Characterization and Eco-friendly Synthesis of TiO_2 Nanoparticles on Steam Activated Carbon Derived from Chrysophyllum albidum Seed Shells for Adsorption and Photocatalytic Degradation of Pollutants

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ABSTRACT

In this work, TiO_2 Nanoparticles (NPs) were synthesized using the green approach and it was loaded on steam-activated carbon from Chrysophyllum albidum seed shells. The TiO_2 NPs, steam-activated carbon, and their composite were characterized for their size, shape, and elemental analysis, using the Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), and Electron Dispersive X-ray analysis (EDX). The SEM micrographs reveal the presence of numerous pores and cavities that can enhance adsorption. The TEM micrograph shows the spherical shapes of the nanoparticles with a size range of 2.80 to 5.80nm. The EDX spectrum of the activated carbon shows a high peak of carbon and oxygen, while that of the TiO_2 nanoparticles and composites reveals a high peak of titanium and oxygen. This study concludes that the green synthesized nanoparticle, Chrysophyllum albidum Seed shell Activated Carbon (CASAC), and CASAC loaded with TiO_2 nanoparticles (CASAC-TiO2NPs) have good capabilities for adsorption or degradation activities.

INTRODUCTION

Nanoparticles (NPs) are gaining huge attention as a rapidly growing class of materials for many applications (Mourdikoudis et al., 2018). Nanoscale materials with a wide range of optical, spectral, conductance and surface characteristics are being extensively studied and applied in various fields, such as electronics, photovoltaic, pollution abatement, corrosion inhibition and drug delivery (Saikumari et al., 2019). Numerous characterization methods have been used to observe the size, crystal structure, elemental composition and a variety of other physical properties of nanoparticles. However, each characterization technique is observed to have different strengths and limitations that complicate the choice of the most suitable method, thus, a synergistic characterization approach is preferred. Two of the main parameters studied in the characterization of NPs are size and shape. The size distribution, degree of aggregation, surface charge and surface area can also be measured, and sometimes, the surface chemistry can be evaluated (Mourdikoudis et al., 2018).

Titanium dioxide nanoparticles are one of the most important ceramic materials that are gaining worldwide attention because of their great industrial applications based on their physical and chemical properties, such as their particle size, surface area, structural composition, thermal stability, and porosity (Shaarawy et al., 2023). TiO_2NPs have also been discovered to be effective in the degradation of several classes of organic compounds, including dyes (Saikumari et al., 2019). The applications of TiO_2 are observed to be dependent on its crystalline structure, morphology and particle size, and the phase stability under different conditions (temperature, pressure, and medium) (Ragupathy et al., 2015 and Mironyuk et al., 2020). TiO_2NPs have been a preferred photocatalytic material because it is inexpensive, strongly oxidizing, easy to prepare, reusable, environmentally friendly, abundant and chemically and biologically inert and with no significant negative health effects (Saikumari et al., 2019; Mironyuk et al., 2020; Konni et al., 2022).

A transmission electron microscope (TEM) has higher resolution and better imaging of internal structures than an SEM. This, therefore, helps the size and shape of the nanoparticles to be more clearly analysed. A scanning electron microscope (SEM) images a pre-selected area of the sample by scanning it with a high-energy beam of electrons, producing signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity. SEM can produce very high-resolution images of a sample surface, revealing details about less than 1 to 5 nm in size. Most SEMs with a magnification range of 20X-30000X with a spatial resolution of 50-100 nm can scan areas which vary from 1 cm to 5μm in width. SEMs also can analyze particular points, as can be seen during EDX operations. Energy dispersive X-ray spectroscopy (EDS or EDX) is an analytical tool for elemental analysis or chemical characterization of a sample. Its characterization abilities are largely due to the fundamental principle that each element has a unique atomic structure, allowing X-rays that are characteristic of an element's atomic structure to be differently identified (Bodolla and Thodeti, 2018). This study aims to report the morphology and elemental composition of Chrysophyllum albidum seed shell activated carbon, green synthesized TiO_2 nanoparticles and their composite.

