



Ultrasound-assisted extraction combined with reverse phase-dispersive liquid-liquid micro extraction as a new approach for preconcentration and spectrophotometric determination of total phenol in marine sediments of Chabahar Bay



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ARTICLE INFO

Article history:

Received 5 March 2016
Received in revised form 1 June 2016
Accepted 2 June 2016
Available online 9 June 2016

Keyword:

Reverse phase dispersive liquid-liquid micro extraction
Total phenol
Marine sediments
4-Aminoantipyrine
Spectrophotometry

ABSTRACT

In this study, Reverse phase dispersive liquid-liquid micro extraction (RP-DLLME) technique have been successfully developed to preconcentrate trace amount of phenol from sediment samples as a prior step to its derivatization with 4-aminoantipyrine and enhanced determination by UV-Vis spectrophotometry after primary ultrasonic extraction. In this procedure, 50 μL 0.7 M NaOH solution was chosen as extraction solvent and other factors including pH, extraction time, concentration of 4-aminoantipyrine, type and volume of dispersive solvents were optimized. Under selected conditions, the limit of detection, the linearity range, relative standard deviation and enrichment factor of method were obtained 15 $\mu\text{g}\cdot\text{kg}^{-1}$, 50–1800 $\mu\text{g}\cdot\text{kg}^{-1}$, 4.8% ($n = 10$) and 33, respectively. Finally, using the high sensitivity, low organic solvent consumption and waste generation method, total phenol content in marine sediments from several locations in Chabahar Bay (southeast Iran) was estimated at 55.8–73.2 $\mu\text{g}\cdot\text{kg}^{-1}$.

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1. Introduction

Phenols (especially chloro- and nitrophenols) are among the major pollutants in the environment because of their toxicity and the extent of uses (Olaniran and Igbinosa, 2011; Zhou et al., 2011). These contaminants can easily enter the environment through water or atmospheric systems in high concentrations due to high solubility in water and their low vapor pressure (Llorca-Pórcel et al., 2009). Soil and sediments are an important natural position for organic matter which existed in the environment (Northcott and Jones, 2000). In fact, sustainability and transfer of contaminants in the marine environment is effectively dependent to their absorption on sediment (Fei et al., 2011). Although most consideration has been focused on the determination of phenolic compounds in aqueous samples, more substituted phenols show limited transport in water and they are more likely absorbed in sediments and soils. Sediments and solids due to their high surface area and surface activities, are as a good adsorbents for phenolic compounds (Fei et al., 2011; Northcott and Jones, 2000; Salgueiro-González et al., 2014).

A great variety of extraction techniques has been applied in the analysis of phenolic compounds in solid matrices. Among others liquid-liquid extraction to organic solvents (Liu et al., 2004; Tölgyessy et al., 2009) Soxhlet extraction (Luque de Castro and García-Ayuso, 1998; Pawliszyn, 2012), supercritical fluid extraction (Chen and Liu, 2005; Li

et al., 2007), accelerated solvent extraction (Petrovic et al., 2002), microwave assisted extraction (Mahugo Santana et al., 2005; Naeni et al., 2012) or ultrasonic solvent extraction (Moreda-Piñeiro et al., 2004; Vagi et al., 2007) have been reported. Since, the major of these methodologies require large amounts of samples, large volumes of poisonous organic solvents, and a long manipulation and extraction time, much attention in sample preparation has focused on miniaturization of the liquid-liquid extraction procedure to provide significant reduction of organic solvent consumption as well as extraction time. Analytical researchers have developed a number of alternative separation methods, such as single-drop micro extraction (Saraji and Bakhshi, 2005), cloud-point extraction (Santana et al., 2004), homogeneous liquid-liquid extraction (Çabuk et al., 2014) and dispersive liquid-liquid micro extraction (DLLME) (López-Darias et al., 2010; Nassiri et al., 2014; Salgueiro-González et al., 2012) which can be used in the case of liquid samples.

Recently, Hashemi et al. (Hashemi et al., 2010) developed a new generation of dispersive liquid-liquid micro extraction (DLLME), termed as reversed-phase (RP) DLLME. The proposed RP-DLLME technique overturns the solvent polarity in the ordinary (normal-phase) DLLME and replaces the toxic solvents with water. In this method, a small volume of water (or an aqueous buffer solution) is dispersed in a lighter-than-water organic solvent with the aim of a moderately polar solvent as the disperser. Therefore, the sedimented phase will be an aqueous micro-drop that can be directly injected into analytical instruments.

