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Analysis of chemical processes for the synthesis of magnetite for biomedical applications.

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Abstract. This article demonstrates the evaluation of wet chemical routes to produce magnetic nanoparticles of iron oxide and surface chemistry characterization by infrared spectroscopy (IR). Its potential use in biomedicine as contrast agents or to deliver drugs in localized medical treatments, which reduce the toxicity associated with cytotoxic drugs, is also evaluated.

1. Introduction

In a broad spectrum of applications, nanotechnology increasingly innovates the biomedical field. Nanoparticles used to detect tumors, activation by alternate magnetic fields as a cancer treatment, and their accumulation, vectorization and functionalization for drug targeting, are some of the more important applications in this field. But equally, their major limitations are studied to optimize their application; among them, the half-life in blood which, when injected into the bloodstream, is recognized and phagocytosed in the liver and then removed, a biological process, which—over long periods—prevents the action of the desired biomedical application [1].

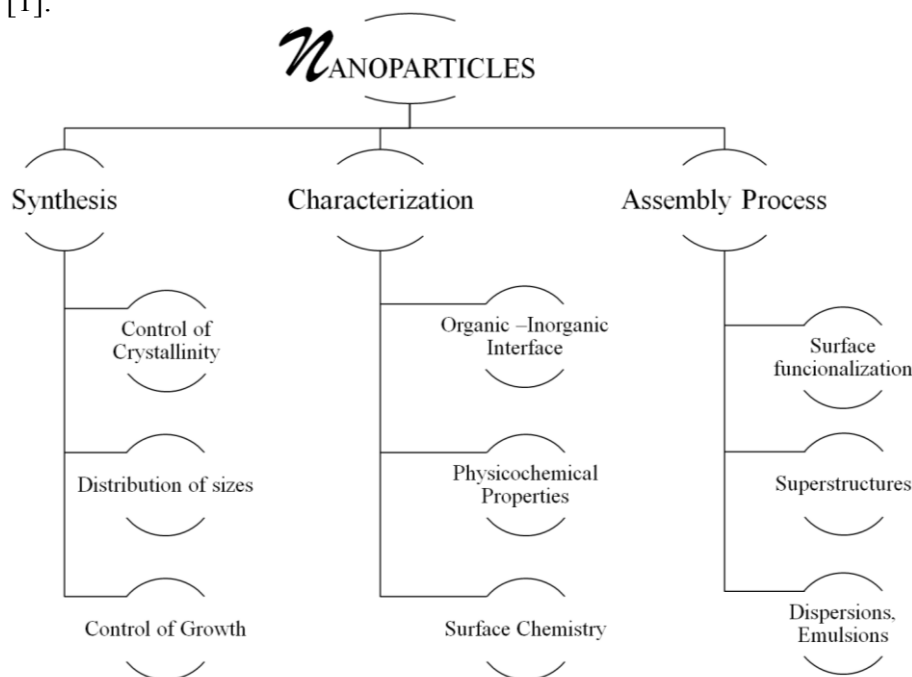


Figure 1. General scheme of synthesis

The half-life of the nanoparticles in contact with the blood depends on their size, the tendency to form aggregates and the nature of their coating. Hence, the importance of studying the feasibility and viability of the chemical pathways used for the synthesis of magnetite, since this will establish the shape, size, distribution, degree of crystallinity and surface chemistry, which determine the magnetic properties of the material and, thus, prolong its half-life to generate applications that impact current biomedical techniques [2].

These nanoparticles are widely used in the market, due to the possibility of their use in high-resolution biomedical images, implemented in the Nuclear Magnetic Resonance techniques, usually employed as contrast agents.

However, the synthesis process presents some problems, such as the lack of control of the average particle size and their broad size distribution.

2. Objectives

In this paper, we analyze and verify the chemical processes associated with the synthesis of magnetic nanoparticles for biomedical applications. It also raises the formulation and design of the experimental setup, the further characterization of the samples, their composition and superficial chemistry through the infrared-spectroscopy (IR) technique and the assessment of potential enhancements to the methods involved and the main variables, as shown in Figure 2.

