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Synthesis and Characterization of Nanostructured Sorbents Derived from Rice Husks using FTIR, SEM, TEM and XRD Approaches

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

This study focuses on the development and characterization of a nanostructured sorbent derived from an agricultural waste. The rice husks were collected from a rice mill, washed and ground into fine powder, and then subjected to chemical and thermal treatments to convert them into nanostructured materials with enhanced surface properties suitable for oil adsorption.

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Characterization of the sorbent was done using scanning electron microscope (SEM), transmission electron microscope (TEM), Fourier Transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD) spectroscopy, energy dispersive X-ray (EDX) spectroscopy and Brunauer–Emmett–Teller (BET) surface analysis. Results obtained confirmed the nanostructural nature of the sorbent, revealing high porosity and the presence of functional groups that promote oil adsorption. The study concludes that the sorbent developed from rice husks could provide an ecofriendly and cost-effective nanomaterial that could effectively be deployed for remediation of oil-contaminated water.

Keywords: Nano sorbent; nano materials; sorption capacity; adsorption isotherm; water remediation.

1. INTRODUCTION

Agro-based companies' produces so much waste during production. Parts of the plants which are non-consumable are referred to as Agro-waste, and consumable parts are the major source of energy for humans. (Yuan et al., 2022). Agro waste is produced as a by-products of agricultural activities such as the processing and manufacturing of the vegetables, fruits poultry, meat, and dairy products (Pattanaik et al., 2019). Agro waste comes in different forms (i) Agricultural residue (ii) Residues from the agricultural industry (Biriniwa et al., 2022). Inappropriate disposal of these agro wastes can harm the earth's surface and groundwater, resulting in contamination. Important biochemicals such as lignin, cellulose, chitin and polyphenolic compounds found in the horticulture, aquatic, or culinary are from agricultural waste (Lu et al., 2022). Agricultural waste materials could be used to make bio fuels as well as organic solvents like ethanol, acetone and butanol. (Ahmed et al., 2022). Agro waste such as Rice husk, Coconut shell, melon husk, wheat straw etc. are used as a replacement for fossil fuel and the manufacturing of nanomaterial. The fabrication of Nano material using Agro-waste as substrates is fast gaining popularity due to its economic, environmental, and technological advantages (Elemike et al., 2022).

As water pollution caused by heavy metal, oil spill and other pollutant has recently become a major environmental and public problem all over the world as a result of rising industry and urbanization, there is a growing interest in the development of efficient and cost-effective nano materials that could serve as sorbents for the remediation of oil contaminated water resources. Nanotechnology is a pivotal field with significant implications for the development and characterization of nanostructured sorbents from agricultural waste materials. It involves the manipulation and utilization of materials at the nanoscale, offering numerous advantages in addressing environmental challenges, such as the remediation of crude oil-contaminated water. Nanotechnology encompasses the design, manipulation, and application of materials and structures at the nanoscale, typically ranging from 1 to 100 nanometers. In the context of sorbent development, nanotechnology enables the creation of nanostructured materials with tailored properties. These materials have high surface areas, unique reactivity, and precise control over their structural and functional characteristics. Nanotechnology holds several key advantages in the development of nanostructured sorbents from agricultural waste. Some of these key advantages include enhanced surface area of the nano-material, controlled properties, and tailored sorbents. A study by Kumar et al. (2019) explored the application of nanotechnology in the development of sorbent materials for the removal of oil and hydrophobic contaminants from water. The research emphasized the potential of nanotechnology in creating advanced sorbents that efficiently capture oil and related pollutants. This study seeks to develop nano-structured sorbents from rice husks, a common agricultural waste, and to characterize the nano-material using FTIR, UV, SEM, TEM and XRD techniques for possible application as a sorbent to remediate oil contaminated water.

2. MATERIALS AND METHODS

2.1 Sample Collection and Pre-Treatment

Rice husks were collected from a rice mill in Owerri Municipal, Imo State, Nigeria. The identity of the sample was authenticated at the Biological Department, Chukwuemeka Odumegwu Ojukwu University, Uli, Anambra State. The rice husk materials were washed with distilled water to remove dirt, dust or any other impurities. The sample was dried in an oven at 60-80°C until it was completely dried. The dried sample was ground into fine powder using a grinder. The ground sample was sieved to ensure that uniform particles between 50-100 microns were obtained.

