



Contents lists available at ScienceDirect

Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy

journal homepage: www.elsevier.com/locate/saa

A quint-wavelength UV spectroscopy for simultaneous determination of dichlorobenzene, chlorobenzene, and benzene in simulated water reduced by nanoscale zero-valent Fe/Ni bimetal



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ARTICLE INFO

Article history:

Received 14 August 2016

Received in revised form 4 March 2017

Accepted 17 March 2017

Available online 18 March 2017

Keywords:

Dichlorobenzene

Determination

Nanoscale zero-valent Fe/Ni bimetal

Spectroscopic analysis

ABSTRACT

A quint-wavelength UV spectroscopy for rapid determination of dichlorobenzene (DCB), chlorobenzene (CB) and benzene (B) was developed for the dechlorination process of DCB reduced by nanoscale zero-valent Fe/Ni bimetal. Based on the absorbance measurements at 260, 269, 277, 290 and 300 nm, the equations for calculating the concentration of DCB and CB and B were established, in which the spectral interference from the nanoparticles could be effectively minimized. The results show that the present method has a good measurement precision (the relative standard deviations are within 2.0%) and accuracy (the recoveries are between 89 and 111%) in the DCB, CB and B's quantification. The present method is simple, rapid, and such a methodology is very suitable to be used for evaluating the dechlorination performance of chlorinated aromatic compounds in the presence of bimetal nanoparticle.

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1. Introduction

Chlorobenzene compounds, including di- and tri-chlorobenzenes, are commonly used as solvents or intermediate in the synthesis of many organic compounds [1]. They are also the important organic chemical raw materials in the production of dyes, pharmaceuticals, pesticides, engineering plastic materials [2,3]. These compounds could enter the environment through various channels and thus contaminate water, soil, sediment, aquifers and air [4]. Because of their toxic, mutagenic, and carcinogenic properties, as well as the poor biodegradability, the chlorobenzene compounds-bearing wastewater has a great negative impact on eco-environment [5,6], and the contents of these compounds in water have been strictly regulated by the US Environmental Protection Agency [7].

Among several technologies aiming at degrading the chlorobenzene compounds into the non-chlorinated compounds during wastewater treatment, the use of nanoscale zero-valent iron (NZVI) with a large specific surface area has been found to be cost-effective for the chlorinated aromatic compounds [8,9]. The performance of the NZVI on the dechlorination could be further improved by doping some metal(s), such as Ni, Pd, Ag, or Cu [10–15], and its physical modifications [16–18]. In this work, the chlorinated aromatic compounds, e.g., dichlorobenzene (DCB) or trichlorobenzene (TCB) is used as a model compound

and it is converted to chlorobenzene (CB), and benzene (B) eventually [19]. By determining the chlorinated species and/or its resulting species during the treatment, the performance of NZVI based bimetal can be examined. Clearly, a simple and efficient analytical method plays an important role for the research in the nanoscale zero-valent Fe/Ni bimetal development.

There are several analytical methods available for determining the chlorinated aromatic compounds in water, in which the sample extraction incorporated with a species separation based technique, i.e., gas chromatography (GC) or high performance liquid chromatography (HPLC) must be involved. For example, the use of methyl tert-butyl ether [20] or a drop-based liquid phase [21] to extract DCB and TCB before the GC measurement. The chlorobenzene compounds can be simply determined by HPLC method with a reversed-phase system (using octadecylsilica gel stationary phase and aqueous methanolic mobile phase) [22]. However, an extraction step, e.g., using 1-butyl-3-methylimidazolium hexafluorophosphate [23], was also mandatory before HPLC measurement. Besides the complicated and time consuming procedures, the major problem in these extraction and separation based methods is the mass loss of these chlorobenzene compounds because they are volatile and thus easy to escape during the solvent extracting and concentrating process. In order to overcome this weakness, the solid phase microextraction (SPME) based headspace GC method has been introduced [24]. However, the method is not suitable to be used in the quantification of the chlorobenzene compounds at the interested concentration range for the nanoscale zero-valent Fe/Ni

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bimetal evaluation, because of the poor repeatability in the SPME process [25,26].

Since aromatic compounds have strong absorption in UV wavelength range, these chlorobenzene compounds can be simply determined spectroscopically [27,28]. However, the major problem in the spectroscopic method is its poor selectivity when the spectrum of the species is overlapped with the co-existing species. Therefore, the conventional method based on the absorbance at a given wavelength fails to be used for the species quantification analysis in such cases. However, the problem could be solved by an appropriated spectroscopic techniques based on the multi-wavelength measurement [29]. A simple and efficient spectroscopic method can greatly facilitate the research in the development of NZVI based bimetal for the dechlorination.

In this study, we proposed a quint-wavelength spectrophotometric method for simultaneously determining DCB, CB, B used for evaluating the performance of the NZVI based bimetal on the DCB's dechlorinating treatment process. Among DCBs, we chose o-dichlorobenzene as the model compound in the catalytical dechlorination. The main focuses were on establishing the equations to calculate the concentrations of DCB, CB and B based on the UV spectroscopic measurement and minimizing the spectral interference from the leached bimetal materials. The precision and accuracy of the method were also evaluated.

2. Experimental

2.1. Chemicals

The chemicals used in this study, including DCB, CB, and B (analytical grade) were purchased from Macklin Reagent Company, China. Ethanol (analytical grade) was purchased from local manufacturer. Deionized water was prepared from Milli-Q Water Systems (Millipore, Bedford, USA) and used in the sample preparation. Nanoscale zero-valent Fe/Ni bimetal were prepared in our lab.

