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Magnetic CeO₂/SrFe₁₂O₁₉ Nanocomposite: Synthesis, Characterization and Photocatalytic Degradation of Methyl Orange

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Abstract

Magnetic nanocomposite CeO₂/SrFe₁₂O₁₉ including a hard magnetic material (SrFe₁₂O₁₉) and soft magnetic material (CeO₂) was prepared by a one-step chemical co-precipitation with high-temperature (900 °C) sintering method. Its structure and properties were studied using Fourier transform infrared (FT-IR), X-ray diffraction (XRD), vibrating-sample magnetometry (VSM), scanning electron microscope (SEM), energy-dispersive X-ray spectroscopy (EDS) and map analysis (MA). The testing results showed that the structure and phase of SrFe₁₂O₁₉ did not change by growth CeO₂ nanoparticles. SEM image and map analysis indicated that SrFe₁₂O₁₉ was distributed between the CeO₂ nanoparticles. Nanocomposite CeO₂/SrFe₁₂O₁₉ showed the quite high magnetic performance of 39.34 emu/g, indicating that the properties of CeO₂/SrFe₁₂O₁₉ were favorable to its separation, recycling and used after photocatalytic process without secondary pollution. In addition, the photocatalytic activity CeO₂/SrFe₁₂O₁₉ for the degradation of methyl orange (MO) was explored under UV light and results revealed the superior photocatalytic performance of MO dye with removal percentage of 88.38% within 90 min.

Keywords CeO₂/SrFe₁₂O₁₉ nanocomposite · Co-precipitation · Photocatalytic activity · Degradation

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1 Introduction

Recently, the environmental pollution due to the rapid growth of different organic pollutions [1–3] of industrial activities has become a prominent issue with a detrimental influence on human life [4–6]. The organic pollutions are stable, non-biodegradable, toxic, carcinogenic and also lead to generation of dangerous by-products using different chemical reactions such as oxidation and hydrolysis [7, 8]. Among the various organic pollutions, the organic dyes such as Congo red [9], methyl orange [8, 10], methylene blue [11], rhodamine B [12] and rhodamine 6G [13] are promising and must be removed from wastewater before discharge to environment. Until now, different techniques such as physical, chemical and biological [14–16] were used to different dyes treatment. Among them, photocatalytic method has attracted more attention because of its low price, non-toxicity and high efficiency [17–19]. Nanostructured metal oxides such as CeO₂ [20], Fe₂O₃ [21] and TiO₂ [22] and their binary nanocomposites such as Bi₂O₃-Al₂O₃ [23], CeO₂-Bi₂WO₆ [24], CeO₂-TiO₂ [25] and TiO₂-ZnO [26] have attracted



much research interest and used in photocatalytic degradation of different organic dyes. Separation of catalyst from aqueous solution is difficult, then probably produced secondary pollution and also increased costs. To overcome these problems, usage of magnetic catalyst was proposed because of simple separation of them by an external magnet [23–26].

Hexaferrite strontium ferrite ($\text{SrFe}_{12}\text{O}_{19}$), as hard magnetic materials, has excellent properties such as large saturation magnetization, good chemical stability, superior coercivity and high uniaxial magnetic crystalline anisotropy [27–31]. In recent years, various binary $\text{SrFe}_{12}\text{O}_{19}$ -based nanocomposites, such as $\text{ZnFe}_2\text{O}_4/\text{SrFe}_{12}\text{O}_{19}$ [32], $\beta\text{-Bi}_2\text{O}_3/\text{SrFe}_{12}\text{O}_{19}$ [33], $\text{Bi}_3\text{O}_4\text{Cl}/\text{SrFe}_{12}\text{O}_{19}$ [34], $\text{SrFe}_{12}\text{O}_{19}@\text{Fe}_3\text{O}_4$ [35], $\text{CoFe}_2\text{O}_4/\text{SrFe}_{12}\text{O}_{19}$ [36–38], $\text{MoS}_2\text{-SrFe}_{12}\text{O}_{19}$ [39] and $\text{BiOCl-SrFe}_{12}\text{O}_{19}$ [40], were synthesized and used in photocatalytic degradation of different organic dyes under light irradiation [32–34].

