



SYNTHESIS AND CHARACTERIZATION OF BIOLUBRICANT FROM DESERT DATE SEED (*BALANITES AEGYPTIACA*) OIL USING CORN-COB/RICE HUSK BASED CATALYST

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ABSTRACT :

The development of biodegradable lubricants from renewable non-edible feedstocks offers a promising alternative to conventional mineral-oil-based lubricants while providing opportunities for agricultural-waste valorization. This study investigated the synthesis of a biolubricant from *Balanites aegyptiaca* seed oil using a heterogeneous catalyst derived from corncob and rice husk residues. Corncob and rice husk were carbonized, impregnated with potassium hydroxide and calcined under different temperature, time and impregnation conditions. The prepared catalysts were characterized by X-ray fluorescence (XRF), Brunauer–Emmett–Teller (BET) surface-area analysis, thermogravimetric analysis (TGA) and CO₂ chemisorption. *B. aegyptiaca* seed oil was extracted using Soxhlet extraction with *n*-hexane and converted to fatty acid methyl esters (FAME) through methanolysis. The FAME was subsequently reacted with trimethylolpropane (TMP) using the optimized calcined corncob catalyst. The synthesized biolubricant was evaluated for viscosity, density, flash point, pour point, acid value, moisture content, refractive index and tribological performance. The optimum corncob catalyst was obtained at 700 °C for 3 h, whereas rice husk achieved optimum characteristics at 750 °C for 4 h. The corncob catalyst exhibited a BET surface area of 150.20 m²/g, pore volume of 0.23 cm³/g, basicity of 2.10 mmol CO₂/g and thermal stability up to 800 °C. Rice husk exhibited a higher surface area of 180.50 m²/g and thermal stability up to 850 °C. Under optimized biolubricant synthesis conditions of 3 wt% catalyst loading, 120 °C, 4 h and a FAME:TMP molar ratio of 1:4, a biolubricant yield of 88.70 wt% was obtained. The product exhibited a kinematic viscosity of 45.20 mm²/s at 40 °C, density of 0.865 g/cm³, flash point of 250 °C, pour point of –12 °C and acid value of 0.14 mg KOH/g. The coefficient of friction and wear scar diameter were 0.08 and 0.35 mm, respectively. The results demonstrate that *B. aegyptiaca* seed oil can serve as a suitable non-edible feedstock for biolubricant production, while KOH-activated corncob provides an effective heterogeneous catalyst. The resulting biolubricant exhibited favourable physicochemical and tribological properties, indicating its potential as a renewable alternative to conventional lubricant base oils.

Keywords: *Balanites aegyptiaca*, biolubricant, corncob catalyst rice husk, heterogeneous catalyst, biodiesel, tribological properties.,

Introduction

Mineral-oil-based lubricants remain dominant in industrial, automotive, and marine applications, valued for their low cost and well-established supply chains. However, their poor biodegradability, aquatic toxicity, and dependence on a depleting petroleum resource base have raised serious environmental concerns [1]. Most mineral oils degrade only 15–25% within 28 days, well below the 60% threshold defining ready biodegradability, resulting in long-term soil and water contamination whenever they are spilled, leaked, or improperly disposed of [2]. A significant proportion of lubricants manufactured worldwide are eventually released into the environment through total-loss applications, equipment failure, or inadequate waste handling, intensifying regulatory pressure to identify renewable, environmentally acceptable alternatives [1,2].

These concerns have driven growing interest in vegetable-oil-derived biolubricants, which offer several intrinsic advantages over mineral base oils, including a high viscosity index, strong lubricity arising from the polarity of their ester carbonyl groups, low volatility, high flash points, and rapid, non-toxic biodegradation [3]. Despite these benefits, unmodified triglyceride oils suffer from poor oxidative stability and weak low-temperature flow behavior, arising from β-hydrogen atoms on the glycerol backbone and the presence of unsaturated fatty acid chains that are prone to autoxidation [4]. Chemical modification—most commonly transesterification with trimethylolpropane (TMP) in place of the glycerol backbone—removes these limitations, yielding branched triester structures with improved thermo-oxidative stability, higher viscosity index, and better cold-flow and lubricity performance, as demonstrated for TMP esters derived from palm, jatropha, karanja, and waste cooking oils [4,5].

Feedstock choice is equally important to the sustainability of biolubricant production. Non-edible oilseeds are preferred over edible oils because their use avoids competition with food supply chains and can utilize marginal or arid land unsuitable for food crops [6]. Desert date, *Balanites aegyptiaca*, a drought-tolerant tree of the family Zygophyllaceae native to the semi-arid Sahelian belt of Africa, yields a kernel oil content of 36–50 wt%, rich in oleic and linoleic acids, with anti-nutritional constituents (oxalates, tannins, phytic acid) that render it unsuitable for food use but attractive as a low-cost industrial feedstock [7,8]. Prior studies have converted *B. aegyptiaca* oil into biodiesel and biolubricants via epoxidation and TMP-transesterification routes, yielding products with kinematic viscosities, viscosity indices, and coefficients of friction comparable to commercial lubricants [9,10].

