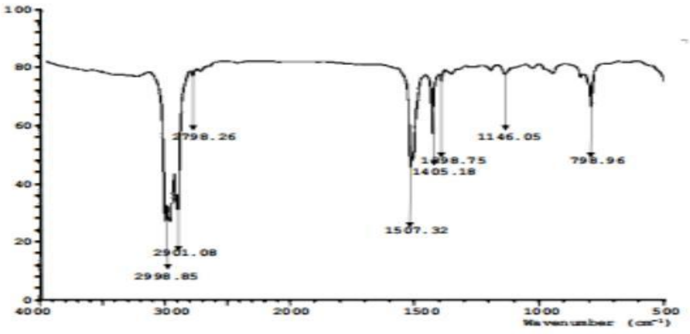

Figure 1: FTIR Spectrum of the Biolubricant

Gas chromatography-mass spectrometry analysis

The chemical composition of the prepared biolubricant was examined. The total ion chromatogram showed sixteen main peaks (Figure 3). Based on the library match, each peak was determined to correlate to the biodiesel's fatty acid methyl ester concentration. The findings indicated that dodecanoic acid, octadecatrienoic acid, octadecenoic acid, and hexadecanoic acid were the main fatty acid components in the esters. During the transesterification process, ethyl ester formation was somewhat less than methyl ester formation. This could be the result of a larger concentration of methoxide radicals than ethoxide radicals. The most prevalent peak heights of the FAME component are easily recognized since they are uniformly spaced. As can be seen above, there is little variation in the biolubricant components' peak heights over range.

Below (Figure 2) are MS-chromatograms that illustrate how the components of the biolubricant samples were separated. The majority of the diesel's constituents are alkanes.

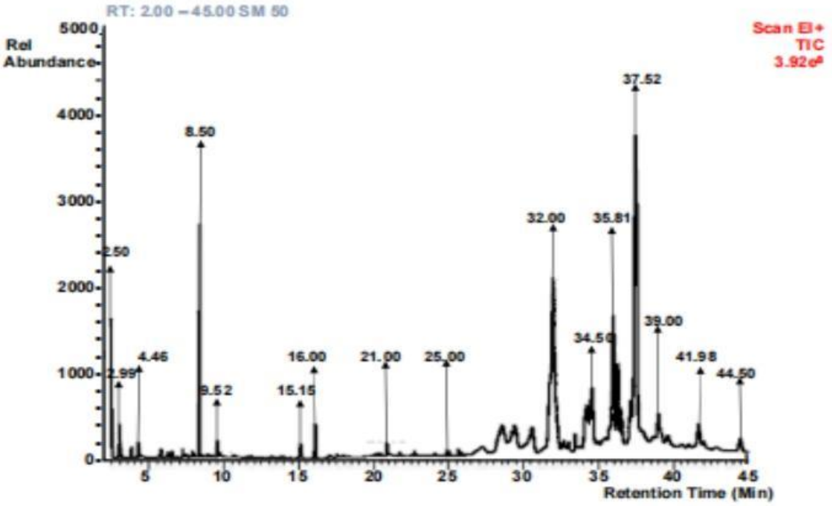

Figure 2: GC/MS Results of Biolubricant

Thermogravimetric analysis

Figure 3 shows the catalyst sample's TGA. A significant decrease in weight was noted within the temperature range of 650^{0}C to 900^{0}C. The loss of organic components from the animal bones may be the cause of this. The material's weight decreased negligibly above 650^{0}C, confirming that animal bones were converted to calcium oxide. Comparable outcomes were documented [16]..

Table 3: Biolubricant TGA Analysis

S/N	Temperature (^{0}C)	Sample
		Biolubricant
1	200.00	75.99
2	250.00	76.03
3	300.00	76.24
4	350.00	76.01
5	400.00	70.86
6	450.00	73.61
7	500.00	70.83
8	550.00	70.89
9	600.00	61.48
10	650.00	33.68
11	700.00	24.15
12	750.00	24.13
13	800.00	24.60
14	850.00	24.76
15	900.00	4.73

