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Sustainable Synthesis of Carbon Nanotubes from Potatoes Peel for Oil Spill Remediation

Afolabi Adenike Mistura [1]; Mansur Yahaya Ibrahim [1]; and Jamilu Usman [2]

[1]Department of Pure and Industrial Chemistry, Sokoto State University, Sokoto. [2]Interdisciplinary Research Center for Membranes & Water Security, King Fahd University of Petroleum and Minerals, Dhahran 31261, Saudi Arabia.

Corresponding author: ymansur64@gmail.com

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Abstract

The environmental challenge posed by oil spills necessitates the development of sustainable remediation strategies. Traditional methods often rely on hazardous chemicals that threaten ecosystems and human health, underscoring the need for alternative solutions. This research aimed to synthesize carbon nanotubes (CNTs) from potato peels using a controlled pyrolysis process and evaluate their potential as eco-friendly adsorbents for crude oil remediation. Potato peels, were carbonized at 300°C. Results revealed that the CNTs exhibited a maximum adsorption capacity of 9538.17 mg/g at 6.0 g dosage, achieving a removal efficiency of 97.997%. FTIR analysis identified functional groups such as hydroxyl (-OH) and carboxyl (-COOH) at the range of 15000 – 25000 cm^{-1} that enhance adsorption, Potato peels' UV-Vis spectra reveal a π-π* transition in carbon-based materials, and a minimal impurity after 220 nm, indicating high purity of CNTs synthesis and SEM confirmed the formation of a porous CNT network. The CNTs performed optimally at pH 6.8, with 95.14% removal efficiency, and at lower temperatures, with 96.77% removal at 20°C. Optimal adsorption was observed at a contact time of 90 minutes, achieving a crude oil removal efficiency of 95.997%. Analysis using Langmuir, Freundlich and Temkin isotherm models revealed high R^2 values across all the three models suggesting complex interactions where both monolayer and multilayer formations occur alongside significant intermolecular forces affecting binding energies which demonstrated the potentials of potato peel-derived CNTs as sustainable, cost-effective adsorbents, offering an eco-friendly approach to oil spill remediation Future research should be focused on optimizing the synthesis process, assess long-term environmental impacts, and conduct cost-benefit analyses.

Keywords: Potatoes, Crude Oil, Carbon Nanotubes, Pyrolysis, Adsorption.

Introduction

The increasing prevalence of crude oil spills poses significant environmental challenges to our environment which necessitated the need for developing effective remediation strategies. Traditional methods for cleaning up oil spills often involve the use of chemical dispersants or physical barriers, which can introduce additional pollutants into marine ecosystems. [1-5] In recent years, there has been a growing interest in sustainable and eco-friendly materials for environmental cleanup applications. One promising avenue is the

utilization of carbon nanotubes (CNTs), renowned for their exceptional mechanical strength, high surface area, and unique adsorption properties. [6-9]
Potato peels are an abundant agricultural byproduct that is often discarded, leading to environmental waste. By converting this organic waste into valuable nanomaterials, we not only mitigate waste but also harness the potential of CNTs in environmental remediation.
This study explores the sustainable synthesis of carbon nanotubes from potato peel waste as a novel approach to address the pressing issue of crude oil contamination as mentioned above. The synthesis process involves pyrolysis, a thermochemical decomposition method that transforms organic material into carbon-rich products at elevated temperatures in an inert atmosphere. This method not only facilitates the production of CNTs but also aligns with principles of green chemistry by minimizing harmful emissions and utilizing renewable resources. The resultant CNTs can be employed as adsorbents to effectively capture and remove hydrocarbons from contaminated water bodies. [10-12]
The aim of this research is to synthesized CNTs from potatoes peels through pyrolysis process, characterization of the produced carbon nanotubes, and their efficacy in cleaning up crude oil spills. By integrating waste valorization with advanced nanotechnology, this research contributes to sustainable practices in environmental management while addressing one of the most critical challenges faced by our planet today.

Methodology

Sample Collection

Potato peels were collected at Sokoto Central Market Sokoto State Nigeria. And crude oil was obtained from Kaduna refinery and Petrochemicals Company, Kaduna State, Nigeria.

Sample Preparation

After collection, the peels were thoroughly washed with clean water to remove any form of impurities and were dried under shade for two weeks in the Laboratory. Collected sample is shown in Figure 1. After drying, the potato peels were ponded using Mortar and Pestle and were sieved into a container to have a very fine powdered sample for synthesis.

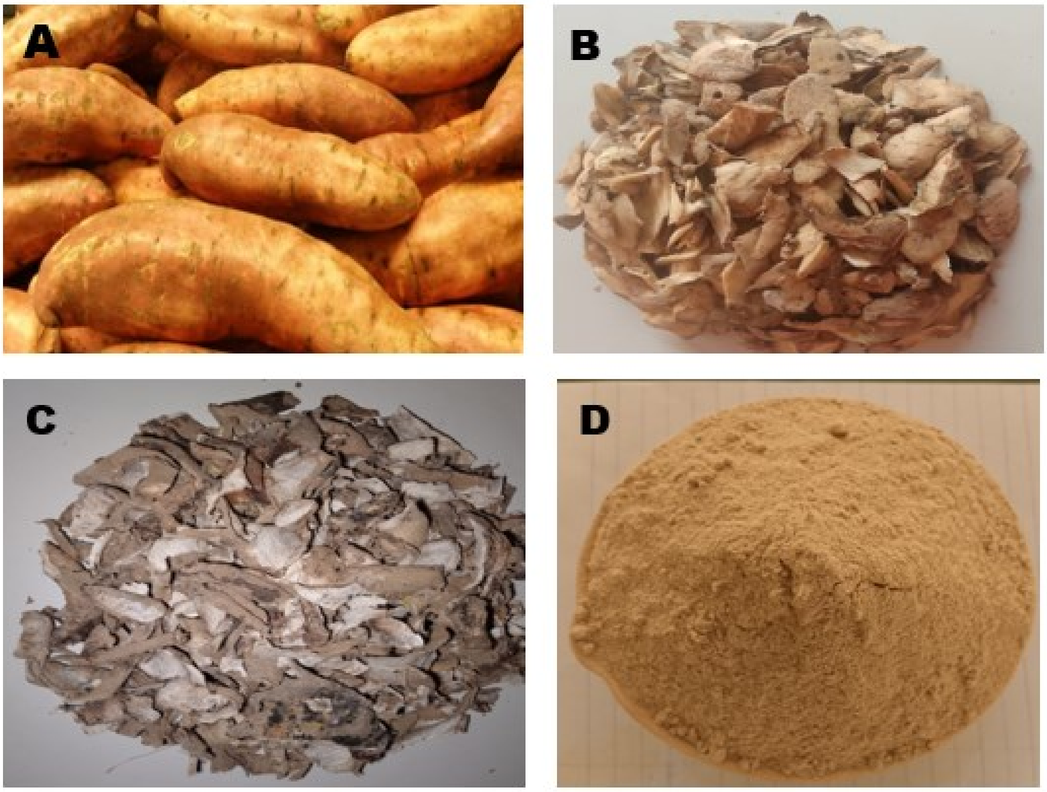

Figure 1: Potatoes Sample: A = Potatoes, B = Fresh Potatoes peel C = Dried Potatoes peel and D= Powdered potatoes peel

Carbonization

900 g of the sample were weighed into crucible and inserted into the muffle furnace, the temperature was set to 300 ^{O}C and carbonized for 2 hours. The samples were then weighed and pounded with Mortar and Pestle to powdered form and sieved to have a very fine powder sample. Figure 2. shows the carbonized samples of potatoes peels

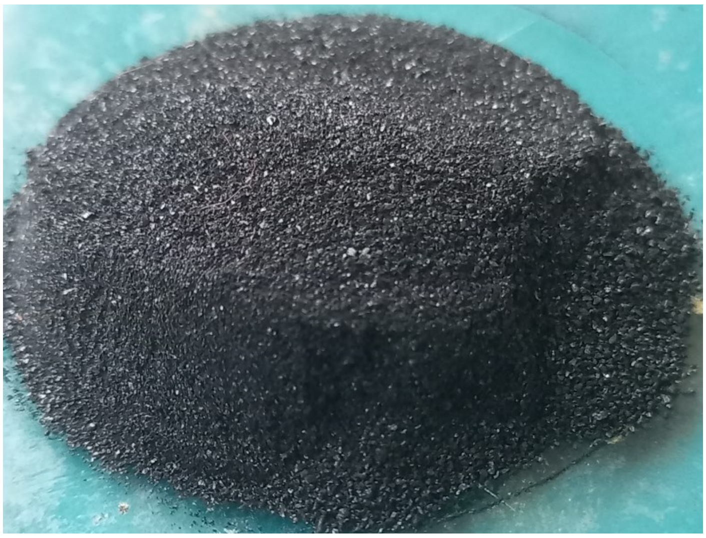

Figure 2: Carbonized samples of Potatoes peels

Synthesis of Carbon Nanotubes from Prepared Samples

The carbonized sample were put into 1000 ml (1L) conical flask, and 60 cl of distilled water was added and shake with hand to mix up and was then agitated at 150 rpm for 2 hours. After mixing well, it was filtered with Whatman filter paper and the residue was dried in a drying oven for 2 days till they dried off and was taken for analysis. Then 30 cl of filtered water was evaporated on the heating mantle using a 500 ml beaker and the CNTs were obtained.