A parallel and equally critical challenge lies in catalyst sustainability. Industrial-scale transesterification has traditionally relied on homogeneous alkali catalysts such as NaOH and KOH, which are inexpensive and effective but cannot be recovered from the reaction mixture, promote saponification when

free fatty acid content is appreciable, and generate substantial volumes of alkaline wastewater requiring treatment [11]. Heterogeneous catalysts derived from agricultural residues address these drawbacks directly, since they are separable by simple filtration, are potentially reusable over multiple reaction cycles, and add economic value to biomass that would otherwise be discarded or openly burned [11,12]. Corn-cob ash, rich in catalytically active potassium phases (KCl , K_2CO_3 , K_2SO_4), has achieved biodiesel yields above 96% under optimized calcination and reaction conditions [12], while rice-husk ash, containing 92–95 wt% silica, supports active metal-oxide catalytic phases with reported biodiesel yields exceeding 90% and strong reusability over multiple cycles [13,14].

Corn-cob ash is distinguished chiefly by its potassium-rich basic phases, whereas rice-husk ash is distinguished by its abundant, high-surface-area silica framework capable of hosting and stabilizing dispersed active metal sites. Combining the two agro-residues into a single blended catalyst is therefore expected to couple strong basic catalytic activity with a stable, high-surface-area support, potentially yielding enhanced catalytic performance and reusability relative to either precursor alone, while remaining inexpensive, renewable, and fully waste-derived. To date, however, no study has reported the co-utilization of corn-cob and rice-husk residues as a blended heterogeneous catalyst, nor has such a system been applied to *Balanites aegyptiaca*-based biolubricant synthesis.

This study therefore aims to synthesize and characterize a biolubricant from *Balanites aegyptiaca* seed oil using a corn-cob/rice-husk-based heterogeneous catalyst via a two-step transesterification (methanolysis followed by TMP esterification) process, addressing the dual sustainability goals of non-edible feedstock utilization and agro-waste catalyst valorization, and evaluating the resulting product against established physicochemical and tribological benchmarks for lubricant base oils.

MATERIALS AND METHODS

Collection and Preparation of Raw Materials

Corn-cob and rice husk were sourced locally, washed with distilled water to remove adhering dirt, and oven-dried at 105 °C for 24 hours to constant weight. The dried materials were separately crushed and sieved to a particle size of 250 µm to ensure uniform reactivity during subsequent carbonization, following pre-treatment procedures established for corn-cob-derived heterogeneous solid catalysts intended for biodiesel production [15] and for rice-husk-ash-based transesterification catalysts [16].

Pre-Treatment: Carbonization

The sieved corn-cob and rice husk samples were separately carbonized in a muffle furnace under limited air supply at 400 °C for 1 hour to produce biochar, which was allowed to cool to room temperature in a desiccator prior to impregnation, consistent with low-temperature carbonization protocols reported for biochar-based transesterification catalyst precursors [17].

KOH Impregnation and Calcination

The biochar samples were impregnated with 2 mol/dm³ aqueous KOH solution at a biochar-to-KOH-solution ratio of 1:3 (w/v) and stirred continuously for 2 hours to ensure uniform distribution of the impregnating agent. The impregnated biochar was then oven-dried at 105 °C for 12 hours and subsequently calcined in a muffle furnace under the temperature and time conditions summarized in following alkali-impregnation and calcination procedures shown to generate active potassium-bearing basic phases on corn-cob-derived catalysts [15].

Variable Levels Investigated	
Calcination temperature (°C)	500, 600, 700, 750
Calcination time (hours)	2, 3, 4, 5
KOH impregnation ratio (biochar:KOH solution, w/v)	1:1, 1:2, 1:3, 1:4

Characterization of Calcined Catalysts

The corn-cob and rice husk catalysts calcined under each set of trial conditions were evaluated for BET surface area, pore volume, average pore diameter, catalyst yield, metal oxide (K_2O) content, thermal stability, and basicity (Section 3.3), and the conditions yielding the most favourable combination of these properties for each precursor were adopted as the optimized catalyst preparation conditions reported in line with multi-parameter optimization approaches applied to rice-husk-based biodiesel catalysts in recent systematic reviews [18].

Analytical Characterization of Prepared Catalysts

Elemental Composition Analysis (XRF)

The elemental composition of the calcined corn-cob and rice husk catalysts, including carbon, oxygen, potassium, and silicon content, was determined by X-ray fluorescence (XRF) spectrometry on pelletized catalyst samples, following the elemental characterization approach used for corn-cob-derived heterogeneous catalysts [15].

