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# Rationally designed In–CeO<sub>2</sub>/g–C<sub>3</sub>N<sub>4</sub> S-scheme heterojunction photocatalyst with tuned redox ability for the photocatalytic degradation of pharmaceutical contaminants

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Herein, the synthesis of an indium-doped cerium oxide/graphitic carbon nitride (In–CeO<sub>2</sub>/g–C<sub>3</sub>N<sub>4</sub>) S-scheme heterojunction aimed at optimizing photocatalytic degradation under visible light for the remediation of pharmaceutical wastewater is reported. The materials were synthesized *via* a hydrothermal process, in which pure CeO<sub>2</sub> and In-modified CeO<sub>2</sub> (In–CeO<sub>2</sub>) were initially synthesized, followed by the incorporation of g–C<sub>3</sub>N<sub>4</sub> to produce the heterojunction. A series of characterization methods, such as X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), and scanning electron microscopy (SEM), validated the effective synthesis and structural integrity of CeO<sub>2</sub>, In–CeO<sub>2</sub>, and In–CeO<sub>2</sub>/g–C<sub>3</sub>N<sub>4</sub>. The optical bandgap of the samples was determined, presenting a reduction from 2.97 eV for CeO<sub>2</sub> to 2.69 eV for In–CeO<sub>2</sub>/g–C<sub>3</sub>N<sub>4</sub>, which facilitated better visible-light absorption. Photocurrent and electrochemical impedance spectroscopy (EIS) characterizations indicated enhanced charge separation and reduced recombination in the In–CeO<sub>2</sub>/g–C<sub>3</sub>N<sub>4</sub> heterojunction. Photocatalytic experiments for the degradation of levofloxacin (LVX) demonstrated that the In–CeO<sub>2</sub>/g–C<sub>3</sub>N<sub>4</sub> heterojunction achieved 85% degradation, significantly higher than those achieved by In–CeO<sub>2</sub> (63%) and CeO<sub>2</sub> (44%), highlighting the enhanced photocatalytic performance of the heterojunction. The higher photocatalytic activity is attributed to the formation of an S-scheme charge migration channel, enabling efficient charge separation. Results indicate that the In–CeO<sub>2</sub>/g–C<sub>3</sub>N<sub>4</sub> heterojunction has great potential for water purification applications, particularly in degrading drug contaminants.

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## 1. Introduction

Utilizing visible light for the photocatalytic degradation of organic pollutants is an economical and sustainable cleantech solution to the escalating environmental pollution and water contamination.<sup>1,2</sup> Despite its promise, the development of photocatalysts is a challenging process because the

conventional semiconductor materials, including TiO<sub>2</sub>,<sup>3,4</sup> ZnO,<sup>5,6</sup> and CeO<sub>2</sub>,<sup>7,8</sup> have low quantum efficiency due to their electronic band structures (wide optical bandgaps), which prevent harnessing the visible part (~43%) of solar light, and their photogenerated charge carriers (electron–hole pairs) recombine rapidly limiting their availability.<sup>9–11</sup>

Cerium oxide (CeO<sub>2</sub>), commonly known as ceria, is an emerging material in the realm of photocatalysts, which is resistant to photocorrosion and is chemically and thermally stable.<sup>12,13</sup> The applicability of ceria as a promising photocatalyst is explicitly limited by its weak visible-light activity and high electron–hole pair recombination, which are crucial factors for visible-light-driven photocatalysis.<sup>14–16</sup> Several investigations, including metal-doping, indicate that cerium oxide modification tunes the electronic structure and enhances the quantum efficiency.<sup>16–19</sup> The rapid electron–hole recombination can be suppressed by coupling ceria with another photocatalytic material through the formation of heterostructure junctions, thereby increasing the availability of charge carriers for photocatalytic reactions.<sup>20–23</sup>

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Assembling two different photocatalytic materials with distinct electronic properties to construct heterostructure junctions enhances charge separation, which is crucial for significant photocatalytic performance.<sup>24–26</sup> These materials possess distinct electronic band structures that facilitate the migration and separation of photogenerated electron–hole pairs, enhancing the photocatalytic performance.<sup>27,28</sup> For instance, S-scheme heterojunction formation involves the interfacial contact of two photocatalysts, in which band structure alignment results in the accumulation of holes in the valence band of one of the combining photocatalysts and electrons in the conduction band of the other photocatalyst, which is mainly compelled by the internal electric field.<sup>29</sup> Graphitic carbon nitride  $g\text{-C}_3\text{N}_4$  ( $g\text{-CN}$ ), a polymeric compound with a layered structure of tri-s-triazine units, exhibits high thermal and chemical stability with excellent visible-light activity. The distinguished properties, which include non-toxicity, a unique metal-free framework, and a semiconductor with a medium band gap, enable its use as a sustainable catalyst for photo-degradation applications. Despite these characteristics,  $g\text{-CN}$  exhibits low quantum efficiency due to a low absorptivity coefficient and sluggish electron–hole separation, which can be improved by modifying  $g\text{-CN}$ . Various reports present  $g\text{-CN}$  as a promising material for the fabrication of heterostructure junctions, as its unique structural framework favours interfacial contact.<sup>30,31</sup> For instance,  $\text{ZrO}_2/g\text{-CN}$ ,<sup>32</sup>  $\text{Ce}(\text{MoO}_4)_2/g\text{-CN}$ ,  $g\text{-CN}/\text{Bi}_2\text{O}_2\text{CO}_3\text{-Bi}_4\text{O}_7$ ,<sup>33</sup>  $\text{Nd}/\text{Ni-LaFeO}_3/g\text{-CN}$ ,<sup>34</sup>  $\text{CdMoO}_4/g\text{-CN}$ ,<sup>35</sup>  $\text{ZnO}/g\text{-CN}$ ,<sup>36</sup>  $\text{Ni-MOF}/g\text{-CN}$ ,<sup>37</sup> and  $\text{ZnAl-LDH}/g\text{-CN}$ <sup>38</sup> include the  $g\text{-CN}$ -based material designs for wastewater decontamination applications. The findings reveal that these composite designs not only increase the optical response but also facilitate charge separation and transport primarily through the formation of heterojunctions.

