

Electrospun polystyrene nanofiber adsorbent for solid phase extraction of phenol as its quinoid derivative from aqueous solutions

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Abstract

In the present study, polystyrene nanofibers (PS NFs) were synthesized by electrospinning method and used as adsorbents in solid phase extraction of phenol from aqueous solutions. Phenol was reacted with 4-aminoantipyrine (4-AAP) reagent in presence of potassium hexacyanoferrate (III). The coloured product was extracted by solid phase extraction using electrospun synthesized polystyrene nanofibers and determined by UV-Vis spectrophotometer. At certain conditions of electrospinning process, the variables affecting the solid phase extraction efficiency were studied and optimized. Under the optimized conditions, (sorbent mass: 0.025 g PS NF, sample flow rate: 2.5 mL min⁻¹, eluent: acetone: NaOH 0.1 M (1:1 v/v)), the linear dynamic range (LDR) and limit of detection (LOD as 3 S_b/m) for extraction of phenol from 50 mL of aqueous solutions were determined as 15-2500 µg L⁻¹ and 10 µg L⁻¹, respectively. The precision (as RSD %) of the extraction method using the proposed adsorbent was lower than 7.6%. Finally, the applicability of the proposed method for the extraction of phenol from industrial aqueous samples was examined and satisfactory results were obtained.

Keywords: Electrospinning; polystyrene; nanofibers; solid phase extraction; phenol.

Introduction

Among many organic chemicals presented in industrial wastewaters, phenol and its derivatives are common compounds of environmental contaminants [1,2]. These compounds are widely used in the manufacture of plastics, epoxies, dyes, drugs, antioxidants, pesticides and in the petrochemical industry [3,4]. Phenol

causes unpleasant smell and taste of drinking water and the presence of it even at low concentrations can make negative effects on different biological processes [1,5]. Therefore, highly reliable analytical techniques are required for the preconcentration and detection of trace amounts of phenol. For the extraction of phenol from aqueous solutions, various extraction methods

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were proposed. One of these methods is solid phase extraction (SPE), which is a very applicable method with reduction in solvent consumption, extraction time and costs [6]. The extraction efficiency often depends on the adsorbent properties, so the nature and the properties of the sorbent materials are the most important part of the SPE system [7,8].

Polymeric materials are often used as sorbent for SPE and their application is reported in various published papers [9,10]. These sorbents are stable in narrow pH range and are capable to extract polar and/or nonpolar compounds from different samples [8]. Hyper-crosslinked polystyrene [11], polystyrene-divinylbenzene (PS-DVB) [12,13], modified PS-DVB [14], DVB copolymers [1], β -cyclodextrin-ionic liquid polymer (β CD-BIMOTs-TDI) [15] and functionalized PS-DVB [16] have been used as SPE sorbents to extract phenols from various water samples. One method for increasing the performance of SPE is the use of nanomaterials as sorbents [17]. In recent years, nanofibers have attracted particular attention due to their large ratio of surface area to volume and excellent structural mechanical properties. The electrospinning is a simple, efficient and inexpensive technique which utilizes high electrostatic forces for producing nanofibers. Nanofibers prepared by electrospinning possess unique characteristics such as large specific surface area, high porosity and flexibility in surface functionalities. Furthermore, a variety of polymers can be used for producing electrospun nanofibers with ability to control the diameters, shapes and porosities [18-22].

In the present study, electrospun polystyrene nanofibers (PS NFs) were synthesized and used as SPE sorbent for the preconcentration and determination

of phenol as its quinoid derivative from aqueous solutions. The surface morphologies and diameter of PS NFs and their chemical structure were characterized by scanning electron microscope (SEM) and Fourier transform infrared spectroscopy (FT-IR), respectively. The experimental factors which could affect the extraction efficiency were investigated and optimized. Finally, the applicability of the proposed SPE sorbent for preconcentration and determination of phenol as its quinoid derivative in real samples was investigated and satisfactory results were obtained.