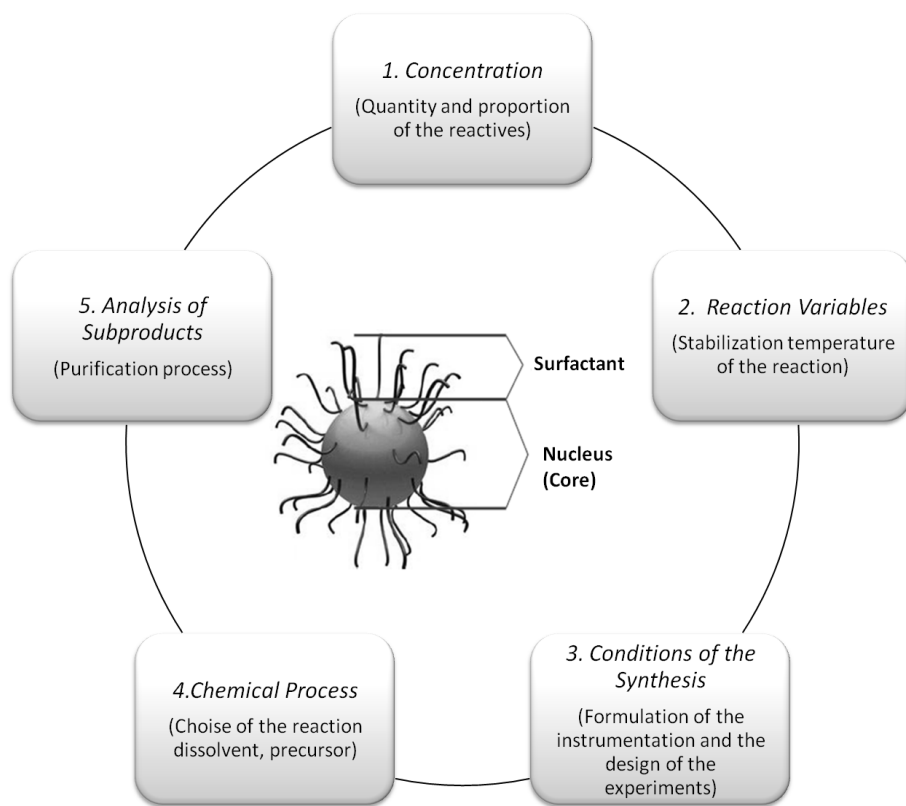


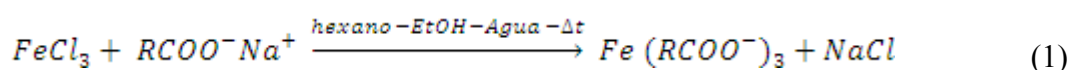
Figure 2. Diagram of the action mechanism in the synthesis of magnetite, with the necessary variables and parameters.

3. Methodology and Formulation of the Chemical Process

There are several methods used to obtain the nanoparticles, among which are physical, chemical, and gas-phase methods. The method used in this work is the synthesis of magnetite through thermal decomposition, described by Roca, A G; Morales, M P; Serna, C [3].

3.1 Description of the Thermal-Decomposition Process in an Organic Iron Precursor

The first stage of the synthesis of magnetite using iron oleate III ($\text{Fe}(\text{ole})_3$) is formed. To do this, we add 2 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and 4 g of sodium oleate to a mixture of dissolvents, formed by 8 ml of EtOH , 6 ml of distilled water and 14 ml of hexane. The mixture was heated to reflux during four (4) hours, as shown in Equation 1.



After the completion of the reaction, the aqueous phase was eliminated and the organic phase was washed several times with deionized water to remove all the ions that remained in the supernatant.

The experimental setup initially began with a heating mantle to 200°C . After characterizing it (thermal profile), it was decided that the assembly would be improved using a thermostatic bath to provide more precise temperature control. We used a glass agitator propeller tip at 200 rpm. A four-mouth, round-bottom reaction flask was designed and constructed to comply with the reaction conditions (hot injection of reagents and surfactants, as well as the conditions of inert atmosphere). A nitrogen-gas flow and a reflux condenser were available and the temperature measurements were obtained with a J-type thermocouple. All the experimental setup and the complete synthesis were performed in a fume hood, due to the volatility of the solvents, as shown in Figure 3.

