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Synthesis of MIL-53(Fe)/SiO_2 composite from LD slag as a novel Photo-catalyst for Methylene Blue degradation

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Highlights

- MIL-53(Fe)/SiO_2 composite was solvo-thermally prepared from LD slag, as a photocatalyst.
- It was characterized by XRD, UV-vis absorbance spectroscopy, SEM-EDS, TEM etc.
- MIL-53(Fe)/SiO_2 composite effectively degraded methylene blue under UV light.
- Maximum degradation of MB was achieved as almost 65 %.

1. Introduction

Metal–organic Frameworks (MOF) are basically multifunctional inorganic-organic hybrid porous and crystalline materials consisting of infinite lattices made up of inorganic secondary building units (SBU) and organic linkers. MIL-53(Fe) is one such MOF, composed of infinite $FeO_4(OH)_2$ cluster connected by 1,4-benzenedicarboxylate (H_2BDC) ligand. Nowadays, application of MIL-53(Fe) in the photocatalytic degradation of organic dyes has obtained extensive attention. To improve its photocatalytic performances, introduction of functional entities into the framework has been attempted [1]. Incorporation of metal oxide into MIL-53(Fe) template forms a composite photocatalyst with improved photocatalytic activity [2]. Therefore, it's worthwhile to directly prepare a MIL-53(Fe) and metal oxide composite from a single raw material rather than incorporation of other material into the framework. In this work, MIL-53(Fe)/SiO_2 composite was prepared from raw LD slag, a steel industry waste containing high Fe_2O_3 and SiO_2, by solvo thermal process. Methylene blue (MB) was chosen as a representative organic pollutant due to its harmful nature towards both aquatic and human life, for studying the photocatalytic activity of MIL-53(Fe)/SiO_2.

2. Methods

MIL-53(Fe)/SiO_2 was synthesized from LD slag by following the typical procedure, where 0.62 g washed LD Slag was firstly dissolved into 21.94 ml of 35% (v/v) HCl in 100^0C so that the Fe_2O_3 present in the raw LD slag gets completely converted to $FeCl_3$ forming a yellowish solution. Then, 0.415 g of H_2BDC and 56 mL of DMF were mixed with the solution and were stirred at room temperature for 2 hours. After that, the clear mixture was transferred into a 100 mL Teflon lined stainless steel autoclave and heated at 170 ◦C for 24 h. Next, the resultant suspension was filtered and washed with hot water to remove calcium terephthalate. Next, the sample was again washed with DMF and C_2H_5OH and the powder sample was kept in an oven dryer at 100 ^{0}C for 10 h.

In the cylindrical glass reactor, 0.1 g MIL-53(Fe)/SiO_2 was added in 200 ml of 10 mg/L MB solution at pH=7. The photocatalytic degradation of MB was then performed in the reactor under UV light ($\lambda \sim 280$ nm) by introducing moderate air of 0.5 LPM at the base of the reactor using a peristaltic pump The suspension was then mixed vigorously in absence of UV light for 30 min to reach adsorption-desorption equilibrium. After irradiation, 3 ml of sample was collected at every 15 min and centrifuged at 10000 rpm for 10 minutes. Supernatant was transferred to a quartz Cuvette and analyzed by UV-vis spectrophotometer.

3. Results and discussion

XRD pattern of the photocatalyst confirms the presence MIL-53(Fe)/SiO_2 composite. FESEM and HRTEM images shows its surface morphology and particle sizes. UV-vis absorbance spectrums of MIL-53(Fe)/SiO_2 analyses its optical properties during the photooxidation process. EDS analysis provides the elemental composition of the composite photocatalyst.

In dark condition for 30 min under vigorous mixing, 14% removal of MB took place due to adsorption process. When the adsorption equilibrium reached, photocatalytic degradation of MB was performed under UV light. Maximum 65% degradation of MB was achieved under UV light irradiation, as shown in figure 1. It can be understood that MB and O_2 molecules are adsorbed on the surface of the MIL-53(Fe)/SiO_2 particle. In addition, the photocatalytic degradation kinetic data is fitted to first-order kinetics plot by the equation [3]:

$$-ln(C/C_0) = kt$$

According to the equation, the rate of MB degradation has been found as 0.0122 min^{-1} which is quite higher than other similar photocatalysts mentioned in the literature.

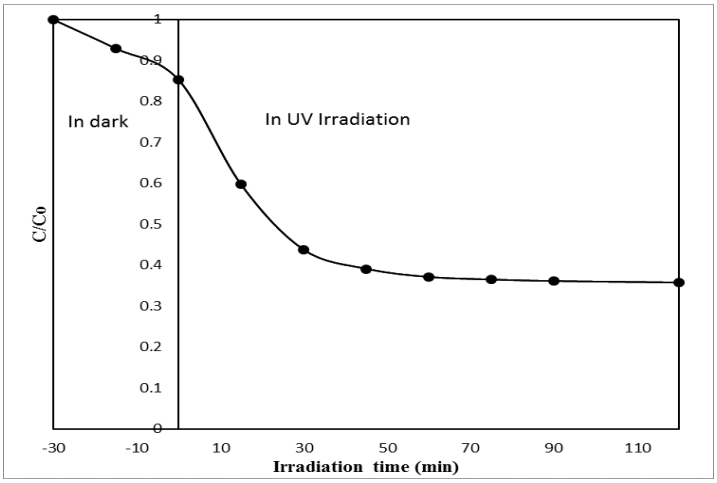

Figure 1. Photocatalytic degradation of MB over MIL-53(Fe)/SiO_2 in UV irradiation.

4. Conclusions

MIL-53(Fe)/SiO_2 composite photocatalyst has been successfully synthesized from LD slag by facile solvothermal method for the first time. The characterizations of MIL-53(Fe)/SiO_2 composites were performed by XRD, SEM/EDS, TEM. The photocatalytic degradation of methylene Blue is carried out in a cylindrical glass reactor under UV light. The maximum degradation is achieved as 65% which is quite high compared to other similar photocatalyst.

References

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Keywords
MIL-53(Fe)/SiO_2 composite, Photocatalysis, LD Slag.