



“Synthesis of Biodiesel from Moringa Oleifera Seeds Oil Using Eggshell-Derived Cao Nanocatalyst: An Eco-Friendly Approach”

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KEYWORDS

Moringa oleifera, biodiesel, eggshell nanocatalyst, transesterification, and Biodiesel

ABSTRACT:

Introduction: The persistent use of fossil fuels has major environmental consequences, particularly due to the emission of greenhouse gases like carbon dioxide (CO₂), which play a key role in global warming and climate change. Biodiesel has become a viable and promising substitute for traditional petroleum diesel.

Objectives: The present study explores the production and evaluation of biodiesel derived from Moringa oleifera seeds oil (MOSO) using a waste eggshell derived nanocatalyst.

Methods: The research focuses on the characterization of the physicochemical properties, fatty acid composition, synthesis of Moringa oleifera biodiesel (MOBD)EN using transesterification and analysis of fuel properties of in comparison with international biodiesel standards (ASTM D6751 and EN 14214) and earlier research reported.

Results: The Moringa oleifera seeds reported to have high oil content of 45.24%. The MOSO was found rich in oleic acid (76.7%) was trans esterified using (CaO)EN. The synthesized (CaO)EN was characterized by DLS, TGA-DTA, SEM and XRD techniques. The resulting (MOBD)EN exhibited favorable properties such as high cetane number (50), appropriate viscosity and density, low ash content (0.18), and a high flash point, making it a safe and efficient fuel alternative. The (MOBD)EN have high calorific value and biodiesel yield i.e. 52kJ/kg and 95.80% respectively.

Conclusion: The overall results confirm the viability of MOSO as a non-edible, sustainable feedstock for biodiesel production. The use of an eco-friendly nanocatalyst derived from eggshells further enhances the environmental and economic feasibility of the process. This study highlights the potential of integrating green chemistry and waste valorization for the development of next-generation biofuels.

Introduction:-

By 2050, the world's population is expected to exceed 9 billion people, leading to higher energy demand and rising costs due to the limited availability of conventional energy sources [1–4]. The continued use of fossil fuels also has serious environmental impacts, especially through the release of greenhouse gases like carbon dioxide (CO₂), which contribute to global warming and climate change [5,6]. Biodiesel has emerged as a promising alternative to petroleum-based diesel. It is biodegradable, non-toxic, and produces much lower emissions of CO₂ and sulfur oxides (SO_x)

[7]. For a biofuel to be a good substitute, it must offer environmental benefits, be cost-effective, provide more energy than it consumes to produce, and be available in large quantities without affecting food supplies. Common sources for biodiesel include edible oils like soybean, rapeseed, canola, sunflower, and palm oil [8]. However, due to rising concerns about food versus fuel use, attention has shifted toward non-edible oils. These are often more affordable and readily available than edible oils [9–12]. Biodiesel is made by a chemical process called transesterification, where oil reacts with alcohol in the presence of a catalyst. This can be done



using either homogeneous or heterogeneous catalysts. Heterogeneous catalysts are preferred because they are easy to separate from the product and can be reused [13]. Calcium oxide (CaO) derived from waste egg shells is one such heterogeneous catalyst that shows promise in biodiesel production. However, unlike homogenous catalysts, heterogeneous catalysts often take longer to complete the reaction. This is mainly because of slow mixing between the three components: oil, alcohol, and catalyst [14]. The low reactivity and small surface area of CaO can reduce its effectiveness, requiring higher amounts of catalyst and longer reaction times, which increases production costs. Improving the basic strength and surface area of CaO can enhance its performance. This can be achieved by reducing the particle size to the nanoscale, which increases the surface area and improves the catalyst's activity [15]. Eggshells, which are usually thrown away as waste in landfills without much processing, are a rich and low-cost source of calcium. They have already been used in animal feed, as a calcium supplement in medicines, as soil enhancers, and even in human nutrition [16-20]. Since eggshells are mainly made of calcium carbonate, which can be converted to CaO, this study focuses on using calcined eggshells as a heterogeneous nanocatalyst to produce biodiesel from *Moringa oleifera* seed oil. The finally produced biodiesel (MOBD)_{EN} is then evaluated and compared with fuel quality standards set by ASTM-D6751 and EN-14214 [21,22].

