



Optimization of ultrasound-assisted dispersive solid-phase microextraction based on nanoparticles followed by spectrophotometry for the simultaneous determination of dyes using experimental design



Arash Asfaram^a, Mehrorang Ghaedi^{a,*}, Alireza Goudarzi^b

^a Chemistry Department, Yasouj University, Yasouj 75918-74831, Iran

^b Department of Polymer Engineering, Golestan University, Gorgan 49188-88369, Iran

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ABSTRACT

A simple, low cost and ultrasensitive method for the simultaneous preconcentration and determination of trace amount of auramine-O and malachite green in aqueous media following accumulation on novel and lower toxicity nanomaterials by ultrasound-assisted dispersive solid phase micro-extraction (UA-DSPME) procedure combined with spectrophotometric has been described. The Mn doped ZnS nanoparticles loaded on activated carbon were characterized by Field emission scanning electron microscopy (FE-SEM), particle size distribution, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR) analyses and subsequently were used as green and efficient material for dyes accumulation. Contribution of experimental variables such as ultrasonic time, ultrasonic temperature, adsorbent mass, vortex time, ionic strength, pH and elution volume were optimized through experimental design, and while the preconcentrated analytes were efficiently eluted by acetone. Preliminary Plackett–Burman design was applied for selection of most significant factors and giving useful information about their main and interaction part of significant variables like ultrasonic time, adsorbent mass, elution volume and pH were obtained by central composite design combined with response surface analysis and optimum experimental conditions was set at pH of 8.0, 1.2 mg of adsorbent, 150 μ L eluent and 3.7 min sonication. Under optimized conditions, the average recoveries (five replicates) for two dyes (spiked at 500.0 ng mL^{-1}) changes in the range of 92.80–97.70% with acceptable RSD% less than 4.0% over a linear range of 3.0–5000.0 ng mL^{-1} for the AO and MG in water samples with regression coefficients (R^2) of 0.9975 and 0.9977, respectively. Acceptable limits of detection of 0.91 and 0.61 ng mL^{-1} for AO and MG, respectively and high accuracy and repeatability are unique advantages of present method to improve the figures of merit for their accurate determination at trace level in complicated materials.

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1. Introduction

The environmental problems associated with presence water contaminants may generate and reasonable and sever problem for marine ecosystem the environmental safety and quality of water media strongly depend on level and types of pollutants, while dyes (synthetic) are toxic to aquatic organisms via entrance to their body through food chain [1].

Therefor assessment of food quality and safety is a great task of scientist and researchers to control the level of most constituents in different media and also to monitor [2] food additives abuse especially non-permitted synthetic dyes (e.g. auramine O, AO). This

is urgent requirement and highly recommended to control and quantify their concentration [3].

Long-term exposure of AO cause teratogenesis and cancer [4–6] that encourage governments to strictly prohibit food additive usage at level higher than threshold limit.

Currently, AO content evaluation needs development sensitive and selective procedure which enable accurate and repeatable monitoring its content at level critical or even lower than [7–9] European Community threshold; (most illegal dyes content is in the range of 0.5–1.0 mg L^{-1}). In real samples complicated matrix and low level of analytes are drawbacks which destroy the monitoring stage and make emphasis to perform preliminary preconcentration and/or separation method [2].

Easley absorption of malachite green (MG) (fungicide and antiseptic agent) in aquaculture by fish and subsequent reduction to the lipophilic leucomalachite green (LMG) cause health and

* Corresponding author.

E-mail addresses: m_ghaedi@mail.yu.ac.ir, m_ghaedi@yahoo.com (M. Ghaedi).

environmental hazards [10]. USA, Europe (Council Directive 96/23/EC) and other countries strictly hindrance from MG application for veterinary treatment due to its carcinogenic and mutagenic properties [11]. Extensive application of seafood led to diminish their content in addition to fast growth of the aquaculture industry which contain high content of MG as low cost, easy availability may generate hazards for different systems [12,13]. AO and MG was quantified by various method and/or instruments like high performance liquid chromatography [2,5,10], liquid chromatography [14,15], spectrophotometry [16,17], capillary electrophoresis [18] and electrochemical determinations [19].

Several AO and MG separation and preconcentration are carried out based on dispersive liquid–liquid microextraction (DLLME) [16,20], cloud point extraction (CPE) [21,22], liquid–liquid extraction (LLE), solid-phase micro-extraction (SPE) and dispersive solid-phase micro-extraction (DSMPE) [16,23–27]. Most of these reports have their own advantages and disadvantages, while amongst SPE procedures show remarks like simplicity, capability to eliminate undesirable matrix components which accelerate and candidate this technique for such propose at aforementioned level [14].

Nano-structure based sorbents due to their unique qualities like wide variety of interactions and high reactive sites are excellent extracting phases in solid phase extraction (SPE) and dispersive solid phase microextraction (DSPME) [17,28]. Nanoparticles as coadjutants sites possible and simplify the extraction procedures, while their bare applicability failure to interact with the target analytes via covalent interactions, electrostatic interactions or simple physisorption. Composite nano-structures based on loading metallic based compound nano-particles loaded on active carbon (AC) simultaneously are able to accumulate both polar and non-polar compounds through bonding to reactive sites of AC and/or metallic based nano-structure.

