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# Water-hyacinth-leaf-like 2D bismuth oxychloride photocatalyst for the degradation of methylene blue and crystal violet dyes

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In this research, we synthesized a water-hyacinth (*Eichhornia crassipes*)-leaf-like 2D bismuth oxychloride ( $\text{Bi}_3\text{ClO}_4$ ) photocatalyst using a simple co-precipitation method. The as-synthesized  $\text{Bi}_3\text{ClO}_4$  is characterized by XRD, Raman spectroscopy, UV-vis spectroscopy, SEM, EDAX and BET analysis. The XRD results showed that the material has a monoclinic crystal structure. Raman spectroscopy confirmed the existence of the vibrational bands of bismuth oxychloride. The BET surface area measurement indicated that the material has a porous structure with a surface area of  $13.68 \text{ m}^2 \text{ g}^{-1}$ . The SEM analysis revealed that the material has a well-defined nanostructured sheet-like morphology. The EDAX study showed that the synthesized material has a well-defined stoichiometry. The as-synthesized bismuth oxychloride photocatalyst exhibited enhanced photocatalytic activity under visible-light irradiation. Complete degradation of crystal violet (100%) was achieved within 75 min, while methylene blue showed 96% degradation within 105 min. These findings highlight the strong potential of bismuth oxychloride as an efficient photocatalyst for wastewater remediation.

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## 1 Introduction

Rapid industrialization and urban expansion have led to the extensive discharge of synthetic dyes into natural water bodies, posing severe environmental and health challenges. Industries such as textile, leather, paper, plastic, and pharmaceuticals are significant contributors to dye-laden wastewater, releasing substantial quantities of colored effluents annually.<sup>1–4</sup> These dyes are often chemically stable, non-biodegradable, and resistant to conventional wastewater treatment processes, enabling them to persist in aquatic ecosystems for long durations.<sup>5–8</sup> The presence of dyes in water bodies not only imparts intense coloration, which reduces light penetration and disrupts photosynthetic activities, but also introduces toxic, mutagenic, and carcinogenic compounds into the environment. Many azo and anthraquinone dyes decompose into aromatic amines, which are harmful to aquatic organisms and may bioaccumulate through the food chain, posing risks to human

health.<sup>9–12</sup> Therefore, the development of efficient and sustainable dye removal technologies has become a critical area of environmental research.

Among various remediation strategies such as adsorption, coagulation, membrane filtration, and biological treatment, advanced oxidation processes (AOPs) have gained significant attention due to their ability to completely mineralize organic pollutants into harmless end-products like  $\text{CO}_2$  and  $\text{H}_2\text{O}$ .<sup>13–16</sup> Photocatalysis, a key AOP, utilizes semiconductor materials to generate highly reactive oxygen species (ROS) under light irradiation, enabling the effective degradation of complex dye molecules.<sup>17–19</sup> Nanomaterials have gained significant attention as promising photocatalysts owing to their high surface-to-volume ratios, tunable electronic structures, and enhanced charge carrier dynamics.<sup>20</sup> In particular, bismuth-based oxyhalides and oxychlorides have attracted increasing interest due to their layered crystal structures, suitable band gaps, and strong internal electric fields that facilitate the efficient separation of photogenerated electron–hole pairs.<sup>21–25</sup>

Recently, bismuth oxychloride derivatives, such as  $\text{Bi}_3\text{ClO}_4$ , have been explored as advanced photocatalysts for dye degradation applications.  $\text{Bi}_3\text{ClO}_4$  exhibits a unique crystal structure that combines favourable light absorption and improved redox capability, enabling the effective generation of reactive oxygen species under visible light irradiation.<sup>26–28</sup> Compared with conventional photocatalysts such as  $\text{TiO}_2$ ,  $\text{BiOCl}$ ,  $\text{Bi}_2\text{O}_3\text{-TiO}_2$ ,  $\text{Bi}_2\text{O}_3/\text{g-C}_3\text{N}_4$ , and  $\text{Zn}_3(\text{VO}_4)_2$ ,  $\text{BiVO}_4$ -derived heterostructures and nanocomposite photocatalysts have exhibited efficient

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Table 1 Comparison of the photocatalytic performance of the as-synthesized Bi<sub>3</sub>ClO<sub>4</sub> nanomaterial with reported photocatalysts

S. no.	Catalyst	Organic pollutant	Light source	Degradation time (min)	Degradation efficiency (%)	Rate constant (min <sup>-1</sup> )	Ref.
1	BiOCl	MB	UV-vis	90	100	0.038	29
2	Bi <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	MB	UV-vis	90	100	0.038	30
3	Bi <sub>2</sub> O <sub>3</sub> /g-C <sub>3</sub> N <sub>4</sub>	MB	Visible	100	98	—	31
4	Zn <sub>3</sub> (VO <sub>4</sub> ) <sub>2</sub>	MB	Visible	120	87	0.010–0.017	32
5	ZnO	MB	Visible	180–300	97–100	—	33
6	BiIO <sub>4</sub> /BiVO <sub>4</sub>	RhB	Visible	60	98	0.05	34
7	BiVO <sub>4</sub> /TiS <sub>2</sub>	RhB	Visible	70	95	—	35
8	ZnO	CV	UV	80	90	—	36
9	ZnO and C/ZnO	CV	UV	60	92	—	37
10	ZnO	CV	UV	30	98	—	38
11	BiVO <sub>4</sub> -NiFe <sub>2</sub> O <sub>4</sub>	CV	UV	60	95	—	39
12	Ag <sub>2</sub> S/NiO-ZnO	RhB	Visible light	120	95	0.0302	40
13	In <sub>2</sub> O <sub>3</sub> /CeO <sub>2</sub>	Congo red (CR), methylene blue (MB)	Visible light	91 (CR) and 130 (MB)	95 (CR) and 94 (MB)	—	41
14	Ag <sub>2</sub> O/ZnO-TiO <sub>2</sub>	RhB	Visible light	100	95	0.0129	42
15	Ag <sub>2</sub> CO <sub>3</sub> -BiVO <sub>4</sub>	Tetracycline	Visible light	80	94	0.0278	43
16	LaFe <sub>2</sub> O <sub>3</sub> /Sb <sub>2</sub> O <sub>3</sub>	Malachite green (MG)	Visible light	88	98 (RhB)	0.003399	44
17	10 mg of Bi <sub>3</sub> ClO <sub>4</sub>	Methylene blue Crystal violet	Visible Visible	105 75	96 100	—	This work This work

degradation of model organic pollutants including methylene blue (MB), rhodamine B (RhB), and crystal violet (CV) under UV or visible irradiation (Table 1).<sup>29–44</sup> Despite achieving high degradation efficiencies ( $\approx 90$ – $100\%$ ), many reported catalysts depend on UV irradiation or longer reaction durations and are associated with the formation of potentially toxic by-products. In this context, the development of simple, visible-light-responsive bismuth-based photocatalysts with rapid degradation kinetics remains an important research objective. Motivated by these considerations, the present work explores Bi<sub>3</sub>ClO<sub>4</sub> as an efficient photocatalyst for the degradation of MB and CV under visible light. The obtained results demonstrate competitive performance compared with previously reported systems, indicating that Bi<sub>3</sub>ClO<sub>4</sub> shows superior and promising visible light responsiveness, making it suitable for solar-driven wastewater treatment processes.<sup>45</sup> Furthermore, the photocatalytic performance of Bi<sub>3</sub>ClO<sub>4</sub> can be significantly enhanced through nanostructuring, defect engineering, and heterojunction formation, which improve light harvesting and suppress charge recombination.<sup>46,47</sup> These attributes make Bi<sub>3</sub>ClO<sub>4</sub>-based nanomaterials promising candidates for next-generation photocatalytic systems aimed at sustainable water purification.<sup>48,49</sup>

In this context, the present research focuses on the synthesis, characterization, and application of Bi<sub>3</sub>ClO<sub>4</sub> nanomaterials for efficient dye degradation. Understanding the structure–property–performance relationship of such nanocatalysts is essential for optimizing their photocatalytic activity and advancing their practical implementation in environmental remediation technologies.

