

Research Article

CuO/NiO-doped ordered mesoporous carbon for high-performance photocatalytic degradation of dye and energy storage application

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ABSTRACT

Advanced nanomaterials with optimized structures, surfaces, porous characteristics, optimum band gaps, and compositions are crucial for wastewater treatment and energy storage applications. Here, we report the soft-template synthesis of CuO/NiO-doped ordered mesoporous carbon (OMC/CuO/NiO), which exhibits a synergistic enhancement in photocatalytic and electrochemical performance. The structure testing verified the creation of regular mesoporous and effective doping of CuO and NiO into the Carbon framework. The photocatalytic activity of the nanocomposite studied was determined by means of the degradation of methylene blue (MB) dye in the presence of UV-visible light. Light absorption and enhanced charge separation increased the degradation efficiency of the OMC/CuO/NiO nanocomposite to 96 % (improved compared to pure OMC of 55.07 %) and facilitated light absorption and charge separation, respectively. In addition to photocatalysis, OMC/CuO/NiO nanocomposite electrode shows excellent performance as a Supercapacitor with a specific capacitance of 564.2 F g⁻¹ (at 5 mVs⁻¹) and 559.1 F g⁻¹ (at 0.9 Ag⁻¹), which is 3.1-fold higher than undoped OMC (179.7 F g⁻¹). Besides, GCD profiles reveal the energy density of 49.6 Wh kg⁻¹ and power density of 359.9 W kg⁻¹, which confirms the potential of the material in the fast-charge-discharge processes. Mechanistic studies validate the fact that synergistic redox interactions between Cu and Ni species facilitate the separation of electrons and holes, the production of reactive oxygen species (ROS), and the enhanced photocatalytic activity. These findings make OMC/CuO/NiO a second-generation dual functional material in the form of sustainable wastewater treatment and superior energy storage technologies.

1. Introduction

The need to use clean water and sustainable energy has created an acute demand of multidimensional materials that can be used to address the environmental and energy concerns [1]. Water pollution has become a serious environmental issue nowadays, which is threatening ecosystems and human health [2–4]. Synthetic dyes are some of the various water contaminants that cause significant problems due to toxicity, chemical stability and biodegradation resistance [5–8]. At the same time, the requirement for sustainable energy technology has increased primarily due to the worldwide energy crisis caused by the depletion of fossil fuels. [9–11]. Therefore, to deal with both challenges, there is a requirement for multifunctional materials that are capable of both pollutant degradation and energy storage.

Photocatalysis is an effective method for treating wastewater. In this process, organic pollutants change into non-toxic chemicals using

energy from the sun. Reactive species, which are created by semiconductors activated by light energy, break down complex contaminants into harmless byproducts [12]. The next-generation energy storage solutions include supercapacitors, or electrochemical capacitors [13,14]. They offer excellent power density, a long cycle life, and fast charge-discharge capabilities, designing nanomaterials with precisely controlled structural and electronic properties that support dual functionality is an important area of research. For instance, these materials can improve energy conversion efficiencies while also offering photonic properties for use in advanced optoelectronic devices and renewable energy technologies. Although there has been considerable progress in developing photocatalysts and supercapacitors, many existing materials struggle with low light absorption, poor charge separation, and limited electrochemical stability. Most current systems do not reach high photocatalytic efficiency (>90 %) and specific capacitance over 500 F g⁻¹. This leaves a significant gap in research for multifunctional materials

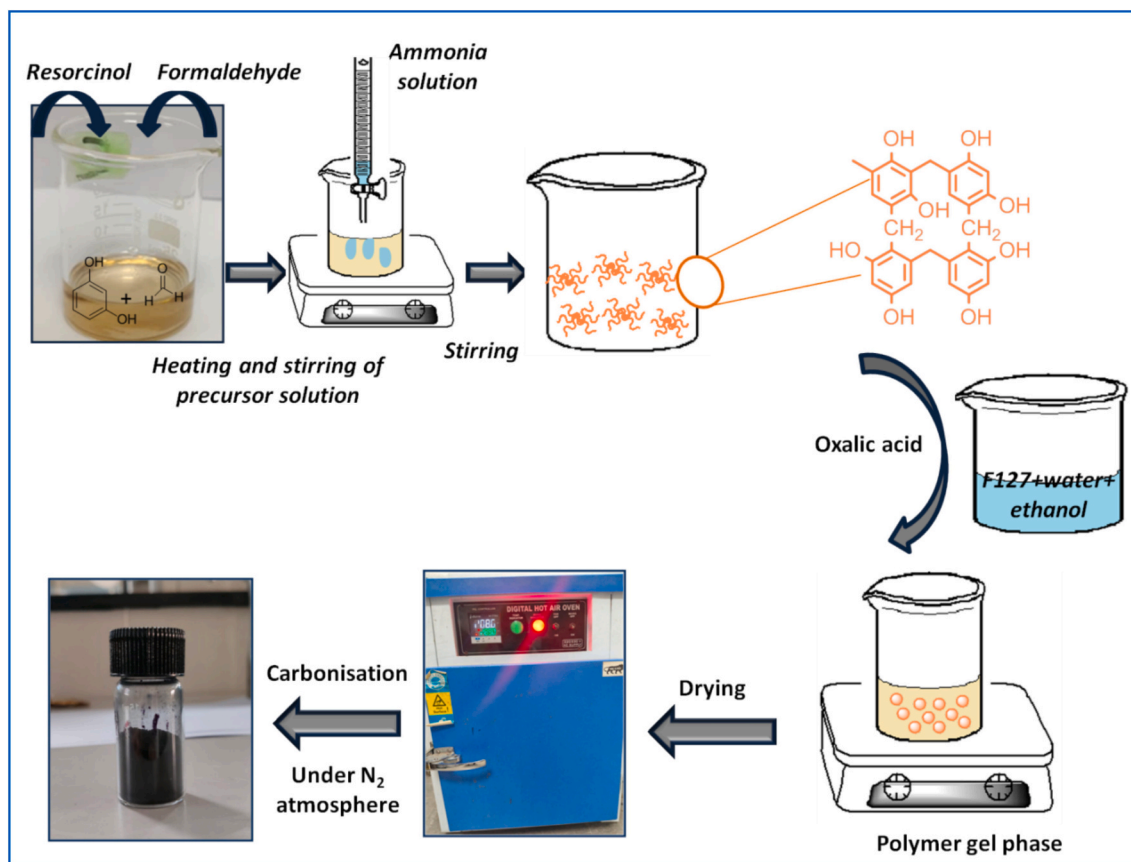
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Scheme 1. Synthetic scheme of Ordered Mesoporous Carbon (OMC).

that can tackle both challenges.

Among various porous carbons, OMC has recently attracted extensive attention as a versatile energy and environmental material with superior performance to conventional porous carbons including activated carbon, CNTs, and graphene. Unlike high-cost and complicated synthesis of CNTs and graphene, OMC can be prepared by cost-effective templating methods [15]. Well-defined mesoporous structure, large surface area, and tunable pore size of OMC enable fast ion diffusion and efficient electron transport, both of which are crucial for both photocatalysis and high-performance supercapacitors. However, pristine OMC usually shows very limited catalytic and electrochemical activity. In this regard, dual-metal doping of Cu and Ni nanoparticles was adopted to introduce abundant redox-active sites and enhance the conductivity. These synergistic $\text{Cu}^{2+}/\text{Cu}^{+}$ and $\text{Ni}^{3+}/\text{Ni}^{2+}$ redox couples further enhance charge transfer, light absorption, and energy storage efficiency [16,17], tunable pore sizes, and outstanding chemical stability. These characteristics promote faster ion diffusion, better electrolyte access, and efficient electron transport-critical parameters for photocatalysis and electrochemical energy storage [18,19].

Soft-templating methods that use block-copolymer, like Pluronic F127 or P123, along with phenol-formaldehyde precursors, provide a more scalable, practical, environmentally friendly, and flexible way to create ordered mesoporous carbon with clear mesostructures. As a result, OMC is a more reliable and structurally beneficial choice than many other carbon-based systems for integrated photocatalytic and electrochemical applications [16,20].

Transition metal oxides such as TiO_2 , ZnO , and MnO_2 have been widely evaluated for photocatalytic or super-capacitive applications; however, their practical performance is often limited by either poor electrical conductivity or large band gaps [21]. To overcome these limitations, OMCs are often modified with transition metal oxides such as Copper oxide (CuO) and Nickel oxide (NiO) to improve their

photocatalytic and electrochemical performance [22,23]. CuO , a p-type semiconductor, has very small bandgap (1.2 to 2.1 eV), high oxygen adsorption capacity, and excellent chemical and thermal stability. NiO demonstrates significant redox activity, effective charge transport capabilities, and enduring electrochemical stability [21,24,25]. By adding these oxides to the OMC matrix, dye degradation and capacitance performance improve. Visible light absorption is better, charge separation becomes easier, and electroactive sites are introduced [26].

Recent studies on single-metal-doped OMC composites typically report photocatalytic efficiencies below 80 % and specific capacitances below 300 F g^{-1} [27,28]. Dual-metal systems have been attempted, but most achieve only moderate activity due to poor redox synergy. In this work, we report a soft-template synthesis of a CuO/NiO -doped ordered mesoporous carbon (OMC/ CuO/NiO) nanocomposite designed for dual functionality in photocatalysis and supercapacitors. By leveraging the synergistic redox activity of Cu and Ni within a conductive mesoporous framework, we achieve 96 % MB degradation under UV-visible light and a specific capacitance of 564.2 F g^{-1} from CV with 559.1 F g^{-1} from GCD, positioning this material among the highest-performing OMC-based systems reported so far.