Based on the facts described above, we synthesized a visible-light-activated  $\text{In-CeO}_2/g\text{-CN}$  S-scheme heterojunction to facilitate charge separation and transport, thereby augmenting the effective photocatalytic removal of pharmaceutical pollutants. The hydrothermal method was used to synthesize pure  $\text{CeO}_2$  and its indium-modified form,  $\text{In-CeO}_2$ , while melamine was calcinated for its thermal polymerization to prepare  $g\text{-CN}$ . Both the components,  $\text{In-CeO}_2$  and  $g\text{-CN}$ , were subjected to ultrasonication to fabricate the  $\text{In-CeO}_2/g\text{-CN}$  heterojunction. The synthesized materials were characterized to investigate their crystal structure formation, thermal stability, microstructure, optical activity, and electrochemical response. The photocatalytic efficiency of  $\text{In-CeO}_2/g\text{-CN}$  was estimated by degrading levofloxacin, a typical pollutant, showcasing the effect of heterojunction formation on photocatalytic performance. The present work aims to significantly contribute to the design of heterojunction materials for the photocatalytic degradation of pharmaceutical contaminants in wastewater remediation.

## 2. Experimental

### 2.1 Chemicals

The chemicals, including ceric ammonium nitrate (Sigma-Aldrich,  $(\text{NH}_4)_2[\text{Ce}(\text{NO}_3)_6]$ ,  $\geq 98.5\%$ ), indium nitrate (Sigma-

Aldrich,  $\text{In}(\text{NO}_3)_3$ , 99.99%), and melamine (Sigma-Aldrich, Powder, 99%), were used in the synthesis of  $\text{CeO}_2$ ,  $\text{In-CeO}_2$ , and  $\text{In-CeO}_2/g\text{-CN}$  materials. Levofloxacin (LVX,  $\text{C}_{18}\text{H}_{20}\text{FN}_3\text{O}_4$ ), ethylenediaminetetraacetic disodium salt (Sigma-Aldrich, EDTA-2Na, 99%), silver nitrate ( $\text{AgNO}_3$ ), isopropyl alcohol (Sigma-Aldrich, IPA, 99%), and benzoquinone (Sigma-Aldrich, BZQ, 99%) were used in the photocatalytic activity investigation. All the standards were prepared in DI water ( $k < 6 \mu\text{S cm}^{-1}$ ).

### 2.2 $\text{CeO}_2$ and $\text{In-CeO}_2$ synthesis

The facile hydrothermal method was applied to synthesize pure  $\text{CeO}_2$  and  $\text{In-CeO}_2$  materials. Initially, 3 g of  $(\text{NH}_4)_2[\text{Ce}(\text{NO}_3)_6]$  was added to 70 mL of DI water while stirring until the formation of a clear solution, followed by the addition of 0.15 g  $\text{In}(\text{NO}_3)_3$ . The pH was raised to  $\sim 10$  with drop-by-drop addition of  $\text{NH}_4\text{OH}$ , and the solution was poured into a Teflon cup enclosed in an autoclave (stainless steel-made), following the heating of the solution at 180 °C for 12 h. The precipitates collected were thoroughly washed with DI water multiple times to neutralize the pH and remove unreacted precursors, and then dried at 55 °C for 2 h.

### 2.3 Preparation of $g\text{-CN}$ powder

The  $g\text{-CN}$  powder was prepared from melamine through thermal condensation.<sup>39</sup> Experimentally, 2 g of the precursor powder was placed in a 25 cc (porcelain) crucible and heated at 550 °C for 6 h at 5 °C  $\text{min}^{-1}$ . Upon the completion of the heating process, the crucible was removed from the furnace and allowed to cool naturally in the air. The pale, yellowish-colored  $g\text{-CN}$  formed was collected and stored in a dry environment.

### 2.4 $\text{In-CeO}_2/g\text{-CN}$ construction

The  $g\text{-CN}$ -based heterojunction of modified  $\text{In-CeO}_2$  was formed using an ultrasonication method. In the experiment, 0.9 g of  $\text{In-CeO}_2$  was poured into DI water (100 mL) and sonicated with an ultrasonic probe for 1 hour. Similarly, in a beaker (100 mL, DI water), 0.1 g of  $g\text{-CN}$  powder was separately sonicated. The resulting suspensions were mixed, followed by sonication again for 1 hour to enhance the interaction and dispersion of  $\text{In-CeO}_2$  with  $g\text{-CN}$ , assembling the  $\text{In-CeO}_2/g\text{-CN}$  heterojunction (Fig. 1).

### 2.5 Characterization

The crystal structure formation and phase purity of the fabricated materials were investigated by powder XRD (X-ray Diffractometer, Shimadzu 6100 AS, Cu-K $\alpha$  radiation,  $\lambda = 0.154$  nm) and FTIR spectroscopy (Shimadzu IRAffinity-1S Spectrophotometer), while for thermal stability, TGA (Thermo Plus Evo, TG8120 Rigaku) was performed. The morphology was explored by SEM (Scanning electron microscope, FEI S50) and optical activity by UV-vis spectroscopy (Double-Beam Spectrophotometer, Jenway/6850). The electrochemical response was recorded on a three-electrode potentiostat (reference electrode =  $\text{Ag}/\text{AgCl}$ , working electrode = indium tin oxide-coated glass, and