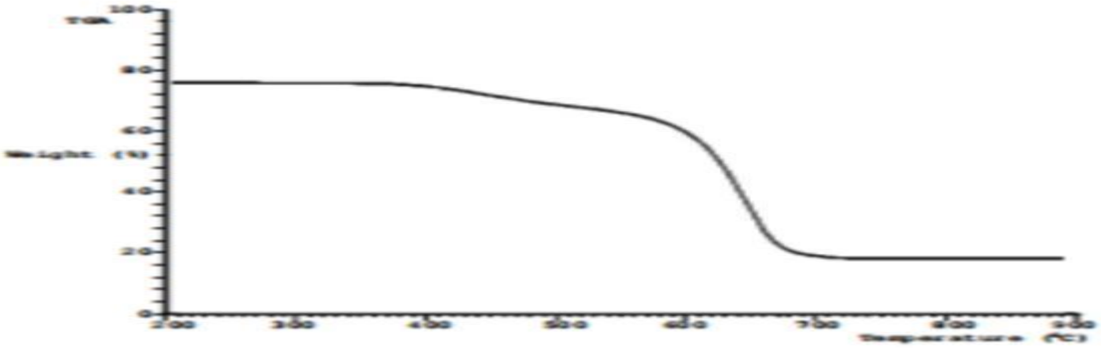

Figure 3: TGA Results of the Biolubricant.

Nuclear magnetic resonance spectrometry

The NMR spectrum exhibits characteristic chemical shifts consistent with the structural framework of fatty acid esters, confirming the successful conversion of waste cooking oil into a biolubricant (Figure 4). The dominant signals in the aliphatic region (0.8 - 1.5 ppm) correspond to terminal methyl and methylene protons of long hydrocarbon chains, indicating the preservation of extended aliphatic structures essential for desirable lubrication properties such as high viscosity index and effective film formation. Signals between 1.6 and 2.5 ppm, particularly the peak near 2.3 ppm, provide strong evidence of ester formation through the presence of α- and β-methylene protons adjacent to the carbonyl group. Additionally, peaks in the 2.0 - 2.8 ppm range suggest the retention of some unsaturation within the fatty acid chains, which may enhance low-temperature performance.

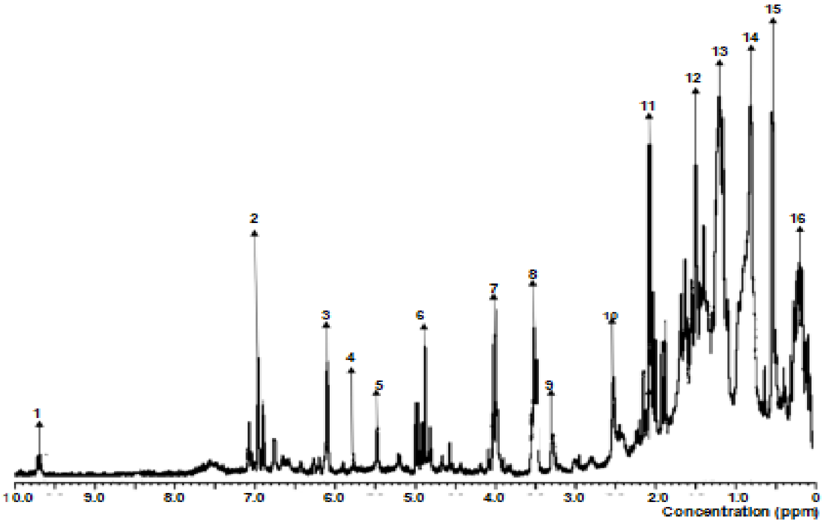

Figure 4: NMR of Biolubricant

In the functional group region (3.5 - 5.5 ppm), resonances associated with methoxy and glyceridic protons indicate substantial conversion of triglycerides into ester-based products, although minor residual glyceridic signals suggest the possible formation of more complex esters. The presence of olefinic protons around 5.3 ppm further confirms carbon - carbon double bonds within the structure. Weak signals in the downfield region (7 - 10 ppm) are attributed to trace oxidation products or impurities, indicating minimal degradation during processing.