To the best of our knowledge, different magnetic composites based $\text{SrFe}_{12}\text{O}_{19}$ were synthesized using sol–gel [32, 38], hydrothermal [34, 35, 37], chemical co-precipitation [36] and characterized. But, there is less reported to binary and ternary composites-based $\text{SrFe}_{12}\text{O}_{19}$ with rare earth transition metal oxide like CeO_2 [18] and Bi_2O_3 [12, 33]. Therefore, in this paper, the $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ binary composite magnetic photocatalyst was prepared by one-step chemical co-precipitation with high-temperature sintering method, characterized and the photocatalytic activity of was evaluated by methyl orange (MO) photodegradation.

2 Experimental

2.1 Materials and Characterization

All the chemical and reagents used in this paper were of high purity and were purchased from Merck and Sigma and used without further purification. FT-IR spectra were recorded on a 5DX FTIR spectrometer (Nicolet Co., USA), using KBr pellets in the range of $4000\text{--}400\text{ cm}^{-1}$. The crystal structure and phases were identified via X-ray powder diffraction (XRD) using a Bruker Advance D8 diffractometer ($\text{Cu K}\alpha$ radiation, $\lambda = 1.54056\text{ \AA}$). The magnetic properties were investigated using a vibrating sample magnetometer (VSM, Meghnatis Daghig Kavir Co., Iran) with applied magnetic field of up to 14 KOe. The morphologies of samples were recorded by scanning electron microscopy (SEM, JEOL-JSM 7600 F).

2.2 Synthesis of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ Nanocomposite

The $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ nanocomposite was synthesized by one-step chemical co-precipitation with high-temperature sintering method. First mixed $\text{Sr}(\text{NO}_3)_2$ (0.211 g), $\text{Ce}(\text{NO}_3)_2 \cdot 9\text{H}_2\text{O}$ (0.868 g) and $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (4.02 g)

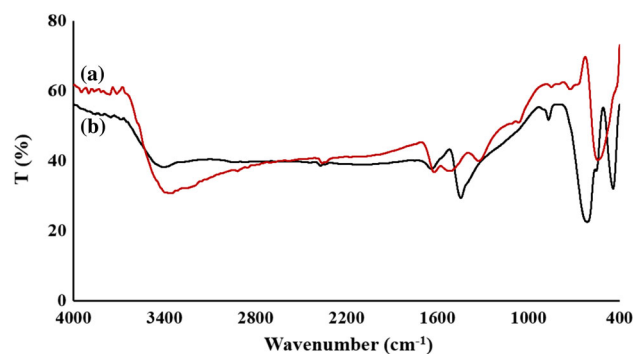


Fig. 1 FT-IR spectra of a CeO_2 and b $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ nanocomposite

with molar ratio of 1:2:10 together. Then added the 50 mL distilled water and stirred for 20 min. After that, under vigorous stirring, the pH value of the solution is achieved to 12 by adding 0.5 M NaOH and the suspension was stirred at $70\text{ }^\circ\text{C}$ for 24 h. Subsequently, the intermediate brown precipitates could be obtained using filtration, washing and drying. Then, the intermediate brown powder was ground by mortar in ceramic crucible and put into a muffle furnace, which was annealed at $900\text{ }^\circ\text{C}$ for 3 h. The black powders washed, dried and characterized.

2.3 Photocatalytic Degradation of Methylene Blue

The photocatalytic activity of the $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ was studied by the degradation of MO under UV light irradiation of a 300 W Xe lamp at the natural pH value. A 50 mL of 20 mg/L MO aqueous solution and suitable amount of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ (0.01 or 0.02 g) were added into container and stirred for 1 h in the dark. After that, the solution was irradiated by UV light. After a given time, about 4 mL of the mixture was withdrawn and the catalyst was separated using an external magnet. Then, the absorption of solution was monitored with a UV–Vis spectrophotometer. The photocatalytic experiments were conducted three times with statistically similar results each time. The degradation rate of MO was calculated by the following equation, where C_o and C_t represent the initial and different reaction time absorbance of MO.

