

RESEARCH ARTICLE

Mesoporous Fe/MCM-41 as Heterogeneous Photocatalyst for the Photodegradation of Methylene Blue

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ABSTRACT:

We have explored the highly efficient and environmentally benign Fe/MCM-41 for methylene blue (MB) dye removal in aqueous solution. The synthesised mesoporous Fe/MCM-41 were characterized XRD, SEM and TEM. The concentration of MB dye removal was estimated from its optical density at $\lambda_{\text{max}} = 665 \text{ nm}$. From the results we infer that the catalyst reveal excellent catalytic property for 95% removal of MB within 75mins, which could be attributed to the adsorptive power of Fe/MCM-41. With the advantages of rapid degradation and efficient magnetic separation, the synthesized material could gain a has the potential to be used as reductant/adsorbent to remove cationic dye molecules effectively and rapidly from drinking water and large scale of industrial wastewater

KEYWORDS: Fe/MCM-4, XRD, Methylene Blue, wastewater treatment.

INTRODUCTION:

Photocatalytic oxidation is one of the most new pollution treatments and is widely applied to water and air purification¹. Colour is the main contaminant to be present in the waste water coming from industries. Dyes are widely used in industries such as textiles, leather, printing, food, and plastics, etc². Various methods developed for the removal of dyes from waste water include adsorption onto solid substrates, chemical coagulation, oxidation, filtration and biological treatment. In recent years photocatalytic processes using titania have been used for important problems of environmental interest like purification of water and air^{3,4} because it has a wide band-gap semiconductor with a large number of applications in catalysis, photovoltaic's, optoelectronics, self-cleaning glass, pigments and many others⁵.

Titania has attracted a great deal of attention since it has high photocatalytic activity and physicochemical stability⁶. Even though it has widespread use of titania was impaired by some flaws of its structure, agglomeration easily happening during the synthesis process and wide band gap (3.2 e.V) which requires ultraviolet irradiation for photocatalytic activation⁷. To overcome this difficult, the discovery of mesoporous materials exhibits wide range of industrial applications since they have high surface area, large pore volume, uniform pores and relatively high thermal stability^{8,9}. Also, mesoporous materials with large surface area and good acidic properties adsorb organic pollutants near to the active sites that results in enhanced photocatalytic degradation rate, moreover the photocatalytic efficiency is also increase by decreasing the electron-hole recombination, facilitating the interfacial charge transfer from the surface of the photocatalyst to the substrate¹⁰. In this work, we have synthesized iron doped mesoporous MCM-41 materials which has been utilized towards the degradation of methylene blue (MB).

MATERIALS AND METHODS:

Fe/MCM-41 materials with different percentage were synthesized by wet impregnation method using sodium metasilicate ($\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$), ferric nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), cetyltrimethylammonium bromide ($\text{C}_{16}\text{H}_{33}(\text{CH}_3)_2\text{N}^+\text{Br}^-$), and sulphuric acid (H_2SO_4). The AR grade chemicals used were purchased from Aldrich & Co., USA.

Synthesis of Fe/MCM-41:

According to our previous report¹¹⁻¹³, we have synthesized Si-MCM-41 by hydrothermal method. Fe/MCM-41 with different weight percentage (10 wt%, 20 wt % and 30 wt %) were obtained by wet impregnation techniques. 1 g of MCM-41 was treated with required amount of 1 M ferric nitrate solution as per loading in ethanol solution (total volume ~25 ml) under stirring at room temperature for 3 h followed by filtration and drying at 80°C. The obtained materials were then calcined at 450°C in air.

Photocatalytic Experiments:

The degradation of methylene blue was studied using Fe/MCM-41 catalyst. A stock solution of MB (1000 Mg/L) was prepared and suitably diluted to the required concentration. All the experiments were performed in a 100 ml beaker in presence of direct solar light in the summer season from 12.00 p.m. to 14.00 p.m. (radiation). At the given time intervals, 2 ml of the suspensions were separated, and then centrifuged to remove the catalyst. The amount of MB present in the water was calculated by Shimadzu UV-3600 Model UV-Visible Spectrophotometer (Japan) and the wavelength of maximum absorption at $\lambda_{\text{max}} = 665 \text{ nm}$ was monitored.

RESULTS AND DISCUSSION:

XRD Pattern of Fe/MCM-41:

The X-ray diffraction pattern of Si-MCM-41 and Fe/MCM-41 with various weight percentage (10%, 20, 30%) were shown in Figures 1. A prominent peak for $hkl = 100$, as well as weaker peaks for $hkl = 110$, 200 and 210 were observed in the samples. The intensity of the (100) diffraction line is higher compared to other hkl peaks. It can be observed that all the above catalysts exhibit a strong peak in the 2θ range of 1.8 to 2.2° due to (100) reflection line and small peaks in the 2θ range of 2.8-4.1° due to higher order (110), (200) and (210) reflections indicating the formation of well-ordered mesoporous materials¹³.