Catalyst characterization

Fourier transform infrared spectrophotometer

The purpose of the FTIR analysis was to find out how the functional groups of the raw and calcined samples had changed. The different functional groups that are present in the sample are represented by the peaks in the spectrum. As seen in Figure 5, the analysis revealed an early broad peak representing O-H bond stretch at 3207.95 cm^{-1}. For the bio catalyst, further notable distinctive peaks were found at 2508.36 cm^{-1}, 1850.01 cm^{-1}, 1428.96 cm^{-1}, etc. Additionally, the biolubricant table revealed that the O-H bond stretch resulted in an early broad

2998.85 cm^{-1}.

Table 4: FTIR Analysis of Bio catalyst

Run #	Peak Wavelength(cm^{-1})	Transmittance (%)	Assignment	Functional group
1	3207.95	62.75	O-H stretching vibrations	Hydroxyl group
2	2508.36	73.98	C-H stretching vibrations	Aliphatic groups
3	1850.01	74.10	C=O asymmetric stretching vibrations of the carboxylate groups	Carboxylic
4	1428.96	66.24	C═O symmetric stretching vibrations of the carboxylate group	Carboxylic
5	1383.37	65.70	C–O asymmetric stretching vibration	Alcohol
6	998.67	40.06	C-O stretching vibration	Ester
7	799.25	56.13	C-H bending vibration	Alkanes
8	658.49	68.21	≡Si–OH bending vibration	Silanol
9	552.50	39.87	-Si-O-Si bending vibration	Silica

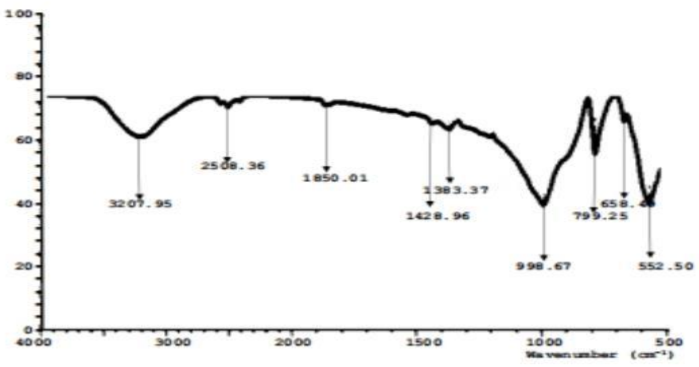

Figure 5: FTIR Spectrum of the Catalyst

Scanning Electron Microscopy Analysis

An invaluable tool for characterizing materials of all sizes and forms, the scanning electron microscope is crucial to the study of nanoscale structures.

The shape of the manufactured samples was ascertained using SEM (SEM thitachi su 3500 scanning microscope, Tokyo, Japan). SEM was used to examine the surface morphology of the catalyst and feedstock, as seen in Figure 6. The presence of diverse pore diameters and particle sizes was revealed by the SEM imaging. The irregular form and texture of the particles are caused by randomly distributed pores in the structure that range in size from micrometers. Particles of all sizes and shapes were present. Higher specific surface areas are produced by the aggregates' reduced size.

Table 5: SEM Analysis for Catalyst

Element	Composition (%)
Carbon	3.98
Oxygen	62.15
Magnesium	7.10
Iron	0.18
Sodium	1.09
Calcium	3.40
Aluminum	0.92
Potassium	7.86
Chlorine	0.30

