

A FIRST COMPARISON BETWEEN THE MICROWAVE ASSISTED CO-PRECIPIATION TECHNIQUE AND THE TYPICAL SOLVOTHERMAL METHOD FOR THE PREPARATION OF $\text{Fe}_3\text{O}_4/\beta\text{-CYCLODEXTRIN}$ CORE/SHELL TYPE NANOPARTICLES

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Abstract. In this work $\text{Fe}_3\text{O}_4/\beta\text{-Cyclodextrin}$ core shell type nanoparticles are being prepared via two different synthesis methods and then the effectiveness of both methods is being discussed according to the resulting properties of the synthesized nanoparticles. The properties of the particles were characterized by FTIR, XRD SEM and VSM. The $\beta\text{-Cyclodextrin}$ (CD) grafted onto nanoparticle surface contributes to an enhancement of the adsorption capacity of Fe_3O_4 nanoparticles because of the strong complexation abilities of the $\beta\text{-CD}$ polymer with metal ions and of the hydrophobic cavity with organic contaminants through host-guest interactions.

The research and development of coated magnetic nanoparticles, recently has found wide application in the field of biomedicine in the construction of sensing devices. However, the magnetic nanoparticle technology can be applied in various fields related to biomedicine and environmental engineering. In this research, $\text{Fe}_3\text{O}_4/\beta\text{-Cyclodextrin}$ nanoparticles are being designed and synthesized via microwave assisted co-precipitation and are finally compared with the mainstream solvothermal method for the preparation of the specific type of magnetic nanoparticles. Referring to the size of the magnetic core of the nanoparticles (paramagnetic in micro scale / 0.1 to 1000 microns or superparamagnetic in nano-scale / 1 to 100 nanometers) and the size distribution of them, which is the backbone of the process, the synthesis can be performed by adapting the parameters of the aforementioned method.

The microwave co-precipitation method is a quite novel method which benefits from the uniform heating properties of the microwave technology. Microwaves are a region of the electromagnetic waves with a wavelength between 0.1 and 100 cm, corresponding to frequencies between 0,3-300 GHz. The operating principle of this method is based on the high power of microwave and the heating from the inside of the body which is penetrated by microwaves, which leads to oscillation of the bonds of the molecules. In fact the bonds which vibrate are those of the fluids even those inherent in the solid bodies and the outcome is the heat of the material.

In this work, the nano-particles designed are strictly magnetic and their core is magnetite with superparamagnetic properties. Magnetite is an iron oxide and a strong ferromagnetic material with the chemical formula Fe_3O_4 . While obtained in nanoparticles <50nm it exhibits superparamagnetic behavior. In magnetite, the O^{2-} and Fe^{2+} , Fe^{3+} ions form an inverse spinel crystallographic structure [1]. As referring to the coating material, cyclodextrins (CDs) are oligosaccharide molecules deriving from the chemical processing of starch. $\beta\text{-CD}$ is a cyclic oligosaccharide consisting of 7 glucopyranose units, which are joined together by α (1-4) linkage forming a torus-shaped ring structure with a hydrophilic exterior and a hydrophobic cavity. It can form inclusion complexes with a wide variety of organic compounds in its hydrophobic cavity through host-guest interactions [2,3]

These fascinating properties make them promising for applications in drug carrier systems, nanoreactors, bioactive supramolecular assemblies, molecular recognition, and catalysis. For biomedical applications, the use of an organic coating compatible with human tissue is necessary and thus the use of materials such as cyclodextrins for coatings is considered more suitable. Considering modern water treatment technologies, functionalized magnetic nanocomposites have received many attentions recently for use in the adsorption of both organic and inorganic pollutants [4,5]. Magnetic nano-adsorbents have the advantages of

both magnetic separation techniques and nano-sized materials, which can be easily recovered or manipulated from complex multiphase systems with an external magnetic field.

Figure 1. illustrates the first characterization results (FTIR and VSM) for nanoparticles synthesized via both techniques. On the left scheme is depicted the effect of preparation time, and temperature modifications on the structure of the nanoparticles for both synthesis methods. From the results it is evident that both the microwave technique and the solvothermal method are quite effective for the preparation of $\text{Fe}_3\text{O}_4/\beta\text{-CD}$ nanoparticles but the synthesized nanoparticles might occasionally not be fully coated if the preparation time and temperature are not precisely adjusted. The VSM results exhibit the superparamagnetic abilities of magnetite nanoparticles in both cases.

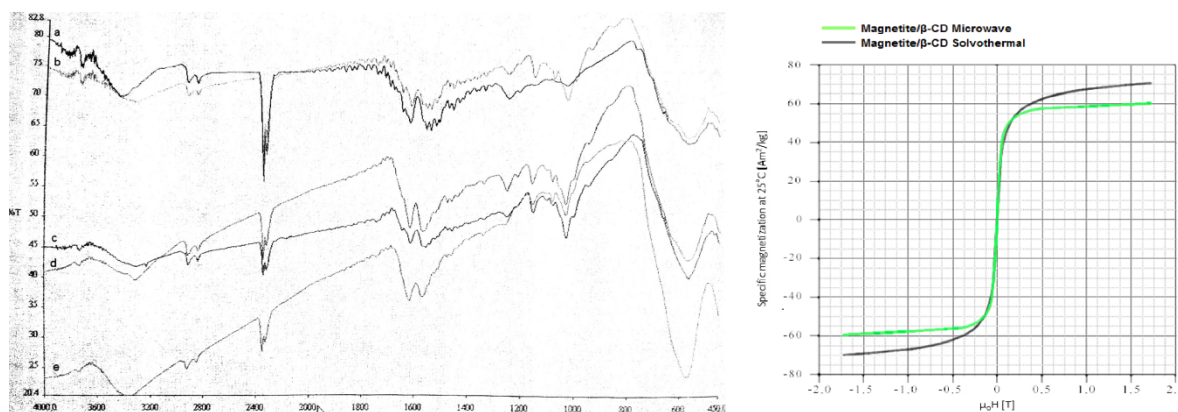


Fig.1. Left: FTIR characterization results for nanoparticles prepared via (a), (e) microwave assisted co-precipitation and (b), (c), (d) solvothermal method with stirring and ultrasound dispersion. Right: VSM characterization results for both types of nanoparticles

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