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Synthesis and Photocatalytic Studies of Fe₃O₄ Nanoparticles

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Abstract

Advances in nanoscience and nanotechnology are centered in the control of size and shape of nanoparticles, as well as attainment of the extended arrangement of nanoparticles in different structures. One dimensional nanostructured magnetic materials are interesting because of their enhanced magnetic properties and potential applications such as information storage and gas sensor. The advanced electronic applications of Fe₃O₄ magnetic nanomaterials are considered to require improvement in the powder processing, particularly, meticulous particle control in the nanometer range and phase purity. Structural properties of Fe₃O₄ nanoparticles were synthesized via sol-gel protocol, the morphology and particles size were determined by X-ray diffraction, scanning electron microscope, transmission electron microscope, energy dispersive X-ray, UV-diffuse reflectance spectrophotometer and particle size analyzer. The structure of as prepared Fe₃O₄ nanoparticles was used for photocatalytic applications.

1. Introduction

Nanomaterials are the most challenging areas of current scientific and technological research because of their tremendous possibilities in novel shapes, structures and the unusual phenomena associated with materials. The field of nanotechnology is one of the most popular areas for current research and development in all technical disciplines. Nanostructured II–VI semiconductors have been studied very intensively in recent time due to their industrial implementation in nanoelectronic devices [1]. The n-type metal oxide semiconductor gas sensors are popular for monitoring caused by toxic and inflammable gases. They exhibit changes in electrical resistance in the presence of toxic and inflammable gases in the ambient atmosphere. The basis of this change in

resistance lies in the reaction between the adsorbed oxygen on the oxide surface and a reducing gas, thus causing a decrease in resistance by releasing electrons back into the oxide [2]. Nanostructures of metal oxides have great attention due to their unique properties in novel applications. The devices based on inorganic materials such as metal oxide semiconductors, which works on principle of the change in conductivity with interaction of molecules [3]. Metal oxides nanostructures offer functionality from electrically conducting to insulating and from highly catalytic to inert properties. One-dimensional nanostructured materials have attracted great attention over the past decade because of their important and often exhibit novel applications such as magnetic, electrical and optical properties. The unique and novel size dependent properties of

magnetic oxides have initiated current worldwide intense research on magnetic nanomaterials [4]. Magnetic particles with sizes in the nanometer scale are now of interest because of their many technological applications and unique magnetic properties which differ considerably from those of bulk materials. Below a critical size, magnetic particles become single domain in contrast with the usual multi domain structure of the bulk magnetic materials exhibiting unique phenomena such as super paramagnetic and quantum tunneling of the magnetization [5]. Iron oxides are one of the most important transition metal oxides of technological importance [6]. Fe₃O₄ is a traditional magnetic material used in wide variety of applications such as electronic ignition systems, generators, vending machines, medical implants, wrist watches, inductor core, transformer circuits, magnetic sensors and recording equipment, telecommunications, magnetic fluids, microwave absorbers and other high-frequency applications [7]. Nanoparticles with novel morphologies and desired compositions have drawn immense attention due to their unique morphology and composition-dependent physicochemical properties and their importance in basic scientific research and potential technology applications [8].

The energy gap (E_g) is an important feature of semiconductors which determines their applications in optoelectronics [9-12]. The UV-Vis absorption spectroscopy is frequently used to characterize semiconductors materials [13], it is easy to extract the E_g values from their absorption spectra knowing their thickness. However,

in colloidal samples, the scattering effect is enhanced since more superficial area is exposed to the light beam. In normal incidence mode, dispersed light is counted as absorbed light and the technique does not distinguish between the two phenomena. On the other hand, it is common to obtain powdered materials, frequently UV-Vis absorption spectroscopy is carried out dispersing the sample in liquid media like water, ethanol or methanol. If the particle size of the sample is not small enough, it precipitates and the absorption spectrum is even more difficult to interpret. In order to avoid these complications, it is desirable to use diffuse reflectance spectroscopy (DRS), which enables to obtain E_g of un-supported materials [14]. The theory which makes possible to use DRS was proposed by Kubelka and Munk [15].

2. Experimental Methods

2.1 Synthesis of Fe₃O₄ Nanoparticles

During the sol-gel protocol of iron nanoparticles, 3.2 g of poly ethylene glycol (PEG) taken in 25 mL of distilled water and stirred for 30 min. Later 3.3 g of FeSO₄.7H₂O in 10 mL of distilled water, 9.6 g of Fe₂(SO₄)₃ in 20 mL of distilled water are stirred separately. Now both FeSO₄.7H₂O and Fe₂(SO₄)₃ solutions were added drop by drop into the poly ethylene glycol solution. Then the solution was stirred for 30 min, ammonia solution is added to maintain pH-10. The mixture was further stirred for 4 h and filtered, washed with distilled water and finally rinsed with acetone and dried in hot air oven at 60 °C for 8 h. The dried compound was calcined at

different temperatures at 300 °C, 500 °C and 700 °C for 4 h to get Fe₃O₄ nanoparticles.