1. Materials and Methods:-

Collection and Preparation of *Moringa oleifera* Seeds

Moringa oleifera seeds were collected from the different location of Jodhpur. The seeds were manually dehulled to remove the outer shells and thoroughly rinsed with water to eliminate dust and other impurities. They were then air-dried under shaded conditions to prevent degradation. Once dried, the seeds were grounded into a fine powder using a mortar and pestle.

Oil Extraction

The powdered seed material was subjected to oil extraction using a Soxhlet apparatus, with *n*-hexane as the solvent. The extraction was carried out for 8–10 hours. Post-extraction, the solvent was removed through evaporation to obtain MOSO.

Preparation of (CaO)_{EN}

Eggshells were sourced from local poultry vendors and thoroughly washed to remove organic residues. The cleaned shells were dried to eliminate moisture and subsequently grounded into a fine powder. This powder was then calcined in a muffle furnace at 900°C for 4 hours to synthesize calcium oxide-based eggshell nanocatalyst (CaO)_{EN}.

Transesterification Process

The transesterification reaction was initiated by mixing the MOSO with methanol in a molar ratio of 1:6. The (CaO)_{EN} was added to this mixture, which was then heated to a temperature range of 60–70°C for 1 to 2 hours to ensure proper mixing and reaction.

Drying and Filtration

The washed biodiesel was dried to eliminate residual moisture and then filtered to remove any remaining solid particulates, yielding purified (MOBD)_{EN}.

Physicochemical Characterization of MOSO

The physicochemical parameters of the crude MOSO, such as saponification value (SV), iodine value (IV), etc. were evaluated using standard methods established by the American Oil Chemists' Society (AOCS, 1997) [23]. The refractive index was determined using an Abbe's refractometer.

Characterization of MOSO by FT-IR

Fourier Transform Infrared Spectroscopy (FT-IR) was employed to identify functional groups present in MOSO, Thermo Fisher Scientific Instrument.

Fatty Acid Profiling of MOSO by GC-MS

The fatty acid methyl ester (FAME)s composition of MOSO was analyzed using a Thermo Scientific TSQ 8000 Gas Chromatography-Mass Spectrometry (GC-MS) system.

Morphological and Thermal Characterization of ESN

DLS- The DLS of the ESN is performed by using Zetasizer Nano ZSP (ZEN 5600) instrument. It used for particle size measurement down to 0.3 nm, zeta potential determination of nanoparticles and surfaces, and other related characterizations.

XRD- The X-Ray Diffractometer (Panalytical X'Pert Pro) is a high-precision analytical instrument used for identifying and characterizing (CaO)_{EN} to determine its



crystal structure, phase composition, and crystallite size.

SEM analysis-The surface morphology of the (CaO)_{EN} catalyst was investigated using Scanning Electron Microscopy (SEM) with a Nova Nano FE-SEM instrument.

TGA-DTA analysis- Thermal stability and decomposition behavior of (CaO)_{EN} analyzed using Thermogravimetric Analysis and Differential Thermal Analysis (TGA-DTA) via a Perkin Elmer Diamond TG/DTA instrument.

Fuel Property Analysis of (MOBD)_{EN}

The produced biodiesel was analyzed for fuel-relevant properties including kinematic viscosity, density, flash point, cetane number, and acid value etc. These tests were performed according to standardized protocols from ASTM- D6751 and European Norm EN-14214. The measured fuel properties of (MOBD)_{EN} were compared with the specifications outlined in ASTM-D6751, EN-14214 standards and earlier reported results to assess its compliance and viability as an alternative biodiesel fuel.

2. Result and Discussion:

Physicochemical Analysis of MOSO

The physicochemical properties of MOSO is given in the table-1. The *M. oleifera* seeds exhibited an impressive oil yield of 45.24%, indicating its potential as a significant source of oil. The moisture content was extremely low (0.02%), suggesting good stability and minimal risk of hydrolytic degradation. Wiltshire FM et al. reported low crude oil yield of *Moringa oleifera* seeds 34.68% and the moisture content was quite high (0.048%) as compared to the present research [24]. SV of 168 mg KOH/g implies the presence of medium-

chain triglycerides, suitable for soap and cosmetic formulations. IV of 70.55 g I₂/100g oil classifies it as a non-drying oil with unsaturation, making it suitable for edible and industrial applications. Fu. X et al., reported high SV, and low IV of MOSO were found to be 189.84 g KOH/kg oil, and 66.78 g I₂/100 g oil, respectively as compared to present work [25]. The refractive index (1.456) and relative density (0.895 Kg/m³) are within the typical range for oils, confirming its purity and identity.

