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# Empowering solar-driven degradation: advancements in lanthanum-doped Bi<sub>2</sub>O<sub>3</sub>/g-C<sub>3</sub>N<sub>4</sub> heterojunctions for high-performance photocatalysis

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The lanthanum-doped bismuth oxide (La-Bi<sub>2</sub>O<sub>3</sub>) anchored g-C<sub>3</sub>N<sub>4</sub> (g-CN) heterojunction-based photocatalyst (La-Bi<sub>2</sub>O<sub>3</sub>/g-CN) has been designed with visible-light activation and prompt interfacial charge transfer. Pure and La-doped bismuth oxide nanomaterials were prepared *via* a hydrothermal method, while g-CN was obtained by thermal polymerization of melamine. The doped material was anchored over the 2D g-CN to make the heterojunction (Type-II) by prolonged ultrasonication. As-synthesized samples were characterized *via* XRD, FT-IR, TGA, SEM, TEM, UV-vis, PL spectroscopy, Mott-Schottky plots, EIS, and photocurrent response to investigate the structural features, thermal endurance, topography, optical response, and interfacial charge mobility. The photocatalyst's efficiency was determined by degrading methylene blue (MB) and ciprofloxacin (CPF) as representative pollutants under visible-light irradiation. The heterojunction material displayed promising photocatalytic activity compared to its pure counterparts and photodegraded 97% of MB and 84% of CPF, following 1st-order kinetics. This remarkable efficiency was due to the band structure tuning of bismuth oxide by La-doping and architecting heterojunction La-Bi<sub>2</sub>O<sub>3</sub>/g-CN with g-CN, which promotes the interfacial charge transfer, delaying recombination of the separated charges. The scavenging results revealed that the hydroxide radicals (HO<sup>•</sup>) and holes (h<sup>+</sup>) correspondingly facilitated the MB and CPF degradation.

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## 1. Introduction

The demand for the development of sustainable technologies for clean energy production and environmental pollution control has sparked substantial interest in visible-light responsive photocatalysis, a process involving a semiconductor material to drive the chemical reaction, presenting a promising approach to these overwhelming challenges.<sup>1</sup> The

efficiency of a photocatalyst significantly hinges on several factors, including light absorption, electron-hole pair generation, and interfacial charge transfer (delaying charge carriers' recombination) to generate the reactive oxygen species.<sup>2,3</sup> The materials, including oxides (ZnO,<sup>4</sup> TiO<sub>2</sub>,<sup>5</sup> Fe<sub>2</sub>O<sub>3</sub>,<sup>6</sup> WO<sub>3</sub>,<sup>7</sup> Bi<sub>2</sub>O<sub>3</sub>,<sup>8</sup> MoO<sub>3</sub>,<sup>9</sup> SnO<sub>2</sub>,<sup>10</sup> and V<sub>2</sub>O<sub>5</sub> (ref. 11)), sulfides (CdS,<sup>12</sup> MoS<sub>2</sub>,<sup>13</sup> ZnS,<sup>14</sup> Bi<sub>2</sub>S<sub>3</sub>,<sup>15</sup> and In<sub>2</sub>S<sub>3</sub> (ref. 16)), selenides (CdSe<sup>17</sup> and ZnSe<sup>18</sup>), and carbonaceous materials,<sup>19</sup> have been explored as photocatalysts. The wide energy bandgaps of the semiconductors limit their visible light absorption, rendering them ineffective and restricting solar light harvesting.<sup>20,21</sup> Another key limitation is the rapid recombination of photogenerated e<sup>-</sup>-h<sup>+</sup> pairs, which hampers the redox reaction to generate the reactive oxygen species.<sup>22-24</sup> Therefore, designing photocatalysts that are visible-light-responsive with enhanced charge separation is crucial to improve their efficiency.

Bismuth oxide Bi<sub>2</sub>O<sub>3</sub>, a semiconductor with thermal and chemical stability, is a promising material for visible-light-responsive photocatalysis as it features resistance to photo-corrosion.<sup>25,26</sup> The band structure of Bi<sub>2</sub>O<sub>3</sub> includes Bi 6s and O 2p hybrid orbitals in the valence-band maxima and Bi 6p orbitals in the conduction-band minima, which support the

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visible light absorption resulting in photoexcitation.<sup>27–29</sup> Nevertheless, the rapid recombination of these charge carriers severely compromises their photocatalytic efficiency. Therefore, the band structure modification to reduce the fast recombination is crucial to designing bismuth oxide-based photocatalysts.<sup>30</sup> Graphitic carbon nitride, a metal-free semiconductor, is a sustainable option for photocatalytic applications. Still, its practical applicability needs further optimization, as its photocatalytic activity is hindered by limited light response and rapid charge-carrier coupling.<sup>31,32</sup> Various investigations have revealed that using g-CN for designing composite-based photocatalysts is a practical approach that tunes the overall catalytic activity of the g-CN-derived material under solar light exposure.<sup>33–35</sup> Composite materials with an active heterojunction architecture exhibit properties tuned for photocatalytic applications, including increased light absorption and efficient charge transfer.<sup>36,37</sup>

In light of the facts mentioned above, we have designed a heterojunction (Type-II) material by anchoring lanthanum-doped bismuth oxide on the 2D graphitic carbon nitride (g-CN) with visible-light activation and prompt interfacial charge transfer for solar light harvesting to derive potential photocatalytic applications. Initially, pure bismuth oxide (abbreviated as BO) and its La-doped counterpart (abbreviated as LBO) were hydrothermally fabricated, while the calcination of melamine synthesized g-CN *via* thermal polymerization. Afterward, using the ultrasonication approach, the doped LBO material was loaded on the g-CN to architect the LBO/g-CN heterojunction.

Physicochemical and opto-electro investigations were performed to prove the nature of the material and validate its suitability for efficient photocatalysis. MB and CPF were degraded as representative organic pollutants over the synthesized photocatalysts to evaluate their catalytic efficiency. The degradation reaction kinetics, reactive oxygen species tracking, and reusability of the heterojunction LBO/g-CN photocatalyst were explored to gain insights into the potential applicability of the designed photocatalyst.

## 2. Experimental

### 2.1 Chemicals

The analytical grade precursors were used for the preparation of pure and lanthanum-doped bismuth oxide nanomaterials, which include lanthanum(III) nitrate hexahydrate ( $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ , Sigma-Aldrich,  $\geq 99.99\%$ ) and bismuth(III) nitrate pentahydrate ( $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ , Sigma-Aldrich,  $\geq 99.99\%$ ). Melamine ( $\text{C}_3\text{H}_6\text{N}_6$ , 99%, Sigma-Aldrich) was used to prepare g-CN. In contrast, MB ( $\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}$ ,  $\geq 82\%$ , Sigma-Aldrich) and CPF ( $\text{C}_{17}\text{H}_{18}\text{FN}_3\text{O}_3$ , Sigma-Aldrich,  $\geq 98\%$ ) were used as representative organic pollutants for investigating the photocatalytic activity of the designed photocatalysts. For the scavenging experiment, isopropyl alcohol (IPA, 99.9%), benzoquinone (BQ, 99%), and ethylenediaminetetraacetic disodium salt (EDTA-2Na, 99%) were used to quench the reactive oxygen species (ROS) generated during MB and CPF degradation.



Fig. 1 Schematic presentation of BO, LBO, g-CN, and LBO/g-CN synthesis.



## 2.2 BO and LBO nanomaterials synthesis

The pure and La-doped nanomaterials, BO and LBO, were synthesized *via* the hydrothermal approach. The bismuth precursor ( $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ ) was dissolved in DI water by magnetic stirring, followed by the addition of a lanthanum precursor ( $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ) for doped counterpart preparation. The solution was stirred (300 rpm) until complete dissolution of precursor salts, and then the  $\text{NH}_4\text{OH}$  was gradually added to raise the pH to  $\sim 9$ . Afterward, the final remedy was charged into a stainless-steel autoclave (Internally Teflon-lined) and heated for 12 hours at  $180^\circ\text{C}$ . The synthesized nanomaterial was thoroughly rinsed with water to wash off the residuals and dried at  $60^\circ\text{C}$ .