2.2 Synthesis of Nano Structured Rice Husk Sorbent Using Bottom up Approach (Chemical Synthesis)

Exactly 500ml of 0.5 M of NaOH was added into 81 g of sample (finely ground rice husk). Precipitate of nano sized particles of rice husk were obtained. The nano sized particles of rice husk formed were collected and dried in an oven (Larkundthod et al., 2022).

2.3 Surface Functionalization of Nano Structured Sorbent

Exactly 500 ml of 0.5 M solution of H_3PO_4 was used to introduce the acid functional group in the nano sorbent. The treated nano sorbent was rinsed with distilled water and dried.

2.4 Characterization of The Nano Sorbent Using FTIR

The rice husk having quantity 2 mg sorbent was mixed with 100 mg of potassium bromide (KBr) to form a paste. The KBr-sorbent paste was pressed into a pellet using a pellet press which created a transparent disc for analysis. The Fourier Transform infrared (FTIR) spectrometer of Model ATP8900plus was turned on, allowed to warm up and after the instrument was calibrated, the sample scanned such that the wavelength was typically 400 – 4000 cm^{-1}, the resolution was 4 cm^{-1} and the number of scan was 16–32 scans for a clear spectrum.

2.5 Characterization of The Nano Sorbent Using TEM

The prepared sorbent was subjected to Transmission Electron Microscopy (TEM) of Model JEM-Z200FSC. In doing so, ethanol was added to the sorbent as a dispersing agent. Ultrasonic bath for 20 minutes was used to ensure that the particles are well-dispersed and do not form aggregates. Carbon-coated copper was used to prepare 400 mesh TEM grids for better conductivity and support. Futher, 2 drops of the dispersed sample suspension were taken with a pipette and was carefully placed onto the surface of the TEM grid. Excess liquid was blotted using filter paper or a fine tissue. The grid was air-dried completely. The TEM instrument was turned on, warmed up and properly calibrated before loading the sample to ensure that the electron beam is focused for high-resolution imaging. The dried TEM grid and the sample were inserted into the sample holder using tweezers. The sample holder was loaded into the microscope for imaging in a vacuum condition.

Fig. 1. A heap of rice husks

2.6 Characterization of The Nano Sorbent Using SEM

Scanning electron microscope (Model No. JSM-IT510) was also used to characterize the sorbent. Exactly 0.5 g of the prepared sorbent sample was attached to the stub of the scanning electron microscope stub and was coated. The sample was loaded into the instrument and imaging parameters were adjusted. High-resolution images were captured to analyse the morphology and structure of the nanostructured sorbent. The images were documented and particle size, shape, and surface features were analysed.

2.7 Characterization of The Nano Sorbent Using XRD

The rice husk sorbent having quality 0.5 g was placed into the X-ray diffraction (XRD) sample holder. The model number of the XRD instrument used was DW-XRD-Y3000. A spatula was used to evenly spread the powder across the surface of the holder. The sample formed a thin, smooth, and flat layer, as an uneven sample can cause inaccurate diffraction patterns. The XRD machine was powered on and allowed to warm up. Before the sample was placed, the XRD system was calibrated using silicon. The prepared sample holder and rice husk sorbent were placed into the XRD machine's sample. The XRD chamber was securely closed, as it needs to be at a specific atmosphere. XRD Parameters were set with 20° scan range. The step size was set to 0.02° for fine resolution. A slower scanning speed of 0.5°/min was set which provides higher resolution but takes more time. After the parameters was set, the scan was Uniates. The XRD will record the intensity of diffracted X-rays as a function of the diffraction angle (2θ), producing a diffraction pattern that represents the material's crystallographic structure. The instrument ran smoothly and the sample remained in place. The XRD software generated a diffraction pattern after the scan was completed, which consisted of peaks corresponding to the crystalline phases in the sample. Crystalline phases were identified to match the diffraction peaks with known crystallographic structures present in the rice husk sorbent.