## 2 Materials and methods

### 2.1 Chemicals

All chemicals used were of analytical reagent (AR) grade, including bismuth nitrate (Bi(NO<sub>3</sub>)<sub>3</sub>; Sigma-Aldrich, 98%

purity), potassium chloride (KCl; Sigma-Aldrich), and formaldehyde (HCHO). Potassium hydroxide (KOH) was used to adjust the pH. The model dyes, crystal violet (CV) and methylene blue (MB), were obtained from E. Merck, India, and used without further purification. Double-distilled water (DDW) was used throughout the experiments.

### 2.2 Synthesis of Bi<sub>3</sub>ClO<sub>4</sub> nanoparticles

Bismuth oxychloride (Bi<sub>3</sub>ClO<sub>4</sub>) was synthesized using a co-precipitation method. Double-distilled water (DDW) was used as the solvent in synthesis and electrochemical studies. In a typical procedure, 0.485 g of bismuth nitrate (Bi(NO<sub>3</sub>)<sub>3</sub>) was dissolved in 10 mL of formaldehyde and stirred vigorously for 30 minutes. After this, 4 M KOH and 2 M KCl were added separately to the solution. The mixture was then stirred for another 30 minutes, during which a white suspension formed, indicating product formation. The resulting precipitate was washed several times with DDW and ethanol to remove impurities and then dried at 100 °C for 6 hours.

## 3 Characterization

### 3.1 Materials characterization

Cu K $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ) was used to obtain X-ray diffraction (XRD) patterns on a Rigaku D/Max-TTR III X-ray diffractometer in the  $2\theta$  range from 5° to 90°. This method was used to ascertain the synthesised material's phase composition and crystalline structure. A Renishaw Raman spectrometer was used to perform Raman spectroscopy measurements, which allowed for the identification of the sample's vibrational modes and chemical bonding information. Field-emission scanning electron microscopy (FE-SEM) on a JEOL JSM-6701F instrument was used to characterise the material's surface morphology. This method provided high-resolution images to



study the particle size, shape, and surface characteristics of materials. The specific surface area and pore properties of the material were ascertained by measuring adsorption and desorption isotherms using a BET flowing gas surface area analyser.

### 3.2 Photocatalytic activity

The photocatalytic activity of  $\text{Bi}_3\text{ClO}_4$  nanoparticles for the degradation of crystal violet (CV) and methylene blue (MB) dyes was evaluated under natural sunlight. This was accomplished by dispersing 10 mg of  $\text{Bi}_3\text{ClO}_4$  nanoparticles in 100 mL of a 10 ppm CV or the MB dye solution. The suspension was magnetically stirred for 60 min in the dark to reach the adsorption equilibrium. To avoid localized overheating, the reaction vessel was kept in a water bath under open-atmospheric conditions with continuous magnetic stirring. Hence, the solution temperature was kept in the range from 27 °C to 30 °C during irradiation. Dark control experiments confirmed negligible dye degradation, indicating that the removal process was predominantly photocatalytic rather than thermally driven. Every 15 minutes, 10 mL of this solution was collected. The solution was centrifuged to extract the  $\text{Bi}_3\text{ClO}_4$  nanoparticles, and the concentration of CV or the MB dye was detected using a UV-visible spectrometer.

## 4 Results and discussion

### 4.1 Physicochemical characterizations

**4.1.1 X-ray diffraction (XRD) of the as-synthesized photocatalyst.** The recorded diffraction pattern agrees well with the standard reference data (PDF no. 00-036-0760), confirming the successful formation of monoclinic  $\text{Bi}_3\text{ClO}_4$ .<sup>50</sup> The XRD pattern exhibits sharp, well-defined peaks at  $2\theta$  values of approximately 9.49°, 19.08°, 21.80°, 22.18°, 23.98°, 28.77°, 29.13°, 31.40°, 31.64°, 37.97°, 38.77°, 43.36°, 45.25°, 46.56°, 50.10°, 52.07°, and 54.85°, indicating the good crystallinity of the prepared sample.

These diffraction peaks can be indexed to the (200), (400), (310), (011), (211), (600), (411), (002), (020), ( $3\bar{1}2$ ), (800), (620), (022), (222), (721), (213), and (431) crystal planes of monoclinic  $\text{Bi}_3\text{ClO}_4$ , respectively, which further validates the phase purity of the synthesized material. No additional impurity peaks were detected, suggesting the formation of single-phase  $\text{Bi}_3\text{ClO}_4$ , as illustrated in Fig. 1a. The relatively narrow peak widths indicate the formation of nanosized crystallites; the average crystallite size can be estimated using the Debye–Scherrer equation, which typically falls in the nanometer range for such systems, supporting the nanoscale nature of the material. The high crystallinity and phase purity revealed by XRD are expected to enhance the separation and transport of photogenerated charge carriers, thereby contributing to improved photocatalytic performance under visible-light irradiation.

The average crystallite size of  $\text{Bi}_3\text{ClO}_4$  was estimated using the Scherrer equation:<sup>51</sup>

$$D = K\lambda/\beta \cos \theta \quad (1)$$

where  $D$  represents the crystallite size,  $K$  is the shape factor (0.94),  $\beta$  is the full width at half-maximum (FWHM) of the diffraction peak,  $\theta$  is the Bragg angle, and  $\lambda$  is the wavelength of the X-ray source (0.15406 nm). Based on this calculation, the average crystallite size of the synthesized  $\text{Bi}_3\text{ClO}_4$  is found to be approximately 5.6 nm. The crystallite size calculated using the Scherrer equation from the (200) diffraction peak at  $2\theta \approx 9.5^\circ$  was found to be of approximately 5.6 nm. This value represents the size of coherent diffraction domains rather than the actual particle dimensions. The larger nanosheet-like structures observed in SEM images (Fig. 4a (50–100 nm)) are likely formed *via* the aggregation of several smaller crystallites during the growth process.

