

Synthesis and Characterization of CuO-Loaded Activated Carbon Derived from Cashew Nut Shells for Methylene Blue Removal

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Abstract

In this study, a copper oxide-loaded activated carbon (AC–CuO) nanocomposite was synthesized using cashew nut shells as a low-cost carbon precursor for the photocatalytic degradation of methylene blue (MB). The prepared nanocomposite was characterized using XRD, FTIR, HRTEM, BET, zeta potential, TGA, and UV–Visible spectroscopy. XRD confirmed the crystalline nature of CuO with an average crystallite size of approximately 16 nm. HRTEM revealed well-dispersed CuO nanoparticles with distinct lattice fringes, confirming their nanoscale crystalline structure. BET analysis indicated a porous structure with a high surface area, while zeta potential measurements showed a negative surface charge favorable for interaction with MB molecules. The AC–CuO nanocomposite achieved 91% degradation of MB within 90 min and followed pseudo-first-order kinetics with a high correlation coefficient ($R^2 = 0.993$). Furthermore, it retained 90% degradation efficiency after five successive cycles, demonstrating good stability and reusability. The results indicate that the cashew nut shell-derived AC–CuO nanocomposite is a promising material for the photocatalytic treatment of dye-contaminated wastewater.

1. INTRODUCTION

The rapid growth of industrialization and urbanization has resulted in the widespread discharge of synthetic dyes into aquatic environments. Among these pollutants, methylene blue (MB), a cationic dye widely used in the textile, paper, and leather industries, has attracted significant attention due to its persistence, non-biodegradability, and potential toxicity to living organisms [1]. Even at low concentrations, MB imparts intense coloration to water, reducing light penetration and disturbing aquatic ecosystems. Furthermore, prolonged exposure to MB can cause harmful effects such as eye irritation, nausea, and respiratory distress in humans. Conventional wastewater treatment methods, including adsorption, coagulation–flocculation, and biological degradation, often prove ineffective in completely removing MB due to its stable aromatic structure and high solubility [2]. These limitations have prompted the exploration of advanced treatment technologies that are not only efficient but also environmentally sustainable [3].

Photocatalysis has emerged as a promising technique for the degradation of organic pollutants, offering the advantages of complete mineralization, reduced secondary pollution, and the use of solar energy as a renewable driving force. Semiconductor metal oxides such as TiO_2 , ZnO , and CuO have been extensively investigated as photocatalysts because of their strong light absorption, stability, and ability to generate electron–hole pairs under irradiation [4]. Among these, copper oxide (CuO), a p-type semiconductor with a narrow band gap ($\sim 1.2\text{--}1.7\text{ eV}$), has attracted increasing attention for visible-light-driven photocatalysis [5]. CuO nanoparticles exhibit excellent electrical conductivity, chemical stability, and catalytic properties, making them highly suitable for wastewater treatment applications. However, challenges such as particle agglomeration, limited surface area, and rapid recombination of photogenerated electron–hole pairs restrict the standalone efficiency of CuO as a photocatalyst [6]. To overcome these limitations, researchers have explored the use of supports or composite materials to stabilize CuO nanoparticles and enhance their photocatalytic performance [7].

Activated carbon (AC) is particularly appealing in this regard due to its high surface area, tunable porosity, and ability to adsorb organic pollutants. By serving as a host matrix, AC not only prevents nanoparticle aggregation but also provides abundant surface-active sites for pollutant adsorption and subsequent degradation [8]. Moreover, its high electrical conductivity facilitates charge transfer, suppressing electron-hole recombination and thereby improving photocatalytic efficiency. Thus, coupling CuO nanoparticles with activated carbon offers a synergistic effect by combining the adsorption capacity of AC with the photocatalytic activity of CuO[9].

Traditionally, activated carbon has been prepared from petroleum-based or expensive precursors, raising concerns regarding cost and environmental sustainability. In recent years, increasing attention has been focused on the utilization of agricultural and biomass wastes as renewable precursors for activated carbon production [10]. Cashew nut shells (CNS), an abundant agro-waste by-product of the cashew processing industry, have emerged as a promising raw material owing to their high carbon content, low cost, and natural availability in tropical countries [11]. Improper disposal of CNS often leads to environmental problems, while their conversion into activated carbon not only helps address waste management issues but also supports circular economy practices. Activated carbon derived from CNS hereafter referred to as cashew nut shell activated carbon (CNSAC), has been reported to exhibit well-developed porosity, high adsorption capacity, and abundant surface functionalities, making it an excellent candidate for supporting nanomaterials in catalytic applications.

2. MATERIALS AND METHODS

2.1 MATERIALS

Copper(II) nitrate trihydrate [$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$] sodium hydroxide (NaOH), and Methylene Blue (MB) dye were procured from reputable chemical suppliers. Cashew nut shells, sourced from local agricultural waste, were thoroughly cleaned to remove debris and impurities before processing. All reagents were of analytical grade and used without further purification.

2.1.1 Preparation of Activated Carbon from Cashew Nut Shells

Cashew nut shells used in this study were collected from Virudhachalam Taluk, located in the Cuddalore district of Tamil Nadu, India. The shells were thoroughly washed with Distilled water to remove adhering dust, dirt, and organic residues. After cleaning, they were oven-dried at 110 °C for 24 hours to ensure complete removal of moisture. The dried shells were then ground and sieved to obtain particles smaller than 2 mm, which served as the raw material for activated carbon preparation.