2. Experimental section

2.1. Materials and methods

The reagents and solvents were used as received without further purification and were of analytical grade. Resorcinol, Pluronic F127 (PEO106-PPO70-PEO106), and metal nitrates, including Copper (II) nitrate trihydrate [$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$] and Nickel (II) nitrate hexahydrate [$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], were procured from Sigma-Aldrich. Formaldehyde (37 wt%), ethanol, oxalic acid, and ammonia solution were purchased from Merck (India). Deionized water was used throughout the study.

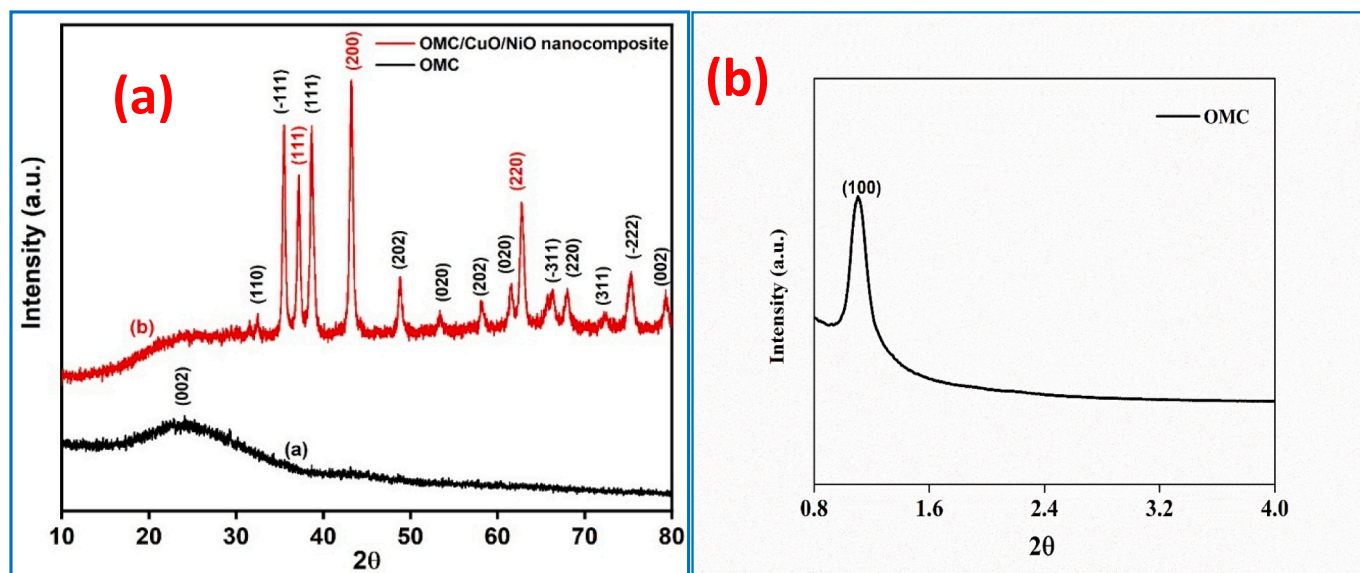


Fig. 1. (a) XRD patterns of OMC and OMC/CuO/NiO nanocomposite showing amorphous carbon structure and crystalline phases of CuO and NiO (b) SAXRD pattern of OMC.

Rigaku Ultima IV diffractometer (Japan) with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) was used to study the X-ray diffraction (XRD) pattern. Surface morphology and elemental composition were examined using a Zeiss Ultra Plus Field-Emission Scanning Electron Microscope with an energy-dispersive X-ray spectroscopy (EDX) detector. High-resolution Transmission electron microscopy (HRTEM) analysis was performed using a FEI Tecnai G2 (USA) at an accelerating voltage of 200 kV. Nitrogen adsorption-desorption measurements were conducted on a Belsorp Mini-X analyzer (BEL Japan Inc.) at 77 K after degassing the samples at 120 °C for 16 h. BET surface area and BJH pore distribution were calculated using BELSORP software.

The photocatalytic activity was evaluated by UV-Vis spectrophotometry (Sytronics Model 117, India) by observing the methylene blue (MB) degradation under UV-visible light. Electrochemical measurements were performed using Autolab PGSTAT 86472 electrochemical workstation in a three-electrode configuration, with an Ag/AgCl reference electrode and a Pt wire as the counter electrode. Cyclic voltammetry (CV) was performed in a 6 M KOH solution.

2.2. Synthesis of Ordered Mesoporous Carbon (OMC)

Synthesis was carried out using modified two-phase method to prepare a 2D hexagonal OMC [29]. Initially, a precursor solution was obtained by dissolving 2.0 g resorcinol in 2.8 mL formaldehyde solution (37 wt%), followed by stirring at 30 °C for 30 min to promote polymerization. After that, 2.5 mL of ammonia solution was added with continuous stirring to obtain RF resol. This was later mixed with a second solution containing 1.1 g of Pluronic F127, 0.2 g oxalic acid and 10 mL deionized water. Upon mixing, the stirring of the homogeneous system was done continuously. After 5–10 min, visible cloudiness was appeared which indicate the beginning of phase separation and micelle formation. To assist the self-assembly of mesophase continuous stirring was done for further 1 h.

The polymeric gel formed was dried at optimum conditions for at least 12 h which was followed by thermal curing at 85 °C for 18 h to stabilize the framework. During carbonization process, the heating rate was controlled at 1 °C min⁻¹ up to 800 °C under continuous flow of N₂ which ensure the complete removal of template residues and stabilization of the mesostructured (Scheme 1). The final product obtained was black mesoporous carbon having a 2D hexagonal framework.

Synthesis of CuO/NiO-Doped Ordered Mesoporous Carbon (OMC/

CuO/NiO).

OMC/CuO/NiO nanocomposites were prepared by post-synthetic metal impregnation further followed by thermal treatment. To avoid any contamination during the process, high-purity ethanol was used as a solvent. To ensure the homogeneous bimetallic distribution inside the mesoporous framework the Cu and Ni precursors were introduced in an equimolar ratio. In this synthetic procedure, 0.7 g OMC was dispersed in 12 mL of ethanol comprising 0.26 g Cu (NO₃)₂·3H₂O and Ni (NO₃)₂·6H₂O. To facilitate consistent metal ion adsorption onto the mesoporous structure, the mixture was stirred for one hour. The obtained slurry was dried at 70 °C under vacuum, after that calcined at 500 °C for 4.5 h under nitrogen atmosphere with a heating rate of 2 °C min⁻¹. This process resulted in integration and in-situ formation of CuO and NiO nanoparticles within the carbon matrix. The obtained OMC/CuO/NiO composite exhibited a black appearance and preserved the highly ordered mesostructure of pure OMC which was further confirmed by XRD and HRTEM analysis.

2.3. Photocatalytic Activity Evaluation

Photocatalytic activity of the prepared sample was assessed by monitoring the degradation of methylene blue (MB) in presence of UV-visible light irradiation. In this experiment, 25 mg of photocatalyst was dispersed in 50 mL solution of MB dye (10 mg L⁻¹) using a quartz reactor. Before irradiation, the suspension was stirred in the dark for 30 min to achieve adsorption-desorption equilibrium. Photodegradation was done using a UV-visible lamp (125 W) positioned 15 cm above the solution surface. At regular time intervals (0–200 min), 5 mL aliquots were withdrawn, centrifuged to remove particulates, and analyzed using a UV-Vis spectrophotometer (Sytronics Model 117, India) by monitoring the absorbance at λ_{max} (664 nm). The photocatalytic efficiency was calculated using the relation;

$$\text{Degradation (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

Where C_0 and C_t are the dye concentrations before and after irradiation.