**4.1.2 Raman spectroscopic analysis.** Raman spectroscopy was employed to further examine the structural order and vibrational characteristics of the synthesized  $\text{Bi}_3\text{ClO}_4$

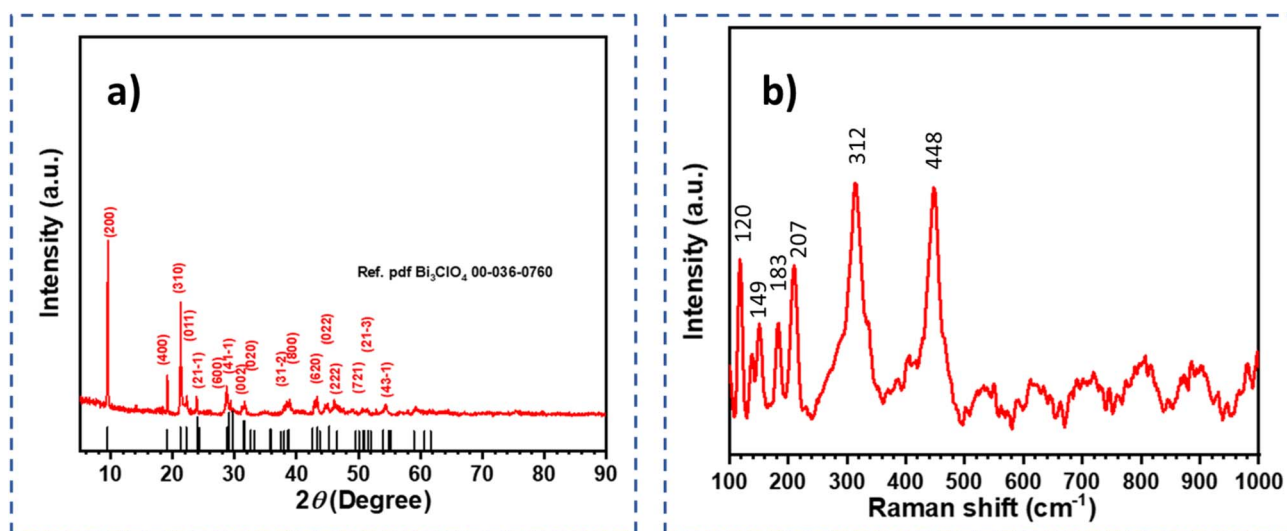


Fig. 1 (a) X-ray diffraction pattern of  $\text{Bi}_3\text{ClO}_4$ . (b) Raman spectrum of  $\text{Bi}_3\text{ClO}_4$ .



nanoparticles, and the corresponding spectrum is presented in Fig. 1b. Several distinct Raman bands are observed at approximately 120, 149, 183, 207, 312, and 448  $\text{cm}^{-1}$ , which are consistent with previously reported vibrational modes of monoclinic  $\text{Bi}_3\text{ClO}_4$ .<sup>52–54</sup> The low-frequency bands in the range of 100–220  $\text{cm}^{-1}$  are mainly associated with lattice vibrations and external Bi–Cl stretching modes with the  $A_{1g}$  symmetry, arising from the motion of  $\text{Bi}^{3+}$  ions against the surrounding  $\text{Cl}^-$  framework. The bands located at  $\sim 183$  and 207  $\text{cm}^{-1}$  can be attributed to internal Bi–Cl stretching and bending vibrations corresponding to the  $A_{1g}$  and  $E_g$  modes of the distorted Bi–Cl polyhedron, respectively. The higher-frequency bands at  $\sim 312$  and 448  $\text{cm}^{-1}$  are assigned to the symmetric and asymmetric stretching vibrations of Bi–Cl bonds within the  $\text{Bi}_3\text{ClO}_4$  lattice, respectively, indicating the presence of well-defined Bi–Cl coordination environments. The relatively sharp and well-resolved Raman peaks suggest the good crystallinity and structural ordering of the synthesized nanoparticles, which is in agreement with the XRD results. Moreover, the absence of additional Raman bands corresponding to possible impurity phases, such as  $\text{Bi}_2\text{O}_3$  or  $\text{BiOCl}$ , further confirms the phase purity of the prepared sample. The slight peak broadening observed in the spectrum may be attributed to the nanoscale crystallite size and lattice strain effects commonly present in nanostructured materials. Such structural features can influence phonon confinement and defect density, which in turn affect charge carrier dynamics during photocatalysis. Therefore, the Raman results, together with XRD analysis, confirm the successful formation of crystalline and phase-pure  $\text{Bi}_3\text{ClO}_4$  with a well-defined monoclinic structure, which is expected to be beneficial for enhanced photocatalytic activity under visible-light irradiation.

**4.1.3 UV-vis absorption analysis of  $\text{Bi}_3\text{ClO}_4$ .** Ultraviolet (UV) spectroscopy is a widely used analytical technique for investigating the optical properties and electronic structure of materials. It is based on the absorption of ultraviolet or visible radiation by a substance, resulting in electronic transitions

between molecular orbitals. The UV-vis absorption spectrum of  $\text{Bi}_3\text{ClO}_4$  (Fig. 2a) exhibits a strong optical absorption in the 420–800 nm wavelength range, indicating that  $\text{Bi}_3\text{ClO}_4$  can efficiently absorb visible light and acts as a solar-light-driven active photocatalyst for dye degradation. The valence band (VB) and conduction band (CB) of  $\text{Bi}_3\text{ClO}_4$  are poised by hybridized Bi 6s/O 2p orbitals and Cl 3d orbitals, respectively. The band gap and absorption coefficient, according to the Kubelka–Munk equation, can be expressed as  $\alpha h\nu = A(h\nu - E_g)^{1/2}$ , where  $\alpha$ ,  $\nu$  and  $h$  represent the absorption coefficient, the frequency of light and Planck's constant, respectively. The Tauc plot (Fig. 2b), constructed by plotting  $(\alpha h\nu)^2$  versus photon energy ( $h\nu$ ), was used to determine the band gap energy. The intercept of the tangent at the x-axis is a good estimate of the band gap  $E_g$ . For the prepared  $\text{Bi}_3\text{ClO}_4$  nanoparticles, a band gap of 2.67 eV is observed. The  $E_g$  value decreases as the crystallite size increases. The band gap of a material is affected by the size of the particles and its crystal structure.

#### 4.1.4 Brunauer–Emmett–Teller (BET) surface area analysis.

It is widely recognized that a higher specific surface area in electrode materials leads to an increased number of accessible active sites, which in turn facilitates charge transfer processes. The textural properties of  $\text{Bi}_3\text{ClO}_4$  were evaluated through nitrogen adsorption–desorption measurements. As shown in Fig. 3a, the adsorption isotherms exhibit a type-III profile accompanied by an H3 hysteresis loop in the relative pressure ( $P/P_0$ ) range of 0.45–1.0, indicating the presence of microporous features.<sup>55</sup>

The pore size distribution curve, derived from the Barrett–Joyner–Halenda (BJH) model (Fig. 3b), shows that the material possesses a broad pore size distribution in the mesoporous region, confirming the formation of a hierarchical porous structure. The Brunauer–Emmett–Teller (BET) specific surface area of  $\text{Bi}_3\text{ClO}_4$  is calculated to be 13.68  $\text{m}^2 \text{g}^{-1}$ . This surface area is moderate due to the stacking of nanosheets, which reduces the accessible surface area but still exposes active edge sites. Several studies on  $\text{BiOCl}$  nanosheets have reported

