



# Hydrothermal fabrication and characterization of novel CeO<sub>2</sub>/PbWO<sub>4</sub> nanocomposite for enhanced visible-light photocatalytic performance

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## Abstract

In this revision, a series of novel visible-light-driven (VLD) CeO<sub>2</sub>/PbWO<sub>4</sub> nanocomposites (NCs) were effectively fabricated by facile hydrothermal preparation way. The UV–Vis absorption spectra exposed that CeO<sub>2</sub> NPs prolonged the adsorption edge of the CeO<sub>2</sub>/PbWO<sub>4</sub> composite to the extensive visible region, which allied to decreases of the bandgap. As-prepared CeO<sub>2</sub>/PbWO<sub>4</sub> NCs revealed superior photocatalytic action under visible-light and could degrade the Methylene Blue (MB) dye solution in 140 min. The photodegradation efficacy of CeO<sub>2</sub>/PbWO<sub>4</sub> NCs was improved catalytic activity, which is around 1.45 and 2.7 times that of CeO<sub>2</sub> and PbWO<sub>4</sub> nanoparticles (NPs) individually. Besides, the CeO<sub>2</sub>/PbWO<sub>4</sub> catalysts display notable stability and reusability performance in four succeeding cycles. The development in the photocatalytic enactment of combined CeO<sub>2</sub>/PbWO<sub>4</sub> nanocomposite could be recognized not only to the sturdy visible-light absorption responses and separating the photoexcited electron–hole pairs. Also, the plausibly systematic illumination of charge transference and exploitation of reactive species for superior photocatalytic action in visible-light have been discussed. It is projected that the CeO<sub>2</sub>/PbWO<sub>4</sub> NCs could be used as effective photocatalysts for promising applications for environmental wastewater refinement.

**Keywords** CeO<sub>2</sub>/PbWO<sub>4</sub> · Nanocomposites · Hydrothermal · Visible light · Photodegradation · Active species

## Introduction

Over the former few years, water contaminants have industrial wastewater become the greatest challenging ecological concerns and thus aroused abundant courtesy in the progress of modern society (Gour and Jain 2019). Wastewater usually covers a huge amount of organic pollutants (such as reactive dyes, pesticides and antibiotics) which is adverse things on aquatic ecologies equilibrium and human healthiness. The textile, paper-making, cosmetics, food industries and dye houses have used countless organic dyes which are the prime causes for the contamination of environmental wastewater due to its toxicity and non-biodegradability is a vital environmental issue (Dhmees et al. 2019). Various traditional systems, such as electrochemical oxidation, membrane filtration, adsorption, chlorination, reverse osmosis,

and photocatalysis, have been agreed to treat the harmful dyes covering wastewater. Amid these skills, semiconductors (SCs) based photocatalysis has drawn increasing interest because it offers a capable substitute strategy to eradicate the dye-containing wastewater since of its high efficacy, green reaction route and moderate reaction settings (Rohini et al. 2017) for solving the recent severe problems of environmental pollution and energy shortages. Unfortunately, the utmost of these physical, chemical and biological systems could custom to secondary impurities simply through degradation manners (Venkatasubramanian et al. 2008). The advanced oxidation practices (AOPs) of semiconductor photocatalysts (PCs) were broadly considered by deprivation of various noxious organic toxins in environment remediation and antimicrobial action (Girase et al. 2011; Depan and Misra 2014; Zhang et al. 2019b; Shanmugam et al. 2020). The photodegradation and mineralization of the dyes by nano-configuration semiconductor in visible-light treated progress has engrossed great concern in modern years. Various metal oxides (such as ZnO, TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, CeO<sub>2</sub>, and WO<sub>3</sub>) have been repeatedly employed as support in heterogeneous catalysis of organic pollutants degradation in the wastewater and antimicrobial activity (Rana et al. 2006; Rawat et al. 2007a;

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Ke et al. 2008; Sunkara and Misra 2008; Xu and Wang 2012; Li et al. 2019).

As a significant rare earth metal oxide chains, cerium oxide ( $\text{CeO}_2$ ), a wide bandgap SCs which fascinates light in the nearby UV and slightly visible region. Likewise, the  $\text{CeO}_2$  has eco-friendly photocatalytic material, has encouraged abundant concern of the researchers owing to its favorable applications, for instance, plentiful oxygen sensors, luminescent things besides admirable chemical constancy, high catalytic action and cost-effective nature (Li et al. 2019; Qi et al. 2019). This material was also functional for solar water splitting ( $\text{Ce}^{4+}/\text{Ce}^{3+}$ ) into hydrogen creation and concerned increasing attention used for deletion of organic pollutants from wastewater owed to its strong light captivation (Priyadharsan et al. 2017a). Though, the key weakness of  $\text{CeO}_2$  is the absence of visible-light consumption since the large bandgap (2.92 eV) and weak separation efficacy of photoexcited carriers hinders have its widespread request in photocatalysis. Hence it is probable to outspread the visible-light captivation skill of  $\text{CeO}_2$  either by doping of metals/nonmetals or fashioning a heterojunction between  $\text{CeO}_2$  and another narrow bandgap SCs to create VLD PCs (Cano-Franco and Álvarez-Láinez 2019a). Unfortunately, the UV region signifies a little amount (3–5%) of photon flux whereas the visible region attains 45% of daylight. So that, it has required to progress the photocatalytic efficacy by doping or coupling the  $\text{CeO}_2$  with narrow bandgap materials which could diminish the recombination of the photogenerated electron/hole pair rate and extend their lifetime important to the excellent light absorption from UV to the visible region for the elimination of organic pollutants (Ma et al. 2019). As a member of the tungstate family, lead tungstate ( $\text{PbWO}_4$ ) is a scientific importance inorganic scintillating semiconductor, which has vast potential applications like humidity identical sensors, solid-state lasers ground, optical fibers, and catalysts (Pourmasoud et al. 2017; Rajendran et al. 2019).  $\text{PbWO}_4$  is utmost smart for high-energy physics uses since of its great density ( $8.3 \text{ g/cm}^3$ ), short decay time ( $< 10 \text{ ns}$  for a large part of light output), high-irradiation destruction resistance, exciting excitonic luminescence, thermo-luminescence, encouraged Raman scattering manners (Yue et al. 2016).

In this present effort, we report novel  $\text{CeO}_2/\text{PbWO}_4$  heterojunction PCs that were effectively fabricated via a simplistic hydrothermal method. The as-obtained nanocomposites (NCs) were categorized by numerous analytical tools such as XRD, FTIR, HR-SEM, HRTEM, PL and UV-DRS spectroscopy were employed to study the crystalline formation, phase configuration, morphology, and optical possessions parallel with  $\text{CeO}_2$  and  $\text{PbWO}_4$  samples. The as-organized  $\text{CeO}_2/\text{PbWO}_4$  NCs were used as a UV and/or visible-light focused photocatalyst towards the photodegradation of MB dye. Coupled with  $\text{PbWO}_4$  on the superficial of  $\text{CeO}_2$  NPs are projected to increase the

surface area of composite providing further response sites owing to lower bandgap and so improve its photocatalytic action. Eventually, a reasonable mechanism and photostability of the catalyst are also anticipated in detail.

## Experimental part

### Materials

Lead nitrate hexahydrate ( $\text{Pb}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ; 99%), Sodium tungstate dihydrate ( $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ , 99%), Ethylene glycol (99%) were procured from Himedia Ltd. Potassium chloride (Merck, 99%), Cerium nitrate, ( $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ; 98%) were obtained since SRL Chem. Limited. Sodium hydroxide (NaOH), isopropanol (IPA), Di-sodium Ethylene Diamine Tetra Acetic Acid (EDTA-2Na), benzoquinone (BQ), and absolute ethanol ( $\text{CH}_3\text{CH}_2\text{OH}$ ) were acquired from SDFCL Chemical Reagent Co., Pvt. Ltd. Methylene blue (MB;  $\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}$ ) dye from SD Fine and was used as received. Deionized water (D.I) was used for the preparation of all solutions. All the chemicals were analytical reagent (A.R) grade also have auxiliary purified before use.