2.4. Electrochemical Testing

Electrochemical workstation Autolab PGSTAT 86472 was used to

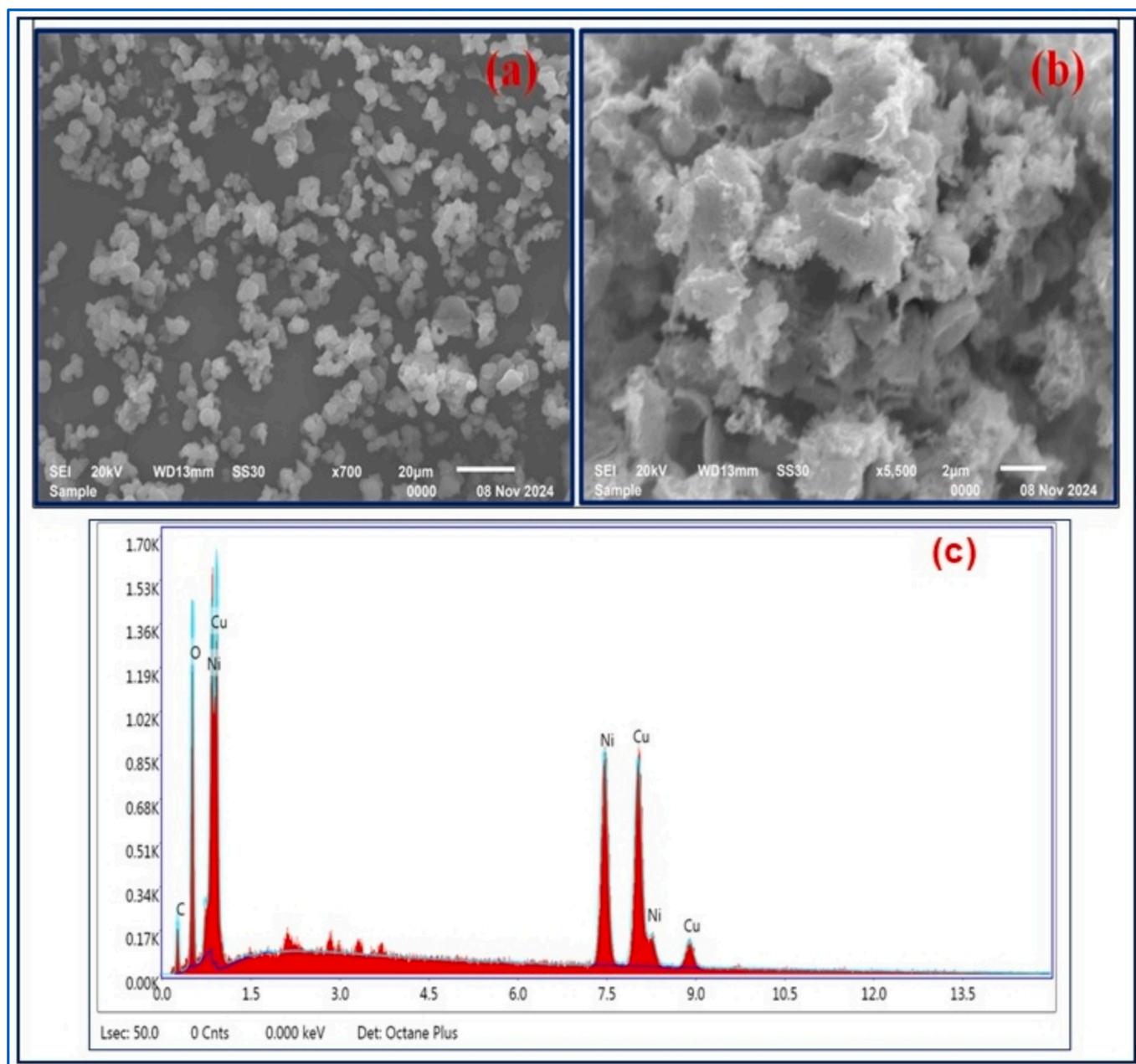


Fig. 2. FE-SEM images of (a) OMC (b) OMC/CuO/NiO nanocomposite, and (c) EDX spectrum confirming the elemental composition and successful impregnation of CuO and NiO nanoparticles in the composite.

evaluate the electrochemical properties of the prepared material in a three-electrode configuration. A platinum wire was served as the counter electrode and an Ag/AgCl (3 M KCl) electrode was served as the reference electrode. In contrast, the active material was coated on a graphite sheet (1.0 cm^2) to prepare the working electrode. The electrolyte used was an aqueous 6.0 M KOH solution [30].

Preparation of Working Electrode.

The fabrication of working electrode was done by mixing 80 wt% active material (OMC/CuO/NiO), 10 wt% acetylene black, and 10 wt% polyvinylidene fluoride (PVDF) in a small amount of *N, N*-dimethylformamide (DMF) to prepare a homogeneous slurry. This slurry was coated onto a graphite sheet (1.0 cm^2) and dried at 65°C for 8 h to ensure complete solvent removal.

Cyclic Voltammetry (CV).

Cyclic Voltammetry (CV) was done in the potential range of -0.2 V to 0.6 V (vs. Ag/AgCl) at different scan rates ($5\text{--}100 \text{ mVs}^{-1}$) to assess the

capacitive behavior.

Galvanostatic Charge–Discharge (GCD).

GCD tests were carried out at the current densities between 0.9 Ag^{-1} to 2.3 Ag^{-1} within the same potential window (-0.2 to 0.6 V).

Energy Density and Power Density.

From GCD data, the energy density ($E, \text{ Wh kg}^{-1}$) and power density ($P, \text{ W kg}^{-1}$) were derived.

3. Results and discussion

3.1. Structural and Morphological Characterization

The wide-angle XRD pattern of synthesized OMC (Fig. 1, curve a) shows a wide diffraction peak around 24.4° attributed to the (002) reflection of graphitic Carbon, suggesting the amorphous nature of the Carbon framework consistent with OMC structures [30,31]. The small-

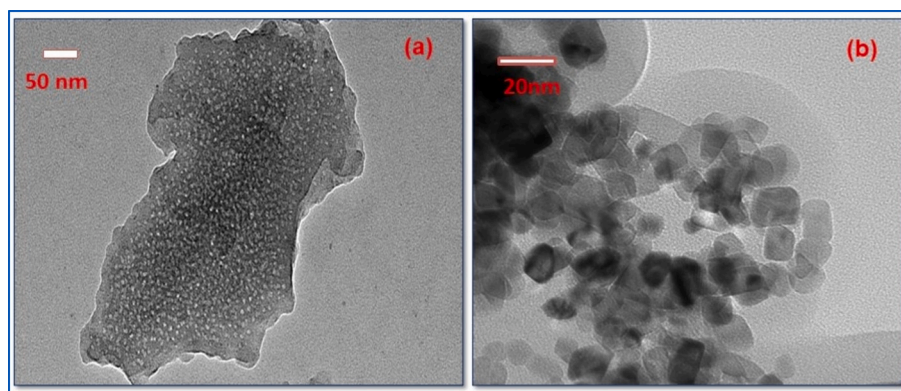


Fig. 3. TEM images of (a) pristine OMC showing a well-ordered mesoporous structure and (b) OMC/CuO/NiO nanocomposite with uniformly distributed Cu and Ni nanoparticles within the Carbon matrix.

angle XRD pattern of synthesized OMC presents a strong diffraction peak nearly at $2\theta = 1.1^\circ$ which can correspond to (100) Bragg reflection of highly ordered mesoporous structure (Fig. 1b) [32]. In contrast, the XRD pattern of the OMC/CuO/NiO nanocomposite (Fig. 1, curve b) displays sharp and well-defined peaks corresponding to crystalline CuO and NiO phases, confirming that incorporation of metal oxides does not disrupt their inherent crystal structure [30,33]. Specifically, diffraction peaks at 32.46° , 35.57° , 38.69° , 49.02° , 53.48° , 58.19° , 61.71° , 66.17° , 68.28° , 72.40° , and 75.21° are indexed to the (110), (-111), (111), (202), (020), (202), (-113), (-311), (220), (311), and (-222) planes of monoclinic CuO, as per JCPDS card no. 80-1916. Additional peaks at 37.15° , 43.15° , and 63.02° correspond to the (111), (200), and (220) planes of cubic NiO, consistent with JCPDS card no. 04-006-6295 [18,24,30]. These results confirm the successful integration of CuO and NiO nanocrystallites into the OMC matrix while retaining their structural integrity and promoting composite crystallinity.

FE-SEM and TEM imaging were employed to determine the microstructure and morphology of the synthesized products [34]. Fig. 2a presents the FE-SEM image of the prepared OMC sample, showing a

panoramic view composed of well-dispersed nanospheres. The presence of Pluronic F127 during synthesis likely acts as a surfactant, reducing the surface tension among aromatic clusters and assisting in even nucleation and growth of spherical nanoparticles. The formation of ordered mesopores within the resol-derived carbon spheres is attributed to the removal of F127 in the process of carbonization [34,35]. The FE-SEM image of the CuO/NiO-doped OMC sample is shown in Fig. 2b, where the particles appear agglomerated, non-uniform, and irregular in shape, reflecting the influence of metal oxide incorporation on surface morphology [36]. EDX analysis of the sample is shown in Fig. 2c, which confirms that CuO and NiO nanoparticles are successfully incorporated into the Carbon framework [37].

Magnified TEM images of OMC are shown in Fig. 3a. The darker area is a carbon matrix, and the lighter areas display the ordered mesopores within the matrix [16]. The well-organized mesoporous architecture improves the effective diffusion of electrolyte ions throughout the matrix which result in enhancing capacitance retention and lowers the impedance associated with electrode kinetics [38]. In the TEM micrograph of the doped OMC/CuO/NiO nanocomposite seen in Fig. 3b, the