$$R(\%) = \{(C_o - C_t) \times 100\} / C_o \quad (1)$$

3 Results and Discussions

3.1 FT-IR

The FT-IR spectrum of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ is shown in Fig. 1. It can be seen that the metal oxygen (Fe–O, Sr–O and Ce–O)

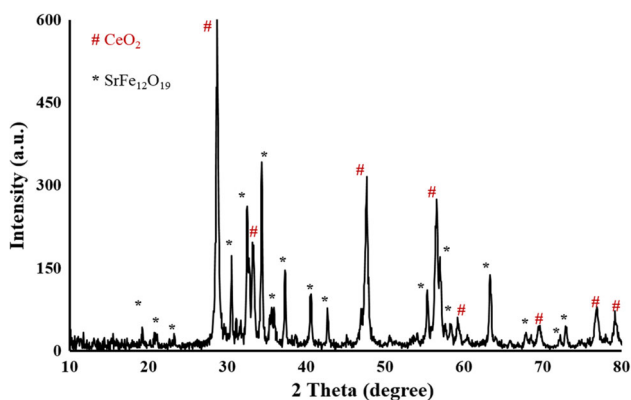


Fig. 2 XRD pattern of CeO₂/SrFe₁₂O₁₉ nanocomposite

stretching vibration frequencies of SrFe₁₂O₁₉ and CeO₂ appeared at 442 and 612 cm⁻¹ [11, 21]. A peak appeared at 867 cm⁻¹ confirms Ce–O stretching vibration [21, 41]. The peak observed at 1446 cm⁻¹ suggests that the existence of stretching vibration of NO₃⁻ ion [21, 41] may be ascribed to the remained of metal nitrate precursors. In addition, two broad peaks appeared at 1639 and 3420 cm⁻¹ confirm the O–H bending and stretching vibration of water molecules adsorbed on the surface of CeO₂/SrFe₁₂O₁₉ nanocomposite [28, 32, 33].

3.2 XRD Pattern

Figure 2 illustrates the XRD pattern of CeO₂/SrFe₁₂O₁₉ nanocomposite. The characteristic peaks of SrFe₁₂O₁₉ were observed at 2θ values of 30.1, 32.1, 34.3, 36.5, 37.3, 41.2, 43.5, 55.7, 56.9, 63.2, 67.8 and 72.9° corresponds to (110), (107), (114), (201), (108), (205), (206), (217), (218), (220), (2014) and (317) crystallographic planes are precisely well matched to the pure hexagonal SrFe₁₂O₁₉ structure with JCPDS card no: 24–1207 [29, 32]. Also, the characteristic

peaks of cubic CeO₂ nanoparticles were observed at 2θ values of 28.8, 33.1, 37.6, 56.7, 59.5, 69.9, 76.9 and 79.2° corresponding to characteristic (111), (200), (220), (311), (222), (400), (331), and (420) crystallographic plane well matched to the pure CeO₂ nanoparticles with JCPDS card no 34–0394 [21, 34, 42].

The average crystallite size of CeO₂ and SrFe₁₂O₁₉ nanoparticles was calculated using Debye–Scherrer formula based on the high intensity peaks appearing at 2θ value of 28.8° for CeO₂ and 34.3° for SrFe₁₂O₁₉ and was measured as 43.52 and 44.13 nm, respectively, which also agree well with the nanoparticle size shown in the SEM images (Fig. 3).

3.3 SEM Images

The morphology of CeO₂/SrFe₁₂O₁₉ nanocomposite is observed by using SEM images as represented in Fig. 3. The images confirm a spherical shape with a rough surface. The average particle size was distributed in the range of 25–50 nm. The molecular mass of SrFe₁₂O₁₉ was higher than that of CeO₂, confirms the SrFe₁₂O₁₉ particles absorbed more electron than CeO₂ particles and causes that the low molecular mass material (CeO₂) displayed more bright. Therefore, the SrFe₁₂O₁₉ regions were possibly dark [32].