## 2.3 Powder g-CN synthesis

The g-CN was fabricated through the thermal condensation of melamine.<sup>38</sup> Experimentally, 5 grams of the melamine powder was transferred into the lidded crucible and introduced into a furnace, followed by calcination at  $550^\circ\text{C}$  with a heating rate of  $10^\circ\text{C min}^{-1}$  for 6 hours to polymerize melamine, forming 2D hexagonal rings of carbon and nitrogen network. The canary-yellowish-coloured g-CN formed was stored in a moisture-free environment.

## 2.4 Fabrication of LBO/g-CN heterojunction

The La-doped nanomaterial LBO was loaded onto the synthesized g-CN sheets to architect the LBO/g-CN heterojunction by ultrasonication. Initially, 460 mg of the LBO and 40 mg of the g-CN were dispersed in DI water in separate beakers by magnetic stirring (500 rpm), followed by sonication in the ultrasonic bath (40 kHz) for  $\frac{1}{2}$  hour at ambient temperature. The suspensions mentioned above were poured into a beaker, and the resultant blend was ultrasonicated for 60 minutes. The composite formed was separated by centrifugation (300 rpm) and dried in the oven (vacuum,  $60^\circ\text{C}$ , 6 hours) to get the designed heterojunction in a ratio of 9 : 1 of LBO to g-CN. The preparation scheme of BO, LBO, g-CN, and LBO/g-CN materials is shown Fig. 1.

## 2.5 Photocatalytic activity (PCA)

The PCA of the designed BO, LBO, and LBO/g-CN was determined by photocatalytic MB and CPF degradation as model dyestuff and antibiotic organic pollutants. Initially, 0.05 g of the catalysts (BO, LBO, and LBO/g-CN) were separately dispersed in 100 mL ( $20\text{ mg L}^{-1}$ ) of the contaminants (MB and CPF) solutions. These suspensions containing the catalysts were kept in

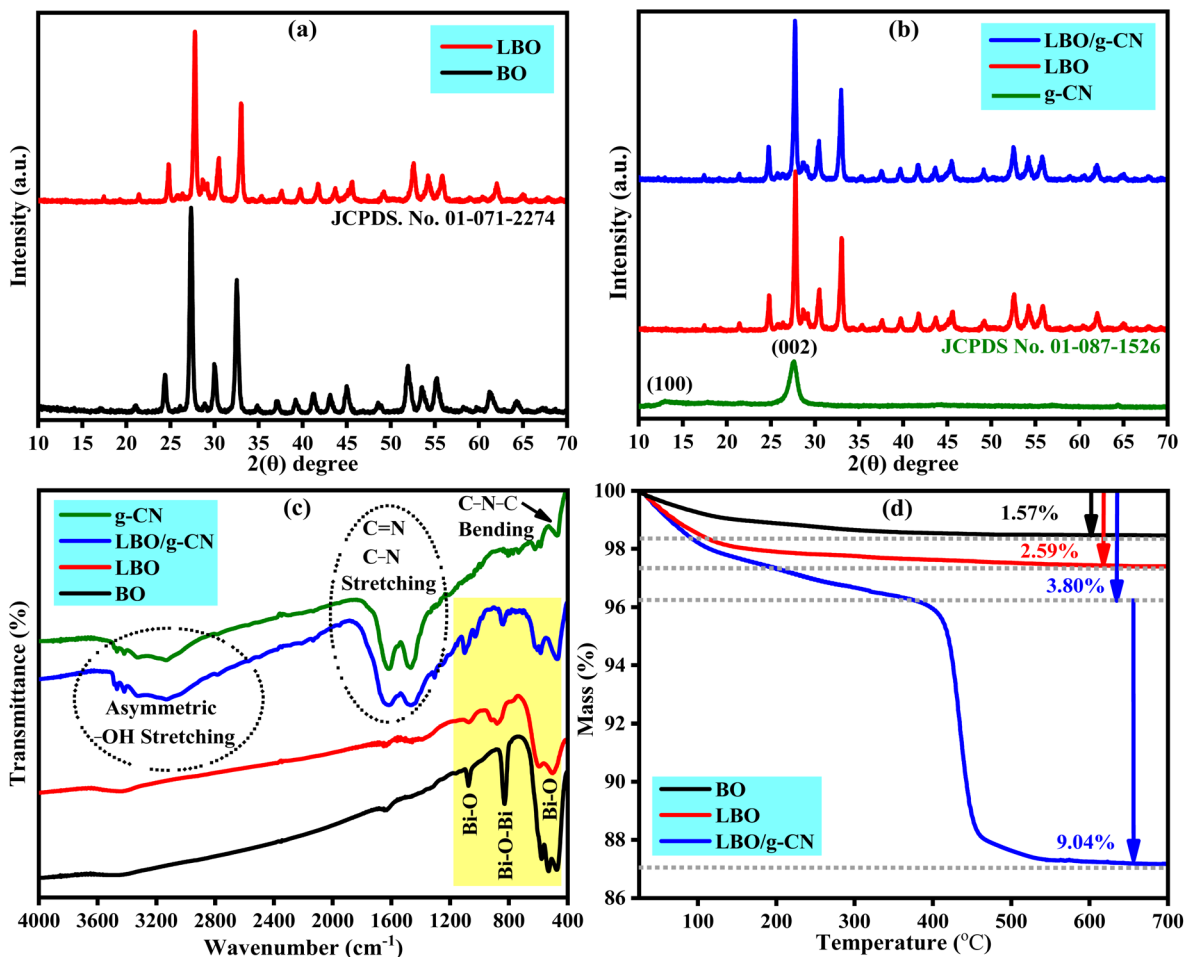


Fig. 2 (a and b) Diffraction patterns of the designed BO, LBO, g-CN, and LBO/g-CN, (c) FT-IR spectra displaying characteristic vibrations, and (d) TGA curves showcasing the thermal stability of the catalysts.



the dark while stirring for adsorption–desorption of pollutant at the catalyst surface. After about 30 minutes, the aliquots were exposed to visible light (Xe lamp, 300 W) to start the photocatalytic degradation of MB and CPF. The small portion from the degrading mixtures was separated at regular time intervals, and the absorbance of the sample drawn was monitored to estimate the concentration of pollutants. The efficiency of the photocatalysts was estimated by calculating the degradation (%) by applying eqn (1), while for the kinetics study of MB and CPF degradation, eqn (2) was applied. The scavenging investigation was performed by adding quenchers, including IPA, EDTA-2Na, and BQ, to the photocatalyst LBO/g-CN.

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

$$-\ln \frac{C_t}{C_0} = Kt \quad (2)$$

### 3. Results and discussion

#### 3.1 Structural characteristics and thermal stability

The synthesized materials' phase formation, purity, and crystallite size were determined from their XRD patterns, which are displayed in Fig. 2(a and b). The Bragg's diffraction at  $2\theta = 21.10^\circ, 34.36^\circ, 32.58^\circ, 34.10^\circ, 37.03^\circ, 39.26^\circ, 41.14^\circ, 43.20^\circ, 44.91^\circ, 48.68^\circ, 51.93^\circ, 53.64^\circ, 55.19^\circ, 61.18^\circ, \text{ and } 64.26^\circ$  corresponds to the (020), (−102), (−211), (−212), (−131), (−222), (−213), (−123), (023), (−232), (−104), (−313), (−224), (232), (−115) crystallographic planes and coincided with the JCPDS No. 01-071-2274 in good agreement, confirming the formation of the designed monoclinic  $\text{Bi}_2\text{O}_3$  phase ( $P2_1/c$ ).<sup>39</sup> The sharp and narrow peaks in the diffraction patterns of the BO, LBO, and LBO/g-CN materials define the well-crystalline nature of these materials. The peaks at  $2(\theta)$  values of  $12.91^\circ$  and  $27.54^\circ$  in the diffraction profile of g-CN correspond to the (100) and (200) crystallographic planes (JCPDS No. 01-087-1527).<sup>40</sup> The slight variation of peak positions and intensity after La-doping and

