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Effect of nucleating agents and stabilisers on the synthesis of Iron-Oxide Nanoparticles-XRD analysis

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Abstract. Iron nanoparticles were made by using the modified coprecipitation technique. Usually the characteristics of synthesised particles depend upon the process parameters such as the ratio of the iron ions, the pH of the solution, the molar concentration of base used, type of reactants and temperature. A modified coprecipitation method was adopted in this study. A magnetic stirrer was used for mixing and the morphology and nature of particles were observed after synthesis. Nanoparticles were characterised through XRD. Obtained nanoparticles showed the formation of magnetite and maghemite under citric acid and oxalic acid as stabilisers respectively. The size of nanoparticle was greatly affected by the use of different types of stabilisers. Results show that citric acid greatly reduced the obtained particle size. Particle size as small as 13 nm was obtained in this study. The effects of different kinds of nucleating agents were also observed and two different types of nucleating agents were used i.e. potassium hydroxide (KOH) and copper chloride ($CuCl_2$). Results show that the use of nucleating agent in general pushes the growth phase of nanoparticles towards the end of coprecipitation reaction. The particles obtained after addition of nucleating agent were greater in size than particles obtained by not utilising any nucleating agent. These particles have found widespread use in medical sciences, energy conservation and electronic sensing technology.

Keywords: nanoparticles; iron oxide; maghemite; magnetite; X-ray diffraction; nucleating agents; stabilisers

1. Introduction

Massart (1981) first discussed the preparation of magnetite nanoparticles using a base solution with iron salts. In last decade, preparation of iron oxide nanoparticles has been extensively studied, not because of its scientific interest but because of the technological advances it is making, Laurent *et al*. (2008). Literature (Hafeli *et al*. 1997, Han *et al*. 2000, Lu *et al*. 2007, Mohammed M. Rahman 2011) reports that these particles found extensive applications in sensing technology, biomedical applications, storage media, water cleaning, magnetic inks. There are various ways to prepare iron oxide nanoparticles such as the coprecipitation method, the thermal decomposition method, the micro-emulsion method and the hydrothermal synthesis method. Details on each process can be found in Drbohlavova *et al*. (2009), Guo *et al*. (2001), Gupta and Gupta (2005),

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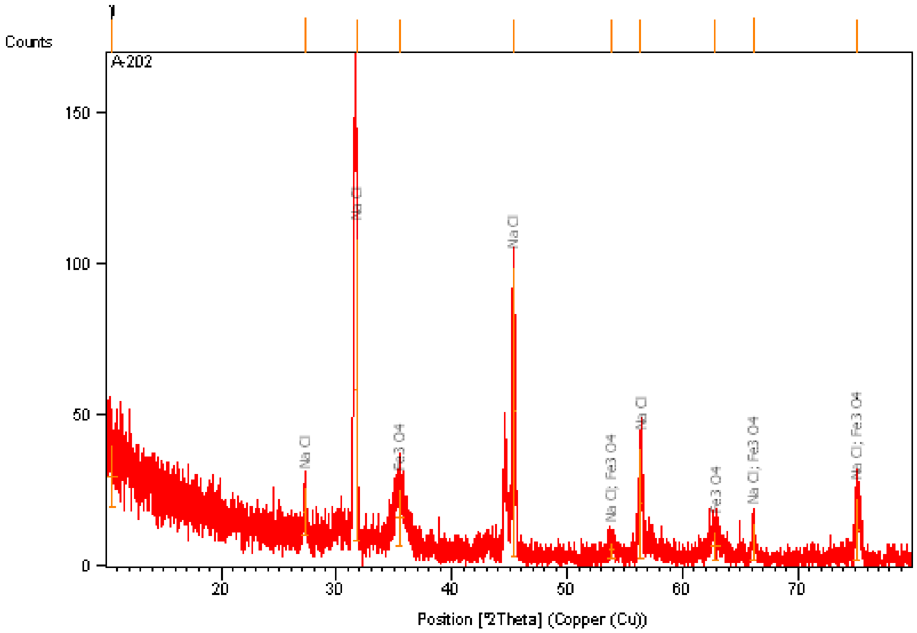

Fig. 1 Sample XRD diffractogram showing the formation of Fe3O4 using citric acid as stabiliser

2.3 X-ray diffraction

X-ray diffraction measurements were carried out on X'pert Pro using Cu Kα radiation. Powdered samples were placed on the sample holder. XRD data was collected in the range of 0 to 80 degrees. Calculations were performed using High score software. The average particle size was estimated by using the Scherer formula. The line width of most intensive peaks was considered for calculation purposes.

3. Result and discussion

The XRD technique was used to identify the size and nature of the obtained particles. Sample 1 was stabilised using citric acid and the XRD pattern (Fig. 1) confirmed the formation of magnetite nanoparticles with size of up to 18 nm (Scherer Formula, Eq. (1)). Sample 2 was stabilised using oxalic acid and the particle size obtained was upto 50 nm, XRD pattern (Fig. 2) confirmed the formation of maghemite.

$$\tau = (K\lambda)/(\beta \text{Cos } \theta) \quad (1)$$

where,

τ is the mean size of the particles,
K is a dimensionless shape factor=0.9
λ is the X-ray wavelength=1.54×10^{-10} A^{o}
β is the line broadening at half the maximum intensity (FWHM)
θ is the Bragg angle