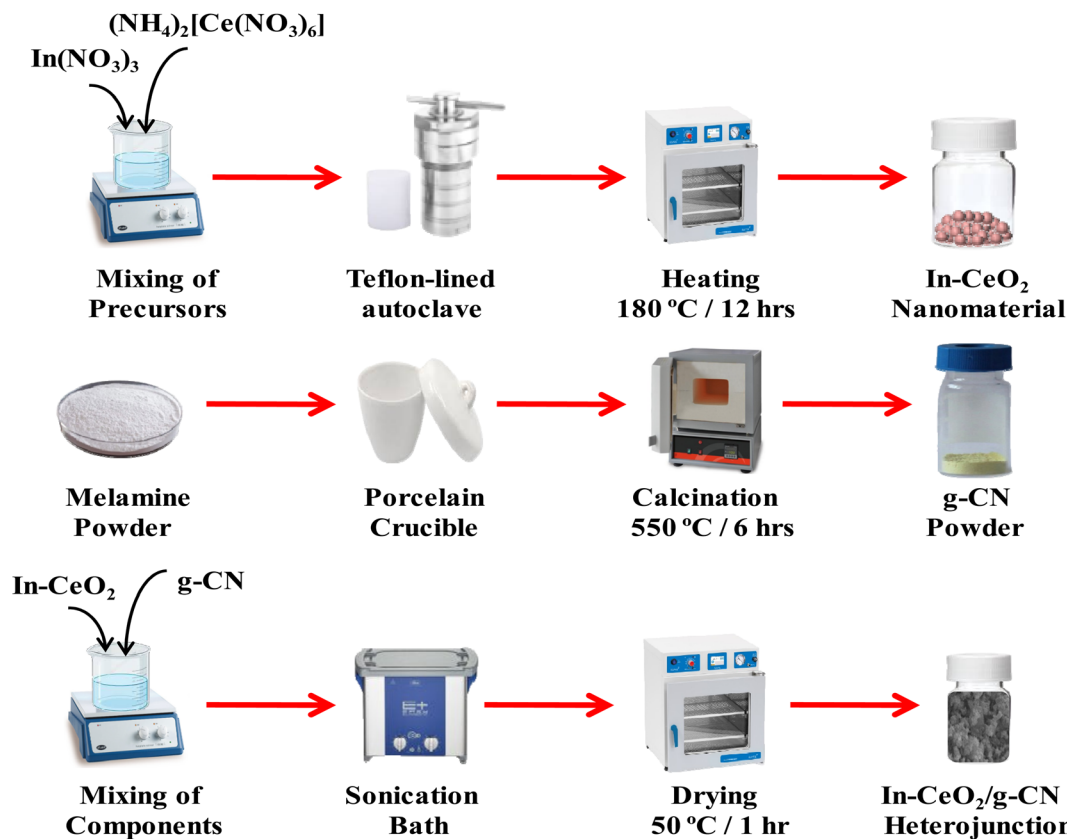


Fig. 1 Schematic of the synthesis of  $\text{CeO}_2$  and  $\text{In-CeO}_2$  (hydrothermal),  $\text{g-CN}$  (calcination), and  $\text{In-CeO}_2/\text{g-CN}$  (ultrasonication).

auxiliary electrode = Pt wire) in 1 M  $\text{Na}_2\text{SO}_4$  electrolytic solution.

## 2.6 Photocatalytic (PC) efficiency

The PC efficiency of  $\text{CeO}_2$ ,  $\text{In-CeO}_2$ , and  $\text{In-CeO}_2/\text{g-CN}$  photocatalysts was evaluated by degrading LVX under a Xe lamp (300 W, UV cutoff filter  $\lambda > 420$  nm) placed 20 cm above the reaction mixture. Initially, 0.05 g of  $\text{CeO}_2$ ,  $\text{In-CeO}_2$ , and  $\text{In-CeO}_2/\text{g-CN}$  were dispersed in LVX ( $10 \text{ mg L}^{-1}$ , 100 mL) solutions in three separate beakers. The resultant mixtures (LVX + photocatalyst) were kept in the dark and stirred for 40 minutes to acquire LVX adsorption-desorption equilibria on the catalyst surface. Afterward, the solutions were exposed to light to initiate LVX degradation, following the separation of samples from the working mixture to investigate the degradation process. The catalyst  $\text{In-CeO}_2/\text{g-CN}$  was recovered by centrifugation and reused for LVX degradation under the same conditions. The percentage degradation ( $D$ ) was calculated using the relation  $D(\%) = (1 - C_t/C_0) \times 100$ ,<sup>40</sup> where  $C_t$  represents the LVX concentration at the specified time interval, while  $C_0$  is its initial concentration.

## 3. Results and discussion

### 3.1 Structural features

The crystal structure formation and phase purity of the synthesized materials were verified *via* XRD patterns ( $2\theta =$

$10^\circ$ – $80^\circ$ ), which are shown in Fig. 2(a). The peaks present at  $2\theta = 28.56^\circ, 33.16^\circ, 47.45^\circ, 56.34^\circ, 59.14^\circ, 69.53^\circ, 76.92^\circ,$  and  $79.12^\circ$ , corresponding to the (111), (200), (220), (311), (222), (400), (331), and (420) diffraction planes, are consistent with the standard data (00-034-0394), confirming the formation of  $\text{CeO}_2$ .<sup>41</sup> The indium-modified material shows a variation in its diffraction peaks, which correspond to the structural changes caused by the different ionic sizes of dopant indium and the cerium host.<sup>42</sup> Notably, the absence of any additional diffraction peaks for pure  $\text{CeO}_2$  and its indium-modified composition  $\text{In-CeO}_2$  confirms the phase purity and insertion of indium ions in the  $\text{CeO}_2$  lattice. The lower-intensity diffraction peaks in the XRD pattern of  $\text{In-CeO}_2/\text{g-CN}$  correspond to the interaction between  $\text{In-CeO}_2$  and  $\text{g-CN}$ , causing microstructural changes.<sup>43,44</sup> The distinct lattice vibrations of the synthesized materials were confirmed from the FTIR spectra, as displayed in Fig. 2(c). The presence of distinctive Ce–O and Ce–O–Ce vibrations at  $460 \text{ cm}^{-1}$  and  $1052 \text{ cm}^{-1}$  confirms the formation of the synthesized materials.<sup>45</sup> The peaks at  $1640 \text{ cm}^{-1}, 1426 \text{ cm}^{-1}, 1324 \text{ cm}^{-1},$  and  $1252 \text{ cm}^{-1}$  in the spectra of  $\text{g-CN}$  and  $\text{In-CeO}_2/\text{g-CN}$  can be attributed to C–N heterocyclic and aromatic stretching vibrations, while the peak at  $810 \text{ cm}^{-1}$  corresponds to the bending vibration (s-triazine units) of the  $\text{g-CN}$  framework.<sup>46–48</sup> The additional –OH stretching and –OH bending observed indicate moisture adsorption on the material's surface.<sup>49,50</sup> The thermal TGA plots showcasing the mass loss of the material against the



