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Phyto-synthesis and characterization of iron-clay nanocomposite using *Elaeis guineensis* Leaves

Mary X Udoh [1], Solomon E Shaibu [1,*], Eno A Moses [1], Emmanuel I Uwah [1], Tijesuni J Adeoye [2] and Comfort O Emmanuel [3]

[1] *Department of Chemistry, University of Uyo, Uyo, Nigeria.*
[2] *Department of Chemistry, University of Vermont, Burlington, VT 05405, USA.*
[3] *Department of Medicine, Grodno State Medical University, Grodno State, Belarus.*

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Abstract

This study presents an eco-friendly synthesis and comprehensive characterization of iron-clay nanocomposites (BFeCN) using *Elaeis guineensis* leaf extract as a natural reducing and stabilizing agent. The phytochemical analysis confirmed the presence of alkaloids, flavonoids, tannins, and phenolics, facilitating the bioreduction of Fe^{3+} to zero-valent iron nanoparticles (FeNPs). The synthesized BFeCN was systematically characterized using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX), and Brunauer–Emmett–Teller (BET) analysis to determine its structural, morphological, and surface properties. The BET analysis revealed a specific surface area of 49.49 m^2/g, a pore volume of 0.138 cm^3/g, and a mesoporous pore size of 121.02 Å, indicating enhanced adsorption potential. The XRD analysis confirmed the successful incorporation of FeNPs within the clay matrix, with key diffraction peaks at 25° and 35.5° (2θ) corresponding to quartz and magnetite phases. The SEM image displayed a rough and highly porous morphology, corroborating the enhanced textural properties of BFeCN while EDX spectra validated the elemental composition, demonstrating a significant iron incorporation that improved adsorption and catalytic properties. These results underscore the potential of BFeCN in pollutant sequestration, offering an environmentally sustainable approach for water remediation and other nanotechnological applications.

Keywords: Phytosynthesis; Iron-clay nanocomposite; *Elaeis guineensis*; Characterization; Eco-friendly materials

1. Introduction

The advent of nanotechnology has opened frontiers of opportunity due to inherent unique properties of materials at the nanoscale, particularly in the remediation of environmental media. It has proven to be a potential technology that is rapidly emerging in wider areas such as medicine, electronics and food technology by manipulating nanomaterials for various applications. Nanotechnology is the engineering and art of developing new materials on a nanoscale. A nanomaterial is a discrete entity with at least one dimension as 100 nm or less. It may include any of the following nano forms: nanoparticles, nanotubes, fullerenes, graphene, nanoclays, nanofibres, nanowhiskers and nanosheets Nanomaterials have enjoyed extensive applications in different spheres as a result of their diversity and chemistry which include but not limited to: water treatment (Vikrant and Kim 2019), metal ion reutilization (Deng et al. 2019), plant disease diagnosis and management (Khan et al. 2019), stem cell research (Aziz et al. 2019; Pacheco-Torgal 2019), construction (Dahlan et al. 2019), and catalysis (Zhu et al. 2019). It has been empirically proven that nanoparticles possess high surface area and potent reactivity. However, their wide applications are hampered by agglomeration and rapid oxidation tendencies (Phenrat et al. 2007) which affect efficiency. Addressing the challenges of agglomeration and

* Corresponding author: Solomon E. Shaibu

rapid oxidation in the synthesis of nanomaterials has necessitated the use of stabilizing agents (Lin et al. 2010) as support in the production of nanocomposites (Liu et al. 2014; Yuan et al. 2009; Kim et al. 2013; Dorathi et al. 2012) with reasonable success. Despite the aforementioned, scanty reports are available for the treatment of polluted water using green and sustainable nanocomposites.