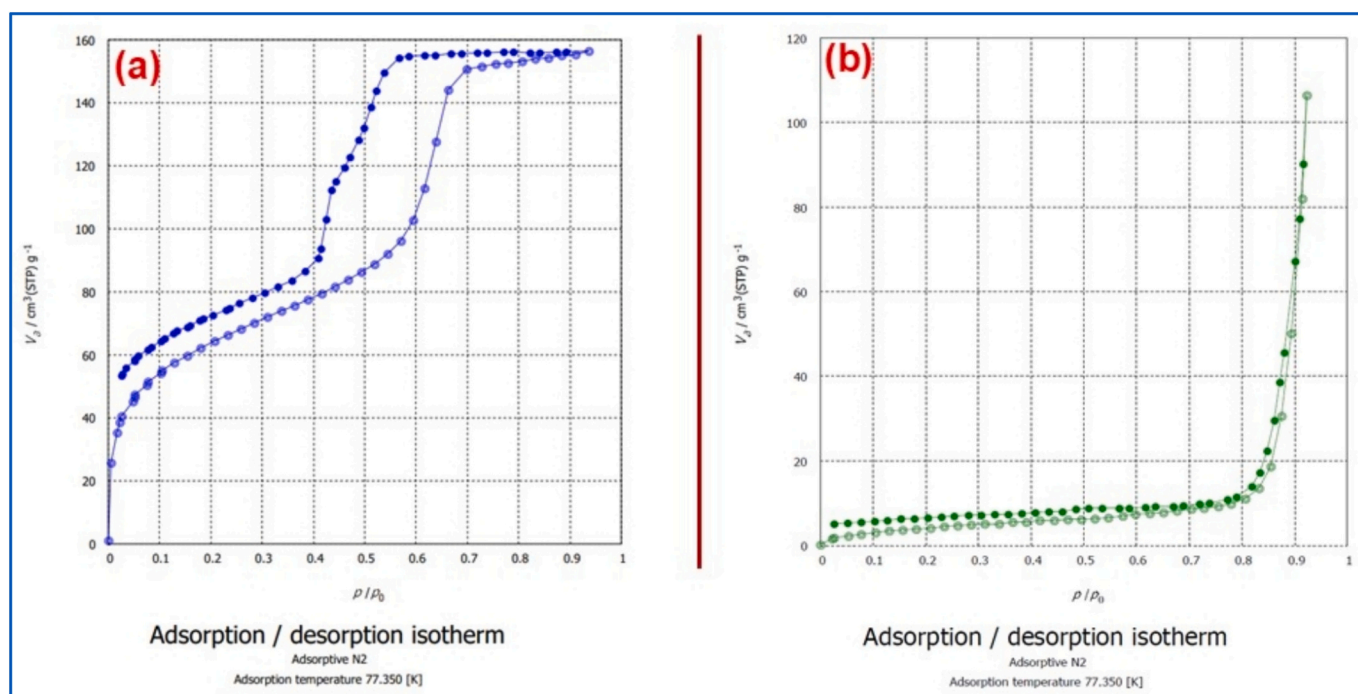


Fig. 4. Nitrogen adsorption-desorption isotherms of (a) OMC and (b) OMC/CuO/NiO nanocomposite measured at 77 K, showing type IV isotherms with H1 hysteresis loops confirming the mesoporous nature of structures.

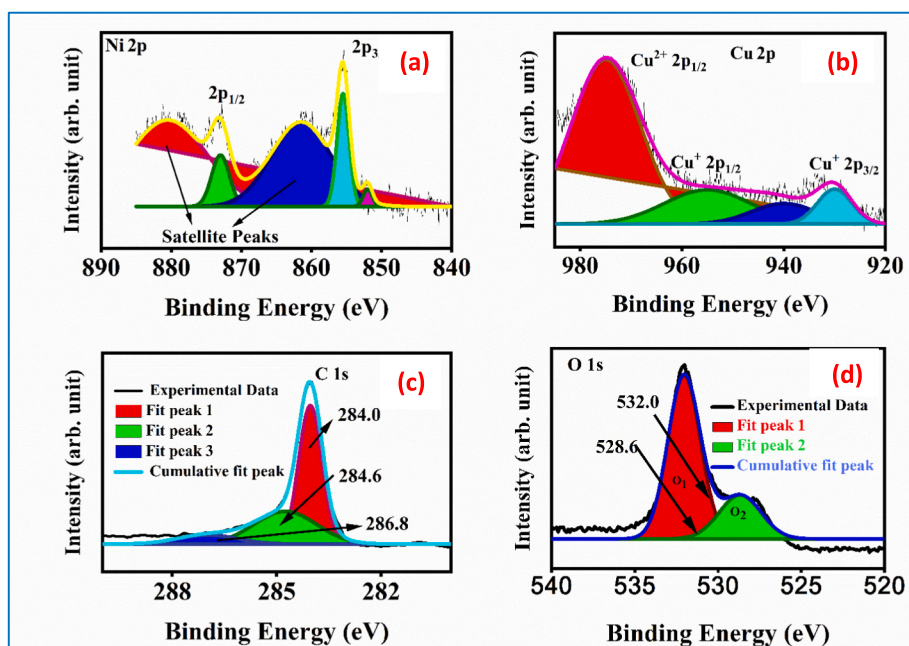


Fig. 5. (a) High-resolution spectrum for Ni 2p (b) Cu 2p spectra (c) C 1 s spectra and (d) O 1 s spectra.

dark areas show the CuO and NiO nanoparticles. These particles are evenly spread throughout the carbon matrix [18]. An ordered mesoporous structure ensures efficient pathways for electrolyte ion diffusion which led to enhanced capacitance retention [38]. The pristine OMC exhibits a highly homogeneous structure, the OMC/CuO/NiO nanocomposite displays distinct encapsulation of copper and nickel oxide nanoparticles (dark regions) within the mesoporous carbon framework (lighter areas), indicating successful integration of metal oxides into the host matrix [39].

The synthesized samples' surface area and pore characteristics were analyzed using nitrogen adsorption-desorption isotherms at 77 K [31]. As shown in Fig. 4a, the isotherm of pure OMC exhibits a type IV curve with a clear H1-type hysteresis loop at relative pressures (P/P_0) > 0.4, characteristic of mesoporous materials [18,40]. The isotherms for both OMC and OMC/CuO/NiO (Fig. 4a and Fig. 4b) display open adsorption-desorption loops, which may be attributed to incomplete degassing during BET measurement, as previously reported by Boutin et al. (2012) and similarly observed in coal-derived samples by Ye et al. (2019) [41].

Upon impregnation of CuO and NiO nanoparticles over the surface of

mesoporous Carbon, the BET surface area significantly decreased from $232.37 \text{ m}^2 \text{ g}^{-1}$ (OMC) to $17.009 \text{ m}^2 \text{ g}^{-1}$ (OMC/CuO/NiO), indicating partial pore blockage. Correspondingly, the total pore volume reduced from $0.2419 \text{ cm}^3 \text{ g}^{-1}$ to $0.1645 \text{ cm}^3 \text{ g}^{-1}$. A slight increase in average pore size was also observed in the doped sample. This increase can be attributed to (i) reduced pore shrinkage due to enhanced rigidity imparted by the embedded metal oxides and (ii) expansion of nanostructures resulting from partial infiltration of Copper and Nickel nitrate precursors into the mesoporous network.

Overall, doping OMC with CuO and NiO results in a notable reduction in both surface area and pore volume. Such changes in textural properties are typically detrimental to electrochemical systems relying solely on physical adsorption; however, in multifunctional systems, where redox activity plays a significant role [42,43].

4. XPS Analysis

X-ray photoelectron spectroscopy (XPS) has been used to thoroughly investigate the surface composition and chemical state of several

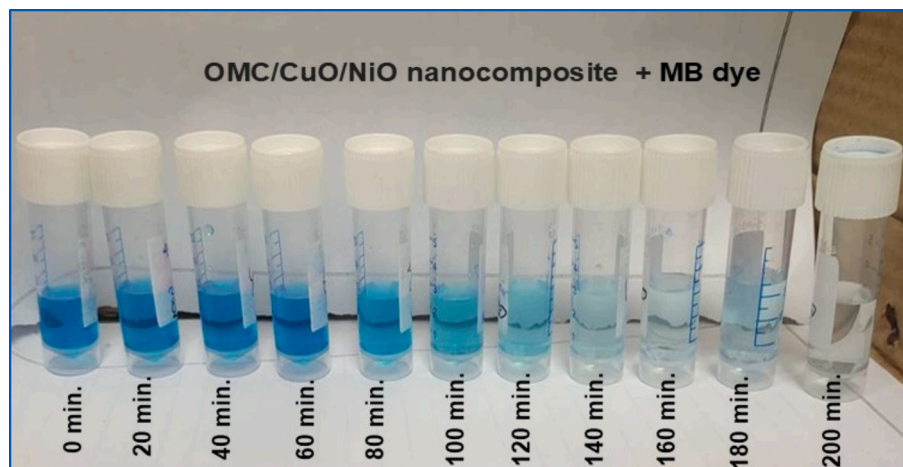


Fig. 6. Visual colour transition of MB dye solution during photocatalytic degradation using OMC/CuO/NiO nanocomposite over time, showing a gradual decrease in colour intensity with increasing irradiation time.

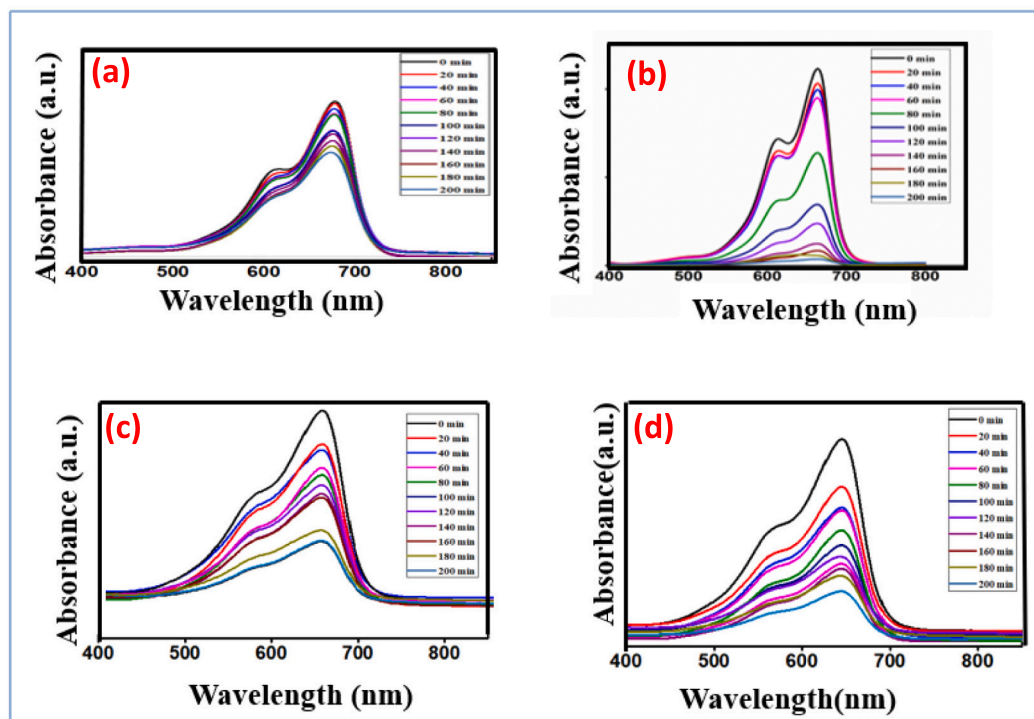


Fig. 7. UV-Vis absorption spectra of MB solution throughout photocatalytic degradation by (a) OMC (b) OMC/CuO/NiO nanocomposite (c) OMC/NiO nanocomposite (d) OMC/CuO nanocomposite over time.