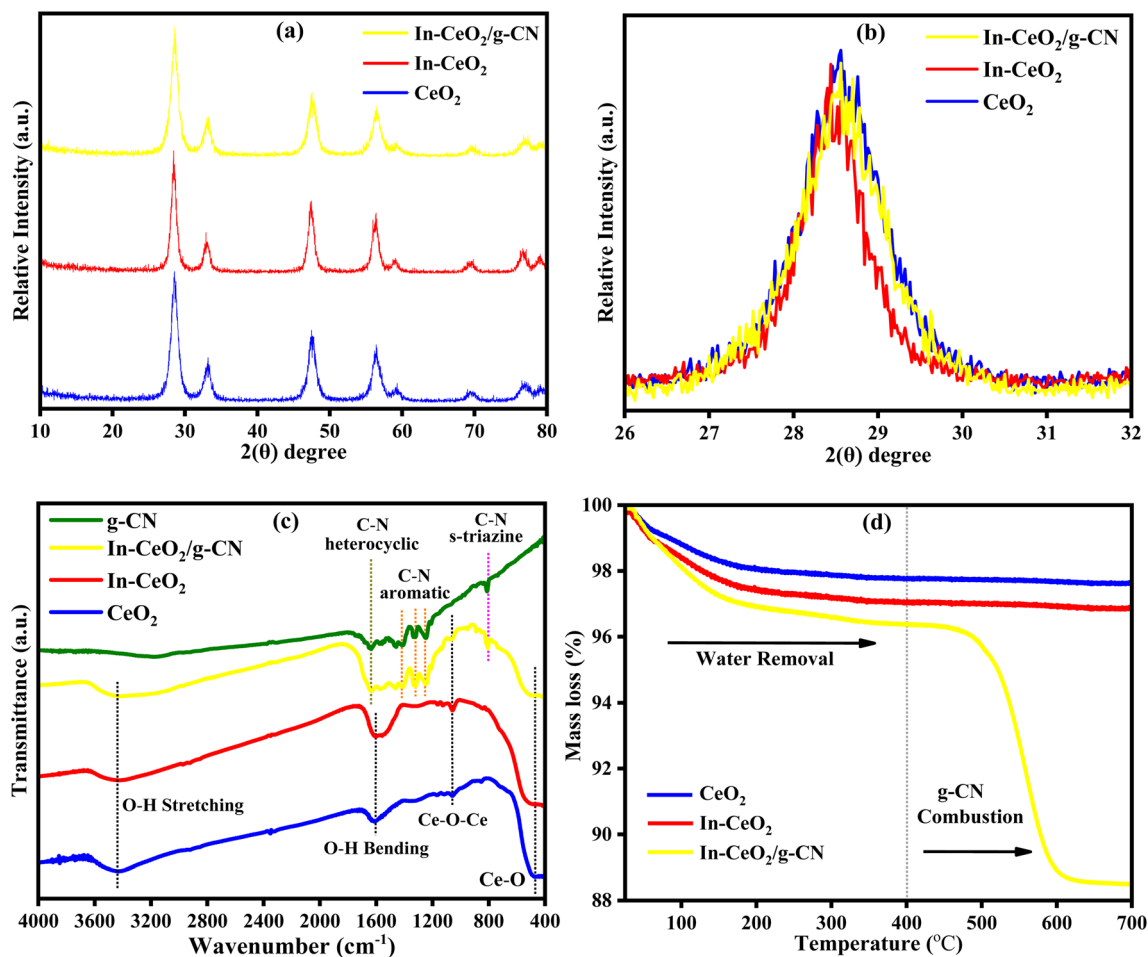


Fig. 2 (a) XRD patterns, (b) enlarged view of the diffraction patterns ( $2\theta = 26\text{--}32^\circ$ ), (c) FTIR spectra, and (d) TGA plots of the synthesized pure  $\text{CeO}_2$ , modified  $\text{In-CeO}_2$ , and the  $\text{In-CeO}_2/\text{g-CN}$  heterojunction.

applied temperature of  $\text{CeO}_2$ ,  $\text{In-CeO}_2$ , and  $\text{In-CeO}_2/\text{g-CN}$  are shown in Fig. 2(d). In the lower temperature zone ( $<300^\circ\text{C}$ ), minute weight losses of 2.29% ( $\text{CeO}_2$ ), 3.03% ( $\text{In-CeO}_2$ ), and 3.60% ( $\text{In-CeO}_2/\text{g-CN}$ ) were observed, which are due to moisture removal. For the g-CN-based heterojunction material, in the higher temperature zone ( $400\text{--}600^\circ\text{C}$ ), 7.93% of weight loss was witnessed, which corresponds to g-CN combustion.<sup>51</sup>

### 3.2 Morphological analysis

The microstructure and morphology of the synthesized  $\text{CeO}_2$ ,  $\text{In-CeO}_2$ , and  $\text{In-CeO}_2/\text{g-CN}$  materials were examined by SEM analysis, and the micrographs are presented in Fig. 3. For  $\text{CeO}_2$ , a fine granular-type aggregation was observed, while for  $\text{In-CeO}_2$ , the symmetry changes to irregular-sized crystallites with varying sizes. Indium modification not only changes the microstructure from granules to crystallites, but also from aggregation to dispersion of the material. The g-CN integration further increases dispersion, preventing agglomeration, as evident in Fig. 3(d), which is substantial for photocatalytic applications.

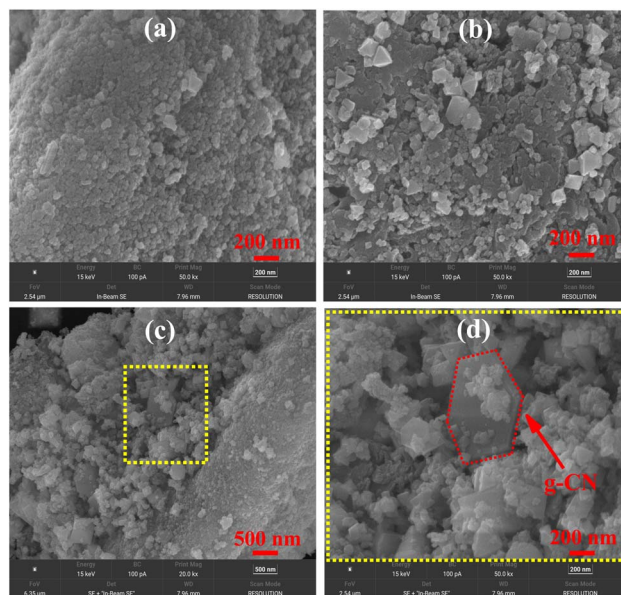


Fig. 3 SEM images of (a)  $\text{CeO}_2$ , (b)  $\text{In-CeO}_2$ , and (c and d)  $\text{In-CeO}_2/\text{g-CN}$ .