MATERIALS AND METHODS

Preparation of the steam-activated carbon

The C. albidum seeds were bought from Wazo market, Ogbomoso town, located at 8.1227° N Latitude and 4.2436° E Longitude, Oyo State, Nigeria. The collected seed samples were properly cleaned, carbonized in a furnace, and activated using a steam reactor. The CASAC was prepared using the modified methods of Amuda et al. (2013) as documented in the research by Amuda et al. (2025). The activated carbon yield is defined as the ratio of the mass of the resultant activated carbon (W1) to that of the precursor (Wo), with both masses being measured on a dry basis. It is therefore calculated using the following formula;

$$Yield\ of\ Activated\ Carbon\ (W_t\%) = \frac{W_1}{W_o} \times 100 \qquad (1)$$

Green synthesis of TiO2 nanoparticles

The green synthesis of TiO_2 NPs was carried out following the modified methods of Olowonyo et. al. (2023). Titanium (IV) oxide bulk powder (0.3g) was used as the precursor, while green tea extract was used as the reducing/capping agent. The synthesized TiO_2 NPs were centrifuged at 3000 rpm, oven dried at 100oC for 7 hours and calcined at 600 0C for 2 hours (Olowonyo et. al., 2023).

Preparation of TiO2 NPs Loaded CASAC (TiO2NPs-CASAC)

During the synthesis process of the nanoparticles, the CASAC powder (15g) was added to the mixture and continuously stirred for another 12 hours for complete loading (Amuda et al., 2025; Olowonyo et al., 2023). The composite was left for 10 hours, after which it was centrifuged at 3000 rpm for 20 min, dried at 110 °C for 3 hours and calcined at 500 °C for 3 hours (Mironyuk et al., 2020; Ouerghi et al., 2022).

Characterization methods

The Transmission Electron Microscopy analysis was carried out using the Verios 460L of the JEM-ARM200F-G TEM. Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy (SEM-EDX) imaging was done using a scanning electron microscope SEM, Hitachi SU 3500 scanning microscope, Tokyo, Japan (Saikumari et. al., 2019; Shaarawy et al., 2023.

RESULTS AND DISCUSSION

Activated Carbon Yield of the prepared CASAC

The carbonised seed shells were weighed to be 170g, while the prepared CASAC was weighed to be 156.72g. The yield of activated carbon is calculated to be 92.19% which indicates high quality of activated carbon (Okoya et al., 2020; Wang et al., 2022). The carbon yield was calculated as follows;

$$Yield\ of\ Activated\ Carbon\ (W_t\%) = \frac{W_1}{W_o} \times 100 \quad (2)$$

Where W_1 is 156.72g and W_0 is 170g;

$$W_t\% = \frac{156.72}{170} \times 100 \quad (3)$$

$$W_t = 92.19\% \quad (4)$$

Characteristics of the steam-activated carbon

The SEM micrograph of the CASAC in Figure 1 shows rough surface with irregular mesopores and micropores. The imaged surface reveals presence of heterogeneous pores and cavities that will aid infiltration of adsorbate solution.

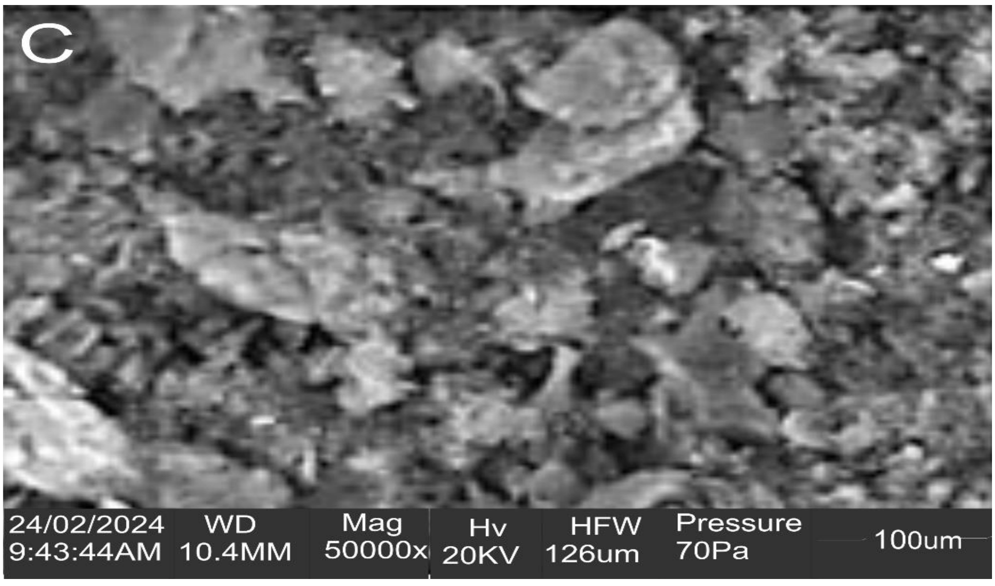

Figure 1: SEM micrograph of CASAC

The EDX spectrum of CASAC (Figure 2) reveals a high percentage of carbon, which is the major component of activated carbon derived from an organic precursor (Muhammad et al., 2024). The spectrum also reveals a high percentage of oxygen, relevant in bond formations and chemical interactions between adsorbent and adsorbate during adsorption processes, thus the presence of oxygen is expected to improve adsorption capacities (Muhammad et al., 2024; Amuda et al., 2025).