Experimental

Chemicals and materials

All chemicals used in this study, including phenol, 4-aminoantipyrine, potassium hexacyanoferrate, ammonium chloride, ammonia, acetone, tetrahydrofuran (THF), dimethyl formamide (DMF), methanol (MeOH) and sodium hydroxide (NaOH) were of analytical reagent grade and were prepared from Sigma-Aldrich (Milwaukee, WI, USA) and Merck (Darmstadt, Germany). PS granules were purchased from Tabriz Petrochemical with industrial code of GPPS 1460 and density: 1.04 kg L^{-1} (Tabriz, Iran). All working solutions were prepared by double distilled water to the required concentrations.

Appropriate amounts of phenol were dissolved in MeOH to obtain stock standard solution with a concentration of 1000 mg L^{-1} . This standard solution was diluted with double distilled water to prepare solutions with the required concentrations.

Instruments

All spectrophotometric measurements were carried out using a UV-Vis spectrophotometer from Mapada Company (6300 PC, China). The

chemical structure of the synthesized electrospun nanofibers were examined using Shimadzu FT-IR (8900, Japan) in the wavenumber range of 400–4000 cm^{-1} . An electrospinning setup was used, which included electrospinning syringe pump (Fanavaran Nano-Meghyas, Iran) and power supply from Fanavaran Nano-Meghyas (Iran). Electrospinning process carried out in a handmade woody box (90 × 50 × 60 cm) in order to protect the users from high voltage. The pH of solutions was adjusted using an EDT pH meter (GP 353, England) equipped with a combined glass pH electrode. The morphologies of the prepared electrospun nanofibers were examined using a Seron Technology SEM (AIS 2100, Korea). Separations were carried out on a handmade glass column (100 mm × 10 mm i.d.). A vacuum pump from Sparmax (TC2000 MV, Taiwan) was used for passing sample solutions through the adsorbent in SPE column.

Preparation of PS nanofibers by electrospinning

PS NFs were synthesized by an electrospinning method according to the following procedure. For the preparation of PS solution (25% w/v), a proper amount of PS granule was dissolved in a mixture of DMF:THF (4:1, v/v) via magnetic stirring at room temperature for 4 h. Then, the viscous solution was loaded into a plastic syringe (10 mL) equipped with a stainless steel flat tip needle with an inner diameter of 0.4 mm that mounted on a syringe pump. The aluminum plate was used as a collector for obtaining nanofiber membranes. The distance between the needle tip and the aluminum collector was kept at 15 cm. A feed rate of 0.1 mL h^{-1} was set on the syringe pump for the polymeric solution delivery. 20 KV voltage was applied on

the polymer solution using an external electric field through the syringe needle. Finally, PS NF membranes were collected on the aluminum foil. Figure 1 shows the scheme of planned electrospinning system.

A scanning electron microscope (SEM) was utilized to characterize the synthesized PS NFs. As illustrated in Figure 2, the diameters of electrospun NFs were in the range of 380-500 nm.

Measurement of phenol content in sample solution

For measuring the concentration of phenol in aqueous solutions, UV-Vis spectrophotometer was utilized using the well-known 4-aminoantipyrine spectrophotometric method that was developed in 1943 by Emerson [23]. This developed method is still in common use mainly due to special features like speed, cost-effectiveness and absence of laborious steps. The reaction between phenol and 4-aminoantipyrine was shown in Figure 3.

For this method, briefly, 50 mL of the sample solution containing 10 mg L^{-1} of phenol and 2 mL of 0.01 mol L^{-1} 4-aminoantipyrine (as derivative reagent), was transferred into a 100 mL beaker. After that, 4 mL of 0.2 g L^{-1} potassium hexacyanoferrate ($\text{K}_3\text{Fe}(\text{CN})_6$) and 2 mL of 0.5 mol L^{-1} ammonium chloride (NH_4Cl) were added. The pH of solution was adjusted in the range of 9-10 by addition of appropriate amounts of ammonia solution (1 M). Then, the obtained solution was stirred at 40-50 °C for 30 min. Finally, the absorbance spectrum of pink solution was recorded by UV-Vis spectrophotometer in the wavelength range of 450-750 nm and $\lambda_{\text{max}} = 515 \text{ nm}$ was used as maximum wavelength for further measurements.