Clay is generally a fine material made of natural rocks or soil that consists of more than one mineral, with small amount of metal oxides and organic matter. Chemically, clay is made up of small crystallites of alumino-silicates of various proportions, with substitutions of iron and magnesium by alkalis and alkaline earth elements (Pavlidou and Papaspyrides 2008). It differs from other fine materials by size and composition. The loading of nanoclays in clay-based nanocomposites is optimized with many better properties as compared to the matrix materials with an eye on a specific application. The incorporation of small amount of organoclays enhances the thermal stability of grease (Kandola et al. 2008; Tcherbi-Narteh 2013). Furthermore, the color retention by nanoclay opens an avenue for application in cosmetics and inks (Natarajan 2015; Patel et al. 2006). The petroleum industry also makes use of organoclays for the removal of hydrocarbons from refineries (Natarajan 2015). The removal of toxic chemicals from pharmaceuticals and pesticides industries is being carried out by organoclays (Chen et al. 2011).

The advancement of nanotechnology has led to the development of various nanomaterials with unique properties suitable for environmental applications. Among these, iron-based nanocomposites have garnered significant attention due to their effectiveness in pollutant removal and catalytic processes (Shaibu et al., 2014). Traditional synthesis methods of these nanomaterials often involve hazardous chemicals and energy-intensive procedures, raising environmental and health concerns. To address these issues, green synthesis approaches have emerged as sustainable alternatives, utilizing natural resources to reduce and stabilize nanoparticles. Combining iron nanoparticles with clay minerals results in iron-clay nanocomposites that exhibit enhanced adsorption capacities and catalytic properties. The clay matrix provides a large surface area and structural support, while the iron nanoparticles contribute to the reactive sites necessary for environmental remediation processes. Such composites have been effectively utilized in the removal of contaminants from water sources. A study demonstrated the successful synthesis of iron nanoparticles supported on clay using palm waste extract, emphasizing the potential of integrating natural materials in nanocomposite fabrication (Dorathi et al. 2012).

Green syntheses methods employ biological entities such as plant extracts, microorganisms, and biopolymers to synthesize nanoparticles. These methods are environmentally friendly, cost-effective, and offer a safer alternative to conventional chemical synthesis. Plant extracts, in particular, are rich in bioactive compounds like polyphenols, flavonoids, and alkaloids, which can act as reducing and capping agents in nanoparticle formation. For instance, the use of green tea extract has been reported in the synthesis of zero-valent iron nanoparticles, highlighting the role of plant-derived compounds in nanoparticle stabilization. Various organisms act as clean, eco-friendly, and sustainable precursors to produce stable and well-functionalized nanoparticles (Sfameni et al., 2023; Raj et al 2022). Plant-mediated approaches are simple, non-toxic, cost-effective, low-cost, and eco-friendly.

Palm leaves are abundant agricultural residues, especially in tropical regions, and are often underutilized. They contain various phytochemicals capable of reducing metal ions to nanoparticles. Research has shown that extracts from palm petioles can be used to synthesize zero-valent iron nanoparticles, which were effective in removing hexavalent chromium from water. This indicates that palm leaf extracts can serve as both reducing and stabilizing agents in the green synthesis of iron-based nanomaterials.

This study aims at developing eco-friendly iron-clay nanocomposite using palm leaf extract. The synthesized nanocomposites were characterized to determine their structural, morphological, and surface properties. The effectiveness of these nanocomposites in environmental applications, particularly in pollutant removal, was also evaluated. By utilizing palm leaves, this work seeks to provide a sustainable approach to nanocomposite synthesis, contributing to waste valorisation and environmental remediation efforts

2. Material and methods

2.1. Sample collection

The clay sample, which contains a significant amount of kaolin, was collected from Ikot Ebom Itam in Itu Local Government Area. The clay found in this vicinity contained approximately 30.7% kaolin, a key ingredient needed for the synthesis of BFeNC (Obot *et al*., 2021). The collected clay sample was sundried to eliminate moisture. Once dry, it was finely ground to increase its surface area, facilitating the subsequent sieving process. Drying the clay removed any interfering moisture during synthesis, while grinding enhanced the clay's adsorption properties by increasing its surface area. The clay was washed with 0.1 M HCl and cleaned with deionized water until the pH of the liquid phase became neutral, as outlined by Zhu *et al*. (2019) and Adebayo *et al., 2*019.