Preparation of Simulated Oil Spill

9.72g of crude oil were added to 500 ml of brine water (0.018 wt% NaCl Conc) to recreate a typical modest oil spill scenario of 9720 mg/L of initial concentration. To create an emulsified solution the immiscible solution was physically shaken for 15 minutes using an agitator as shown in the Figure 3 below.

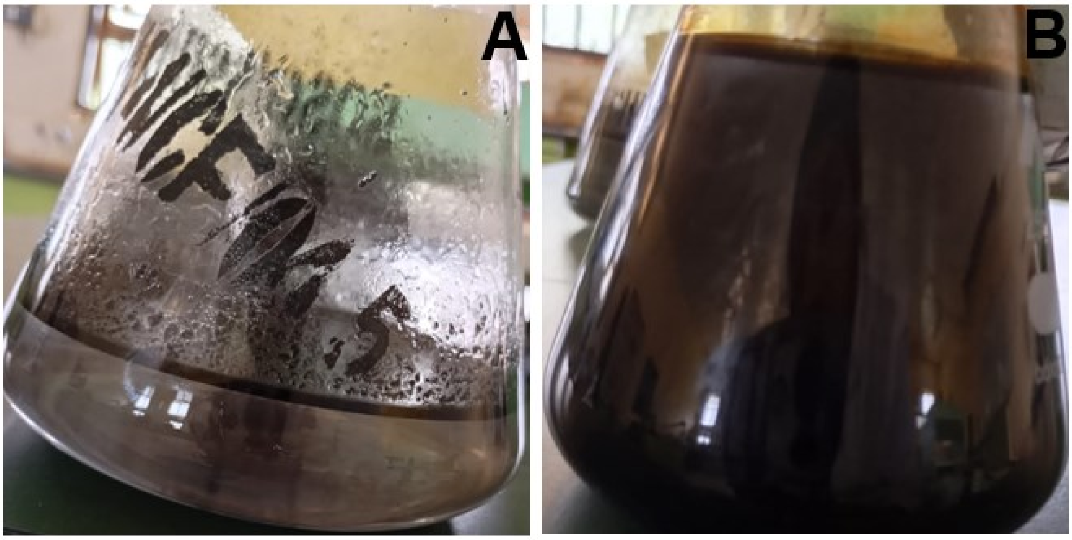

Figure 3: Simulated crude oil spill for batch adsorption experiment: (A=before agitation and B= after agitation)

Batch and Sorption Studies

Effect of Adsorbent Dosage on Adsorption of Crude Oil onto the Synthesized CNTs

A 100 mL sample of a simulated oil spill with an initial crude oil concentration of 9720 mg/L was measured into 500 mL beakers. Different dosages of synthesized CNTs were tested as adsorbents, starting with 2.0 g. Each mixture was agitated in a speed-controlled multipurpose oscillator at 250 rpm for 5 minutes to allow the adsorption process to reach equilibrium.

After agitation, the CNTs were filtered using Whatman filter paper and a suction funnel. The residual oil-in-water solution was homogenized with a magnetic stirrer for 15 minutes to form an emulsified solution. The equilibrium concentration of the emulsified solution was then measured using a UV-Vis spectrophotometer at a wavelength of 400 nm. This process was repeated for various CNT dosages: 2.0 g, 3.0 g, 4.0 g, 5.0 g, and 6.0 g.

For each adsorbent dosage, the amount of crude oil adsorbed per unit mass of CNTs at equilibrium, qa (mg/g), and the percentage of crude oil removal were calculated using Equations 1 and 2:

$$qa = \frac{(Co - Ca)\ x\ V}{M} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

$$\%\ Removal\ of\ Crude\ Oil\ = \frac{C_O - C_e}{C_O} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots . (2)$$

Where; Co = Initial concentration of solution (mg/L), Ca = Equilibrium concentration at adsorbent dosages (mg/L), V = Volume of the solution (mL) and M = Mass of Adsorbent used (g).

Effect of Contact Time on the Adsorption of Crude Oil onto the Synthesized CNTs

A 100 mL sample of a simulated oil spill with an initial crude oil concentration of 9720 mg/L was measured into a 500 mL beaker. To this, 2.0 g of synthesized CNTs was added as the adsorbent. The mixture was agitated in a speed-controlled multipurpose oscillator at 200 rpm for 15 minutes to allow adsorption to reach equilibrium.

After agitation, the CNTs were filtered using Whatman filter paper and a suction funnel. The residual oil-in-water solution was homogenized with a magnetic stirrer for 15 minutes to form an emulsified solution. The equilibrium concentration of the emulsified solution was determined using a UV-Vis spectrophotometer at a wavelength of 400 nm.

This process was repeated for different contact times (30 minutes, 45 minutes, 60 minutes, 75 minutes, and 90 minutes) and for different dosages of CNTs (2.0 g, 3.0 g, 4.0 g, 5.0 g, and 6.0 g), for CNTs derived from potato peels.

For each contact time and adsorbent dosage, the amount of crude oil adsorbed per unit mass of CNTs at equilibrium, qa (mg/g), and the percentage removal of crude oil were calculated using Equations 3 and 4:

$$qt = \frac{(Co - Ct)\ x\ V}{M} \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (3)$$

$$\%\ removal\ of\ crude\ oil = \frac{(Co - Ce)}{Co} x\ 100 \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (4)$$

Where; Co = Initial concentration of solution (mg/L), Ct = Concentration of solution at time T, (mg/L), V = Volume of the solution (mL) and M = Mass of Adsorbent used (g).

Effect of pH on the Adsorption of Crude Oil onto the Synthesized CNTs

A 100 mL sample of a simulated oil spill with an initial crude oil concentration of 9720 mg/L was placed in a 500 mL beaker. To this, 2.0 g of synthesized CNTs, derived from potato peels, was added as the adsorbent. The mixture was agitated at 200 rpm for 15 minutes to allow adsorption to occur. After agitation, the CNTs were filtered using Whatman filter paper, and the residual oil-in-water solution was homogenized. The equilibrium concentration of the emulsified solution was determined using a UV-Vis spectrophotometer at a wavelength of 400 nm.

This process was repeated for different pH levels: acidic conditions (pH 1.2, 0.8, and 0.6), achieved by adding 10 mL, 20 mL, and 30 mL of acid, respectively, and basic conditions (pH 10.50, 10.20, and 10.00), achieved by adding 10 mL, 20 mL, and 30 mL of base, respectively. For each pH level, the adsorption capacity at equilibrium, qa (mg/g), and the percentage removal of crude oil were calculated using Equations 3 and 4 above:

Effect of Temperature on the Adsorption of Crude Oil onto the Synthesized CNTs

A 100 mL sample of a simulated oil spill with an initial crude oil concentration of 9720 mg/L was measured into a 500 mL beaker. To this, 2.0 g of synthesized CNTs derived from potato peels was added as the adsorbent. The mixture was agitated in a speed-controlled multipurpose oscillator at 200 rpm for 15 minutes to allow the adsorption process to reach equilibrium.

After agitation, the CNTs were filtered using Whatman filter paper and a suction funnel. The residual oil-in-water solution was homogenized with a magnetic stirrer for 15 minutes to form an emulsified solution. The equilibrium concentration of the emulsified solution was measured using a UV-Vis spectrophotometer at a wavelength of 400 nm.

