Ti doped BaMnO₃ perovskite structure as photocatalytic agent for the degradation of noxious air and water pollutants

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Abstract

Photocatalytic degradation of noxious water and air impurities like Methylene blue (MB), Rhodamine B (RhB), NO_x, and CO is carried out by pristine and Ti-doped BaMnO₃ (BaMn_{0.85}Ti_{0.15}O_{2.93}, BMT). Perovskite BMT ceramic mixed with equimolar ratio of zeolite is used for investigation of water and air pollutant degradation. The synthesized Ti-doped BaMnO₃ (BaMn_{0.85}Ti_{0.15}O_{2.93}, BMT) perovskite was characterized using X-ray diffraction (XRD) and FE-SEM techniques. The XRD pattern of Ti-doped BaMnO₃ shows the formation of two phases, namely BaMnO_{2.92} and BaMn_{0.85}Ti_{0.15}O_{2.93} and BMT. The photocatalytic degradation of noxious water and air pollutants were analyzed using UV–Vis and XPS for doped and pristine BaMnO₃. From obtained results, it is observed that the synthesized BMT shows enhanced photocatalytic degradation (~99%) as compared to BaMnO₃ toward noxious water impurities like RhB and MB dyes within 270 and 150 min under sunlight. The synthesized BMT compound shows the absorbance spectrum in the wavelength range of 200–600 nm. The UV–Vis DRS confirms the absorption edges for the compound above 400 nm i.e., visible light absorber with a bandgap 2.48 eV. On the other hand, XPS analysis of bulk BMT-zeolite on exposure to sunlight shows the complete degradation of adsorbed NO_x gases from the BMT-zeolite surface as compared to the unexposed zeolite sample surface. Therefore, BMT can be used as active photocatalytic agent for the remediation of environmental noxious water and air pollutants.

Keywords Ti-doped BMT · Zeolite · Methylene blue · Rhodamine B · NO_x · CO · Photocatalyst · X-ray photoelectron spectroscopy

1 Introduction

India being a developing nation and fast-growing economy, revolutionizing its infrastructure in the form of high rise buildings, residential colonization, and industrialization, etc. This rapidly changing infrastructure leads to the emission/release of several air and water pollutants into the environment. Air pollutants, like NO_x, SO_x, and CO were emitted in the environment from vehicular traffic,

possess severe health hazards. While water pollutants i.e., metal ions, dyes, pesticides PAHs, etc. when released in the aquatic ecosystem gets accumulated in the food chain and hampers the ecological balance [1].

In terms of air pollutants, NO_x gases are one of the significant contributors to air pollution. The annual average concentrations of NO_x gases globally are in the range of 20–90 ppb but can exceed up to 1 ppm or higher level due to excessive use of fuel-based transport carriers. The

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high level of NO_x gases leads to the greenhouse effect, ozone layer depletion, smog, and overall ecological imbalance. As per the Central Pollution Control Board (CPCB), New Delhi, India, the concentration of NO_2 and particulate matter (PM_{10}) in the air have exceeded their prescribed limits [9, 35]. These pollutants not only alter the ecological balance but also degrades the indoor air quality (IAQ) associated with building structures in urban areas [21]. As a result, several health issues like pulmonary and respiratory disorders were observed, and approximately 500,000 cases were reported every year in India [31].

In terms of water pollutants, inorganic impurities including metal ions [15, 19, 36] and organic impurities like toxic dyes [3, 13] are the major contributors of water pollution. The effluents of various textiles and dye industries are rich in colored dyes content, and upon entering the aquatic ecosystem, it deteriorates the water quality. Noxious dyes like Rhodamine B (RhB) and methylene blue (MB) were widely used in various industries for coloring purposes [22], these dyes on releasing in water forms complexes with different metal ions, which in turn accumulates in the food web [16].