2.2. Extract preparation and phytochemical analysis of *Elaeis guineensis* Leaves

Fresh leaves of *Elaeis guineensis* were sourced from the Ekamba Nsukara located in Uyo Local Government Area (LGA) of Akwa Ibom state, Nigeria. The collected plant material was transported to the Chemistry Laboratory at the University of Uyo, Uyo, Nigeria. The leaf samples were thoroughly washed with distilled water, cut into small pieces, and air-dried. Subsequently, the dried leaves were finely ground and subjected to maceration by immersing 60 g of the powdered *Elaeis guineensis* leaves in water for 48 hours with slight heating at 50 °C for 20 minutes. This extract was then utilized for phytochemical analysis, as described by Enin *et al.,* 2013, to assess the presence of various bioactive compounds such as alkaloids, flavonoids, terpenoids and steroids, saponins, tannins, phenolic compounds, coumarins and carbohydrates. Qualitative phytochemical analysis was done according to standard protocols as described previously (Edeoga et al., 2005, Raaman (2006) and Karthishwaran et al., 2010). The extract was filtered and used for the synthesis of BFeNC.

2.3. Synthesis of BFeNP

A 200 ml of *Elaeis guineensis* extract was mixed with 2 M of 500 ml iron (iii) chloride ($FeCl_3$) and 5 g of pulverized clay. The bioactive compounds in the *Elaeis guineensis* leaf extract, such as flavonoids and terpenoids, acted as reducing agents, facilitating the reduction of Fe^{3+} from the iron (iii) chloride (Singh *et al*., 2024). The reduction of Fe^{3+} led to the formation of zero-valent FeNPs (Fe^0) within the clay slurry. To ensure the optimal formation and growth of FeNPs, the mixture of *Elaeis guineensis* leaf extract, $Fe(NO_3)_2$ and clay were incubated at an optimized temperature of 50 - 60 °C under continuous stirring for 4 hours to promote the uniform distribution and adequate bonding of the nanoparticles to create the BFeNC. This meticulous mixing process results in the formation of BFeNC, where the FeNPs are evenly dispersed throughout the clay.

2.4. Characterization of BFeNC

Different techniques were employed to characterize the prepared BFeNC. Fourier Transform Infrared (FTIR) spectra were obtained using a Bruker Platinum ATR Tensor 27 spectrometer in single mode at 4 cm^{-1} resolution, with a range of 4000-300 cm^{-1} and an average of 32 scans per sample. X-ray diffraction (XRD) was utilized for identifying unknown crystalline materials (e.g., minerals and inorganic compounds), characterizing crystalline materials, measuring sample purity, and determining unit cell dimensions. A Bruker multifunctional Powder x-ray diffractometer D8-Advance was employed using Cu-Kα radiation (λ= 0.1506 nm). The analysis was performed using locked coupled mode scanning at 40 kV, 30 mA, and a scanning speed of 5°/min. The size, shape, and surface morphology of the catalysts were assessed using a TESCAN Vega 3 LMH Scanning Electron Microscope (SEM) with a working distance of 15 mm and a voltage of 20 kV. Image acquisition was carried out at various magnifications (10, 20, 50, and 100 μm) with the samples mounted on a carbon tape-fast sample holder. High-Resolution Transmission Electron Microscopy (HRTEM) and Energy Dispersive X-ray Spectroscopy (EDS) were employed to determine the composition and distribution of the elements.

3. Results and discussion

3.1. Phytochemical Analysis of *Elaeis guineensis* extract

The phytochemical analysis of *Elaeis guineensis* extract revealed the presence of several bioactive compounds in the plant as presented in Table 1.

The results indicated the presence of alkaloids in the plant, which aligns with the findings of Phin et al., 2013. Alkaloids are nitrogenous compounds with potential pharmacological activities, including antimicrobial and anti-inflammatory effects (Enin et al., 2021 & Pandey and Kumar, 2024). The result also revealed the presence of flavonoids in the plant. Flavonoids are polyphenolic compounds known for their antioxidant, anti-inflammatory, and anticancer activities (Yuan et al., 2024). The presence of steroids in the plant was also revealed from the results, including anti-inflammatory and immunomodulatory effects (Shaikh and Patil, 2020). As shown in Table 1, it was observed that saponins were absent in *Elaeis guineensis*. Saponins are known for their anti-inflammatory and antimicrobial properties (Phin et al., 2013). The result also revealed the presence of tannins in the plant. Tannins have both antioxidant and antibacterial properties. According to Dubale et al. (2023), the presence of alkaloids, flavonoids, and steroids, in the plant can link the plant to numerous health benefits, implying that *Elaeis guineensis* may have potential as a medicinal plant. Further investigation into these compounds' properties could lead to new therapeutic and medicinal applications.