Table 1: Physicochemical properties of MOSO.

Parameters	MOSO
Oil (%)	45.24
Moisture content (%)	0.02
Saponification value (mg KOH/g)	168
Iodine value (gI ₂ / 100g oil)	70.55
Refractive index	1.456
Relative density (Kg/m ³)	0.895
FFAs (%)	0.52
Unsaponifiable matter (%)	0.85

FT-IR Analysis of (MOSO):-

The FT-IR spectral analysis of MOSO revealed the presence of key functional groups characteristic of natural triglyceride-based oils. The interpretation of the FT-IR peaks is given in the table 2. The spectra for the same is given in the figure-1.

Table-2 FT-IR Interpretation with Wavenumber Ranges

Wavenumber (cm ⁻¹)	Range	Functional Group / Vibration	Interpretation
3000 – 3500		=C–H stretching	Indicates cis double bonds (unsaturated fatty acids).
2750 – 2950		C–H stretching (–CH ₂ – and –CH ₃)	Aliphatic hydrocarbon chains (common in fatty acids).
1600 – 1850		C=O stretching	Ester carbonyl group from triglycerides.
1250 – 1500		CH ₂ bending (scissoring) CH ₃ symmetric bending (umbrella)	Methylene groups in long carbon chains



	mode)	Methyl groups of fatty acids.
720 – 730	(CH ₂) _n rocking	Long-chain methylene groups.

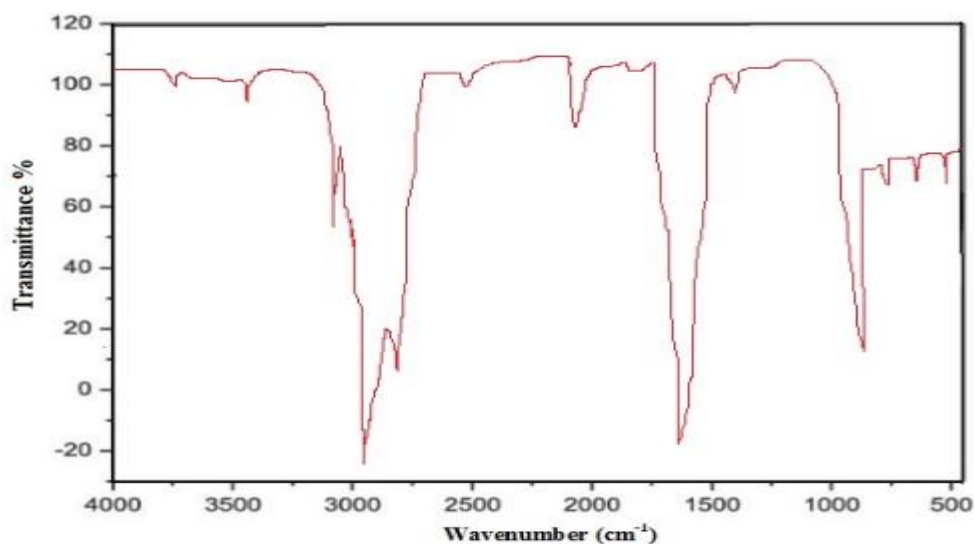
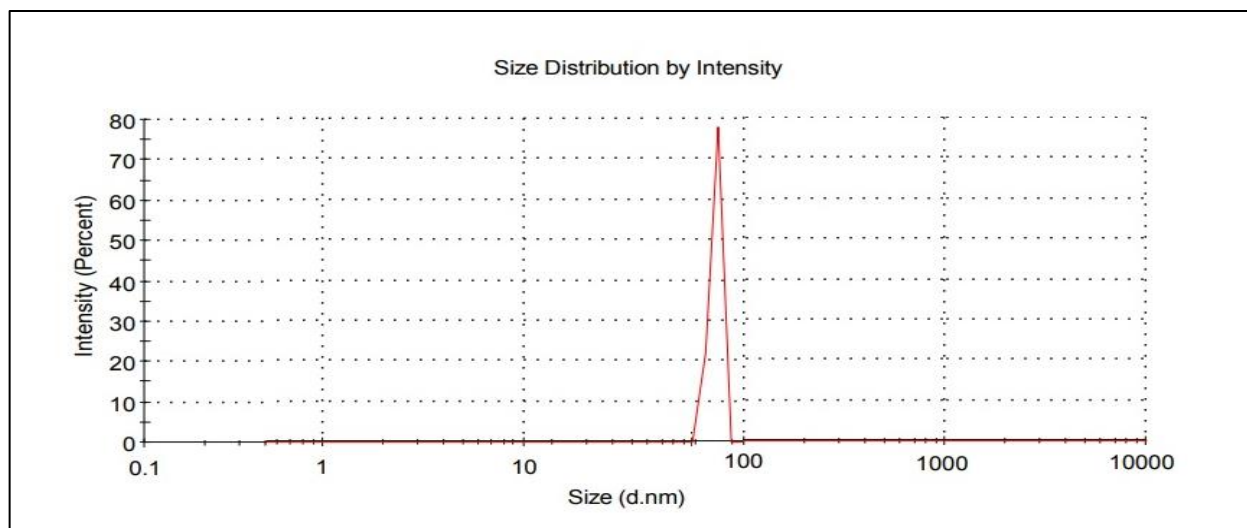