Sonochemistry as ongoing area in chemistry and technology is based on application of ultrasound to accelerate the rate of reaction and also to enhance the mass transfer in physical or chemical based procedure [29]. Cavitation which is due to micro-bubbles in liquid by exposure to a large negative pressure led to generation of very fine emulsions and subsequently very large interfacial contact areas between the liquids and a corresponding dramatic increase in the mass transfer between the two immiscible phases. This leads to an increase in the extraction efficiency of the procedure in a minimum time. For these reasons, ultrasound-assisted dispersive solid phase micro-extraction (UA-DSPME) has been developed [30].

Multivariate technique extensively and usefully are applied in optimization of procedures through fast, economic and effective pathway and allows more than one variable to be optimized simultaneously. [31–33]. In this technique the different groups of designs such as Plackett and Burman and CCD design was used to determine the estimated effect of variables. CCD contains an imbedded factorial design with center points and is augmented with a group of axial points which make the design rotatable. One of the merits of CCD is to reduce the number of experiments in the studies with a large number of factors and levels. In addition, CCD can provide high quality predictions in studying linear, quadratic and interaction effect factors which influence a system; whereas interactions are unobserved in the normal orthogonal design and single factor tests [30,34].

This article the applicability of ultrasound-assisted dispersive solid phase micro extraction using Mn doped ZnS nanoparticles loaded on activated carbon (ZnS: Mn-NPs-AC) as sorbent for the preconcentration of AO and MG in water samples following their subsequent quantification with spectrophotometry. The ZnS: Mn-NPs-AC was synthesized and characterized by FE-SEM, particle size distribution, XRD and FT-IR analysis.

2. Experiment-materials and methods

2.1. Chemicals and reagents

The cationic dye auramine-O (AO) and malachite green (MG) (Fig. 1a) (Merck, Germany) were purchased from the market, and were used without any further purification. The stock solutions (10 mg L^{-1}) of AO and MG dyes were prepared by dissolving accurately weighed quantity of dye in 100 mL of double distilled deionized water. The experimental solutions of desired concentration were prepared accordingly by diluting the stock solution with distilled water. Zinc sulfate (ZnSO_4), sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) were purchased from Scharlau Company. Manganese sulfate ($\text{MnSO}_4 \cdot 2\text{H}_2\text{O}$) were purchased from Sigma Company. Dimethylformamide, acetone, acetonitrile, ethanol, methanol and Activated carbon was purchased from Merck Company.

2.2. Instrumentation

The absorbance of dye solution was measured at optimum wavelength, λ_{max} of 617 and 429 nm for MG and AO, respectively using a UV-Vis spectrophotometer (Perkin Elmer Lambda 25). The pH of the working solutions was adjusted to the desired values with dilute HCl or NaOH using a pH-meter (Metrohm 686, Switzerland). A Hermle Labortechnik GmbH centrifuge model Z206A (Germany) was used to accelerate the phase separation. Nanoparticles morphology was studied using field emission scanning electron microscope (FE-SEM: Hitachi S-4160), which is operated at an accelerating voltage of 15 kV. X-ray diffraction (XRD, Philips PW 1800) patterns are recorded from 20° to 70° using Cu $K\alpha$ radiation (40 kV and 40 mA) as the X-ray source ($\lambda = 1.54 \text{ \AA}$). Fourier transform infrared (FTIR) spectrophotometer (Perkin Elmer-Spectrum RX-IFTIR) was used to characterize the functional groups present in the adsorbent. An ultrasonic bath with a heating system (Tecno-GAZ SPA Ultra Sonic System) at 40 kHz frequency and 130 W power was used for the ultrasound-assisted adsorption procedure.

2.3. Synthesis of ZnS: Mn-NPs-AC

The Mn doped ZnS nanoparticles (ZnS: Mn-NPs) was synthesized and subsequently was loaded on activated carbon (AC) as follows: first 30 mL of 1.0 mol L^{-1} ZnSO_4 solution was mixed with 30 mL of 0.5 mol L^{-1} $\text{MnSO}_4 \cdot 2\text{H}_2\text{O}$ solution and diluted with extra 130 mL deionized water. In the next step, 60 mL of 0.5 mol L^{-1} $\text{Na}_2\text{S}_2\text{O}_3$ solution was added to above mixture drop-by-drop along strong stirring during 40 min and reaction solution was stirred at 70°C for 20 h. Then, 20 g AC as well as 200 mL deionized water was added to above mixture in the Erlenmeyer flask and stirred for 3 h at 30°C . After 3 h, the obtained Mn doped ZnS-NPs-AC were filtered and washed several times by distilled water and then dried at 80°C for 24 h.

2.4. Extraction procedure

An aliquot of 12 mL of aqueous sample (or standard solution) at pH 8.0 in 15 mL screw-cap glass test tube was contacted with 1.2 mg of ZnS: Mn-NPs-AC. The glass tube was placed on a vortex for 2.5 min lead to complete and efficient dispersion of the ZnS: Mn-NPs-AC in aqueous media. Subsequent sonication of mixture for 3.7 min lead to acceleration of mass transfer from aqueous media to the adsorbent. The extracted analytes was finally separated from the sample matrix by centrifugation (3000 rpm, 4 min) and subsequently supernatant solution was discarded by Pasteur pipette. In the next step, the adsorb analytes was elutes

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