### Preparation of $\text{CeO}_2/\text{PbWO}_4$ photocatalyst

In a typical synthesis, 0.03 mol of  $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  was ultrasonically dissolved in 100 mL of D.I water, then 10 mL  $\text{NH}_4\text{OH}$  was gradually dropped directly into the above reaction mixture for pH extended at  $\sim 12$  under constant stirring for 30 min. Lastly, the composed precipitants (Cui et al. 2019) were kept dry at  $60 \text{ }^\circ\text{C}$  for 8 h and extra calcined at  $400 \text{ }^\circ\text{C}$  for 1.5 h to attain  $\text{CeO}_2$  NMs. In this research, the  $\text{CeO}_2$  blended  $\text{PbWO}_4$  NMs, via 0.03 mol of  $\text{Pb}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and 0.03 mol of  $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$  solution was added by 50 ml of D.I water. And 1 mol (50 mL) of  $\text{NH}_4\text{OH}$  solution was additional in the pioneer solution, although the pH value was touched at  $\sim 11$ – $12$ . Afterwards, being stirred for 3 h, the 0.1 g of as-obtained  $\text{CeO}_2$  NMs was auxiliary added in the upstairs produces and formerly stirred for 2 h. Then, the reaction mixer was relocated to heat-treat by  $160 \text{ }^\circ\text{C}$  aimed at 24 h in a 250 mL Teflon-lined stainless steel autoclave. Lastly, the autoclave was then cooled to room temperature, hence the as-attained  $\text{CeO}_2/\text{PbWO}_4$  precipitate was centrifugated and rinsed carefully with ethanol/D.I. water further dry at  $70 \text{ }^\circ\text{C}$  for 8 h. Besides, the  $\text{CeO}_2/\text{PbWO}_4$  NCs was attained (Jeyakanthan et al. 2018). Advising to this outline, the pristine  $\text{PbWO}_4$  NPs was also attained via without accumulation of  $\text{CeO}_2$  NMs.

## Characterization of the obtained samples

The crystal organization and phase of as-obtained nanocomposite was estimated by X-ray diffraction (Rigaku Miniflex-II; X-ray diffractometer) over CuK $\alpha$  radiation in the 2 $\theta$  range from 10 to 80°. FTIR revisions were done by Perkin Elmer RX-1 FTIR spectrophotometer. Surface morphologies and microstructure of as given NMs were scrutinized via high-resolution (HR-SEM; HITACHI S-3000 H) scanning electron microscope and high-resolution transmission electron microscopy (HR-TEM; JEM-2011; JEOL-Japan) instruments. Energy-Dispersive X-ray spectroscopy (EDX-attached with HRSEM) was used to evaluate the elemental compositions of the NCs. To measure the optical assets of the attained samples were considered by a UV–Vis DRS spectrophotometer (UV2550 model, Shimadzu—Japan). Photoluminescence (PL) spectrophotometry was performed with a (Perkin-Elmer-LS 100) to determine the electron–hole recombination rate at an excitation series of  $\sim$ 342 nm. The optical absorption of dye degradation samples was performed via a UV–Vis spectrophotometer (UV; Perkin-Elmer Lambda-19). The absorption spectra in the photodegradation rate process of MB dye solutions also measured with a UV–Vis (Perkin Elmer-Lambda 35) spectrometer.

## Description of the photocatalytic activity of MB dye

The photodegradation performing of as-attained samples (50 mg) was measured via deprivation of MB dye (20 ppm; 100 mL solution; 10 mg/L) under visible-light exposure (300 W Xe lamp by  $\lambda > 420$  nm cutoff filter in a Pyrex photocatalytic vessel). Preceding to exposure, the suspensions stayed constant magnetically stirred for around 30 min in the dark to certify that the dyes might extend the absorption–desorption balance on the photocatalyst superficial and dyes (Ali Baig et al. 2020). At certain time pauses of 20 min irradiation, 2.5 mL of aliquots were collected. The degraded resolutions were explored by UV–Vis absorption peaks of corresponding MB dye (wavelength at  $\sim$ 663.5 nm) (Zelege and Kuo 2019). The photodegradation efficacy was extent as resulting formula, Efficiency (%) =  $(C_0 - C_t)/C_0 * 100$ , wherever  $C_0$  and  $C_t$  exist the absorbance rate of dyes solution earlier and afterwards destruction. Finally, through the degradation manner, photocatalyst was separated from the reaction blend and dried to succeed in the reusability trials (Zhang et al. 2017).

## Active species trapping experiments

To detect that the reactive species, certain caused/trapping mechanism during the photocatalytic manner, 1 mM of EDTA-2Na IPA and BQ and were added as scavengers of holes ( $h^+$ ) hydroxyl radicals ( $\bullet OH^-$ ) and superoxide

radical ( $\% O_2\bullet^-$ ) exclusively surveyed by the photocatalytic valuations, hence to catch the detection of dynamic reactive species (Cardillo et al. 2016).

## Results and discussion

### Crystal structure investigation

To explore the crystal constructions and phase composition of the as-obtained samples, XRD evaluations were carried out. As exposed in Fig. 1a, the key diffraction peaks are indexed to the (111), (200), (220), (311), (222), (400) and (311) crystal planes, which was in moral contract with the typical cubic crystalline phase structure pattern of CeO<sub>2</sub> (JCPDS card No: 89-8436) (Syed Khadar et al. 2019). For PbWO<sub>4</sub>, the diffraction peaks (Fig. 1b) and consistent planes of (112), (004), (200), (204), (220), (116) and (312), respectively, could be well-indexed to the pure tetragonal stoltzite phase (JCPDS card No. 19-0708) (Xiong et al. 2015). In Fig. 1c, designating a good crystallinity of CeO<sub>2</sub>/PbWO<sub>4</sub> NCs revealed the association with both the distinctive diffraction peaks of CeO<sub>2</sub> and PbWO<sub>4</sub> crystalline phases. The solid and sharp diffraction peaks proposed that the CeO<sub>2</sub>/PbWO<sub>4</sub> nanocomposite was fine crystalline in nature. No characteristic impurity peaks from other crystalline forms were detected which proves that the CeO<sub>2</sub>/PbWO<sub>4</sub> NCs has high purity. The average crystallite size (D) of as-prepared NCs was intended by the full width half maximum (FWHM) using Scherer's equation (Ramos-Corella et al. 2019), The average crystalline sizes for the pristine CeO<sub>2</sub>, PbWO<sub>4</sub> and CeO<sub>2</sub>/PbWO<sub>4</sub> nanocomposite was found to be 24, 28 and 21.5 nm, individually. The intensity variance and peak extending of the CeO<sub>2</sub>/PbWO<sub>4</sub> NCs are ascribed to a substantial reduction of crystallite sizes (Table 1).