2.2 Characterization Techniques

X-ray powder diffraction data were recorded on Siemens (D5000) diffractometer using CuK α radiation ($\lambda = 1.5406 \text{ \AA}$) in the range of $2\theta = 2-65^\circ$. Particle size was recorded by particle size analyzer Horiba SZ100. The morphological analysis of the samples was done by using transmission electron microscope (TEM). A drop of the reaction mixture was placed over carbon-coated copper grids and the solvent was allowed to dry. Images were acquired on a Philips Technai-FE 12 TEM (120 kV). The energy dispersive X-ray spectroscopy (EDX) analysis was performed by scanning electron microscopy (SEM) with a Model S520 (Hitachi, Japan) equipped with an EDX detector (Oxford LINK-ISIS 300). The EDX spectrum was measured at 10 kV accelerating voltage.

3. Results and Discussion

3.1 X-Ray Powder Diffraction

XRD patterns of Fe₃O₄ nanoparticles as-synthesized and calcined at different temperatures at 300 °C, 500 °C and 700 °C. From the patterns of samples, it was found that all the different peaks at (110), (024), (116) corresponds to 36°, 48°, 54°. For calcined samples we observed peaks (104), (110), (024), (116) corresponds to 30°, 36°, 48°, 54° to the inverse cubic spinel structure of Fe₃O₄. In the above spectra phase identification is one of the most important uses in XRD.

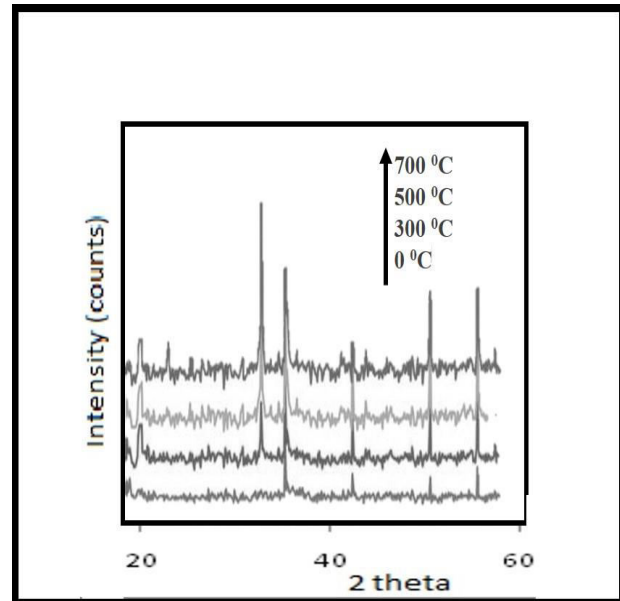


Fig. 1 XRD pattern of a) as-synthesized and (b-d) annealed at different temperatures of Fe₃O₄ nanoparticles

As shown in Fig. 1, XRD pattern of iron nanoparticles after annealing the XRD patterns well indexed to the cubical spinel phase of magnetite and no other peaks observed in as-synthesized material.

3.2 UV-DRS

The UV-DRS spectra of the composites, recorded in the %reflectance mode between 350 to 700 nm are shown in Fig. 2. The optical band gap energy (E_g) is determined for direct and/or indirect band gap. The UV-DRS spectra of the iron nanoparticles, recorded in the %reflectance mode between 350 to 700 nm are shown in Fig. 2. Reflectance band edge of the as synthesized and different annealing temperature of the iron nanoparticles show

broad absorption slopes implying the presence of multiple phases in the bulk. cases of transitions.

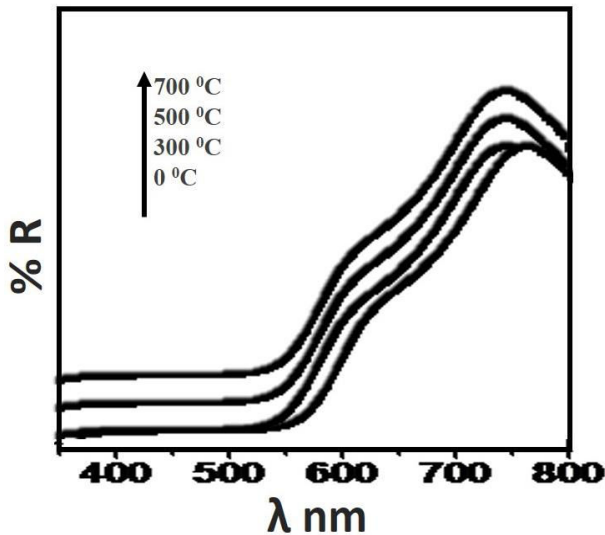


Fig. 2. Representative Diffused reflectance UV-Vis spectra of the iron nanoparticles collected in the reflectance mode

3.3 Particle Size Analysis

Particle size distribution of Fe₃O₄ nanoparticles are shown in the Fig. 3 as-synthesized and calcined between 300-700 °C. It is observed that as-synthesized sample as per average particle size of 11 nm and calcined at 700 °C as average particles size of 23 nm. It is also seen that particle size increases with increase in calcination temperature.

