Effect of Fe$_3$O$_4$ crystallite size on the Rhodamine B degradation

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Abstract

Fe$_3$O$_4$ particles showed catalytic efficiency on Rhodamine B degradation with strong dependence on the nanograin size.

Introduction

The degradation of organic pollutants from wastewater is a growing research topic due to its ecological and environmental relevance. Organic dyes like Rhodamine B are widely used in textiles industries, and it is very important their removal from wastewater$^1$. Magnetite (Fe$_3$O$_4$) particles have been used as catalyst in Fenton reaction for dyes degradation$^2$. In this work the degradation of RhB under visible light, using H$_2$O$_2$ as the oxidation reagent in a photo-Fenton reaction was used as a model reaction to investigate the Fe$_3$O$_4$ catalytic activity in different particles sizes and crystallite sizes. The particles were synthesized by modified solvothermal route to obtain different size of particles. The degradation tests were carried out using the same concentration of particles for all tests.

Results e Discussion

Three different sizes samples of Fe$_3$O$_4$ were synthesized by modified solvothermal method. In our methodology the temperature of dissolution of iron salt precursors was changed, keeping constant the reaction temperature in autoclave. This synthesis parameter unprecedented allowed controlling the size of particles. Particles of 100 nm, 350 nm and 750 nm using temperature of 140 °C, 80 °C and 25 °C, respectively. The SEM image reveals nanostructured particles showing rough surface, the particles are made of aggregates of nanoparticles of magnetite, the nanograin. The XRD data confirms the pattern of Fe$_3$O$_4$. The crystallite size was calculated by Scherrer equation and the values were 77 nm to the 750 nm particles (Fe$_3$O$_4$-750), 70 nm to the 350 nm particles (Fe$_3$O$_4$-350) and 13 nm to the 100 nm particles (Fe$_3$O$_4$-100). RhB was used as organic dye to degradation reaction by H$_2$O$_2$ under UV-Vis radiation. Without Fe$_3$O$_4$ (Figure 1a) particles, the degradation is less than 5%, considered negligible, being attributed to the low oxidation potential of H$_2$O$_2$ as compared to hydroxyl radicals (OH). When Fe$_3$O$_4$-100 was irradiated under visible radiation, without H$_2$O$_2$ (Figure 1b) it was observed a little RhB degradation, approximately 10%, due to adsorption of RhB in Fe$_3$O$_4$ catalyst. The RhB degradation of the 700 and 350 nm particles (Figure 1c and 1d, respectively) is similar, around 70% in 60 min. Although these particles have different sizes, there is almost no difference in their crystallite size (nanograin), thus this slight difference is not enough to promote higher catalytic activity for the 350 nm microspheres. However, the degradation of RhB by H$_2$O$_2$ using 100 nm magnetite particles under Vis irradiation reached 100% in 60 min (Figure 1e), which showed the 100 nm Fe$_3$O$_4$ has more catalyst efficiency in RhB degradation. The smaller crystallite (13 nm) contributes to enhance the catalytic effect in these particles in RhB degradation due to the higher surface/volume ratio. Furthermore, the catalysts can be conveniently separated by applying an external magnetic field.

Figure 1. Degradation of RhB under different conditions: (a) RhB/H$_2$O$_2$/vis; (b) RhB/Fe$_3$O$_4$-100/vis; (c) RhB/Fe$_3$O$_4$-750/H$_2$O$_2$/vis; (d) RhB/Fe$_3$O$_4$-350/H$_2$O$_2$/vis; (e) RhB/Fe$_3$O$_4$-100/H$_2$O$_2$/vis

Conclusions

The Fe$_3$O$_4$ with controlled particles size by temperature of dissolution of iron salt precursors showed efficient on RhB degradation by photo-Fenton reaction, confirming the crystallite size influence on catalytic activity of the particles. The most efficient degradation was 100 nm particles, 13 nm crystallite size. As smaller the crystallite is higher is the catalytic activity.

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