Titanium	4.64
Sulphur	6.73
Phosphorus	1.60
Copper	0.03

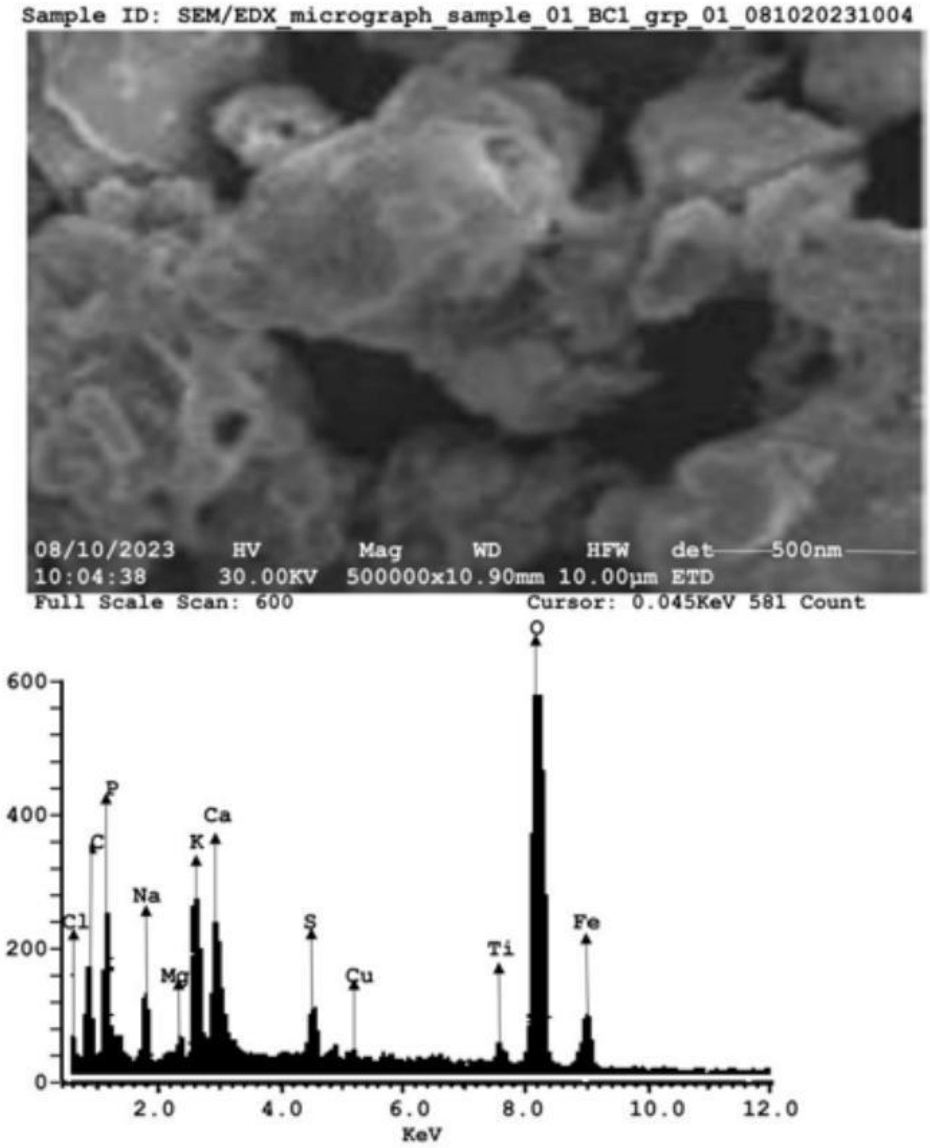

Figure 6: SEM Imaging of Catalyst

Brunauer-Emmet-Teller Surface Area Analysis

Tables 6 and 7 display the surface characteristics and pore diameter data, respectively.

Table 6: BET Surface Parameters

Methods	Surface Parameters		
	Sample weight (g)	Saturated pressure (kPa)	Degassing Time (hr)
N_2 Adsorption-desorption	0.30	95.00	3.00

Table 7: BET Surface Area and Pore Values

Surface area ($m^2\ g^{-1}$)	Pore volume (($cm^3\ g^{-1}$)	Pore Diameter (nm)
3.1076	0.0493	19.308

The BET analysis of the prepared catalyst showed a surface area of 3.1076 m^2g^{-1} and a micro pore volume of 0.0493 cm^3g^{-1} which allows easy diffusion of reactants into the interior surface of the catalyst. The generated catalyst's surface area of 3.1076 m2g-1 and micropore volume of 0.0493 cm^3g^{-1}, as shown by the BET study, facilitate facile reactant diffusion into the catalyst's interior surface. The BET method's intrinsic error-introducing elements, such as the assumption of a perfect condition, the effectiveness of the desiccators utilized, and the purity of the salts used, resulted in an average inaccuracy of +9.0667% of the actual BET surface area values expected. The BET method used gave average error of +9.0667% of the actual BET surface area values expected due to some inherent error introducing factors associated with the method such as: ideal situation assumption, efficiency of the desiccators used, purity of the salts used.

X-Ray Fluorescence

The Table's 7 conclusion indicates that the highest ingredients in the doors catalyst were silicon oxide (32.47%), CaO (28.11%), and aluminum oxide (8.14%). The basic strength of the catalyst is the sole thing that these metal oxides do [18]. One clue that calcium oxide made from animal bones is composed of CaO is the presence of silicon oxides and magnesium oxide as the main constituents. With strong basic and acidic properties, this biocatalyst is advantageous for the synthesis of biolubricants.