Keeping in view the above toxic pollutants, a photocatalytic material that led to the degradation of air as well as water pollutant is the need of today, and it is one of the costs effective approach to minimize the effect of air and harmful water-borne pollutants on human health [37, 38]. TiO_2 (nanoparticles) is one of the most widely and extensively studied photocatalyst because of its chemical stability, non-toxicity [5, 34]. Perovskite, ceramic oxide catalysts with structural formula ABO_3 (A being divalent cation and B is tetravalent cation) discovered in the 1970s, was used for the NO and CO oxidation and since then have been a subject of extensive study [14, 17, 18, 30, 32]. Kim and co-workers used Lanthanum manganese oxides with Pt-based catalyst to carry out the photocatalytic degradation of NO [20]. As of now, perovskite-based catalyst are gaining more attention in recent times, and they are currently being used as active photocatalytic agent individually for the degradation of noxious impurities like NO_x and soot removal from automotive emissions and dyes removal from aqueous system [6, 39].

Although a lot of efforts have been made on the photocatalytic degradation of air pollutants and water pollutants individually, several researchers have studied detailed mechanisms of degradation of dyes from the textile industry, such as acid green (AG) and formic acid in the presence of photocatalyst viz. Co-ZSM-5 and Fe/TiO_2 , respectively [2, 4]. These type of studies comes under organic–inorganic compound systems. The other applications of organic–inorganic composites include optical devices, electronic conductors, coatings, mechanically robust filters. The

composites provide chemical and thermal stability to the systems [25–29].

In the present work, we have synthesized the Ti-doped BaMnO_3 perovskite ceramic mixed with equimolar ratio of zeolite material i.e., $\text{BaMn}_{0.85}\text{Ti}_{0.15}\text{O}_{2.93}$ (BMT) which has the potential for the removal of air pollutants like NO_x , CO and water pollutants like Methylene blue (MB) and Rhodamine B (RhB) together.

2 Materials and methods

2.1 Experimental

The zeolite for the adsorption of toxic gases has been synthesised from class F fly ash consisting mainly of alumina, silica, and low calcium content and NaOH in 1:1.2 ratio. Synthesis of zeolite was done in two steps using the solid-state route. Firstly, 10 g of fly ash was thoroughly mixed with 12 g of NaOH in an agate mortar and pestle. The obtained powder mixture was heated at 600 °C for 4 h. Post heat treatment, the color of the fused mixture turned from grey to light green. The fused mixture was cooled to room temperature, and again ball milled to obtain fine powder. The powder mixture was dissolved in 100 ml of distilled water and stirred at room temperature for 8 h. The mixture was then heated at 100 °C for 12 h in an oven followed by normal cooling up to room temperature. The obtained mixture was then filtered and washed with distilled water in order to remove the excess NaOH. Finally, it was dried at 80 °C overnight for obtaining the zeolite powder.

The synthesis of Ti 0.15 mol fraction doped BaMnO_3 was done by solid-state reaction route. The oxide powder of BaCO_3 (Alfa Aesar, purity > 99.5%), MnO_2 (Alfa Aesar, purity > 99%) and TiO_2 (Alfa Aesar, purity > 99%) were used as precursor materials. The stoichiometric amounts of precursor materials were mixed thoroughly in an agate mortar and pestle using acetone as medium to ensure uniform mixing. Then, the powder mixture was calcined at 920 °C in a muffle furnace for 6 h in air atmosphere. The synthesized sample BMT and BM samples were tested with MB and RhB dye in the presence of sunlight and for photocatalytic degradation activity.

The equimolar amounts of zeolite and Ti 0.15 mol fraction doped BaMnO_3 powder were mixed and compacted into cylindrical pellets of diameter 10 mm using a hydraulic press. Zeolite pellets of 10 mm diameter were also prepared for NO_x adsorption study. Finally, the catalyst mixed with zeolite pellets were exposed to ambient air pollution and Bharat Stage (BS-IV) two wheeler emissions whose norms are described by Central Pollution Control Board (CPCB) New Delhi, India, mentioned elsewhere [10].

The total time of exposure to zeolite-BMT samples was 1 h for NO_x , SO_x , CO and other pollutants for adsorption. The sample was kept in sunlight for 6 h for photocatalytic characterization.

2.2 Characterization techniques

The x-ray diffraction study (XRD) of the Ti-doped BaMnO_3 was done by Rigaku X-ray diffractometer (Rigaku, ULTIMA IV) equipped with Cu K α (1.5418 Å) radiation source and nickel K β filter. The XRD data for BMT was collected in the 2θ range of 10° – 60° with a scan rate of $4^\circ/\text{min}$. The microstructures of the sample were analyzed using field emission scanning electron microscope (FE-SEM) (MIRA3 TESCAN) at an accelerating voltage of 10 kV.