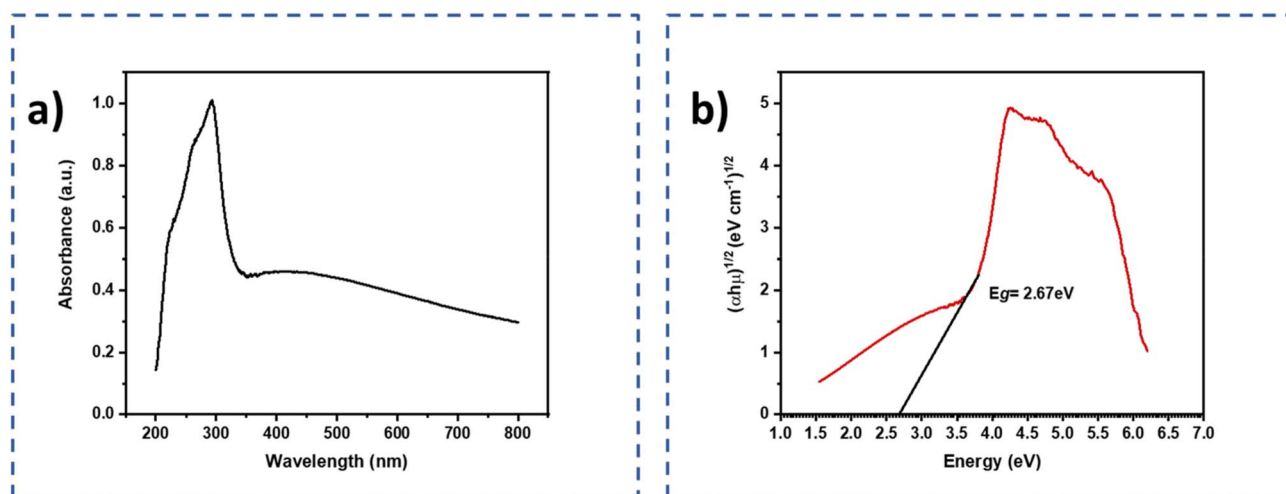


Fig. 2 (a) UV-vis absorption spectrum and (b) Tauc plot  $(\alpha h\nu)^2$  vs. photon energy (eV) of the  $\text{Bi}_3\text{ClO}_4$  photocatalyst.



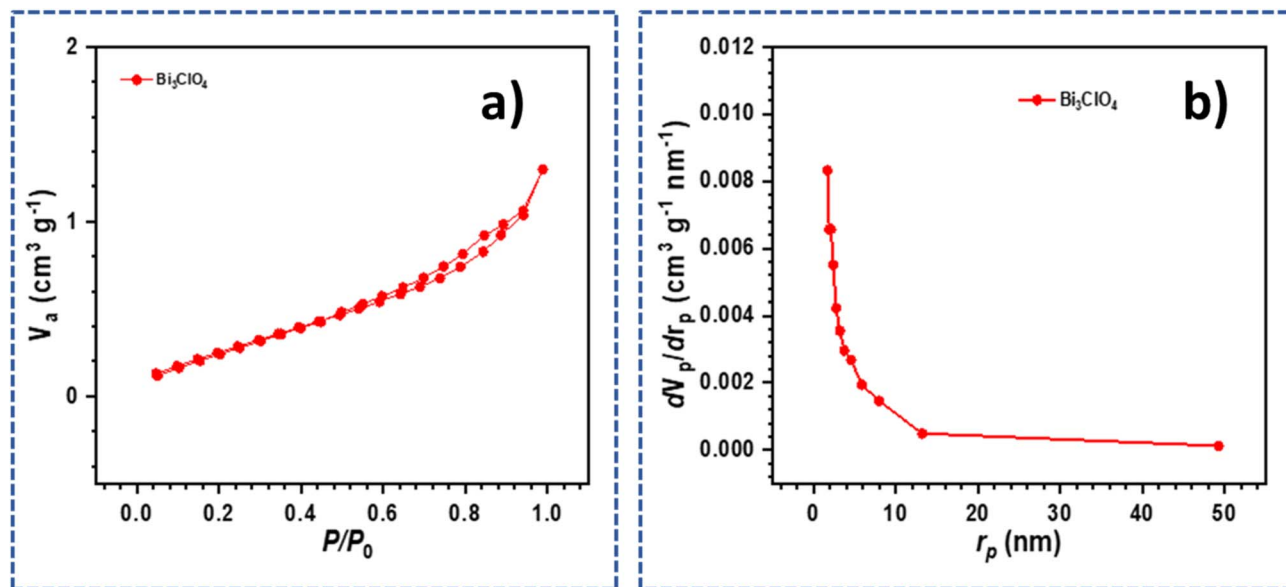


Fig. 3 (a) N<sub>2</sub> adsorption isotherm and (b) pore size variation of Bi<sub>3</sub>ClO<sub>4</sub>.

comparable or even lower values. For instance, P-BiOCl nanosheets exhibit a surface area of 13.4 m<sup>2</sup> g<sup>-1</sup>,<sup>56</sup> while hierarchical BiOCl structures show values as low as 7.3 m<sup>2</sup> g<sup>-1</sup> (ref. 57) and even 3.49 m<sup>2</sup> g<sup>-1</sup>, depending on the morphology and stacking of nanosheets.<sup>58</sup> Therefore, the obtained surface area of bismuth-based photocatalysts indicates the formation of nano-sized particles with accessible surface sites.<sup>59</sup> The presence of mesopores and interparticle voids can significantly enhance the mass transport of dye molecules and provide more adsorption sites for photocatalytic reactions. Furthermore, the mean pore radius remains nearly unchanged, suggesting that the observed increase in the surface area is mainly due to particle size reduction, improved dispersion, and structural reorganization rather than the creation of new pore channels. The enhanced surface area and mesoporous architecture are expected to play an important role in improving photocatalytic performance by providing more active reaction sites, shortening charge-carrier diffusion pathways, and facilitating the adsorption of organic dye molecules on the catalyst surface. In addition, the porous structure can promote light harvesting *via* multiple scattering within the pores, thereby increasing the probability of photon absorption. These combined structural and textural advantages are beneficial for the efficient separation and transfer of photogenerated electron-hole pairs, ultimately contributing to the improved photocatalytic activity of Bi<sub>3</sub>ClO<sub>4</sub> under visible-light irradiation.<sup>60</sup>

**4.1.5 Field-emission scanning electron microscopy analysis (FE-SEM).** The surface morphology and microstructural characteristics of the synthesized Bi<sub>3</sub>ClO<sub>4</sub> sample were thoroughly examined using field-emission scanning electron microscopy (FESEM), and the representative images are displayed in Fig. 4a–d. The low-magnification FESEM image (Fig. 4a) reveals that the Bi<sub>3</sub>ClO<sub>4</sub> sample is composed of densely packed, irregularly arranged petal-like structures forming a hierarchical assembly. These structures appear to be loosely

stacked and interconnected, producing a three-dimensional architecture with numerous open spaces and channels. Such a morphology indicates that the growth process favours anisotropic crystal formation, leading to the development of layered units that aggregate into larger clusters. A closer inspection at higher magnification (Fig. 4b) clearly shows that the material is constructed from ultrathin nanosheets that are curled, folded, and randomly oriented. These nanosheets resemble the natural petal-like structure of water hyacinth leaves (Fig. 4c), forming a flower-like or lamellar architecture. The sheets are not perfectly flat; instead, they exhibit bending, wrinkling, and edge curling, which suggests a very low thickness and high flexibility. The lateral size of these nanosheets typically ranges from 50 to 100 nm, while their thickness remains in the 5–10 nm scale. The interweaving of such thin sheets generates a porous framework with abundant voids between adjacent layers. This open and hierarchical nanosheet assembly is advantageous because it provides a large accessible surface area, promotes effective light scattering within the structure, and facilitates rapid diffusion of reactant molecules during photocatalytic processes. In the petal-like arrangement of the nanosheets, individual sheets overlap and radiate outward in a manner similar to the natural water hyacinth (*Eichhornia crassipes*) plant's leaves. This biomimetic morphology significantly increases the density of exposed edge sites, surface defects, and unsaturated coordination centers. These features are particularly beneficial for photocatalytic applications, as they can serve as active sites for adsorption and surface reactions. In addition, the intimate contact between adjacent nanosheets is expected to provide efficient pathways for charge transport, thereby reducing the recombination probability of photogenerated electron-hole pairs. The hierarchical stacking and porous arrangement also enhance photon harvesting by allowing multiple internal reflections of incident light, ultimately improving the photocatalytic efficiency of the Bi<sub>3</sub>ClO<sub>4</sub> material.



