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The Significance Of Cellulose Aerogel In Oil Adsorption From PWW And Its Effect On Oil Viscosity

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Abstract— *This study investigates the use of cellulose aerogel, derived from agricultural waste, as a sustainable solution for removing oil from produced wastewater (PWW) generated during oil extraction. Conventional oil removal methods are often inefficient and expensive, whereas cellulose aerogel demonstrated a high crude oil absorption capacity of 24.4 g/g more than double that of polypropylene fibers (≈10 g/g). Scanning Electron Microscopy (SEM) revealed a highly porous structure with 97.3% porosity, enabling efficient oil uptake. Viscosity testing showed a significant reduction in the viscosity of oil contaminated water, from 42 Pa·s to 0.0027 Pa·s at 60°C, facilitating improved oil recovery. The aerogel maintained 85% of its performance after five reuse cycles and reached saturation within 20 minutes, confirming its rapid and reusable nature. These findings highlight the potential of cellulose aerogel as an effective and environmentally friendly alternative for oil pollution remediation. Further research is recommended to assess its scalability and performance in real-world applications.*

Key words: *Cellulose Aerogel, Oil Adsorption, Wastewater Treatment, Nanocellulose, Environmental Remediation.*

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1) INTRODUCTION:

Oil spills are a critical environmental challenge, causing significant harm to marine ecosystems and surrounding habitats. The release of hydrocarbons into aquatic environments can lead to severe ecological consequences, including the disruption of local wildlife and the degradation of water quality. With thousands of oil spills occurring annually due to increased extraction, transportation activities, and natural leaks, effective remediation strategies are essential to mitigate these impacts. The complexity of oil spill cleanup has prompted researchers to explore various materials and techniques for oil absorption. Among these, sorbents have emerged as a key solution. These materials work by absorbing oil from the water's surface, thereby reducing the potential for further environmental contamination. Various types of sorbents exist, including natural options such as straw, hay, and sawdust, alongside synthetic alternatives like polypropylene fibers. Each type has its advantages and limitations, but many traditional methods face challenges related to cost, efficiency, and environmental impact. In recent years, cellulose has gained attention as a promising material for oil spill remediation. This naturally occurring polymer, found in plant cell walls, is lightweight, biodegradable, and abundant, making it an attractive candidate for developing effective oil sorbents. Specifically, cellulose aerogel—an advanced form of cellulose characterized by its highly porous structure—has shown exceptional oil absorption capabilities. The high surface area and voids within the aerogel allow it to absorb significant amounts of oil, making it suitable for addressing spills in various aquatic environments [1]. This paper aims to investigate the use of cellulose aerogel for oil absorption, focusing on its effectiveness, impact on oil viscosity, and potential as a cost-effective remediation solution. The approach utilizes cellulose derived from agricultural waste, aligning with sustainability goals and promoting the circular economy. By transforming agricultural byproducts into valuable materials for pollution control, we can reduce waste while developing innovative strategies for environmental protection. The study's objectives include preparing cellulose from readily available agricultural waste, characterizing the resulting aerogel using techniques such as scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), Fourier-transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD). Additionally, the research will evaluate the cellulose aerogel's oil absorption capacity and its effect on the viscosity of different oil types. Moreover, the feasibility of implementing cellulose aerogel in practical applications will be assessed. The project aims to establish a straightforward extraction process that can be easily replicated in various settings, including local laboratories and communities. By demonstrating the efficacy of cellulose aerogel in treating oily water, this research seeks to provide a viable alternative to conventional oil spill remediation technologies, which often involve expensive equipment and complex procedures. Ultimately, this study underscores the importance of developing sustainable and effective solutions for oil spill remediation. By leveraging natural materials like cellulose aerogel, we can enhance oil absorption capabilities while minimizing environmental impact and costs. The findings will contribute to ongoing efforts in environmental conservation and highlight the potential of innovative materials in addressing one of the pressing challenges of our time: water pollution caused by oil spills. Through this research, we aim to promote cleaner waterways and protect vital ecosystems for future generations [2].