For the confirmation of photocatalytic activity of the prepared $\text{BaMn}_{0.85}\text{Ti}_{0.15}\text{O}_{2.93}$ (BMT) and BaMnO_3 (BM) compound, the UV–visible diffuse reflectance spectra (DRS) of the prepared photocatalyst were recorded using a Shimadzu UV-2450 UV–Vis spectrophotometer over the wavelength range of 200–800 nm. BaSO_4 was used as a reference material for baseline correction.

2.3 Photocatalytic degradation studies

The photocatalytic study of BMT was investigated using 5 ppm of RhB and MB dyes as model pollutant for dye degradation method. Natural sunlight was used as a radiation source for the photocatalytic reaction. In this test, 0.1 g of the catalyst was added in 100 ml of dye solution. Prior to irradiation, the catalyst-dye suspensions were magnetically stirred for 1 h in the dark to ensure the establishment of adsorption–desorption equilibrium between the catalyst and dye molecules. For monitoring the dye degradation with time, an aliquot (~ 3 ml) from the dye-catalyst suspension was periodically sampled and centrifuged at 8200 rpm for absorbance measurements on a Shimadzu 2450 UV–visible spectrophotometer. A blank experiment was always performed on the dye solutions in the absence of the catalyst under identical experimental conditions.

The following expression has been used for calculating the degradation efficiency.

$$\% \text{ of degradation} = (1 - C/C_0) \times 100$$

where, C_0 and C are the initial dye concentration and concentration at different intervals of time, respectively.

The chemical state of zeolite constituents was analysed by X-ray photoelectron spectroscopy (XPS) (PHI Versa Probe 5000 II (ULVAC- PHI). A set of survey and core level spectra were recorded under ultra-high vacuum (UHV) conditions. Al K α anode (1486.6 eV) was used as an X-ray generation source. For a broad survey spectrum, the pass

energy of the analyser was kept at 187.5 eV whereas, the core level the spectra were acquired at pass energy of 23.75 eV to achieve better resolution. The C1s spectrum reference at 284.5 eV was used for peak shift correction of other constituent elements [23]. In the curve-fitting procedure both Gauss and Lorentz type of function was used to obtain a better fit, and the background treatment was done by Shirley function to the original data. MULTIPAK (PHI Inc., USA) software has been used for de-convolution and spectra analysis.

3 Results and discussion

3.1 Characterization studies

The X-ray diffraction pattern of 0.15 mol fraction Ti doped BaMnO_3 obtained at room temperature is shown in Fig. 1. The formation of the desired Ti doped BaMnO_3 along with some evidence of additional phases is evident from Fig. 1. The obtained XRD pattern was analysed and indexed using X'pert High score plus software and PDF 2 database, respectively. The peak analysis shows the existence of $\text{BaMn}_{0.85}\text{Ti}_{0.15}\text{O}_{2.93}$ (ICSD file no. 98-016-9113) and $\text{BaMnO}_{2.92}$ (ICSD file no. 98-017-3245). Both $\text{BaMn}_{0.85}\text{Ti}_{0.15}\text{O}_{2.93}$ and $\text{BaMnO}_{2.92}$ have hexagonal crystal structure. The XRD and other studies of the synthesised zeolite used for remediation of noxious pollutants have been mentioned elsewhere [12, 33]. Figure 2 shows the XRD pattern for synthesised $\text{BaMnO}_{2.98}$. From the indexed XRD pattern, it is clearly evident that the formation of additional phases along with $\text{BaMnO}_{2.98}$ (ICSD file no. 98-001-9048) has appeared viz. BaO (ICSD file no. 98-017-3922), MnO_2 (ICSD file no. 98-007-3716) and $\text{Ba}_2\text{Mn}_8\text{O}_{16}$ (ICSD file no. 98-006-2096).