2.8 Characterization of The Nano Sorbent Using Bet

0.5 g of the nano sorbent was placed into a sample tube designed for BET analysis. The sample was degassed at 200°C for 4 hours by heating it under vacuum. This step was critical to remove any adsorbed gases and moisture that could interfere with the BET measurements. The BET instrument was turned on and was allowed to stabilize. The instrument was properly calibrated and the gas supply was connected to flowing at the correct pressure. The degassed sample was loaded to the sample tube into the BET instrument sample holder. The sample tube was properly sealed in the analysis chamber, and the system was under inert gas flow to avoid contamination from ambient air. Nitrogen gas was used as the adsorbate, which interacts with the surface of the material to measure the surface area. The pressure range was set to 0.3 of the adsorbate gas. The instrument introduced nitrogen gas into the chamber in controlled amounts, allowing it to adsorb onto the surface of the sample at different pressures. The BET analyser recorded the amount of gas adsorbed and desorbed at different pressures, generating data that describes the material's surface area and porosity. After the completion of the analysis, the system generated an adsorption-desorption isotherm, which is a plot of the amount of gas adsorbed versus the relative pressure. Using the BET equation, the system automatically calculates the specific surface area (m²/g) of the rice husk sorbent based on the isotherm data and in agreement with the equation that follows;

$$\text{Specific surface area (BET)} = \frac{1}{\mathrm{V}[(\mathrm{P0}/\mathrm{P})-1]} - \frac{\mathrm{C}-1}{\mathrm{VmC}} \cdot \frac{P}{Po} + \frac{1}{\mathrm{VmC}}$$

Where *V* is the volume of gas adsorbed at pressure PPP, *Vm* is the volume of gas required to form a monolayer, *Po* is the saturation pressure of the gas, *C* is the BET constant related to the energy of adsorption, and $\frac{P}{Po}$ is the relative pressure.

3. RESULTS AND DISCUSSION

3.1 Synthesized Nano Structured Sorbent

Table 1 shows the parameters involved in the synthesis of the nano sorbent

3.2 Surface Functionalization of Rice Husk Sorbent

When 500 mL of 0.5 mol of H_3PO_4 was added to rice husk nano sorbent, phosphates functional groups were also introduced to enhance the reactivity, solubility and binding affinity. The added phosphates did not significantly increase the molecular weight of the nano rice husk sorbent because the phosphate group (PO_4^{3-}) is relatively small and light compared to the molecules they modify. The mass of a phosphate group is only about 95 Da (Daltons), which is a minimal addition to the overall molecular weight of large molecules or complex systems like proteins or functionalized surfaces. The primary purpose of phosphates is not to increase weight but to change the chemical reactivity, which enables more control over interactions in processes such as binding, catalysis, or surface activation. The synthesis reaction is stated as follows;

$$Rice\ husk\ (Silica; SiO_2) + NaOH + H_3PO_4 \\ \rightarrow Nano\ rice\ husk\ sorbent\ (Silica \\ - phosphate - Na)$$

The balanced equation is stated as follows;

$$SiO_2(Rice\ Husk) + 0.5\ NaOH + 0.25\ H_3PO_4 \\ \rightarrow SiO_2PO_3Na\ (Nano\ Rice\ Sorbent) \\ + 2.5\ H_2O$$

3.3 Fourier Transform Infrared Spectrum of The Rice Husk Nano Sorbent

The Fourier infrared spectrum of the rice husk nano sorbent is presented in Fig. 2.