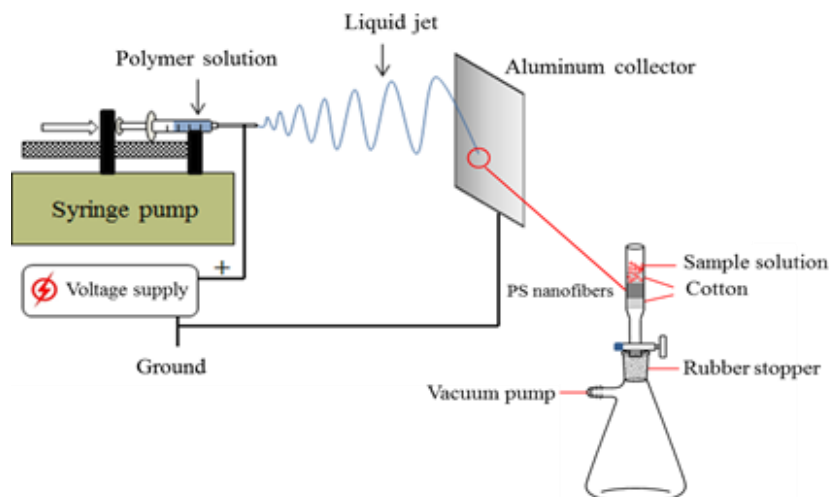


Figure 1. The schematic of electrospinning system for producing PS NFs with SPE applications

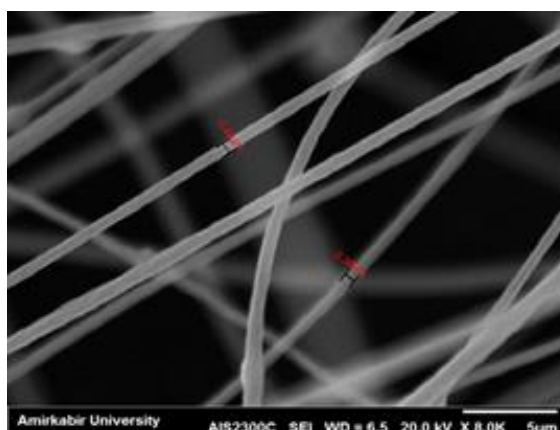


Figure 2. The SEM images of polystyrene nanofibers

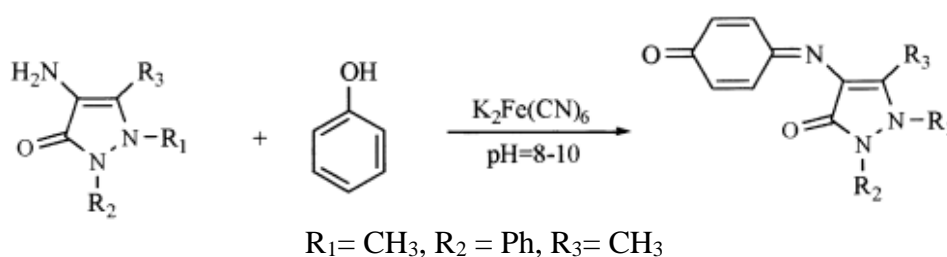


Figure 3. Reaction of 4-aminoantipyrine with phenol

Solid phase extraction using PS NF sorbents

SPE was performed on a handmade glass column with an effective length of 100 mm and inner diameter of 10 mm equipped with a controllable valve in order to adjust the sample flow rates. An appropriate amount of electrospun PS

NFs was firmly packed into the glass column between two pieces of cotton to retain the sorbent in the column. The column linked to a vacuum pump using a vacuum Erlenmeyer. Before use, SPE column was conditioned by passing 2 mL of both MeOH and double distilled water respectively to clean the sorbent from

