Synthesis and Characterisation of Fe_3O_4 Nanoparticles

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Abstract- **Magnetite (Fe_3O_4) nanoparticles were prepared by the chemical co-precipitation method from ferrous/ferric mixed salt-solution in alkaline medium. The conditions for the preparation of Fe_3O_4 nanoparticles were optimized and Fe_3O_4 nanoparticles were successfully prepared at optimum pH 11, initial temperature of 45 °C and at stirring rate of 500 rpm. The structure, morphology and optical properties of as prepared nanoparticles were characterized with the help of XRD, SEM-EDX, FT-IR and UV-Vis-DRS spectra. The band gap energy (Eg) from absorption spectrum was evaluated as 2.4 eV by the Tauc equation. Therefore the semiconductor nanoparticles can play a suitable role as a photocatalyst under light irradiation.**

Keywords: magnetite nanoparticles; photocatalyst; co-precipitation

I. INTRODUCTION

The synthesis of transition metals and their oxide nanoparticles (NPs) is of importance in nanotechnology research due to their potential and practical applications [1–6]. Among the materials, magnetite (Fe_3O_4) is a common magnetic iron oxide that has a cubic inverse spinel structure with oxygen forming a face-centered cubic closed packing and iron cations occupying interstitial tetrahedral sites and octahedral sites [7].

Fe_3O_4 nanoparticles have been the subject of intense interest because of their potential applications in several advance technological areas due to their promising physical and chemical properties. Generally, these properties depend on the size and structure of particles [8,9]. Fe_3O_4 nanoparticles find wide applications in the field of biomedical, as anticancer agent [10, 11], corrosion protective pigments in paints and coatings [12]. The magnetic atoms or ions in such solid materials are arranged in a periodic lattice and their magnetic moments collectively interact through molecular exchange fields, which give rise to a long-range magnetic ordering.

Magnetite (Fe_3O_4) is an important magnetic material that is widely used in different applications such as magnetic sensors [13], high density magnetic recording media [14], printing ink [15], ferrofluids [16], magnetic resonance imaging [17], catalysts [18] and especially in the biomedical field [19,20] due to its chemical stability and biocompatibility. There have been several methods reported to synthesize Fe_3O_4 powders, including co-precipitation [21], oxidation of $Fe(OH)_2$ by H_2O_2 [22], microemulsion [23], hydrothermal synthesis [24], sol–gel method [25], thermal decomposition of alkaline solution of Fe^{3+} chelate in the presence of hydrazine [26], pyrolysis of EDTA ferric sodium salt [27], reduction of hematite by CO [28], and organic solution phase decomposition of $Fe(acac)_3$, or $Fe(CO)_5$ [29–31] Thus, an efficient, economic, scalable, and non toxic, synthesis of Fe_3O_4 nanoparticles is highly preferred for potential application and fundamental research. However, some of these methods encountered problems with complicated equipment and/or long preparation time caused by multiple processing steps, deeming them economically unfeasible for large-scale production. Co-precipitation method is a method of synthesis of Fe_3O_4 which is easy to do with the success rate from 96 to 99.9%. Chemical co-precipitation can produced fine, stoichiometry particles of single and multi-component metal oxides. Chemical coprecipitation method has the potential to meet the increasing demand for the direct preparation of well dispersed (water-base) Fe_3O_4 nanoparticles and offer a low-temperature alternative to conventional powder synthesis techniques in the production of nanopaticles, and the sizes of nanopaticles can be well controlled. In this study, Fe_3O_4 magnetic nanoparticles were fabricated by co-precipitation method. UV, FTIR, SEM, XRD, and EDAX were used to characterize the synthesized nanoparticles.