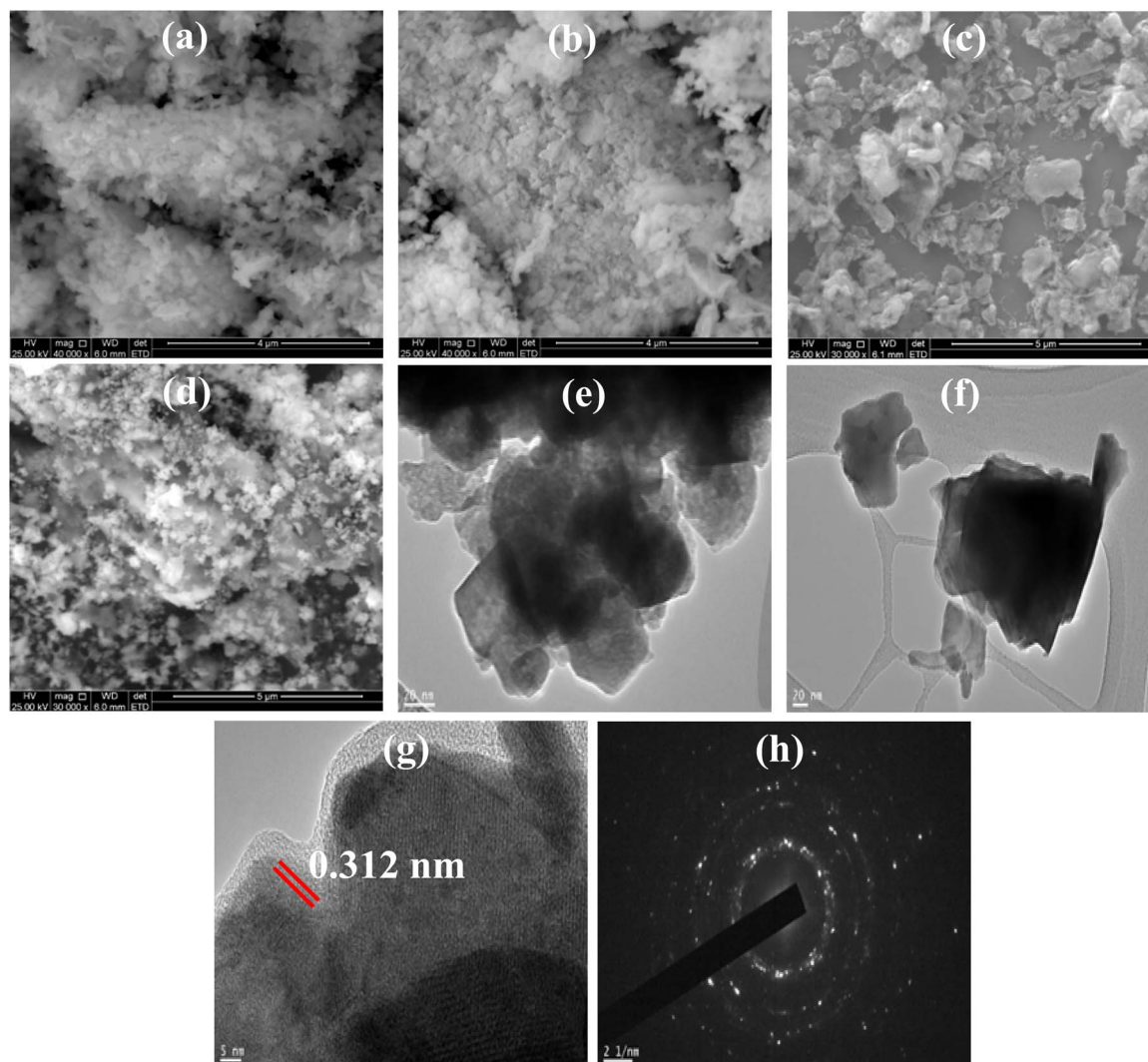


Fig. 3 SEM images of (a) BO, (b) LBO, (c) g-CN, and (d) LBO/g-CN, and TEM images of (e) LBO/g-CN, (f) g-CN, and (g and h) HRTEM and SAED pattern of LBO/g-CN.



anchoring doped material on g-CN reflects a change in crystallographic characteristics, primarily due to structural deformation leading to defective regions.<sup>41–43</sup> The average crystallite sizes (calculated by applying Scherrer's formula  $D = 0.9\lambda/\beta \cos \theta$  (ref. 44)) of BO, LBO, and LBO/g-CN were 24.41 nm, 26.31 nm, and 29.45 nm, respectively, which is possibly due to lattice strain relaxation, which facilitates the crystallite growth.<sup>45</sup>

The functional properties of the synthesized materials with characteristic metal–oxygen vibrations were confirmed by FT-IR spectra, as displayed in Fig. 2(c). The characteristic Bi–O stretching ( $\sim 1077 \text{ cm}^{-1}$ ), Bi–O–Bi stretching ( $\sim 830 \text{ cm}^{-1}$ ), and Bi–O bending ( $\sim 522 \text{ cm}^{-1}$ ) in the fingerprint region confirm the formation of the designed material.<sup>46–48</sup> In the FT-IR pattern of the synthesized g-CN, the C=N stretching ( $\sim 1623 \text{ cm}^{-1}$ ), C–N stretching ( $\sim 1469 \text{ cm}^{-1}$ ), and C–N–C bending ( $\sim 481 \text{ cm}^{-1}$ ) are typical peaks of g-CN, confirming its synthesis.<sup>49,50</sup> The wide band in the high-frequency range ( $3200\text{--}3600 \text{ cm}^{-1}$ ) is owing to –OH stretching and appeared due to adsorbed moisture.<sup>51,52</sup> The decrease in vibrational peak intensity of g-CN in LBO/g-CN, in contrast to its pure counterpart, confirms the interaction between these two materials forming LBO/g-CN heterostructures.<sup>53</sup>

The TAG profiles of BO, LBO, and LBO/g-CN are displayed in Fig. 2(d). The insignificant mass loss of 1.57% and 2.59% for BO and LBO materials was due to moisture removal, confirming the phase stability of the materials. For the LBO/g-CN hetero-junction architect, mass loss in two temperature zones was observed. The first mass loss (3.80%) was due to the removal of adsorbed moisture, while the second mass loss (9.04%) in the temperature range of  $400\text{--}600 \text{ }^\circ\text{C}$  was carbon content combustion of g-CN.<sup>54</sup> The mass loss observed for the g-CN removal was much closer to the amount used for LBO/g-CN architecting.

### 3.2 Morphological study

The SEM and TEM images of as-synthesized BO, LBO, g-CN, and LBO/g-CN are presented in Fig. 3. The pure bismuth oxide exhibits dispersion of irregular-sized microcrystallites featuring irregular symmetry. La-doping affected the overall size of crystallites, but the irregularity of size and symmetry was retained, as evidenced by the SEM image of the LBO material. The synthesized g-CN features dispersed flakes that have an irregular shape. In contrast, by forming a composite with LBO to architect the LBO/g-CN hetero-junction, the g-CN flakes were