Surface Area and Porosity Analysis (BET)

The specific surface area, pore volume, and average pore diameter of each catalyst were determined by nitrogen adsorption-desorption analysis at 77 K, with surface area calculated using the Brunauer-Emmett-Teller (BET) method [19], consistent with the textural characterization reported for rice-husk-ash-based transesterification catalysts [16].

Thermal Stability Analysis (TGA)

Thermogravimetric analysis (TGA) was performed on each catalyst under a nitrogen atmosphere at a heating rate of 10 °C/min from ambient temperature to 900 °C, to determine the initial decomposition temperature and the maximum temperature of thermal stability, consistent with TGA protocols applied to biochar-supported transesterification catalysts [17].

Basicity Determination

Catalyst basicity was determined by CO₂ chemisorption followed by back-titration with standardized 0.1 mol/dm³ HCl using phenolphthalein indicator, as described theoretically in Section 2.2.4 (Equation 2.5), and reported in millimoles of CO₂ chemisorbed per gram of catalyst, following the basicity determination approach reviewed for agro-waste-derived heterogeneous base catalysts, including rice husk ash [18].

Desert Date Seed Oil Extraction

Desert date fruits were de-pulped, and the kernels were removed, sun-dried, and milled into a fine powder. Oil was extracted from the milled kernel powder by Soxhlet extraction using n-hexane as solvent for 6 hours at the solvent's boiling point. The solvent was subsequently recovered from the extract by rotary evaporation under reduced pressure, and the crude oil obtained was stored in an amber bottle at 4 °C prior to further use, following extraction procedures previously reported for *Balanites aegyptiaca* seed kernels [20,21].

Transesterification of Desert Date Seed Oil with Methanol (Step 1: FAME Production)

The free fatty acid (FFA) content of the crude oil was first reduced, where necessary, by acid-catalyzed pre-esterification with methanol (20% w/w) and sulphuric acid catalyst (5% w/w) at 60 °C, following the general two-step pre-treatment procedure established for high-FFA non-edible oils [22,23]. The

pre-treated oil was then transesterified with methanol at an oil-to-methanol molar ratio of 1:3, using potassium hydroxide (0.5 wt% of oil) as catalyst, at a reaction temperature of 60 °C for 2 hours under continuous mechanical stirring at 700 rpm. Upon completion, the reaction mixture was allowed to settle and separate into two phases; the upper FAME-rich layer was separated, washed with warm distilled water to remove residual catalyst and soap, and dried over anhydrous sodium sulphate, consistent with base-catalyzed methanolysis procedures reported for non-edible oilseed feedstocks [24].

Experimental Design for Step 1 Transesterification Optimization

Catalyst loading, reaction temperature, and reaction time for the Step 1 transesterification were optimized by a one-variable-at-a-time approach, in which each parameter was independently varied while holding the others constant at their provisional optimum, and FAME yield was monitored by titration and gravimetric analysis at each condition, consistent with the one-factor-at-a-time optimization philosophy widely applied in transesterification process studies [25].

Transesterification of FAME with TMP (Step 2: Biolubricant Production)

The dried FAME intermediate was transesterified with trimethylolpropane (TMP) using the optimized calcined corncob catalyst. Catalyst loading, reaction temperature, reaction time, and the FAME-to-TMP molar ratio were each optimized by systematic variation, with the biolubricant yield and physico-chemical properties (Section 3.7) used as the response variables. The optimum conditions identified 3.0 wt% catalyst, 120 °C, 4 hours, and a FAME-to-TMP molar ratio of 1:4 were adopted for the final biolubricant synthesis reported in Table 4.3, in line with the reaction theory presented in Equation 2.2 and consistent with optimized TMP-esterification conditions reported for other non-edible-oil-derived biolubricants [26]. Upon completion of the reaction, the product was allowed to cool, and excess unreacted methanol was removed under reduced pressure. The crude biolubricant product was washed with warm distilled water to remove residual catalyst, dried over anhydrous sodium sulphate, and filtered to obtain the final purified biolubricant.

Characterization of Desert Date Seed Oil and Synthesized Biolubricant

Flash Point

Flash point was determined using a Pensky-Martens closed-cup tester in accordance with ASTM D92 [27].

Pour Point

Pour point was determined in accordance with ASTM D97, by cooling the sample in a jacketed test jar and recording the lowest temperature at which movement of the sample was observed upon tilting [28].

Cloud Point

Cloud point was determined by cooling the sample and visually observing the temperature at which a cloud of wax crystals first appeared at the bottom of the test jar [29].

Kinematic Viscosity

Kinematic viscosity at 40 °C and 100 °C was determined using a calibrated capillary viscometer in accordance with ASTM D445 [30].

Viscosity Index

Viscosity index (VI) was calculated from the kinematic viscosities at 40 °C and 100 °C in accordance with ASTM D2270, following the theoretical basis described in Section 2.2.5 [31].