Chemical activation was carried out using sodium hydroxide (NaOH) as the activating agent due to its effectiveness in enhancing the surface area and porosity of the carbon material. Methylene Blue (MB), a cationic dye with the molecular formula $\text{C}_{16}\text{H}_{18}\text{N}_3\text{SCL}$, was employed as a model pollutant to evaluate the adsorption efficiency of the synthesized activated carbon.

All reagents, including NaOH and hydrochloric acid (HCl), were of analytical grade ($\geq 99\%$ purity) and procured from SD Merck Fine Chemicals, India. The chemicals were used without further purification. Prior to use, all glassware was acid-washed, and only deionized water was utilized throughout the study to prevent contamination and ensure consistency during solution preparation and sample dilution.

2.1.2 Synthesis of CuO Nanoparticles on Activated Carbon

To synthesize CuO-loaded activated carbon, an aqueous solution of copper(II) nitrate trihydrate was added dropwise to a suspension of cashew nut shell-derived activated carbon under continuous stirring. Sodium hydroxide (NaOH) was then added gradually to maintain a basic pH, promoting the precipitation of CuO nanoparticles. The resulting mixture was aged, filtered, and thoroughly washed with deionized water to remove residual impurities. The obtained product was dried and subsequently calcined at 400 °C to achieve the formation of crystalline CuO nanoparticles uniformly distributed on the activated carbon surface.

3. RESULTS AND DISCUSSION

3.1 TGA / DTA analysis

The thermal behavior of the CNSAC–CuO nanocomposite was investigated using thermogravimetric analysis (TGA) and differential thermal analysis (DTA), as shown in Figure 1. The TGA curve exhibits a multi-step weight-loss pattern as the temperature increases from room temperature to 1000 °C. The initial minor weight loss below 150 °C can be attributed to the evaporation of physically adsorbed water molecules and moisture present on the surface of the sample [15]. A sharp and significant weight loss is observed between 200 °C and 300 °C, which corresponds to the decomposition of organic functional groups and residual volatile matter present in the activated carbon matrix. This transition is accompanied by a strong exothermic peak in the DTA curve, suggesting the combustion of carbonaceous species and the release of trapped gases. Beyond 300 °C, a gradual and continuous weight loss is observed up to 800 °C, which can be associated with the slow degradation of more stable oxygen-containing groups, such as carboxyl, carbonyl, and phenolic groups, along with partial structural rearrangements within the activated carbon [16].

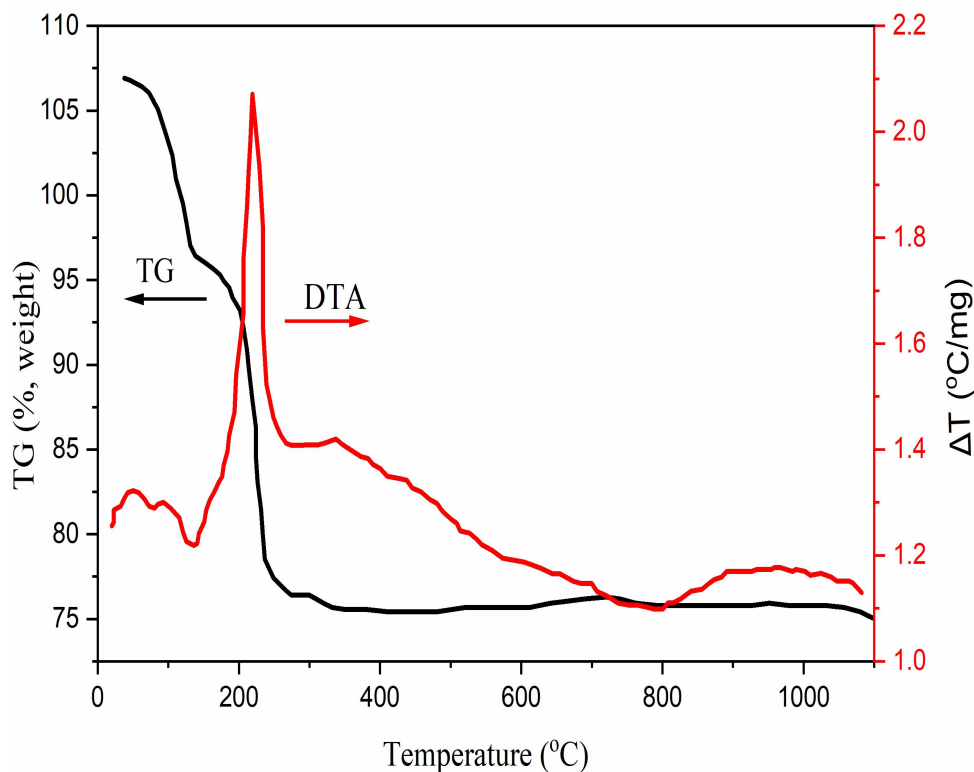


Figure 1. TG/DTA curves of the CNSAC-CuO nanocomposite.

