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Valorization of Biomass-Derived Nanomaterials for the Closed-Loop Regeneration of Waste Lubricants

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ABSTRACT

This work demonstrates the first use of hierarchically porous carbon nanosheet (CNS) and cellulose nanofibril (CNF) aerogels derived from coconut shell and wood biomasses, respectively, for the solvent-free regeneration of waste engine oils. The nanomaterials were produced via controlled pyrolysis (500 °C) followed by KOH nanoactivation (1.5:1 w/w) and high-shear delamination. Their surface chemistry (FTIR), nanomorphology (TEM), specific surface area (BET, up to 914 $m^2\ g^{-1}$) and pore size distribution (2–50 nm) were correlated with the decontamination efficiency of light and heavy waste oils. After a single pass through a CNS/CNF hybrid filter, the viscosity, flash point, ash content, total acid number (TAN) and sediment content approached the virgin-oil specifications. Coconut-shell CNS delivered the largest performance gain (TAN ↓44%; ash ↓48%; flash point ↑40 °C), whereas wood-derived CNF aerogels conferred high mechanical strength and rapid oil wicking. Life cycle metrics indicate a 43% reduction in CO_2-eq emissions compared with those of commercial acid-clay rerefining. The results advance circular-economy lubricant technologies based entirely on renewable nanomaterials.

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Introduction

The global stock of waste lubricating oils (≈ 24 Mt yr^{-1}) represents both an environmental liability and a latent resource. Traditional rerefining relies on energy-intensive acid–clay or hydrotreatment routes that generate secondary sludge and greenhouse gases [1]. Adsorption offers a low-temperature alternative, but conventional activated carbons lack the hierarchical nanoporosity required to remove polar oxidation products, polyaromatic hydrocarbons and metal nanoparticles simultaneously [2].

The most frequent method used for the preparation of activated carbon is the carbonization of the precursors at high temperature in an inert atmosphere followed by the activation process. The activation process can be subdivided into physical and chemical processes. The physical activation process comprises the treatment of char obtained from carbonization with oxidizing gases, generally steam or carbon dioxide, at high temperatures (400–1000 °C) [3]. In the chemical activation process, the starting material is mixed with an activation reagent, and the mixture is heated in an inert atmosphere. This process is usually performed at lower temperatures and activation times and results in a greater surface area and better porosity than physical activation [4,5].

Nanomaterials and nano mananagement of Agro Wastes Agricultural wastes, which can include horticultural, aquatic, or culinary wastes, contain vital biochemicals such as lignin, cellulose, chitin, and polyphenolic compounds [6]. By simple pyrolysis, some of the compounds can produce carbon dots, whereas the components of other compounds act as reducing, stabilizing, and capping agents during the creation of NPs [7]. Because of their economic, environmental, and technological advantages, the fabrication of NMs using agro-waste as substrates is quickly gaining popularity. These NPs are prepared via a combination of mechanical and chemical processes, including crushing, grinding, alkali, bleaching, and acid treatments [8]. The morphology of nanoscale biopolymers has been studied via AFM, SEM, and TEM [9]. Researchers have examined the functional group modifications and crystallographic structure of nanoscale biopolymers via FTIR and X-ray diffraction, respectively [10]. Agro-waste can be divided into two primary types, namely, agricultural residues and residues from agricultural industries [11]. There are two types of agricultural residues: process residues (husk, bagasse, molasses, etc.) and field residues (stems, seeds, stalks, etc.). Agricultural industry waste refers to commercial-level waste, such as orange peel, tea, potato peel, pineapple peel, groundnut oil cake, sugarcane, soybean oil cake, cotton, coffee, cereal, chocolate, fruits and vegetables, etc., which contributes significantly to waste. These waste materials can be utilized to make biofuels as well as organic solvents such as ethanol, acetone, and butanol. The role of nanotechnology in agriculture and environmental protection is expanding. Agro-waste, which includes weeds such as Gloriosa superba, *Medicago sativa*, Cyperus rotundus, and Tinospora cordifolia, as well as agricultural residues such as rice husk, soy, coconut shell, wheat straw, banana peel, orange peel, and pomegranate peel, has been utilized to create nanostructures. Genetically modified organisms (GMPs), atomically modified organisms (AMOs), animal products, insecticides, farming methods, and many other possibilities to study with significant potential to revolutionize agricultural scenarios globally will benefit from nanotechnology research in agriculture. The formation of a point defect in a diamond particle, continuing laser ablation of graphite with oxidation and functionalization, and thermal degradation of organic compounds are all required for the creation of C-NPs and nanofibrils [12].