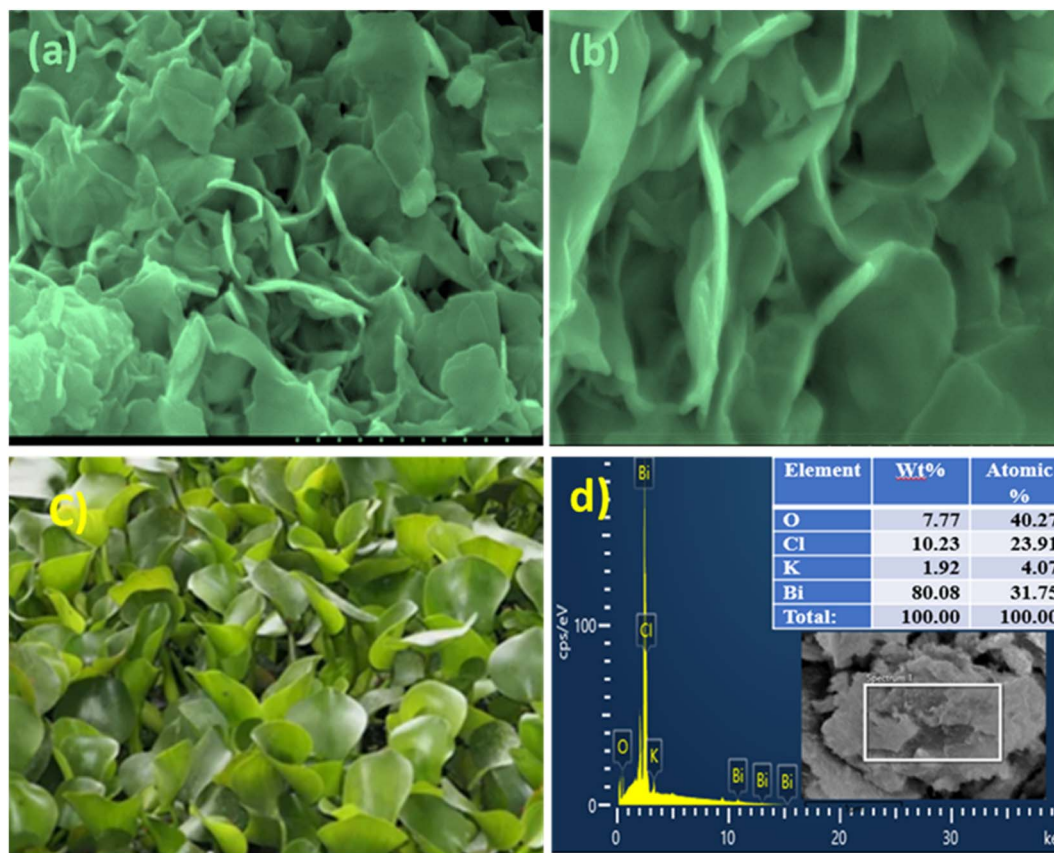


Fig. 4 (a and b) SEM images of  $\text{Bi}_3\text{ClO}_4$ ; (c) photograph of water hyacinth leaves (*Eichhornia crassipes*); (d) EDS spectrum of  $\text{Bi}_3\text{ClO}_4$ , with inset showing the corresponding SEM image of the selected area.

The elemental composition and chemical purity of the  $\text{Bi}_3\text{ClO}_4$  sample were analyzed by energy-dispersive X-ray spectroscopy (EDS), as presented in Fig. 4d. The EDS spectrum confirms the presence of Bi, O, and Cl as the primary elements, which is consistent with the expected composition of  $\text{Bi}_3\text{ClO}_4$ . A small signal corresponding to K is also observed, which may originate from residual precursor salts or synthesis-related additives. The quantitative analysis indicates that Bi is the dominant element, accounting for the majority of the weight percentage, while oxygen and chlorine are present in appropriate proportions to form the oxychloride structure. The absence of any additional impurity peaks suggests that the synthesized sample possesses high chemical purity. Furthermore, the inset of the mapping region demonstrates that the elements are uniformly distributed throughout the selected area, confirming compositional homogeneity at the microscale.

Overall, the FESEM and EDS analyses reveal that the synthesized  $\text{Bi}_3\text{ClO}_4$  exhibits a well-defined hierarchical nanosheet-assembled morphology resembling water-hyacinth-like petals. This unique architecture provides several structural advantages, including a high specific surface area, abundant exposed active sites, improved light-harvesting capability, and efficient charge-transfer pathways. Such morphological and compositional features are expected to play a crucial role in enhancing the photocatalytic activity and overall functional

performance of the  $\text{Bi}_3\text{ClO}_4$  material under visible-light irradiation.

#### 4.2 Photocatalytic degradation of the CV dye

The photodegradation of crystal violet (CV) was examined, and additional studies on the degradation of organic dyes over  $\text{Bi}_3\text{ClO}_4$  were performed under UV and visible-light irradiation. Crystal violet was chosen as one of the additional contaminants among various anionic dye types. A blank test was also done to make sure the dye was not photodegrading on its own. Excellent visible-light photocatalytic activity is demonstrated by  $\text{Bi}_3\text{ClO}_4$ , and it requires only 75 minutes to completely break down different contaminants. Under visible-light illumination, the photocatalytic degradation rates of pollutants were still higher than those under UV-light illumination, as shown in Fig. 5a–c. The photocatalytic activity of  $\text{Bi}_3\text{ClO}_4$  was tested for degrading the CV dye under visible-light irradiation, and the results are displayed in Fig. 5b. It can be seen that under dark conditions, only negligible amounts of CV are degraded. In contrast to the dark condition, the degradation activity under the light irradiation is extremely enhanced, achieving an approximately 100% degradation efficiency within 75 min in the presence of the  $\text{Bi}_3\text{ClO}_4$  photocatalyst. Fig. 5c shows the rate constant of the photocatalytic reaction, calculated from the slope of the plot of  $\ln(C_0/C_t)$  versus irradiation time, where  $C_0$  is the initial CV