3.4 EDX/elemental Mapping

Elemental analytical data (Sr, Ce, Fe and O) and distribution of them on CeO₂/SrFe₁₂O₁₉ nanocomposite were obtained by EDX/elemental mapping as represented in Figs. 4 and 5, respectively. Analysis was conducted at many different points and the atom percentage of each element is shown in the inset of Fig. 4. As seen in Fig. 5, the distribution of all atoms in CeO₂/SrFe₁₂O₁₉ nanocomposite is almost homogeneity and confirms the preparation of core–shell CeO₂/SrFe₁₂O₁₉ nanocomposite.

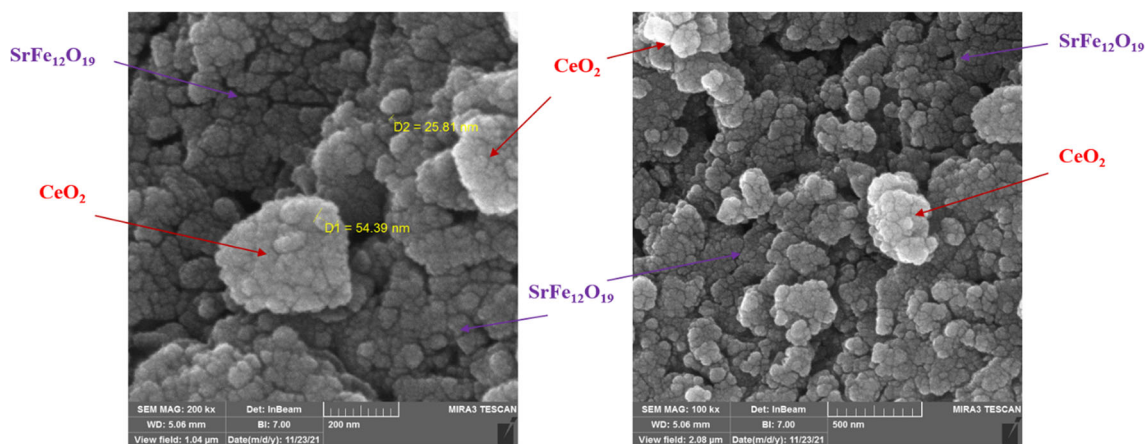
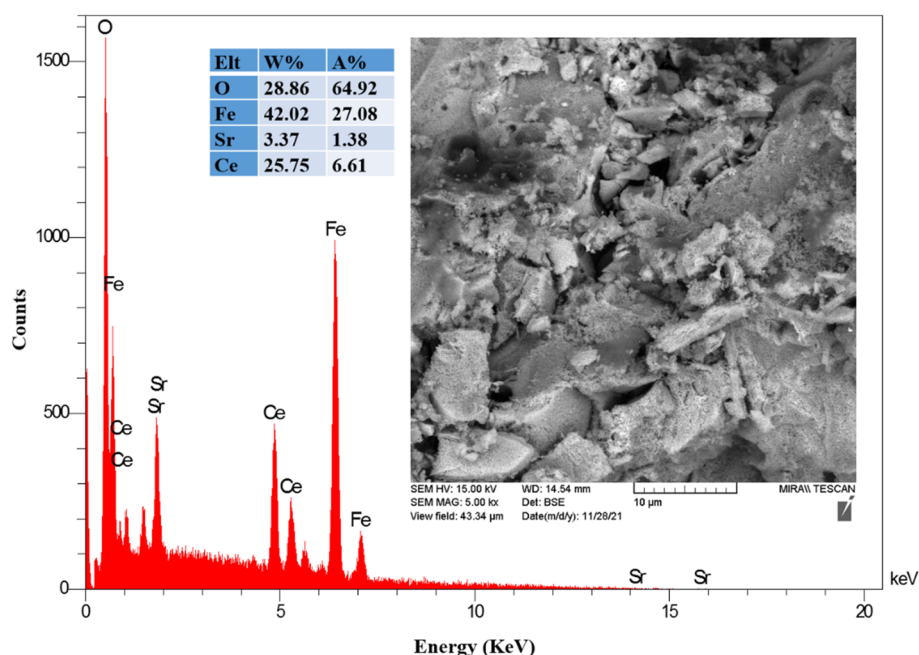