This process was repeated at various temperatures: 100°C, 75°C, 50°C, 25°C, and 10°C, using different adsorbent dosages (2.0 g, 3.0 g, 4.0 g, 5.0 g, and 6.0 g), separately for CNTs synthesized from potato peels.

Reuse of synthesized CNTs Adsorbent for continues Crude Oil Clean Up

The reusability of CNTs synthesized from potato peels was investigated to assess their effectiveness in repeated cycles of crude oil adsorption. After the initial adsorption process, 100 mL of simulated oil spill solution with an initial concentration of 9720 mg/L was treated with 2.0 g of CNTs. The mixture was agitated at 200 rpm for 15 minutes to ensure equilibrium. After the adsorption, the CNTs were separated from the solution using Whatman filter paper and a suction funnel. To regenerate the spent CNTs, they were rinsed with an ethanol to effectively remove the adsorbed oil. Following this, the CNTs were thoroughly rinsed with distilled water and then dried in an oven at 100°C to restore their adsorption capacity. This regeneration process was repeated for subsequent adsorption cycles to evaluate the efficiency of the CNTs in crude oil removal after each reuse.

Chemical Characterization

Chemical characterization techniques are crucial for determining the structure, composition, and physical properties of the synthesized CNTs from potato peels. These techniques provide insight into the surface functional groups, morphology, and crystallinity of the nanomaterials. The key methods used in this research are:

UV-Visible Spectroscopy (UV-Vis)

UV-Vis spectroscopy is used to study the optical properties of CNTs by measuring the absorbance of ultraviolet and visible light. This method helps determine the electronic transitions in the CNTs, providing information on their band gap and the presence of π-electrons in the conjugated carbon system. The CNTs synthesized from potato peels were dispersed in a suitable solvent, such as ethanol or distilled water. The dispersion is then placed in a quartz cuvette, which was inserted into the UV-Vis spectrophotometer (Shimadzu UV-2600, Shimadzu Corporation, Japan). The spectrometer records the absorbance of light at different wavelengths, typically ranging from 200 to 800 nm. This data is used to analyze the electronic properties of the CNTs, helping to confirm their structure and the presence of any defects or impurities. [13]

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is used to identify the functional groups present in the CNTs by measuring the infrared absorption or transmission spectrum. This method is based on the fact that different chemical bonds absorb light at specific frequencies, allowing for the identification of the functional groups present on the surface of the CNTs. A small sample of the synthesized CNTs is mixed with potassium bromide (KBr) powder and pressed into a pellet. The pellet is then placed in the FTIR spectrometer (PerkinElmer Spectrum Two, PerkinElmer, Inc., US), which emits infrared radiation through the sample. The absorbance of specific wavelengths is recorded to identify functional groups such as hydroxyl (-OH), carboxyl (-COOH), and carbonyl (C=O) that may be present on the surface of the CNTs. [14]

Scanning Electron Microscopy (SEM)

SEM is employed to observe the surface morphology and structure of the CNTs. It works by scanning the surface of a sample with a focused beam of electrons. When these electrons interact with the atoms of the material, they produce signals that can be used to generate high-resolution images, revealing details about the sample's topology. A small amount of the synthesized CNTs was mounted on a conductive tape and coated with a thin layer of gold to prevent charging under the electron beam. The sample was placed inside the SEM chamber (JEOL JSM-IT500, JEOL Ltd., Japan), and an electron beam was directed onto the surface of the CNTs. The resulting secondary electrons were detected and processed to produce images of the nanotubes' structure, size, and distribution. SEM allows for the examination of the nanoscale structure of the CNTs, ensuring that the synthesis process results in the desired nanomaterials. [15]

X-Ray Diffraction (XRD)

XRD is a non-destructive technique used to determine the crystallinity and phase composition of CNTs. The method relies on the diffraction of X-rays by the crystalline planes of the material. The angle and intensity of the diffracted X-rays provide information about the atomic structure of the sample. powdered sample of the synthesized CNTs is placed on a sample holder and subjected to X-ray radiation (PANalytical X'Pert Pro, Malvern Panalytical, Netherlands). As the X-rays interact with the crystalline lattice of the CNTs, they produce a diffraction pattern that is recorded by the detector. The resulting pattern is analyzed to determine the crystalline structure, phase composition, and interlayer spacing of the carbon nanotubes. XRD also helps in assessing the purity of the synthesized CNTs, as any impurities or amorphous carbon phases can be detected. [16]

Results and Discussions

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Figure 3, reveals key functional groups that are significant for their application as carbon nanotube precursors in crude oil spill cleanup. The spectrum shows two major peaks at 3392.4 cm^{-1} and 1638.3 cm^{-1}, representing O-H stretching vibrations and C=O stretching, respectively. These groups play a vital role in adsorption, enhancing surface interaction with oil molecules during the remediation process.

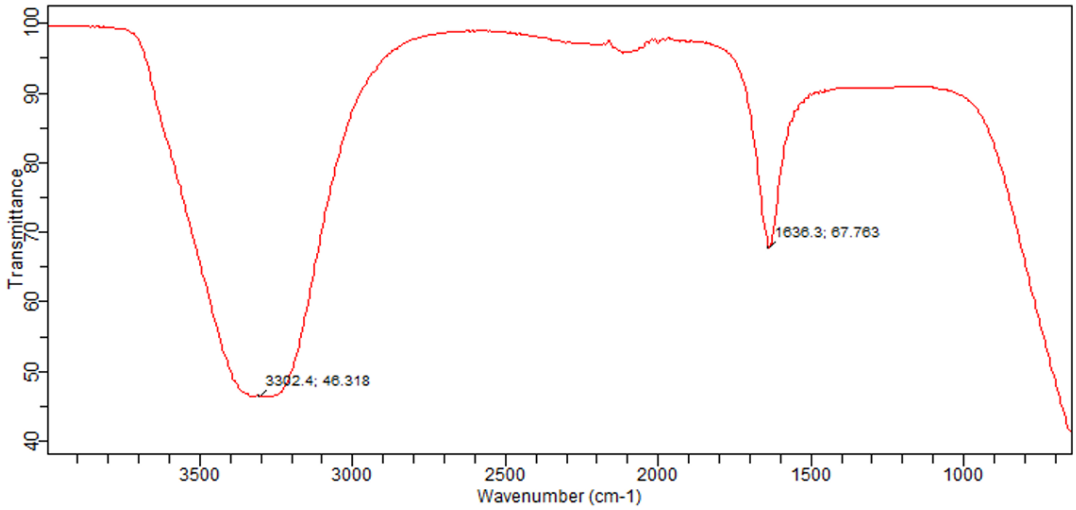

Figure 3: FTIR Spectra of Free Potatoes Peels (Control)

The broad absorption band at 3392.4 cm^{-1} is attributed to hydroxyl (O-H) groups, typically found in cellulose, hemicellulose, and lignin, components of potato peels. These groups increase hydrophilicity and surface interaction, which enhances oil absorption efficiency in biosorbents used for oil spill remediation. The presence of hydroxyl groups indicates potential adsorption sites that bind oil molecules through hydrogen bonding and Van der Waals forces. [17]

The peak at 1638.3 cm^{-1} corresponds to the C=O stretching of carboxyl groups, which likely originate from pectin and organic acids present in the biomass. Similar findings have been reported in studies using agricultural wastes for oil spill cleanup. The carboxyl groups improve the biosorbent's affinity for hydrophobic crude oil compounds, aiding in efficient removal from water. These groups can form ester linkages with crude oil, further supporting the adsorption process. [18]

The transmittance values of 46.318% and 67.763% suggest moderate intensity for these functional groups, which aligns with studies showing that moderate functional group intensities contribute to substantial adsorption capacities in biosorbents. Such a composition is essential for enhancing surface area and porosity when these materials are carbonized into nanotubes. [19]

Interestingly, the absence of significant peaks in the fingerprint region (below 1500 cm^{-1}) indicates a lack of complex inorganic impurities. This purity is advantageous as impurities often diminish the material's effectiveness in oil adsorption, as noted in previous studies. [20] The functional groups observed in the FTIR spectrum of potato peels are similar to those found in other biomass materials, which are often used for carbon nanotube synthesis.