### FT-IR analysis

To decide the proper functional groups in final catalysts samples, FT-IR spectra of as-obtained CeO<sub>2</sub>, PbWO<sub>4</sub> and CeO<sub>2</sub>/PbWO<sub>4</sub> NCs were perceived in the series of 400–4000 cm<sup>-1</sup> and shown in Fig. 2. An exact extensive band of 3100–3650 cm<sup>-1</sup> is recognized to the typical surface hydroxyl (O–H) stretching mode (Saravanakumar et al. 2019). The bending vibration group of actually adsorbed water (H<sub>2</sub>O) molecules are besides observed at 1635–1670 cm<sup>-1</sup> band. An increase in the number of surfaces -OH groups could expand the photocatalytic action. The existence of sharp dominated absorption peaks on 570–730 cm<sup>-1</sup> which is linked to the metal–O (Ce–O, W–O, Pb–O) bonds/stretching vibration are confirmed that the prepared NCs (Syed Khadar et al. 2019). Usually, spinel oxide and metal–oxygen broadening frequencies are perceived in

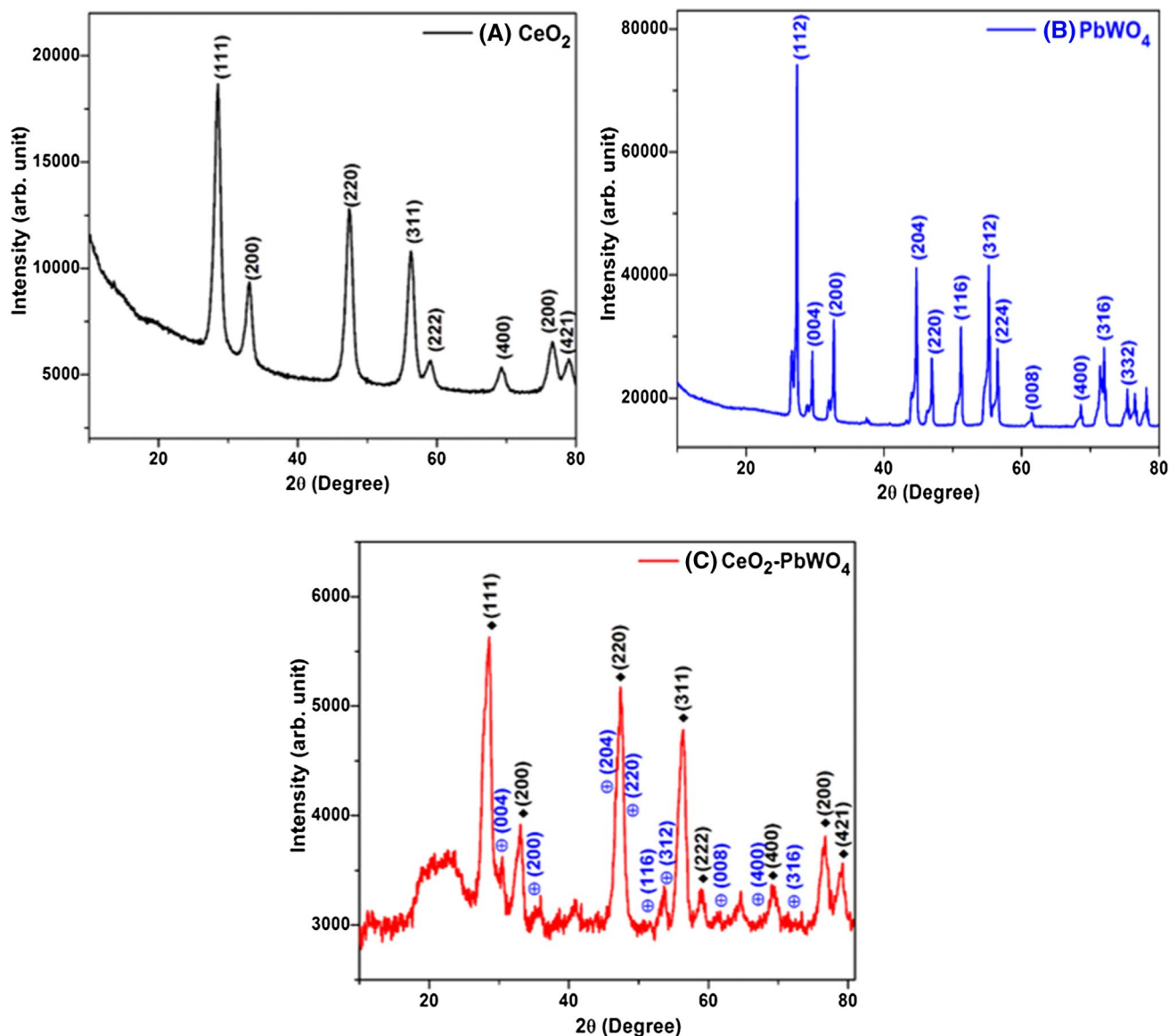


Fig. 1 XRD pattern of as obtained nanomaterials

**Table 1** Comparison of visible-light assisted MB dye photodegradation rate (%) over earlier reported nanocomposite materials

S. No	Nanomaterials	Irradiation time (mins.)	Degradation efficiency (%)	References
1	CeO <sub>2</sub>	180	86	(Reddy Yadav et al. 2017)
2	NiWO <sub>4</sub>	330	60	(AlShehri et al. 2017)
3	g-C <sub>3</sub> N <sub>4</sub> /BaTiO <sub>3</sub>	360	76	(Xian et al. 2015)
4	g-C <sub>3</sub> N <sub>4</sub> /CeO <sub>2</sub>	120	74	(Kumar et al. 2013)
5	CdS/TiO <sub>2</sub>	310	62	(Wei et al. 2014)
6	α-Fe <sub>2</sub> O <sub>3</sub> /TiO <sub>2</sub>	160	80	(Li et al. 2016)
7	CeO <sub>2</sub> /PbWO <sub>4</sub>	140	94	This work



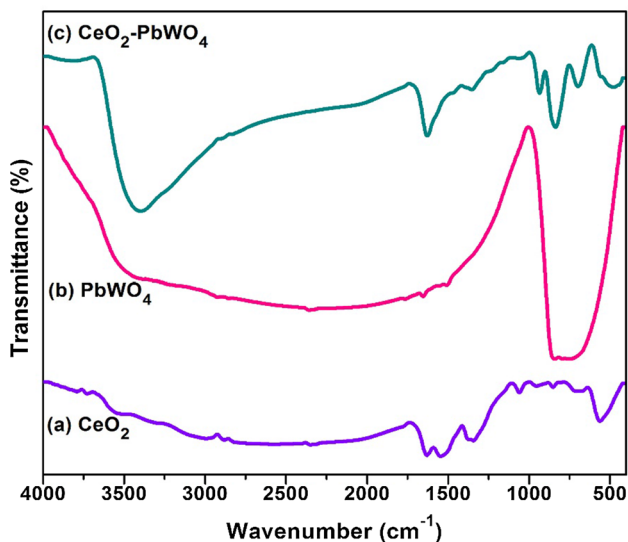


Fig. 2 FT-IR spectra of as-prepared samples

the peak range of 650–850  $\text{cm}^{-1}$ . No auxiliary absorption group was sensed in the experimental FTIR spectrum (Rana et al. 2005b).

### Morphology and microstructure analysis

To acquire detailed evidence about exterior morphology and microstructure of the  $\text{CeO}_2$ ,  $\text{PbWO}_4$  and  $\text{CeO}_2/\text{PbWO}_4$  NCs were inspected by HRSEM and HRTEM. Figure 3a the HRSEM images of pristine  $\text{CeO}_2$  demonstrates spherical shaped aggregates morphology. Figure 3b for  $\text{PbWO}_4$  shows a uniform ball-shaped structure was obtained. Also the Fig. 3c the  $\text{CeO}_2/\text{PbWO}_4$  NCs displays the high agglomeration of  $\text{CeO}_2$  NPs rendered with non-uniform spherical fashioned aggregates (Ramasamy Raja et al. 2019). The elemental composition and purity analysis of  $\text{CeO}_2/\text{PbWO}_4$  NCs have been determined from EDX extents. The EDX inquiry authorizes the existence of all essential elements