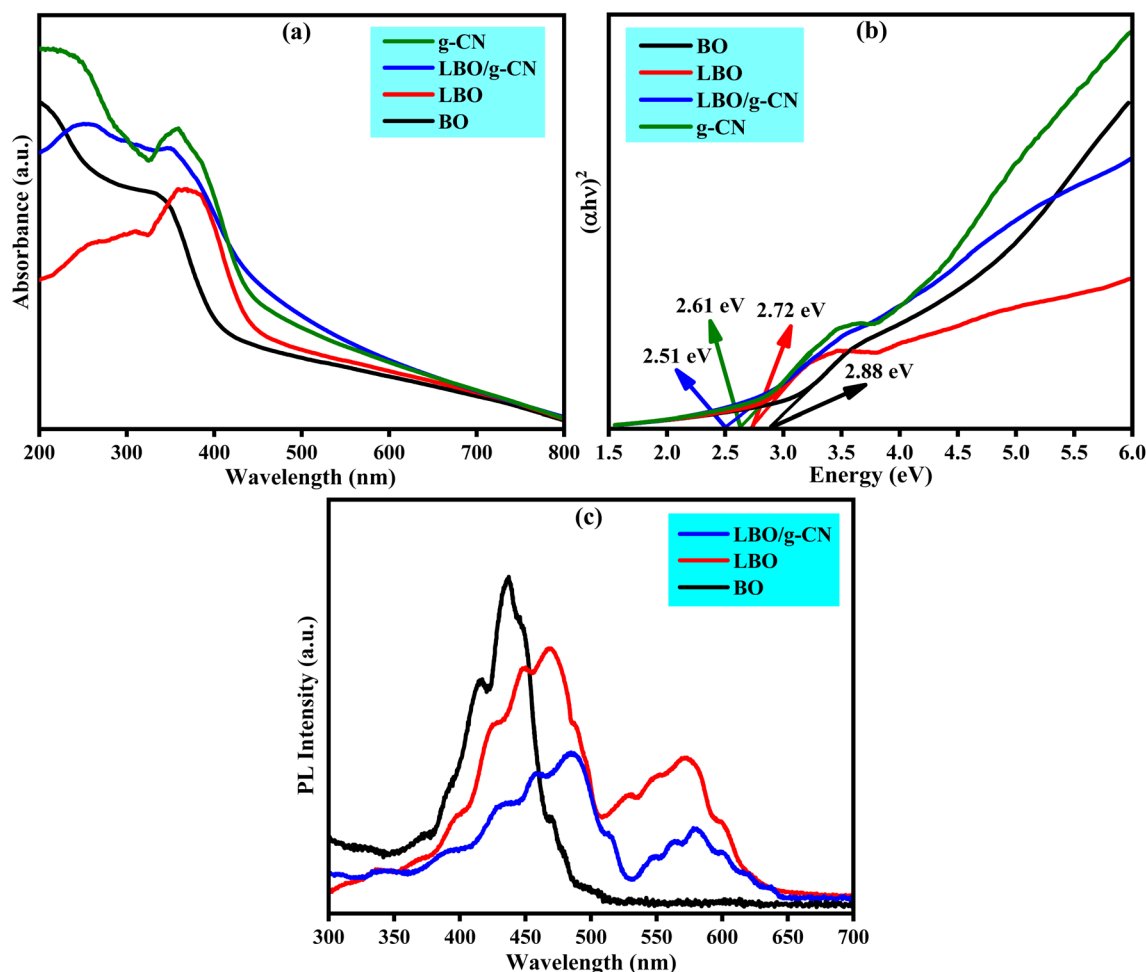


Fig. 4 (a) Absorption (200–800 nm) spectra of BO, LBO, g-CN, and LBO/g-CN, (b) Tauc plots displaying the optical bandgaps, and (c) PL spectra of the catalysts.



covered with the dispersion of LBO crystallites, representing the uniformity of the composite. This dispersion will facilitate the interfacial charge transfer by delaying the possible charge recombination, which is promising for photocatalytic activity.<sup>55</sup> The TEM images of pure g-CN and LBO/g-CN heterojunction materials are displayed in Fig. 3(e and f). The LBO material is a hybrid with g-CN flakes that have a crystalline nature. The lattice fringes feature an interplanar spacing of 0.312 nm corresponding to the (−102) plane, as showcased in the HRTEM image of LBO/g-CN.

### 3.3 Optical properties

The optical activity, which plays a substantial role in visible-light-driven catalytic activity, was investigated by recording the absorption ( $\lambda = 200\text{--}800\text{ nm}$ ) of the designed BO, LBO, g-CN, and LBO/g-CN materials, and the absorption spectra are displayed in Fig. 4(a). The pure BO material shows an absorption edge near the start of the visible light region, which is redshifted by La-doping, as can be evident from the absorption of LBO material. This shift is exhibited due to band structure deformation by generating sub-states within the valence and

conduction bands, which triggers visible light activation.<sup>56,57</sup> The pure g-CN displays visible light activation, which was further increased by anchoring the LBO material. In contrast to its pure counterparts, the assembled heterojunction LBO/g-CN with enhanced visible light activity could be a potential photocatalyst for visible light harvesting. The optical energy ( $E_g$ ) gap was determined by the Tauc plot equation (Fig. 4(b)), and its values were 2.88 eV, 2.72 eV, 2.61 eV, and 2.51 eV for BO, LBO, g-CN, and LBO/g-CN. The decline in  $E_g$  can be credited to an active heterojunction architecture with the conduction of separated charges, which is promising for solar-driven applications.<sup>58,59</sup> The suppression of the charge recombination phenomenon was studied by the PL spectroscopic analysis, and the results are depicted in Fig. 4(c). The radiant decay was of high intensity for the pure BO material, which highlights the rapid recombination that was delayed by La-doping and suppressed significantly by the heterojunction LBO/g-CN formation as the PL intensity declines after modifications. The heterojunction construction prevents the recombination by spatial separation of the charge carriers, where the negative electrons move to the semiconductor with a lower conduction band and the positive holes

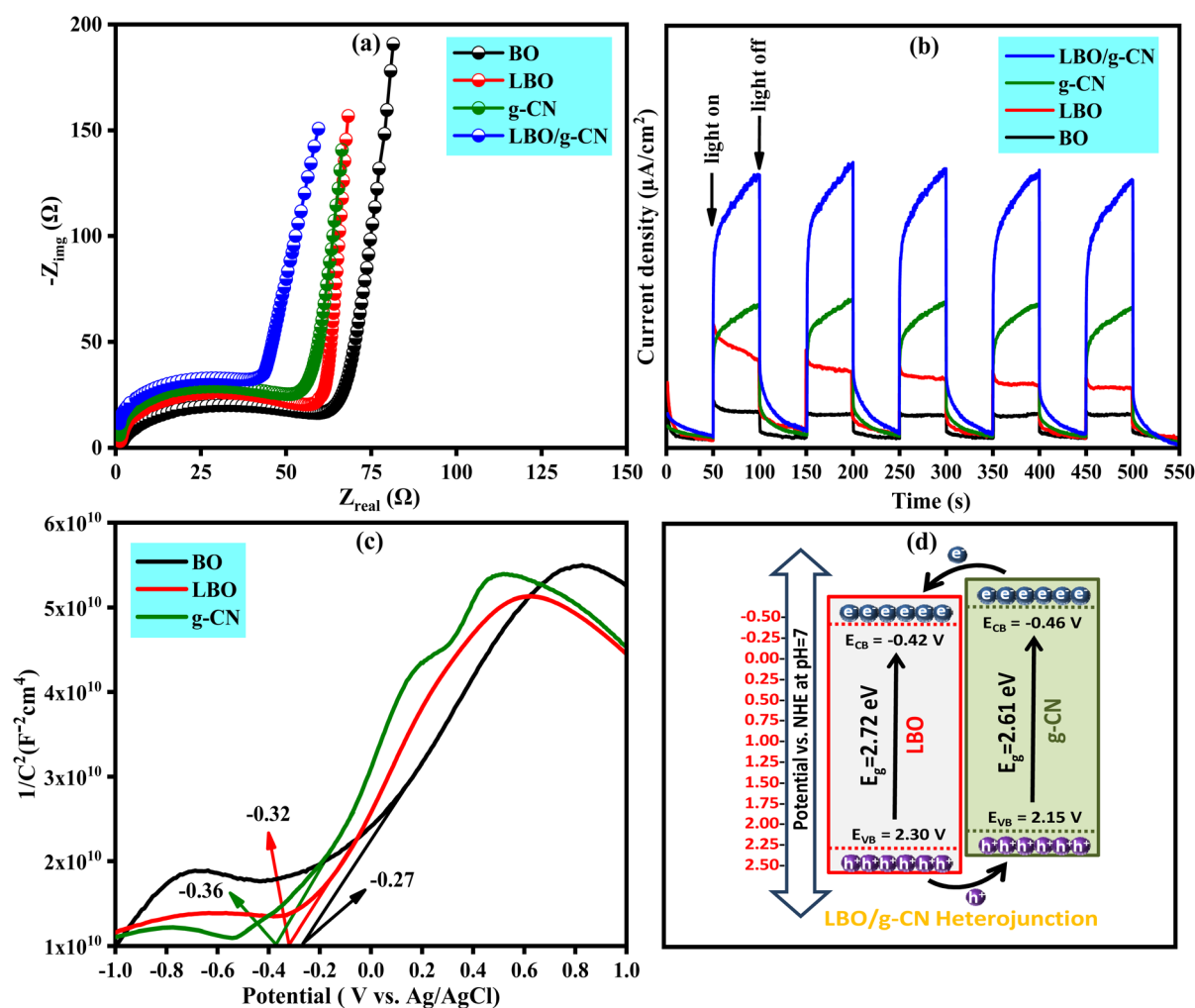


Fig. 5 (a) EIS Nyquist plots, (b) photocurrent response during light-on light-off cycles, (c) Mott–Schottky plots, and (d) schematic display of the designed LBO/g-CN heterojunction (Type-II).