possible interferences and conditioning it (conditioning step). For each extraction, to 50 mL of the sample containing phenol (in the range of 15-2500 $\mu\text{g L}^{-1}$), 2 mL of 0.01 mol L^{-1} 4-aminoantipyrine (as derivative reagent), 4 mL of 0.2 g L^{-1} $\text{K}_3\text{Fe}(\text{CN})_6$ (as oxidant) and 2 mL of 0.5 mol L^{-1} NH_4Cl were added. The pH of solution was adjusted in the range of 9-10 by ammonia solution (1 M) and solution was stirred at 40-50 $^\circ\text{C}$ for 30 min. After the formation of colored product, the solution was passed through the PS NF column at the rate of 2.5 mL min^{-1} by vacuum pumping (loading step). After passing of mixture through the sorbent, pink colored product was adsorbed on the surface of PS NFs and separated from aqueous solution. Then, the loaded solution was eluted using 4

mL of a mixture of acetone: 0.1 mol L^{-1} NaOH (1:1 v/v) as an effective eluent solvent. Finally, the analyte concentrations in the eluent solution were determined by UV-Vis spectrophotometer.

Results and discussion

FT-IR spectra of PS NFs

The FT-IR spectra of PS NFs is shown in Figure 4. The strong absorbance band at 698.18 cm^{-1} is attributed to single ring styrene monosubstituted. The bands at 1490 and 1448 cm^{-1} are attributed to the C=C of the aromatic ring and the band at 2920 cm^{-1} is related to the C-H with SP^2 spin. Also, C-H vinyl functional group was observed at 1026 cm^{-1} .

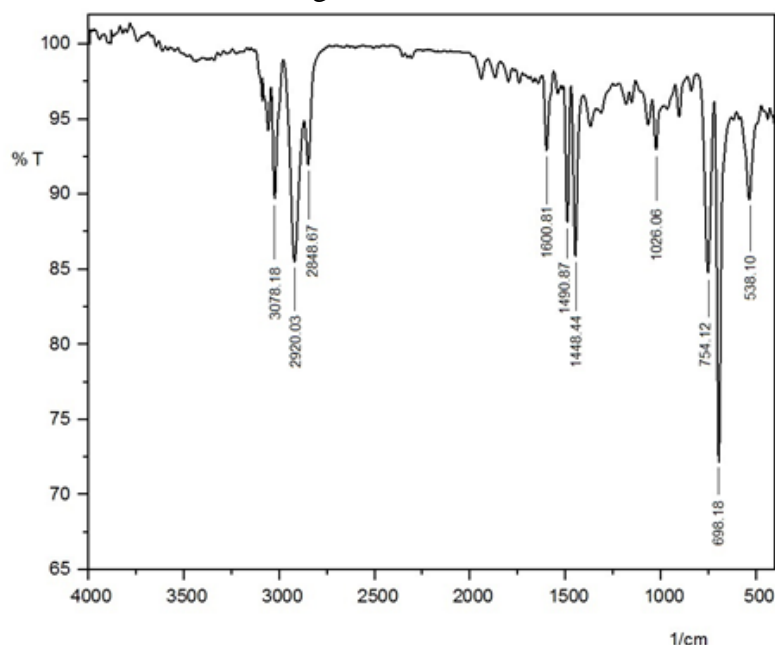


Figure 4. FT-IR spectra of PS NFs

During the reaction of phenol with 4-aminoantipyrine reagent, the amino group of reagent is oxidized by $\text{Fe}(\text{CN})_6^{3-}$ to an imino cation which attacks electrophilically to the *para*-position of the phenolic ring. Oxidation of the resulting amino product by $\text{Fe}(\text{CN})_6^{3-}$ leads to the *p*-quinoid species.

All of experimental parameters affecting the extraction performance were investigated and optimized using univariate method with 10 mg L^{-1} concentration of phenol as follows.

Effect of PS NF's mass on the SPE efficiency

The mass of PS NF sorbent plays an important role in the SPE method because target analyte should be adsorbed completely with sufficient PS NFs. In this study, the sorbent mass of PS NFs was investigated in the range of 0.005 – 0.05 g. As shown in Figure 5, the extraction efficiency increased with the

increasing weight of the PS NFs up to 0.025 g. But, the extraction efficiency of phenol decreased with increasing amounts of sorbent more than 0.025 g due to the inadequate eluent volume and difficulty of passing of sample solution through the column. Thus, 0.025 g of PS NF was selected as the optimum mass for subsequent extractions.