2. EXPERIMENTAL

2.1 Materials

Ferric nitrate nona-hydrate ($Fe(NO_3)_3{\cdot}9H_2O$), Ferrous ammonium sulphate [$Fe(NH_4)(SO_4)$], Ammonium hydroxide (25%), Sodium hydroxide (NaOH), were purchased from Merck chemical company (Darmstadt, Germany)

2.2 Synthesis of Fe_3O_4 nanoparticles

Fe_3O_4 nanoparticles were synthesized by the chemical coprecipitation method. Aqueous solution of $Fe(NO_3)_3$ and [$Fe(NH_4)(SO_4)$] by a molar ratio of $Fe^{2+}/Fe^{3+} = 1/2$ was prepared and kept at room temperature. For this purpose, 8.08g $Fe(NO_3)_3.9H_2O$ and 3.921g [$Fe(NH_4)(SO_4)$] were dissolved in 100ml deionized water under nitrogen gas with vigorous stirring in a magnetic stirrer at 80ºC. After that, 100 ml of 25% $NH_3.H_2O$ was added to the solution until the pH of the solution reached to 11. The color of bulk solution turned from orange to black immediately. The magnetite precipitate was separated from the solution by centrifuge at 5000 rpm for 5 min and then washed with deionized water and ethanol independently and finally dried at 25°C under vacuum.

2.3 Characterization

The UV-Vis spectral analysis was performed on a JASCO, V-530 spectrophotometer. FT-IR spectra for the nanoparticles were recorded with Thermoscientific, Nicolet iS5 spectrometer in the region of 400-4000 cm^{-1}. X-Ray Diffraction patterns were recorded on XPERT-PRO diffractometer using Cu-Kα radiation of wavelength 1.54 A^0. SEM images (model: VEGA 3 TESCAN) along with EDAX (Bruker) were recorded. The elemental composition of the synthesized catalysts was analyzed using EDX (JOEL Model JED-2300).

3. RESULTS AND DISCUSSION

3.1. X-ray diffraction (XRD) studies

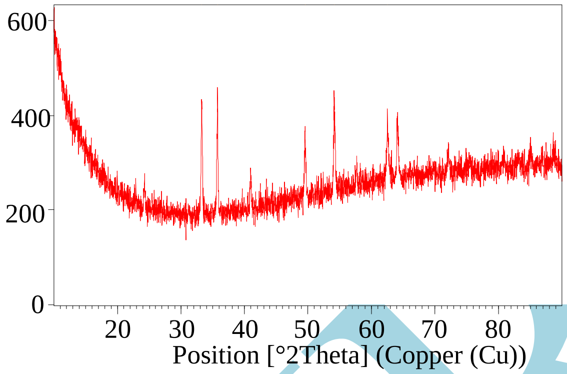

Figure 1. XRD pattern of Fe_3O_4 nanoparticles

The XRD of the sample (fig.1) shows the formation of Fe_3O_4, based on the comparison of their XRD patterns with the standard pattern of Fe_3O_4 (JCPDS 79-0418) [32]. The diffraction peaks at 30, 35.4, 43, 53.4, and 62.5 corresponding to (220), (311), (400), (422), (440) are quite identical to characteristic peaks of the highly crystalline cubic spinel structure of Fe_3O_4. It is clear that only the phase of Fe_3O_4 is detectable. There is no other phase such as $Fe(OH)_3$ or Fe_2O_3, which are the usual products in a chemical co-precipitation procedure. The positions and relative intensities of the reflection peak of Fe_3O_4 NPs agree with the XRD diffraction peaks of standard Fe_3O_4 samples, indicating that the black colored magnetic powders are magnetite nanoparticles. Sharp peaks also suggest that the Fe_3O_4 nanoparticles have good crystalline structure.

3.2. UV–visible spectroscopic studies

The UV–visible spectrum of Fe_3O_4 nanoparticles (fig. 2) shows an absorption band in the region of 330–450 nm, which originates primarily from the absorption and scattering of UV radiation by magnetic nanoparticles, which is in accordance with the previously reported literature [33]. The absorption band at 330 nm indicates the formation of nanosized particles.