elements present in sample. The X-ray photoelectron spectroscopy (XPS) spectrum of Ni 2p (Fig. 5a) shows characteristic peaks at binding energies of approximately 852 eV, 855 eV, 861 eV, 873 eV, and 880 eV, which correspond to different chemical states of nickel. The peak observed at around 852 eV is attributed to the Ni 2p_{3/2} level of metallic nickel (Ni⁰), indicating the presence of unoxidized nickel species. In contrast, the peak appearing at approximately 855 eV, along with its associated satellite peak at about 861 eV, corresponds to Ni²⁺ species such as nickel oxide (NiO) or nickel hydroxide (Ni(OH)₂). These satellite peaks arise due to shake-up processes, which are characteristic of transition metal oxides and occur because of energy loss related to d-d electronic excitations. The higher binding energy peaks at 873 eV and 880 eV are assigned to the Ni 2p_{1/2} component and its corresponding satellite, respectively. The spin-orbit splitting between the Ni 2p_{3/2} and Ni 2p_{1/2} peaks (approximately 17 eV) is consistent with literature values for nickel compounds. Overall, the presence of both metallic (Ni⁰) and oxidized (Ni²⁺) peaks in the spectrum indicates that the sample surface contains nickel in mixed oxidation states, suggesting partial oxidation of metallic nickel or the coexistence of Ni and NiO phases. (See Fig. 6.)

The XPS spectrum (Fig. 5b) of Cu 2p confirms the coexistence of Cu⁺ and Cu²⁺ oxidation states in the sample. The peaks observed at binding energies of 829 eV and 840 eV correspond to the Cu⁺ 2p_{3/2} and Cu⁺ 2p_{1/2} levels, respectively, indicating the presence of monovalent copper species without any shake-up satellites. The additional peaks at 854 eV and 872 eV are attributed to the Cu²⁺ 2p_{3/2} and Cu²⁺ 2p_{1/2} levels, accompanied by weak shake-up features, confirming the presence of divalent copper. Thus, the XPS analysis concludes that the sample contains both Cu⁺ and Cu²⁺ species, indicating a mixed-valence state of copper resulting from partial oxidation of Cu⁺ to Cu²⁺.

The XPS spectrum of C 1s (Fig. 5c) clearly reveals the presence of carbon atoms in different chemical environments. The main peak observed at a binding energy of 284.0 eV corresponds to sp²-hybridized carbon (C—C or C=C) bonds [44], which represent graphitic or hydrocarbon-type carbon and form the dominant component of the spectrum. The second peak appearing at 284.6 eV is associated with sp³-

hybridized carbon (C—C or C—H), typically arising from amorphous carbon or surface contamination. Another peak located at a higher binding energy of 286.8 eV is attributed to C—O or C—O—C functional groups, indicating the presence of oxygen-bonded carbon species such as alcohols or ethers. Overall, the C 1s spectrum confirms that the surface carbon is mainly composed of graphitic carbon along with a smaller contribution from oxidized carbon species. The presence of these oxygen-containing groups suggests mild surface oxidation or exposure of the sample to air, resulting in the formation of C—O bonds.

The XPS spectrum of O 1s (Fig. 5d) shows two peaks at 528.7 eV and 532.0 eV, corresponding to lattice oxygen (O_l) and surface adsorbed oxygen species (O_s), respectively. The lattice oxygen is associated with metal–oxygen bonds within the crystal structure, while the surface oxygen species (such as –OH or adsorbed O₂) are highly reactive and play a crucial role in photocatalysis. The presence of both types of oxygen enhances charge separation and promotes the generation of reactive oxygen species, thereby improving the overall photocatalytic activity of the material.

The XPS analysis confirms the coexistence of multiple oxidation states of Ni and Cu, along with the presence of lattice and surface oxygen species, which collectively support the enhanced photocatalytic degradation of dyes. The mixed oxidation states of Ni²⁺/Ni⁰ and Cu²⁺/Cu⁺ facilitate efficient charge transfer and redox cycling, reducing electron–hole recombination and promoting the generation of reactive radicals during photocatalysis. The lattice oxygen (O_l) ensures structural stability, while the surface adsorbed oxygen species (O_s) actively participate in forming reactive oxygen species (ROS) such as •OH and O₂^{•−}, which degrade dye molecules. Additionally, the presence of oxygenated carbon species improves surface adsorption of dyes and enhances interfacial charge transfer. Altogether, the XPS results confirm that the synergistic interaction among Ni, Cu, O, and C components contributes to superior photocatalytic dye degradation efficiency.

5. UV – Vis Spectroscopy Analysis

The optical band gap energies of OMC, OMC/CuO, OMC/NiO and OMC/CuO/NiO nanocomposites were systematically investigated using ultraviolet–visible diffuse reflectance spectroscopy (UV–Vis DRS). The corresponding absorption spectra obtained were employed to construct Tauc plots (refer to Fig. S1), which is used for the determination of the materials' optical band gaps. OMC exhibited band gap energy of 1.57 eV, making it active under ultraviolet (UV) irradiation. However, this relatively wide band gap restricts its photocatalytic efficiency within the visible light spectrum. In contrast, OMC/NiO, OMC/CuO and OMC/CuO/NiO nanocomposites demonstrated a significantly band gap of 2.8 eV, 2.1 eV, 1.97 eV which enables enhanced absorption in the visible region and exhibits improved photocatalytic efficiency. A reduced band gap is advantageous as it lowers the threshold energy required for electron excitation, enabling the material to harness lower-energy photons and broaden its absorption range from the UV into the visible spectrum. The incorporation of CuO and NiO into OMC to form nanocomposites resulted in a progressive decrease in band gap energies. The significant reduction in band gap can be attributed to the synergistic electronic interactions and strong interfacial interaction between the two semiconductors. Upon heterojunction formation, the conduction and valence band edges of OMC and CuO and NiO undergo realignment through Fermi level equilibration, which promotes efficient charge separation and accelerated interfacial charge transfer. Among the synthesized composites, the OMC/CuO/NiO nanocomposite exhibited the lowest band gap of 1.97 eV. This narrowed band gap enhances the material's capability to harvest both UV and visible light, thereby facilitating the excitation of electrons from the valence band to the conduction band. As a result, OMC/CuO/NiO nanocomposite generates a higher density of photogenerated electron–hole pairs, which markedly improves its photocatalytic efficiency. Moreover, the intimate interfacial contact between OMC and CuO, NiO promotes rapid interfacial charge transfer which suppresses electron–hole recombination. This efficient charge separation further drives the formation of reactive oxygen species (ROS)—including hydroxyl ($\bullet\text{OH}$) and superoxide ($\bullet\text{O}_2^-$) radicals which play a crucial role in the oxidative degradation of organic dye molecules. So OMC/CuO/NiO nanocomposite is the promising candidate for the photodegradation of organic pollutants in environmental remediation applications.

6. Photocatalytic Performance

The photocatalytic degradation of MB dye was examined under UV–visible light to assess the performance of the synthesized OMC, OMC/CuO/NiO, OMC/NiO and OMC/CuO nanocomposite (Fig. 7). For each evaluation, total of 25 mg of the composite was dispersed in 50 mL of MB solution (10 ppm), and then magnetically stirred in the dark for 30 min to establish adsorption-desorption equilibrium. After this step, the mixture was irradiated using a UV lamp 15 cm above the reaction cell. Aliquots were withdrawn at regular intervals (0 to 200 min) to track the degradation process. As illustrated in Fig. 7, the UV–Vis absorption spectra showed a steady decrease in the characteristic absorption peak of MB at 664 nm, confirming effective photodegradation over time. The corresponding degradation profile (Fig. S2a) demonstrates a significant decline in the C/C_0 ratio, confirming the continuous reduction in MB concentration with increasing irradiation time.