Fig. 3 SEM images of CeO₂/SrFe₁₂O₁₉ nanocomposite

Fig. 4 EDS spectrum of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ nanocomposite



3.5 VSM

The magnetic hysteresis loop of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ nanocomposite is represented in Fig. 6. As shown in Fig. 6, the hysteresis loop area of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ nanocomposite is wide with saturation magnetization (M_s) of 39.34 emu/g, and a remnant magnetization (M_r) of 21.5 emu/g indicates that the sample is a kind of hard-magnetic materials [12, 32, 33]. The coercivity (H_c) of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ nanocomposite is 5384 Oe, indicating that it possesses a good anti-demagnetization ability, which was beneficial to its recycling from solution after used as photocatalyst using external magnet and also reused it again in photodegradation of different organic dyes such as methylene blue [32, 33] and rhodamine B [12, 34].

3.6 Photocatalytic Activity

Methyl orange is an aromatic anionic dye which is stable, toxic and colorant and causes serious environmental problems [43, 44]. Then, removal of MO dye is a major environmental issue before discharging to receiving water using different route such as adsorption [45–47] and photocatalytic degradation [10, 20, 48]. Recently, photocatalytic degradation of MO has been applied by different compounds such as transition metal oxides Fe_2O_3 [20, 21, 30], ZnO [48] and TiO_2 [22], binary heterojunction $\text{Bi}_2\text{O}_3\text{-Al}_2\text{O}_3$ [23], $\text{AgBr/g-C}_3\text{N}_4$ [49] and TiO_2/ZnO [26], strontium hexaferrite [28], ternary heterojunction $\text{Ag/TiO}_2/\text{biochar}$ [10], attapulgite- $\text{SnO}_2\text{-TiO}_2$ [50] $\text{AgBr@Ag}_3\text{PO}_4/\text{Fe}_3\text{O}_4$ [51] and

PEDOT/NiO/Fly ash [52], $\text{CdS/CeO}_2/\text{Ag}_3\text{PO}_4$ [53] and also quaternary heterojunction $\text{PANI-CdS/CeO}_2/\text{Ag}_3\text{PO}_4$ [54].

The photocatalytic property of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ nanocomposite on photodegradation of MO under UV light was analyzed by monitoring of UV-Vis spectra of MO solution (Fig. 7). As seen in Fig. 7, the maximum value of absorbance peak of MO solution is observed at 472 nm that the peak only become decrease slowly with respect to the increases in irradiation time, confirming sufficient photocatalytic degradation of MO dye. Also, the peak did not shift and no other peaks were appeared, predicting that the pure photochemical reaction was happened [33].

3.7 Effect of Irradiation Time and Catalyst Dose

The influence of irradiation time and catalyst dose on photocatalytic performance of MO is represented in Fig. 8. In the first 5 min of irradiation time, about 32.12% and 43.46% of MO was degraded using 0.01 and 0.02 g of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ as photocatalyst, respectively. While at 90 min irradiation time, the percentage of MO photodegradation were 73.26 and 88.38%, respectively.

Compared with the photodegradation performance of other compounds (Table 1), it was noted that the $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ exhibited remarkably better photocatalytic activity.

The photodegradation performance of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ was evaluated for 5 cycles. After each experiment, the photocatalyst collected by centrifuged, washed twice with distilled water, dried at 80 °C and then used for degradation of MO in similar conditions. Reusability performance results are