Figure -1: -FT-IR Spectra of MOSO

Characterization of (CaO)_{EN}:**Dynamic light scattering (DLS) analysis:-**

Dynamic light scattering (DLS) is a fast and non-invasive tool used to measure particle size, size

distribution and stability in solutions or suspensions during nanomaterial preparation [26]. The DLS analysis of (CaO)_{EN} is shown in the figure-2

Figure 2: - DLS analysis of (CaO)_{EN}

**Interpretation:**

- The eggshell nanocatalyst particles have a mean hydrodynamic diameter of approximately 90–100 nm.
- The narrow and sharp peak indicates a uniform particle size distribution (monodisperse system), which is ideal for catalytic applications.
- No significant peaks at higher sizes suggest minimal agglomeration and good dispersion in the medium.

XRD Interpretation of (CaO)_{EN}:

X-ray diffraction (XRD) is a nondestructive technique used to evaluate the structures, phases, crystal

orientations, and other structural data of crystalline materials [27]. The X-ray diffraction (XRD) pattern presented corresponds to a nanostructured material derived from eggshell, commonly composed of calcium carbonate (CaCO₃) for the (CaO)_{EN} is shown in the figure-3 After calcination or modification for catalytic purposes, eggshells converted to CaO. The XRD pattern shows sharp and intense diffraction peaks at 2θ values corresponding to the following planes: (111), (200), (220), (311), and (222) w.r.t 38°, 45°, 65°, 78°, and 90° respectively. These peaks are indicated that metallic nanoparticles supported on the eggshell-derived catalyst and it has a highly crystalline CaO or CaCO₃ phases with embedded or surface-deposited metallic nanostructures.