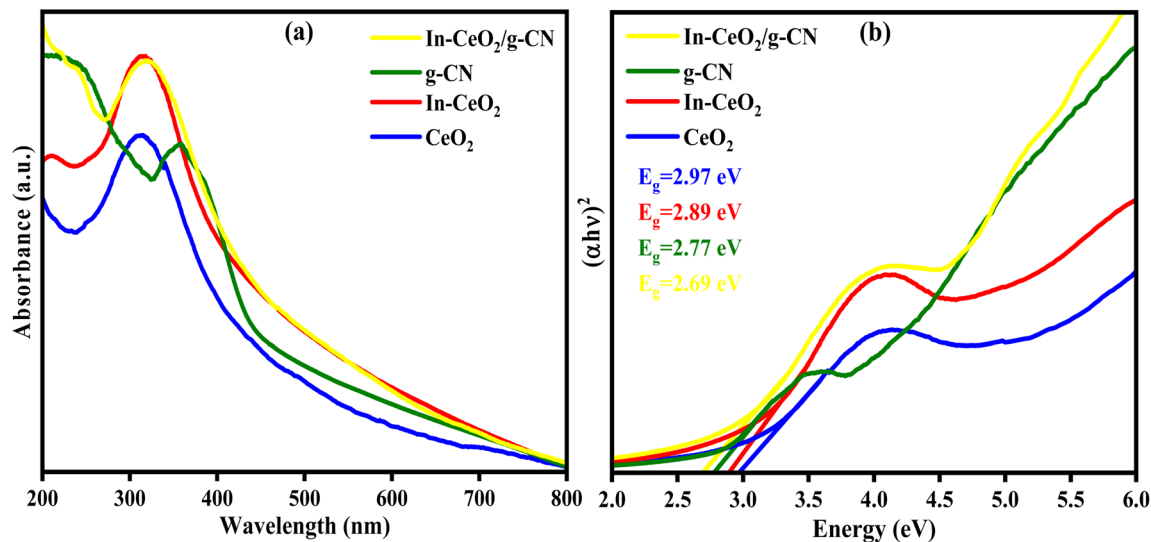


Fig. 4 (a) UV-visible absorption spectra and (b) bandgap calculation from the Tauc plot model of the synthesized CeO<sub>2</sub>, In-CeO<sub>2</sub>, g-CN, and In-CeO<sub>2</sub>/g-CN.

### 3.3 Optical properties

The optical properties of CeO<sub>2</sub>, In-CeO<sub>2</sub>, g-CN, and In-CeO<sub>2</sub>/g-CN materials were investigated by UV-vis absorption spectroscopy, and the absorption spectra observed for different materials are presented in Fig. 4(a). The synthesized materials feature absorption edges extending into the visible region. Notably, indium-modification and heterojunction formation with g-CN considerably enhances visible-light absorption, as evident from the red-shift in the absorption edge.<sup>52,53</sup> Fig. 4(b) presents the Tauc plots generated to calculate the optical bandgap ( $E_g$ ),<sup>54</sup> and the materials CeO<sub>2</sub>, In-CeO<sub>2</sub>, g-CN, and In-CeO<sub>2</sub>/g-CN feature an  $E_g$  of 2.97 eV, 2.89 eV, 2.77 eV, and 2.69 eV, respectively. The In-CeO<sub>2</sub>/g-CN heterojunction ( $E_g = 2.69$  eV) exhibits increased absorption, facilitating the separation of electron-hole pairs under visible light and rendering it promising for photocatalytic applications.

### 3.4 Electrochemical response

The electrochemical responses of CeO<sub>2</sub>, In-CeO<sub>2</sub>, g-CN, and In-CeO<sub>2</sub>/g-CN materials were investigated to elucidate charge transfer kinetics and electron-hole recombination rates. The EIS results, presented as Nyquist plots in Fig. 5(a), reveal a distinct electrochemical response. The semicircle diameter in the Nyquist plot reflects the charge transfer resistance ( $R_{ct}$ ) at the working electrode-electrolyte interface.<sup>55</sup> Notably, the In-CeO<sub>2</sub>/g-CN heterojunction exhibits a smaller semicircle diameter compared to its components, g-CN and In-CeO<sub>2</sub>, indicating lower  $R_{ct}$  and faster charge transfer kinetics.<sup>56-58</sup> The light-on photocurrent generation for the materials CeO<sub>2</sub>, In-CeO<sub>2</sub>, g-CN, and In-CeO<sub>2</sub>/g-CN is presented in Fig. 5(b). The significantly increased photocurrent density observed for In-CeO<sub>2</sub>/g-CN can be attributed to enhanced charge separation due to band structure alignment, which suppresses rapid electron-