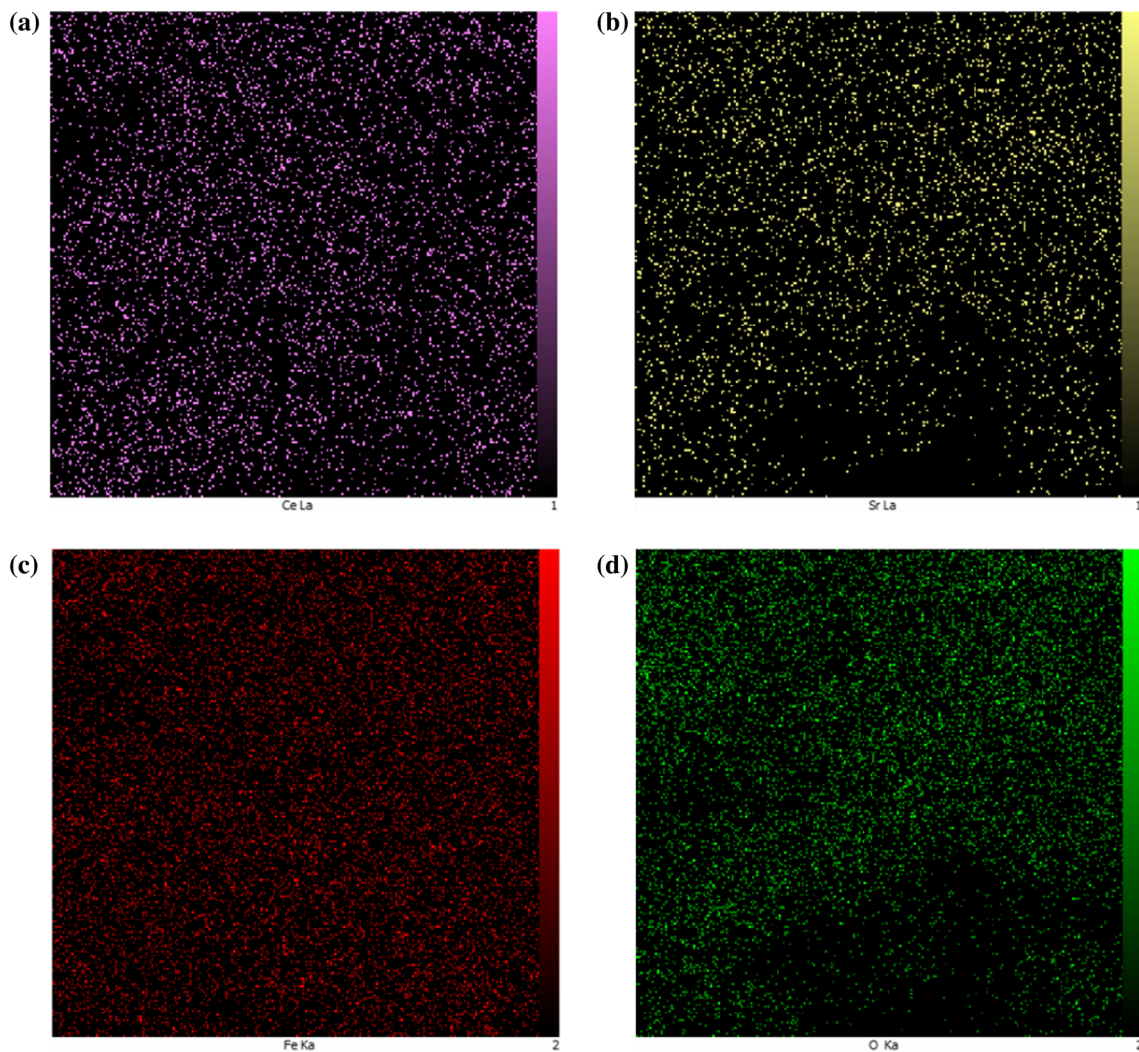


Fig. 5 Elemental distribution **a** Ce, **b** Sr, **c** Fe and **d** O images of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ nanocomposite

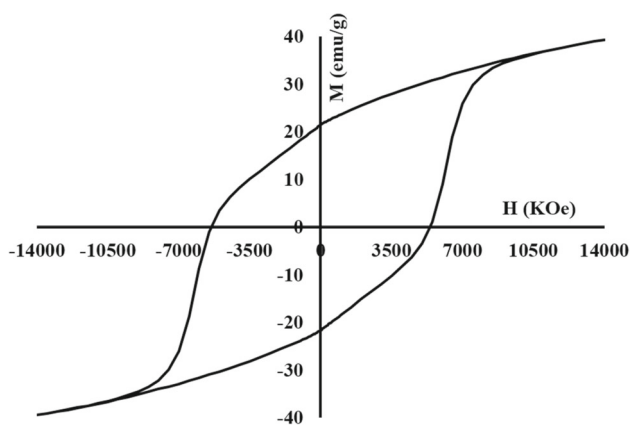


Fig. 6 Hysteresis loops of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ nanocomposite