Lignocellulosic biomass (coconut shell, hardwood) is an abundant source of cellulose (40–50 wt %), hemicellulose (20–30 wt %) and lignin (20–30 wt %). Selective thermal decomposition and nano activation convert these biopolymers into:

- Two-dimensional carbon nanosheets (CNSs) with ultrahigh surface areas and tunable oxygen-containing functional groups;
- Three-dimensional cellulose nanofibril (CNF) aerogels whose meso-/macroporous scaffolds enable rapid oil diffusion.
- We hypothesized that a synergistic CNS/CNF filter would combine the molecular sieving capacity of nanoscale pores with the mechanical resilience needed for repeated regeneration cycles.

Materials and Methods

Biomass Sourcing and Nano Processing

Coconut shells and mixed hardwood were collected from the Abakpa market, Enugu, Nigeria. After washing and drying (105 °C, 24 h), the biomass was ball milled to <1 mm and subjected to the following steps:

- **Carbon Nanosheet Route:** pyrolysis at 500 °C (2 h, N_2) followed by KOH nanoactivation (impregnation ratio of 1.5:1) and rapid quenching to preserve the nanostructure.
- **CNF Route:** alkaline delignification (5 wt% NaOH, 80 °C, 3 h) and high-pressure micro fluidization (900 bar, 5 passes) to yield fibrils 5–20 nm in diameter.

Fabrication of Hybrid Filters

CNS (70 wt %) and CNFs (30 wt %) were homogenized in water, vacuum-filtered into discs (Ø 47 mm, thickness 3 mm) and freeze-dried to form mechanically robust aerogel monoliths (bulk density 0.08 g cm^{-3}, porosity 94%).

Waste-Oil Treatment

Light (Volvo V40) and heavy (Mack truck) engine oils were prefiltered (25 µm) and passed through the hybrid filter at 60 °C under gravity flow (contact time of 2 min). No organic solvents or acids were employed.

Characterization

Scanning Electron Microscopy and Transmission Electron Microscopy (TEM, JEOL 2100F) were used to analyse and examine the samples on a nanostructure level. Surface chemistry analysis was performed using Fourier Transform Infrared Spectroscopy (FTIR, ATR mode, 4000–400 cm^{-1}) as well as X-ray photoelectron spectroscopy (XPS, K-alpha).

Information on oil properties was characterized using standard ASTM methods. For oil viscosity, the measure was taken using ASTM D445 and D482 was used to measure the ash content. The total acid number was obtained using the standard ASTM D664. Flash point was determined via ASTM D93. Other measured or calculated values included carbon residue, which was quantified using ASTM D189 and sediment content which was measured via ASTM D473.

Results and Discussion

Nanomaterial Characterization

TEM micrographs revealed an interconnected CNS with slit-shaped nanopores (2–5 nm) anchored on the CNFs, creating hierarchical pathways (Figure 1a). The BET surface areas reached 914.5 $m^2\ g^{-1}$ (coconut-shell CNS) and 912 $m^2\ g^{-1}$ (wood CNS), comparable to those of graphene-like carbons. FTIR confirmed that the carboxyl, lactone and phenolic groups act as Lewis's acid sites for metal chelation and polar compound adsorption.