The FTIR analysis suggests that control potato peels exhibit the necessary functional groups, such as hydroxyl and carboxyl, which are crucial for crude oil adsorption applications. When processed into carbon nanotubes, potato peels are expected to demonstrate high efficiency in oil spill remediation, similar to other agricultural waste-derived materials. These findings emphasize the feasibility of using potato peels as a sustainable and cost-effective solution for oil spill cleanup. [21-22]

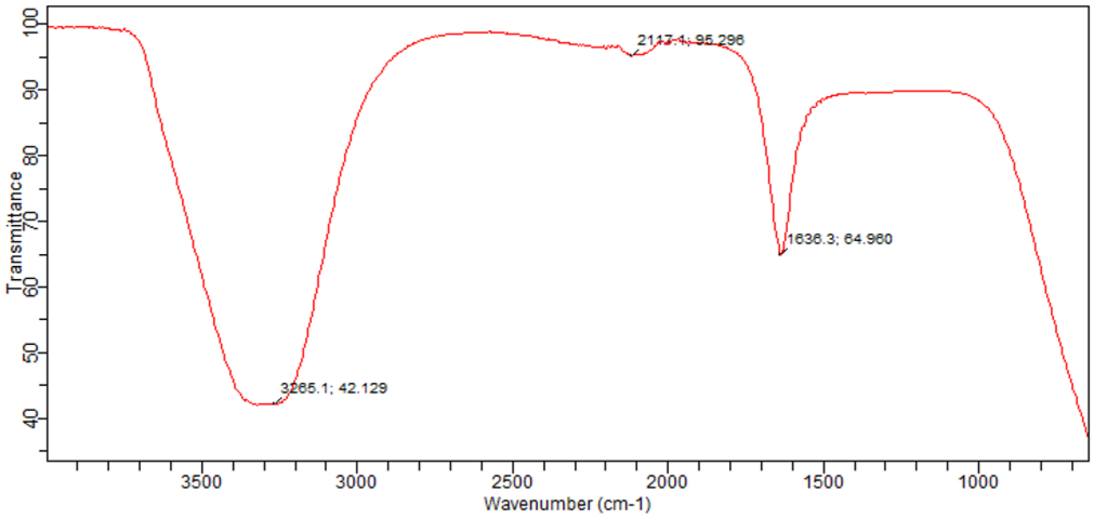

Figure 4: FTIR Analysis of Potatoes Peels Synthesized CNTs

Figure 4 reveals the FTIR analysis results for Potatoes Peels Synthesized CNTs. The spectrum displays prominent peaks at 3265.1 cm^{-1}, 2117.1 cm^{-1}, and 1636.3 cm^{-1}, corresponding to O-H, C≡C, and C=C stretching vibrations, respectively. These functional groups enhance the surface characteristics of the synthesized CNTs, improving their potential for oil adsorption during remediation. [23]

The broad peak at 3265.1 cm^{-1}, with a transmittance of 42.129%, indicates the presence of hydroxyl (O-H) groups, which are typical in biomass-derived materials. These hydroxyl groups enhance the hydrophilicity of the CNTs, promoting better interaction with water molecules during oil-water separation. However, the retention of these oxygen-containing functionalities in the CNT structure is beneficial as it ensures a balance between hydrophilic and hydrophobic interactions, essential for efficient oil spill cleanup. [24]

At 2117.1 cm^{-1}, with a high transmittance of 95.298%, the spectrum shows the presence of C≡C stretching vibrations, which are characteristic of alkyne groups and indicate sp-hybridized carbon chains. These peak highlights the graphitic nature of the synthesized CNTs, which is essential for adsorbing non-polar crude oil molecules. The formation of these carbon-carbon triple bonds demonstrates successful carbonization, which significantly enhances the hydrophobicity of the material, making it more effective for oil spill remediation. [25]

The peak at 1636.3 cm^{-1}, with a transmittance of 64.980%, corresponds to C=C stretching, a functional group typical of alkene structures. These unsaturated carbon chains enhance the hydrophobicity of the CNTs, increasing their interaction with non-polar crude oil molecules. The presence of these functional groups ensures that the CNTs have both high oil adsorption efficiency and structural stability in aquatic environments, making them ideal for large-scale oil spill cleanup efforts [26]. This aligns with previous studies, which have demonstrated that carbon nanomaterials with unsaturated carbon structures are highly efficient in adsorbing hydrocarbons from contaminated water. [27]

Thus, the FTIR analysis of potato peels synthesized CNTs suggests that these materials possess a unique combination of functional groups, making them promising candidates for sustainable crude oil spill remediation. The hydroxyl, alkyne, and alkene groups present in the CNTs provide a dual functionality—hydrophilicity for water interaction and hydrophobicity for oil adsorption—which enhances the overall efficiency of the material in oil spill cleanup. [28]

Ultraviolet-Visible Spectroscopy (UV-Vis) Analysis Results

UV-Vis is an analytical technique that measures the absorption of ultraviolet and visible light by a material. It provides information about the electronic transitions and energy gaps in materials, commonly used to study optical properties, chemical bonding, and concentration of compounds in solutions.

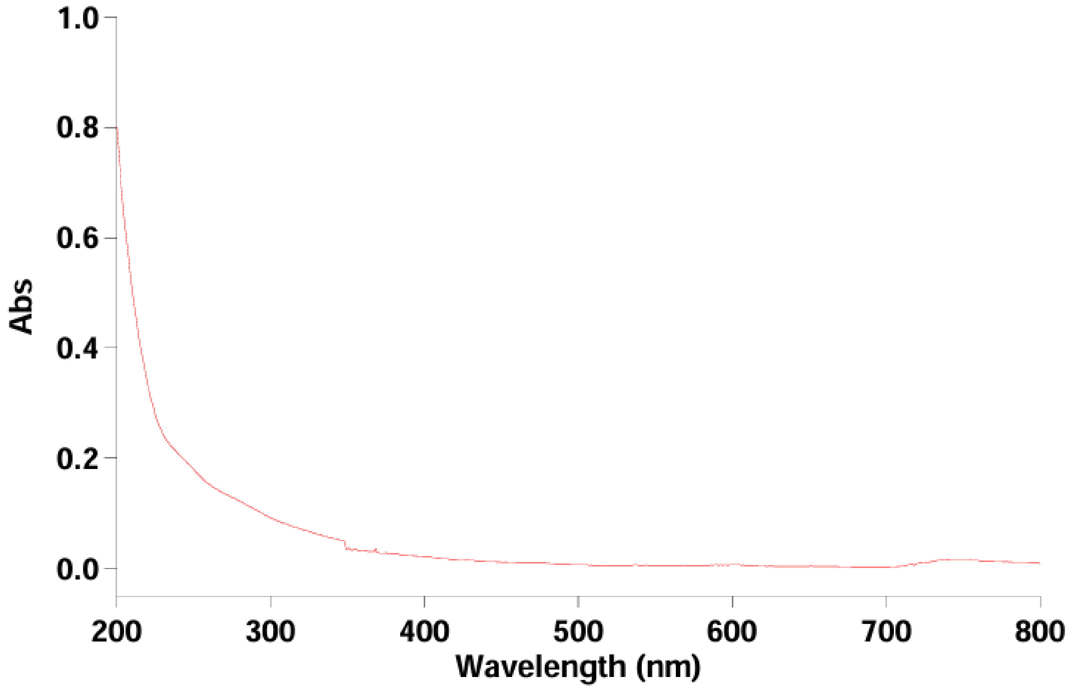

Figure 5: Ultraviolet Visible Spectra of Control Potatoes Peels

Figure 5, displayed the UV-Vis Spectra of Control Potatoes Peels which demonstrates significant absorbance in the UV region between 200-300 nm, with a rapid decline beyond 300 nm. The high absorbance in the ultraviolet region can be attributed to the presence of organic compounds and lignin in the potato peels, which contain chromophore groups capable of absorbing UV radiation. This behavior is typical for biomass materials, which have been widely reported to exhibit peaks in the lower wavelength region due to π-π* transitions within aromatic compounds. [29]

The rapid drop in absorbance beyond 300 nm is indicative of the absence of metallic impurities or nanostructured materials like CNTs, which typically exhibit broad absorption across the visible to near-infrared (NIR) region. In comparison to the UV-Visible spectra of synthesized CNTs from the same biomass, this control spectra highlights the transformation that occurs during the carbonization and CNTs synthesis process. The conversion of lignocellulosic materials in potato peels to CNTs would significantly enhance the absorbance beyond 300 nm due to the increased π-electron conjugation and the formation of sp2 hybridized carbon structures, as previously reported in other studies. [30] Moreover, the control spectrum serves as a baseline to demonstrate the effectiveness of the carbonization process for CNT synthesis. The absence of absorbance in the visible and NIR regions suggests that untreated potato peels lack the conductive and optical properties required for crude oil spill remediation. The synthesized CNTs, on the other hand, possess these enhanced properties, as evidenced by shifts in absorption peaks and increased absorbance across a broader spectrum, which are crucial for hydrophobicity and sorption capacity in oil spill cleanup [31]. This aligns with research showing that the introduction of nanomaterials significantly enhances the surface area and adsorption capabilities of materials used for environmental remediation. [32]