Fig. 1 XRD pattern of synthesised Ti doped BaMnO_3

Fig. 2 XRD pattern of BaMnO_3 along with the formation of additional phases

The FE-SEM images of synthesised BMT composition is shown in Fig. 3. The microstructure clearly reveals the formation of flake-like nature of the grains with the particle size in the range of 1–2 μm lengths. Whereas, the synthesised zeolite for the adsorption of toxic air pollutants shows different morphological features and is described elsewhere [33].

The optical absorption spectra of the BMT is shown in Fig. 4a. The UV–Vis DRS shows the absorption edges for the compound above 400 nm ($\lambda_{\text{edges}} > 400 \text{ nm}$), which confirms the absorption in the visible region. The exact bandgap energy of these types of compounds is calculated from the Tauc plots as shown in Fig. 4b. The best fit for $(ah\nu)^n$ versus $h\nu$ was obtained for $n=2$, which suggests a direct bandgap transition. The calculated bandgap energy of the compound is 2.48 eV, conforming that the synthesized BMT compound can act as a photocatalytic agent.

3.2 Photocatalytic degradation of water pollutants

The synthesised photocatalytic agent i.e. BMT is tested for the photodegradation of organic dye pollutants i.e. Methylene blue (MB) and Rhodamine B (RhB) under natural sunlight-irradiation. To check the photocatalytic activity of the synthesised compound, 10 ml of dye solution with corresponding 10 mg of BMT is added in the solution and kept in sunlight to observe the color change of the MB and RhB dye. The gradual degradation of noxious dyes with respect to time is shown in Figs. 5a–f and 6a–f. The complete photocatalytic degradation of MB and RhB dyes takes within 270 min and 150 min, respectively. On the other hand, when BaMnO_3 (BM) is used as photocatalytic agent, then the only 20% degradation of RhB takes place in 150 min, and 40% degradation of MB dye takes place in 270 min. The results of RhB and MB dye tested on BaMnO_3 (BM) and Ti 0.15 mol fraction doped BaMnO_3 (BMT) has been plotted in Fig. 7a–d. To determine the photo stability of RhB and MB, blank tests (without catalyst) were carried out under identical experimental conditions, and results showed that there was no self-degradation of dyes.

The photocatalytic reaction rate constant for degradation of RhB and MB with BMT was represented by Langmuir–Hinshelwood equation:

$$\ln(C_0/C) = kt \quad (1)$$

where, C_0 is the initial RhB/MB concentration, C is the RhB/MB concentration at irradiation time t , and k is the rate constant for the degradation of RhB/MB dye. Figure 7a, b show a linear plot between $\ln(C_0/C)$ and irradiation time for BM and BMT, indicating that the photocatalytic degradation of RhB and MB follows pseudo-first-order kinetics. The degradation rate constants are calculated to be 0.0214 min^{-1} (with BMT) and 0.0014 min^{-1} (with BM) for MB and 0.0336 min^{-1} (with BMT) and 0.0009 min^{-1} (with

Fig. 3 FE-SEM images synthesised BMT compound

Fig. 4 UV-Vis DRS of **a** synthesized BMT and **b** corresponding Tauc plot

BM) for RhB dye respectively. A blank test has also been carried out on MB and RhB dye in a similar environment as reference.

The primary underlying mechanism for degradation of pollutant dyes may be explained as follows. When the incident photons interact with the photocatalyst (BMT in the present study), the electrons lying in the valance band (VB) are excited to the conduction band (CB). This leads to the creation of holes in the valance band. The excited electrons and hole may either recombine with each other or diffuse to the BMT (photocatalyst) surface, thus reacting with the adsorbed pollutant (MB/RhB) molecules. The photosensitisation of the dye molecules can provide photocatalyst (BMT) with additional electrons which are capable of creating radicals viz $\cdot\text{O}^{2-}$. These reactive radicals can effectively decompose organic pollutants. The whole photodegradation process depends upon various parameters viz. pH of the solution, initial dye concentration, reaction temperature, and irradiation intensity of the light [7].