I. EXPERMINTAL METHEDOLOGY:

A. Material:

Mechanicals Material:

palm leaves, mill machine, analytical balance, Soxhlet flask, Soxhlet extraction, pipette dropper, distilled water, filter paper, funnel, oven, conical flask, lab spatula, a magnetic stirrer, magnetic hot plate, Sonication, centrifuge, sieve, glass beakers, crude oil, Cellulose aerogel and Methyl trimethoxy silane (MTMS).

Chemical Material:

Ethanol, Toluene, Sodium Hydroxide, Acetate Acid, H_2O_2, H_2SO_4, NaCl, Urea solution.

B. Methdology:

1. Extraction of Nanocellulose from plant west (palms leaves):

Figure 1: palm leaves

Figure 2: mill machine

Figure 3: after grinding

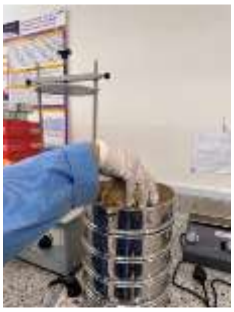

Figure 4: separating sieve

Figure 5: sample size 0.212

Figure 6: sample size 0.251

Figure 7: sample size 0.425

Figure 8: weight sample

Take 10 grams of 0.251mm size of palm leaves particles with 10% (10 grams) sodium hydroxide and 200 ml of distilled water and heat the mixture to 160 ° C for two hours in a hot magnet plate, then wash the sample with distilled water, filter it, and dry the fibers well in Oven one night. On the second day 10 grams of the sample was combined with 200 milliliters of 20% H2SO4 and heated to 250 °C for 1 hour while stirring moderately. 500 mL of cold distilled water was added to the mixture to stop the reaction. The pH of the concentrate cake was adjusted to 7-8 using 5% w/v NaCl.

The resulting suspension will then undergo a 5 10 min centrifugation process at 6000 rpm. The supernatant will then be depleted, replenished with DI water, and the sample placed in the oven one night [3].

Figure 9: Weight of NaOH

Figure 10: After 2 hr

Figure 11: Filtration

Figure 12: Drying sample in the oven

Figure 13: H2SO4

Figure 14: 2hr in hot plate

Figure 15: Centrifuge

Figure 16: pH analysis

2. Alkalization:

Twenty grams of sodium hydroxide were accurately measured using an analytical balance and placed into three glass beakers, each containing 300 ml of distilled water. A magnetic stirrer was added to facilitate even heat distribution, and the beakers were heated to 80 °C for one hour. After this period, the samples were washed with hot distilled water to neutralize the pH. This washing process was repeated until the samples were free of impurities, colourless, and had a normal pH. The samples were then filtered using filter paper and a funnel, dried, and placed in the oven overnight [4].


Figure 17: Weight of NaOH


Figure 18: The sample in solution


Figure 19: The sample after 2hr


Figure 20: Filtration the sample


Figure 21: After the filtration


Figure 22: After washing


Figure 23: Measuring the pH


Figure 24: Dring samples

3. **Bleaching:**

A solution consisting of acetate acid 20 ml and H_2O_2 20 ml was prepared in a glass beaker, and a magnetic stirrer was placed inside it to stir the sample and prevent its adhesion, then it was placed on the hot magnetic plate and the temperature was fixed at 90 degrees Celsius for 6 hours. Then cold water was added to stop the reaction, and the sample was separated using a centrifuge for 10 minutes. After separating the sample, it was placed in the oven for one night to dry it [5].


Figure 25: Heating sample


Figure 26: Stop reaction


Figure 27: Centrifuge separation


Figure 28: Separate the sample


Figure 29: After filtration

C. Characterization of Nano cellulose

Scanning electron microscopy (SEM), X-ray diffraction (XRD) And watch the fibers break down into cellulose nanofibers with different dimensions ranging from 20- 25 nm. Therefore, in this case, high cellulose fibers were obtained in the extracted samples, and the amount of carbohydrates did not change much.