The relatively stable profile above 800 °C indicates the thermal stability of the CNSAC-CuO nanocomposite. The final residual weight confirms the presence of thermally stable CuO nanoparticles embedded within the carbon matrix, demonstrating their ability to withstand high temperatures without significant decomposition. The thermal stability of the CNSAC-CuO nanocomposite is significantly improved compared with that of pure activated carbon, as the CuO nanoparticles act as reinforcing agents that

inhibit the complete burnout of the carbon structure. This synergistic effect enhances thermal durability and may contribute to the suitability of the material for high-temperature applications. In addition, the strong exothermic feature observed in the DTA profile reflects the catalytic effect of CuO in promoting the oxidation of carbonaceous species, which may be beneficial for photocatalytic and catalytic applications.

3.2 X-RAY DIFFRACTION ANALYSIS

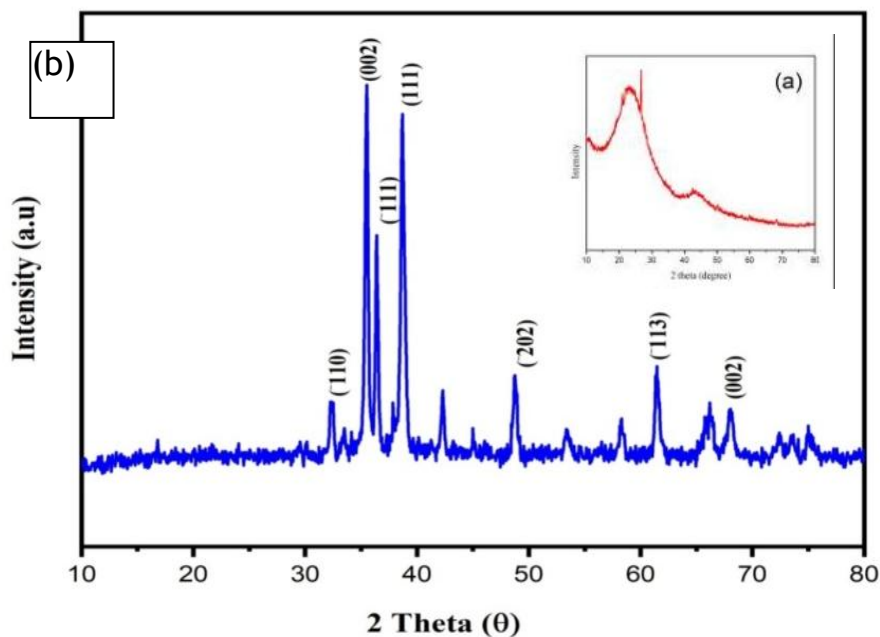


Figure 2. XRD patterns of (a) pure AC derived from cashew nut shells and (b) CNSAC-CuO nanocomposite

The X-ray diffraction (XRD) patterns of pure activated carbon (AC) derived from cashew nut shells and the CNSAC-CuO nanocomposite are

shown in Figure 2(a) and 2(b), respectively. The broad diffraction hump observed for pure AC (Figure 2a) in the range of 20°–30° (2θ) indicates the

amorphous nature of the carbon structure. In contrast, the CNSAC–CuO nanocomposite (Figure 2b) exhibits several sharp and intense diffraction peaks, confirming the successful incorporation and crystalline nature of CuO nanoparticles within the carbon matrix. The prominent diffraction peaks at 2θ values of approximately $32.4^\circ, 35.4^\circ, 35.4^\circ, 38.7^\circ, 48.7^\circ, 61.5^\circ$ and 65.7° correspond to the (-110) , (002) , (-111) , (111) , (-202) , (-113) , and (002) planes of monoclinic CuO, which are in good agreement with JCPDS Card No. 45-0937 [17]. The absence of additional impurity peaks suggests that the synthesized CuO nanoparticles are phase-pure. The average crystallite size of the CuO nanoparticles, calculated using the Debye–Scherrer equation [18], was found to be 16 nm, confirming their nanocrystalline nature.

Compared with chemically synthesized CuO nanoparticles reported in the literature, the present biosynthesis approach using cashew nut shell-derived activated carbon not only produced smaller-sized particles but also offered improved crystallinity[19]. The carbon matrix derived from cashew nut

shells plays a dual role: (i) acting as a reducing and stabilizing agent during the synthesis of CuO nanoparticles, and (ii) providing a high-surface-area support that enhances nanoparticle dispersion and prevents agglomeration. This sustainable route thus demonstrates a green and low-cost alternative to conventional chemical synthesis.

The incorporation of CuO nanoparticles into activated carbon significantly enhances its functional properties. In particular, the CNSAC–CuO nanocomposite exhibits promising photocatalytic potential due to the synergistic interaction between CuO and the carbon matrix. The high surface area of activated carbon facilitates the efficient adsorption of dye molecules, while the CuO nanoparticles promote photodegradation under visible-light irradiation. This combined mechanism makes the material a suitable candidate for wastewater treatment applications, particularly for the removal of toxic organic dyes.