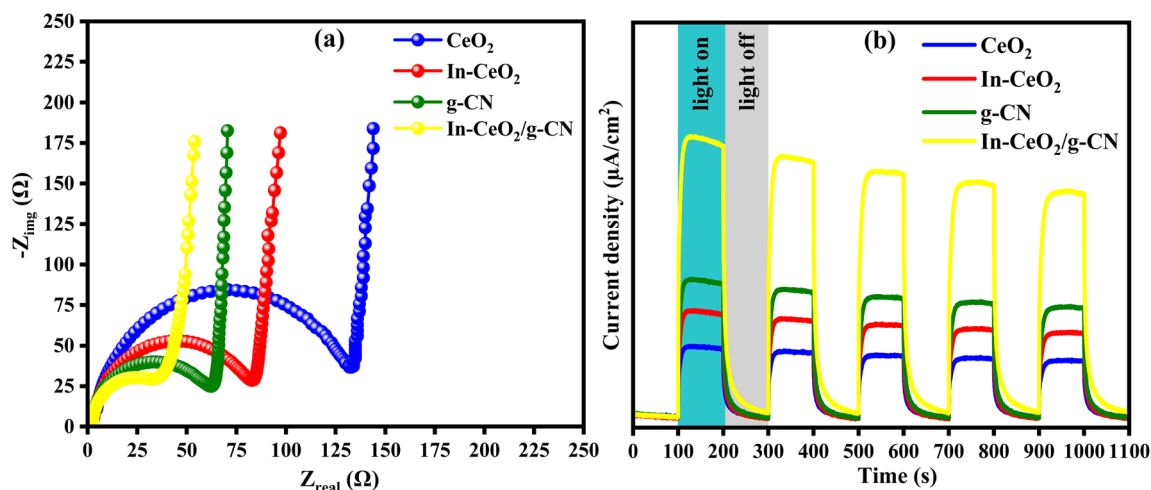


Fig. 5 (a) Nyquist plots and (b) photocurrent responses of the CeO<sub>2</sub>, In-CeO<sub>2</sub>, g-CN, and In-CeO<sub>2</sub>/g-CN materials.



hole recombination.<sup>59,60</sup> The heterojunction formation enhances charge transport, thereby increasing the availability of electron-hole pairs or their participation in the photocatalytic process.

### 3.5 PC activity

The PC activity of CeO<sub>2</sub>, In-CeO<sub>2</sub>, and In-CeO<sub>2</sub>/g-CN materials was estimated by degrading LVX. The UV-vis absorption data of the LVX samples separated from the aliquot at specified time intervals (in the presence of In-CeO<sub>2</sub>/g-CN) are presented in Fig. 6(a). The absorption intensity at  $\lambda_{\max}$  corresponds to the LVX concentration, which decreases, indicating LVX degradation. The absorption intensity rapidly declines in the presence of the photocatalyst In-CeO<sub>2</sub>/g-CN as compared to In-CeO<sub>2</sub> and CeO<sub>2</sub>, which degrade LVX to a lower extent. In Fig. 6(b), the LVX degradation (%) over the synthesized catalysts is presented, which follows the order, CeO<sub>2</sub> (44%) < In-CeO<sub>2</sub> (63%) < In-

CeO<sub>2</sub>/g-CN (85%). The order of photocatalytic efficiency is consistent with the optical properties and electrochemical responses of the photocatalysts, which feature facilitated electron-hole separation and transport in In-CeO<sub>2</sub>/g-CN, revealing its promising potential for photocatalytic applications.

The kinetics study of LVX degradation over the photocatalysts, CeO<sub>2</sub>, In-CeO<sub>2</sub>, and In-CeO<sub>2</sub>/g-CN, is shown in Fig. 7(a), which presents 1st-order kinetics for LVX degradation. The rate constants were calculated as 0.0075 min<sup>-1</sup>, 0.0123 min<sup>-1</sup>, and 0.0198 min<sup>-1</sup>, corresponding to CeO<sub>2</sub>, In-CeO<sub>2</sub>, and In-CeO<sub>2</sub>/g-CN. The scavenging study presented in Fig. 7(b) for LVX degradation in the presence of a photocatalyst with the highest photocatalytic efficacy, In-CeO<sub>2</sub>/g-CN, was performed using EDTA, AgNO<sub>3</sub>, IPA, and BQ for trapping holes (h<sup>+</sup>), electrons (e<sup>-</sup>), hydroxyl radicals (HO<sup>•</sup>), and superoxide radicals (<sup>•</sup>O<sub>2</sub><sup>-</sup>), respectively. In the presence of AgNO<sub>3</sub>, the decline in LVX degradation was not significant, but in the

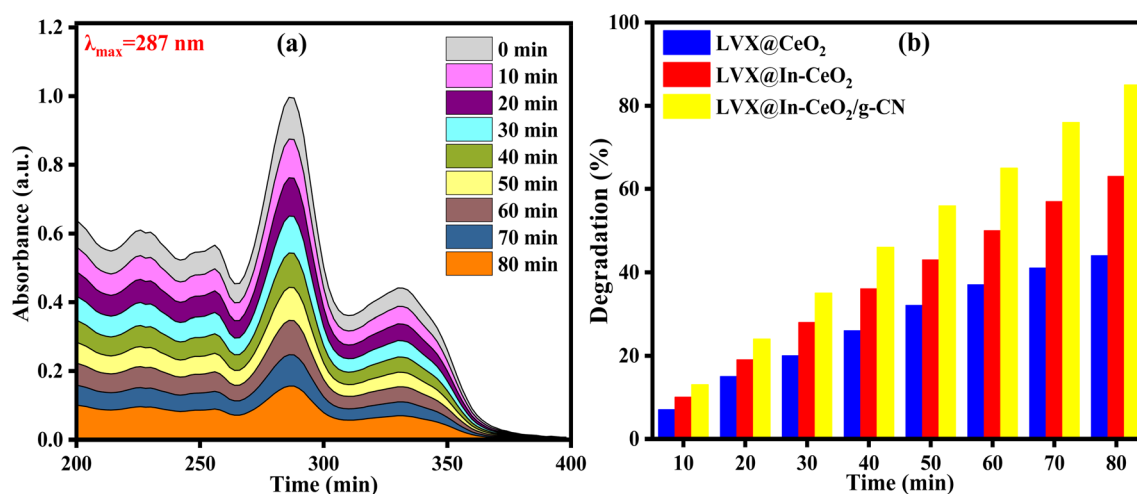


Fig. 6 (a) UV-vis absorption spectra of the samples collected during LVX degradation in the presence of In-CeO<sub>2</sub>/g-CN and (b) comparison of the catalytic efficiency.

