P19 – Characterization of solid magnetic nanoparticles by high resolution continuum source electrothermal atomic absorption spectrometry (HR-CS-ETAAS)

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Abstract

Nanometer-sized materials have attracted substantial interest in the scientific community because they offer a highly active surface area to volume ratio enable them to have a wide range of potential applications because their great extraction capacity and efficiency. Magnetic nanoparticles (MNPs), as a new kind of nanometer-sized materials, are superparamagnetic, which means that they are attracted to a magnetic field, but retain no residual magnetism after the field is removed. Therefore, suspended superparamagnetic particles adhered to the target can be removed very quickly from a matrix using a magnetic field.[18] 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.[19] The excellent properties of MNPs are strongly influenced by the particle size. Transmission electron microscopy (TEM), scanning electron microscopy (SEM) and X-ray diffraction (XRD) are the main techniques used for the characterization and observation of the size and shape of the MNPs. The objective of this study was to develop a method for direct determination of Fe concentration and particle size of solid MNPs by application of solid sampling high resolution continuum source graphite furnace atomic absorption spectrometry (HR-CS-GFAAS). A new strategy in evaluating the area and the slope of the obtained absorbance signals for a line of Fe with low sensitivity was developed for the determination of both, iron concentration in solid MNPs and their average particle size. For this purpose, five furnace program parameters, atomization temperature, heating rate, and pyrolysis temperature, heating rate and hold pyrolysis time, were optimized with the employ of two multiple response surface designs. The response parameters chosen were: atomization signal area/weighted mass of MNPs and the inverse of the slope of the atomization signal/weighted mass of MNPs. The optimized parameters are shown in Table 1.

Table 1. Optimized graphite furnace program

Step	Temperature (°C)	Heating time (°C s^{-1})	Hold time (s)
Drying	150	5	35
Pyrolysis	1050	75	5
Auto-zero	1050	0	5
Atomization	2500	1275	12
Cleaning	2600	500	4

With these optimized parameters good calibration curves 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 MNP size was validated by SEM. This method is being employed in the optimization of the synthesis of MNPs by the coprecipitation method.

Key Words: HR-CS-GFAAS, MNPs, Fe concentration, particle size

[18] J.S. Suleiman, B.Hu, H. Peng, C. Huang, Talanta 77 (2009) 1579-1583.

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