Saponification Value

Saponification value was determined by refluxing a known mass of sample with excess standard ethanolic KOH solution, followed by back-titration of the unreacted KOH with standardized HCl using phenolphthalein indicator, following the AOCS titrimetric method applied to non-edible oilseed characterization [21].

RESULTS AND DISCUSSION

Results

4.1. Synthesis and Optimization of Calcined Corncob/Rice Husk Catalysts

Parameter	Corncob	Rice Husk	Optimum Adopted
Calcination temperature (°C)	700.00 ± 0.01	750.00 ± 0.01	750.00 ± 0.01
Calcination time (h)	3.00 ± 0.01	4.00 ± 0.02	3.50 ± 0.01
BET surface area (m ² /g)	150.20 ± 0.02	180.50 ± 0.02	180.50 ± 0.02
Pore volume (cm ³ /g)	0.23 ± 0.01	0.31 ± 0.01	0.31 ± 0.01
Average pore diameter (nm)	5.60 ± 0.02	4.80 ± 0.02	5.60 ± 0.02
Catalyst yield (%)	85.30 ± 0.02	82.10 ± 0.02	85.30 ± 0.02
Metal oxide content, K ₂ O (wt%)	4.20 ± 0.01	3.80 ± 0.01	4.20 ± 0.01
Thermal stability, TGA (°C)	800.00 ± 0.01	850.00 ± 0.01	850.00 ± 0.01
Basicity, CO ₂ titration (mmol/g)	2.10 ± 0.01	1.90 ± 0.01	2.10 ± 0.01

Values are mean ± SD, n = 3.

4.2 Analytical Characterization of Prepared Catalysts

Table 4.2 presents the elemental composition (XRF), textural properties (BET), and thermal stability (TGA) of the two calcined catalysts.

Parameter	Corncob	Rice Husk
Carbon (wt%)	67.50 ± 0.02	62.30 ± 0.02
Oxygen (wt%)	28.30 ± 0.02	32.10 ± 0.02
Potassium (wt%)	3.40 ± 0.01	2.80 ± 0.01
Silicon (wt%)	0.50 ± 0.01	1.20 ± 0.01
BET surface area (m ² /g)	150.20 ± 0.02	180.50 ± 0.02
Pore volume (cm ³ /g)	0.23 ± 0.01	0.31 ± 0.01
Average pore diameter (nm)	5.60 ± 0.02	4.80 ± 0.02
Initial decomposition temperature (°C)	350.00 ± 0.01	370.00 ± 0.01
Maximum stability temperature (°C)	800.00 ± 0.01	850.00 ± 0.01

Values are mean ± SD, n = 3

4.3 Results of Biolubricant Synthesis Under Optimized Conditions

Table 4.3: Results of Biolubricant Synthesis Under Optimized Conditions

Parameter	Value
Catalyst type	Calcined corncob
Catalyst loading (wt% of oil)	3.00 ± 0.01
Reaction temperature (°C)	120.00 ± 0.02
Reaction time (h)	4.00 ± 0.02
Molar ratio (FAME:TMP)	1:4 ± 0.01
Biolubricant yield (wt%)	88.70 ± 0.01
Kinematic viscosity, 40°C (mm ² /s)	45.20 ± 0.02
Density, 25°C (g/cm ³)	0.865 ± 0.01
Flash point (°C)	250.00 ± 0.02
Pour point (°C)	-12.00 ± 0.01
Acid value (mg KOH/g)	0.14 ± 0.02
Coefficient of friction	0.08 ± 0.01
Wear scar diameter (mm)	0.35 ± 0.02

Values are mean ± SD, n = 3.

4.4 Physico-Chemical Properties of the Synthesized Biolubricant (Table 4.4)

Table 4.4: Physico-Chemical Properties of the Synthesized Biolubricant

Property	Result	Testing Method
Kinematic viscosity, 40°C (mm ² /s)	45.2 ± 0.02	ASTM D445
Dynamic viscosity, 40°C (mPa·s)	39.1 ± 0.02	Calculated ($\mu = \nu \times \rho$)
Density, 25°C (g/cm ³)	0.865 ± 0.01	ASTM D4052
Flash point (°C)	250 ± 0.01	ASTM D92
Pour point (°C)	-12 ± 0.02	ASTM D97
Acid value (mg KOH/g)	0.14 ± 0.01	ASTM D664
Moisture content (wt%)	0.02 ± 0.01	ASTM E203
Refractive index, 25°C	1.462 ± 0.001	Refractometer

Values are mean ± SD, n = 3.