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In the proposed method, by reducing the required organic solvent and sample amount in ultrasound-assisted extraction method, sensitivity loss is compensated by its combining with reverse phase dispersive liquid–liquid micro extraction. Due to our knowledge this type of combination between ultrasonic solvent extraction and RP-DLLME was performed for the first time. Extracted total phenols in marine sediment samples after derivatization with 4-aminoantipyrine was determined spectrophotometrically. Although the dispersive liquid–liquid micro extraction combined with UV–Vis spectrophotometry was optimized for the determination of phenols in seawater (Nassiri et al., 2014) there are no reports on RP-DLLME and spectrophotometric determination of phenols in soil and sediment. Thus, in order to ensure the best simulation of real samples, RP-DLLME was optimized for preconcentration and spectrophotometric determination of phenols derivatized with 4-AAP. Finally, the optimized method was used to measure the total concentration of phenols in sediment of Chabahar Bay (southeast Iran) for the determination of environmental hazards.

2. Experimental methods

2.1. Instrumentation

Spectrophotometry was carried out on a UNICO S2100 UV–Vis spectrophotometer equipped with a 10 μL quartz microcell (model Q-01701). A Centurion Scientific K3 series K241R centrifuge was used to accelerate phase separation. A TPS WP-80 digital pH meter was used for pH adjustments. A 100 μL Hamilton syringe (Hamilton Company, Nevada) was used for phase separation of collected sediments. A freezer dryer (model OPR-FDB-5503) was utilized to drying sediments and an ultrasonic bath (model PARSONIC 7500S) was used to primary extraction of Phenol from sediment samples.

2.2. Reagents

A 1000 mg/L standard solution of phenol (Merck, Darmstadt, Germany) was prepared by dissolving in *n*-hexane-dichloromethane (50:50, v/v). Working solutions were prepared daily by dilution of this stock solution. 4-Aminoantipyrine, potassium peroxodisulfate, sodium hydroxide and hydrochloric acid were purchased from Merck (Darmstadt, Germany) or Fluka (Buchs, Switzerland) and used for derivation

purposes as received. Dichloromethane, *n*-hexane, which were used as ultrasonic extraction solvents, were from Merck (Darmstadt Germany) or Fluka (Buchs, Switzerland) and were used after cleaning up with ultrapure water. Analytical grade ethanol, acetone, ethyl acetate and acetonitrile (Merck and Fluka) were used as received.

2.3. Sediments collection

Marine sediments were collected from 10 different location of Chabahar bay coastline (Fig. 1) at spring 2015. All samples were transferred in labeled bags and placed in a cooler packed with ice during transportation to the laboratory. They were freeze-dried, passed through a 0.2 mm- sieve, and carefully preserved in glass bottle with Al-foil cover at 4 °C until laboratory analysis.

2.4. Ultrasonic extraction

The initial extraction of analyte from the sediments was carried out by the standard of EPA method 3550C, so that 1 g of the dried sediment samples was added to 5.0 mL of dichloromethane-*n*-hexane (50:50, v/v) and the mixture was shaken by sonication for 15 min using an ultrasonic extractor. After centrifugation at 3500 rpm for 15 min, the supernatant was decanted in to 15 mL conical glass sample tube and preserved for the next step.

2.5. RP-DLLME and derivatization procedure

An amount of 5.0 mL of standard solution (containing 1 mg/L of phenol in *n*-hexane-dichloromethane (50:50, v/v)) or real sample (5.0 mL of the solution, which have been prepared in the previous step) was placed in the glass test tube with a lengthened conical bottom. In a microvial, 300 μL Acetonitrile (as disperser) and 50 μL 0.7 M NaOH solution (as extraction solvent) were mixed, and the mixture was rapidly injected into the diluted sample by a 2-mL polyethylene syringe. Accordingly, the extraction solvent (water) was dispersed into the sample as very fine droplets, and a cloudy solution was formed. The glass test tube was fitted inside the centrifuge tube and centrifuged for 5 min at 4000 rpm. The sedimented phase was completely transferred to a micro vial using a 100- μL micro syringe and 50 μL of 0.9% w/v 4-aminoantipyrine (4-AAP) and 50 μL of 1.4% w/v potassium



Fig. 1. Locations of coastal Chabahar bay sampling sites (1: 25°0.07075 N, E 61°0.41965, 2: N 25°0.146563, E 61°0.180761, 3: 25°0.35961 N, E 60°0.311075, 4: 25°0.292438 N, E 60°0.601179, 5: 25°0.313012 N, E 60°0.616975, 6: 25°0.3094833 N, E 60°0.624362, 7: 25°0.355048 N, E 60°0.602569, 8: 25°0.436504 N, E 60°0.487577, 9: 25°0.354396 N, E 60°0.434637, 10: 25°0.35916 N, E 60°0.311075.)

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