to the semiconductor with a higher valence band, preventing their fast recombination.<sup>60</sup>

### 3.4 Electrochemical properties

The as-designed materials BO, LBO, and LBO/g-CN were subjected to electrochemical characterizations, including EIS, photocurrent response, and Mott-Schottky analysis, to get insights into the charge transfer characteristics, which significantly drive photocatalytic performance. The electrochemical measurements were performed using modified indium-tin-oxide ITO glass, ITO-BO, ITO-LBO, ITO-g-CN, and ITO-LBO/g-CN as working electrodes on a three-electrode potentiostat. The auxiliary and reference electrodes were a Pt-plate and Ag/AgCl in 1 M Na<sub>2</sub>SO<sub>4</sub> electrolyte. The Nyquist plots for BO, LBO, g-CN, and LBO/g-CN materials are featured in Fig. 5(a). The Nyquist semicircle arc radii are inversely proportional to the material's interfacial charge (electron) transfer.<sup>61</sup> The smaller Nyquist radii for the LBO/g-CN heterojunction indicate a higher electrode reaction rate, reflecting lower charge transfer resistance at the photocatalyst-electrolyte interface due to a more rapid interface electron transfer.<sup>62</sup>

The photocurrent response was recorded under a visible-light source to explore the dynamics of charge separation and

transfer, and the photocurrent generated during light-on and light-off cycles for the sample BO, LBO, g-CN, and LBO/g-CN materials is displayed in Fig. 5(b). The greater photocurrent intensity for the LBO/g-CN heterojunction, in contrast to the BO, LBO, and g-CN, showcases higher charge separation and transfer due to the low recombination rate of the charge carriers.<sup>63,64</sup> The results reveal prompt interfacial charge separation and transfer for the architected LBO/g-CN heterojunction, which are promising for photocatalytic activity.

The Mott-Schottky analysis revealed the semiconductor nature and the synthesized materials' band structure. The Mott-Schottky plots generated for the BO, LBO, g-CN, and LBO/g-CN are displayed in Fig. 5(c). The tangents drawn to the linear part of the curves at the horizontal axis exhibit a positive slope, which features n-type semiconductor formation.<sup>65</sup> The flat-band potentials ( $E_{fb}$ ) of BO, LBO, and g-CN were determined to be  $-0.27$  V,  $-0.32$  V, and  $-0.36$  V, respectively. The  $E_{fb}$  is almost equal to the Fermi level for n-type semiconductors, and its conduction band potential ( $E_{CB}$ ) is generally 0.1 V more negative than its  $E_{fb}$ .<sup>66</sup> Following this general agreement,  $E_{CB} = E_{fb} - 0.1$ , the  $E_{CB}$  of the LBO and g-CN are  $-0.42$  V and  $-0.46$  V, while their  $E_{VB}$  was determined by using the relation  $E_{VB} = E_{CB} + E_g$ , where  $E_g$  is the optical energy bandgap value.<sup>67,68</sup> The  $E_{VB}$  was

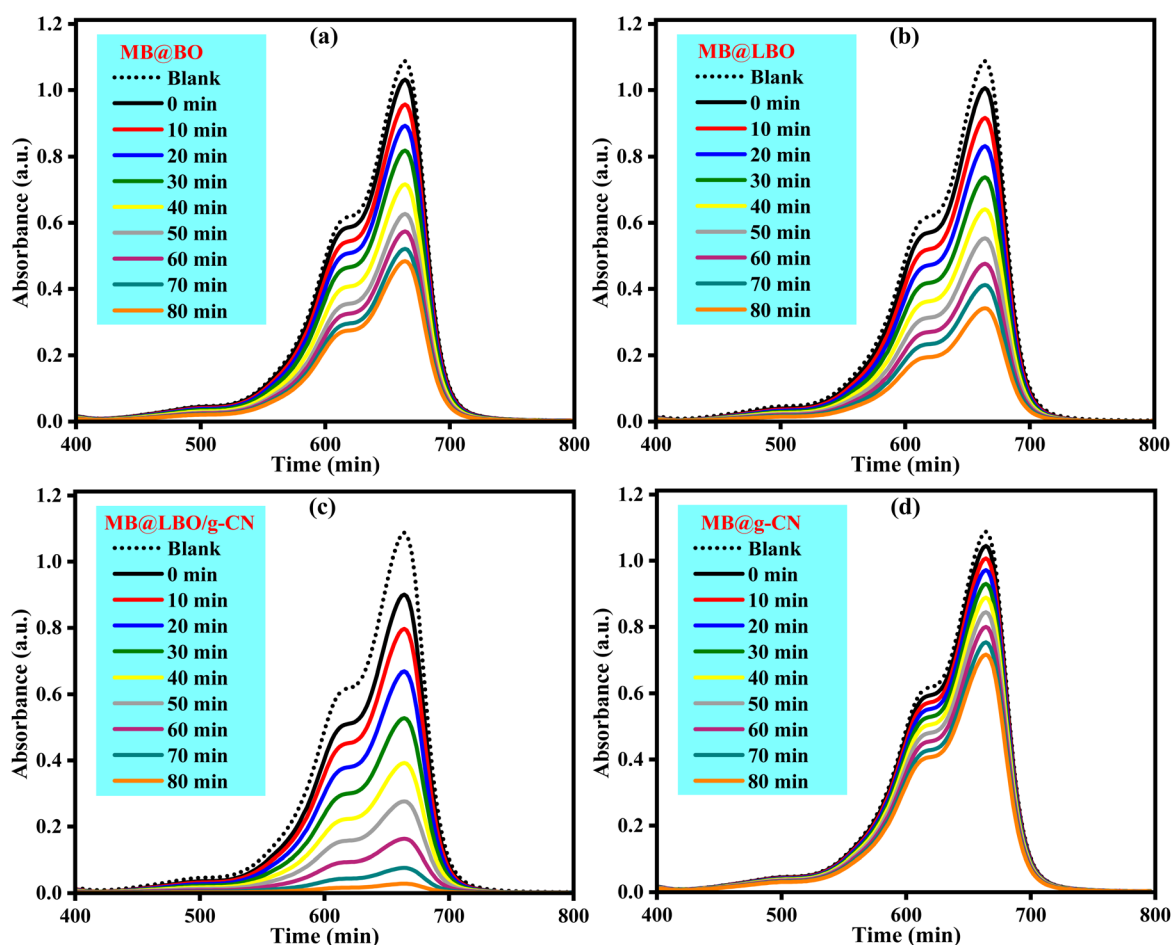


Fig. 6 UV-vis absorption (overlaid) of the samples separated from the aliquot during MB photodegradation over defined time intervals in the presence of (a) BO, (b) LBO, (c) LBO/g-CN, and (d) g-CN.