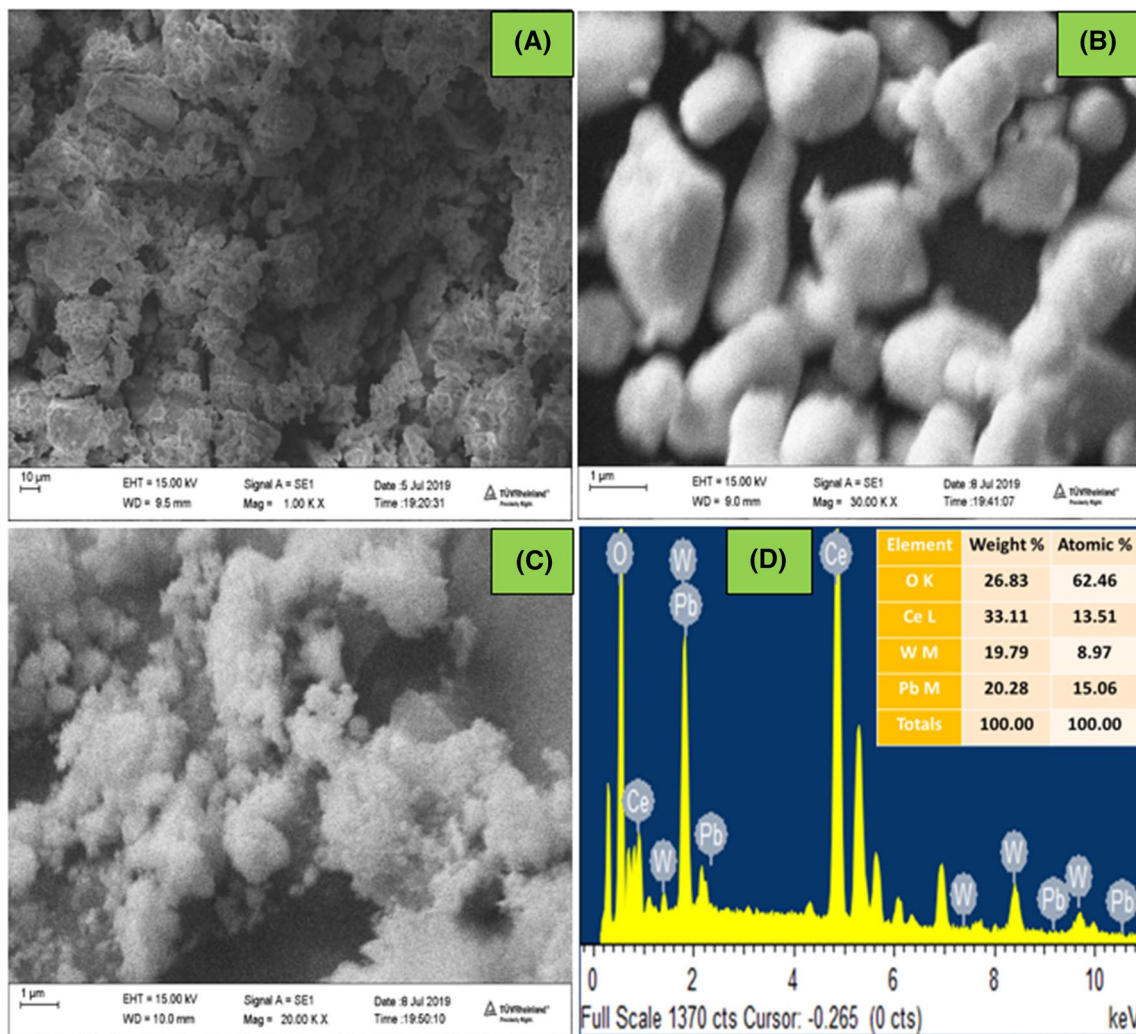


Fig. 3 HRSEM micrographs of as-synthesized a  $\text{CeO}_2$  b  $\text{PbWO}_4$  c  $\text{CeO}_2/\text{PbWO}_4$  NCs and d EDX spectrum of the  $\text{CeO}_2/\text{PbWO}_4$  NCs

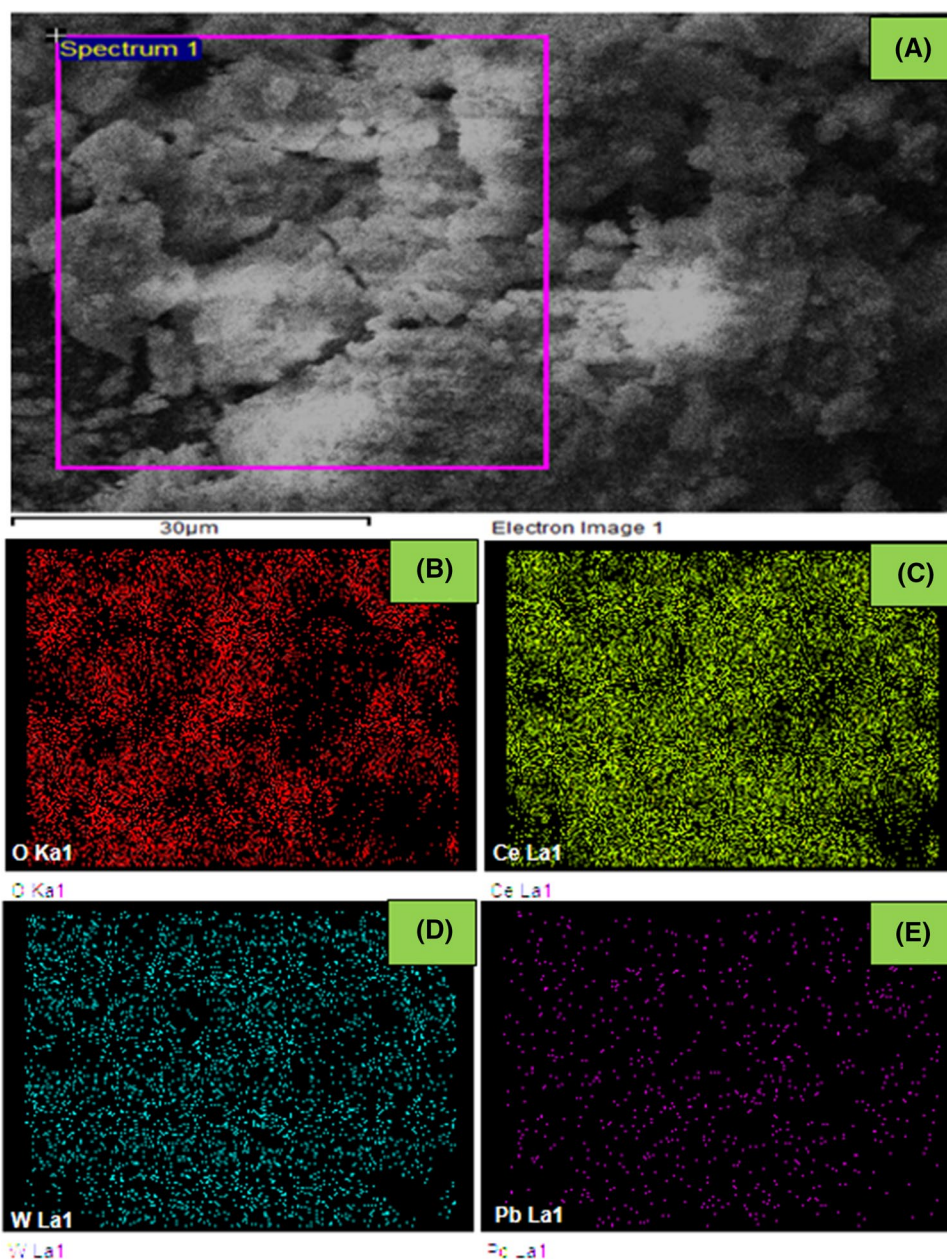
such as Ce, W, Pb, and O individually from  $\text{CeO}_2/\text{PbWO}_4$  NCs as described in Fig. 3d, also the weight % are detected and inserted in Fig. 3d (Lan et al. 2018). Moreover, the EDX elemental mapping analysis characterizes that the distribution of W, Ce, O and Pb elements separately and as illustrated in Fig. 4. It is well evident that  $\text{PbWO}_4$  and  $\text{CeO}_2$  are evenly circulated in the  $\text{CeO}_2/\text{PbWO}_4$  NCs. Furthermore, the EDX spectra and corresponding elemental mapping outcomes of as-obtained  $\text{CeO}_2/\text{PbWO}_4$  NCs which are very pure and no other impurities are found. The HRTEM analysis of  $\text{CeO}_2/\text{PbWO}_4$  NCs is publicized in Fig. 5a–b. The HRTEM micrograph indicates that the number of  $\text{CeO}_2$  fine NPs has indeed deposited compactly on the  $\text{PbWO}_4$  surface structure

and also homogeneous dispersion nature were forming the nano-sized composite (Aboutaleb and El-Salamony 2019). Moreover, the surface has several irregular small granules with additional agglomeration and the shape is more or less spherical. The intimate contact amid  $\text{PbWO}_4$  and  $\text{CeO}_2$  facilitates the separation of the photoexcited carriers, which favours the enhancement of photocatalytic concert (Hezam et al. 2017).