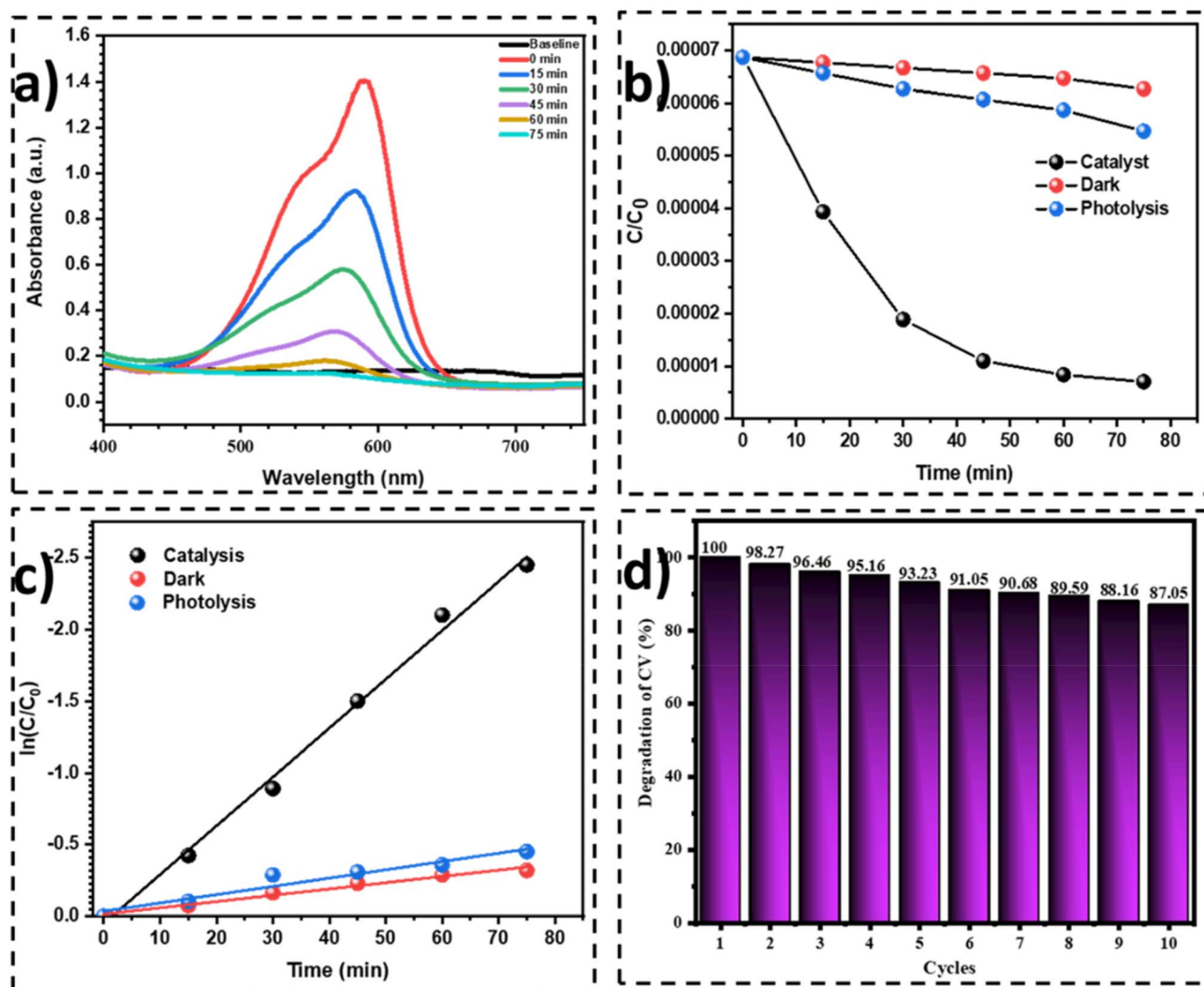


Fig. 5 (a) UV-visible spectra of the photocatalytic degradation of the CV dye, (b) time-evolved photocatalytic degradation of CV under visible light irradiation, (c) rate of chemical kinetics of the Bi<sub>3</sub>ClO<sub>4</sub> NPs during photocatalytic CV degradation, and (d) bar chart of the reusability test of Bi<sub>3</sub>ClO<sub>4</sub> NPs for the CV dye degradation.

concentration and  $C_t$  is the concentration of CV at time  $t$ . Therefore, the slope of the  $\ln(C/C_0)$  vs.  $t$  curve may be used to determine the rate constant  $k$ , which is  $0.050 \text{ min}^{-1}$ , indicating that the Bi<sub>3</sub>ClO<sub>4</sub> photocatalyst's ability to degrade the CV dye improved when exposed to visible light. The stability and reusability of the photocatalyst were assessed over ten successive photocatalytic cycles during the regeneration process (Fig. 5d). After each cycle, the synthesized photocatalyst was washed with room-temperature distilled water and ethanol and then dried at 60 °C to avoid any possible structural or surface modification. The degrading efficiency of crystal violet decreased from 100% to 87.05% after 10 cycles, which is a small 12.95% decrease. The result suggests that the produced catalysts have superior chemical stability and can potentially be recycled during the purification of wastewater containing organic colours. According to the Langmuir–Hinshelwood model, the photocatalytic degradation of organic substrates generally follows pseudo-first-order kinetics at low concentrations. Under such conditions, the reaction rate can be expressed

as a function of the pollutant concentration.<sup>61</sup> The stated pseudo-first-order equation is as follows:

$$\ln\left(\frac{C}{C_0}\right) = -kt. \quad (2)$$

The photocatalytic kinetics were analysed using the Langmuir–Hinshelwood pseudo-first-order model, expressed as  $\ln(C_0/C_t)$  versus time. The reaction rate constant ( $k$ ) was determined from the slope of the linear plot. As shown in Fig. 5c, a strong linear relationship between  $\ln(C_0/C_t)$  and time ( $t$ ) was observed for Bi<sub>3</sub>ClO<sub>4</sub> during the reaction stage ( $R^2 = 0.9931$ ), whereas photolysis and dark conditions exhibited lower linearity with  $R^2$  values of 0.97476 and 0.92056, respectively.

#### 4.3 Photocatalytic degradation of the MB dye

Visible light was used for the degradation of methylene blue (MB) in the presence of the Bi<sub>3</sub>ClO<sub>4</sub> catalyst. Among the



different kinds of anionic dyes, MB was selected as one of the extra pollutants. The examination of  $\text{Bi}_3\text{ClO}_4$ 's photocatalytic activity for the MB dye degradation under visible light irradiation is presented in Fig. 6a–c.  $\text{Bi}_3\text{ClO}_4$  showed excellent visible-light photocatalytic activity, and it took only 105 minutes to fully degrade pollutants. As seen in Fig. 6a, the photocatalytic degradation rates of contaminants under visible light irradiation were still higher than those under UV light illumination. Within 105 minutes, the  $\text{Bi}_3\text{ClO}_4$  disintegration rate reached 96% due to the acceptance of the electrons produced by the  $\text{Bi}_3\text{ClO}_4$  particles, which inhibits the production of charges. Under dark conditions, only negligible amounts of MB were degraded. In contrast to the dark condition, the degradation activity was extremely enhanced under light irradiation, achieving an approximately 96% degradation efficiency in the presence of the  $\text{Bi}_3\text{ClO}_4$  photocatalyst. The results are shown in Fig. 6b. The durability of the  $\text{Bi}_3\text{ClO}_4$  photocatalyst was investigated through ten repeated photocatalytic cycles, demonstrating its stability during the regeneration process (Fig. 6d).

After ten cycles, the degradation efficiency of methylene blue dropped by 9%, from 96% to 87%. The outcome indicated that the generated catalysts had better chemical stability and could be recycled to purify wastewater containing organic colours. The dependency of the photocatalytic reaction rate on the organic pollutant concentration at low substrate concentrations could be represented using a pseudo-first-order equation. As shown in Fig. 6c, a linear relationship between  $\ln(C_0/C_t)$  and time ( $t$ ) was observed for  $\text{Bi}_3\text{ClO}_4$  during the reaction stage ( $R^2 = 0.9097$ ), while photolysis and dark conditions exhibited  $R^2$  values of 0.97631 and 0.78367, respectively. The reaction rate constant ( $k$ ) was determined from the slope of the  $\ln(C_0/C_t)$  versus time plot, yielding a value of  $0.054 \text{ min}^{-1}$  for  $\text{Bi}_3\text{ClO}_4$ .