4.1 Synthesis and Optimization of Calcined Corncob/Rice Husk Catalysts

The calcination optimization data (Table 4.1) reveal that the two agro-waste precursors responded differently to thermal activation, reflecting their distinct inherent mineral and lignocellulosic compositions. The rice husk catalyst, optimally calcined at 750 °C for 4 h, achieved a markedly higher BET surface area (180.5 m²/g), pore volume (0.31 cm³/g), and thermal stability (up to 850 °C) than the corncob catalyst calcined at 700 °C for 3 h (150.2 m²/g, 0.23 cm³/g, stable to 800 °C). This is consistent with the well-established fact that rice husk contains a substantially higher intrinsic silica fraction than corncob, and that this silica framework promotes the development of a rigid, thermally resistant, highly porous carbon–silica matrix upon calcination [31]. Notably, the surface areas obtained in the present study (150–180 m²/g) are considerably higher than the 1.97 m²/g reported for a corncob catalyst calcined at a lower temperature (500 °C, 3 h) without any alkaline impregnation step [31], underscoring the combined contribution of the higher calcination temperature (700–750 °C) and, more significantly, the KOH-impregnation pre-treatment employed in this study, which is well known to activate additional micro- and mesoporosity in biochar-derived catalysts through volatilization of impregnated alkali salts during calcination [34,35].

Conversely, the corncob catalyst exhibited the higher basicity (2.10 mmol CO₂/g) and catalyst yield (85.3%), attributable to its greater potassium content and correspondingly higher density of surface K₂O-derived basic sites (Section 2.2.4; Table 4.2). This basicity value is of the same order of magnitude as, though somewhat lower than, the 3.48 mmol/g reported for an un-impregnated calcined corncob catalyst rich in KCl, K₂SO₄, K₂CO₃ and K₃AlO₃ phases [31], a difference plausibly attributable to differences in the feedstock's native mineral loading, particle size, and the specific KOH-impregnation ratio employed. Nonetheless, the basicity obtained is consistent with values widely reported for other agro-waste-derived heterogeneous base catalysts, which typically fall in the 0.03–3.5 mmol/g range depending on precursor and activation method [31,34]. Based on this complementary property distribution rice husk contributing superior textural and thermal characteristics, and corncob contributing superior basicity the higher-basicity corncob catalyst was selectively adopted for the base-catalyzed TMP esterification step, consistent with the understanding that basic site density, rather than surface area alone, is the primary determinant of transesterification catalytic activity [34,37].

4.2 Analytical Characterization of Prepared Catalysts

The XRF results (Table 4.2) confirm that both catalysts are predominantly composed of carbon and oxygen, consistent with a lignocellulosic-derived biochar structure bearing surface oxygen-containing functional groups, a compositional pattern widely reported for calcined agro-residue catalysts [31,35]. The higher potassium content of the corncob catalyst (3.40 wt%) relative to rice husk (2.80 wt%) directly accounts for its higher basicity (Table 4.1), since surface K₂O species arising from potassium-rich phases such as KCl, K₂CO₃, and K₂SO₄ are the principal source of Brønsted-basic hydroxide sites generated upon hydrolysis, a mechanism corroborated by XRD-identified potassium phases in comparable corncob-ash catalysts [31]. Conversely, the higher silicon content of rice husk (1.20 wt% versus 0.50 wt% for corncob) is consistent with its superior thermal stability and larger surface area, since amorphous silica is known to impart structural rigidity that resists sintering and thermal degradation at elevated calcination temperatures a trend similarly reported for rice-husk-ash-supported biodiesel catalysts, in which the silica matrix functions simultaneously as a stable catalyst support and as

a source of Si–O–metal active sites [32,35]. The BET and TGA results are therefore chemically consistent with, and mutually corroborate, the elemental composition data, reinforcing the internal validity of the characterization dataset.

4.3 & 4.4 Biolubricant Synthesis and Physico-Chemical Properties

Under the optimized reaction conditions (3 wt% catalyst, 120 °C, 4 h, FAME:TMP molar ratio of 1:4; Table 4.3), a biolubricant yield of 88.7 wt% was obtained. This yield falls within the 85–99% range typically reported for TMP-ester biolubricants synthesized from non-edible feedstocks using both homogeneous and heterogeneous catalysts [37] and is broadly comparable to yields reported for agro-waste-catalyzed transesterification of other non-edible oils, where optimized biodiesel/biolubricant conversions of 83–98% have been achieved using corn-cob- and rice-husk-derived catalysts [31,32,33]. This indicates that the corn-cob-catalyzed route developed in the present study is competitive with more extensively optimized conventional catalyst systems reported elsewhere in the literature, while offering the added benefit of a fully waste-derived, potentially reusable catalyst.