Thus, the UV-Visible spectra of the control potato peels serve as an essential reference for assessing the transformation and effectiveness of the synthesized carbon nanotubes, particularly in the context of crude oil spill remediation. The spectroscopic changes post-synthesis will confirm the structural and functional enhancements provided by CNTs, further supporting their role in sustainable environmental solutions. [33]

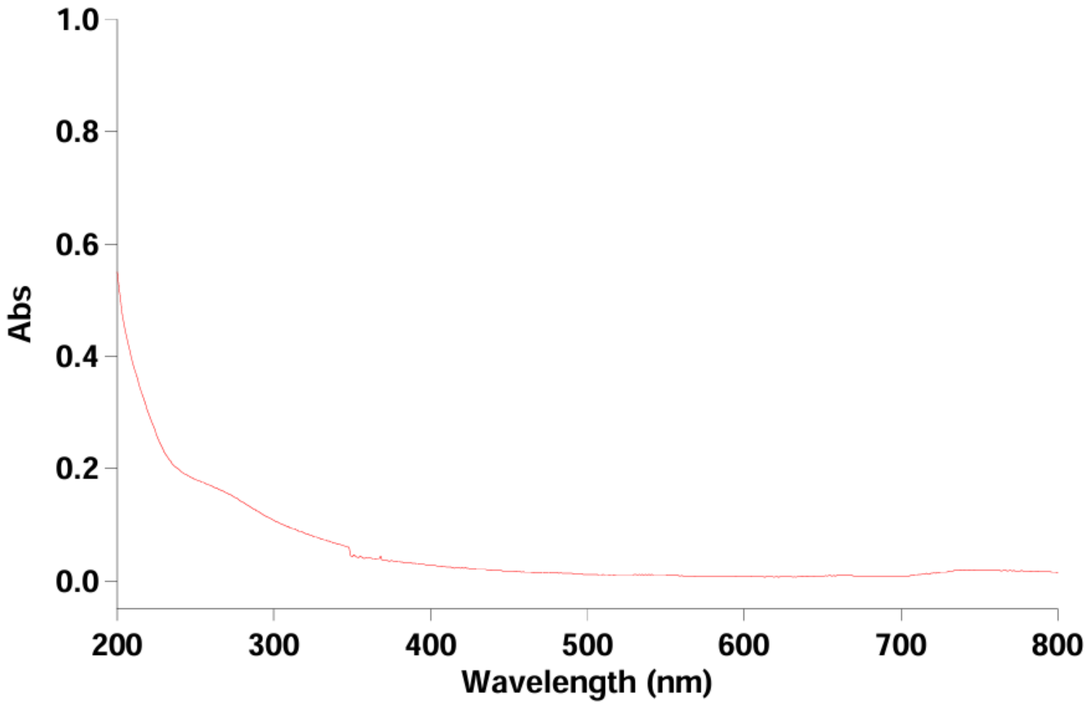

Figure 6: Ultraviolet Visible Spectra of Potatoes Peels Synthesized CNTs

In Figure 6, the Ultraviolet Visible spectra of the synthesized CNTs from potato peels show a notable absorption peak around 210-220 nm, which is characteristic of π-π* transitions in carbon-based materials. This peak suggests the presence of aromatic systems in the synthesized CNTs, consistent with sp^2 hybridized carbon structures. The steep decline in absorbance after 220 nm indicates minimal impurity, suggesting that the synthesis process was effective in producing CNTs with high purity. [34]

The UV-Vis spectra observed here align with those reported in similar studies, where biomass-derived CNTs exhibited strong absorption in the ultraviolet region due to their conjugated π systems. Studies have shown that biomass waste such as potato peels can serve as an effective precursor for producing CNTs with desirable structural and optical properties, as demonstrated by the characteristic absorption peaks in the UV range [35-36].

This reinforces the viability of using agricultural waste as a sustainable source for nanomaterials, reducing the environmental burden associated with crude oil spill clean-up. [37]

The relatively low absorbance beyond 400 nm further confirms that the synthesized CNTs from potato peels do not exhibit significant absorption in the visible light range. This result is consistent with previous research on the optical properties of carbon-based nanomaterials synthesized from renewable sources, which typically display strong ultraviolet absorption due to their unique electronic structures [38-39]. The absence of strong absorption in the visible region suggests the formation of well-structured CNTs without excessive defects or impurities, which is crucial for applications such as oil spill remediation where purity and efficiency of sorbents are key. [40]

Moreover, the use of potato peels as a precursor for CNT synthesis contributes to the sustainability of the process. The conversion of this waste into valuable nanomaterials reduces waste accumulation while providing an eco-friendly alternative for nanotechnology applications. This aligns with recent research that emphasizes the importance of green synthesis approaches in CNT production for environmental applications, including oil spill clean-up. [41]

The UV-Vis spectra of CNTs synthesized from potato peels confirm the presence of well-formed carbon nanostructures with strong ultraviolet absorption and minimal impurities. These properties make them highly suitable for use in environmental remediation, particularly in crude oil spill clean-up, where the high surface area and adsorption capabilities of CNTs are crucial for efficient pollutant removal. [42-43]

Scanning Electron Microscopy (SEM) Results

SEM is a powerful imaging tool that provides high-resolution, three-dimensional images of the surface morphology and texture of materials. By scanning a focused electron beam across a sample, SEM allows researchers to study the material's microstructure, particle size, and surface features, crucial for evaluating its mechanical and physical properties.

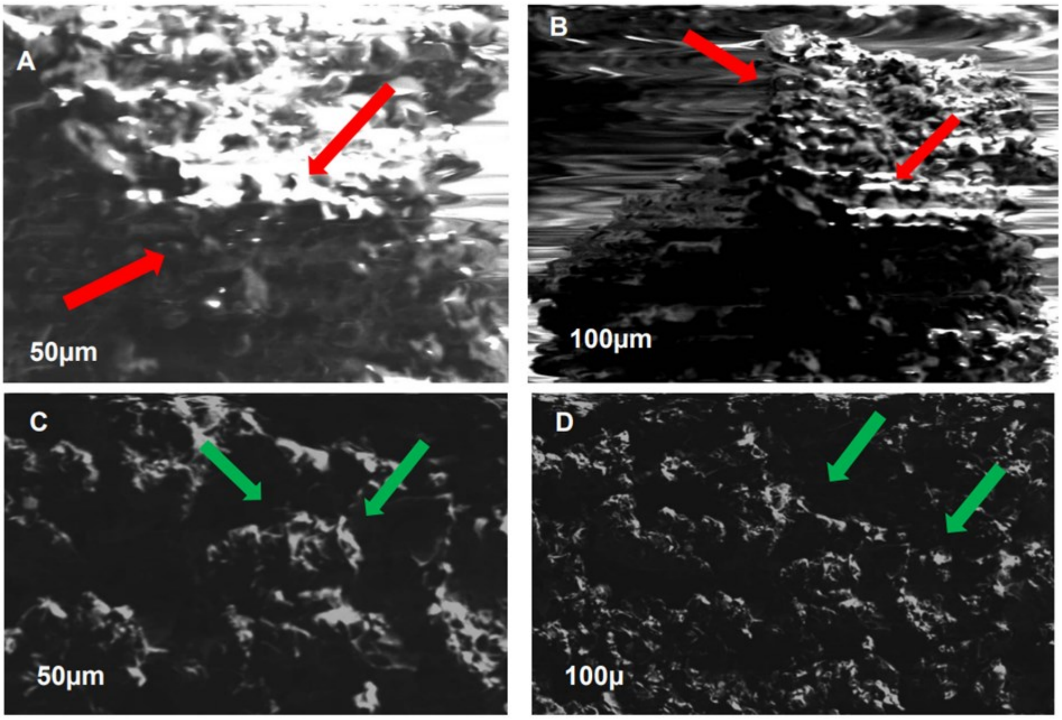

Figure 7: SEM Images of Potatoes Control (A and B) and Synthesized CNTs (C and D)