### Optical properties

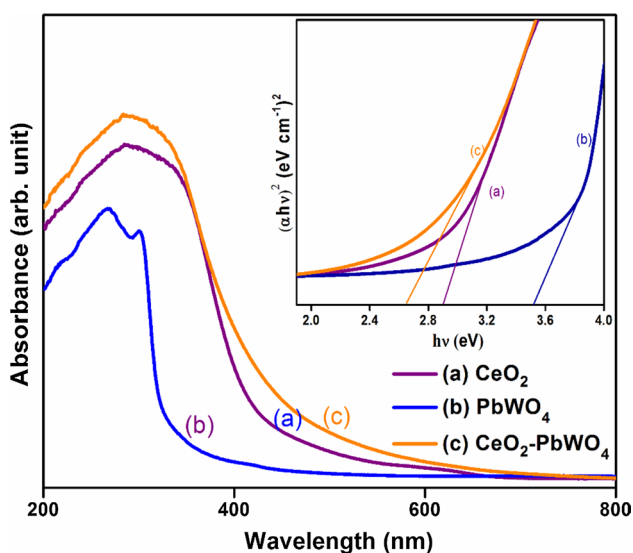
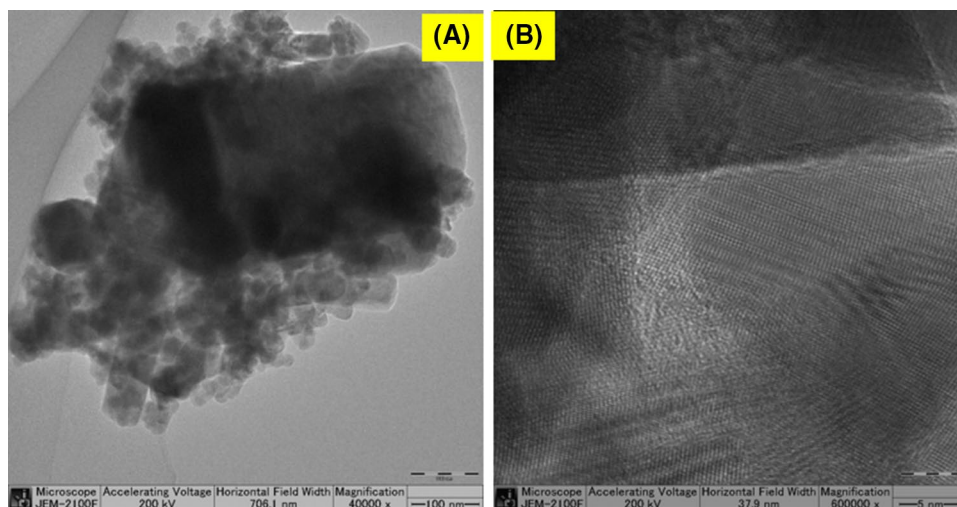
The optical properties and energy bandgap of the as-obtained samples have very important to determine the

**Fig. 4** EDX elemental mapping analysis of  $\text{CeO}_2/\text{PbWO}_4$  nano-composite





**Fig. 5** HRTEM images of as-obtained CeO<sub>2</sub>/PbWO<sub>4</sub> NCs



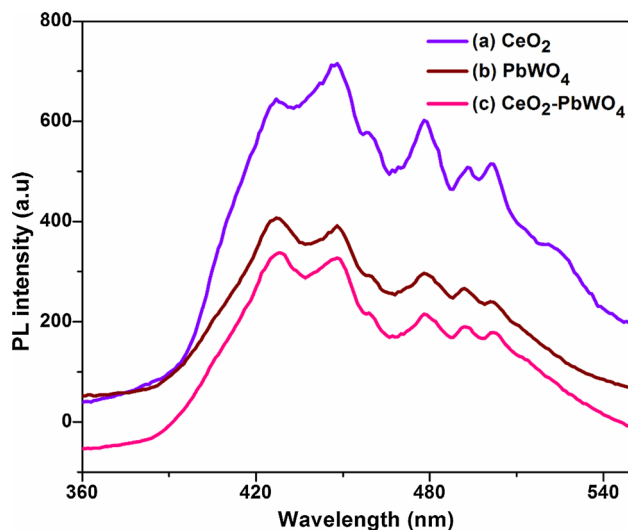
**Fig. 6** a UV–Vis DRS absorption spectra b Tauc's plots of the CeO<sub>2</sub>, PbWO<sub>4</sub> and CeO<sub>2</sub>/PbWO<sub>4</sub> NCs

photocatalytic behaviors (Liang et al. 2017) were studied by UV–Vis DRS as exhibited in Fig. 6. The CeO<sub>2</sub>, PbWO<sub>4</sub> and CeO<sub>2</sub>/PbWO<sub>4</sub> nanomaterials (NMs) were shown in strong absorption ability in the wavelength range of 200–800 nm. Related with pristine CeO<sub>2</sub> and PbWO<sub>4</sub>, the CeO<sub>2</sub>/PbWO<sub>4</sub> NCs consume broader absorption competence (Rana et al. 2005a; Cano-Franco and Álvarez-Láinez 2019b), which is superior visible-light harvesting capacity and redshift (~290–418) of absorption edge implying that the CeO<sub>2</sub>/PbWO<sub>4</sub> NCs owns admirable visible-light dynamic photocatalytic action (David et al. 2018; Wang et al. 2019). The bandgap energy was determined by fitting the absorption facts since the direct transition equation,  $(\alpha h\nu) = A(h\nu - E_g)^n$ , where  $h$  refers the Planck's constant,  $\alpha$  stands for absorption coefficient,  $E_g$  stands for bandgap (eV),  $\nu$  has shorted in the

frequency of vibration,  $A$  is the relatively constant. Also,  $n$  refers could have values  $\frac{1}{2}$  and  $2$  contingent on the kind of inter-band conversion, i.e., direct and indirect allowed transition, individually. The bandgap energy ( $E_g$ ) values are optically deduced from the Tauc plots and the graph plotted by  $(\alpha h\nu)^2$  versus the photon energy ( $h\nu$ ). The  $E_g$  values of CeO<sub>2</sub>, PbWO<sub>4</sub> and CeO<sub>2</sub>/PbWO<sub>4</sub> heterojunction NCs are nearly 2.92, 3.52 and 2.68 eV, separately were exposed in insert of Fig. 6. However, the CeO<sub>2</sub> combined PbWO<sub>4</sub> NCs could reduce the bandgap energy of CeO<sub>2</sub>/PbWO<sub>4</sub> NCs which is owed to an energy transition since the visible region triggered by an active band of PbWO<sub>4</sub> effectively deposited on the CeO<sub>2</sub> surface (Liu et al. 2019). In the combined effect of light, absorption would have an obvious effect while catalytic action towards the degradation of dyes qualified to absorption of quite visible-light to probably make more charge carriers (Velusamy and Lakshmi 2017).