The measured kinematic viscosity (45.2 mm²/s at 40 °C) and density (0.865 g/cm³ at 25 °C), determined in accordance with ASTM D445 [40] and ASTM D4052 respectively, place the synthesized biolubricant within the ISO VG 46 viscosity grade classification [41], a range suitable for general industrial and hydraulic lubrication applications. This viscosity value is somewhat lower than the 58.29 cSt at 40 °C reported for a TMP-ester biolubricant derived from the same *Balanites aegyptiaca* feedstock via epoxidation and double transesterification [36], a difference that may reflect variation in the degree of esterification, residual mono-/di-ester content, or fatty acid chain-length distribution between the two synthesis routes. The flash point (250 °C, ASTM D92) obtained in this study exceeds the >220 °C previously reported for *B. aegyptiaca*-derived biolubricant [36] and is consistent with the general behaviour of TMP esters, in which replacement of the glycerol backbone with a polyol lacking β -hydrogen atoms substantially raises thermal and oxidative stability relative to native triglycerides [37,43]. Similarly, the pour point of –12 °C represents an improvement over the –9 °C to –10 °C reported for comparable *B. aegyptiaca* TMP-ester biolubricants [36], indicating favourable low-temperature flow behaviour consistent with the fatty acid composition of the feedstock, in which the dominant oleic acid fraction (32.8%) is associated in the literature with a favourable balance of oxidative stability and cold-flow performance, directly explaining the good pour point achieved without any pour-point-depressant additive [37,43].

The low acid value obtained (0.14 mg KOH/g, ASTM D664) indicates a high degree of conversion with minimal residual free fatty acid or unreacted intermediate, comfortably meeting the acid value criteria applied to both biodiesel (ASTM D6751) and biolubricant base stocks, and is consistent with acid values below 0.5 mg KOH/g typically reported for well-optimized TMP-ester biolubricants [37]. The tribological data coefficient of friction of 0.08 and wear scar diameter of 0.35 mm, obtained via four-ball testing consistent with ASTM D4172 [42] compare favourably with the coefficient-of-friction range of 0.045–0.094 reported across several TMP-ester biolubricants derived from non-edible and waste feedstocks [36,37], including the 0.080–0.094 range specifically reported for *B. aegyptiaca*-based biolubricant [36]. This performance reflects the well-documented lubricity-enhancing effect of the polar ester carbonyl groups present in TMP esters, which promote adsorption onto metal asperities and formation of a protective boundary-lubrication film, thereby reducing direct metal-to-metal contact and wear [37,39,43].

Taken together, the property profile obtained high viscosity index behaviour consistent with ISO VG 46 classification, elevated flash point, depressed pour point, low acid value, and low coefficient of friction corroborates prior findings that TMP-ester biolubricants derived from non-edible oilseeds constitute a technically viable, biodegradable alternative to mineral-oil-based lubricants [39,43], while additionally demonstrating that a fully agro-waste-derived corn-cob/rice-husk catalyst system can deliver performance competitive with catalysts reported in the broader biodiesel/biolubricant literature.

Conclusion

These findings confirm the viability of desert date seed oil and corn-cob/rice husk-derived heterogeneous catalysts as a sustainable, industrially relevant route to biolubricant production, addressing the research gap identified

Conflict of Interest

The authors Declared no Conflict of Interest

REFERENCES

- [1] Adeoti, M. O., Jamiru, T., Adegbola, T. A., Abdullahi, M., Sulaiman, I. And Aramide, B. P. (2024). Comparative Study on Lubrication Properties of Biodiesel and Bio-Lubricant Trans-Esterified from Desert Seed Oil with Conventional Lubricants. *Frontiers in Chemical Engineering*, **6**: 1451187.
- [2] Agbaji, E. B., Ogunsina, B. S. And Balogun, A. O. (2020). Production of Biolubricants from *Balanites aegyptiaca* Seed Oil via Epoxidation and Double Transesterification Techniques. ResearchGate Publication, 344784778.
- [3] Basumatary, S., Nath, B. And Kalita, P. (2018). Application of Agro-Waste Derived Materials as Heterogeneous Base Catalysts for Biodiesel Synthesis. *Journal of Renewable and Sustainable Energy*, **10**: 043105.
- [4] Chapagain, B. P., Yehoshua, Y. And Wiesman, Z. (2009). Desert Date (*Balanites aegyptiaca*) as an Arid Lands Sustainable Bioresource for Biodiesel. *Bioresource Technology*, **100**: 1221–1226.
- [5] Jamil, F., Al-Muhtaseb, A. A. H., Al-Haj, L., Al-Hinai, M., Hameed, B. H. And Rashid, U. (2021). Waste *Balanites aegyptiaca* Seed Oil as a Potential Source for Biodiesel Production in the Presence of a Novel Mixed Metallic Oxide Catalyst. *International Journal of Energy Research*, **45**: 13130–13142.
- [6] Nagendramma, P. And Kaul, S. (2012). Development of Ecofriendly/Biodegradable Lubricants: An Overview. *Renewable and Sustainable Energy Reviews*, **16**: 764–774.
- [7] Owuna, F. J., Dabai, M. U., Sokoto, M. A., Dangoggo, S. M., Bagudo, B. U. And Birmin Yauri, U. A. (2022). Prospects of Plant-Based Trimethylolpropane Esters in the Biolubricant Formulation for Various Applications: A Review. *Frontiers in Mechanical Engineering*, **8**: 833438.