#### 4.4 Reactive species capturing experiment

The scavenger-based trapping experiments were carried out to identify the main reactive species. Ethylenediaminetetraacetic acid (EDTA), *p*-benzoquinone (BQ), sodium azide ( $\text{NaN}_3$ ), and isopropyl alcohol (IPA) were used as scavengers for

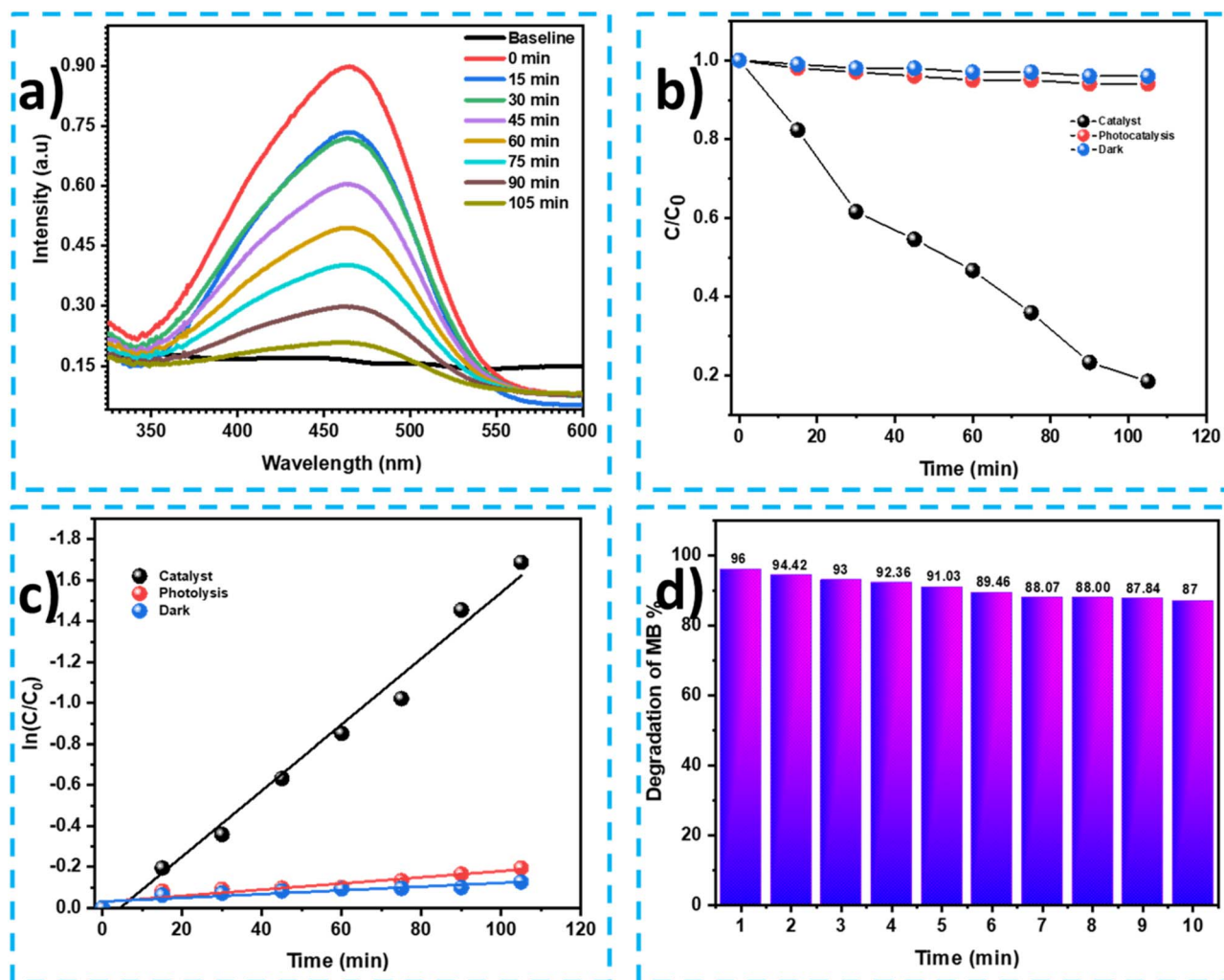


Fig. 6 (a) UV-visible spectra of the photocatalytic degradation of the MB dye, (b) time-evolved photocatalytic degradation of MB under visible light irradiation, (c) first-order kinetics of the photodegradation of the MB dye on  $\text{Bi}_3\text{ClO}_4$ , and (d) bar chart of the reusability test of  $\text{Bi}_3\text{ClO}_4$  NPs for the MB dye degradation.



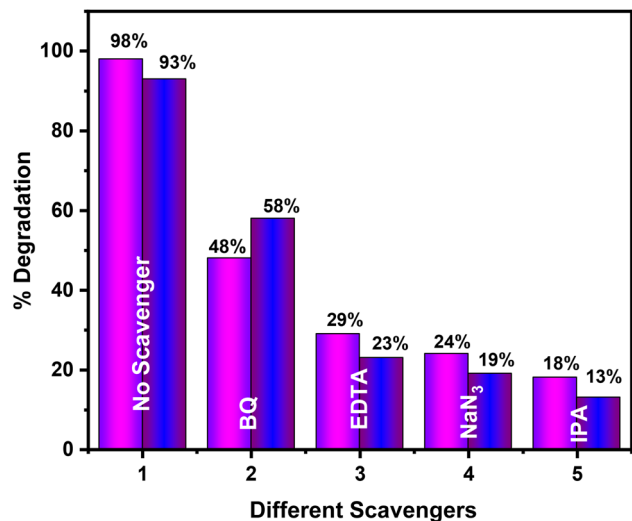
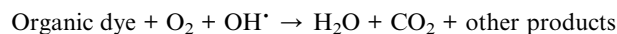
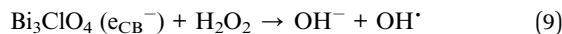
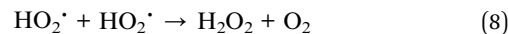
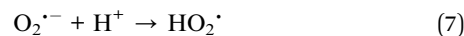
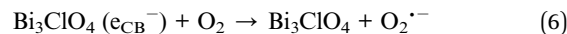
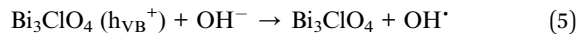
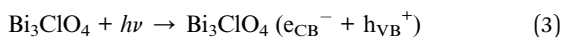


Fig. 7 Photocatalytic degradation efficiency of Bi<sub>3</sub>ClO<sub>4</sub> under sunlight irradiation in the presence of different scavengers: BQ (O<sub>2</sub><sup>•-</sup>), EDTA (h<sup>+</sup>), NaN<sub>3</sub> (<sup>1</sup>O<sub>2</sub>), and IPA (•OH).

photogenerated holes (h<sup>+</sup>), superoxide radicals (O<sub>2</sub><sup>•-</sup>), singlet oxygen (<sup>1</sup>O<sub>2</sub>), and hydroxyl radicals (•OH), respectively. In each experiment, 10 mL of 0.1 mol L<sup>-1</sup> solutions of EDTA, BQ, NaN<sub>3</sub>, and IPA was added separately to crystal violet (CV) and methylene blue (MB) dye solutions.