After 200 min of irradiation, the degradation efficiencies of the OMC/CuO, OMC/NiO, and OMC/CuO/NiO catalysts were 78.2 %, 76.2 %, and 96 %, respectively, whereas the undoped OMC achieved only 55.07 %. These results clearly demonstrate that the incorporation of metal oxides enhances the photocatalytic performance, with the OMC/CuO/NiO composite exhibiting the highest activity among all the tested materials. This improvement is attributed to presence of CuO and NiO, which contribute to more effective charge carrier separation, a reduction in bandgap energy, and enhanced light harvesting. In addition, the

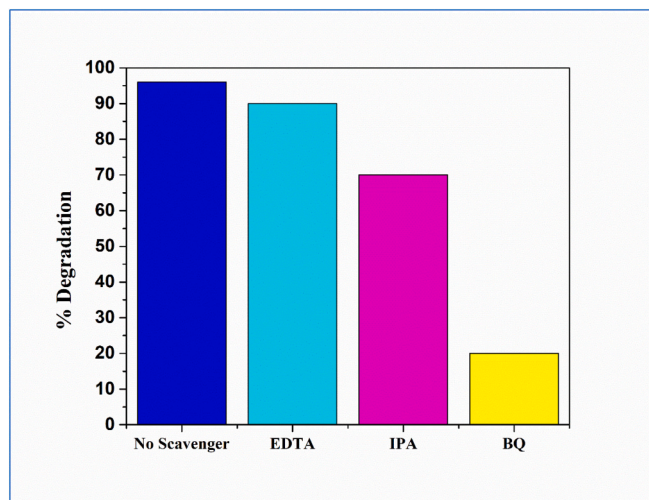


Fig. 8. Percentage degradation of MB dye with several scavengers.

introduction of oxygen vacancies and subtle structural modifications further strengthens the photocatalytic performance by enabling faster electron–hole migration and promoting active surface interaction. The study looked at the degradation rates using a pseudo-first-order model. The plot of $\ln(C/C_0)$ versus irradiation time (Fig. S2b) depicted excellent linearity, confirming that the reaction adheres to first-order kinetics. The calculated R^2 values for OMC/CuO, OMC/NiO and OMC/CuO/NiO nanocomposite are 0.99, 0.98 and 0.96 respectively, further validating the applicability of this model. These results show that the OMC/CuO/NiO nanocomposite has excellent photocatalytic efficiency. This highlights its potential as a strong option for treating dye-contaminated wastewater using light.

6.1. Scavengers test

During the photocatalytic degradation of MB dye, different reactive radical species are generated, which play an essential role in facilitating the degradation process (Fig. 8). A scavenger study was performed with the OMC/CuO/NiO catalyst to elucidate the primary reactive species responsible for this activity. In this investigation, selective scavengers were employed to capture specific reactive species: benzoquinone (BQ)

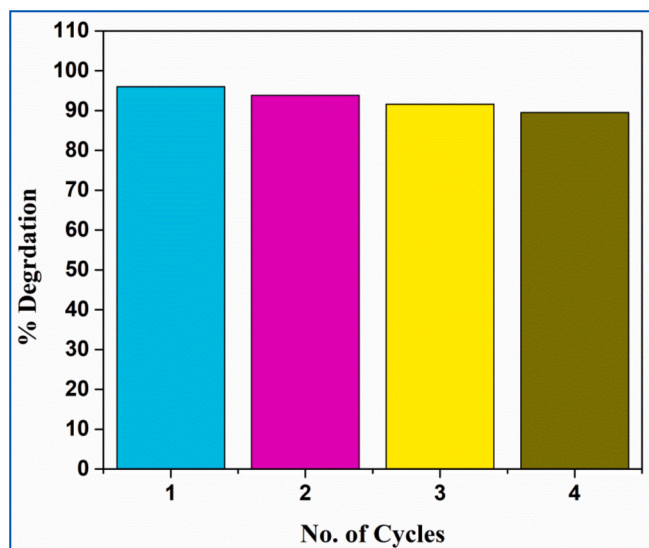


Fig. 9. Study of the reusability of OMC/CuO/NiO nanocomposites for MB dye degradation.

Table 1
Photocatalytic degradation efficiency, synthesis method, and potential applications.

S. No.	Material	Dye Degradation (%)	Degradation Time (min)	Catalytic dosage	Source	Method used	Dye used	Analyte	Ref.
1.	NiO	~88	~0.1 mg	Halogen lamp		Hydrothermal	Methylene Blue	Cationic	[21]
2.	ZnO/NiO	~85	180-	–		One-pot solvothermal	Methylene Blue	Cationic	[36]
3.	NiO nanostructures	80	120 2 g L ⁻¹	UV tube (400 W)		Thermal decomposition	Rhodamine B	Cationic	[45]
4.	NiO nanoparticles	79.5	60 50 mg	Xe arc (300 W)		Green soft chemical	Methylene Blue	Cationic	[46]
5.	OMC/TiO ₂ composite	~77	18,050 mg	Sunlight		Soft template method	Rhodamine B	Cationic	[27]
6.	CuO nanoparticles	80	120-	–		Biogenic synthesis	RY160	Anionic	[37]
7.	OMC/CuO/NiO nanocomposite	96	120 -			Soft template method	Methylene Blue	Cationic	Present work

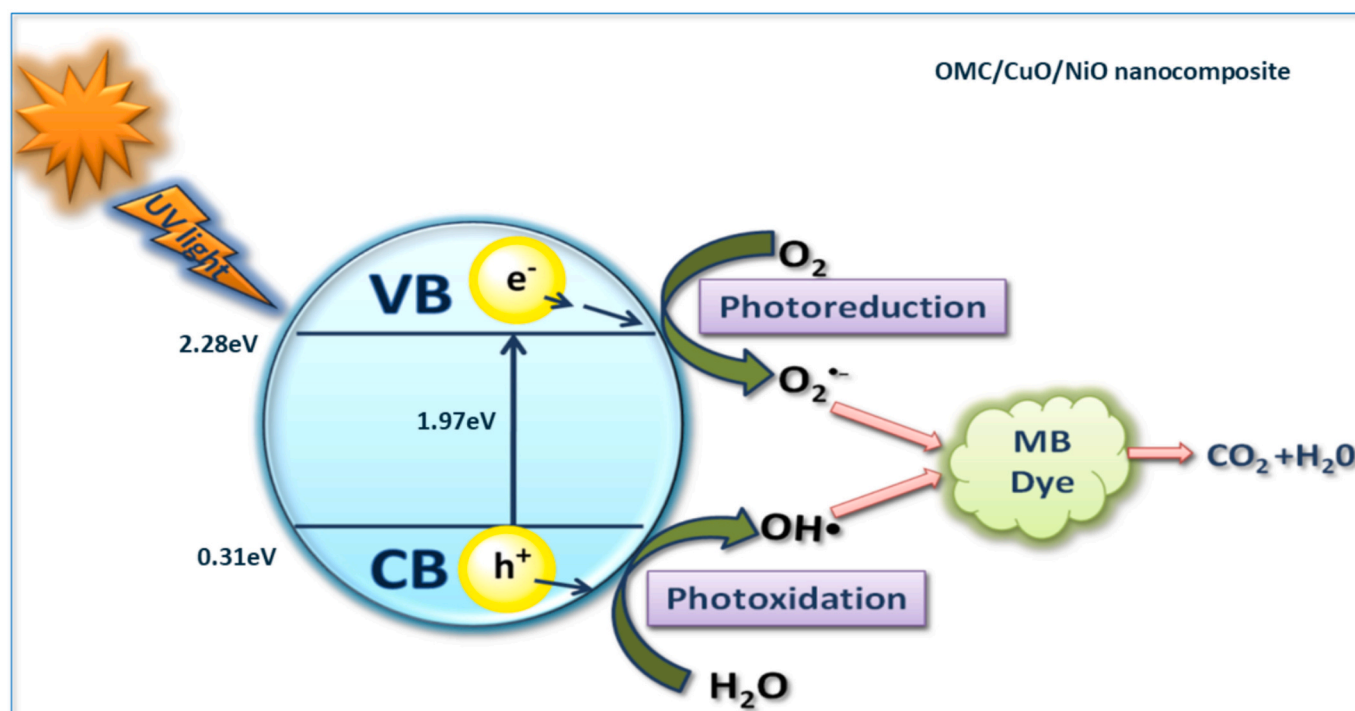


Fig. 10. Suggested mechanism for the photocatalytic degradation of MB dye by OMC/CuO/NiO nanocomposite under UV–visible light irradiation.

was used to capture superoxide radicals ($\bullet\text{O}_2^-$), ethylenediaminetetraacetic acid (EDTA) for photogenerated holes (h^+), and isopropanol (IPA) for hydroxyl radicals ($\bullet\text{OH}$). This approach facilitated a systematic explanation of the specific contributions of individual reactive species to the overall photocatalytic activity. The lowest degradation efficiency was attained in the presence of BQ, suggesting that $\bullet\text{O}_2^-$ plays a major role in dye degradation process. On the other hand, medium decline in degradation efficiency was attained with the use of IPA which highlights that $\bullet\text{OH}$ radicals in this process are not the main active but are reactive species but EDTA shows the highest degradation efficiency, which indicates that h^+ , plays a minor role in the photocatalytic degradation.

6.2. Reusability test

The recyclability and long-term stability of a catalyst are key factors that affect its practical use and cost-effectiveness. In this study, we assessed the stability of the OMC/CuO/NiO nanocomposite by testing its photocatalytic efficiency in degrading aqueous MB solutions. The experiment involved multiple cycles of photocatalytic reactions under UV light. We measured the catalyst's efficiency at various intervals.

As shown in Fig. 9, the nanocomposite proved to be very stable during all the experimental cycles. The OMC/CuO/NiO catalyst underwent four consecutive photocatalytic cycles to test its reusability. After each cycle, we easily recovered the sample. We thoroughly washed the catalyst with ethanol after each run and reused it for the next cycles. The results indicated a small decrease in photocatalytic activity, about 6.5 % after four cycles. This decline mainly resulted from typical material loss during recovery. We compared these results with existing literature in Table 1.

This slight decrease highlights the nanocomposite's strong structural stability and consistent photocatalytic performance under UV light. These findings suggest that the OMC/CuO/NiO nanocomposite has great potential for long-term use in wastewater treatment. Consistent performance over repeated cycles is essential for both environmental sustainability and operational efficiency.

of the developed OMC/CuO/NiO nanocomposite are provided to provide a comprehensive overview of the material's performance.

