ICAM2009

11th International Conference on Advanced Materials

Rio de Janeiro Brazil
September 20 - 25

A Chemometric study of iron oxide nanoparticles synthesis

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Abstract – In this work Fe_3O_4 nanoparticles were obtained from a suspension of $FeSO_4.7H_2O$ under sonication, with addition of an NaOH solution. The synthesis parameters were optimized using a chemometric approach based on a factorial design, which allows verification of individual variables and their interactions, and permits achieving optimal conditions saving time and costs. It was observed that the nature of the base and its concentration play key roles in phase formation and crystallite size. Moreover, an important synergetic effect of base concentration and ultrasound frequency was observed.

The possibilities of application for iron oxide nanoparticles have greatly increased in recent years. In the clinical area, these particles are being used in a variety of ways, notably as contrast agents for magnetic resonance imaging (MRI). Nanometric-scaled materials for MRI contrast agents are generally magnetite-like structures (Fe_3O_4) with 10-300 nm in diameter[1]. It is well established that synthesis conditions determine particle size and morphology – and consequently their applications. In this work nanoparticles of Fe_3O_4 were obtained from a suspension of $FeSO_4.7H_2O$ under sonication, with addition of an NaOH solution. The nanoparticle synthesis was studied for the first time using a chemometric approach based on a factorial design. The factorial design is an advantageous strategy to optimize synthesis parameters because it allows verifying the effects of the individual variables and their interactions, and permits achieving optimal conditions, saving time and costs.

For the full 2^3 factorial planning, the variables studied were the type of base (NaOH 1.0 $mol.L^{-1}$ or $N_2H_4.H_2O$), ultrasound frequency (40 or 593 KHz) and reaction time (40 or 80 min); crystallite size was the monitored response. The results of the first planning showed that Fe_3O_4 was obtained only when NaOH was employed, suggesting that the nature of the base is a relevant factor. A second planning was elaborated considering the NaOH concentration (0.5 and 1.0 $mol.L^{-1}$), in addition to ultrasound frequency and reaction time; crystalline Fe_3O_4 was obtained and no secondary phase was observed for all 8 experiments.

Moreover, smaller crystallite sizes (21.6 nm) were achieved employing the following experimental conditions: NaOH 1.0 $mol.L^{-1}$, 593 KHz and 40 min. Concerning statistical analysis, it was verified that the base concentration was the most import factor, with a negative effect, i.e., the higher the base concentration, the smaller the crystallite size (Fig. 1). Likewise, the ultrasound frequency is a relevant factor and presents a negative effect. It is interesting to point out that the interaction of these two factors is quite important, and positive, indicating a synergetic interaction.

Particle morphology was examined by SEM (Fig. 2), which showed clusters (around 50 nm) of iron oxide nanoparticles.

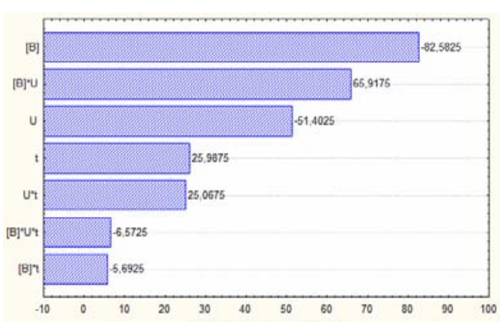

Fig. 1: Pareto chart of effects. [B] refers to base concentration, U is ultrasound frequency, and t is time.

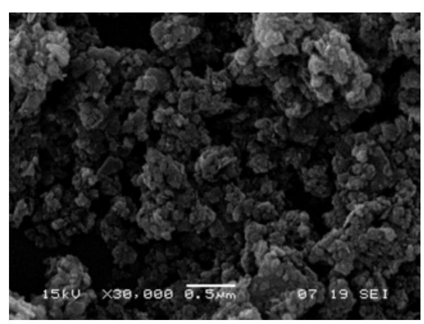

Fig. 2: SEM picture of magnetite nanoparticles.

References

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