



Magnetic dispersive solid phase extraction with graphene/ZnFe₂O₄ nanocomposite adsorbent for the sensitive determination of mercury in water and fish samples by cold vapor atomic absorption spectrometry

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ARTICLE INFO

Keywords:

Mercury
Graphene/ZnFe₂O₄ nanocomposite
Solid phase extraction
Fast contact time
CV-AAS

ABSTRACT

In this study, the magnetic graphene/ZnFe₂O₄ composite material was synthesized and characterized by X-ray diffraction, scanning electron microscopy equipped with energy dispersive (X-ray spectrometry), Brunauer, Emmett and Teller surface area and zeta potential measurements. This material was investigated for the first time as an adsorbent for the magnetic dispersive solid phase extraction of Hg(II) ions from various samples. Mercury determination in solutions was made by using cold vapor atomic absorption spectrometry. The experimental conditions affecting the preconcentration of mercury were optimized. They were found as follows: sample pH, 6; adsorption and elution contact time, 3 min; elution solvent, 5 mL of 2 mol L⁻¹ HCl. The preconcentration factor, detection limit, and precision (as RSD%) of the method were found to be 30-fold, 10 ng L⁻¹, and 2.7% for mercury, respectively. The developed method was successfully verified by analyzing certified reference materials (DOLT-4 Dogfish Liver and NCS ZC 81002b Human Hair) and by recovery experiments in SPS-WW1 wastewater, tap water, well water, wastewater and fish samples.

1. Introduction

Mercury (Hg) is one of the most toxic elements affecting the health of human and the ecosystem is released into the environment from both anthropogenic and natural sources such as the weathering of rocks, volcanic events, geothermal activity, coal and oil combustion, waste incineration, metal refining and manufacturing [1–3]. The toxicity of Hg depends on the chemical form in which it is taken up by the human body. Generally, organic forms of Hg are much more toxic than inorganic forms [4]. The prominent mercury sources for human come from dental amalgams, pharmaceuticals, cosmetics, and food, primarily contaminated fish [5]. Hg is also transferred into the human food chain through crops and water. It has been reported to cause various neurodegenerative diseases, such as, amyotrophic lateral sclerosis, Alzheimer's disease, and Parkinson's disease [1]. Long-term dietary consumption of mercury-contaminated food can lead to a series of symptoms of chronic mercury poisoning and cause damage to the human body, and even lead to death [6]. The WHO guidelines prescribe a health limit value of 1 µg/L for Hg in drinking water [4]. The development of reliable methods for mercury determination in water and food samples is of great importance due to their high toxicity and biomagnification factor (up to 10⁶) [3, 4, 7]. In the Codex Standard, the

permissible level of inorganic mercury in salt, food grade is 0.1 mg kg⁻¹ [8].

The preconcentration and separation of analytes prior to measurement are frequently needed even with a highly sensitive detection system because of the extremely low concentration levels of mercury in many types of samples and its potentially complicated matrix [3]. The solid phase extraction (SPE) method has superior properties such as rapid phase separation, high selectivity, low cost, small use of organic solvents, simple extraction, high recovery, high preconcentration factor and automation with more detection techniques [9–12]. Magnetic SPE (MSPE) is a new type of SPE based on the combination of magnetic inorganic material and non-magnetic adsorbent. An adsorbent is placed in a solution or suspension containing the target analytes. After adsorption of the analytes, the adsorbent is separated from the solution using an external magnetic field. The MSPE has an excellent adsorption efficiency and provides rapid separation from the sample matrix by an external magnetic field instead of filtration or centrifugation [13–15].

The exploration of nanomaterials is an active research area in SPE. These new materials as adsorbent in sample preparation are important to obtain more selective materials with higher adsorption capacity. They have a high specific surface area, chemical stability, and a lot of adsorption sites [14, 16, 17]. Nano-sized particles are characterized by

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enhanced reactivity, a large surface area to volume ratio which speeds up sorption kinetics, and unique thermal, mechanical and electronic properties [1, 18]. Rapid mass transfer can be obtained due to the sufficiently large contact area between the sorbents and the analytes, which is beneficial for rapid equilibrium [13]. The most important products of this sorbent group are carbon-based nanomaterials. These materials include fullerenes, graphene and graphene based materials, carbon nanotubes, carbon nanocones-disks and nanohorns, carbon nanofibers, as well as their functionalized forms. However graphene, a two-dimensional material with a single layer framework of six membered carbon rings, possesses excellent thermal, mechanical and electrical properties [19]. Graphene and graphene-based materials have attracted considerable attention as a novel material for use in sample

preparation due to their remarkable properties. These materials have many advantages such as large specific surface area, high adsorption capacity, strong affinity, good reusability and reproducibility [15, 17]. They have been used as SPE adsorbents for environmental and food analyses [20–24]. The introduction of magnetic properties into graphene is important to separate the mother solution from the adsorbent. Various magnetic nanoparticles combined with graphene have been used successfully as adsorbents for removing organic compounds. Yamaguchi et al., [25] synthesized magnetic a MnFe_2O_4 -graphene hybrid and used it as an attractive adsorbent for the removal of glyphosate from contaminated water for water treatment and purification processes. Ye et al., [26] used graphene- Fe_3O_4 for the preconcentration of phthalate acid from water prior to gas chromatography-mass