This spectrum in Fig. 2 is an FTIR (Fourier Transform Infrared) spectrum, for the nano sorbent derived from rice husk (RH). A cursory look at the spectrum will reveal a broad absorption peak at 3439.00 cm⁻¹ which is characteristic of the O-H stretching vibrations, suggesting the presence of hydroxyl groups (–OH) typically associated with water molecules or alcohols, indicating the possible presence of surface hydroxyl groups and moisture adsorbed on the rice husk nano-sorbent. There is also a peak at 2926.29 cm⁻¹ which is suggests the C-H stretching vibrations of aliphatic $–CH_2$ groups. It represents the organic components inherent in rice husk, such as cellulose, hemicellulose, and lignin. The absorption peak at 2369.33 cm⁻¹ can be attributed to the presence of atmospheric CO_2 and some organic components present within the material. It's not commonly associated with functional groups typical of nano-sorbents but might arise due to environmental contamination or byproducts of processing. The peak that occurred at 1728.45 cm⁻¹ represents the C=O stretching vibrations, typically associated with ester and carbonyl groups (–COOR and –C=O). This indicated the presence of organic compounds and residues in the rice husk and the functional groups introduced during the modification process of the nano-sorbent. There is also a peak at 1637.57 cm⁻¹ which is commonly assigned to C=C stretching in alkenes or the bending mode of adsorbed water molecules. It can also be linked to the aromatic structures in lignin, which is one of the primary components of rice husk. The peak at 1514.38 cm⁻¹ is characteristic of aromatic ring vibrations, particularly from lignin, which is a major component of rice husk. It also indicates functionalization involving aromatic compounds. The absorption band or peak at 1427.00 cm⁻¹ is likely due to C-H bending vibrations in $–CH_2$ or $–CH_3$ groups, which are common in cellulose and lignin structures. The peak found at 1381.79 cm⁻¹ is attributed to O-H bending vibrations, possibly from phenolic groups or adsorbed water (Singh et al., 2021). It may also relate to the presence of cellulose and hemicellulose in the rice husk. There is a peak at 1329.00 cm⁻¹ and this peak is characteristic of C-O stretching vibrations of alcohols, carboxylic acids, and phenolic groups. It indicates the presence of oxygenated functional groups in the material. The peak at 1256.29 cm⁻¹ may correspond to C-O stretching or C-O-C linkages in polysaccharides, often found in cellulose and hemicellulose, which are major components of rice husk, while the peak at 1039.46 cm⁻¹ is indicative of C-O stretching vibrations, particularly in polysaccharides, and could also be associated with Si-O-Si stretching, suggesting the presence of silica in the rice husk, which is a known constituent.

Table 1. Parameters of the synthesized risk husk sorbent

S/N	Parameters	Value/Amount
1	Weight of rice husk sample	81 g
2	Volume of 0.5 M solution of NaOH	500 ml
3	Moles of NaOH that reacted	0.25 mol
4	Moles of rice husk (SiO_2) that reacted	0.125 mol
5	Mass of rice husk (SiO_2) that reacted	7.51 g
6	Mass of sorbent (Na_2SiO_2) produced	15.26 g