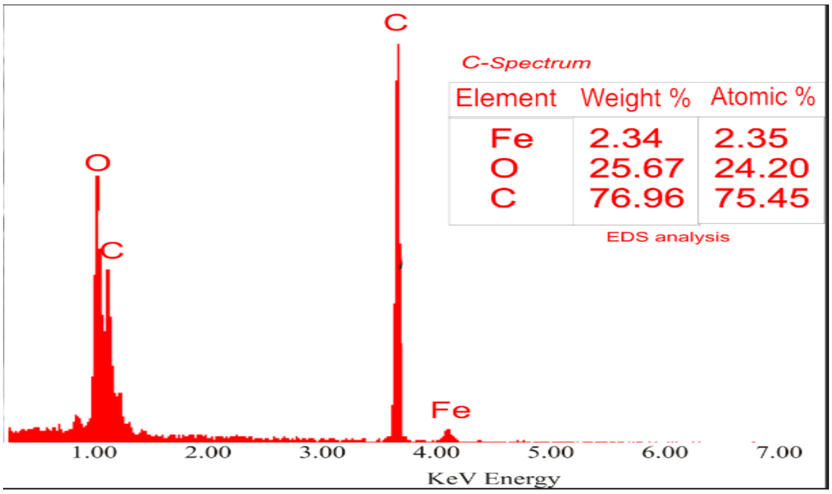

Figure 2: EDX Spectrum of CASAC

Characteristics of the green-synthesised TiO2 nanoparticles and composite

TEM was employed as one of the reliable techniques for nanoparticles characterization to obtain high accuracy of the actual particle size and shape pattern (Droepenu et al., 2022). The size of the TiO_2NPs ranges from 2.80 to 5.80nm and the shape is seen to be spherical as revealed by the TEM micrograph in Figure 3. The SEM micrograph of TiO_2 NPs in Figure 3 reveals large and small spherical-shaped crystals and irregular clusters of agglomerated particles, which could be a result of the water content from the plant extracts used and drying conditions employed. (Singh et al., 2020; Irshad et al., 2021; Shimi et al., 2022; Verma et al., 2022; Droepenu et al., 2022).

