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Utilization of Lignin from Oil Palm Empty Fruit Bunches (OPEFB) for the Fabrication of Eco-Friendly Superhydrophobic Sponges

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Abstract

The escalation of crude oil exploitation poses significant risks of leakage and oil spills in the oceans. Therefore, this research aims to synthesize superhydrophobic sponges utilizing lignin derived from Oil Palm Empty Fruit Bunch (OPEFB) waste. Lignin was isolated from OPEFB with the addition of 15% NaOH, followed by neutralization and purification using H_2SO_4, which yielded a recovery of 30.3%. Subsequently, melamine sponges were modified via a facile dip-coating technique using a mixture of lignin, Polydimethylsiloxane (PDMS), and (3-aminopropyl)triethoxysilane (APTES). The physicochemical properties and material performance were characterized using ^{1}H-NMR, FTIR, and Water Contact Angle (WCA) measurements. ^{1}H-NMR analysis confirmed the successful isolation of the lignin structure, while FTIR spectra verified the effective deposition of the silane-lignin layer on the sponge skeleton. Contact angle analysis results indicated a significant surface transformation, where the sponge shifted from a superhydrophilic nature 0° to a highly superhydrophobic state (170.91°). With such performance, this material holds great potential as an effective, eco-friendly adsorbent for oil spill remediation in aquatic environments.

Keywords: OPEFB, Lignin, Superhydrophobic Sponge, Oil Spill Remediation.

INTRODUCTION

The rapid escalation of global industrial activity and the intensification of petroleum exploration in recent decades have precipitated severe environmental consequences, most notably the increasing risk of oil leaks and spills in aquatic ecosystems. This phenomenon poses a persistent threat to marine biodiversity and public health (Sha et al., 2024). Crude oil spills are also categorized as hazardous and toxic waste under code D221. This classification is predicated on the physicochemical characteristics of crude oil, which is reactive, highly toxic, flammable, and contains polycyclic aromatic compounds with potential carcinogenic properties (Lu & Yuan, 2018). The complexity of managing such hazardous waste necessitates the development of remediation technologies that are not only effective in separating oil from water but also efficient in terms of operational costs. Consequently, a persistent challenge lies in developing materials that balance low production costs with high absorption efficiency to mitigate the catastrophic impacts of oil spills.

Various conventional strategies have been employed to address oil spills, including chemical dispersants, containment booms, skimmers, inorganic sorbents, and bioremediation approaches (J. Wang et al., 2021). Therefore, the use of advanced sorbents is regarded as one of the most viable and scientifically promising solutions for effective oil spill cleanup. The primary advantages of the adsorption method include its high adsorption capacity, ease of field application, potential for reusability, and superior environmental friendliness compared to chemical dispersants, which often leave toxic residues (Zhao et al., 2021).

Commonly investigated absorptive materials include nanoparticles, two-dimensional (2D) materials,

and three-dimensional (3D) materials. Oleophilic nanoparticles with high adsorption capacity, such as zeolite, activated carbon, and silica, have been extensively investigated for oil removal. However, powders or nanoparticles are often challenging to recover from water bodies after absorption, potentially causing secondary pollution (J. Wang et al., 2021).

Moreover, two-dimensional materials such as synthetic films, meshes, and fabrics have exhibited superior oil–water separation performance. Nevertheless, the oil storage capacity of 2D materials is relatively limited due to their thin structural nature. Therefore, recent research attention has pivoted towards hydrophobic porous 3D materials due to their outstanding oil- and organic solvent-absorption performance. 3D hydrophobic materials possess a large internal void volume, enabling significant storage capacity and the ability to absorb and desorb oil immediately. Several advanced 3D materials developed include carbon nanotube (CNT) foams, rubber/graphene composites, cellulose aerogels, and graphene aerogels. Despite their high performance, the synthesis of these materials poses challenges of complex operations, high production costs, and the use of chemical agents that may cause environmental pollution. These factors create technical and economic barriers to large-scale application in open-ocean oil spill response (J. Wang et al., 2021).