Table 1 Preliminary Phytochemical Analysis of *Elaeis guineensis*

GROUP OF PHYTOCHEMICALS	TEST	RESULTS
Alkaloids	Mayer's test, Iodine test	+
Coumarins	NaOH test	+
Carbohydrates	Benedict's test, Molish test	+
Flavonoids	Shinoda test	+
Phenolics	Lead acetate	+
Tanins	Ferric chloride ($FeCl_3$)	+
Steroids	Salkowski's test	+
Terpenoids	Salkowski's test	+

+: present

3.2. Brunauer–Emmett–Teller (BET) surface area

The BET analysis of the synthesized BFeCN provided critical insights into its surface area, pore size, and pore volume—parameters pivotal for evaluating its adsorption capabilities as shown in Figure 1. The BET-specific surface area of the BFeCN was determined to be 49.49 m^2/g, with a pore size of 121.02 Å and a pore volume of 0.138 cm^3/g. A higher specific surface area enhances the adsorption capacity of materials by increasing the number of available active sites (Qu et al., 2021). This is particularly beneficial for applications such as pollutant removal, where adsorption is the key mechanism. The measured surface area indicates that the incorporation of iron nanoparticles effectively improved the clay's structural properties, likely by increasing porosity and creating additional microporous regions. The pore size of 121.02 Å classifies the material as mesoporous, which is advantageous for adsorbing medium-sized molecules, such as organic pollutants and heavy metals, commonly found in environmentally polluted media (Shaibu et al., 2014 & 2015). The pore volume of 0.138 cm^3/g further supports the material's capacity to accommodate a substantial amount of adsorbate molecules. This sharp increase in surface area and pore volume highlights the synergistic effects of incorporating iron into the clay, resulting in improved performance in applications requiring high surface area and porosity (Orok et at., 2024).