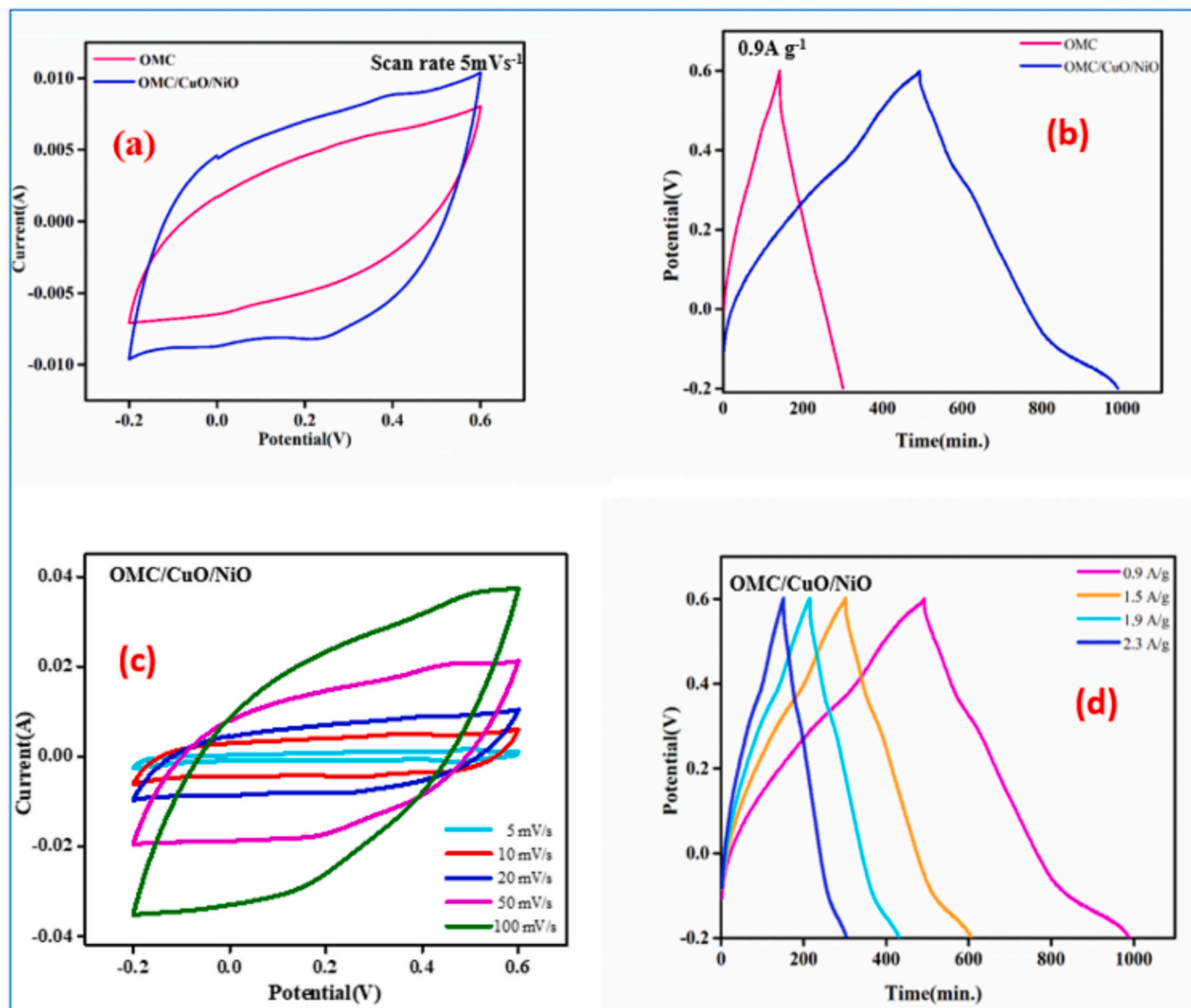


Fig. 11. (a) The CV curves of OMC/CuO/NiO nanocomposite at scan rate 5 mV s⁻¹ (b) Comparison of charge discharge cycle OMC and OMC/CuO/NiO nanocomposites at 0.9 A g⁻¹ (c) CV curves of OMC/CuO/NiO nanocomposites (d) GCD curves of OMC/CuO/NiO nanocomposites.

6.3. Suggested mechanism for photocatalytic degradation of methylene blue dye

Based on the results of this study, the proposed band alignment and charge transfer mechanism for the photocatalytic degradation of methylene blue (MB) dye by OMC/CuO/NiO nanocomposite is illustrated in Fig. 10. When exposed to light, CuO and NiO semiconductors absorb photons with energy equal to or greater than the bandgap. This process excites electrons from the valence band (VB) to the conduction band (CB), generating electron-hole pairs (e^-/h^+), as shown in Eqs. (2) and (3). The OMC framework improves charge separation by enabling fast electron transport and reducing recombination. The photocatalytic degradation of MB dye with the OMC/CuO/NiO nanocomposite occurs through a series of photophysical and redox processes stimulated by UV-visible light. When exposed to ultraviolet light, electrons in CuO and NiO are photoexcited from the valence band (VB) to the conduction band (CB), leaving holes behind in the VB. This step is essential for photocatalytic activity. The band gap energy of pure OMC is 1.57 eV, while OMC/CuO and OMC/NiO show band gaps of 2.1 eV and 2.8 eV, respectively. For the OMC/CuO/NiO nanocomposite, the band gap

decreases to 1.97 eV. This reduction in band gap is due to the electronic band structure alignment and the interactions between OMC and CuO, NiO. When a heterojunction forms, the conduction and valence band edges of the two semiconductors reorganize to balance their Fermi levels. This shift causes bending of the bands at the interface, creating an internal electric field. This field helps separate photogenerated charge carriers effectively and changes the band edge positions, narrowing the band gap. These structural and electronic changes improve the light-harvesting ability and charge transport properties of the nanocomposite, enhancing its overall photocatalytic performance. The edge positions of the conduction band (CB) and valence band (VB) were estimated using the following empirical relations:

$$E_{CB} = \chi - E_e - 0.5E_g$$

$$E_{VB} = E_{CB} + E_g$$

Where E_{CB} = Conduction band potential, E_{VB} = Valence band potential, χ = electronegativity value of photocatalyst, E_e = Energy of free electrons, E_g = Band gap energy of photocatalyst. The CB and VB potentials of OMC were calculated as 0.51 eV and 1.57 eV, while for OMC/

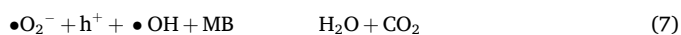
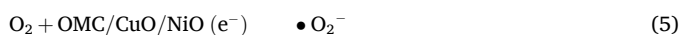
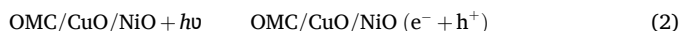
Table 2

Electrochemical parameters, synthesis method, and potential applications of the developed OMC/CuO/NiO nanocomposite to provide a comprehensive overview of the material's performance.

S. No.	Material	Doping	Specific Capacitance (F g ⁻¹)	Synthesis method	Application	Ref.
1.	AC	Mn, Cu	AC – 173 AC@ Mn–Cu– 418	Green synthesis	Supercapacitors	[52]
2.	Cu	Ni, O	Cu/ Ni/O -507	Co-Precipitation	Supercapacitors	[53]
3.	OMC	N/B	OMC- 143	Ionothermal strategy	Supercapacitors	[54]
4.	OMC	NOMC, POMC, NPOMC	N/B OMC- 272 NOMC- 176 POMC- 184 NPOMC- 220	Nanocasting	Supercapacitors	[50]
5.	Mesoporous carbon	N	N-MC- 299	In situ doping	Supercapacitors	[55]
6.	OMC/Graphene composite	N	NOMC-67 GO-N-OMC-252	Evaporation-Induced Self-assembly	Supercapacitors	[56]
7.	OMC	N/O	N/O/OMC- 229	Solid-Solid grinding method	Supercapacitors	[57]
8.	OMC	B	B-OMC-246	Nanocasting	Supercapacitors	[28]
9.	OMC	N/P	NPOMC-140	Chemical Vapour deposition	Supercapacitors	[58]
10.	Mesoporous carbon	N	N-MC - 191.8	Hydrothermal	Supercapacitors	[59]
11.	OMC	CuO/NiO	OMC-175 OMC/CuO/NiO-572	Two-phase method	Supercapacitors	This work

CuO/NiO were found to be +0.31 eV and + 2.285 eV respectively. Upon exposure to light, both CuO and NiO become photoactivated, leading to the creation of electron-hole pairs. The electrons generated in the conduction band (CB) of the CuO/NiO within the OMC/CuO/NiO composite then move toward the CB of the ordered mesoporous carbon (OMC), as shown in Eq. (9). This electron transfer happens due to the favorable band alignment between the materials, which promotes efficient charge separation and improves the overall photocatalytic performance of the composite system. The photogenerated electrons reduce dissolved oxygen molecules to create superoxide radicals ($\bullet\text{O}_2^-$), while the holes oxidize water molecules or surface hydroxyl groups to produce hydroxyl radicals ($\bullet\text{OH}$).