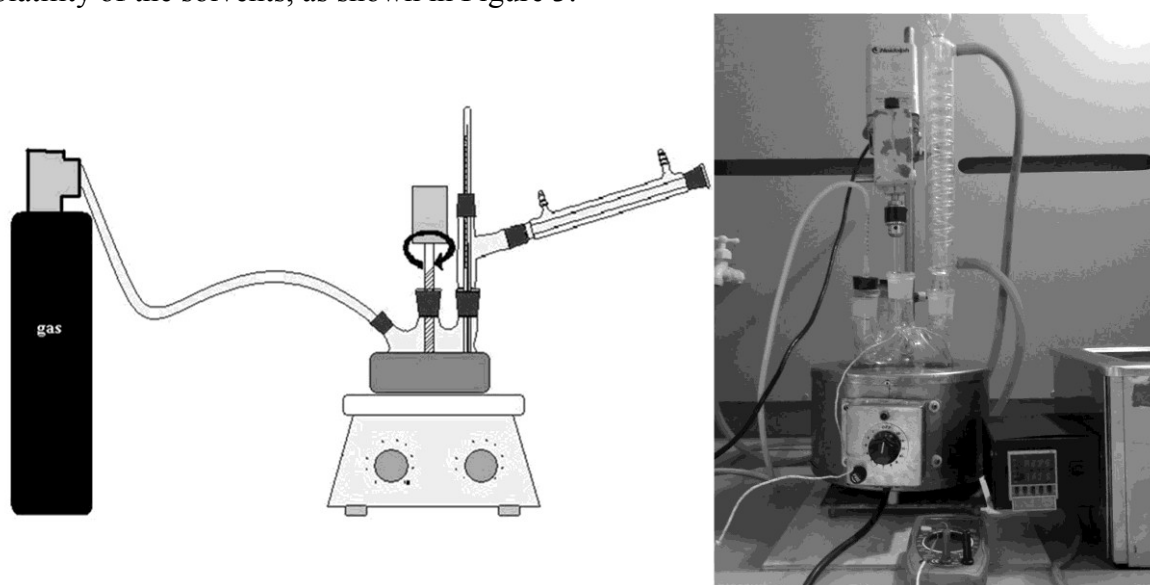


Figure 3. Experimental setup of the thermal-decomposition method in an organic iron precursor

3.2 Description of the Salt Coprecipitation Method

This method involves the addition of an Fe^{2+} and Fe^{3+} salt solution on a basic medium in excess. The advantages of this method are its simplicity and low cost. The method is described by García, L A; Rodríguez, O S; Betancourt, R; Saldívar, R [4].

For this reason, we performed an optimization of the method. The chemical-coprecipitation technique consists of mixing a 2.333 g ferric-chloride-hexahydrate solution ($FeCl_3 \cdot 6H_2O$) and 0.8580 g of ferrous-chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$) with an 0.1 M concentration and a molar relation of 2:1, with continuous mechanical agitation. Later, this mixture was heated to a temperature of 70°C. A 10% volume solution of sodium hydroxide ($NaOH$) was added carefully from a burette. Instantly, we observed the formation of a dark precipitate as the base fell into the mixture, producing flocculation. The precipitate was washed several times with distilled water to remove the ions and the sodium hydroxide in the supernatant. The washed magnetite was kept in suspension to facilitate its peptized process.

The surfactant preparation was carried out through the peptization method. Oleic acid as the surfactant, hexane as the carrier liquid and suspended magnetite as the magnetic material were used.

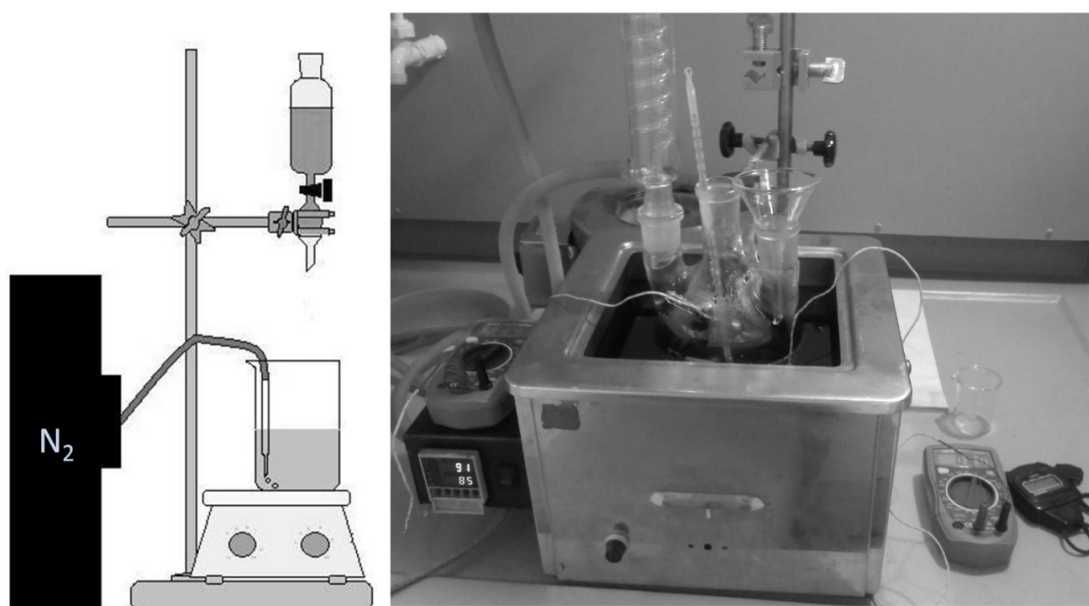


Figure 4. Experimental setup for the synthesis of magnetite, through the coprecipitation method

4. Characterization

We observed that the superficial chemistry of the magnetite presented an absorbance peak at 610 nm, which corresponds to a coordinated double bond ($C=O$) (Figure 5b), which means a coating of oleic acid to this substance, showing the steric isolation required. This also shows that the coprecipitation and thermal decomposition results belong to the bands of natural absorbance of magnetite (Figure 5a). Similarly, we compared two samples of magnetite

powder, produced by the methods of coprecipitation and thermal decomposition, noting that the latter method is much closer to the values reported in the literature [5].