Amidst the limitations of these advanced materials, commercial sponges have emerged as compelling alternatives. Among these 3D materials, melamine sponges act as a superior candidate due to their specific physical properties, including low density, excellent mechanical elasticity, commercial availability at low cost, and high porosity and absorbency (Deng et al., 2023; Qiao et al., 2025; Zhang et al., 2019). The melamine sponge is chemically defined by a three-dimensional copolymeric network, synthesized via the polycondensation of formaldehyde, sodium bisulfite, and melamine (Mako & Edyta, 2023). This microscopic structure provides an ideal framework for fluid retention. However, when sponges are directly applied for oil spill recovery, they inherently absorb large amounts of water due to the amphiphilic nature of their surfaces (Sun et al., 2021). The presence of secondary amine and hydroxyl groups on the melamine skeleton makes the material amphiphilic, meaning it can absorb both water and oil simultaneously. However, this amphiphilicity makes it difficult for sponges to selectively absorb oil from water, limiting their practical application in real-world environments (J. Wang et al., 2021).

To overcome this challenge, recent studies have focused on surface modification of sponges to alter their hydrophilic surfaces and render them superhydrophobic. Superhydrophobic materials have become a research hotspot for oil-water separation due to their unique wettability (Fang et al., 2023). A superhydrophobic surface provides the sponge with strong water repellency during oil absorption, ensuring that only the oil phase penetrates the sponge pores. However, fabricating a stable superhydrophobic layer presents its own challenges, as most of the superhydrophobic layer is easily destroyed and desorbed due to weak adhesion between the coating and the sponge skeleton. To enhance the stability of the superhydrophobic layer, it is essential to fabricate a robust coating on the sponge surface (J. Wang et al., 2021).

A prevalent strategy to mitigate interfacial instability involves the use of adhesive linkers. However, reliance on traditional adhesives (e.g., silanes, fluorides, epoxy resins) is increasingly untenable due to their significant ecological footprint, economic constraints, and rigorous preparation protocols. In addition, excessive adhesive can bind many hydrophobic materials and block the sponge's channels, reducing its oil-absorption capacity. Therefore, the development of a facile, green synthesis route that avoids compromising the material's porosity remains a pivotal challenge in hydrophobic sponge engineering (J. Wang et al., 2021). In the pursuit of greener functional agents, one promising modification approach involves using lignin, a natural aromatic polymer abundant in nature (Liu et al., 2022). Ideally suited for superhydrophobic fabrication, lignin possesses a high density of hydroxyl sites that serve as effective anchors for chemical grafting, while its otherwise limited hydrophilicity simplifies the surface modification process (Chen et al., 2023; Sun et al., 2021; Tan et al., 2023).

A sustainable and abundant biorefinery feedstock, oil palm empty fruit bunches (OPEFB) are a lignin-rich (20–32%) by-product of palm oil processing that currently lacks high-value applications (Buchori et al., 2025; Puasa, Ahmad, Zakaria, Khalil, et al., 2022a; Puasa, Ahmad, Zakaria, Shaharuddin, et al., 2022a). The urgency for value-added utilization is underscored by the massive accumulation of palm oil waste in Indonesia (Febriani Paula Koloay & Beatrik Manuhutu, 2024; Jainal et al., 2023; Zul Amraini et al., 2022). A study by Sun et al. (2021) demonstrated that lignin-based melamine sponges successfully achieved an oil separation efficiency of over 98.6% and withstood extreme environmental conditions.

However, no specific studies on the use of lignin isolated from OPEFB for the fabrication of superhydrophobic sponges have been reported. The utilization of lignin from OPEFB not only contributes to sustainable palm waste management but also provides an economical, biodegradable green material solution (Tan et al., 2023).

Based on this background, this research aims to develop a method for isolating lignin from OPEFB as the active ingredient in the fabrication of superhydrophobic sponges via a facile impregnation technique. The lignin isolation process commences with the hydrolysis of OPEFB waste, followed by acidification precipitation with sulfuric acid (H_2SO_4). The isolated lignin is subsequently fabricated onto melamine sponges with the addition of Polydimethylsiloxane (PDMS) using a dip-coating technique. In this system, PDMS serves as a bonding layer between lignin and the melamine sponge, enhancing hydrophobicity and improving weather resistance in the superhydrophobic sponge (Fang et al., 2023; Yang et al., 2024). PDMS is selected as it possesses superior properties compared to polyurethane, rubber, gels, or fabrics due to its elasticity, flexibility, stability, transparency, and biocompatibility (Y. Wang et al., 2024). Characterization processes are conducted via Fourier Transform Infrared (FTIR) analysis, proton Nuclear Magnetic Resonance (^{1}H-NMR), and water contact angle (WCA) measurements to assess the surface hydrophobicity performance quantitatively. Through this approach, it is expected to create a new generation of adsorbent materials that are not only technically effective in addressing oil spills but also ecologically sustainable and economically viable.