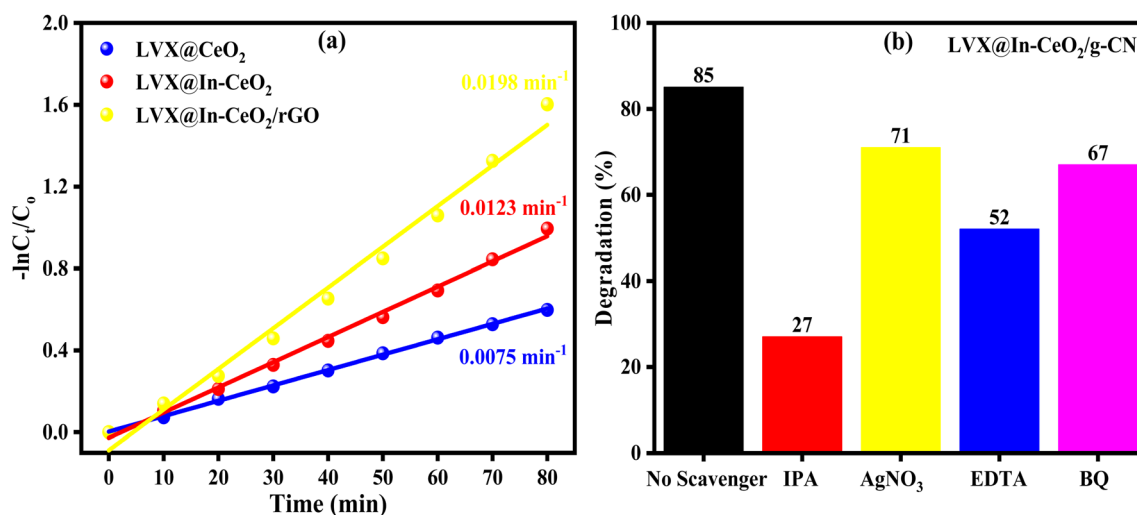


Fig. 7 (a) Kinetic studies of LVX degradation over CeO<sub>2</sub>, In-CeO<sub>2</sub>, and In-CeO<sub>2</sub>/g-CN, and (b) scavenging studies over In-CeO<sub>2</sub>/g-CN.



Table 1 Detailed comparison of the present work with previous studies

Sr. no.	Photocatalyst	Pollutant	Conditions	D(%)	References
1	Ag-doped g-CN/Biochar	Ciprofloxacin (CIP)	50 ppm CIP, 50 mg catalyst, visible-light, 4 h	70%	61
2	ZnO/g-CN	Methyl orange (MO)	10 ppm MO, 50 mg catalyst, 400 W lamp	83.71%	62
3	Ni-doped $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> /g-CN	Ciprofloxacin (CIP)	10 mg per L CIP, 15 mg catalyst, solar light $\sim 660 \text{ Wm}^{-2}$	82.1%	63
4	g-CN/Bi <sub>2</sub> O <sub>2</sub> CO <sub>3</sub>	Carbamazepine (CBZ)	20 ppm CBZ, 1 g of catalyst, sunlight, 180 minutes	98%	64
5	ZnCr <sub>2</sub> O <sub>4</sub> /g-CN	Ciprofloxacin (CIP)	10 mg per L CIP, 75 mg of catalyst, Halogen lamp, 120 minutes	74.36%	65
6	Bi <sub>2</sub> MoO <sub>6</sub> /g-CN	Ciprofloxacin (CFX)	10 mg per L CFX, 0.05 g of catalyst, LED light, 90 minutes	89.04%	66
7	In-CeO <sub>2</sub> /g-CN	Levofloxacin (LVX)	10 mg per L LVX, 50 mg catalyst, Xe lamp (300 W, UV cutoff filter $\lambda > 420 \text{ nm}$ ), and 80 minutes	85%	Present study

presence of IPA, the degradation dropped considerably. The scavenging investigation presents the contribution of active species in the order  $e^- < h^+ < \cdot\text{O}_2^- < \text{HO}\cdot$  for LVX degradation. A detailed comparison of the present work with previous findings is presented in Table 1.

Electronic band structure alignment and energy band energies hold vital significance in designing heterojunction materials with a specific scheme for charge migration. To get insights into the mechanism of LVX degradation over In-CeO<sub>2</sub>/g-CN, the energy band values, VB maxima ( $E_{\text{VB}}$ ) and CB minima

