P4 - Optimization of the synthesis of functionalized magnetic nanoparticles and their characterization

M.T. Siles Cordero, M.M. López Guerrero, E.I. Vereda Alonso, A. García de Torres, J.M. Cano Pavón

Department of Analytical Chemistry, Faculty of Sciences, University of Málaga Andalucía Tech. Campus de Teatinos S/N. 29071 Málaga. España. Email: mtsiles@uma.es

Abstract

Magnetic nanoparticles (MNPs), a new kind of nanometer-sized material, are widely used in the fields of biotechnology and biomedicine. These particles are superparamagnetic, which means that they are attracted to a magnetic field, but retain no residual magnetism after the field is removed. In recent years, MNPs have been studied because of their potential applications as magnetic carries for various biomedical uses such as cell and DNA separation, drug delivery system, magnetic resonance imaging, bio separation, and preconcentration of various anions and cations.[16] Magnetite, Fe_3O_4 is the magnetic material most used in biomedical application due to its several interesting properties such as great chemical stability, low toxicity, high saturation magnetization to allow its manipulation with an external field, ability to bond with different molecules to surface functionalized, biocompatibility and the heating ability in presence of a field, which made it an candidate for hyperthermia treatment.[17] The quality of the MNPs is extremely sensitive to synthesis conditions, such as reaction time, temperature, reagent concentration, quality of solvent, etc. In order to optimize the synthesis to obtain a high yield with the smallest size of these nanoparticles, a factorial design 24 (16 experiments) was developed. Two level factorial designs have many advantages, for example they are more efficient than studying one factor at a time. A factorial design allows the effect of several factors and even interactions between them to be determined with the same number of trials as are necessary to determine any one of the effects by itself with the same degree of accuracy. The selected factors and their corresponding ranges were determined after preliminary experiments. These factors were the reaction time, volume and concentration of NH_3. The lower and upper values given to each factor are shown in Table 1.

Table 1. Factor levels in the screening

Variable	Lower	Upper
Reaction time (min)	15	60
NH_3 volume (mL)	10	40
NH_3 concentration (%)	10	30

In order to quantify the synthesis yields a new method by Fourier transform infrared spectrometry (FTIR) has been developed for the direct determination of Fe-O by absorbance measurements in KBr pellets. The procedure is based on the use of the ratio between the absorbance of the characteristic band of iron oxide and three characteristics bands of a nitrate internal standard added to samples.

Simple linear regression (SLR) and multiple linear regression (MLR) were proved with each of the three area ratios chosen between the analyte and the internal standard, obtaining the best results with the MLR method.

For validation purposes the different yields of the synthesis were analysed by the proposed IR method and HR CS ETAAS using solid samples auto sampler. The results obtained were satisfactorily compared. At the same time, the size of the MNPs obtained was determined by Scanning Electron Microscopy (SEM).

Keywords: MNPs, coprecipitation synthesis, experiments design

Acknowledgements The authors thank the Spanish Ministerio de Ciencia y Tecnología (MCyT project no. CTQ2013-44791-P) for supporting this study and also FEDER funds. And Universidad de Málaga, Plan Propio.

---

[16] M.H. Mashhadizadeh, Z. Karami, J. Hazard. Mat., 190 (2011) 1023-1029.

[17] P.E. García Casillas, C.A. Rodríguez González, C. A. Martínez Pérez, Infrared Spectroscopy of Functionalized Magnetic Nanoparticles, In: Infrared Spectroscopy – Materials Science, Engineering and Technology, Ed by Prof. T. Theophile (2012) ISBN 978-953-51-0537-4.