3.3 FTIR Analysis

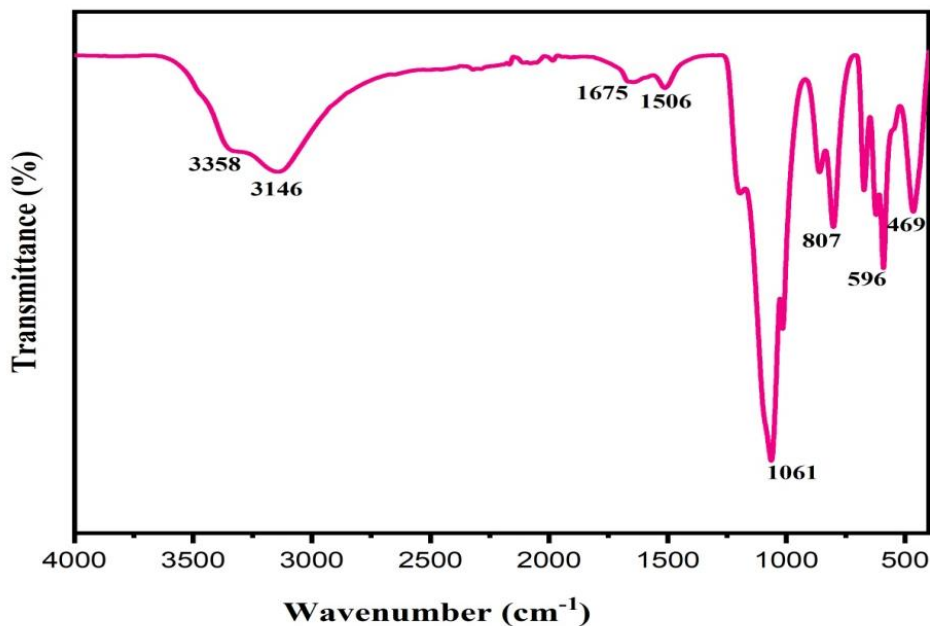


Figure 3. FTIR spectra of the CNSAC–CuO nanocomposite.

Figure 3 displays the FTIR spectra of the CNSAC–CuO nanocomposite, highlighting several distinct absorption bands. The broad bands at 3358 cm^{-1} and 3146 cm^{-1} are attributed to O–H stretching vibrations, indicating the presence of adsorbed water or hydroxyl groups on the surface of the nanocomposite[20]. The weak peaks at 1675 cm^{-1} and 1506 cm^{-1} correspond to C=O and C=C stretching vibrations, respectively, likely arising from carbonaceous components in the composite [21]. The prominent peak at 1061 cm^{-1} can be assigned to C–O stretching vibrations. Most notably, the strong absorption bands at 807 cm^{-1} , 596 cm^{-1} , and 469 cm^{-1} are characteristic of Cu–O stretching vibrations, confirming the formation of copper oxide within the activated carbon matrix [22].

The presence of O–H and C–O-related vibrations demonstrates that the CNSAC–CuO nanocomposite contains surface functional groups capable of facilitating interactions with water and organic molecules, which may be beneficial for catalytic and adsorption processes. The sharp bands attributed to Cu–O vibrations at 807 cm^{-1} , 596 cm^{-1} , and 469 cm^{-1} confirm the successful formation of the CuO phase, as these values are in good agreement with reported literature values for monoclinic CuO. The

observed carbonyl and aromatic C=C peaks at 1675 and 1506 cm^{-1} suggest the presence of residual carbon functionalities from the activated carbon component, which may further contribute to surface reactivity. Collectively, these spectral features indicate that the CNSAC–CuO nanocomposite possesses rich surface chemistry and a well-defined CuO structure, supporting its potential for photocatalytic and environmental applications.

3.4 UV–Visible Analysis

The optical properties of the CNSAC–CuO nanocomposite were examined using UV–Visible spectroscopy, and the results are shown in Figure 4(a). The absorption spectrum exhibits a sharp and intense absorption peak centered at 293 nm , which corresponds to the electronic transition of CuO nanoparticles [23]. The strong absorption in the UV region indicates the formation of nanosized CuO particles and quantum confinement effects [24]. The broad absorption tail towards the visible region further confirms the effective interaction between CuO nanoparticles and the activated carbon matrix, suggesting their potential for photocatalytic activity under solar irradiation.