The SEM images (A and B) in Figure 7, captured at 500x magnification with a scale of 50 and 100 µm, shows the surface morphology of untreated potato peels. The visible surface texture is marked by a relatively smooth and compact structure, typical of unmodified plant-based materials. While the material exhibits some degree of surface roughness as can be seen in the arrowed pointed areas, it lacks the extensive porosity and nanoscale features required for high-efficiency adsorption applications, such as crude oil spill cleanup. This baseline observation highlights the need for further treatment and modification to enhance the adsorption properties of potato peels as a precursor for CNTs. [44]

Potato peels are rich in organic carbon content, making them suitable candidates for the sustainable synthesis of CNTs. However, in their raw form, as shown in this SEM image, the potato peels have limited surface area and microporosity. According to recent studies, chemical and thermal treatment processes, such as pyrolysis, can significantly improve the surface structure of potato peels, transforming them into CNTs with highly porous and hydrophobic surfaces that are optimal for oil spill remediation. The smooth morphology observed here indicates that raw potato peels, while naturally abundant and cost-effective, require activation to enhance their adsorption capabilities. [45]

The use of agricultural waste materials like potato peels for CNT synthesis aligns with ongoing research focused on sustainable environmental technologies. Previous studies have demonstrated that CNTs derived from potato peels possess excellent oil adsorption properties, primarily due to their high surface area and hydrophobicity post-treatment. The SEM image of the untreated potato peel serves as a starting point for understanding the material's natural limitations before processing. Through carbonization and activation processes, the surface roughness and porosity can be significantly improved, resulting in a material that is not only effective in crude oil spill cleanup but also supports waste valorization efforts. [46]

The SEM image in Figure 7, captured at 1500x magnification with a scale of 50 µm, provides a detailed view of the surface morphology of carbon nanotubes synthesized from potato peels. The image reveals a complex, intertwined network of CNTs, showcasing a high degree of surface roughness and porosity. This enhanced morphology is crucial for environmental applications, particularly for the effective adsorption of hydrophobic contaminants such as crude oil. The presence of numerous surface features and the tubular structure indicate successful synthesis and activation, positioning the material as a potent candidate for oil spill remediation. [47]

Sustainable synthesis methods for carbon nanotubes from agricultural waste, like potato peels, have garnered significant attention due to their cost-effectiveness and environmental benefits. The transformation from raw potato peels to CNTs involves thermal and chemical treatments that promote the formation of a porous network, significantly increasing the surface area available for adsorption. Studies have demonstrated that CNTs synthesized from biomass exhibit superior adsorption characteristics, largely attributed to their high aspect ratios and unique surface functionalities. The image exemplifies how the synthesis process can unlock the potential of potato peels as an eco-friendly adsorbent for oil spills. [48]

The ability to convert agricultural waste into valuable nanomaterials aligns with the principles of sustainability and circular economy. By synthesizing CNTs from potato peels, not only is waste minimized, but new functional materials are also created that can address pressing environmental issues, such as oil spills. Research indicates that these biomass-derived CNTs not only have enhanced structural properties but also exhibit excellent hydrophobicity, making them suitable for capturing and retaining oil in contaminated environments. The SEM image of synthesized CNTs from potato peels underscores the effectiveness of utilizing waste materials in nanotechnology, supporting innovative approaches to environmental remediation. [49]

X-ray Diffraction (XRD) Results

XRD is a non-destructive technique that reveals the crystallographic structure of materials by analyzing how X-rays are diffracted by the atomic planes in a sample. It is used to identify the crystalline phases present in a material and to determine its lattice parameters, helping to understand the structure and purity of synthesized materials.

Table 1: XRD Peak list, Qualitative Analysis and Percentage of the Synthesized CNTs

Phase name	Formula	2θ	Figure of merit	Percentage (%)
Graphite-2H	C	26.526(10)	0.513	24
Pentlandite, syn	$Fe_4.8Ni_4.2S8$	50.06(2)	1254	21
Sylvite, syn	K Cl	28.33(5)	1.165	25
Quartz, syn	SiO_2	26.526(10)	1.459	5
Aluminum Phosphate Hydrate	$Al PO_4 x H_2O$	50.06(2)	3.007	6.7
Mascagnite	$(NH4)_2 SO_4$	28.33(5)	2.927	19.5

Table 1, presents the X-ray Diffraction analysis of CNTs synthesized from potato peels, showcasing a variety of crystalline and amorphous phases. The prominent peak at approximately 26.6° corresponds to the (002) plane of graphite, indicative of the formation of graphitic carbon. This is a critical finding, as the presence of graphitic structures enhances the mechanical and electrical properties of the CNTs, making them ideal candidates for environmental applications such as crude oil spill remediation. Previous research has demonstrated that sustainably sourced CNTs, like those from potato peels, possess excellent adsorption properties due to their high surface area and tunable porosity. [49]

In addition to the graphite phase, the XRD pattern reveals peaks corresponding to aluminum phosphate hydrate, pentlandite, and other mineral phases at 50.06°. The presence of aluminum phosphate is noteworthy, as it suggests that the synthesis process may have preserved some inorganic constituents from the potato peels, which could enhance the structural integrity and thermal stability of the CNTs. Previous studies have shown that such inorganic impurities can play a significant role in improving the overall performance of carbon-based materials, particularly in applications related to oil adsorption and environmental cleanup. The retention of these phases can also provide additional functional sites for interactions with oil molecules, further improving the efficacy of the CNTs in spill response scenarios. [50]

The detection of quartz and sylvite in the XRD analysis indicates the presence of silica and potassium chloride minerals, respectively. These minerals are often found in plant materials and can influence the physicochemical properties of the synthesized CNTs. The presence of quartz has been linked to increased hydrophilicity, which may assist in the dispersion of CNTs in aqueous environments, an essential factor in their practical application for oil spill cleanup. The combination of graphitic structures and the mineral content from potato peels not only contributes to the material's mechanical strength but also enhances its capability to interact with hydrophobic substances like crude oil. [51]

The X-ray diffraction (XRD) analysis of CNTs synthesized from potato peel waste indicates the presence of graphitic carbon and certain mineral phases, highlighting the successful synthesis of CNTs with desirable structural features. This synthesis process, which relies on agricultural waste, promotes sustainability in material production. The graphitic structure of these CNTs improves their mechanical and chemical stability, which is advantageous for environmental remediation applications, especially in adsorbing and cleaning up oil spills. Utilizing potato peels as a precursor for CNTs not only offers an eco-friendly alternative but also adds value to waste materials, contributing to sustainable environmental solutions.

From the percentage composition of the sample displayed in table 1, the presence of graphite-2H indicates that the synthesized CNTs may have a layered structure similar to that of graphite. The 2H notation refers to the hexagonal crystal system typical of graphite. A peak corresponding to this phase would typically appear around 26° 2θ, indicating a well-defined crystalline structure. The percentage composition of 24% suggests that a significant portion of the material is composed of this phase, which may contribute to the electrical conductivity and structural integrity of the CNTs.

With a composition of 21%, pentlandite is a nickel-iron sulfide mineral often associated with nickel deposits. Its presence could indicate residual minerals from the potato peel synthesis process or impurities in the CNTs. Peaks for pentlandite would typically be observed at specific angles related to its cubic crystal system. Sylvite, with a composition of 25%, is a potassium chloride mineral commonly found in evaporitic environments. Its presence may suggest that potassium ions were incorporated during synthesis or are residual from the precursor materials used in synthesizing CNTs from potato peels.[52]

Quartz, comprising 5% of the sample, is one of the most abundant minerals in Earth's crust and can be present due to contamination or as an inherent component in plant materials like potato peels. Quartz peaks are generally sharp and well-defined due to its high crystallinity.

Aluminum Phosphate Hydrate compound's presence at 6.7% indicates that aluminum ions were likely involved during synthesis, possibly contributing to catalytic activity during CNT formation or acting as a stabilizer for the synthesized material. Finally, Mascagnite appears at 19.5%, suggesting ammonium sulfate was either introduced during synthesis or formed as a byproduct when using organic precursors like potato peels rich in nitrogen compounds. Generally, the XRD patterns from our research displayed distinct peaks corresponding to specific 2θ values, which indicate the presence of crystalline phases. A prominent peak around 26.6° (2θ), which corresponds to the (002) plane of graphite signifies that the synthesized material has a crystalline graphitic structure, characteristic of high-quality CNTs. Additional peaks at different angles, may correspond to residual biomass or other carbonaceous materials that did not convert into CNTs during synthesis.