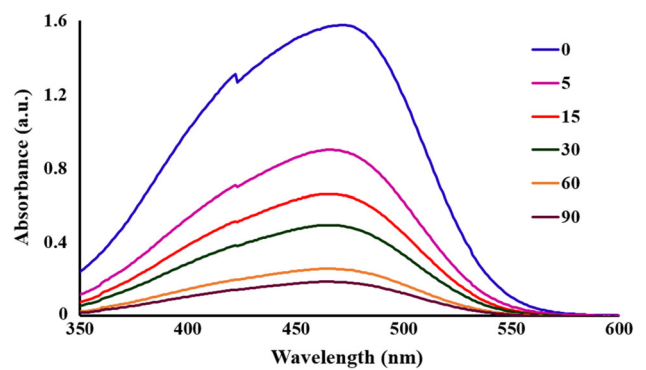


Fig. 7 Time-dependent UV-Vis spectra of MO solution in the presence of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ nanocomposite

shown in Fig. 9. As can be seen in Fig. 9, it was clear that $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ as photocatalyst showed an excellent

activity after 5 cycles and a slight amount ($< 5\%$) in photodegradation activity was observed.

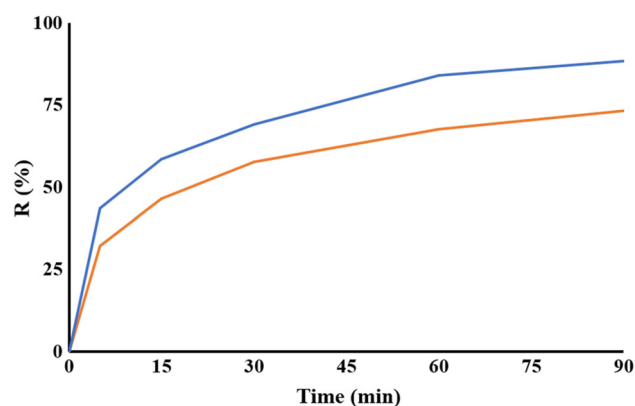


Fig. 8 The effect of irradiation time on removal percentage of MO dye in the presence of **a** 0.01 and **b** 0.02 g of CeO₂/SrFe₁₂O₁₉ nanocomposite

Table 1 The photodegradation performance of CeO₂/SrFe₁₂O₁₉ and other compounds

Adsorbent	Photodegradation percentage (%)	Reference
CeO ₂ /SrFe ₁₂ O ₁₉	88.38	This work
Fe ₂ O ₃	95%	[21]
TiO ₂ /ZnO	97%	[26]
Bi ₂ O ₃ -Al ₂ O ₃	60	[23]
MoS ₂ /Fe ₃ O ₄	79	[54]
TiO ₂ powder	24	[22]
Ag/TiO ₂ /biochar	97.5	[10]
α-Fe ₂ O ₃ @carbon core-shell	85	[55]
SrFe ₁₂ O ₁₉	95	[28]