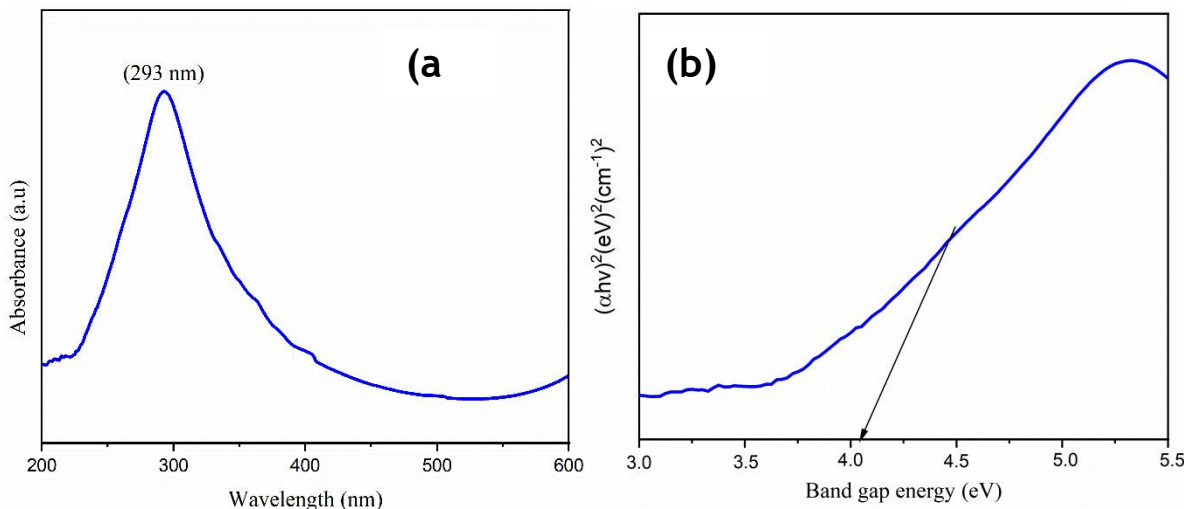


Figure 4. (a) UV-Visible spectrum and (b) band gap energy of the CNSAC-CuO nanocomposite.

The optical band gap of the CNSAC-CuO nanocomposite was determined using the Tauc relation, $(\alpha h\nu)^2 = A(h\nu - E_g)$, where α is the absorption coefficient, $h\nu$ is the photon energy, and E_g is the band gap energy. The extrapolation of the linear region of the Tauc plot (Figure 4b) gives a band gap value of 4.0 eV. This relatively higher band gap compared with bulk CuO (~1.2–1.7 eV) can be attributed to the nanoscale size of the CuO particles, which induces strong quantum confinement [25]. The presence of activated carbon plays a vital role in enhancing light absorption and charge separation. The porous structure of carbon increases the effective surface area, while its conductive nature facilitates electron transfer from the excited CuO nanoparticles, thereby suppressing the recombination of electron-hole pairs. This synergistic effect between activated carbon and CuO not only tunes the optical properties but also improves the photocatalytic efficiency of the CNSAC-CuO nanocomposite for dye degradation and other environmental remediation applications.

3.5 ZETA POTENTIAL AND DLS ANALYSIS

The surface charge and stability of the CNSAC-CuO nanocomposite were examined using zeta potential analysis (Figure 5a). The measured zeta potential value was -27 mV, indicating that the nanocomposite carries a moderately high negative surface charge. Generally, zeta potential values greater than ± 25 mV are considered to provide good electrostatic repulsion, thereby preventing particle aggregation [26]. Hence, the obtained value confirms that the CNSAC-CuO nanocomposite exhibits appreciable colloidal stability in aqueous suspension. The negative surface charge originates from the adsorption of hydroxyl and oxygen-containing functional groups on the nanocomposite surface, which is further enhanced by the activated carbon matrix.

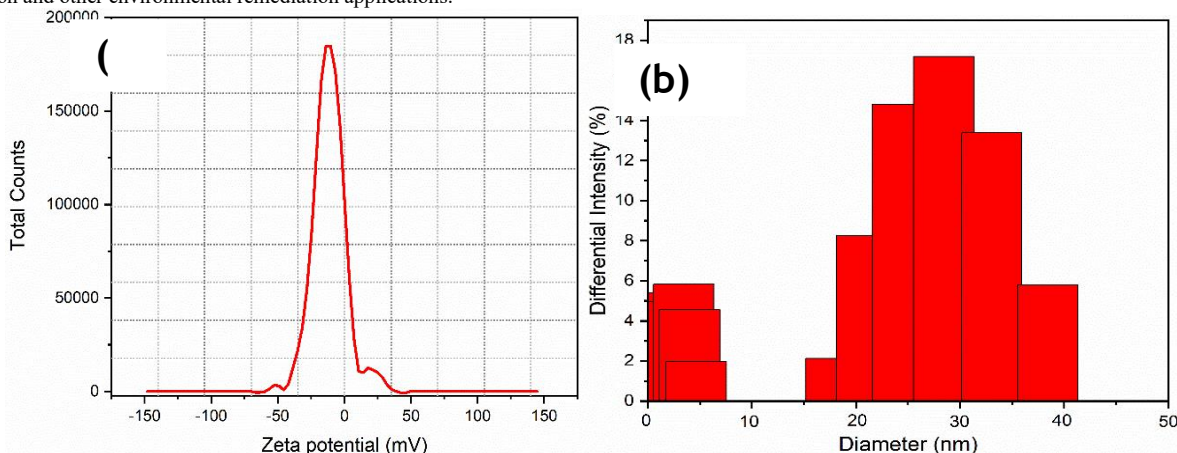


Figure 5. (a) Zeta potential and (b) DLS size distribution of the CNSAC-CuO nanocomposite.

Dynamic light scattering (DLS) analysis was performed to determine the hydrodynamic particle size distribution of the CNSAC-CuO nanocomposite (Figure 5b). The histogram indicates a bimodal distribution, with particle sizes mainly ranging between 20–35 nm and a significant population around 28–30 nm. A small fraction of particles in the 5–10 nm range was also detected, which can be attributed to well-dispersed CuO nanocrystals embedded within the porous carbon structure. The observed particle sizes are slightly larger than the crystallite size calculated from XRD (~16 nm), which is expected because DLS measures the hydrodynamic diameter, including solvation layers and possible clustering of nanoparticles in suspension.