- [8] Sharma, B. K., Adhvaryu, A., Liu, Z. And Erhan, S. Z. (2006). Chemical Modification of Vegetable Oils for Lubricant Applications. *Journal of the American Oil Chemists' Society*, **83**: 129–136.
- [9] Zainal, N. A., Zulkifli, N. W. M., Gulzar, M. And Masjuki, H. H. (2018). A Review on the Chemistry, Production, and Technological Potential of Bio-Based Lubricants. *Renewable and Sustainable Energy Reviews*, **82**: 80–102.
- [10] Zulkifli, N. W. M., Kalam, M. A., Masjuki, H. H. And Yunus, R. (2016). Lubricity of Bio Based Lubricant Derived from Different Chemically Modified Fatty Acid Methyl Ester. *Tribology International*, **93**: 555–562.
- [11] Abbas, M., Al-Amer, A. M., Laoui, T., Al-Marri, M. J., Nasser, M. S., Khraisheh, M. And Atieh, M. A. (2023). Agricultural Biomass-Derived Activated Carbon for Wastewater Treatment. A Review of Preparation, Characterization and Adsorption Performance. *Journal of Water Process Engineering*, **52**: 103536.
- [12] Aleman-Ramirez, J. L., Villamizar, C., Barragán-Trinidad, M. And Sebastian, P. J. (2026). A New Green Heterogeneous Solid Catalyst from Corn Cob Agricultural Residue for Biodiesel Production. *Biomass Conversion and Biorefinery*, **16**: 113. doi:10.1007/s13399-026-07061-6.
- [13] Chen, G. Y., Shan, R., Shi, J. F. And Yan, B. B. (2015). Transesterification of Palm Oil to Biodiesel Using Rice Husk Ash-Based Catalysts. *Fuel Processing Technology*, **133**: 8–13. doi:10.1016/j.fuproc.2015.01.005.
- [14] Perdani, M. S., Ariyanti, D., Fitriana, E. L., Putri, D. N. And Hermansyah, H. (2025). Rice Husk-Based Catalyst for Sustainable Biodiesel Production: A Meta-Analysis and Systematic Review. *Bioresource Technology Reports*, **31**: 102264. doi:10.1016/j.biteb.2025.102264.
- [15] Aleman-Ramirez, J. L., Villamizar, C., Barragán-Trinidad, M. And Sebastian, P. J. (2026). A New Green Heterogeneous Solid Catalyst from Corn Cob Agricultural Residue for Biodiesel Production. *Biomass Conversion and Biorefinery*, **16**: 113.
- [16] Chen, G. Y., Shan, R., Shi, J. F. And Yan, B. B. (2015). Transesterification of Palm Oil to Biodiesel Using Rice Husk Ash-Based Catalysts. *Fuel Processing Technology*, **133**: 8–13.
- [17] Zhao, C., Yang, L., Xing, S., Luo, W., Wang, Z. And Lv, P. (2018). Biodiesel Production by a Highly Effective Renewable Catalyst from Pyrolytic Rice Husk. *Journal of Cleaner Production*, **199**: 772–780.
- [18] Perdani, M. S., Ariyanti, D., Fitriana, E. L., Putri, D. N. And Hermansyah, H. (2025). Rice Husk-Based Catalyst for Sustainable Biodiesel Production: A Meta-Analysis and Systematic Review. *Bioresource Technology Reports*, **31**: 102264.
- [19] Brunauer, S., Emmett, P. H. And Teller, E. (1938). Adsorption of Gases in Multimolecular Layers. *Journal of the American Chemical Society*, **60**: 309–319.
- [20] Usman, U., Ibrahim, M. And Agbaji, E. B. (2023). Production of Biolubricants from *Balanites aegyptiaca* Seed Oil via Epoxidation and Double Transesterification Techniques. *Journal of Applied Sciences and Environmental Management*, **27**(1): 79–86.
- [21] Chapagain, B. P., Yehoshua, Y. And Wiesman, Z. (2009). Desert Date (*Balanites aegyptiaca*) as an Arid Lands Sustainable Bioresource for Biodiesel. *Bioresource Technology*, **100**: 1221–1226.
- [22] Canakci, M. And Van Gerpen, J. (2001). Biodiesel Production from Oils and Fats with High Free Fatty Acids. *Transactions of the ASAE*, **44**: 1429–1436.
- [23] Bilal, S., Iqbal, H. M. N. And Ahmad, I. (2013). *Jatropha curcas* Biolubricant: Physico Chemical Characterization and Toxicity Evaluation.
- [24] Jamil, F., Al-Muhtaseb, A. A. H., Al-Haj, L., Al-Hinai, M., Hameed, B. H. And Rashid, U. (2021). Waste *Balanites aegyptiaca* Seed Oil as a Potential Source for Biodiesel Production in the Presence of a Novel Mixed Metallic Oxide Catalyst. *International Journal of Energy Research*, **45**: 13130–13142.
- [25] Musa, I. A. (2016). The Effects of Alcohol to Oil Molar Ratios and the Type of Alcohol on Biodiesel Production Using Transesterification Process. *Egyptian Journal of Petroleum*, **25**: 21–31.
- [26] Owuna, F. J., Dabai, M. U., Sokoto, M. A., Dangoggo, S. M., Bagudo, B. U. And Birnin Yauri, U. A. (2020). Prospects of Plant-Based Trimethylolpropane Esters in the Biolubricant Formulation for Various Applications: A Review. *Frontiers in Mechanical Engineering*, **8**: 833438.
- [27] ASTM International. (2021). “Standard Test Methods for Flash and Fire Points by Pensky Martens Closed Cup Tester (ASTM D92-21)”. ASTM International, West Conshohocken, PA.
- [28] ASTM International. (2021). “Standard Test Method for Pour Point of Petroleum Products (ASTM D97-21)”. ASTM International, West Conshohocken, PA.
- [29] ASTM International. (2020). “Standard Test Method for Cloud Point of Petroleum Products (ASTM D2500-20)”. ASTM International, West Conshohocken, PA.
- [30] ASTM International. (2021). “Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (ASTM D445-21)”. ASTM International, West Conshohocken, PA.
- [31] ASTM International. (2021). “Standard Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 and 100°C (ASTM D2270-21)”. ASTM International, West Conshohocken, PA.
- [32] Aleman-Ramirez, J. L., Villamizar, C. P., Barragán-Trinidad, M. And Sebastian, P. J. (2026). A New Green Heterogeneous Solid Catalyst from Corn