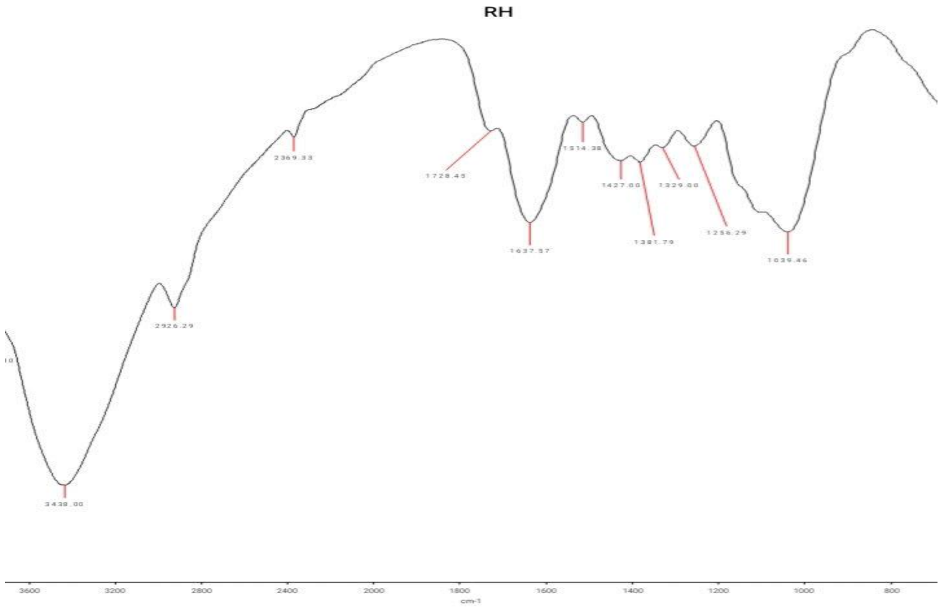


Fig. 2. FTIR spectrum of the rice husk sorbent

The FTIR spectrum above shows that the rice husk nano-sorbent retains significant organic and inorganic components. The O-H, C-H, and C=O functional groups are prevalent, showing the presence of cellulose, hemicellulose, and lignin. Additionally, the peaks related to Si-O indicate the presence of silica, which is a major component of rice husk ash. The presence of carbonyl groups (C=O) and aromatic structures might indicate modifications or the retention of the natural structure of lignin. This spectrum confirms that the rice husk-based nano-sorbent contains multiple functional groups, making it potentially effective for adsorbing contaminants such as heavy metals or organic pollutants in crude oil remediation applications. The observed functional groups can facilitate interactions with a variety of pollutants, particularly through hydrogen bonding, van der Waals forces, and ion exchange mechanisms.

3.4 Brunauer-Emmett-Teller (BET)

Table 2 shows the Brunauer–Emmett–Teller (BET) data for the nano sorbent derived from rice husk.

In Table 2, nano rice husk sorbent has a reasonably high surface area and this indicates that it will exhibit improved adsorption capacity. The nano rice husk sorbent exhibits the following attributes;

1. High surface area: 680.5 m^2/g (BET) and 999.9 m^2/g (Single Point)
2. Microporous structure: t-Plot Micropore Area (20.4 m2/g) and Micropore Volume (0.13044 cm3/g)
3. Narrow pore size distribution: Average pore width (30.34 Å - 30.42 Å)
4. High pore volume: 0.6035 cm3/g (Single Point) and 0.52222 cm3/g (BJH Desorption)

3.5 Scanning Electron Micrograph of The Rice Husk Nano Sorbent

The image in Fig. 3 shows the scanning electron micrograph of the rice husk sorbent.

The SEM image shows an irregular, flaky structure, which is common for processed rice husk at the nanoscale. The fragmented appearance and the layered formation shows that the rice husk has been broken down either through mechanical grinding and chemical treatment. The roughness and the porosity visible in the image indicate a high surface area, making it suitable for adsorption and catalysis. The gaps and cracks visible in the image imply a porous structure. Rice husk, especially after chemical and thermal processing, can become highly porous. This porosity increases the adsorption capacity, which can be useful in water purification (e.g., removing dyes or heavy metals), air filtration, or as a support material for catalysts.

Rice husk (Fig. 3) is composed mainly of silica (SiO_2), along with organic compounds like lignin and cellulose. The SEM image, combined with techniques like Energy Dispersive X-ray Spectroscopy (EDX), was used to confirm the presence of silica, which often dominates the structure after thermal treatment. The nano-scale structure seen here is the result of extracting silica from the husk, by burning off the organic components (leaving nano-silica) or through other chemical treatments. Nano-silica derived from rice husk is used in reinforcing materials like polymers and concrete to improve their mechanical properties. The material also serves in biochar applications, helping with soil amendment and as a carbon-rich material for environmental remediation. Its high surface area and silica content make nano rice husk ideal for use in catalysts and energy storage materials (e.g., lithium-ion batteries, supercapacitors). Heating rice husk removes organic matter and results in the formation of nano-silica, which explains the highly structured, granular morphology in the SEM image above. Treated rice husk with acids or bases removed unwanted organic components and enhanced its silica content, leading to a more refined, nanoporous structure. Reduced rice husk to nanoscale mechanically created fragmented, layered particles similar to what's observed in the SEM image. The SEM image above of nano rice husk reveals a highly fragmented, porous, and irregular structure typical of rice husk after processing. This nano structure, with its high surface area and porous nature, is useful adsorption, catalysis, and composite reinforcement.