Fig. 5 Photocatalytic degradation of methylene blue (MB) dye using synthesized BMT under natural sunlight

Fig. 6 Photocatalytic degradation of Rhodamine B (RhB) dye using BMT under natural sunlight

3.3 XPS analysis of photocatalytic degradation of air pollutants

XPS analysis is carried out in order to confirm the adsorption and degradation of NO_x and other gases on

Fig. 7 **a** Log plot of photocatalytic degradation of MB dye by BM and BMT as a function of time. **b** Log plot of photocatalytic degradation of RhB dye by BM and BMT as a function of time. **c** Photo-

catalytic degradation of MB for BM and BMT compound and **d** photocatalytic degradation of RhB dye for BM and BMT compound

zeolite-BMT surface. The results obtained is shown in Fig. 8a–d. For detailed analysis, the core level spectra of C1s, N1s, and O1s were plotted in Fig. 8a, b, d and analysed using MULTIPAK software.

For BMT-zeolite composition, the N 1s peak is analyzed as shown in Fig. 8b. After the deconvolution, the presence of NO and N₂O is confirmed [8]. The absence of NO₂ peak in the N1s spectrum confirms the photocatalytic activity of BMT. The adsorption analysis of NO_x and other air pollutants on zeolite surface has been done in detail and mentioned elsewhere [8, 11]. The various binding energy peak values and the quantitative estimation of respective pollutants for BMT added zeolite have been listed in Table 1. Figure 8c shows the survey spectrum for BMT composition for elemental identification. Among the three nitrogen oxides, N₂O is responsible for the greenhouse effect, NO and NO₂ are major air pollutants that may cause serious health issues if exposed for a large continuous period of time. Similarly, the presence of carbon monoxide (CO)

has been confirmed by the deconvolution of C1s spectrum shown in Fig. 8a [24]. The other peaks, such as C–C and carbonates (–CO₃–) have also been confirmed in the C 1s spectrum. The presence of SiO₂, carbonates, and other metal oxides, which are part of zeolite composition, have been shown in O 1s peak in Fig. 8d. The presence of CO has also been confirmed in O 1s spectrum.

A generalised proposed mechanism for degradation of NO₂ gas as air pollutant may be explained by the following equations:

Fig. 8 Core level spectra of **a** C1s, **b** N1s, **c** Su1s and **d** O1s. The de-convolution of respective spectrum shows the presence of NO_x, CO, and other pollutants adsorbed and effect of BMT photocatalyst in NO₂ degradation as evident in N1s spectrum

Table 1 Semi-quantitative analysis for NO_x and other air pollutants adsorption on BMT added zeolite

Compound/pol- lutants	Binding energy peaks (eV)	(At%) (BMT- zeolite)
NO	401.02 (present work, [8])	23.21
NO ₂	405.3 (present work, [8])	–
N ₂ O	399.3 (present work, [8])	76.79
CO	285.8 (present work, [24])	14.73
–C–N–	287.17 ([23])	9.40
–CO ₃ –	289.03 ([23])	18.89

Oxidation using hydroxyl radicals : OH[•]

The by-products formed are non-toxic and environmentally acceptable in the form of calcium nitrates [11].

4 Conclusion

Ti 0.15 mol fraction doped BaMnO₃ perovskite ceramic mixed with equimolar ratio of zeolite material is synthesized and investigated as photocatalytic agent for the remediation of noxious environmental pollutants like MB, RhB and NO_x CO etc. BMT when applied as photocatalyst for the photodegradation of organic dyes i.e. MB and RhB, results in 100% degradation in 270 and 150 min, respectively. On the other hand, XPS analysis has revealed the adsorption of NO, CO, N₂O, and degradation of NO₂ on the zeolite-BMT surface under sunlight radiation. The quantitative study of BMT-zeolite composition confirmed the adsorption N₂O (76.79 at%), NO (23.21 at%), and CO (14.73 at%). Apart from sulfur and NO₂ (0 at% due to complete degradation), all other CO and NO_x related compounds were analyzed. Hence, the bulk BaMn_{0.85}Ti_{0.15}O_{2.93} with zeolite can act as a potential photocatalyst material for remediation of noxious impurities like MB, RhB, and NO_x from wastewater and air, respectively.

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Authors' contribution SS and IT were responsible for the proper implementation of the experimental design. SS, HR, RSB, and SKP carried out the effective parameter studies for noxious air pollutants and its data interpretation while IT, SM, and SR carried out the effective parameter studies of noxious water pollutants and its data interpretation. SS wrote the initial manuscript while other authors help him in the final editing and formatting of the manuscript.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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