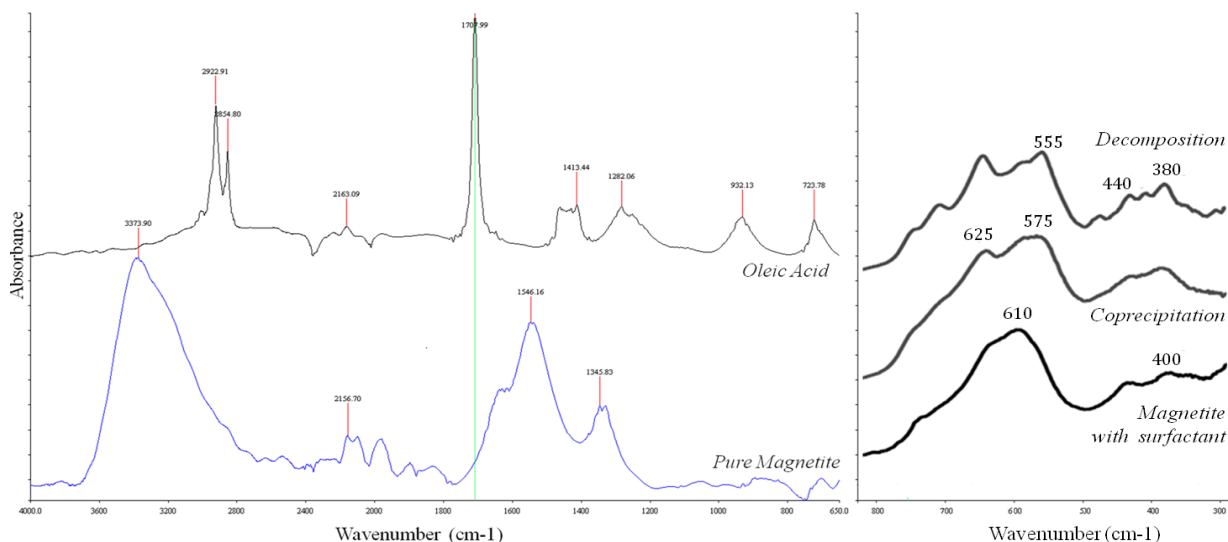


Figure 5. Results of the Infrared Spectroscopy. a) Absorbance peaks for the reactants and the products. b) Absorbance peaks of the products obtained through different chemical pathways.

5. Results and Discussion

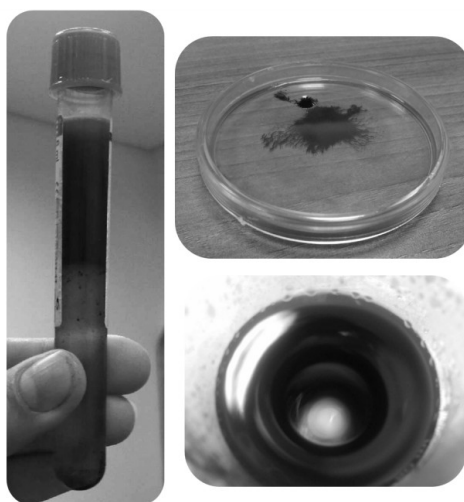


Figure 6. Experimental results obtained: Magnetite in the carrier medium

Figure 6 shows the experimental results of the synthesis of magnetite through thermal decomposition in the middle of an organic precursor. It shows the hydrophobic character of the colloid, due to an organic (hexane) carrier medium. This feature implies further exchange in an aqueous medium, since the suspension must be biocompatible. Additionally, it shows the peptization process of the magnetite, after six (6) hours of continuous agitation and constant temperature. As shown in Figure 5, the results of the Infrared Spectroscopy (IR) for these final products correspond to magnetite with surfactant on its surface [6].

6. Conclusions

In evaluating the viability and feasibility of the chemical pathways to synthesize magnetite, we present the following results:

In thermolysis with organic precursors, we observed that, although the method is viable, it requires production time of more than six (6) hours and the difficulty of maintaining a stable temperature of more than 300°C hinders its implementation. Similarly, we propose changing the solvent (hexane) in future tasks because of the difficulty of finding it in the local market, due to restrictions in handling.

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8. References

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