D. Fabrication of Recycled Cellulose Aerogels

Recycled cellulose fibers (20 g, 2% weight) were sonicated for 10 minutes in a solution containing 1.9% sodium hydroxide and 10% urea. The mixture was then placed in a coil refrigerator for over 24 hours to facilitate gel formation. Afterward, the solution was frozen for two days and then thawed at room temperature, followed by immersion in 99% ethanol to induce coagulation. A beaker was used as a template to control the thickness of the sample. Once coagulated, the gel was immersed in deionized water for two days to achieve solvent exchange and then freeze-dried for two days. To enhance the oil absorption capacity of the cellulose aerogels, they were modified with methyltrimethoxysilane (MTMS), which improves hydrophobicity. The cellulose aerogels were placed in a glass container, and MTMS was added in specific quantities based on the desired 70° hydrophobicity. The precise amount of MTMS was calculated according to the volume of cellulose aerogel. The MTMS and aerogels were thoroughly mixed to ensure an even coating, and the mixture was observed for any reactions. After modification, the cellulose aerogels were removed from the MTMS solution and placed on filter paper to absorb any excess liquid. The aerogels were then allowed to cure and dry, after which they were weighed using an analytical balance [6].

Figure 30: Recycled cellulose fibers

Figure 31: The sample in the solution

Figure 32: The sample in freezer

Figure 33: Ethanol to coagulate

E. Characterize the cellulose aerogel:

Examination of cellulose aerogel using SEM tests: Using field-emission scanning electron microscopy (FE-SEM), aerogel samples were studied. Prior to FE-SEM, the samples were stored in a dry cabinet. Then, using sputtering, a thin gold layer was applied to them. take pictures of the aerogels' structural details [7].

F. Crude Oil Absorption Test:

Carefully put the modified cellulose aerosols into the oil and allow it to absorb the oil. Stir the mixture to facilitate absorption and ensure that the aerogels are saturated with oil. After an absorption period, the cellulose aerogel was removed from the crude oil. Place them on filter paper to remove the oil and weigh the aerogels using an analytical balance. The crude oil adsorption capabilities of the aerogel samples were investigated using a modified ASTM F726-06 test method 5,10,20,37 examining the aerogel particles for their ability to absorb crude oil and separate from water [8] [9].

Figure 34: *Crude Oil Absorption*

II. RESULTS AND DISCUSSION:

- **SEM analysis of Cellulose extraction:**

Cellulose nanoparticles can be analysed using SEM (Scanning Electron Microscopy), which reveals detailed information about their size, shape, distribution, and composition. Cellulose is a primary component of plant cell walls, alongside wax, pectin, lignin, and hemicellulose, constituting about 25 to 50 percent of a plant's total weight. In this study, SEM was conducted on samples collected from palm fronds after wax removal and thorough cleaning to ensure clarity and accuracy without impurities. The SEM scans provided images of varying dimensions for each sample, allowing for the identification of nanoparticles. The obtained images, presented in the figure, show cellulose particles of different shapes and sizes. Various magnifications were used, including 1000, 3000, 5000, 10000, and 20000 [7] [10].