The combination of zeta potential and DLS results confirms the successful formation of the nanosized CNSAC-CuO nanocomposite with moderate stability in aqueous dispersion. The particle size range and stabilization mechanism make the nanocomposite highly suitable for photocatalytic

applications, where both dispersion and surface reactivity play critical roles in enhancing dye degradation efficiency.

3.6 HR-TEM Analysis

The HR-TEM images presented in Figure 6a and Figure 6b show that the CNSAC-CuO nanocomposite is predominantly spherical in morphology and forms loosely aggregated clusters, with a uniform distribution across the imaging field. The high-resolution micrograph (Figure 6b) reveals clear lattice fringes, highlighted by yellow arrows, confirming the crystalline nature of the CuO nanoparticles [17]. These well-defined interplanar spacing validate the successful incorporation of crystalline CuO within the activated carbon matrix.

The particle size histogram (Figure 6c) indicates that the nanoparticles are distributed primarily between 10 and 40 nm, with a mean diameter of 23

nm. The relatively narrow size distribution demonstrates that the synthesis protocol effectively controlled particle growth and prevented excessive aggregation, yielding a uniform nanostructure. The TEM observations confirm that the CNSAC–CuO nanocomposite exhibits nanoscale dimensions, high crystallinity, and uniform morphology—all desirable features for catalytic, electronic, and biomedical applications. The mean particle size of 23 nm is in good agreement with earlier reports on CuO-based nanomaterials, suggesting that the cashew nut shell (CNS)-derived activated carbon effectively acted as both a stabilizing and dispersing agent during nanoparticle formation.

Furthermore, the presence of well-defined lattice fringes indicates that the nanoparticles possess a highly ordered crystal structure, which is essential

for enhancing activity and stability in functional applications. The narrow size distribution enhances reproducibility and ensures consistent performance in repeated catalytic cycles. Importantly, the nanoscale size, high crystallinity, and large surface-to-volume ratio of the CNSAC–CuO nanocomposite contribute directly to its photocatalytic efficiency. Smaller, uniformly sized nanoparticles provide abundant active sites, facilitate efficient light absorption, and improve electron–hole separation while reducing charge recombination. These structural and morphological characteristics collectively enhance photocatalytic degradation processes, highlighting the CNSAC–CuO nanocomposite as a promising candidate for environmental remediation and other advanced nano technological applications.

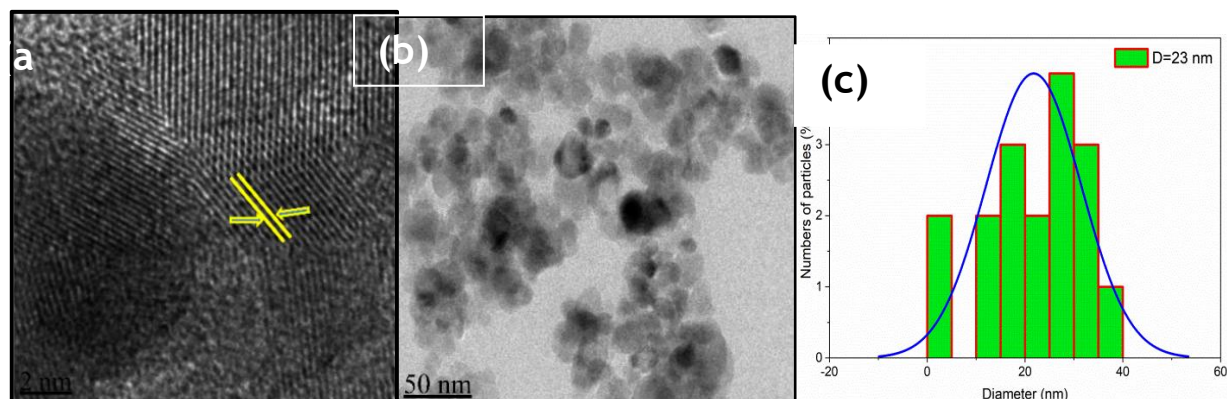


Figure 6. (a–b) HR-TEM images and (c) particle size histogram of the CNSAC–CuO nanocomposite.