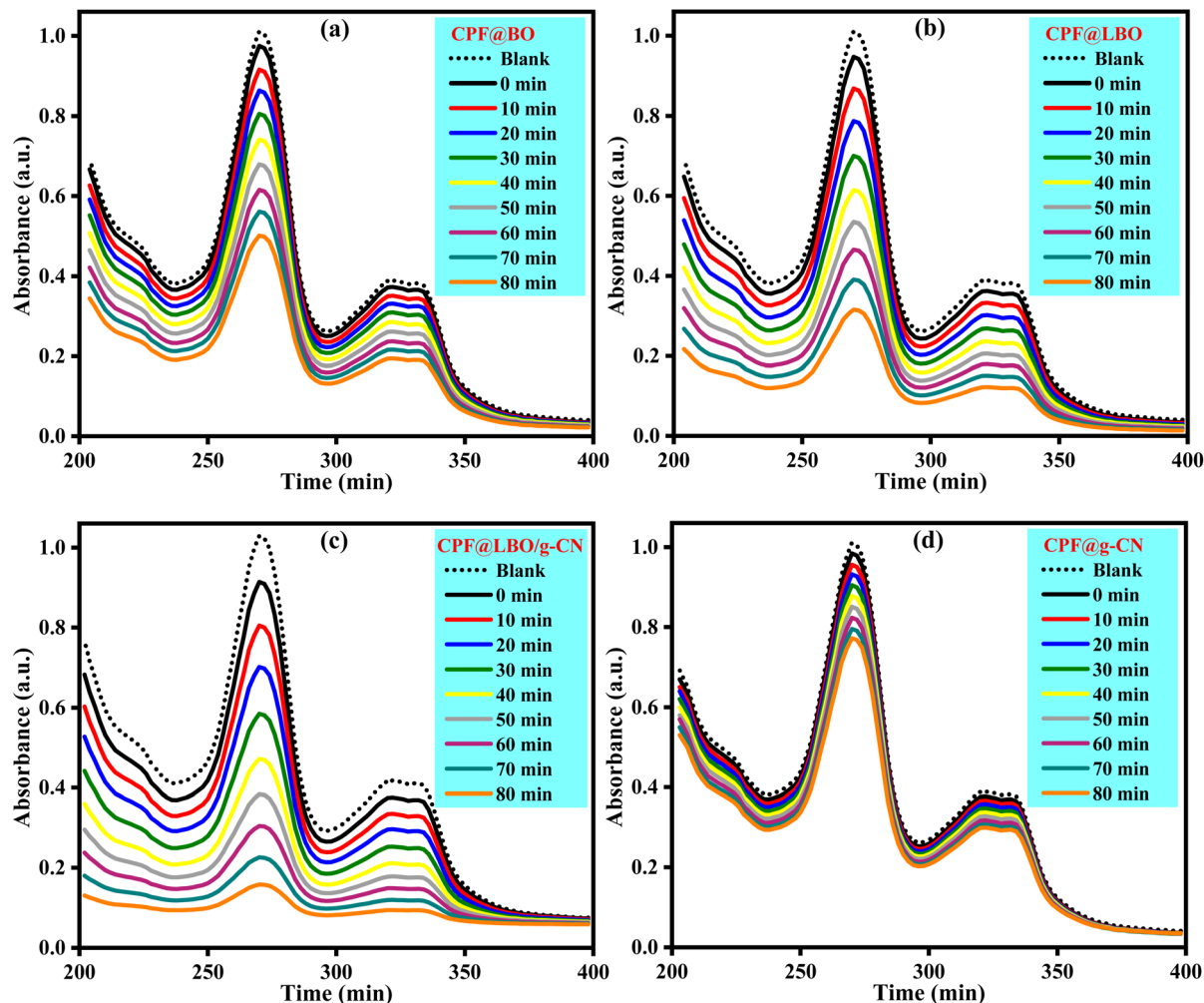


Fig. 7 UV-vis absorption (overlaid) of the samples separated from the aliquot during CPF photodegradation over defined time intervals in the presence of (a) BO, (b) LBO, (c) LBO/g-CN, and (d) g-CN.

calculated to be +2.30 V and +2.15 V for LBO and g-CN, respectively. Based on the band structure, the designed LBO/g-CN photocatalyst is diagrammatically presented in Fig. 5(d), showcasing improved charge separation due to heterostructures assembling for the photocatalytic activity.

### 3.5 Photocatalytic performance

The as-designed photocatalysts, including BO, LBO, g-CN, and LBO/g-CN, were exposed to visible light to investigate their photocatalytic activity for MB and CPF degradation. The results for degrading the typically targeted pollutants were compared to reveal the impact of La-doping and heterojunction designing with the g-CN material.

**3.5.1 MB degradation.** MB was degraded in the presence of BO, LBO, g-CN, and LBO/g-CN photocatalysts. The absorbance spectra ( $\lambda = 400\text{--}800\text{ nm}$ ) of the samples separated from the aliquot at the defined time intervals (after every 10 minutes) are presented in Fig. 6(a–c). The MB degradation without the catalyst was negligible, as no decrease in MB concentrations was observed, which indicates that the MB is not prone to

degrade under the same conditions without a photocatalyst. The decline in the absorption peak intensity at  $\lambda_{\text{max}}$  showcases a decrease in the MB over time, which is more prominent for the heterojunction-based photocatalyst LBO/g-CN, highlighting its significant photocatalytic performance.

**3.5.2 CPF degradation.** CPF was degraded in the presence of as-designed catalysts to explore the photocatalytic performance and the non-specific pollutant degradation nature of the prepared photocatalyst. The absorption spectra ( $\lambda = 400\text{--}800\text{ nm}$ ) taken at different time intervals are overlaid and presented in Fig. 7(a–c). The sharp decline in CPF concentration in the presence of LBO/g-CN demonstrates the higher photocatalytic activity of the designed heterojunction.

**3.5.3 Kinetics of degradation.** The reaction kinetics of MB and CPF degradation were examined by applying the kinetics model, and the best fit was the 1st-order kinetics ( $-\ln C_t/C_0$  vs.  $t$ ) model as presented in Fig. 8(a and b).<sup>69</sup> The rate constant ( $k$ ) was calculated from the slope of the linear fit of  $-\ln C_t/C_0$  against time. The  $k$  values for MB degradation in the presence of BO, LBO, and LBO/g-CN catalysts were  $0.010\text{ min}^{-1}$ ,  $0.016\text{ min}^{-1}$ ,