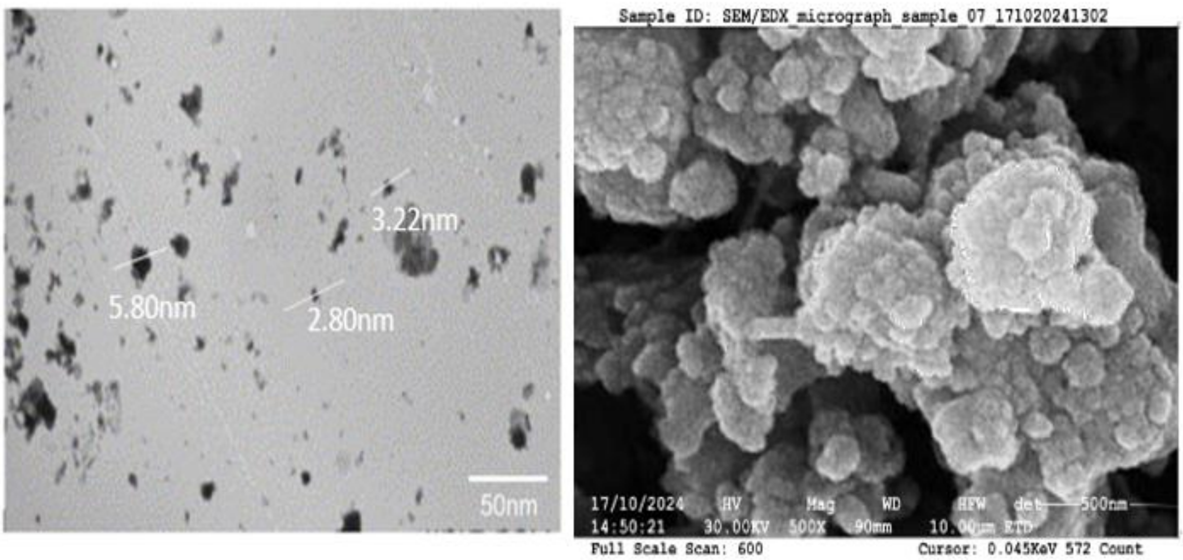

Figure 3: TEM and SEM micrograph of TiO_2 NPs

According to literature, green synthesized TiO_2 NPs often contain large crystal structures and it could be because of the phytochemicals present in the capping agent used for its synthesis (Irshad et al., 2021). The SEM micrograph of TiO_2 NPs – CASAC in Figure 4 also showed an irregular surface with several mesopores and micropores, agglomerated clusters, and some spherical particles. TiO_2 NPs – CASAC reveals surfaces that are filled with crevices and pores that could improve the adsorption efficiency of the composite.

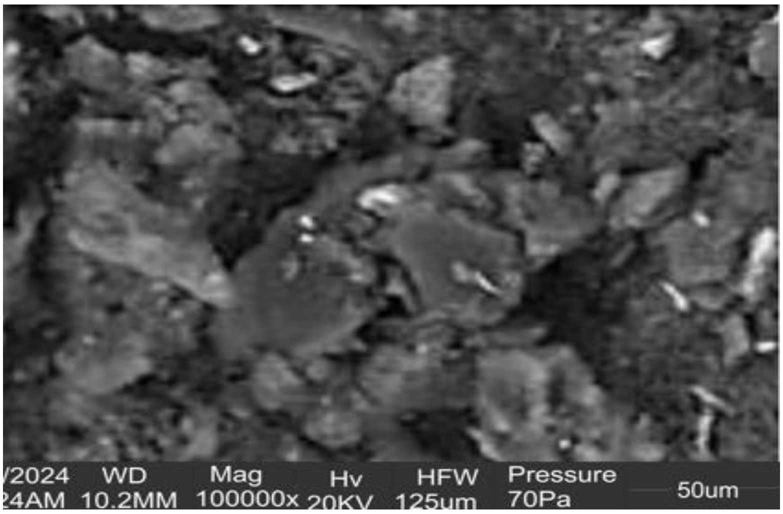

Figure 4: SEM micrograph of TiO2 NPs-CASAC

Based on the atomic weight composition of TiO_2 NPs, high peaks of oxygen and titanium were observed in the EDX spectrum of TiO_2 NPs in Figure 5 (Shimi et al., 2022). The EDX spectrum of the TiO_2 NPs – CASAC in Figure 5 also confirms the presence of titanium and oxygen. Also, low peaks of companion or ally elements such as S, Mg, Ca, Cl, Si, and phosphorus were observed, and this could indicate sustainability and efficiency of the synthesis method (Irshad et al., 2021).

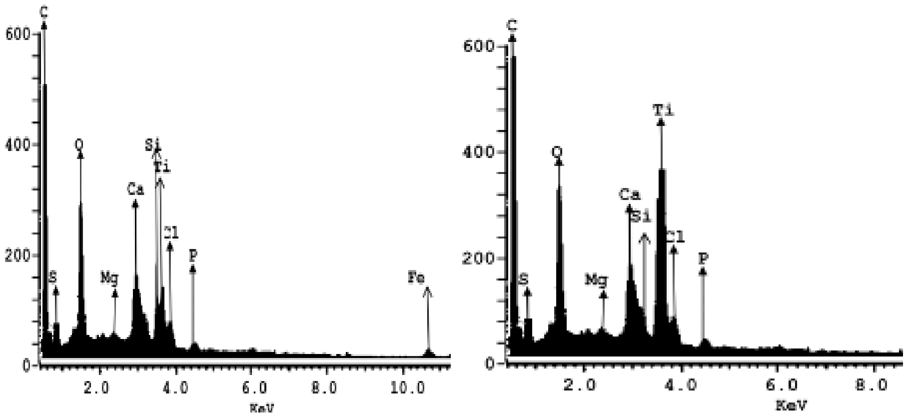

Figure 5: EDX spectrum of (A)TiO_2 NPs-CASAC and (B)TiO_2 NPs

CONCLUSION

This study observed the morphology of Chrysophyllum albidum seed shell activated carbon, green synthesized TiO2 nanoparticles and their composite. The SEM micrographs reveal the presence of several pores and cavities in the activated carbon, the nanoparticles, and the composite, which will enhance infiltration of adsorbate solution through the pores and therefore, promote adsorption capacities. The presence of oxygen will also aid chemical interactions and thus improve the adsorption capacity.

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