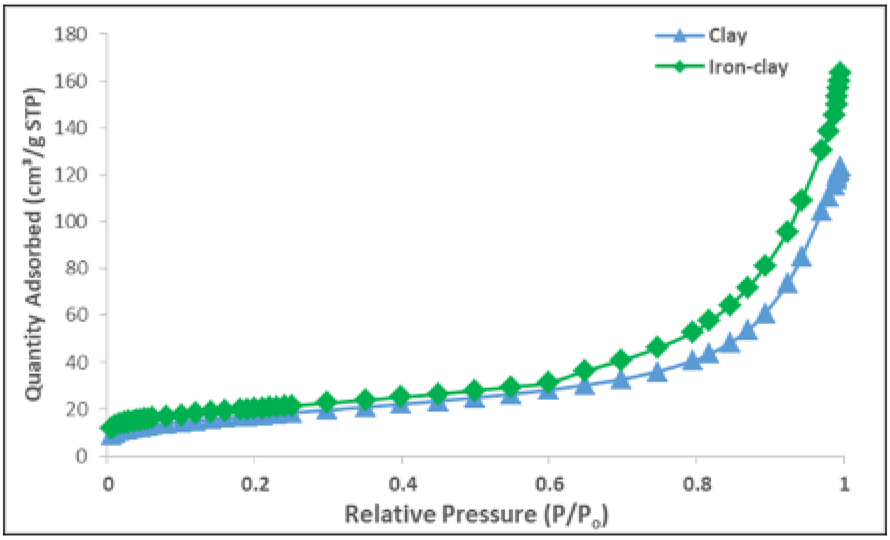

Figure 1 BET Analysis of clay and BFeCN

3.3. Energy Dispersive X – Ray Spectroscopy (EDX)

The elemental analysis of the clay and the FeCN using energy dispersive X – ray spectroscopy is as shown by the spectra in Figure 2. The EDX spectrum of natural clay identified the dominant elements; silicon (Si), oxygen (O), aluminum (Al), potassium (K) and sodium (Na). Silicon (17.8%) and aluminum (19.1%) are key constituents of aluminosilicate minerals (quartz, kaolinite and montmorillonite), which form the structural basis of the clay material, consistent with the report of Sabbagh et al. (2019). The high silicon content confirms the presence of silica (SiO_2), a primary component of clays and other silicate-based materials. The detection of oxygen and aluminum in the clay sample indicates the presence of oxide compounds, particularly silicates and aluminates. This finding supports the identification of the sample as clay, specifically in the form of aluminum silicates such as kaolinite or montmorillonite. These compounds are common in natural clays and contribute to the material's thermal stability, plasticity and ion-exchange properties (Singh et al., 2019; Irabor & Unuigbe, 2023). However, the spectrum revealed the presence of significant impurities, suggesting that the sample is not a pure form of aluminosilicate clay. The analysis also revealed the presence of iron due to the incorporation of iron within the clay matrix, confirming the successful synthesis of the nanocomposite. Additionally, the EDX results validated the successful synthesis of the FeNC, demonstrating its enriched elemental composition. The iron incorporation enhances its adsorption, magnetic properties, and overall applicability in pollutant removal, corroborating the effectiveness of using clay in environmental remediation strategies.

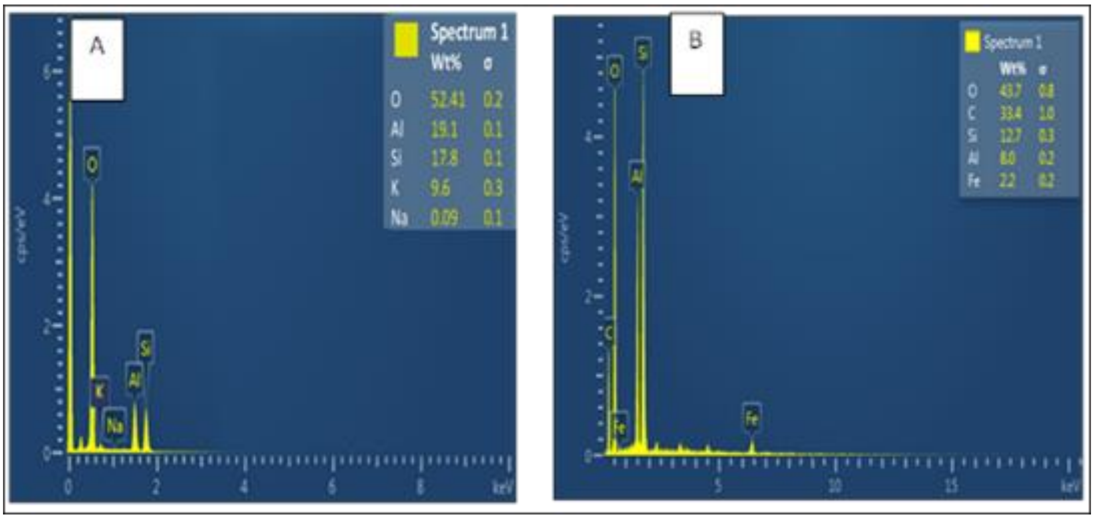

Figure 2 Energy-Dispersive X-ray (EDX) spectroscopic analysis of (a) raw clay (b) BFeCN

3.4. Scanning electron microscopy (SEM)

The Scanning Electron Microscope (SEM) was used to analyze BFeCN at 20.0 kV accelerating voltage, with a working distance (WD) of 15.31 mm and a magnification of 2.00 kx. The detector used is a secondary electron (SE), which provides detailed surface morphology and structural characteristics, highlighting the effects of iron nanoparticle incorporation. The image observed in the BFeCN revealed a rough, irregular surface with numerous granular and agglomerated particles. The texture suggests a material with a porous or fractured nature. The individual grains appear to be of varying sizes, and some seem fused or clustered. The SEM micrographs in Figure 3 also revealed the presence of large voids and irregular particle clusters. The rough surface and interparticle gaps suggest macropores, which contribute to enhanced fluid transport and higher adsorption capacity for larger molecules. These porous structures provide ample active sites for pollutant binding and facilitate the diffusion of adsorbates, aligning with observations by Ahmad and Haseeb, (2015). In addition, the SEM analysis of the BFeCN validated the successful synthesis of a structurally optimized composite. The observed porous architecture and surface roughness enhance its adsorption capacity and functional properties, making it a promising candidate for environmental remediation. These results are consistent with existing literature like the structural and adsorption studies of Fe-modified montmorillonite for dye removal (Adebayo et al., 2021).