3.4 Photocatalytic Application

The photocatalytic performances of Iron nanoparticles were demonstrated by room temperature UV-light degradation of MB. In a typical photocatalytic reaction 20 mg of iron nanoparticles was ultrasonically

dispersed in a 20 mL of aqueous MB (1 x 10⁻⁵ M) in a quartz reactor. The solution was magnetically stirred for 30 minutes in dark to saturate the adsorption/desorption of MB solution on catalyst. After 30 minutes of stirring the solution was irradiated with UV light accompanied by proper stirring to ensure uniform exposure of the suspension throughout the process. The reactor temperature is maintained low by circulating cold water. At periodic time interval 1.5 mL of aqueous MB solution was collected from the photo reactor and centrifuged to separate out any inorganic particles and the UV absorption was recorded. The degradation of MB was monitored by observing the characteristic absorption peak of MB at about 660 nm.

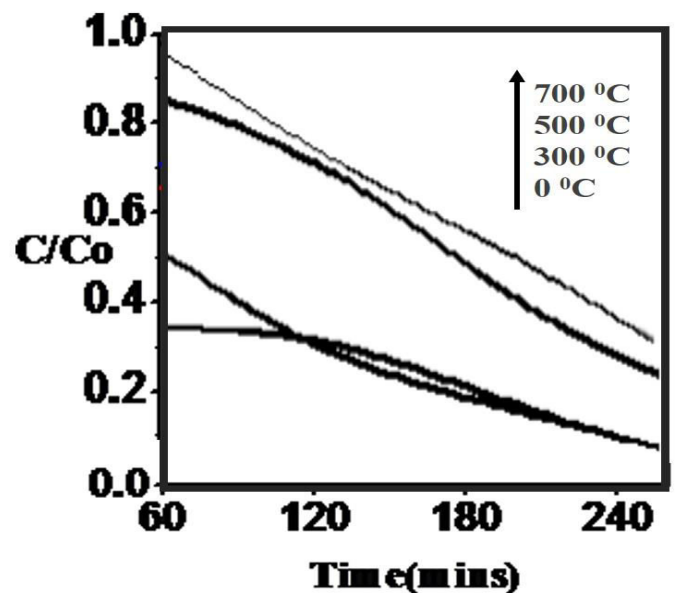


Fig.3. UV-visible spectroscopic changes of photocatalytic degradation

The UV-spectra clearly shows the peak at λ max of 660 nm gradually decreasing with increasing illumination time

indicating that the MB undergoes an obvious degradation with time. Fig. 10 depicts the approximate linear relationship of C/C_0 versus the irradiation time t . It can be expressed as follows: $\ln(C/C_0) = kt$, where k is the degradation reaction rate constant, C_0 and C are the initial concentration and the concentration at reaction time t for MB, respectively and the results are displayed in Fig. 10. The apparent decomposition rate constants as the evidence for the raspberry nanoparticles assembled found to be MB/iron nanoparticles suspensions were irradiated for 350 nm. The sample with the largest rate constant has the highest catalytic activity. The experimental results reveal the superior photocatalytic performances of F-300 material.

Generally, the photocatalytic efficiency of semiconductors is mainly dependent on the separation of photo generated electron-hole pairs and the transfer of the separated electrons from the photocatalyst to the organic pollutant. In our study, the sizes of the Iron nanoparticles as a photocatalyst play important roles in improving the photocatalytic performance. The ultrafine nanoparticles are responsible to reduce the recombination opportunities of electron-hole pairs. Nanoparticles assembled spheres are beneficial by increasing the absorption and increasing the number of reaction sites. Hence, the sample calcined at 300 °C shows excellent photocatalytic efficiency towards MB.

4. Conclusion

DRS is more convenient technique to characterize unsupported nanomaterials than UV-Vis absorption spectroscopy, since

it takes advantage of the enhanced scattering phenomenon in powder materials. Effects of light scattering in the absorption spectra of powder samples dispersed in liquid media can be avoided using DRS. If the absorption peak is not well resolved, even the use of derivative of absorption spectra does not guarantee the exact estimation of E_g , and can lead erroneous conclusions. Finally, the DRS technique does not require a powder sample to be dispersed in any liquid medium, so the material is not contaminated or consumed.

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