Table 2. Brunauer–Emmett–Teller (BET) data for nano sorbent derived from rice husk

Property	Value
BET Surface Area	680.5 m^2/g
Langmuir Surface Area	52.24 m^2/g
Micropore Area	20.4 m^2/g
Pore Volume	0.6035 cm^3/g
Average Pore Width	30.34 Å

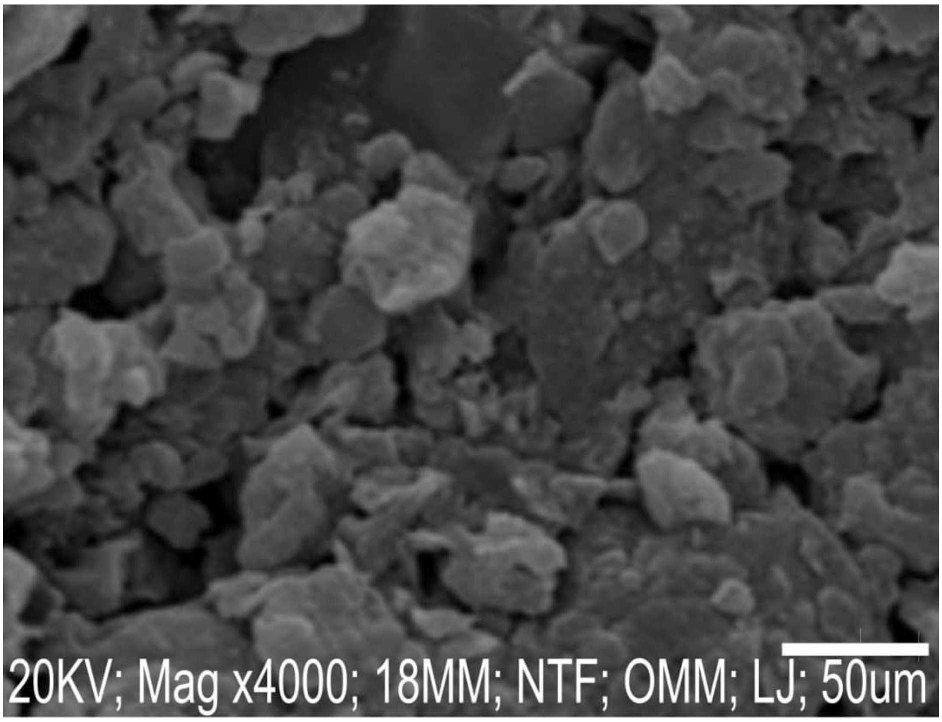


Fig. 3. Scanning Electron Micrograph (SEM) of the rice husk sorbent after using activating agent

3.6 Transmission Electron Micrograph of The Rice Husk Nano Sorbent

The transmission electron microscopy (TEM) image above shows the morphology of nano-sorbent material, derived from rice husk (Fig. 4). The TEM image shows spherical nanoparticles with varying diameters. The sizes indicated in the image are 9.22 nm and 12.54 nm, suggesting that the sorbent particles are within the nanoscale range. The size distribution of these particles is important because it affects the surface area available for oil adsorption. The small particle sizes and large distribution of dark spots (likely representing nanoparticles) imply a high surface area, which is a desirable trait for sorbents. A higher surface area allows for more interaction points for oil molecules, enhancing the material's ability to remove oils from water. The arrangement of the particles shows a porous structure. The pore structure is vital for the adsorption of larger oil molecules, like those found in AGO and PMS, trapping them effectively in the nanomaterial. The relatively uniform size and spherical shape of the particles can contribute to consistent adsorption performance. A uniform particle distribution ensures that the sorbent material works effectively over a wide area when dispersed in contaminated water.

Rice husk-derived nano-sorbents contain silica (SiO_2) and carbon-based materials. The use of rice husk for oil removal takes advantage of its silica content, which enhances its hydrophobic properties, making it effective in repelling water and attracting oil molecules. The TEM image reveals that the rice husk-derived nano-sorbent possesses key characteristics like nanoscale particle size, high surface area, and probable porosity, which are crucial for efficiently removing hydrophobic oils such as AGO and PMS from water. These properties made this material highly effective for environmental cleanup applications.

3.7 Energy Dispersive X-ray (EDX) Spectrum of The Rice Husk Nano Sorbent

The image in Fig. 5 below shows the spectrum obtained from the EDX spectroscopic analysis of the rice husk nano sorbent.