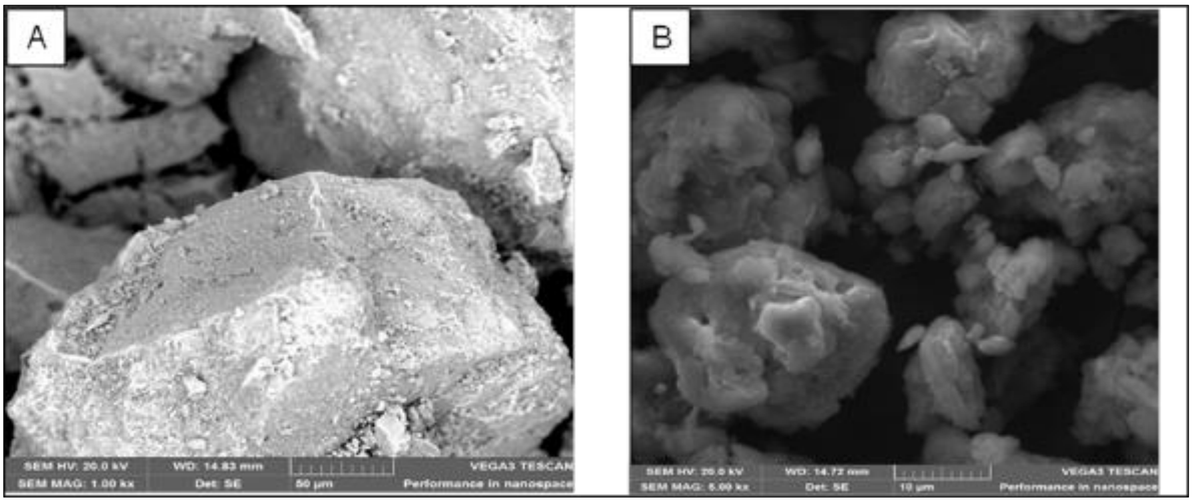

Figure 3 SEM images of (a) raw clay and (b) BFeCN

3.5. Fourier Transform Infrared Spectroscopy (FTIR)

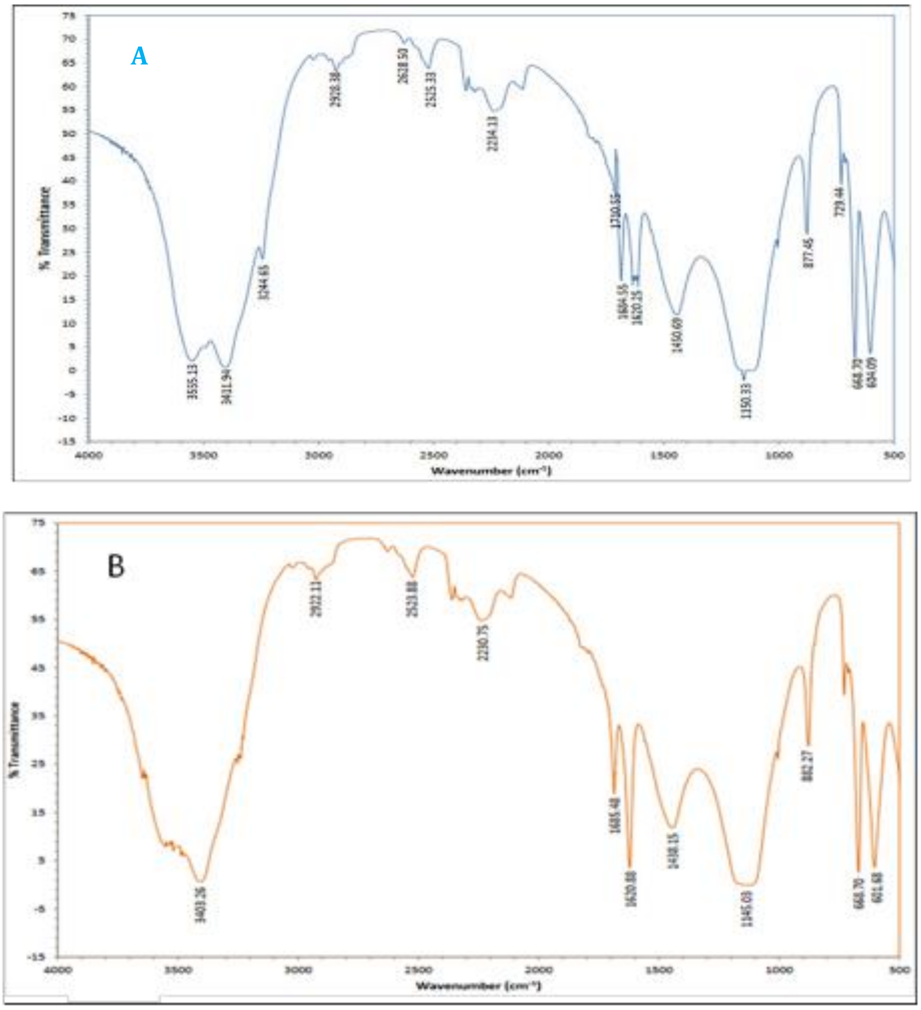

Figure 4 FTIR Spectra of a) Clay and b) BFeCN

The FTIR analysis of the iron-clad nanocomposite (BFeCN) elucidated the functional groups present and their roles in enhancing the material's adsorption capabilities (Regmi et al., 2012; Shu et al., 2020; Usman et al., 2021; Zhao et al., 2019). The spectra of clay and BFeCN in Figures 4a and 4b respectively exhibited several characteristic peaks corresponding to various functional groups critical for pollutant interaction and adsorption. Key peaks included O-H stretching vibrations (3400-3700 cm^{-1}) and C-H stretching vibrations (2900-3000 cm^{-1}), indicative of hydroxyl and aliphatic hydrocarbons groups, respectively. The O-H stretching bands are known for their hydrophilic nature and ability to form hydrogen bonds, facilitating the adsorption of polar molecules. The C-H stretching bands contribute to the hydrophobic interactions of the material. The peaks ranging from 1670-1690 cm^{-1} indicate the presence of –C=C stretching vibrations of aromatic groups. These groups play a vital role in chelating metal ions and adsorbing organic pollutants, as corroborated by Amarasinghe et al. (2009) who reported similar adsorption mechanisms in clay materials. Peaks ranging from 800 -1200 cm-1 represent Si – O in-plane stretching and Al – OH – Al bending vibrations of the clay material. This is in agreement with the previous submissions of Oguz et al. (2016) and Rezender et