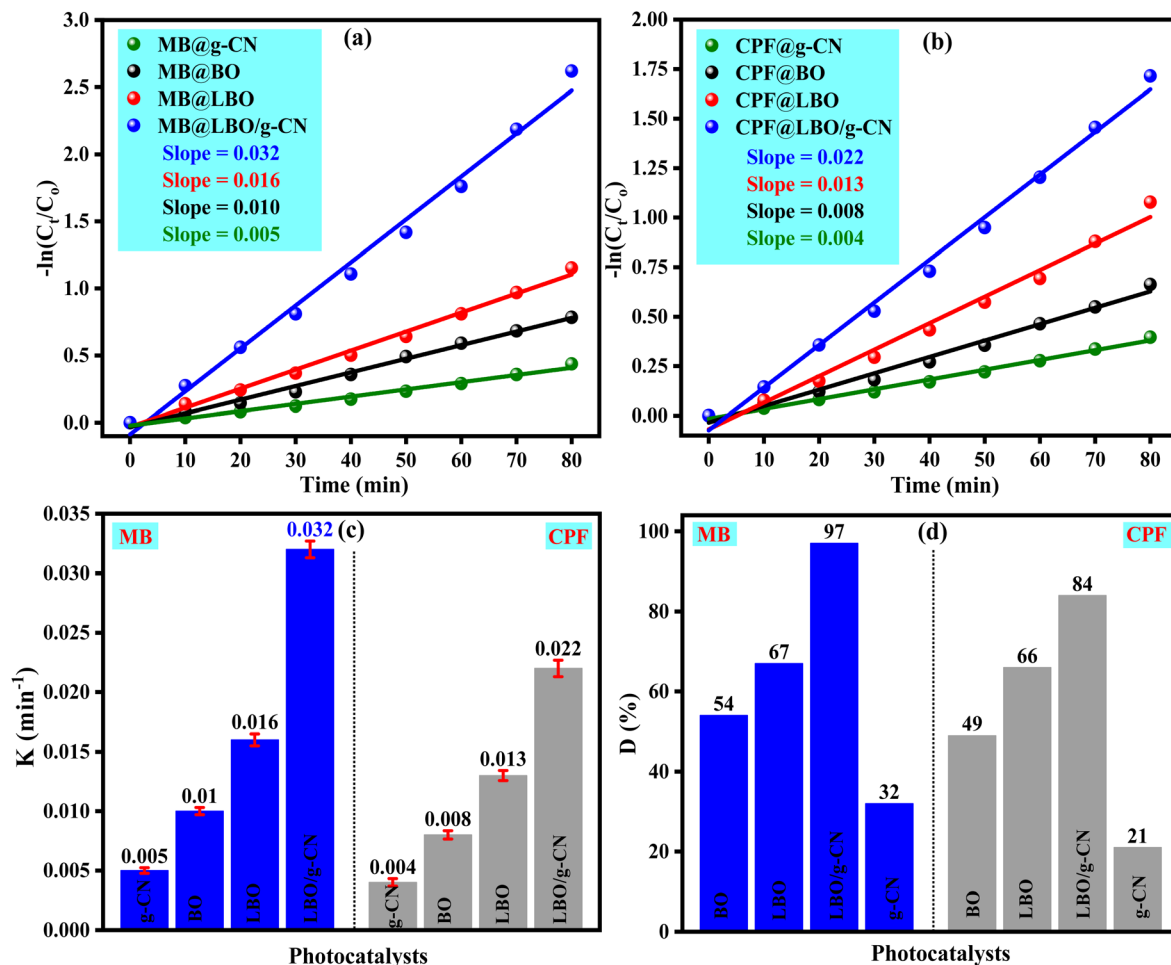


Fig. 8 (a and b) Kinetics study of MB and CPF degradation and (c and d) calculated rate constants and  $D$  (%) of MB and CPF over the g-CN, BO, LBO, and LBO/g-CN.

and  $0.032 \text{ min}^{-1}$ , while for CPF degradation, the rate constants were  $0.008 \text{ min}^{-1}$ ,  $0.013 \text{ min}^{-1}$ , and  $0.022 \text{ min}^{-1}$  in the presence of catalysts in a similar manner. The degradation ( $D$ , %) achieved for the targeted pollutants in the presence of BO, LBO, and LBO/g-CN catalysts was 54%, 67%, and 97% for MB, while CPF degraded to 49%, 66%, and 84%. The remarkable photocatalytic activity of the designed heterojunction LBO/g-CN for

the degradation of both MB and CPF discloses its promising potential in the photocatalytic removal of organic pollutants. This improved efficiency can be attributed to the tuned optical response and prompt charge separation and transfer dynamics.

The detailed comparison of the present study with  $\text{Bi}_2\text{O}_3$ -based photocatalysts is summarized in tabular form to showcase the efficiency of LBO/g-CN (Table 1).

Table 1 Comparative analysis of present work with previous studies

Sr. no.	Photocatalyst	Typical pollutant	Light source, catalyst dose, and pollutant concentration	Degradation (%) / time (min)	Reference
1	$\text{CuO}/\text{Bi}_2\text{O}_3$	Rhodamine B	Visible-light, 35 mg, 10 ppm	95.6%/180 min	70
2	$\text{Ce}-\text{Bi}_2\text{O}_3$	Methyl orange	Visible-light, 1 g, 20 ppm	83%/120 min	71
3	$\text{Se}-\text{Bi}_2\text{O}_3$	Methylene blue	Visible-light, 0.05, 3.2 ppm	88%/270	72
4	$\text{Au}-\text{Bi}_2\text{O}_3$	Rhodamine B	Visible-light, 1 g, 4.8 ppm	80%/180 min	73
5	$\text{Bi}_2\text{O}_3/\text{biochar}/\text{g}-\text{C}_3\text{N}_4$	Tetracycline	Visible-light, 0.06 g, 20 ppm	86.7%/30 min	74
6	$\text{Bi}_2\text{O}_3/\text{activated carbon}$	Methyl orange	Visible-light, 0.2 g, 200 ppm	72.5%/80 min	75
7	$\text{Bi}_2\text{O}_3/\text{MWCNTs}$	Cr(vi)	Visible-light, 1 g, 10 ppm	87.48%/480 min	76
8	$\text{Bi}_2\text{O}_3@-\text{g}-\text{C}_3\text{N}_4$	Tetracycline	Visible-light, 0.5 g, 10 ppm	80.2%/50 min	77
9	$\text{La}-\text{Bi}_2\text{O}_3/\text{g}-\text{C}_3\text{N}_4$	Methylene blue/Ciprofloxacin	Visible light, 0.05 g, and 20 ppm	97%/84%/80 min	Present study



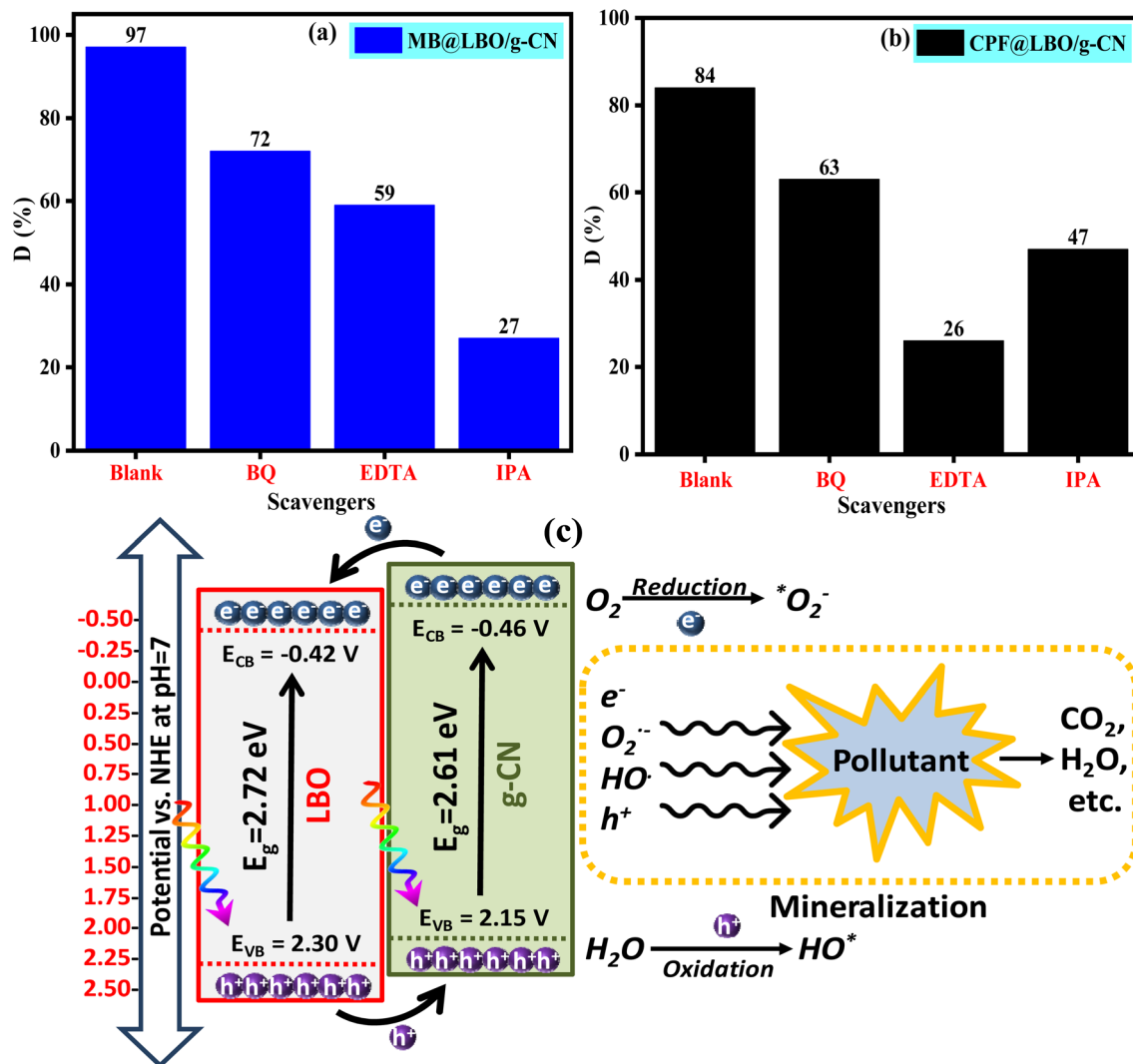


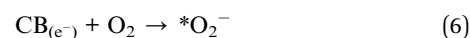
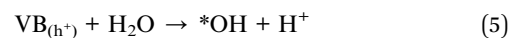
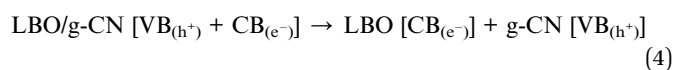
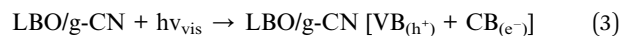
Fig. 9 (a) MB and (b) CPF degradation over catalyst LBO/g-CN along with scavengers, and (c) schematic display of degrading MB and CPF over the heterojunction LBO/g-CN.