## Photoluminescence analysis

The photoluminescence (PL) system is also an effective approach to assess the separation ability of photoexcited electron–hole ( $e^-h^+$ ) pairs since it directly related to photocatalytic efficacy. The PL spectrum of the as-synthesized pristine CeO<sub>2</sub>, PbWO<sub>4</sub> and CeO<sub>2</sub>/PbWO<sub>4</sub> heterojunction NCs were investigated as shown in Fig. 7. The PL emission intensity of CeO<sub>2</sub>/PbWO<sub>4</sub> nanocomposite was lesser than that of pristine CeO<sub>2</sub> and PbWO<sub>4</sub>, signifying that the coupling of PbWO<sub>4</sub> NPs might reduce the fluorescence from the CeO<sub>2</sub> NPs while the recombination rate of ( $e^-h^+$ ) pairs is seriously reserved and extend the lifetime of charge carriers (Jeyakanthan et al. 2018). It is well established that coupling of PbWO<sub>4</sub> NPs onto the superficial of CeO<sub>2</sub> donated for reduced recombination rate of photoexcited charges related to CeO<sub>2</sub>. This reduction might be accredited to (i) a greater amount of nominal defects and (ii) effectual charge



**Fig. 7** Photoluminescence emission spectra of as-prepared samples

separation of  $\text{CeO}_2/\text{PbWO}_4$  NCs (Koli and Kim 2019). The detected stronger characteristic PL near-band-edge emissions ranges of  $\sim 440\text{--}510$  nm (448, 487 and 503 nm) for the visible region (apparently excitation peak at  $\sim 342$  nm). Normally, the effective charge separation and inhibited ( $e^-h^+$ ) recombination rate by coupling of  $\text{CeO}_2/\text{PbWO}_4$  NCs was auspicious for enhancing the photocatalytic efficacy of  $\text{CeO}_2$  NPs (Lu et al. 2020).

### Photocatalytic activity

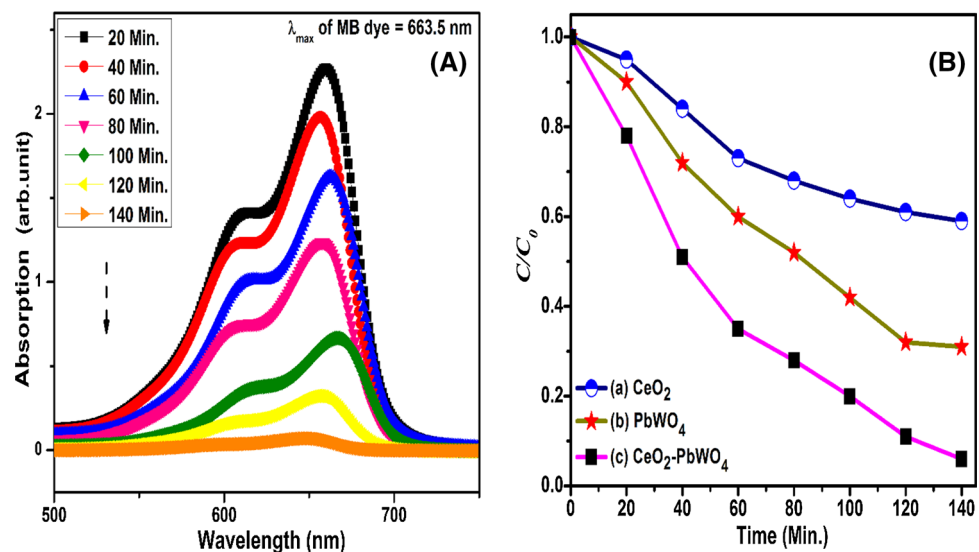
The photocatalytic performance was verified against the deprivation of MB aqueous dye in the existence of as-synthesized  $\text{CeO}_2$ ,  $\text{PbWO}_4$  and  $\text{CeO}_2/\text{PbWO}_4$  PCs under visible-light exposure. It is seen from Fig. 8a, the characteristic

UV–Vis absorption peak of MB dye solution at  $\sim 663.5$  nm has constantly reduced by  $\text{CeO}_2/\text{PbWO}_4$  photocatalyst and hence the supreme degradation efficacy almost 94% was degraded within 140 min. The photocatalytic proficiency of pristine  $\text{CeO}_2$  and  $\text{PbWO}_4$  for MB dye degradation was 42% and 58% in identical exposure time separately. The  $C/C_0$  (where  $C_0$  = absorption intensity of the initial dye solution and  $C$  = main absorption peak intensity of dye) has schemed vs. the wavelength as exposed in Fig. 8b. Related to the blank photodegradation testing of MB dye showed almost no obvious degradation in presence of a catalyst under the dark condition, along with an absence of catalyst and without catalyst in the light source, hence the curves could be neglected (Wen et al. 2018). Mostly, the MB organic dyes could be photodegraded by three conceivable reactions containing photolysis, photosensitization, and photocatalysis also. Largely, degradation efficacy was improved via accumulation of  $\text{CeO}_2/\text{PbWO}_4$  photocatalyst effort might be the outcomes from (i) synergetic influence of the two metal oxides, (ii) leads to the decreases of bandgap energy, (iii) hindrance of the recombination rate of photoexcited ( $e^-h^+$ ) pairs separation amid  $\text{CeO}_2$  and  $\text{PbWO}_4$  heterojunction, (iv) accessibility of surface reactive sites, (v) development of light absorption ability of photoexcited charges generated in the visible-light (Rawat et al. 2007b). The primary absorbance of the peak disappeared entirely after 140 min of visible-light exposure in the  $\text{CeO}_2/\text{PbWO}_4$  photocatalyst which specifies the cleavage of conjugated chromosphere structure of MB dye and exchange into small aromatic intermediary (Rožić et al. 2019).

### Kinetics of MB dye photodegradation

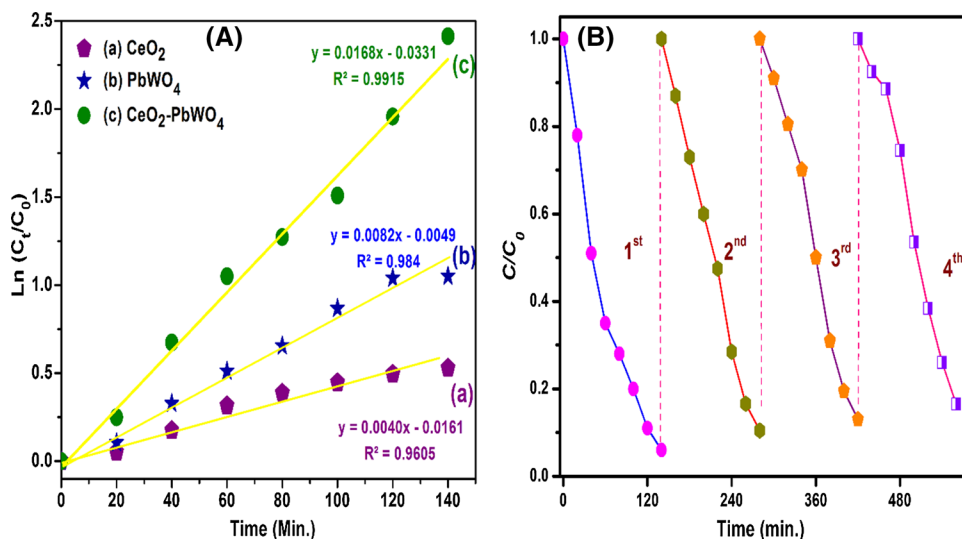
The photocatalytic reaction kinetics of MB dye in visible-light, overall the photocatalysts, was considered by a

**Fig. 8 a** UV–Vis absorption spectra of MB dye solution using  $\text{CeO}_2/\text{PbWO}_4$  NCs under visible-light exposure **b** Photodegradation rate of prepared photocatalysts





**Fig. 9 a** Photodegradation linear plot  $\ln(C_0/C_t)$  versus time of as-prepared photocatalytic samples **b** Reusability performance of  $\text{CeO}_2/\text{PbWO}_4$  photocatalyst