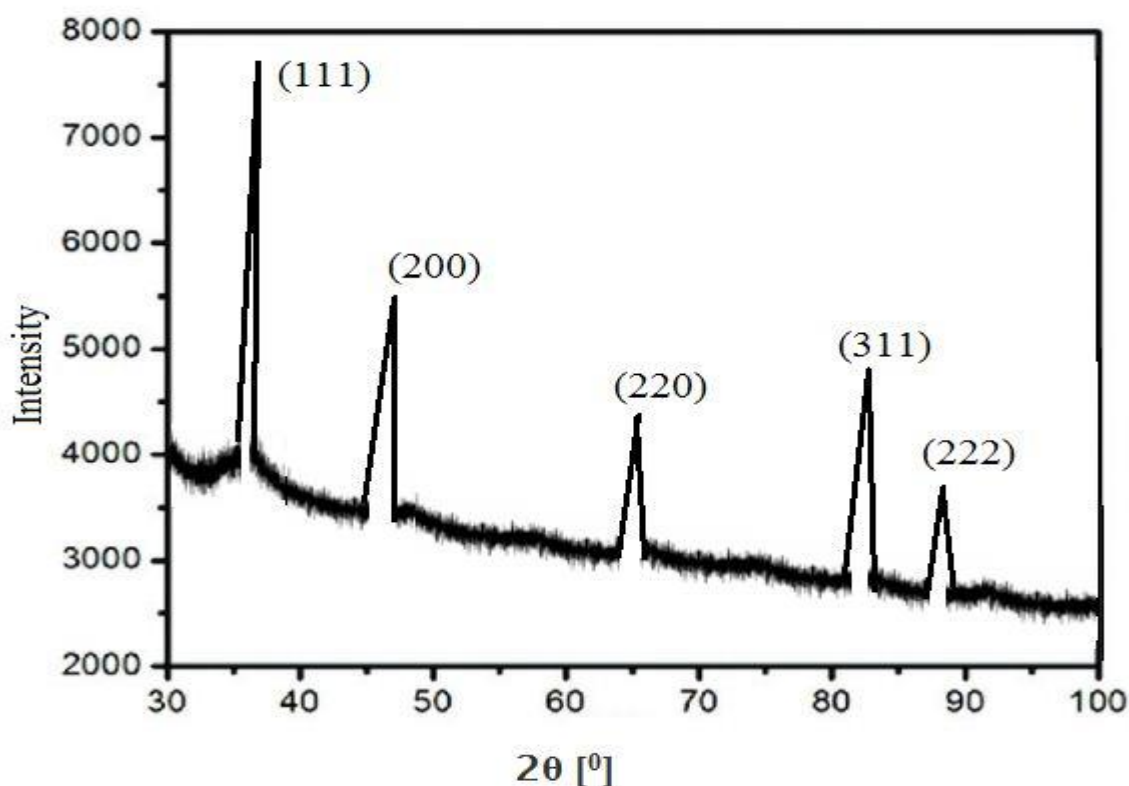


Figure 3:- XRD analysis of (CaO)_{EN}

SEM Analysis

Scanning electron microscopy (SEM) is highly versatile methodologies for 2D and 3D materials characterization [28]. SEM images of (CaO)_{EN} shown in the figure-4 confirm that the (CaO)_{EN} has a highly porous, uneven,

and fine structure suitable for catalytic applications. The nano-scale size and surface irregularities improve the surface-to-volume ratio, making the catalyst effective in reactions such as transesterification and biodiesel synthesis.

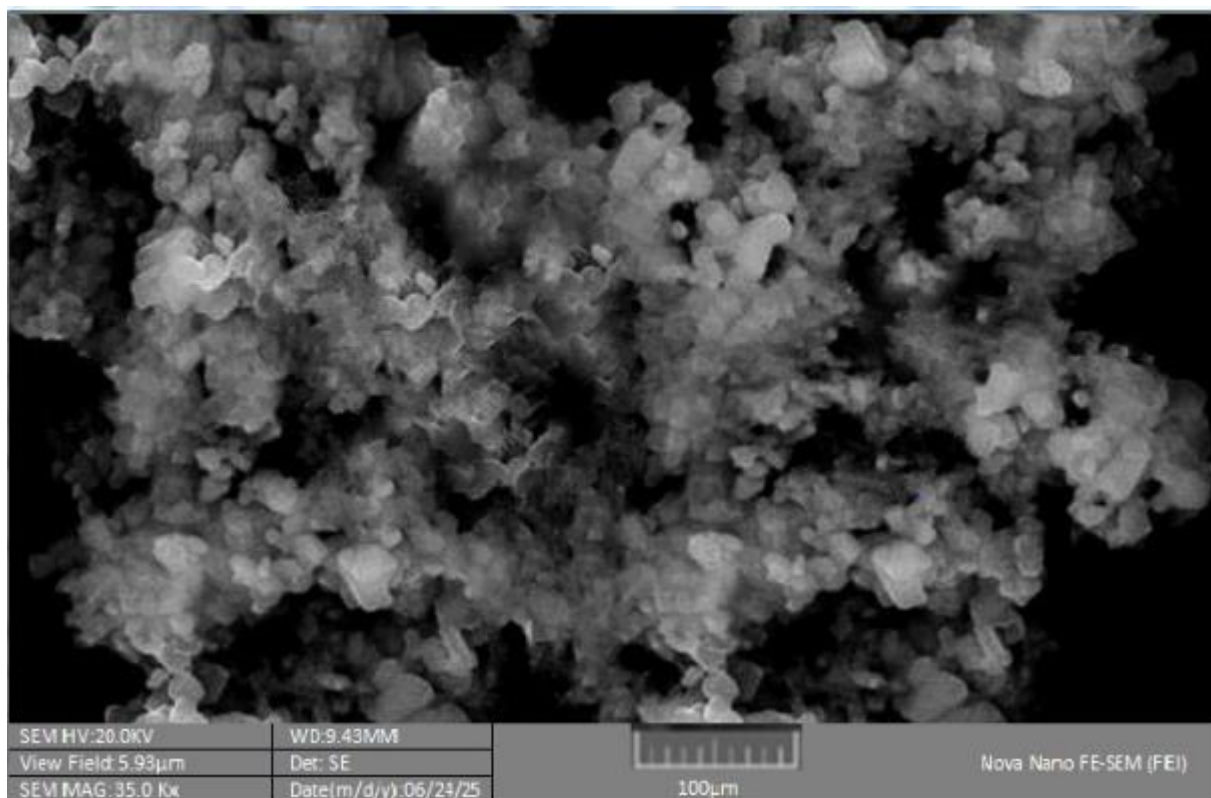


Figure 4: SEM analysis of (CaO)_{EN}

TGA-DTA Interpretation of Eggshell Nanocatalyst:

Thermogravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate) or as a function of time (with constant temperature and/or constant mass loss) [29]. Differential thermal analysis (DTA) is a thermal analysis technique, the term “differential” indicates that

the difference in behavior between the material under study and a supposedly inert reference material is examined [30].

The thermal behavior of the (CaO)_{EN} was analyzed using Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) shown in the figure 5 and table -3. The results are presented showed mass loss (TGA curve, red) and thermal events (DTA curve, blue) as functions of temperature.

Table-3: Thermal Analysis Table of Eggshell Nanocatalyst

Temperature Range (°C)	TGA Observation (Mass Loss %)	DTA Observation (Thermal Event)	Interpretation
30–150°C	Minor weight loss (~1–3%)	Small endothermic peak	Evaporation of physically adsorbed water and volatile moisture.
150–600°C	Gradual mass loss (~3–5%)	Broad thermal fluctuation	Decomposition of organic matter, e.g., proteins or membranes in eggshell.
600–850°C	Continued slight mass decrease	Broad endothermic curve	Thermal rearrangement and phase transitions before major decomposition.
850–950°C	Major mass loss	Sharp endothermic	Decomposition of CaCO ₃ to CaO + CO ₂ . This



	(~40–45%)	peak	is the key transformation.
950–1200°C	Mass stabilizes (~50% remains)	Exothermic peaks observed	Crystallization/sintering of CaO phase, indicating catalyst formation is complete.

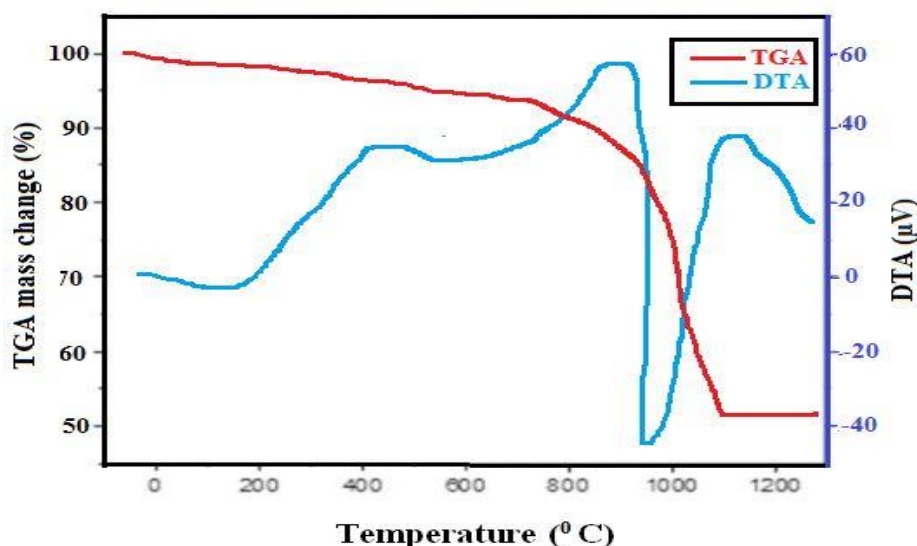


Figure: 5 TGA-DTA analysis of (CaO)_{EN}

Fatty acids composition:

The fatty acids composition of MOSO is shown in the table 4 and GC-MS spectrum for the same is shown in figure-6. The fatty acid profile of MOSO revealed a high concentration of oleic acid (76.7%), indicating its potential a monounsaturated oil with excellent oxidative stability. The similar results for the MOSO with oleic acid fatty acid comprising 70.85% of total oil followed by palmitic acid (8.98%) reported by Fu. X et al. [25]. Similarly 74.41 % oleic acid reported by Mofijur M et al [31]. Saturated fatty acids such as palmitic acid (5.72%) and stearic acid (4.85%) are present in moderate amounts, contributing to the oil's structural and thermal properties. The low levels of

polyunsaturated fatty acids linoleic acid (0.57%) and linolenic acid (0.26%) further supported its stability and extended shelf life. Arachidic acid (3.1%) adds to the unique fatty acid composition, while the remaining 8.8% includes minor components that may influence flavor and functional properties. It is ideal for the oil used for production of biodiesel to have relatively larger percentage of monounsaturated fatty acids than polyunsaturated fatty acids. This is because oil containing relatively high proportion of polyunsaturated fatty acids tends to exhibit a poor oxidation stability and it can compromise the fuel properties such as kinematic viscosity and impair fuel quality [32-34]. Other fuel properties which are negatively affected by high degree of unsaturation are density and cetane number [35].

Table-4: Fatty acids composition of MOSO (uncorrected weight percent) determined by GC-MS

Fatty acids (%)	MOSO
Palmitic acid (C _{16:0})	5.72
Stearic acid (C _{18:0})	4.85

Oleic acid(C _{18:1})	76.7
Linoleic acid (C _{18:2})	0.57
Linolenic acid (C _{18:3})	0.26
Arachidic acid (C _{20:0})	3.1
Others	8.8

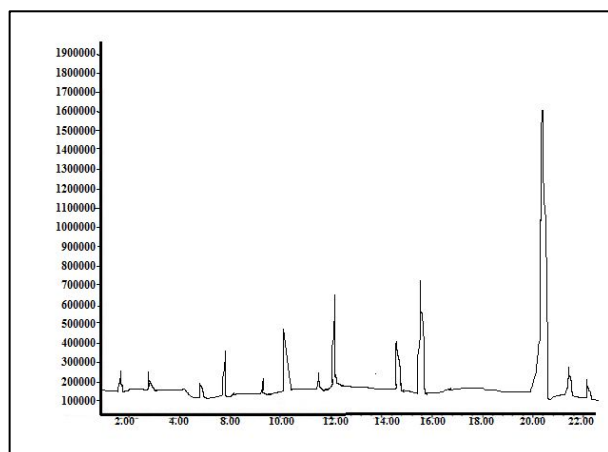


Figure 6- GC-MS spectrum of MOSO

Fuel properties analysis of (MOBD)_{EN}:

Table-5 presented a detailed comparison of the fuel properties of (MOBD)_{EN} with international standards such as ASTM D6751, EN 14214 and earlier research conducted by Azad AK et al (2005) and Rashid U et al (2011) to assess its suitability as a biodiesel [21,22,36,37].

The cloud point and pour point of (MOBD)_{EN} were recorded as -8°C and -12°C respectively, which fall within the EN standard range (-3°C to -12°C and -15°C to -16°C). These values suggest good cold flow behavior, making (MOBD)_{EN} suitable for use in colder environments.

The flash point of (MOBD)_{EN} was observed at 150°C , well above the EN minimum requirement of 101°C , indicating a low risk of fire hazard during storage and handling.

Table-5: Fuel properties of (MOBD)_{EN} compared with ASTM-D6751, EN 14214 standards and earlier research reported.^[21, 22,36,37]

Fuel properties	(MOBD) _{EN}	MOBD ^[36,37]	ASTM-D6751 ^[21]	EN 14214 ^[22]
Cloud point ($^{\circ}\text{C}$)	-8	18	-3 to -12	-----
Pour point ($^{\circ}\text{C}$)	-12	17	-15 to -16	-----
Density (Kg/m ³)	880	---	880	860–900
Flash point ($^{\circ}\text{C}$)	150	---	130	Min. 101
Fire point ($^{\circ}\text{C}$)	120	---	-----	-----

The fire point was recorded at 120°C , further confirming the safe handling characteristics of (MOBD)_{EN}.

The kinematic viscosity of (MOBD)_{EN} was $2.8\text{ mm}^2/\text{s}$, which is well within the ASTM D6751 standard range of $1.9\text{--}6.0\text{ mm}^2/\text{s}$, suggesting proper fuel flow and injector performance. The viscosity at 40°C was found to be $4.5\text{ mm}^2/\text{s}$, aligning well with the EN 14214 acceptable range of $3.5\text{--}5.0\text{ mm}^2/\text{s}$.

The ash content of (MOBD)_{EN} was $0.018\text{ wt}\%$, which is below the EN limit of $0.020\text{ wt}\%$, indicating low levels of inorganic contaminants and reduced risk of engine deposits.