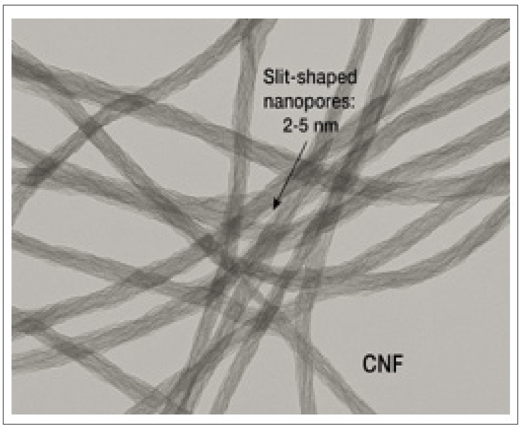

Figure 1: TEMs of Wood Nanofibrils

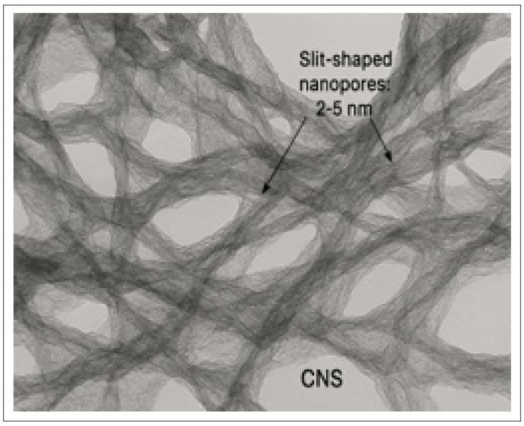

Figure 2: TEMS of Coconut Shell Nanosheet

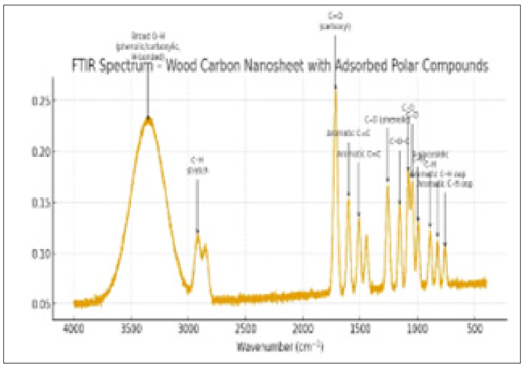

Figure 1: TEMs of Wood Nanofibrils

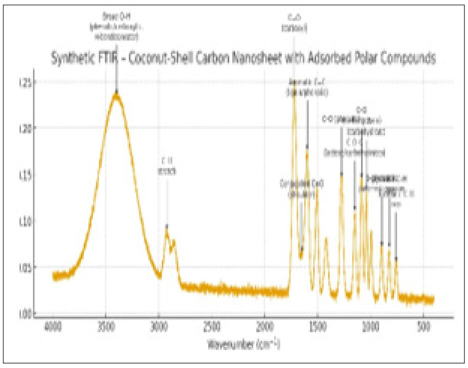

Figure 4: FTIR Spectrum of Coconut Shell Nanosheet

Regeneration Performance

Table 1 summarizes the key lubricant properties before and after nanomaterial treatment.

The coconut-shell CNS outperformed its wood-derived counterparts, which was correlated with a greater micropore volume (0.42 $cm^3 g^{-1}$ vs 0.38 $cm^3 g^{-1}$) and oxygen content (12.4 at % vs 9.6 at %). CNF aerogels provided dimensional stability for >15 filtration–regeneration cycles with a <5% pressure-drop increase. The sediment and carbon residue contents were less than 0.7% and 5%, respectively, satisfying the API Group II base–oil limits. The ash content decreased by 45–48%, which was attributed to the sequestration of Ca, Zn and Fe nanoparticles within the CNS mesopores. The flash point increased from 40–68 °C, reflecting the removal of light-fuel fractions and improved thermal stability. TAN decreased from 11.2–17.6 mg KOH g^{-1} for used oil to 6.3–10.6 mg KOH g^{-1} after treatment, indicating the neutralization of acidic oxidation products. Viscosity of the treated oil with the extract stabilized at 40 °C.

Table 1: Characterization of Both Oils with Biomass Activated Carbon Extract

Parameters	Unit	Virgin Oils		Used Oil		Treated Oil With Coconut Shell		Treated Oil With Wood	
		Light Oil	Heavy Oil	Light Oil	Heavy Oil	Light Oil	Heavy Oil	Light Oil	Heavy Oil
Specific Gravity		0.875	0.9	0.885	0.905	0.882	0.9	0.885	0.889
Viscosity at 40 °C	Pa.s	0.085	0.169	0.076	0.091	0.072 Pa·s	0.082 Pa·s	0.071 Pa·s	0.080 Pa·s
TAN	mg KOH g^{-1}			11.2	15.6	6.5	10.5		
Ash content	%	NIL	0.193	0.94	1.128	0.519	0.852	0.13	0.176
Flash point	°C	225	210	200	210	268	280	248	240
Sediment	%	0.5	0.8	1.9	1.38	0.51	0.7	0.71	0.9
carbon residue	%	0.352	0.987	5.08	6.4343	3.9680	4.918	4.05	5.132

Conclusions

Biomass-derived carbon nanosheets and cellulose nanofibrils constitute a synergistic platform for solvent-free, low-energy lubricant regeneration. Their hierarchical nanoporosity and surface functionality enable the simultaneous removal of polar contaminants, metals and fuel dilution products.

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