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Preparation and characterization of Fe_2O_3 nanoparticles

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Abstract. Nano particles hematite (Fe_2O_3) with good crystallinity were prepared by Pechini method. Hematite (Fe_2O_3) has emerged as a promising photo-electrode material due to its significant light absorption, chemical stability in aqueous environments, and ample abundance. Photoelectrochemical cells offer the ability to convert electromagnetic energy from our largest renewable source, the Sun, to stored chemical energy through the splitting of water into molecular oxygen and hydrogen. The structure and the size of the Fe_2O_3 nanoparticles were analyzed by SEM and XRD. The UV-Vis optical absorption of the samples was also investigated. These methods help to define the obstacles that remain to be surmounted in order to fully exploit the potential of this promising material for solar energy conversion.

1. Introduction

Many industrial dyes are toxic and carcinogenic. The organic dyes present in industrial wastewater often pose significant threats against human health and environmental pollution control. Therefore, it is important to remove organic dyes from wastewater. However, wastewater exhibits stable behavior under harsh conditions and resists biodegradation,making it difficult to remove organic dyes easily [1, 2, 3, 4, 5, 6, 7, 8]. Many wastewater treatment methods have been explored for example the adsorption technique, but this method is now becoming unpopular because it is expensive, and the adsorbent has low recyclability. Photocatalysis is an environment friendly process that utilizes irradiation energy for catalytic reactions. Hence, photocatalytic technology has been widely investigated for applications to the decomposition of pollutants. Researchers are especially interested in developing photocatalysts that can extend the absorption wavelength into the visible-light region [1, 2, 9, 10]. Hematites (Fe_2O_3) are minerals belonging to the group of iron-oxide minerals; they have a hexagonal structure and exhibit paramagnetic behavior. Moreover, they show strong catalytic activity, widely and easily available, and are extremely environment friendly. In particular, hematite may be a promising candidate for visible-light photocatalysis it can absorb visible light, collect up to 45% of solar-spectrum energy, and is one of the cheapest semiconductor materials available [2, 9, 10, 13].In this study, hematite nanoparticles are employed for the removal of Rhodamine B (RhB) dye by photocatalytic decomposition. RhB is nonbiodegradable and extensively used in the industry; therefore, it is selected as the model contaminant .

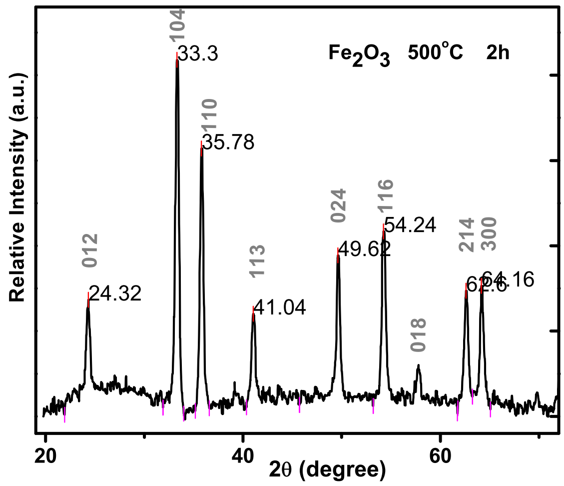

Figure 1. X-ray powder diffraction patterns of Fe_2O_3 prepared by the polymeric precursor method at 500 C.

2. Experimental

The polymeric precursor method (pechini) is based on the polymerization of metallic citrate by ethylene glycol[11, 12]. A hydrocarboxilic acid such as citric acid is normally used to chelate cations in an aqueous solution.The method is based on an intensive blending of positive ions in a solution, controlled transformation of the solution into a polymer gel, removal of the polymer matrix and development of an oxide precursor with a high degree of homogeneity. During the synthetic process, metal salts or alkoxides are introduced into a citric acid solution with ethylene glycol. The formation of citric complexes is believed to balance the difference in individual behavior of ions in solution, which results in a better distribution of ions and prevents the separation of components at later process stages.

3. Results and discussion

The XRD pattern (1) of the annealed sample shows the formation of phase pure hematite.shows the XRD pattern of the dark red-brown materials, Fe_2O_3 obtained after $500C$. The XRD spectrum of the nanoparticles shown in figure (1) contains nine peaks, which are clearly distinguishable [13]. All of them can be perfectly indexed to rhombohedral Fe2O3 in peak position (JCPDS No. 24-0072), space group$R\overline{3}$(148). The spectrum contains no impurity phase. Furthermore, the relative intensity of the (110) diffraction peak is greater than that of polycrystalline Fe_2O_3 power. The fact indicates that the nanoparticles Fe_2O_3 crystals may develop preferentially rather than randomly.