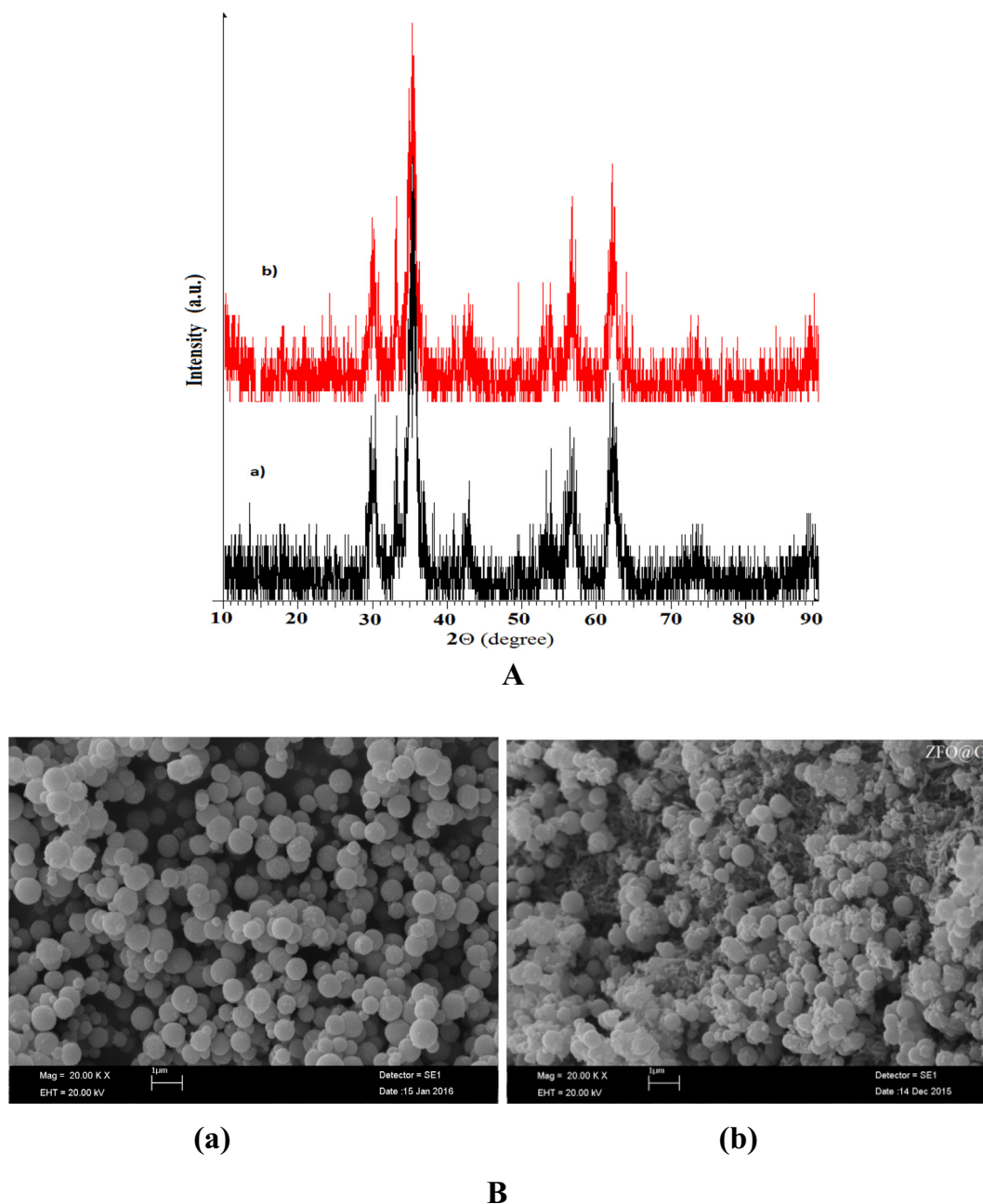


Fig. 1. A. XRD patterns of (a) ZnFe_2O_4 and (b) $\text{G/ZnFe}_2\text{O}_4$.

B. SEM images of (a) ZnFe_2O_4 (b) $\text{G/ZnFe}_2\text{O}_4$ materials.

C. (a) EDX spectra of ZnFe_2O_4 , (b) $\text{G/ZnFe}_2\text{O}_4$, (c) electron probe micro analysis images of Zn, C, O and Fe elements for $\text{G/ZnFe}_2\text{O}_4$ composite.

D. N_2 adsorption/desorption isotherm of $\text{G/ZnFe}_2\text{O}_4$,

E. The zeta potential versus pH curves for ZnFe_2O_4 and $\text{G/ZnFe}_2\text{O}_4$.

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