3.7 BET Analysis

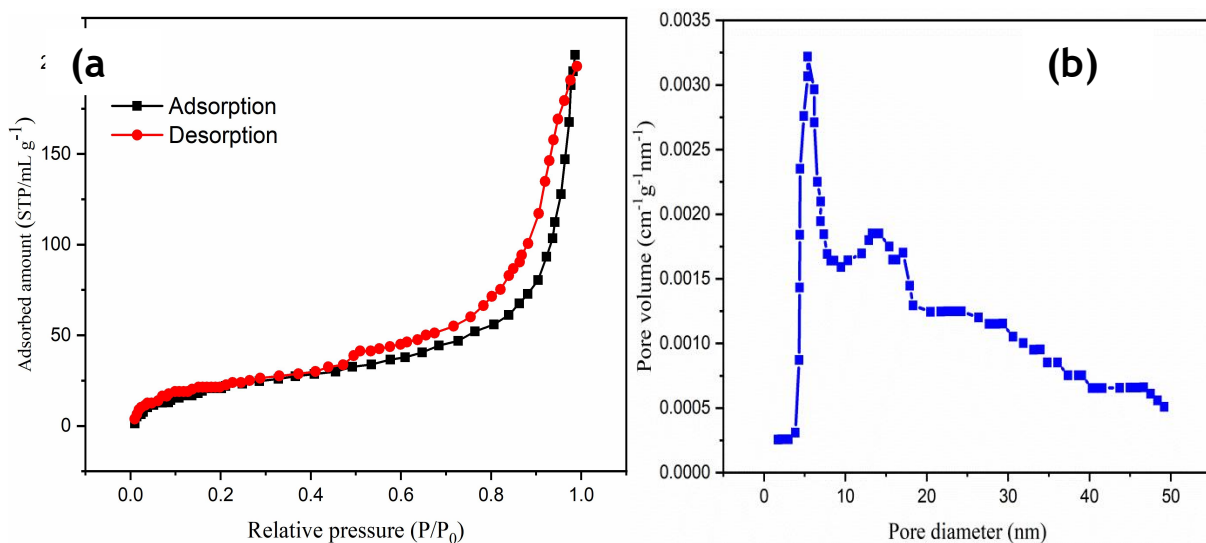


Figure 7. (a) BET surface area and (b) BJH pore size distribution analysis of the CNSAC–CuO nanocomposite.

Figure 7a presents the N_2 adsorption–desorption isotherms of the CNSAC–CuO nanocomposite, which exhibit a Type IV curve with an H3 hysteresis loop, characteristic of mesoporous materials with slit-like pores [27]. The corresponding BJH pore size distribution (Figure 7b) shows a predominant pore diameter of about 5 nm, with a secondary broad distribution around

13–15 nm extending up to 50 nm, confirming the mesoporous nature of the nanocomposite. Such a mesoporous structure provides more active sites and facilitates the diffusion of dye molecules to the catalyst surface, which supports efficient photocatalytic degradation

3.9 PHOTOLUMINESCENCE Analysis

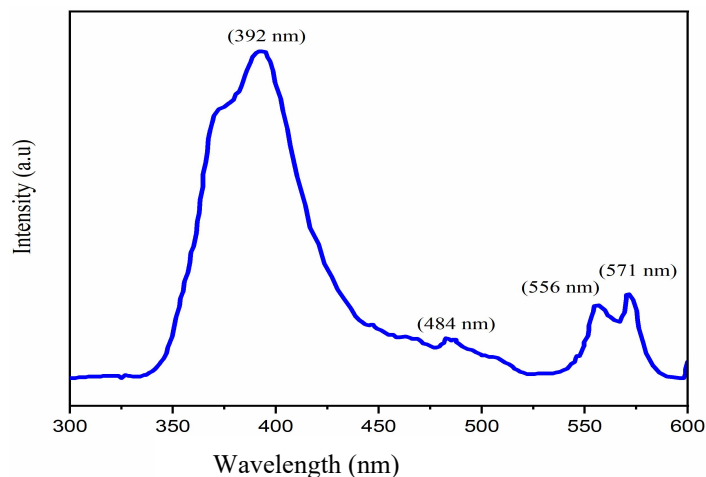
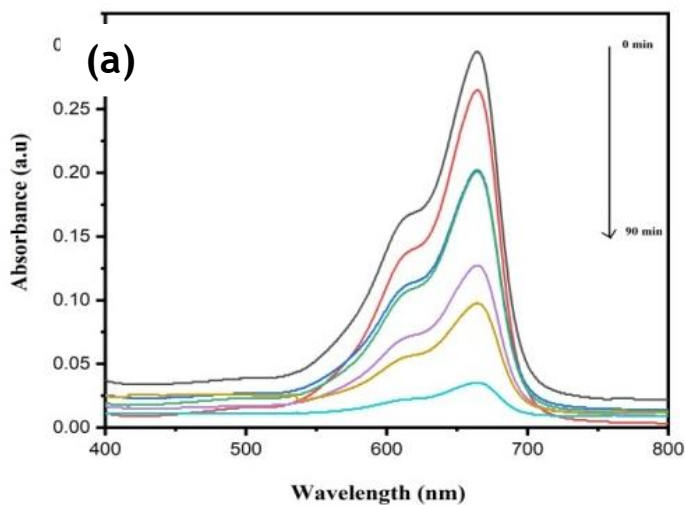


Figure 8. PL spectrum of the CNSAC-CuO nanocomposite.

Figure 8 presents the photoluminescence spectrum of the CNSAC-CuO nanocomposite, which displays a strong emission peak at 392 nm along with weaker emissions at 484, 556, and 571 nm. The intense peak at 392 nm corresponds to near-band-edge transitions of CuO nanoparticles [28]. The weaker emissions at longer wavelengths are commonly associated with intrinsic defects such as oxygen vacancies, surface traps, or interfacial states [29]. Although defects are typically linked to recombination centers, in photocatalytic materials such defect states can trap photogenerated charge carriers and prolong their lifetime, thereby reducing charge recombination and supporting photocatalytic activity [30].