Table 8: XRF Catalyst Analysis

S/N	Basic Oxides	Formulae	% Composition
1	Silicon Oxide	SiO_2	32.47
2	Aluminum Oxide	Al_2O_3	8.14
3	Magnesium Oxide	MgO	2.83
4	Calcium Oxide	CaO	28.11
5	Iron Oxide	Fe_2O_3	6.74
6	Potassium Oxide	K_2O	7.65
7	Sodium Oxide	Na_2O	1.18
8	Phosphorus Oxide	P_2O_5	2.27
9	Zinc Oxide	ZnO	0.05
9	Barium Oxide	BaO	0.05
10	Manganese Oxide	MnO	3.71
11	Titanium Oxide	TiO_2	0.29
14	Sulphide	SO_3	3.09
15	Loss of Ignition	LOI	3.39

X-ray diffraction analysis

The calcined sample's x-ray diffraction (XRD) pattern is displayed in Figure 7. The catalyst's XRD examination reveals a distinct peak representing the separated metal oxide crystalline phases of oxygen and calcium, which is consistent with the catalyst's produced composition. Comparably steep peaks were also seen for the animal bones; from a chemical perspective, these bones could be used to make CaO, which could be used as a catalyst to produce biolubricant. These peaks resemble those that have been documented in comparable circumstances [17]..

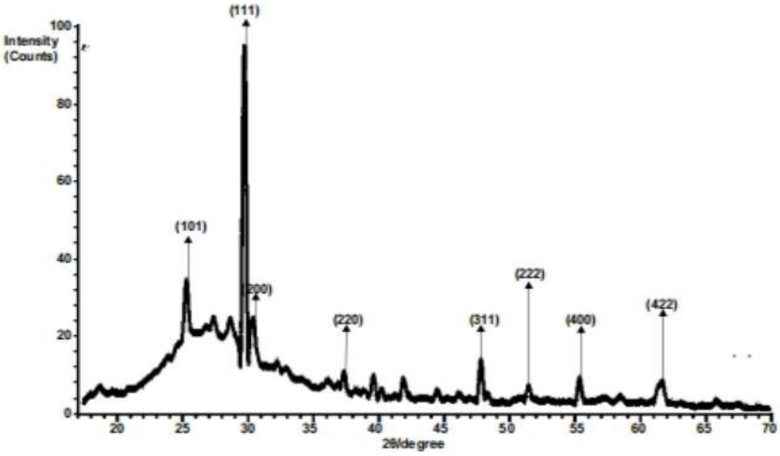

Figure 7: XRD for Calcined Catalyst

CONCLUSION

A viable method for producing lubricants in a sustainable manner is the synthesis of a biolubricant from leftover cooking oil by employing calcium oxide obtained from animal bones as a heterogeneous catalyst. By using used cooking oil as a feedstock, the environmental impact of disposing of it is lessened, and lubricant synthesis can be accomplished at a reasonable and sustainable cost. An affordable and environmentally beneficial substitute for conventional catalysts is CaO obtained from animal bones. It has been demonstrated that the CaO catalyst works very well for transesterification of used cooking oil, with a high conversion rate and good product yield. Its appropriateness for usage is demonstrated by the synthesized biolubricant's performance evaluation and characterization utilizing a variety of analytical techniques and physio-chemical property tests. show that it is appropriate for use in a range of industrial settings. The synthetic biolubricant is a great substitute for lubricants that are sold in stores since it has good viscosity index, oxidation resistance, and thermal stability. The SEM study demonstrated the high surface area and huge pore volume of the catalyst that was produced by calcining animal bones. This means that with low-quality feedstock, such used cooking oil, animal bones can be a good replacement for convectional catalysts. The TGA study revealed that the generated catalyst exhibited a high degree of thermal stability as well.

All things considered, this work offers a sustainable method for turning leftover cooking oil into biolubricants by employing a calcium oxide catalyst made of animal bones. This strategy offers a great chance to advance sustainable development while lowering contamination of the environment and reliance on nonrenewable resources.

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