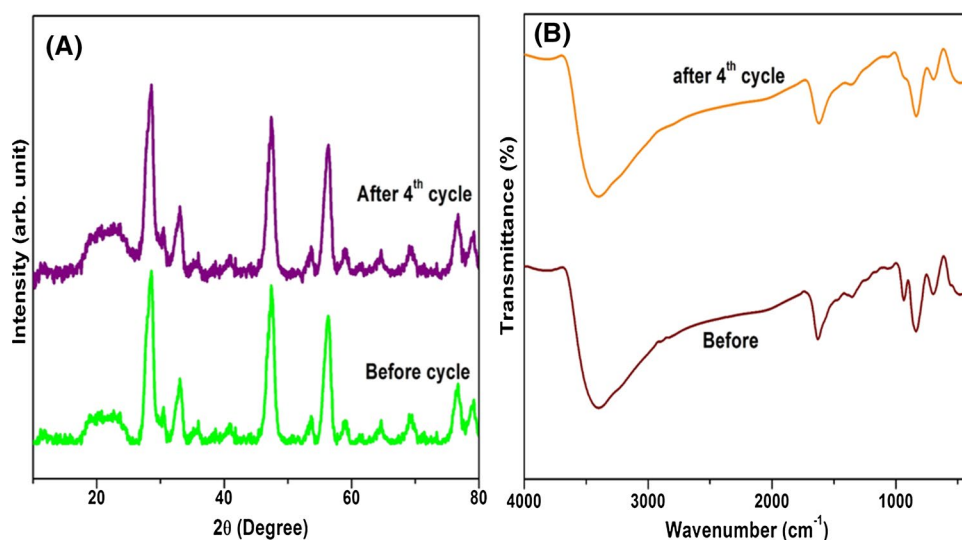


consistent pseudo-first-order kinetic equation of  $\ln C_0/C_t = kt$  using  $\text{CeO}_2$ ,  $\text{PbWO}_4$  and  $\text{CeO}_2/\text{PbWO}_4$  PCs as shown in Fig. 9a. Here,  $k$  refers to a reaction rate constant,  $C_0$  and  $C_t$  are the initial and residual concentrations of MB aqueous dye solution at the agreeing time, and  $t$  (min) was initiate to be linear regression (Yu et al. 2013; Xu et al. 2020). The obvious rate constants for  $\text{CeO}_2$ ,  $\text{PbWO}_4$  and  $\text{CeO}_2/\text{PbWO}_4$  PCs were calculated as 0.0004, 0.0082, and 0.0164  $\text{min}^{-1}$ . The  $k$  value has increased in the order of  $\text{CeO}_2 < \text{PbWO}_4 < \text{CeO}_2/\text{PbWO}_4$ . It has decided that  $\text{CeO}_2/\text{PbWO}_4$  PCs has a greater rate constant and it has 4.2 and 2.1 fold enrichment linked to that of pristine  $\text{CeO}_2$  and  $\text{PbWO}_4$  PCs, separately. Also, the  $\text{CeO}_2/\text{PbWO}_4$  NCs owns expressively high photocatalytic dye degradation effectiveness as compared with related metal oxides and reported in other nanocomposites (Kumar et al. 2013; Wei et al. 2014; Xian et al. 2015; Li et al. 2016; AlShehri et al. 2017; Reddy Yadav et al. 2017).

**Reusability of the photocatalyst**

To further explore the stability and reusability were of great consequence factor for its practical application, and the Fig. 9(b) has exhibits the recycling investigates of chief  $\text{CeO}_2/\text{PbWO}_4$  PCs was repeatedly used (Shanmugam et al. 2019). For both cycles, the  $\text{CeO}_2/\text{PbWO}_4$  PCs were recycled via centrifuging, washing and drying for the next cycling runs. For the  $\text{CeO}_2/\text{PbWO}_4$  PCs, the MB dye photodegradation still existing good stability with only about 5% decreases from the initial activity (94%) during the 4th recycle. The activity loss has which largely might be owed to the photo-corrosion of the catalyst by light in  $\text{CeO}_2/\text{PbWO}_4$  photocatalyst (Yu et al. 2015). As well, the XRD and FTIR analysis of  $\text{CeO}_2/\text{PbWO}_4$  PCs were surveyed further to prove the stability of before and after the photocatalytic replies were carried out. As shown in Fig. 10a, b the XRD and FTIR

**Fig. 10 a** XRD pattern **b** FTIR spectra of before and after catalytic reacted photocatalyst



outcomes might designate that no notable changes are witnessed after 4th recycles, representing the structural stability of the  $\text{CeO}_2/\text{PbWO}_4$  PCs has measured (Saravanakumar et al. 2016). These consequences propose that the  $\text{CeO}_2/\text{PbWO}_4$  PCs used as effective superior photo-stability, and suitable recyclable PCs might also be reused constantly for wastewater treatment under visible-light contact (Vignesh et al. 2019).

### Reactive species study

As well known, to validate the radicals of nanocomposites in the photodegradation progression, the trapping tests of reactive species are executed by utilizing light drive. Photo-excited ( $e^-h^+$ ) pairs thereby form numerous reactive species for instance ( $\% \text{O}_2\bullet^-$ ) and hydroxyl radical ( $\% \bullet\text{OH}^-$ ), hence these reactive species could source in the decomposition of the organic toxins (Negi et al. 2019). Figure 11 as could be seen that photodegradation rate of as-obtained PCs has faintly reductions resultant from the adding of EDTA-2Na, hence validating that  $h^+$  are not a major reactive species in this concerned systems. Likewise, the degradation rate is slightly declined when the addition of IPA, hence the  $\% \bullet\text{OH}^-$  is minor/deliberate part of the allied photocatalytic system. This is because the (VB) valence band potential of  $\text{CeO}_2$  and  $\text{PbWO}_4$  NPs have conjugated into the  $\% \text{OH}/\bullet\text{OH}^-$  also being  $\% \bullet\text{OH}^-$  cannot be fashioned. Though, the addition of BQ initiated significantly suppress the photocatalytic movement from 91 to 26% as presented in the tentative results. Based on the trapping investigation effects, it's resolved that  $\% \text{O}_2\bullet^-$  radicals are the dominant reactive species ( $\text{O}_2 + e^- = \% \text{O}_2\bullet^-$ ) accountable for the decomposition of  $\text{CeO}_2/\text{PbWO}_4$  photocatalytic scheme (Ravishankar et al. 2015).