METHODOLOGY

Materials and Instrumentals

The primary raw material used in this study was OPEFB waste collected from Katingan Regency, Central Kalimantan Province, Indonesia. The chemical reagents employed included sodium hydroxide (NaOH), H_2SO_4, n-hexane, PDMS, and (3-aminopropyl)triethoxysilane (APTES). All chemicals were of analytical grade and sourced from Merck. Additionally, commercially available melamine sponges were utilized as the supporting substrate, and distilled water was used throughout the experimental procedures as a solvent.

The physicochemical properties of the synthesized materials were characterized using several analytical techniques. Functional groups were elucidated using a Bruker Tensor II Fourier Transform Infrared (FTIR) spectrometer equipped with an Attenuated Total Reflectance (ATR) accessory, operating within a spectral range of 4000–300 cm^{-1}. Structural confirmation was further supported by ^{1}H NMR spectroscopy performed on a Bruker Avance Neo 500 MHz instrument. To assess surface wettability and quantify the superhydrophobic nature of the sponges, WCA measurements were conducted via the sessile drop method. This procedure involved depositing water droplets onto the material surface and capturing digital images, which were subsequently processed and analyzed using ImageJ software to determine the precise contact angle values (Pramudita et al., 2024).

Methods

Lignin Isolation and Purification

The isolation of lignin commenced with the mechanical grinding of OPEFB, followed by sieving through an 80-mesh screen to ensure particle uniformity. The extraction process was performed by reacting 165 g of the prepared OPEFB powder with 1320 mL of 15% NaOH solution at 80°C for 2 hours under continuous stirring. After thermal treatment, the mixture was retrieved for 24 hours to facilitate sedimentation, then filtered. The resulting filtrate was acidified by slow addition of H_2SO_4 to pH 2 to induce precipitation. This suspension was centrifuged at 3,000 rpm for 20 minutes to recover the crude lignin. To enhance purity, the crude precipitate was redissolved in 1 N NaOH solution (pH 10.5–11.5) and filtered to remove insoluble impurities. The filtrate was then subjected to a second acidification step using 10 M H_2SO_4 until pH 2, and the mixture was centrifuged again under the same conditions. The final lignin precipitate was washed with distilled water until a neutral pH was obtained and oven-dried at 60°C for 4 hours. The structural properties of the isolated lignin were subsequently characterized using FTIR spectroscopy and ^{1}H-NMR.

Fabrication of Sponges Superhydrophobic

The fabrication process commenced with the pre-treatment of the melamine sponge substrate. The melamine sponge was thoroughly washed with distilled water and subjected to mechanical agitation using a shaker for 30 minutes to eliminate surface impurities, followed by oven-drying to remove residual moisture. The hydrophobic coating solution was formulated by dispersing 0.5 g of lignin and 1 g of PDMS containing 10% (v/v) APTES into 30 mL of n-hexane solvent. Subsequently, the pre-treated sponge

was fully immersed in this suspension to ensure uniform coating of the porous structure. To stabilize the coating and induce cross-linking, the sample underwent thermal curing at 80°C for 2 hours, resulting in the formation of a superhydrophobic surface. The physicochemical properties and wettability of the modified sponge were finally characterized using FTIR spectroscopy and WCA measurements.

RESULTS AND DISCUSSION

Lignin Isolation

The isolation of lignin was performed using the Kraft pulping method, which comprises two primary stages: alkaline extraction and acid precipitation. In the initial stage, lignin was separated from the lignocellulosic matrix using 15% NaOH. The separation of lignin is achieved via alkaline treatment (NaOH), which hydrolyzes the ester cross-links within the cellulose-lignin complex, effectively solubilizing the lignin for removal (Buchori et al., 2025; Fernanda et al., 2025). The mechanism is driven by the dissociation of NaOH into Na^+ and OH^- ions. The hydroxyl ions, OH-, attack the phenolic hydroxyl groups within the lignin structure, inducing deprotonation and the cleavage of ether linkages (such as α- and β-aryl ethers). This reaction generates reactive phenolate intermediates and soluble sodium lignate salts, thereby effectively solubilizing the lignin and detaching it from the OPEFB fibers (M. Aditya Pradana, Hosta Ardhyananta, 2017). Subsequently, the purification and recovery of lignin were achieved by acidifying the filtrate with H_2SO_4 to pH 2. This pH level represents the optimum condition for precipitation, where the protonation of lignin molecules suppresses their solubility and maximizes the recovery rate (Fitri Rizkiana et al., 2024). Falah et al. (2022) reported that lower pH values correlate with increased lignin yield (Falah et al., 2022; Ridho et al., 2022). Through this procedure, the final yield of isolated lignin obtained was 30.3%. This value is competitive when compared to similar isolation methods reported in recent literature. For instance, Medina et al. (2015) and Muryadi et al. (2021) achieved lignin yields of 28.89% and 30%, respectively (Medina et al., 2015; Muryadi et al., 2021). Furthermore, the theoretical lignin content in OPEFB biomass is known to range from 10% to 34.7% (Aulia et al., 2024; Puasa, Ahmad, Zakaria, Khalil, et al., 2022b; Puasa, Ahmad, Zakaria, Shaharuddin, et al., 2022b) (Puasa et al., 2022; Aulia, 2024). Therefore, the obtained yield is considered substantial as it reflects a high recovery rate relative to the raw material's composition