3.10 PHOTOCATALYTIC STUDY

Figure 9(a) presents the UV-Visible absorption spectra of MB dye over a wavelength range of 400–800 nm, showing a distinct absorption peak at around 665 nm, with a shoulder near 615 nm that is characteristic of MB. The uppermost spectrum corresponds to the initial MB solution without the catalyst. The spectra recorded at 15 min intervals from 15 to 90 min under sunlight in the presence of the CNSAC-CuO nanocomposite show a gradual reduction in absorbance as the photodegradation of MB dye progresses. The CNSAC-CuO nanocomposite achieved a degradation efficiency of approximately 90% after 90 min. This decrease in absorbance indicates the degradation of the dye over time, highlighting the effectiveness of the CNSAC-CuO nanocomposite in degrading MB through the photodegradation process. As the reaction time increases, the absorbance peak gradually diminishes, confirming the decolorization of MB.



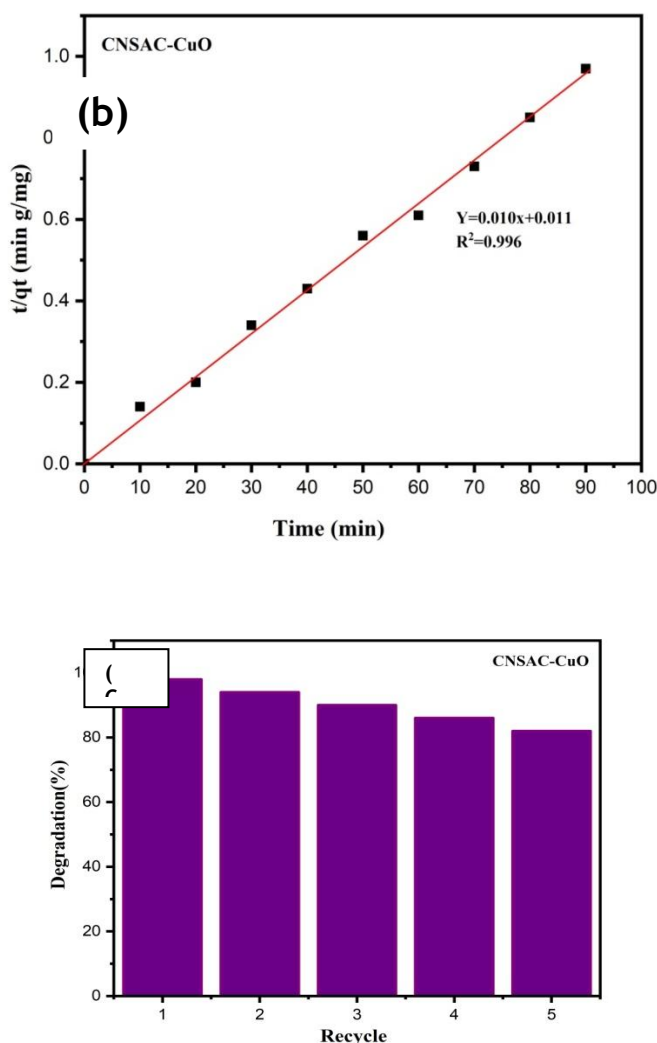


Figure 9. (a) UV–Visible absorption spectra, (b)Pseudo second order model for the adsorption of MB, and (c) reusability of the CNSAC–CuO nanocomposite.

Figure 9(b) illustrates the plot of t/q_t against contact time, representing the adsorption kinetics of MB dye onto the CNSAC–CuO nanocomposite in the dark. A linear relationship is observed, and the linear regression equation, $y = 0.010x + 0.011$, with an R^2 value of 0.996, suggests a strong fit. This indicates that the adsorption follows pseudo-second-order kinetics, where the adsorption rate depends on the number of available active sites on the catalyst surface. This supports the conclusion that MB is efficiently adsorbed onto the CNSAC–CuO surface, which facilitates the subsequent photocatalytic degradation.

Figure 9(c) shows the reusability of the CNSAC–CuO nanocomposite over five cycles of degradation. The degradation efficiency remains high, decreasing only slightly from approximately 97% in the first cycle to approximately 91% in the fifth cycle. This suggests that the CNSAC–CuO nanocomposite possesses good stability and reusability, which are important factors for practical applications in wastewater treatment [31]. The slight decrease in degradation efficiency over successive cycles can be attributed to possible surface deactivation or loss of active sites on the nanocomposite, which is a typical behavior in catalyst reuse studies.

4. CONCLUSION

In this study, a CNSAC–CuO nanocomposite was synthesized from cashew nut shell-derived activated carbon. XRD confirmed the crystalline nature of CuO with an average crystallite size of 16 nm, and TGA/DTA showed good thermal stability. UV–Visible and PL

analyses indicated strong UV absorption and suitability for photocatalytic use. The nanocomposite degraded 91% of MB dye within 90 min under sunlight, and the adsorption of MB onto the nanocomposite followed the pseudo-second-order model. It also retained about 91% efficiency after five cycles. Overall, CNSAC–CuO is a promising and reusable material for dye degradation and wastewater treatment.

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