### 3.5.4 Proposed mechanism of MB and CPF degradation.

The photocatalytic MB and CPF degradation involve the production of reactive oxygen species, including superoxide radical anions ( ${}^*O_2^-$ ) and hydroxyl radicals ( $HO^*$ ) by the redox reaction of photo-separated charges, electrons in the conduction band, and holes in the valence band (electron-hole pair). Mechanistically, LBO/g-CN, under visible light exposure, undergoes electron excitation, and in turn, the electron is promoted to the conduction band  $E_{CB}$  ( $e^-$ ), generating holes in the valence band  $E_{VB}$  ( $h^+$ ). The slight difference in the  $E_{CB}$  and  $E_{VB}$  of the LBO and g-CN mitigates the recombination of separated charges by transferring and allowing these charge carriers to participate in a redox reaction, generating reactive oxygen species, which degrade the MB and CPF. The mechanism of MB and CPF degradation over LBO/g-CN catalyst is summarized in eqn (3)–(10).<sup>78–82</sup>

The production of these radical species and their relatively active involvement in MB and CPF degradation was tracked by

scavenging investigation. The catalyst LBO/g-CN, along with scavengers, including IPA for hydroxide radicals quenching, EDTA for holes trapping, and benzoquinone for superoxide radical anions trapping,<sup>83</sup> were added to the MB/CPF solution, and the degradation achieved is presented in Fig. 9(a and b). The MB degraded to 72%, 59%, and 27%, while CPF showed 63%, 26%, and 47% degradation in the presence of BQ ( ${}^*O_2^-$  quencher), EDTA ( $h^+$  quencher), and IPA ( ${}^*OH$  quencher) over the catalyst LBO/g-CN.



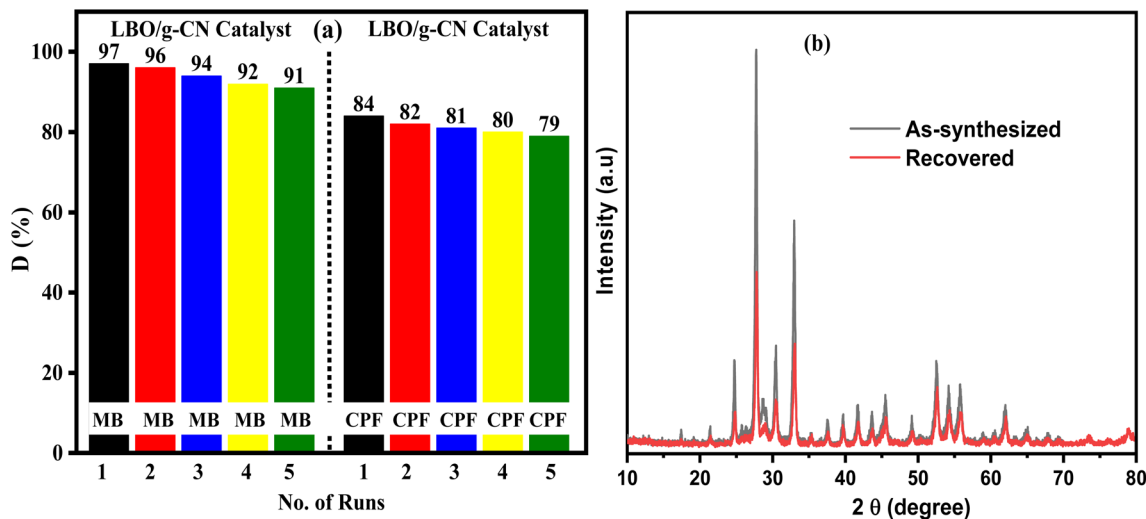
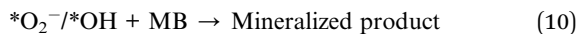
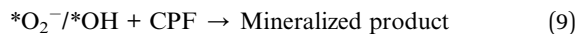
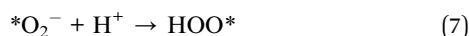


Fig. 10 (a) MB and CPF degradation (%) achieved during recycling of the LBO/g-CN catalyst and (b) diffraction pattern comparison of the as-synthesized and used photocatalyst to witness its stability.



**3.5.5 Reusability and stability.** The catalyst LBO/g-CN was recovered for MB and CPF degradation and reused to assess its stability and non-photocorrosive nature for practical applicability as a stable and reusable photocatalyst. After completing every cycle, the LBO/g-CN was recovered by centrifuging the aliquot after washing and oven drying before the subsequent cycle use. The catalyst features stability without relinquishing its efficiency (Fig. 10(a)), as the degradation (%) decrease was not substantial. The minor reduction in the efficiency can be attributed to catalyst dosage loss during its recovery or washing. After completing five cycles, the catalyst was recovered again, and stability was further explored by comparing the XRD patterns of the recovered catalyst to that of as-synthesized LBO/g-CN. No noticeable change was observed in the diffraction pattern of the recovered catalyst (Fig. 10(b)), which witnesses the stability of the designed heterojunction LBO/g-CN.

## 4. Conclusion

Pure bismuth oxide and its lanthanum-doped composition were fabricated by the economical and facile hydrothermal route, following the decoration of La-doped counterparts on g-CN by ultrasonication to assemble the heterojunction LBO/g-CN. XRD and FT-IR revealed the structural characteristics of the designed material, confirming the formation of BO, LBO, g-CN, and LBO/g-CN. The assembled LBO/g-CN exhibits excellent thermal stability, as witnessed by the TGA analysis. The SEM and TEM

results revealed the decoration of LBO material over the 2D g-CN leaves. The band structure of bismuth oxide was tuned by La-doping, which red-shifted the absorption edge, featuring absorption in visible light. The electrochemical properties revealed prompt interfacial charge transfer and a lower recombination rate for the LBO/g-CN heterojunction, reflecting its promising potential for catalytic applications. The designed heterojunction LBO/g-CN shows significantly enhanced MB and CPF degradation compared to its non-assembled counterparts, disclosing its tuned optical activity and prompt interfacial charge transfer. In visible light irradiation for 80 minutes, MB and CPF degraded to 97% and 84%, following 1st-order kinetics. Scavenging studies disclose the significant contribution of reactive oxygen species, hydroxide radicals for MB, and holes in CPF degradation. The reusability of the LBO/g-CN catalyst without relinquishing its efficiency showcases its stability. The designed recoverable heterojunction LBO/g-CN with visible light activation and prompt interfacial charge transfer could be a promising photocatalyst for the photocatalytic degradation of toxic organic compounds.

## Author contributions

M. M. Rashed (project administration, methodology, funding acquisition, resources). Mohamed Abdel Rafea (data curation, conceptualization). Mohamed I. Attia (data curation, formal analysis). Mohamed R. El-Aassar (writing – original draft, data curation, formal analysis). Tasneem I. Hussein (conceptualization, validation). Salma Saddeek (validation, formal analysis). Imran Shakir (writing – review & editing). Mazen R. Alrahili (visualization). Muhammad Aadil (supervision).

## Conflicts of interest

There are no conflicts to declare.



## Data availability

Data will be available upon request.

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