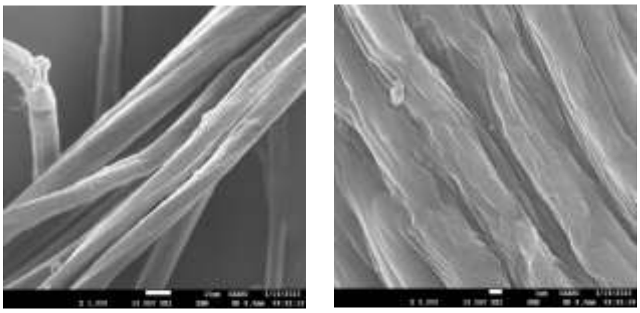

Figure 35: SEM analysis for Sample 1

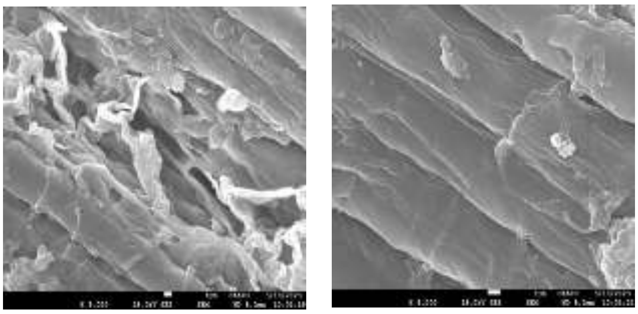

Figure 36: SEM analysis for sample 2

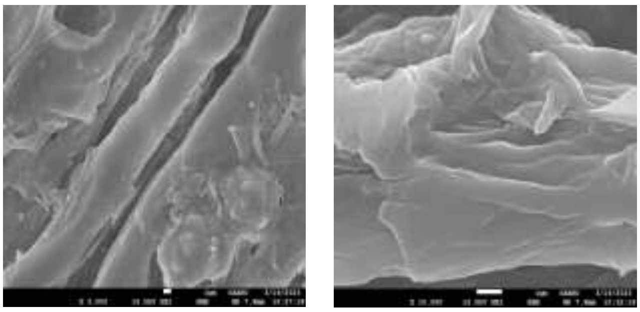

Figure 37: SEM analysis sample 3

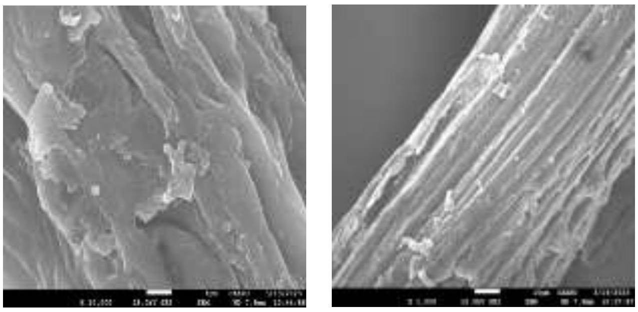

Figure 38: SEM analysis sample 4

- **EDX analysis of Cellulose extraction:**

Cellulose is a complex carbohydrate that serves as the main structural component of plant cell walls, consisting of long chains of linearly connected glucose units. The goal of analysing cellulose using energy-dispersive X-ray spectroscopy (EDX) is to understand its elemental composition and properties. Samples of cellulose were obtained from sources such as palm leaves, and the cellulose was purified into a thin film suitable for analysis. The EDX results reveal various peaks, which provide information about the elemental composition of the cellulose, including the presence and concentration of different elements. The peaks indicate the elements present, allowing for insights into the material's characteristics. The degree of crystallinity, referring to the order within the cellulose chains, can also be inferred from the elemental analysis. Given the high cellulose content in the sample, a significant percentage of compound C is evident [7] [10].