The spectrum above shows the highest peak in the spectrum corresponds to silicon, which dominates the sample with a weight percentage of 75.56% and atomic percentage of 60.20%. Silicon's presence is vital in improving the surface area and adsorption capacity for removing oils from water. Oxygen is the second most abundant element (10.37% weight, 25.25% atomic), indicating the presence of oxides and hydroxyl groups, which can enhance the hydrophilic nature of the material, making it more effective in interacting with water and the oils. Aluminum (Al) present at 5.40% by weight (4.20% atomic), aluminum originate from natural mineral components within the husk that enhance the mechanical properties and

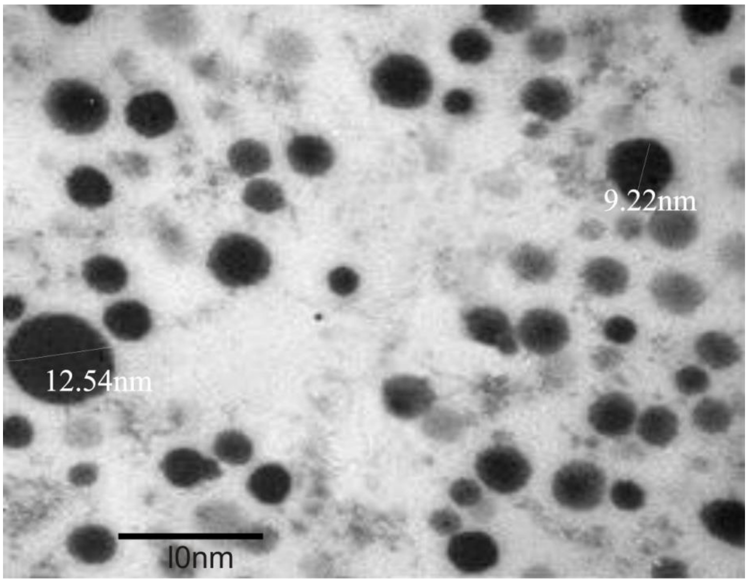
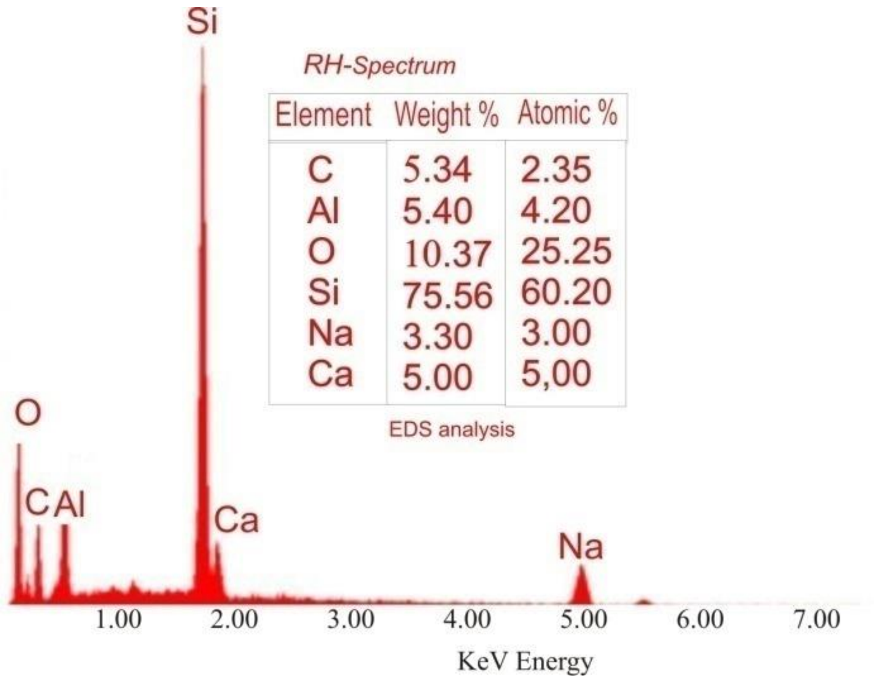


Fig. 4. Transmission Electron Microscopy (TEM) of the rice husk sorbent after using activating agent



Element	Weight %	Atomic %
C	5.34	2.35
Al	5.40	4.20
O	10.37	25.25
Si	75.56	60.20
Na	3.30	3.00
Ca	5.00	5,00

Fig. 5. EDX (Energy Dispersive X-ray Spectroscopy) of rice husk nano sorbent

chemical stability of the nano-structured material. Aluminum oxides are also known for their adsorptive properties. Carbon (C) at 5.34% by weight (2.35% atomic), the presence of carbon suggests that the organic matrix of the rice husk remains in the nano-structure. Carbon also contribute to the hydrophobic interactions with oil, which is essential in oil sorption.