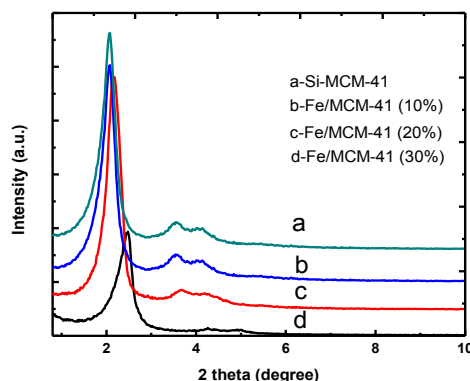


Figure 1: XRD pattern of Si-MCM-41 and Fe/MCM-41

Morphological study:

The size and morphology of Fe/MCM-41 was investigated with SEM and the image are presented in Figure 2. As depicted in the SEM image, the formation of orderly growth of pure hexagonal phase of the molecular sieves has been shown. It can also be seen that after incorporation of metal in the framework, the samples show irregular morphology. The TEM image of the Fe/MCM-41 was shown in Figure 3, which clearly indicate formation of well defined hexagonal array of channel characteristic of the MCM-41 structure¹⁴.

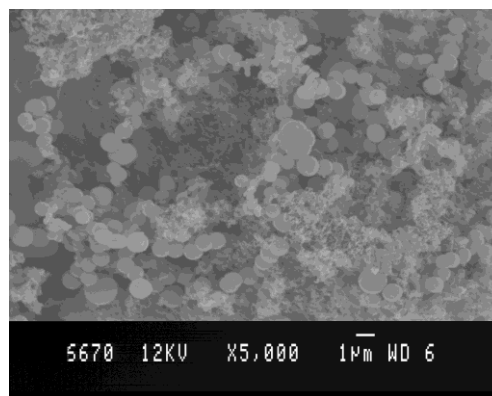


Figure 2: SEM Image of Fe/MCM-41

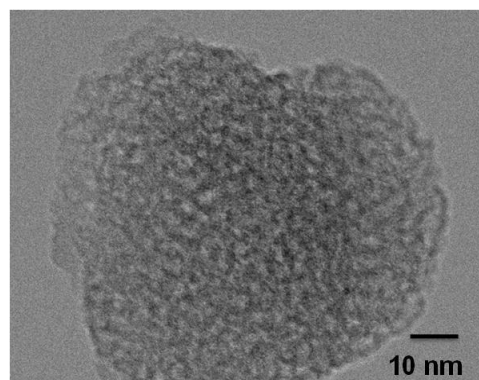


Figure 3: TEM Image of Fe/MCM-41

Photocatalytic activity of the synthesized Fe/MCM-41 catalyst:

Effect of catalyst dosage:

The effect of the Fe^{3+} ions on degradation of methylene blue under direct sun light was evaluated by varying the Fe/MCM-41 catalyst concentration 10%, 20% and 30% at 100 mg of methylene blue concentration. The degradation efficiency increases on increasing the concentration of Fe^{3+} ions due to enhanced generation of OH radicals (Fig.4). Further increase in the concentration, degradation efficiency decrease because of Fe^{3+} act as a filter of sun light¹⁵.

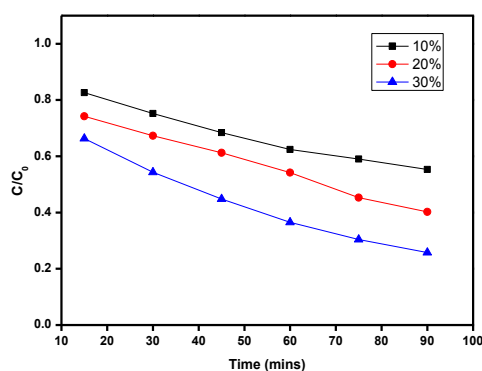


Figure 4: Effect of Fe^{3+} concentration on degradation

Effect of H_2O_2 :

The effects of H_2O_2 concentrations on the degradation rate of MB were investigate to explain the role of H_2O_2 . The degradation efficiency at various concentrations of H_2O_2 was shown in Fig.5, it was observed that the degradation efficiency initially increased with an increase in the H_2O_2 concentration up to 6ml/L. At low concentration, H_2O_2 cannot generate enough hydroxyl radicals so rate of the reaction is low. Above optimum level of H_2O_2 concentration the decrease in decolorization occurs due to the scavenging effect of excess H_2O_2 , which decreases the number of hydroxyl radicals in the solution¹⁶.

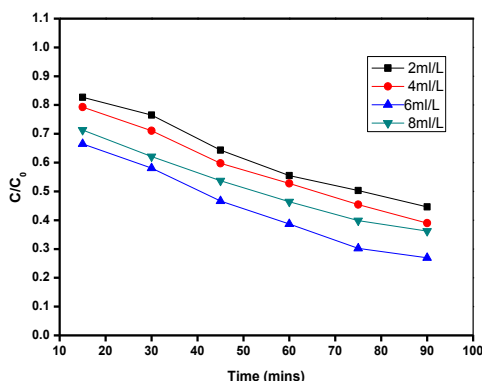


Figure 5: Effect of H_2O_2 on degradation efficiency

CONCLUSION:

We have concluded that the synthesized Fe/MCM-41 photocatalyst by hydrothermal method. The XRD pattern of Fe-MCM-41 shows the formation of well-ordered mesoporous materials. The TEM image clearly indicates the well defined hexagonal array of channel. The catalytic efficiency of the synthesized sample was studied on the removal of MB under sun light and optimized various parameters like H_2O_2 and catalyst dosage. From the results we confirmed the synthesized material shows to be highly efficient for removal of organic dyes in water by catalytic degradation.

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