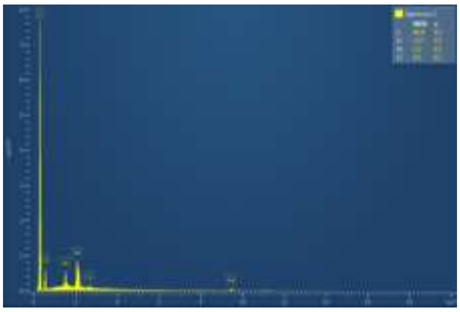

Figure 39: EDX analysis sample 1

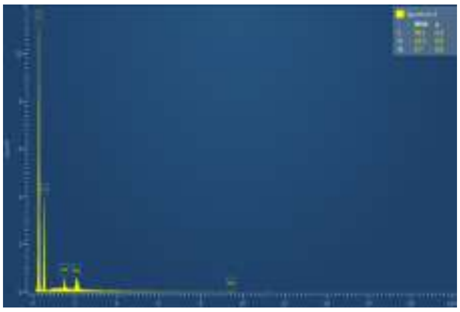

Figure 40: EDX analysis sample 2

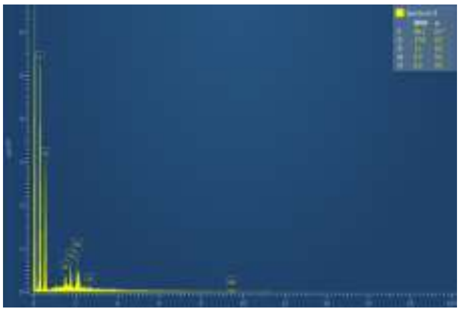

Figure 41: EDX analysis sample 3

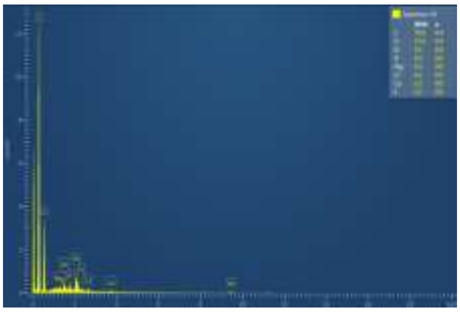

Figure 42:EDX analysis sample 4

As a result, it was obtained that sample No. 1 and sample No. 4 contain the largest percentage of cellulose, which is 80% and 60%.

- **XRD analysis of Cellulose extraction:**

X-ray diffraction (XRD) is a common technique for examining the crystal structure and chemical composition of materials, including cellulose molecules. Cellulose, a polysaccharide found in plant cell walls, is primarily composed of natural fibers. The sample underwent XRD analysis, and the results are illustrated in the figure below. The diffraction peaks indicate that cellulose possesses a distinct crystalline structure. The XRD pattern of cellulose shows characteristic peaks due to the repeating units of cellulose chains within the crystal lattice, typically appearing at angles of 20° and 25°. These results confirm a high concentration of cellulose particles in the sample. Additionally, the presence of other peaks in the XRD pattern may reveal other crystalline phases or impurities, providing further insights into the composition and characteristics of the cellulose particles [7] [10].

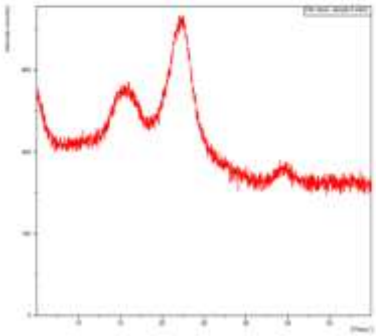

Figure 43: XRD analysis sample 1,2,3

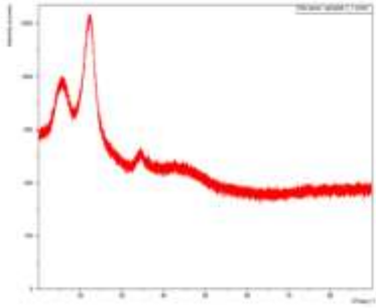

Figure 44: XRD analysis sample 4

- **SEM analysis of cellulose aerogel:**

Scanning field emission electron microscopy (FE-SEM) was utilized to investigate cellulose aerogel samples after they were made hydrophobic. The contact angle increased to 152°, and the crude oil-water separation efficiency reached 50%. SEM provided structural images of the aerogels, revealing a network of uniformly interconnected cellulose fibers that form the unique porous structure of the aerogel. The recycled cellulose fibers contributed to the creation of a significant three-dimensional network in the 38 aerogels examined. These gel nanoparticles exhibit low density and a high porosity of 97.3%. All aerogels displayed a homogeneous structure with highly porous networks, indicating minimal shrinkage during the freeze-drying process. This approach presents a simple, efficient, and environmentally friendly method for preparing cellulose-CNT composite aerogels, which are highly porous solids containing three-dimensional networks of air-filled pores. Their distinctive characteristics include low density (0.004 - 0.500 cm^3) and substantial internal space [7] [10].