Batch Adsorption Study

Effect of Adsorbent Dosage

The effect of adsorbent dosage on the adsorption of crude oil was determined by the use of different adsorbent dosage (2.0 g, 3.0 g, 4.0 g, 5.0 g and 6.0 g) of synthesized potatoes peel CNTs, the amount of crude oil adsorbed per unit mass of its adsorbent dosage at equilibrium

q_a (mg/g) and the percentage removal of crude oil was determined using equation 1 and 2 above.

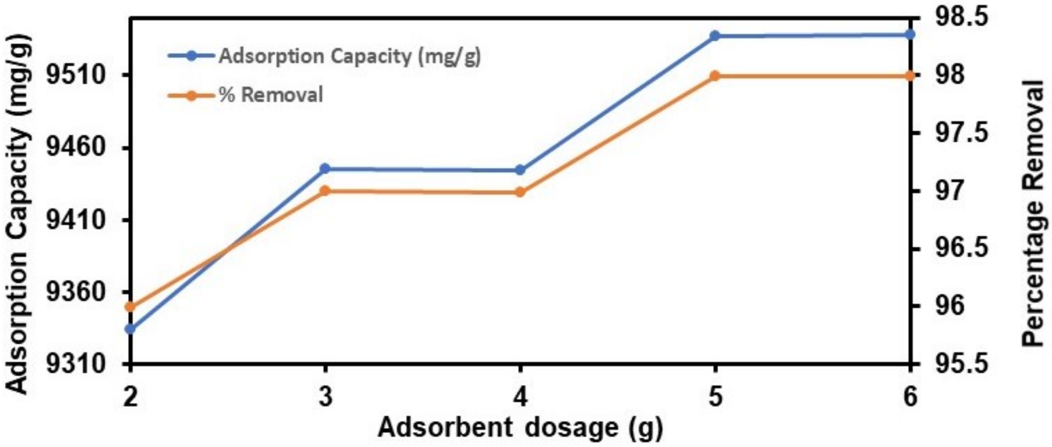

Figure 8: Graphical presentation of the result obtained on effect of adsorbent dosage on the adsorption of crude oil onto synthesized CNTs

Figure 8, presents the relationship between the dosage of potatoes peels CNTs and the effectiveness of crude oil adsorption. As the adsorbent dosage increases from 2 g to 5 g, both the percentage removal of crude oil and the adsorption capacity (measured in mg/g) show a significant upward trend. This indicates that as more adsorbent is introduced, the available surface area for adsorption increases, leading to enhanced crude oil removal. This trend aligns with research findings, where increasing adsorbent dosage typically enhances removal efficiency due to the higher availability of active adsorption sites. [53-54]

At the higher dosage levels, particularly from 5 g to 6 g, the graph shows a plateau, where both the percentage removal and adsorption capacity stabilize at around 98% and 9550 mg/g, respectively. This suggests that beyond a certain dosage, the system reaches an equilibrium, and additional adsorbent does not result in significant further improvement. This behavior is consistent with the concept of saturation in adsorption processes, as noted in studies where once the active sites are fully occupied, adding more adsorbent has little effect on the removal rate [55]. This implies that beyond 5 g, the adsorbent's capacity to adsorb crude oil is maximized.

The use of potatoes peels as a sustainable source of CNTs for oil spill cleanup is particularly promising due to their low cost and eco-friendly nature. The findings from this graph reinforce the potential of this bio-waste material in environmental cleanup applications, aligning with studies that emphasize the effectiveness of bio-based CNTs in adsorbing hydrocarbons from aqueous environments [56-57]. This highlights the importance of optimizing adsorbent dosage to achieve the best balance between efficiency and cost in practical applications of crude oil spill remediation.

Effect of pH

Figure 9 illustrates the influence of pH levels on the efficiency of crude oil removal and adsorption capacity of CNTs synthesized from potato peels. The results reveal that as the pH increases from 6 to 6.8, both the percentage removal of crude oil and adsorption capacity consistently improve. This trend aligns with findings in similar studies, which suggest that acidic to near-neutral pH conditions favor the adsorption of hydrophobic contaminants, such as crude oil, onto CNTs due to changes in the surface charge and functional groups on the adsorbent material. At lower pH values, competitive interactions with protons may reduce the adsorption sites available for crude oil molecules, but as the pH increases, these sites become more accessible, enhancing the adsorption performance [58].

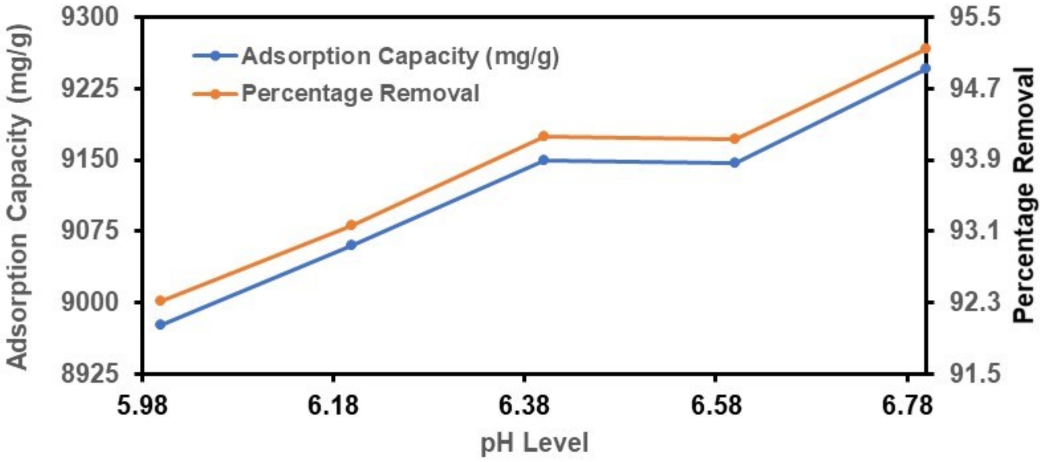

Figure 9: Graphical presentation of the result obtained on effect of pH on the adsorption of crude oil onto synthesized CNTs

The graph shows a relatively stable adsorption efficiency between pH 6.2 and 6.6, followed by a noticeable increase from pH 6.6 to 6.8. This behavior is consistent with the surface chemistry of CNTs derived from biowaste materials, as the ionization of surface groups like hydroxyl and carboxyl groups typically intensifies in slightly alkaline conditions, allowing improved interaction with non-polar molecules like oil. Studies have documented that the increased pH may lead to enhanced electrostatic interactions and hydrophobic effects, both of which are critical for binding crude oil molecules onto CNT surfaces [59]. Consequently, a slightly higher pH could optimize the adsorption process without significantly affecting the structural stability of the CNTs.

These findings underscore the potential of potato peel-derived CNTs in crude oil remediation, especially in environments with near-neutral pH levels. This pH-dependent behavior aligns with similar observations in CNT adsorption research, where bio-synthesized CNTs exhibit robust oil adsorption properties due to the intrinsic compatibility of carbon-based materials with hydrophobic pollutants.[60]

Effect of Temperature

Figure 10 illustrates the effect of temperature on the adsorption capacity and percentage removal of crude oil using CNTs synthesized from potato peels. The data show a clear inverse relationship between temperature and adsorption performance: as the temperature increases from 20°C to 100°C, both the adsorption capacity and the percentage removal decrease steadily.