Interpretation of ^{1}H-NMR

^{1}H-NMR spectroscopy was performed to verify the chemical structure of the isolate. The obtained spectrum exhibited resonance patterns highly characteristic of the lignin structure (Figure 1). In the downfield region, chemical shifts in the range of δ 6.5−8.0 ppm indicated the presence of aromatic protons, which constitute the fundamental lignin skeleton. Side-chain proton signals observed at δ 4.0−5.0 ppm were associated with the Ph−CH_2−$OCOCH_3$ moiety. Specific peaks in the δ 4.2−4.5 ppm range were attributed to hydroxyl groups. The region between δ 3.0−4.0 ppm corresponded to protons involved in ether linkages (C−O−C). A prominent signal at δ 3.5 ppm confirmed the presence of methoxyl groups (−OCH_3) directly attached to the aromatic ring. As shown in Figure 2, in the upfield region, chemical shifts at δ 1.6−2.1 ppm and δ 2.1−2.45 ppm corresponded to aliphatic and phenolic hydroxyl groups, respectively. On the other hand, the range of δ 0.8−2.0 ppm represented aliphatic protons associated with various methyl and methylene groups (C−CH_2 −C and −CH_3).

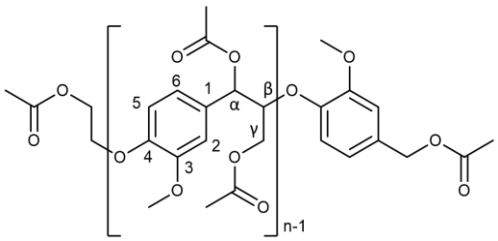

Figure 1. Structure of OPEFB Lignin

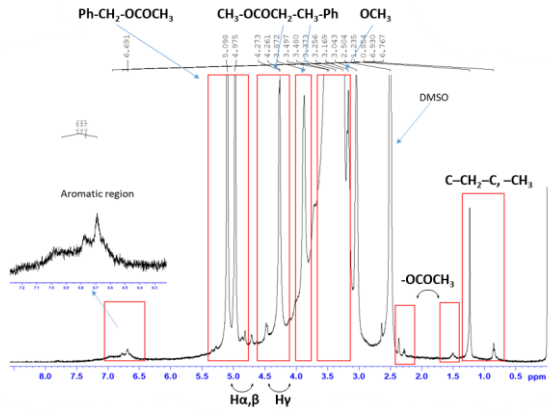

Figure 2. ^{1}H-NMR Spectrum of OPEFB Lignin

FTIR Spectral Analysis

Comparative FTIR analysis revealed that while the lignin and the superhydrophobic sponge share analogous functional group profiles, distinct spectral shifts and intensity variations were observed, indicative of chemical modifications. Both materials exhibited characteristic absorption bands at 2914 and 2887 cm^{-1}, corresponding to C–H stretching vibrations of alkyl groups (Li et al., 2025; Liu et al., 2022). A notable difference was observed in the hydroxyl region, the broad O–H stretching band at 3470 cm^{-1}, which was prominent in the lignin spectrum, appeared significantly diminished in the superhydrophobic sponge, reflecting a reduction in surface hydrophilicity (Utami et al., 2024). In the fingerprint region, aromatic skeletal vibrations were detected at 1626, 1241, and 1034 cm^{-1} for lignin. In contrast, the sponge displayed shifted peaks at 1545, 1268, and 993 cm^{-1}. Specifically, the shift of the aromatic C-O stretch band (syringyl unit) from 1241 cm^{-1} (lignin) to 1268 cm^{-1} (superhydrophobic sponge) indicates the interaction of these groups (Shi et al., 2019).