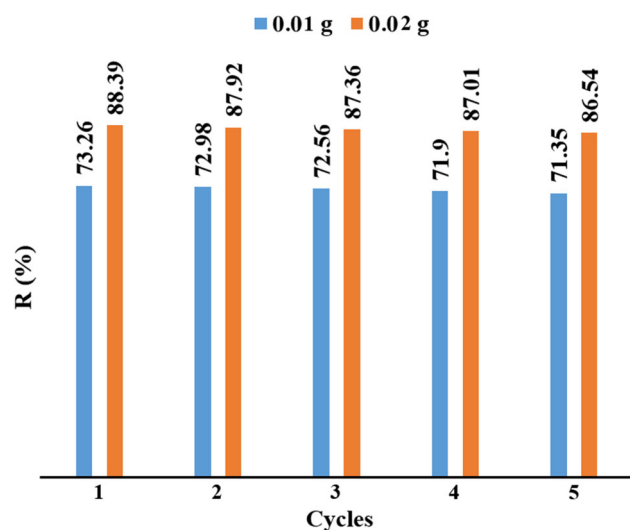


Fig. 9 Reusability experiment of CeO₂/SrFe₁₂O₁₉ nanocomposite for 5 cycles

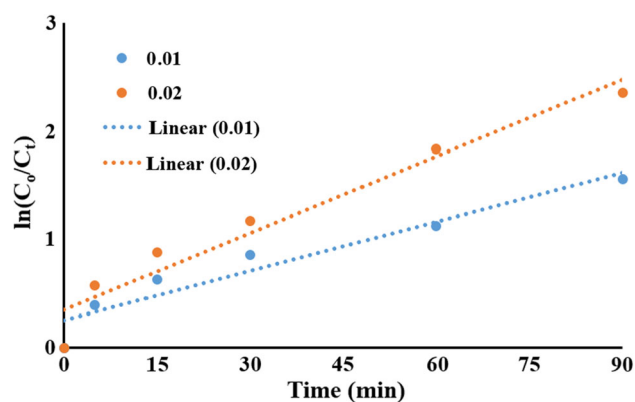
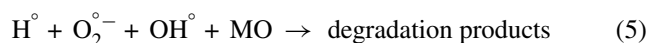
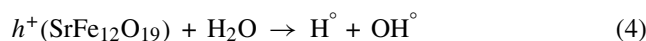
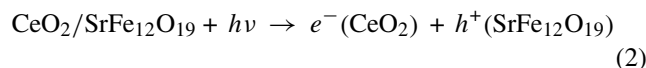


Fig. 10 Pseudo-first-order plots of MO degradation of **a** 0.01 and **b** 0.02 g of CeO₂/SrFe₁₂O₁₉ nanocomposite

3.8 Langmuir Isotherm

The kinetic analysis of the degradation of MO dye using CeO₂/SrFe₁₂O₁₉ nanocomposite by Langmuir model was investigated and the linear relationship between $\ln(C_0/C_t)$ and time is confirmed by pseudo first-order kinetic as shown in Fig. 10.

A suitable mechanism for the photodegradation of methyl orange using CeO₂/SrFe₁₂O₁₉ nanocomposite was proposed based on the above results, as demonstrated by following equations [11, 21, 28, 30, 32]:



For photodegradation of organic dyes using different photocatalysts, there are several steps [28] such as diffusion and adsorption of dye on the surface of catalyst, adsorption of light by catalyst for preparation of e^-/h^+ pair (charge separation), various reactions to produce active radicals and finally degradation of dye by active radicals H° , $\text{O}_2^{\circ-}$ and OH° [11, 21, 28, 30, 32].

4 Conclusions

We have successfully prepared a magnetic nanocomposite CeO₂/SrFe₁₂O₁₉ photocatalyst by a one-step chemical coprecipitation with high-temperature sintering method. High magnetic performance of CeO₂/SrFe₁₂O₁₉ predicts the ease separation of CeO₂/SrFe₁₂O₁₉, to its recycling and reused

without secondary pollution. The photocatalytic potential of $\text{CeO}_2/\text{SrFe}_{12}\text{O}_{19}$ illustrated that the percentage of MO degradation was up to 88.38% after UV light irradiation for 90 min. The photocatalytic data revealed that the degradation of MO followed by Langmuir isotherm as pseudo-first-order kinetic model.

Author Contribution S.A. Jasim, A.M. Abdulhadi, M.E. Al-Gazally and T. Alawsai collected the FT-IR, XRD and VSM analysis, revised the paper. I. Patra and H. Sharma revised the paper, recorded the SEM/EDX and DSC analysis. H.T. Mohammed, S.A. Hussein, U.S. Altamari, A.T. Hammid designed and collected the photodegradation data, revised the paper. C. Chem supervised, wrote and revised the paper. All authors discussed the results and contributed to the final manuscript.

Declarations

Conflict of interest The authors declare that there is no conflict of interest.

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