al. (2018). These functional groups enhance the composite's reactivity by providing active sites for pollutant binding, supporting its potential in environmental applications. The integration of iron nanoparticles may also have contributed to modifications in the chemical environment, as evidenced by slight shifts in peak positions compared to unmodified clay. In addition, the FTIR analysis confirmed the presence of functional groups essential for adsorption processes, validating the material's suitability for pollutant removal. These findings align with previous studies by Djomgoue and Njopwouo (2013), emphasizing the application of FT-IR spectroscopy for clay surface characterization.

3.6. X-ray diffraction analysis (XRD)

The XRD diffractograms of the clay and BFeCN (Figures a and b), as compared with the reflection patterns of the Joint Committee on Powder Diffraction Standards (JCPDS) card no. 04-0783 and 06–0696 reveal key information about their mineralogical composition and crystallinity. The most prominent peak, occurring around 25° (2θ), is characteristic of quartz (SiO_2) with high intensity and suggests that quartz is a major phase in the sample. Other smaller peaks between 5° and 10° (2θ) may indicate the presence of montmorillonite or illite, which are typical clay minerals. Montmorillonite, in particular, often shows peaks in this region due to its layered, expandable structure. Additional peaks observed around 15° (2θ) are likely attributed to kaolinite, as it is known for its characteristic peaks at both 12° and 25° (2θ), consistent with the reports of Obot et al. (2021) and Shaibu et al. (2022).