The AV was 0.08 mg KOH/g , slightly higher than the EN maximum of 0.050 mg KOH/g , which may indicate the presence of FFAs and a potential need for purification or antioxidant treatment to prevent degradation during storage.

The CN of (MOBD)_{EN} was 50, which meets the ASTM-D6751 minimum requirement (47) but is slightly below the EN-14214 minimum (51). Nonetheless, it still indicates acceptable ignition quality and performance.

The CV of 52 MJ/kg reflects high energy content as compared to earlier reported results (48 MJ/kg), making (MOBD)_{EN} a strong alternative to conventional diesel in terms of power output.

Lastly, the biodiesel yield of 95.80% slightly higher the previous investigation reported (94.30%) demonstrated an efficient conversion process, indicating the feasibility of large-scale production of (MOBD)_{EN} with high yield.



Kinematic Viscosity (mm ² /s)	2.8	---	1.9–6.0	-----
Viscosity (mm ² /s)	4.5	4.8	-----	3.5-5.0
Specific gravity (g/cm ³)	0.885	0.875	-----	-----
Ash content (wt%)	.018	---	0.050 max	Max. 0.02
Carbon residue (wt%)	0.045	---	0.050 max	-----
Acid value (mgKOH/g)	0.08	0.38	0.050 max	0.050 max
Cetane number	50	67	47min	51min
Calorific value (KJ/Kg)	52	48		
Biodiesel yield	95.80%	94.30%		

3. Conclusion:-

The present study confirms that (MOBD)_{EN} exhibited promising potential as a sustainable and eco-friendly alternative to fossil fuels. The fatty acid composition of MOSO is characterized by a high content of monounsaturated fatty acids especially oleic acid (76.7) contributes significantly to the oxidative stability and desirable fuel characteristics of the resulting biodiesel. The fuel properties of (MOBD)_{EN}, including its density, viscosity, flash point, and cetane number were found to be within or close to the limits set by international standards such as ASTM D6751, EN 14214. The reported fuel properties are found improved as compared to earlier reported results. In addition, the study successfully utilized an eggshell-derived nanocatalyst, rich in calcium oxide (CaO), for the transesterification process. The DLS result confirmed nanoscale size (~100 nm) of the (CaO)_{EN}. The XRD results of (CaO)_{EN} demonstrated good crystallinity and structural features desirable for catalytic applications. This supported the effective transformation of waste eggshell into a value-added heterogeneous nanocatalyst. This heterogeneous catalyst not only proved to be effective in achieving high biodiesel yield 95.80% and calorific value 52 KJ/Kg but also offered the advantages of reusability, low cost, and reduced environmental impact. The use of a waste-derived catalyst supported the green chemistry approach and enhances the sustainability aspect of the process. Overall, the integration of a non-edible feedstock (MOSO), favorable fatty acid profile, and a waste-derived

nanocatalyst in biodiesel production presents a viable pathway for the development of cost-effective, high-quality, and environmentally responsible biofuels. Further research and process optimization can pave the way for large-scale commercial applications of Moringa-based biodiesel.

4. ABBREVIATIONS:

AV-ACID VALUE

(MOBD)_{EN}- MORINGA OLEIFERA BIODIESEL DERIVED FROM EGGHELL NANOCATALYST

AOCS- AMERICAN OIL CHEMIST'S SOCIETY

MOSO- MORINGA OLEIFERA SEED OIL

ASTM- AMERICAN SOCIETY FOR TESTING MATERIAL

(CAO)_{EN}- CALCIUM OXIDE NANOCATALYST DERIVED FROM EGGHELL

XRD- X-RAY DIFFRACTION

TGA-DTA- THERMOGRAVIMETRIC ANALYSIS AND DIFFERENTIAL THERMAL ANALYSIS

SEM- SCANNING ELECTRON MICROSCOPY

DLS- DYNAMIC LIGHT SCATTERING

CV- CALORIFIC VALUE

CN- CETANE NUMBER

EN- EUROPEAN NORMS



FAMEs - FATTY ACIDS METHYL ESTERS

FFAs- FREE FATTY ACIDS

FT- IR - FOURIER TRANSFORMS INFRARED SPECTROSCOPY

GC- MS- GAS CHROMATOGRAPHY MASS SPECTROMETRY

IV- IODINE VALUE

SV- SAPONIFICATION VALUE

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6. **Conflict of Interest:** There is no conflict of interest.

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