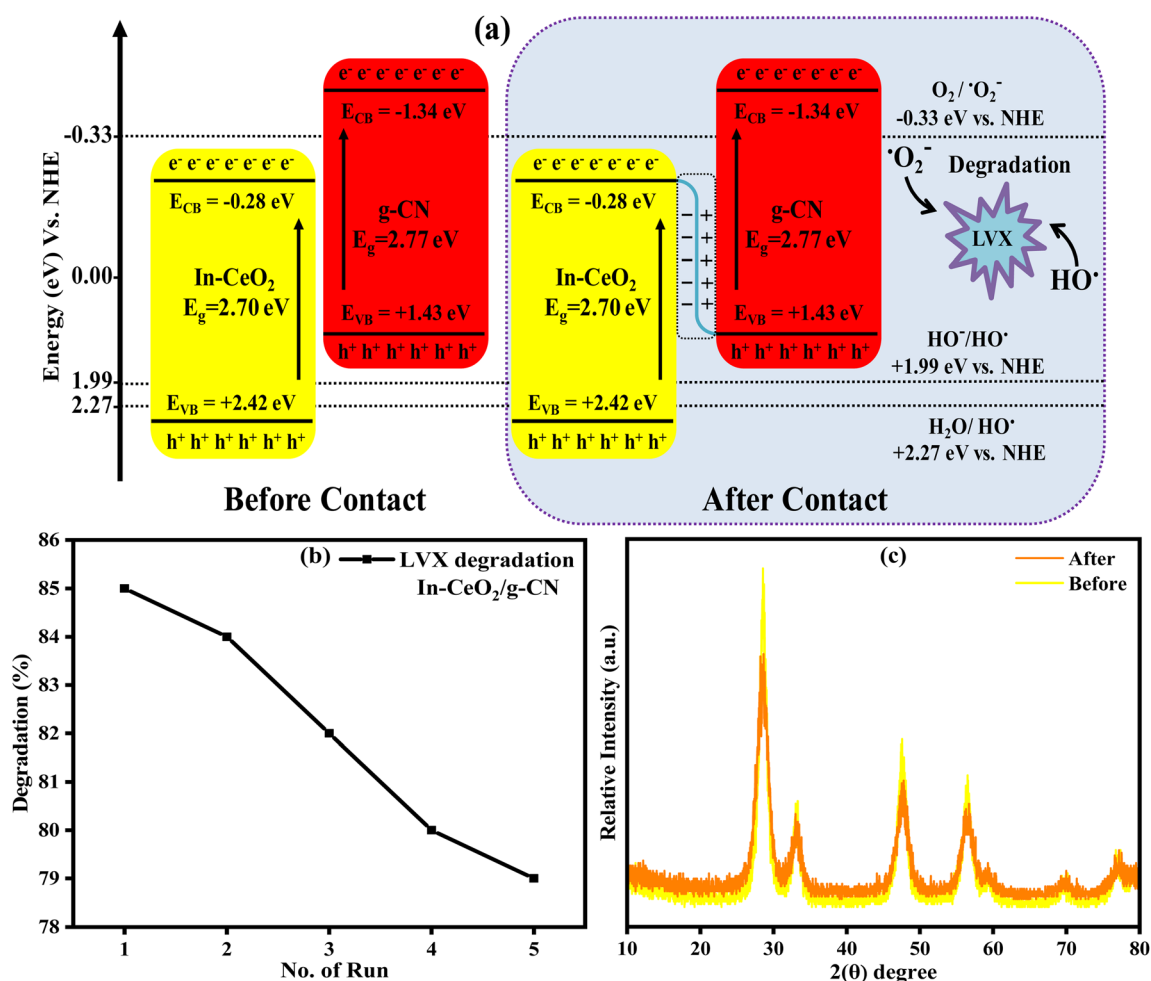


Fig. 8 (a) Schematic of the proposed S-scheme In-CeO<sub>2</sub>/g-CN heterojunction and (b) reusability and (c) before-and-after XRD pattern alignments to study the stability of the catalyst.



( $E_{CB}$ ) of g-CN and In-CeO<sub>2</sub> were explored by applying the relations:<sup>67,68</sup>

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

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

Here, “ $\chi$ ” is electronegativity, and  $E_e = 4.5$  eV denotes the energy of a free electron on the hydrogen scale. The calculated energy band values were  $E_{VB} = +2.42$  eV for In-CeO<sub>2</sub> and  $E_{CB} = -0.28$  eV, while  $E_{VB} = +1.43$  eV and  $E_{CB} = -1.34$  eV for g-CN. Thermodynamically, pure In-CeO<sub>2</sub> is unable to generate  $\cdot O_2^-$  radicals due to  $E_{CB}$  being higher than the required reduction potential ( $O_2/\cdot O_2^- = -0.33$  eV vs. NHE,<sup>69</sup>). Pure g-CN is unable to generate HO $\cdot$  radicals as it has an  $E_{VB}$  value lower than the required oxidation potential ( $HO^-/HO\cdot = 1.99$  eV and  $H_2O/HO\cdot = 2.23$  eV vs. NHE,<sup>70</sup>). But the scavenging studies reveal the generation of both  $\cdot O_2^-$  and HO $\cdot$  radicals, which degrade LVX. As shown in (Fig. 8), the low-redox-potential carriers,  $e^-$  of In-CeO<sub>2</sub> and  $h^+$  of g-CN, recombine, leaving behind the active  $e^-$  (g-CN) and  $h^+$  (In-CeO<sub>2</sub>),<sup>71,72</sup> which participate in the reduction and oxidation reactions producing active radical species that degrade LVX.

The reusability and structural stability of In-CeO<sub>2</sub>/g-CN were studied by LVX degradation in different runs, and the results are presented in Fig. 8(b and c). The photocatalyst retains ~95% of its initial activity after multiple cycles, indicating good structural stability and recyclability; minor losses are likely due to insufficient catalyst recovery. The diffraction pattern of the recovered catalyst displays its structural stability, as no significant change was detected.

## 4. Conclusion

Pure CeO<sub>2</sub> and its indium-modified composition, In-CeO<sub>2</sub>, were hydrothermally fabricated, while g-CN was synthesized through melamine calcination. The In-CeO<sub>2</sub> material was dispersed on g-CN through ultrasonication to construct the S-scheme In-CeO<sub>2</sub>/g-CN heterojunction. XRD and FTIR analyses verified the formation of CeO<sub>2</sub>, In-CeO<sub>2</sub>, and In-CeO<sub>2</sub>/g-CN, with thermal stability indicated by TGA. SEM micrographs showcased the dispersion of In-CeO<sub>2</sub>, with g-CN preventing agglomeration. The In-CeO<sub>2</sub>/g-CN heterojunction increased visible-light absorption under  $\lambda > 420$  nm illumination and facilitated charge separation *via* band structure alignment, forming an S-scheme of charge migration. The PC efficiency was evaluated by LVX degradation in the presence of catalysts, CeO<sub>2</sub>, In-CeO<sub>2</sub>, and In-CeO<sub>2</sub>/g-CN. The In-CeO<sub>2</sub>/g-CN catalyst exhibited a maximum LVX degradation of 85% ( $0.0198 \text{ min}^{-1}$ ) compared to CeO<sub>2</sub> and In-CeO<sub>2</sub>, which degraded LVX to an extent of 44% ( $0.0075 \text{ min}^{-1}$ ) and 63% ( $0.0123 \text{ min}^{-1}$ ), respectively. The formation of the In-CeO<sub>2</sub>/g-CN S-scheme heterojunction enables the recombination of low-redox-potential charge carriers (with low redox ability), preventing active charge carriers (high redox ability) from recombining and making them available for photocatalytic reactions. Despite the improved activity, the study is limited to a single pollutant and

Xe-lamp illumination conditions; future work will evaluate performance under solar-simulated visible light and conduct advanced interfacial analyses such as XPS.

## Author contributions

Mazen R. Alrahili (writing – original draft, validation), M. Abdel Rafea (funding acquisition, project administration, resources), Magdi E. A. Zaki (investigation, formal analysis), M. Khairy (data curation, validation), Mohamed. R. El-Aassar (investigation, data curation), Sultan Albarakati (software, methodology), Imran Shakir (visualization, formal analysis), Abdullah K. Alanazi (writing – review & editing), Muhammad Aadil (supervision).

## Conflicts of interest

There are no conflicts to declare.

## Data availability

Data will be available upon request.

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