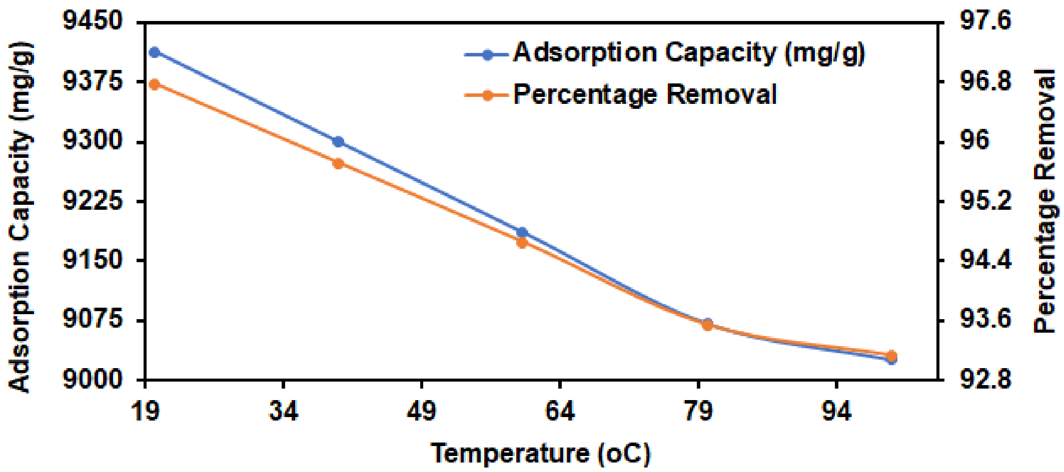

Figure 10: Graphical presentation of the result obtained on effect of temperature on the adsorption of crude oil onto synthesized Potatoes peels CNTs

At 20°C, the CNTs exhibit their highest adsorption capacity and removal efficiency, capturing around 9412.64 mg/g with a removal rate of approximately 96.77%. However, at 100°C, adsorption capacity reduces to around 9026.04 mg/g, with removal efficiency dropping to 93.13%. This trend suggests that the adsorption process may be exothermic, with lower temperatures favoring the adsorption of crude oil onto the CNTs. Similar findings in the literature report that higher temperatures increase the kinetic energy of molecules, which can lead to weaker interactions between the adsorbate (crude oil) and the adsorbent (CNTs), reducing the overall adsorption efficiency. [61- 62]

The practical implication of this temperature sensitivity is crucial for optimizing crude oil cleanup strategies. For regions with variable temperatures or in situations where temperature control is feasible, these CNTs from potato peels would be most effective in cooler conditions. At higher temperatures, adsorption efficiency decreases, potentially due to the desorption effect where crude oil molecules are more likely to escape from the CNT surface. [63-64]

This highlights the importance of deploying potato peel-derived CNTs in settings where environmental temperatures can be maintained at lower levels to maximize adsorption efficiency. Furthermore, the decrease in adsorption efficiency at higher temperatures aligns with the general understanding that physical adsorption processes often decline as thermal energy disrupts weak van der Waals forces between the adsorbate and adsorbent. [65]

Effect of Contact Time

The graph in Figure 11 illustrates the effect of contact time on the adsorption efficiency of crude oil onto CNTs synthesized from potato peels. As time progresses from 30 to 90 minutes, the adsorption capacity (mg/g) and percentage removal (%) both increases, suggesting that the contact time plays a significant role in the adsorption process. Initially, the rate of adsorption appears slower, which could be attributed to limited surface area exposure of the synthesized CNTs to the crude oil. However, as the contact time extends, the adsorption rate accelerates, achieving a higher percentage removal and capacity, reaching approximately 96% removal and over 9300 mg/g capacity at 90 minutes. This trend aligns with studies suggesting that prolonged contact time allows for greater interaction between adsorbent and pollutant molecules, enhancing removal efficiency and adsorption capacity.

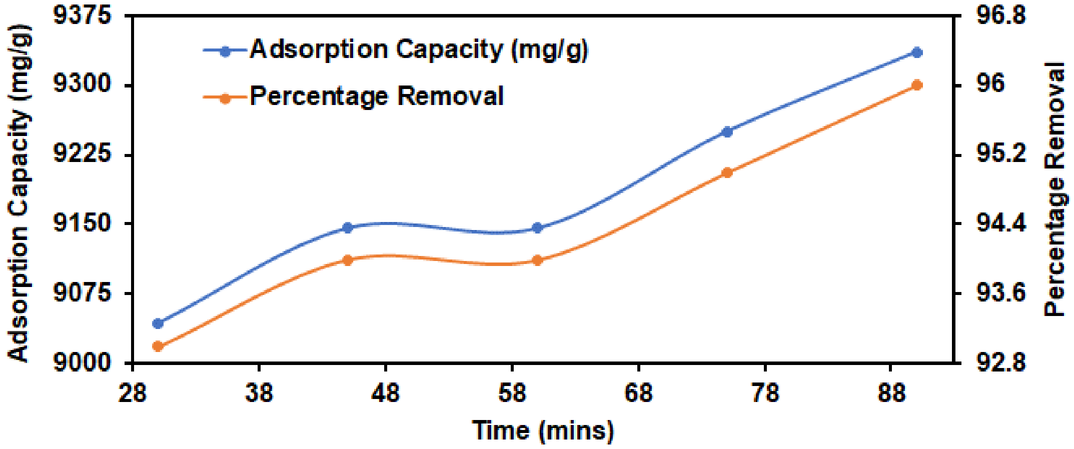

Figure 11: Graphical presentation of the result obtained on effect of contact time on the adsorption of crude oil onto synthesized potatoes peels CNTs

This result aligns well with findings from other research on the utilization of sustainably sourced carbon-based adsorbents for oil spill cleanup. Carbon nanotubes derived from agricultural waste, such as potato peels, have shown promising adsorption properties, largely due to their high surface area, porosity, and functional groups that interact with crude oil molecules. Studies have highlighted that carbon materials derived from such waste sources can serve as eco-friendly, cost-effective alternatives for traditional adsorbents in oil spill remediation. [66]

Batch Adsorption Kinetics

To research the sorption mechanism and potential rate-controlling phases, such as mass transport and chemical reaction processes involved in crude oil adsorption from aqueous solution. The data obtained were analyzed using Langmuir, Freundlich, and Temkin isotherm models. The constants for each model were calculated using nonlinear regression analysis. The adsorption kinetic model constants for the removal of crude oil are shown in Table 2.

Table 2: Adsorption kinetic model constants for the removal of crude oil using Potatoes Peel Synthesised Carbon Nanotubes

Langmuir Isotherm	Qmax (mg/g)	KL (L/mg)	R^2
	130	0.05	0.998
Freundlich Isotherm	**n**	**KF (mg/g)**	**R^2**
	1.5	28	0.764
Temkin Isotherm	**AT (mg/g)**	**BT (J/mol)**	**R^2**
	9	2	0837

The synthesis of carbon nanotubes from potato peels not only provides an eco-friendly alternative but also enhances waste valorization efforts. The high Qmax value from the table indicates that these CNTs can effectively remove significant quantities of crude oil from contaminated water bodies, making them suitable candidates for remediation applications.

The comparison among different isotherm models reveals insights into the nature of interactions between crude oil molecules and the surface of CNTs derived from potatoes peel. The high R^2 values across all the three models (with Langmuir Isotherm having the best value) suggest complex interactions where both monolayer and multilayer formations occur alongside significant intermolecular forces affecting binding energies. This complexity often arises in real-world scenarios where various factors such as temperature, pH, and chemical composition influence how effectively an adsorbent can capture crude oil from aqueous solutions.

Literature review has revealed that majority of biomass-based adsorbents had excellent adsorption capacity and removal efficiency (>85 %). The adsorption process for all the adsorbents mainly followed the Langmuir isotherm model and correlated well with the pseudo-second order kinetic model. They believed that sorption occurs primarily through intra-particle diffusion, film diffusion, and ion exchange. [67]

Our results are in agreement with Ahmed et al., findings in their sorption studies in which R^2 values were $0 < R^2 < 1$ and the values of n were more significant than 1, demonstrating the favorable sorption of the crude oil on the adsorbents. [68]

It can be concluded that our results align with existing literature on CNTs' capabilities while highlighting their potential use in environmental applications.

Conclusion

The study demonstrates the synthesis of CNTs from potato peels, an agricultural waste material, using a controlled pyrolysis process. The CNTs were evaluated for their potential use in crude oil spill remediation. The CNTs showed high adsorption performance, with a maximum capacity of 9538.17 mg/g and a removal efficiency of 97.997% at a dosage of 6.0 g. They performed best in slightly alkaline conditions and cooler environments, with optimal contact time of 90 minutes. The data was analyzed using Langmuir, Freundlich, and Temkin isotherm models, revealing complex interactions and intermolecular forces affecting binding energies. The results highlight the effectiveness of potato peel-derived CNTs as eco-friendly and sustainable adsorbents for crude oil spill remediation, addressing environmental challenges and waste valorization. Future research should be focused on optimizing the synthesis process, assess long-term environmental impacts, and conduct cost-benefit analyses.

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