# Optimization of the synthesis of Magnetic Nanoparticles by Solid Sampling High Resolution Continuum Source Graphite Furnace Atomic Absorption Spectrometry and Multi-Response Surface Methodology

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**SUMMARY:**

Magnetic nanoparticles (MNPs) are a new kind of nanometer-sized materials superparamagnetic with potential applications as magnetic carries for various biomedical uses, wastewater remediation, preconcentration of various anions and cations, etc.[1] The excellent properties of MNPs are strongly influenced by the size of the nanoparticles. Another important factor is the amount of iron present. In this work a simple, rapid and inexpensive approach was developed for direct determination of Fe concentration and particle size of solid MNPs by solid sampling high resolution continuum source graphite furnace atomic absorption spectrometry (SS-HR-CS-GFAAS). A new strategy in evaluating area and slope of the obtained absorbance signals for a line of Fe (352.614 nm) with low sensitivity was developed for both determinations. For this purpose, five furnace program parameters, atomization heating rate, atomization temperature, pyrolysis heating rate, pyrolysis temperature and hold pyrolysis time, were optimized with the employ of two multiple response surface designs. The response variables chosen were: atomization signal area/weighted mass of MNPs and the inverse of the upslope of the atomization signal/weighted mass of MNPs. With the optimized furnace parameters, good calibration curves ($R > 0.997$) were obtained with liquid iron standards (for Fe determination) and with MNPs samples with certified size particle (for size particle determination). The determination of the MNPs size was validated by transmission electron microscopy (TEM).

The developed method was used in the optimization of the synthesis of MNPs by a multi-response surface methodology. The MNPs were synthesized by a modified chemical co-precipitation method, where three factors (reaction time, volume and concentration of $NH_3$) were investigated using two responses variables (Fe concentration and 1/size of MNPs). From this study was concluded that the

highest Fe concentrations were obtained with low volumes of $NH_3$, and the smallest sizes of MNPs were obtained with high $NH_3$ concentrations. Therefore, the optima esperimental conditions obtained were: reaction time: 55 min; $NH_3$ volume: 10 mL; and $NH_3$ concentration 30%. The synthesis of MNPs with the optimized parameters result in MNPs with around 65% in Fe and a size of 17 nm. Optimum size ranges from 13 to 18 nm are preferable for their use as absorbent in solid phase extraction because of their higher magnetic moment per particle.[2]

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