These reactive oxygen species (ROS) are very oxidative and can break down MB dye molecules into smaller, less harmful parts. Eventually, this process turns them into CO_2 and H_2O (Fig. 10). Also, oxygen vacancies in the CuO and NiO lattices help trap charges and boost the generation of more ROS. The combined effects of the metal oxides and the conductive OMC matrix greatly improve photocatalytic performance. This is shown by the higher degradation efficiency and the shorter half-life of the dye. This mechanism explains why the OMC/CuO/NiO composite has better photocatalytic activity than pure OMC. [47].



7. Electrochemical performance

The electrochemical properties of the synthesized samples were analyzed through cyclic Voltammetry (CV) [48,49]. CV curves were recorded in a three-electrode configuration at various scan rates (5–100 mVs^{-1}) with 6.0 M KOH serving as the electrolyte. The specific capacitance (C_v) from CV was calculated using Eq. (8):

$$C_v = \frac{\int IdV}{Sm\Delta V} \quad (8)$$

Here in above equation C_v = specific capacitance measured from CV

curves in F g^{-1} , I = current in A; ΔV = applied potential window in V; dV = represents the differential change in voltage, m = mass of the sample loaded on the surface of the electrode in mg; and S = scan rate in mVs^{-1} .

As illustrated in Fig. 11a and Fig. 11c, the CV curves of OMC and OMC/CuO/NiO nanocomposite exhibit nearly rectangular shapes with redox peaks, signifying the coexistence of electric double-layer capacitance and pseudocapacitance arising from Faradaic reactions of Cu and Ni species. However, pure OMC is more rectangular with a lower current response, confirming its predominantly double-layer capacitance behavior. Among the studied materials, the OMC/CuO/NiO nanocomposite exhibited the maximum specific capacitance of 564.2 F g^{-1} at a scan rate of 5 mVs^{-1} , surpassing that of pristine OMC, which showed a specific capacitance of 179.7 F g^{-1} at the same scan rate. Similarly, the specific capacitance C_s from GCD curves was determined in F g^{-1} using Eq. (12): [50].

$$C_s = \frac{I \times \Delta t}{m \times \Delta V} \quad (9)$$

I = current applied in A; m = mass deposited on electrode in mg; ΔV = applied potential window in V; Δt = discharge time in sec. [51]

$$E = \frac{1}{2 \times 3.6} \times C \Delta V^2 \quad (10)$$

$$P = 3600 \times \frac{E}{t} \quad (11)$$

The specific capacitance (C , F g^{-1}) was derived based on the analysis of the discharge curves using Eq. (9) at a current density of 0.9 A g^{-1} as shown in Fig. 11b. The OMC/CuO/NiO electrode delivers a specific capacitance of 559.1 F g^{-1} , which is $3.1 \times$ greater than pristine OMC (178.8 F g^{-1}). Furthermore, the OMC/CuO/NiO electrode achieves an energy density (E) of 49.69 Wh kg^{-1} at a power density (P) of 359.9 W kg^{-1} , as calculated using Eq. (10) and Eq. (11) confirming its potential for high-performance energy storage applications and has been compared with existing literature in Table 2. Fig. 11d shows the GCD curves OMC/CuO/NiO electrodes recorded at various current densities between 0.9 A g^{-1} to 2.3 A g^{-1} within the potential window ranging from -0.2 V to 0.6 V . The OMC/CuO/NiO electrode exhibits nearly triangular, symmetric charge-discharge profiles, indicating excellent electrochemical reversibility and stable capacitive behavior.

In a two-electrode setup, galvanostatic charge-discharge (GCD) is utilized to evaluate the electrochemical performance of the samples. GCD serves as a method to ascertain the capacitance performance as per Eq. (12)

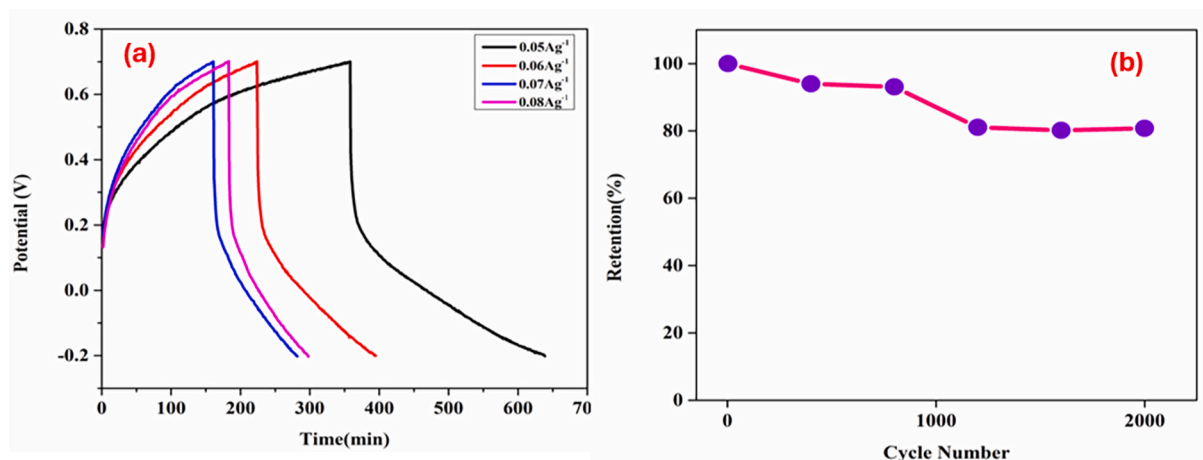


Fig. 12. (a) GCD curve of OMC/CuO/NiO nanocomposite in two electrode system at the various current density (b) Cyclic stability of OMC/CuO/NiO nanocomposite from 2 to 2000 cycles.

$$C_s = 4 \frac{I \times \Delta t}{m \times \Delta V} \quad (12)$$

Here, I denotes “current” measured in amperes, t represents “discharge time” in seconds, m indicates the “active mass” applied to the electrode, and V refers to the “working potential window.” The capacitance performance is derived from the GCD curves and is expressed in $F\ g^{-1}$. Fig. 12a illustrates the GCD curves for OMC/CuO/NiO composite within the potential range of $-0.2\ V$ to $+0.8\ V$ at a current density of $0.05\ A\ g^{-1}$, $0.06\ A\ g^{-1}$, $0.07\ A\ g^{-1}$, $0.08\ A\ g^{-1}$. The specific capacitance values for OMC/CuO/NiO composite are $83.8\ F\ g^{-1}$ at $0.05\ F\ g^{-1}$. The energy density and power density for OMC/CuO/NiO composite is $6.2\ Wh\ kg^{-1}$ and $79.3\ W\ Kg^{-1}$.

The cyclic stability of the OMC/CuO/NiO composite electrode, illustrated in Fig. 12b, was assessed by continuously monitoring cyclic voltammetry (CV) curves within a potential range of $-0.2\ V$ to $0.8\ V$ in a $6.0\ M\ KOH$ electrolyte. The electrode demonstrated remarkable electrochemical stability, preserving 92.0 % of its initial capacitance after 400 cycles, 90.1 % after 800 cycles, 88.2 % after 1200 cycles, 85.4 % after 1600 cycles, and 80.1 % even after 2000 continuous cycles. This gradual reduction in capacitance with cycling suggests minimal structural degradation or loss of active material; however, the electrode continues to exhibit good long-term durability, thereby confirming its suitability for stable supercapacitor application.

8. Conclusions

In this study, CuO/NiO-doped ordered mesoporous carbon (OMC/CuO/NiO) was effectively synthesized through the soft-template method and its dual applicability in photocatalysis and energy storage was demonstrated. Structural, morphological, and optical analyses confirmed that Cu and Ni nanoparticles were uniformly incorporated into the OMC matrix without disrupting its ordered mesoporous architecture. The OMC/CuO/NiO nanocomposite achieving a 96 % degradation efficiency of the MB dye, exhibited remarkable photocatalytic activity, which is attributed to its enhanced light harvesting, optimized band gap, and efficient charge separation. In addition to its photocatalytic capability, the material showed impressive electrochemical performance, delivering a specific capacitance of $564.2\ F\ g^{-1}$ (CV), $559.1\ F\ g^{-1}$ (GCD), and an energy density of $49.6\ Wh\ kg^{-1}$ at a power density of $359.9\ W\ kg^{-1}$. The utilization of inexpensive carbon precursors and earth-abundant transition metals (Cu and Ni) renders the OMC/CuO/NiO nanocomposite economically advantageous alternative to conventional photocatalysts such as TiO_2 , ZnO , and noble-metal-based systems, as well as electrode materials derived from high-cost compounds like RuO_2 , Co_3O_4 , or graphene. Moreover, carbon-based

frameworks are generally non-toxic, further enhancing the practical appeal of the OMC/CuO/NiO composite.

Future work: Subsequent studies will focus on optimizing the Cu/Ni doping ratio to further enhance the photocatalytic and electrochemical performance of the material. Additional research will focus on assessing the long-term stability of the material.

Overall, these findings underscore the dual functionality of CuO/NiO-doped OMC, positioning it as a highly promising candidate with strong potential for sustainable wastewater remediation and advanced energy storage devices.

CRediT authorship contribution statement

Anjali Bhardwaj: Writing – original draft, Methodology, Investigation, Funding acquisition, Conceptualization. **Harish Kumar:** Writing – review & editing, Resources, Methodology, Formal analysis, Conceptualization. **Ashok Kumar Sharma:** Writing – review & editing, Supervision, Resources, Methodology, Investigation, Formal analysis, Conceptualization.

Ethical approval

No such data applies to human and animal studies. This work has not been submitted to nor appeared in a journal or as part of thesis work.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.inoche.2025.115997>.

Data availability

Data will be made available on request.

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