2.2. Sample Preparation

The stock solutions ($500 \mu\text{mol} \cdot \text{L}^{-1}$) of DCB and CB were prepared by accurately measuring $28 \mu\text{L}$ of DCB and $25 \mu\text{L}$ of CB respectively, into two separate 500 mL volumetric flasks which was filled with 5 mL ethanol in advance. The stock solution of B ($1670 \mu\text{mol} \cdot \text{L}^{-1}$) was prepared by adding $80 \mu\text{L}$ pure solvent (with micro syringe) in a 500 mL flask.

A set of standard solutions for DCB and CB ($15, 30, 60, 100, 150 \mu\text{mol} \cdot \text{L}^{-1}$), and B ($50, 100, 150, 200, 250 \mu\text{mol} \cdot \text{L}^{-1}$) were prepared by adding different volumes ($1.0, 2.0, 4.0, 6.0,$ and 10.0 mL) of DCB or CB

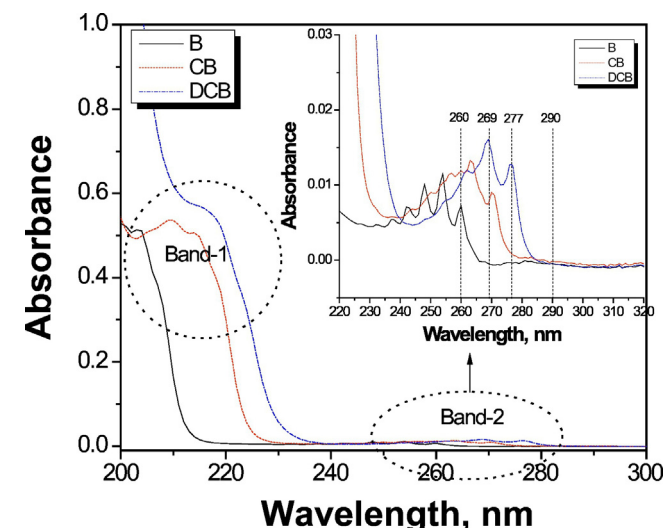


Fig. 1. UV spectra of DCB, CB and B (insert: the enlarged spectra for Band-2).

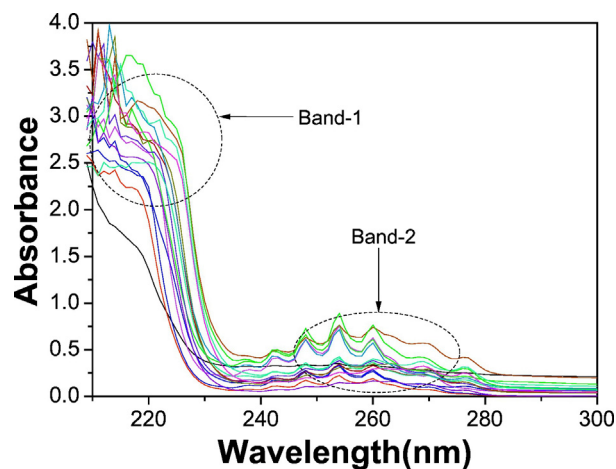


Fig. 2. UV spectra of DCB, CB, and B mixture solutions.

or B stock solution into five 50 mL volumetric flasks, filling with deionized water and mixing well.

2.3. The Preparation of Nanoscale Zero-valent Fe/Ni Bimetal and Its Application

Microfibrillated cellulose (MFC) [30] was used as the supporting material for the nano-Fe/Ni bimetal, in which the molar ratios of Fe to Ni were from 9:1 to 7:3. In the beginning of the dechlorination process, 0.10 g of bimetal reductant was added to a DCB containing solution in a quartz cell bottle (custom made, also for UV/Vis spectroscopy) with no headspace. And then the cap was screwed for avoiding the oxygen and escape of DCB, CB and B. The reaction was conducted at a given temperature with a magnetic agitation.

2.4. Spectroscopic Measurement

A UV/Vis spectrophotometer (Agilent 8453 UV-Visible System, USA) equipped with a $\Phi 2 \text{ cm}$ custom-made quartz cell bottle was used for the experiments. This special cell bottle was cylindrical with height of 7.7 cm, equipped with a wrap cap and inner silicon rubber cushion in order to keep DCB, CB and B from evaporating. Absorption spectra of sample solutions were recorded over the wavelength range of 200–1000 nm. Distilled water was used as the blank in the measurement. In the application, the supernatant of the solution in quartz cell bottle

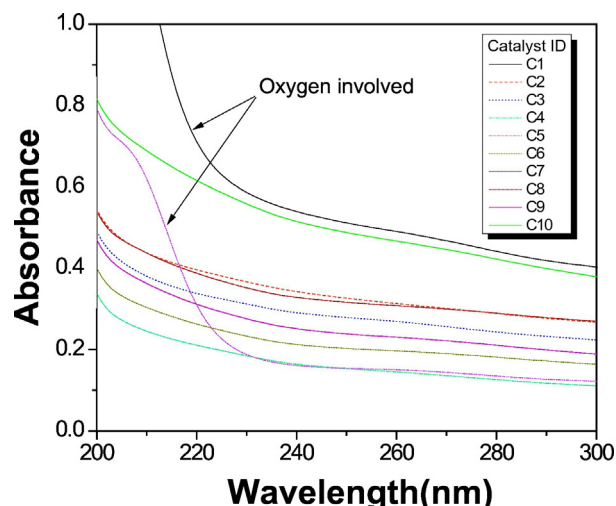


Fig. 3. Spectra of the blank solutions containing the different nano-metallic nanoparticle.

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