The BFeCN diffractogram in Figure 5b shows a peak at 250 which remains dominant, indicating quartz is still a major phase. The increased peak intensity at lower angles (150) suggests structural changes, possibly due to iron incorporation into the clay matrix. The emergence of peaks around 350-550 suggests new phases, potentially iron oxide. However, the peak at 35.5° (2θ) corresponds to the (311) reflection of magnetite (Fe_3O_4), indicating partial oxidation of the iron nanoparticles (Dadashi et al., 2015; Shaibu et al., 2014). Additionally, the XRD analysis confirms the successful synthesis of the FeCN (check formula) with well-defined crystalline phases of iron. These structural enhancements contribute to the composite's improved adsorption and catalytic properties, validating its potential for environmental applications such as pollutant removal and water treatment.

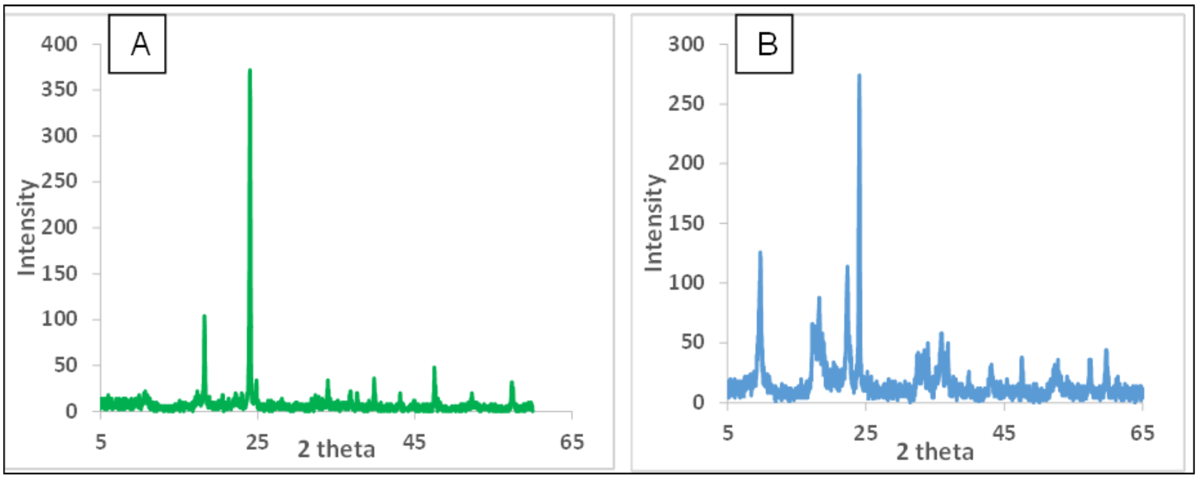

Figure 5 XRD image showing the composition of (a) clay and (b) BFeCN

4. Conclusion

This study successfully demonstrated the green synthesis of iron-clay nanocomposites (BFeCN) using palm leaf extract as a natural reducing and stabilizing agent. The characterization results confirmed the successful incorporation of iron nanoparticles onto the clay matrix, with enhanced surface properties and uniform distribution. The eco-friendly synthesis approach eliminates the need for hazardous chemicals, making it a sustainable and cost-effective method for nanomaterial production. The synthesized nanocomposite exhibits promising structural and textural properties, making it suitable for various environmental applications, particularly in pollutant removal. The utilization of palm leaves not only provides a sustainable alternative to conventional synthesis methods but also contributes to agricultural waste valorisation. Future studies will focus on optimizing synthesis conditions and exploring the nanocomposite's performance in real-world environmental applications. The findings of this research highlight the potential of green nanotechnology in developing effective and environmentally friendly materials for pollution control and sustainable development.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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