Calcium (Ca) present at 5.00% by weight and 5.00% at atomic; Sodium (Na) present at 3.30% by weight (3.00% atomic); sodium indicates residual salts from the preparation process. Sodium is known to affect the ionic strength of the solution, potentially influencing the adsorption efficiency.

3.8 X-Ray Diffraction (Xrd) Spectrum for The Rice Husk Nano Sorbent

Fig. 6 shows the XRD spectrum for the rice husk nano structured sorbent.

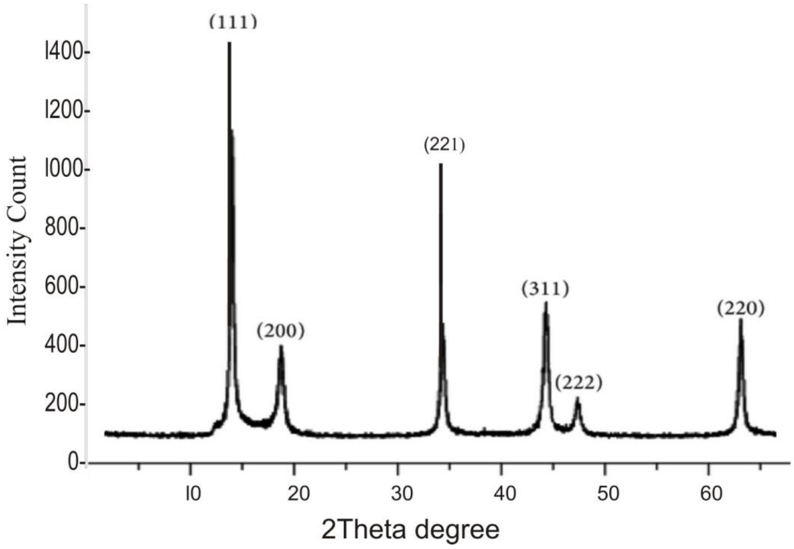


Fig. 6. XRD (X-ray Diffraction) spectrum for rice husk nano sorbent

From the XRD spectrum in Fig. 6, the presence of sharp peaks at 20°, 30°, 50°, and 60° shows a crystalline structure, with the most prominent peaks assigned to different planes, namely, 111 and 221 at higher peak, 200, 311, 222, 220 at lower peak. The XRD spectrum showed that the rice husk nano-sorbent is highly crystalline, which will contribute to its high efficiency in removing oil contaminants from water. The sorbent's structure allows it to trap oil molecules in its porous framework, aided by van der Waals forces and hydrophobic interactions.

4. CONCLUSION

This study has successfully developed and characterized nanostructured sorbent from an agricultural waste (rice husks). Environmentally friendly and cost-effective methods were used. The thermal and chemical treatment processes facilitated the transformation of this waste material into highly porous structures with a significant surface area, ideal for oil adsorption. The data from the characterization studies revealed that the nanostructured sorbent exhibited nano-scale structures with enhanced surface properties, including high porosity and functional groups (such as hydroxyl and carboxyl groups) that contribute to oil adsorption. The prepared adsorbent may be applied for the remediation of crude oil-contaminated water.

5. RECOMMENDATIONS FOR FURTHER STUDIES

This study makes the following recommendations;

1. There is need for optimization of the sorbent production processes, particularly in terms of the thermal and chemical treatment conditions, to enhance the sorbents' structural and adsorption properties.

2. Research should be conducted to explore the scalability of the production process of the sorbent for large-scale applications, particularly in industrial oil spill clean-ups.

3. Field application studies should be carried out in real-world oil spill scenarios to evaluate the practical applicability of this sorbent, considering different environmental conditions such as temperature, pH, and salinity.

4. Other types of agricultural wastes e.g. coconut shells, palm kernel husks, etc. should be explored as potential sources of nanostructured sorbents for crude oil remediation.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc) and text-to-image generators has been used during writing or editing of this manuscript.

COMPETING INTERESTS

Authors have declared that they have no known competing financial interests or non-financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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