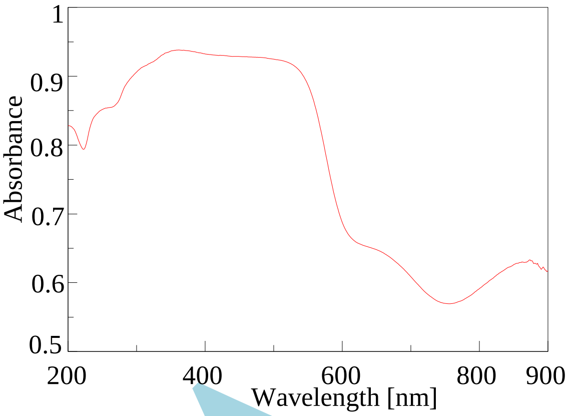

Figure 2. UV-Vis spectrum of Fe_3O_4 nanoparticles

3.3. FT-IR spectroscopic studies

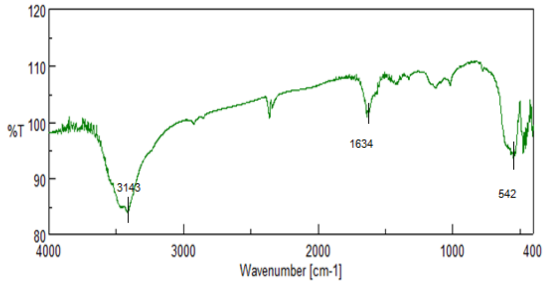

Figure 3. FT-IR spectrum of Fe_3O_4 nanoparticles

FT-IR spectra of Fe_3O_4 nanoparticle was depicted in figure 3. The characteristic band at 542 cm^{-1} was due to the Fe-O bond. The peak at 3410 cm−1 is ascribed to the stretching vibrations of hydrogen-bonded surface water molecules and hydroxyl groups. The peak at 1634 cm^{-1} is assigned to the O–H bending.

3.4. Scanning Electron Microscopy (SEM) Analysis:

SEM image of Fe_3O_4 (fig. 4) shows that spherical Fe_3O_4 nanoparticles obtained by this method are uniform in both morphology and size, but agglomerate to some extent because the extremely small particle size can get great specific surface area and surface energy. The picture appears that Fe_3O_4 particles composed of small particle, and the nanoparticles are uniform and monodispersed.

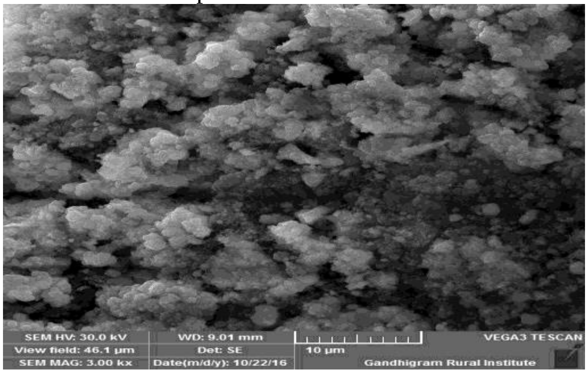

Figure 4. SEM image of Fe_3O_4 nanoparticles

3.5. EDAX Analysis

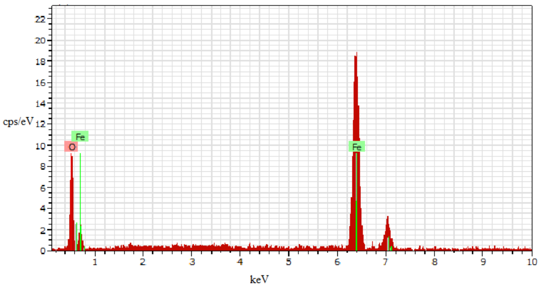

Figure 5. EDAX image of iron oxide nanoparticles

The elemental composition of Fe_3O_4 nanoparticles was qualitatively determined by energy dispersion spectrum (EDS) as shown in figure 5. It shows that Fe and O peak are obtained and atomic (%) ratio of Fe / O = 66.88/33.38

4. CONCLUSION

In summary, we reported chemical co-precipitation approach, which is simple and effective technique for preparing magnetite nanoparticles .The XRD and SEM results show that homogeneous sized Fe_3O_4 nanoparicles with spherical shape can be obtained. The synthetic process used is economical and environmental friendly, as it involves inexpensive and less toxic iron salts, which make Fe_3O_4 NPs as a promising material for engineering applications.

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