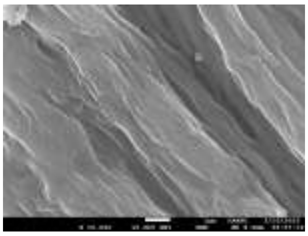

Figure 45: SEM analysis for cellulose aerogel sample 1

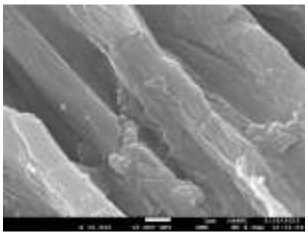

Figure 46: SEM analysis for cellulose aerogel sample 2

Figure 47: SEM analysis for cellulose aerogel sample 3

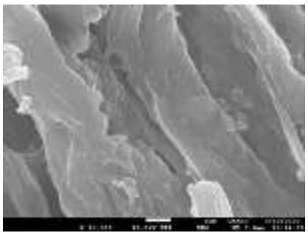

Figure 48: SEM analysis for cellulose aerogel sample 4

- **Crude oil adsorption:**

To investigate the behaviour of crude oil adsorption on MTMS-coated aerogel, the absorption time was measured in minutes. The results show that the medium readily absorbs crude oil, becoming fully submerged after four minutes, indicating a strong adsorption affinity. High absorption rates were observed, with saturation reached within approximately 20 minutes, marking the initial phase of adsorption. When compared to commonly used sorbents for oil spill cleanup, such as polypropylene fiber mats, the aerogel demonstrated nearly double the effectiveness. The low viscosity of the crude oil significantly influences its absorption behaviour. The oil absorption mechanism relies on capillary action, as well as van der Waals forces, significant hydrophobic interactions, pore shape, and oil viscosity. In this case, the viscosity of the oil is a key factor affecting absorption capacity, with lower viscosity facilitating better penetration into the aerogel's pores. Moreover, the influence of temperature on crude oil adsorption indicates that the absorptive capacity increases with rising temperatures. At 10 °C, the oil transforms into a gel with a high viscosity of 42 Pa, which reduces absorption capacity due to difficulty penetrating the pores. As the temperature increased to 25, 40, and 60 °C, the viscosity of the crude oil decreased to 0.0090, 0.0049, and 0.0027 Pa·s, respectively, allowing for easier and faster diffusion into the porous matrix of the aerogel [8] [9].

III. CONCLUSION:

In conclusion, there are significant environmental issues related to oil extraction and water pollution that have negative impacts on both the environment and the economy. Traditional methods of treating oil-contaminated water are often unsustainable, expensive, and have negative environmental effects. Nanotechnology, specifically the use of cellulose aerogel, shows promise in addressing these challenges by effectively removing oil from water [11]. Cellulose aerogel, derived from plant waste, is a cost-effective and environmentally friendly option for oil-water separation. The use of nanoparticles in this process has shown efficient oil absorption without the need for complex chemical treatments. The research conducted in this study involved the extraction of nanocellulose from palm leaves and its transformation into cellulose aerogel, which was then tested for its ability to absorb oil. Various characterization tests were conducted to confirm the composition and properties of the extracted cellulose aerogel. The study demonstrated that cellulose aerogel can effectively clean wastewater and produce oil absorbents suitable for different oil viscosities. The research presented in 17 analyzed journals covered various topics related to cellulose recovery, cellulose aerogels, and oil-water separation. The significance of this project lies in the development of a cost-effective and environmentally friendly method for oil-water separation using cellulose aerogel [12].

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