Furthermore, the superhydrophobic sponge exhibited a sharp, dominant peak at 804 cm^{-1}, attributed to N–H wagging vibrations likely originating from the melamine substrate and APTES functionalization. In contrast, lignin showed a much weaker signal at 889 cm^{-1}, suggesting differences in amine/amide content (Figure 3). These spectral alterations confirm the successful deposition of a coating layer comprising PDMS and APTES onto the melamine sponge, which introduces new functional groups and masks the original surface characteristics of the lignin.

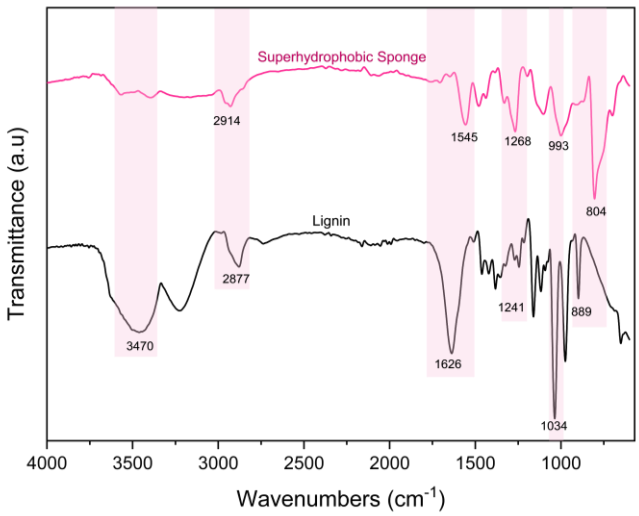

Figure 3. FTIR Spectra of Lignin and Superhydrophobic Sponges

Contact Angle Analysis

Surface wettability analysis via WCA measurements is the definitive method for characterizing the physicochemical interaction between a liquid phase and a solid surface. This parameter functions not merely as a geometric descriptor but also as a crucial predictive index for evaluating material performance across various applications, including corrosion resistance, impact resistance, and, specifically, oil-water separation efficiency. According to established surface chemistry principles, the wetting behavior of a solid surface is classified into distinct regimes based on the magnitude of the contact angle (θ). As systematically delineated by Wang (2024), a surface is categorized as superhydrophilic when $\theta < 10°$, hydrophilic when $\theta < 90°$, hydrophobic when the angle lies between 90° and 150°, and superhydrophobic when θ exceeds the critical threshold of 150° (Y. Wang et al., 2024).

In the context of melamine sponges, the pristine material inherently possesses high surface energy and porosity, leading to immediate water absorption. As reported in previous investigations by Mako´s-Chełstowska (2023), pure unmodified melamine sponges demonstrate a WCA of 0°, indicating a superhydrophilic nature that naturally limits their selectivity in distinguishing between oil and water phases (Mako & Edyta, 2023). In sharp contrast to the pristine substrate, this study's experimental results reveal a dramatic transformation in surface properties following lignin-based modification. The functionalized sponge exhibited an exceptional WCA of 170.91° (Figure 4). This experimental value far surpasses the 150° threshold required for superhydrophobicity. The drastic transition from 0° in the raw material to 170.91° in the modified sample confirms the successful deposition of low-surface-energy agents, specifically lignin and PDMS, onto the sponge skeleton. This substantial increase in contact angle indicates that the coating effectively masked the melamine's hydrophilic groups, resulting in a surface with extreme water repellency. Consequently, these findings validate that the lignin-modified sponge exhibits superior superhydrophobic properties, making it a highly efficient candidate for selective oil-absorption applications.

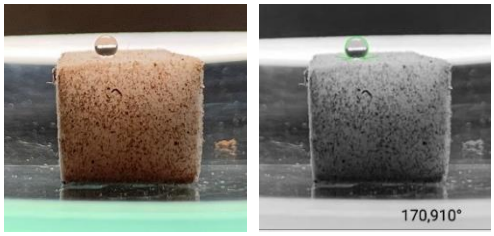

Figure 4. Contact Angle of Superhydrophobic Sponges

CONCLUSION

This study successfully demonstrated the use of OPEFB waste to produce superhydrophobic sponges. Lignin was effectively isolated with a 30.3% yield and utilized to coat melamine sponges via a facile dip-coating method with PDMS and APTES. The modification resulted in a dramatic change in wettability, increasing the WCA from 0° to an exceptional 170.91°, as validated by ^{1}H-NMR and FTIR analyses. Consequently, this lignin-based sponge offers a sustainable, low-cost, and highly efficient solution for oil spill remediation, effectively promoting a circular economy approach to agro-industrial waste management.

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