The role of reactive species in the photocatalytic degradation process was investigated using various scavengers under sunlight irradiation (Fig. 7). The photogenerated holes (h<sup>+</sup>) was evaluated using EDTA as a hole quencher. During CV degradation, the presence of EDTA results in only a slight reduction in removal efficiency, implying that holes contribute marginally to the oxidation process. In contrast, a substantial decline in MB degradation was observed upon EDTA addition, indicating that photogenerated holes play a significant role in the degradation pathway of MB. The involvement of singlet oxygen (<sup>1</sup>O<sub>2</sub>) was examined using sodium azide (NaN<sub>3</sub>). The pronounced suppression of MB degradation in the presence of NaN<sub>3</sub> confirms that singlet oxygen acts as a major reactive species in MB decomposition. However, its effect during CV degradation is comparatively minor, revealing only a secondary contribution. Notably, the addition of isopropanol (IPA), a well-known hydroxyl radical (•OH) scavenger, leads to a dramatic decrease in the degradation efficiencies of both CV and MB. This strong inhibitory effect demonstrates that hydroxyl radicals are the predominant reactive oxygen species governing the photocatalytic degradation of both dyes. The proposed photocatalytic reaction pathways are outlined in eqn (3)–(9).

#### 4.5 Reaction mechanisms of the Bi<sub>3</sub>ClO<sub>4</sub> photocatalyst for the removal of organic dyes



A plausible photocatalytic degradation mechanism of Bi<sub>3</sub>ClO<sub>4</sub> nanoparticles can be explained based on photoinduced charge generation and subsequent reactive oxygen species (ROS) formation. Upon irradiation with photons possessing energy equal to or greater than the band gap energy ( $E_g$ ), Bi<sub>3</sub>ClO<sub>4</sub> absorbs light and undergoes electronic excitation. Electrons from the valence band (VB) are promoted to the conduction band (CB), simultaneously generating positively charged holes (h<sup>+</sup>) in the VB (eqn (3)). This process results in the formation of electron–hole pairs within the semiconductor matrix. Following excitation, the photogenerated charge carriers migrate toward the surface of the catalyst. The conduction band electrons interact with dissolved molecular oxygen adsorbed on the catalyst surface, leading to the formation of superoxide radical species (O<sub>2</sub><sup>•-</sup>) (eqn (4)–(6)). Concurrently, the valence band holes react with surface-adsorbed water molecules or hydroxide ions to generate highly oxidative hydroxyl radicals (•OH) (eqn (5)). The formed superoxide radicals may further participate in secondary reactions. In the presence of protons (H<sup>+</sup>), O<sub>2</sub><sup>•-</sup> can be converted into hydroperoxyl radicals (•OOH), which subsequently undergo disproportionation or reduction steps to produce hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>). Hydrogen peroxide can then decompose, either photolytically or *via* electron-mediated pathways, yielding additional hydroxyl radicals (•OH) (eqn (7)–(9)). These reactive oxygen species, particularly hydroxyl radicals, possess strong oxidative potential and are primarily responsible for the breakdown and mineralization of organic dye molecules. The overall degradation pathway thus involves sequential charge generation, surface redox reactions, and ROS-mediated oxidation of pollutants.

Based on the active species trapping experiments and band energy level analysis, a plausible photocatalytic degradation mechanism of crystal violet (CV) and methylene blue (MB) over Bi<sub>3</sub>ClO<sub>4</sub> is proposed in the Fig. 8. The trapping results reveal that the photogenerated holes (h<sup>+</sup>) in the VB are the dominant active species responsible for the degradation of both CV and MB. These highly oxidative holes can directly oxidize dye molecules adsorbed on the catalyst surface. Meanwhile, the photoinduced electrons in the CB react with dissolved oxygen (O<sub>2</sub>) to generate superoxide radicals (O<sub>2</sub><sup>•-</sup>), which further participate in the degradation process. Additionally, hydroxyl radicals (•OH) may be formed either *via* the oxidation of surface-adsorbed H<sub>2</sub>O/OH<sup>-</sup> by h<sup>+</sup> or through subsequent reactions involving O<sub>2</sub><sup>•-</sup>. Although O<sub>2</sub><sup>•-</sup> and h<sup>+</sup> contribute to dye



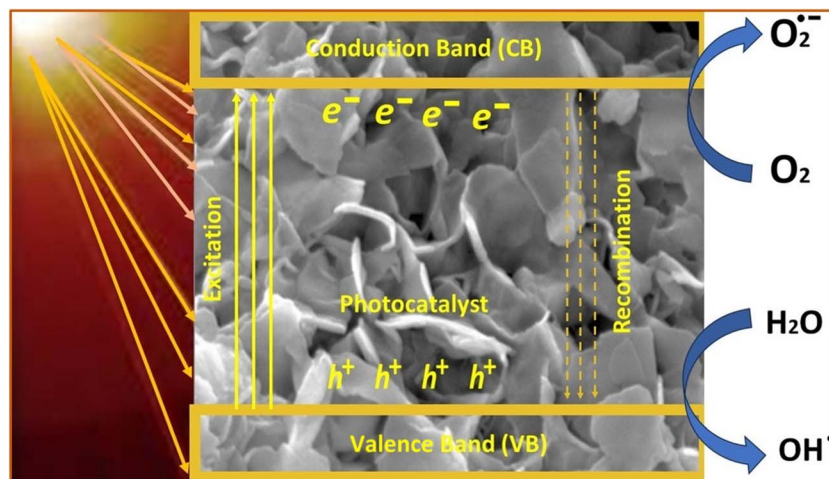


Fig. 8 Schematic of the photocatalytic production of radicals for the degradation of dyes under visible light irradiation.

decomposition, their roles are secondary compared to the direct oxidation by hydroxyl radicals ( $\bullet OH$ ), as evidenced by the scavenger experiments. Therefore, the photocatalytic degradation of CV over  $Bi_3ClO_4$  is primarily governed by the strong oxidative ability of hydroxyl radicals ( $\bullet OH$ ), whereas the degradation of MB is controlled by the combined action of  $\bullet OH$ , photogenerated holes ( $h^+$ ), and superoxide radicals ( $O_2^{\bullet -}$ ), enhancing the overall degradation efficiency.

## 5 Conclusion

In conclusion, this study successfully synthesized bismuth oxychloride ( $Bi_3ClO_4$ ) nanomaterials by a co-precipitation method for the efficient photocatalytic degradation of dyes. The synthesized material has a monoclinic structure and a crystallite size of 5–6 nm. Different characterization techniques, including XRD, Raman spectroscopy, UV-visible spectroscopy, FESEM, and EDS, confirm the successful synthesis, structural integrity, elemental composition, and nanosheet-like morphology of the nanomaterials. The monoclinic bismuth oxychloride ( $Bi_3ClO_4$ ) demonstrates superior performance, achieving nearly 100% degradation of crystal violet (CV) dye within 75 minutes and 96% degradation of the MB dye within 105 minutes of visible light irradiation. These remarkable results highlight the potential of the monoclinic bismuth oxychloride ( $Bi_3ClO_4$ ) nanomaterial as a promising photocatalyst for environmental remediation and wastewater treatment applications.

## Author contributions

Rajvardhan K. Chougale: methodology, writing – original draft, Prashant D. Sanadi: resources, Sharadchandra S. Patil: resource, Umesh S. Siddharth: resource, Pravin P. Pawar: resource, Babasaheb D. Bhosale: supervision, and Ganesh S. Kamble: methodology, supervision, project administration, validation and revision.

## Conflicts of interest

There is no actual or potential conflict of interest in relation to this article.

## Data availability

Original data were generated and analysed in this study. All data are provided within the manuscript.

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