The nanoparticles size was roughly estimated to be about $60 - 70nm$ from the SEM data, as shown in figure 2 Such a type of unique morphology was observed for the first time for iron oxide with excellent magnetic properties. SEM images also clearly showed the nano-structural homogeneities and remarkably unique neck-structured morphology with average size of $50 - 60nm$. Although the neck-structure shaping mechanism is unclear, low temperature during autoclave, long time oxidation and annealing may be responsible to the neck-structure formation. Further study is needed to clarify the mechanism. The figure 2a) shows the sample without addition of NH_4 at a temperature of $500C$ this presents an irregular shape of the nanoparticles, while figure 2 b) is Fe_2O_3 with addition of NH_4 at a temperature of $500C$,these nanoparticles have a more regular basis.

Figure 3 shows the absorption spectra of the RhB solution during different irradiation times.

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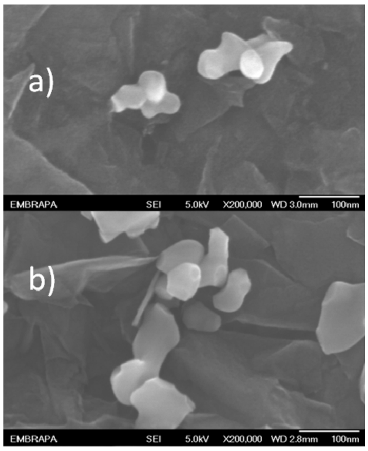

Figure 2. Examples of hematite nanoparticule morphologies: SEM images of hematite prepared by the thermal oxidation method (top image Fe_2O_3, bottom image Fe_2O_3 with NH_4). Temperature $500C$.

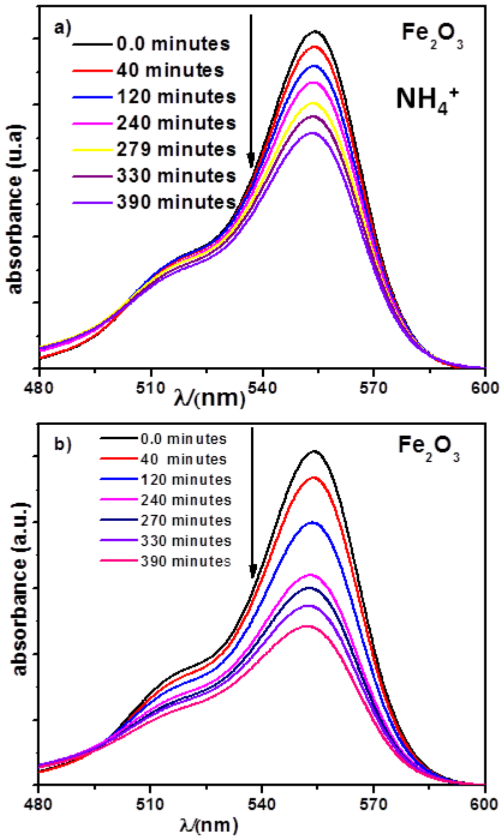

Figure 3. (Color online) Absorption spectra of rhodamine B aqueous solution in presence of nanoparticles of Fe_2O_3 , using $\lambda = 350nm$ for the illumination. a) with addition of NH_4, b) without added NH_4.

In this work, experiments were performed in three different conditions, namely: (1) without catalyst, (2) in the presence of Fe_2O_3 with PH=7 as catalyst and (3) in the presence of

Fe_2O_3 nanoparticles as catalyst. UV irradiation of RhB in the absence of hematite catalyst showed almost a negligible variation in the absorbance maximum. However, when hematite nanostructures were used as photocatalyst, the absorption maximum (at $\lambda = 550$ nm) steadily decreased upon irradiation with UV, without any obvious peak shift of the absorption maximum. This suggests that the complete degradation and decolourization of aqueous RhB was purely due to the catalyst under UV irradiation. This work reports a facile and simple method for the large scale synthesis of hematite nanostructures. The prepared hematite nanostructures showed excellent photocatalytic activity towards Rhodamine B, which was believed to be due to the high surface area. Among these two structures of the hematite, the Fe_2O_3 without NH_4 showed relatively faster photocatalytic activity towards RhB.

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