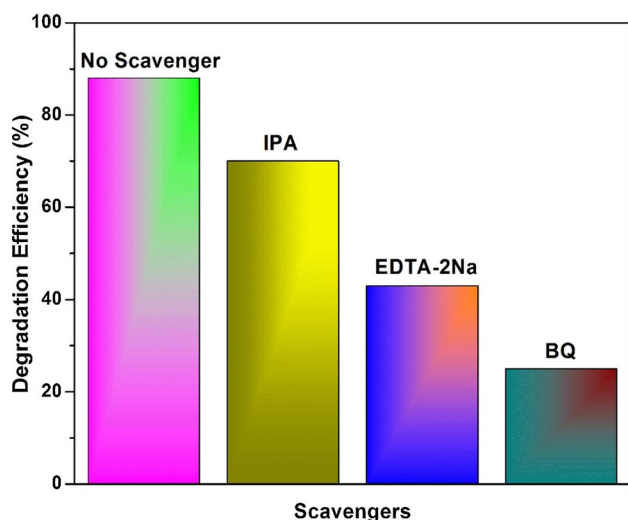


Fig. 11 Effect of scavengers on the MB dye photodegradation process

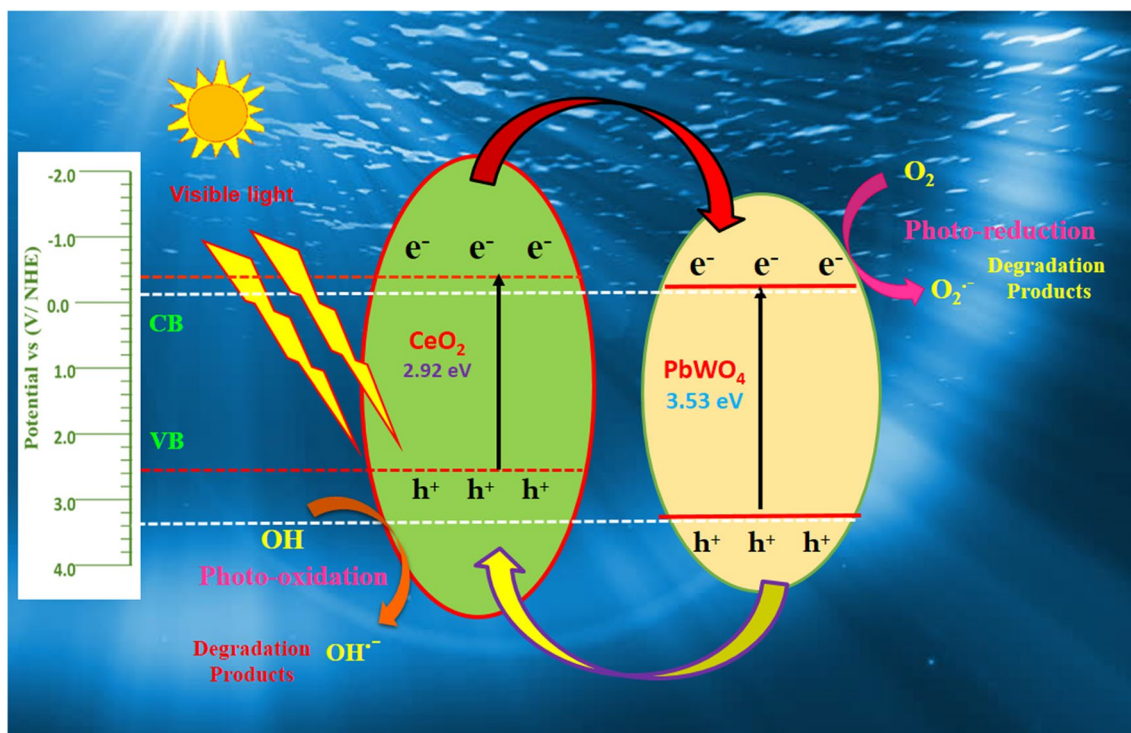
### Possible photocatalytic mechanism

To obtain better consideration on the  $\text{CeO}_2/\text{PbWO}_4$  PCs heterojunction, their valence band (VB) and conduction band (CB) edge potentials were intended via Mulliken electronegativity theory from the Eqs. (1) and (2)

$$E_{VB} = X - E_e + 0.5E_g \quad (1)$$

$$E_{CB} = E_{VB} - E_g \quad (2)$$

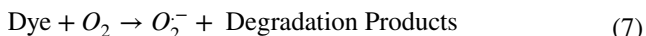
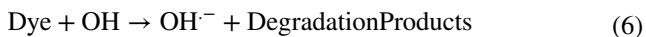
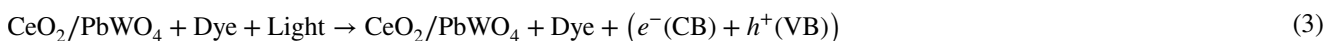
wherever  $\chi$  states the absolute electronegativity of certain semiconductors ( $\chi$  is 5.56, 6.15 eV for  $\text{CeO}_2$  and  $\text{PbWO}_4$ , singly). And the  $E_{VB}$ ,  $E_{CB}$ ,  $E_g$  and  $E_e$  are VB potential, CB potential, estimated optical bandgap of  $\text{CeO}_2$  and  $\text{PbWO}_4$ , and energy of free electrons vs. hydrogen scale (4.5 eV), separately (Channei et al. 2019). Agreeing to the beyond equations, the  $E_{VB}$  and  $E_{CB}$  band edge potential values were assessed to be +2.52 and -0.11 eV for  $\text{CeO}_2$  and as -0.40 and +3.41 eV for  $\text{PbWO}_4$ , separately. From the above-mentioned results, the probable photocatalytic mechanism (Fig. 12) comprises by visible-light which leads to the conception of a photoexcited electron ( $e^-$ ) flowing towards CB of  $\text{CeO}_2$  and thereby the VB of  $\text{PbWO}_4$  acts as a drop for the hole ( $h^+$ ) (Tomova et al. 2015). Thus, the  $e^-$  and  $h^+$  could be proficiently divided; the  $e^-h^+$  pairs are essentially gathered in the CB of  $\text{CeO}_2$  and the VB of  $\text{PbWO}_4$ , individually. It is important to note that Ce element has two valence state ( $\text{Ce}^{4+}$  and  $\text{Ce}^{3+}$ ), and  $\text{Ce}^{4+}/\text{Ce}^{3+}$  combine owns with multiple roles in endorsing the separation of photoexcited  $e^-h^+$  pairs of  $\text{CeO}_2/\text{PbWO}_4$  PCs, by which the photocatalytic performance is enriched (Misra et al. 2012; Priyadharasan et al. 2017b; Wang and Tian 2020). Essentially,  $\text{Ce}^{3+}$  could effortlessly trap the  $\text{O}_2\bullet^-$  making chemical adsorption of oxygen on the exterior of  $\text{CeO}_2$  centered on the  $\text{Ce}^{3+} + \text{O}_2 = \text{Ce}^{4+} - \% \text{O}_2\bullet^-$ , which stimulates photocatalytic oxidation response. Instead of  $\text{Ce}^{3+}$ , which comprises oxygen defects could also absorb visible-light to harvest the photoexcited electron ( $e^-$ ) (Ali Baig et al. 2021). All these gathered electrons ( $e^-$ ) and  $\text{Ce}^{3+}$  own a strong reduction capacity was augmented easily and speedily decrease the absorbed  $\text{O}_2$  on  $\text{CeO}_2$  to cause active superoxide ( $\% \text{O}_2\bullet^-$ ) radicals (Cano-Franco and Álvarez-Láinez 2019). This is because the VB band edge potential of  $\text{CeO}_2$  (+2.52 eV vs. NHE) develops more positive than  $\text{PbWO}_4$  (-0.11 eV vs. NHE), which is beneficial for MB dye deprivation (Fukumura et al. 2017). While electron in the CB supports an  $\text{O}_2$  molecule to form an  $\text{O}_2\bullet^-$  radicals and formerly  $\text{O}_2\bullet^-$  will respond with surface water molecules ( $\text{H}_2\text{O}$ ) to yield the  $\bullet\text{OH}^-$  radical. Lastly, the reactive radicals respond with MB dye molecules decayed to the intermediates or degradation yields over the highly oxidizing species ( $\text{O}_2\bullet^-$ ,  $\bullet\text{OH}^-$  etc.). The appropriate photocatalytic reaction progression (Reddy



**Fig. 12** Possible mechanism for the photoexcited electron–hole separation of the degradation of MB dye by CeO<sub>2</sub>/PbWO<sub>4</sub> photocatalytic interface under visible-light

Yadav et al. 2016; Zhang et al. 2019a) could be also agreed upon by the succeeding Eqs. (3–7)

purity, surface morphology, chemical composition and optical belongings were examined and debated in detail.



**Conclusions**

To sum up, CeO<sub>2</sub>/PbWO<sub>4</sub> heterojunction photocatalyst was effectively formed via a facile and effectual hydrothermal scheme. The CeO<sub>2</sub>/PbWO<sub>4</sub> nanocomposite was characterized by a range of techniques to study its phase

In specific, CeO<sub>2</sub>/PbWO<sub>4</sub> photocatalyst displayed the utmost photodegradation efficacy for 94% of MB aqueous dye within 140 min in visible-light treatment which is greater than other as-obtained samples. The degradation rate constant was closely 4.2 and 2.1 fold greater than those of pristine CeO<sub>2</sub> and PbWO<sub>4</sub> PCs singly. Also, •OH radicals were enabled in the key reactive species in the photodegradation route and the plausible degradation pathways have also been projected. Moreover, the optimized CeO<sub>2</sub>/PbWO<sub>4</sub> photocatalyst presented an outstanding photocatalytic steadiness also reusability upto four consecutive cycles. The developed photocatalytic activities were recognized to the pairing of CeO<sub>2</sub> and PbWO<sub>4</sub> NPs were initiated by superior charge separation/transfer, reduced bandgap, and strong visible-light absorption proficiency thus suppressing the recombination of photoexcited charges. This study promotes the novel CeO<sub>2</sub>/PbWO<sub>4</sub> NCs with outstanding visible-light photocatalytic action

and admirable stability was estimated to inspire potential environmental applications in may near future.

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## Declarations

**Conflict of interest** The authors have declared no conflict of interest.

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