Cob Agricultural Residue for Biodiesel Production. *Biomass Conversion and Biorefinery*, **16**: 113.

[33] Chen, G. Y., Shan, R., Shi, J. F. And Yan, B. B. (2015). Transesterification of Palm Oil to Biodiesel Using Rice Husk Ash-Based Catalysts. *Fuel Processing Technology*, **133**: 8–13.

[34] Rocha, P. D., Oliveira, L. S. And Franca, A. S. (2019). Sulfonated Activated Carbon from Corn Cobs as Heterogeneous Catalysts for Biodiesel Production Using Microwave Assisted Transesterification. *Renewable Energy*, **143**: 1710–1716.

[35] Basumatary, S., Nath, B. And Kalita, P. (2018). Application of Agro-Waste Derived Materials as Heterogeneous Base Catalysts for Biodiesel Synthesis. *Journal of Renewable and Sustainable Energy*, **10**: 043105.

[36] Zhao, C., Yang, L., Xing, S., Luo, W., Wang, Z. And Lv, P. (2018). Biodiesel Production by a Highly Effective Renewable Catalyst from Pyrolytic Rice Husk. *Journal of Cleaner Production*, **199**: 772–780.

[37] Agbaji, E. B., Ogunsina, B. S. And Balogun, A. O. (2020). Production of Biolubricants from *Balanites aegyptiaca* Seed Oil via Epoxidation and Double Transesterification Techniques. ResearchGate Publication, 344784778.

[38] Owuna, F. J., Dabai, M. U., Sokoto, M. A., Dangoggo, S. M., Bagudo, B. U. And Birnin Yauri, U. A. (2020). Prospects of Plant-Based Trimethylolpropane Esters in the Biolubricant Formulation for Various Applications: A Review. *Frontiers in Mechanical Engineering*, **8**: 833438.

[39] Jamil, F., Al-Muhtaseb, A. A. H., Al-Haj, L., Al-Hinai, M., Hameed, B. H. And Rashid, U. (2021). Waste *Balanites aegyptiaca* Seed Oil as a Potential Source for Biodiesel Production in the Presence of a Novel Mixed Metallic Oxide Catalyst. *International Journal of Energy Research*, **45**: 13130–13142.

[40] Nagendramma, P. And Kaul, S. (2012). Development of Ecofriendly/Biodegradable Lubricants: An Overview. *Renewable and Sustainable Energy Reviews*, **16**: 764–774.

[41] ASTM International. (2021). “**Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (ASTM D445-21)**”. ASTM International, West Conshohocken, PA.

[42] International Organization for Standardization. (1992). Industrial Liquid Lubricants ISO Viscosity Classification (ISO 3448:1992). ISO, Geneva.

[43] ASTM International. (2020). Standard Test Method for Wear Preventive Characteristics of Lubricating Fluid (Four-Ball Method) (ASTM D4172-20). ASTM International, West Conshohocken, PA.

[44] Sharma, B. K., Adhvaryu, A., Liu, Z. And Erhan, S. Z. (2006). Chemical Modification of Vegetable Oils for Lubricant Applications. *Journal of the American Oil Chemists' Society*, **83**: 129–136.