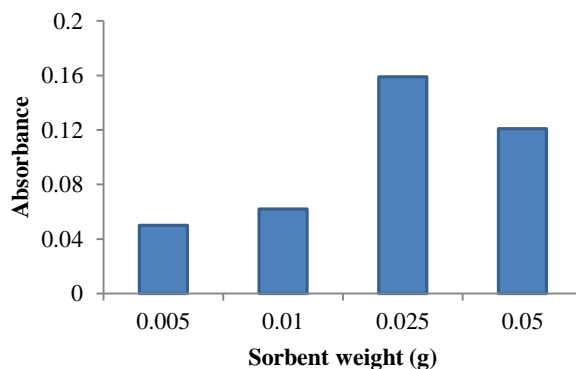


Figure 5. Effect of sorbent mass on the extraction efficiency. Extraction conditions: 50 mL of 10 mg L⁻¹ of phenol, Sample flow rate: 2.5 mL min⁻¹, Eluent: 4 mL acetone: NaOH 0.01 M (1:1 v/v)

Effect of sample flow rate on the SPE efficiency

In SPE system, the sample flow rate through the PS sorbent not only affects the extraction efficiency, but also controls the extraction time. The effect of sample flow rate on the extraction efficiency was studied in the range of 2.5-10 mL min⁻¹. According to the

results (Figure 6), the extraction efficiency increased with the reducing sample flow rate. This can be explained by more time available between the sorbent and adsorbate in lower flow rates. Consequently, 2.5 mL min⁻¹ sample flow rate was the most favorable flow rate and used for further studies.

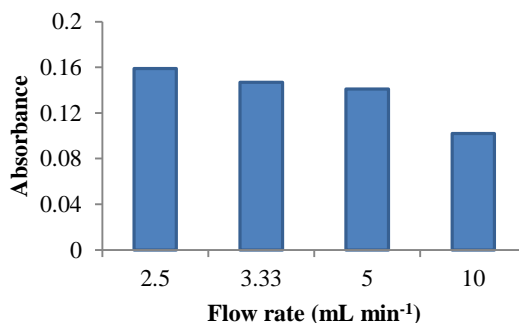


Figure 6. Effect of the sample flow rate on the extraction efficiency. Extraction conditions: 50 mL of 10 mg L⁻¹ of phenol, Sorbent weight: 0.025 g PS, Eluent: 4 mL acetone: NaOH 0.1 M (1:1 v/v)

Effect of eluent type and volume on the SPE efficiency

After the adsorption stage, column and sorbent should be eluted with a specific

volume of suitable eluent. This step is dependent on the chemical nature of the target analyte. Organic solvents like methanol, acetone, chloroform, HCl

(0.01 mol L⁻¹), NaOH (0.01 mol L⁻¹) and mixture of acetone: NaOH were studied as eluents for the desorption studies. The results showed that the mixture of acetone: NaOH 0.01 M obtained the best desorption. In order to optimize the composition of eluent mixture, different ratios of acetone: NaOH 0.01 M (2:2, 3:1, 1:3, 3.5:0.5 v/v) were investigated under the optimized conditions (50 mL of 10 mg L⁻¹ of phenol, sample flow rate: 2.5 mL min⁻¹, sorbent mass: 0.025 g PS NF, eluting volume: 4 mL). Also, different NaOH concentrations in the range of 0.0001- 0.1 mol L⁻¹ were investigated (4 mL of acetone: NaOH, 1:1 v/v). The best extraction efficiency was achieved with addition of 0.1 mol L⁻¹ NaOH to eluent.

On the basis of the obtained experimental results, the mixture of 1:1 (v/v) acetone: NaOH 0.1 M was chosen as the best eluent.

To investigate the eluent volume, acetone: NaOH 0.1 mol L⁻¹ (1:1 v/v) volumes in the range of 3-6 mL were passed through the SPE column. According to the results, the preconcentration of phenol increased while volume of eluent enhanced from 3 to 4 mL. At higher volumes, the preconcentration was reduced due to the more volumes of solvent. Therefore, in all subsequent experiments, 4 mL of acetone: NaOH 0.1 mol L⁻¹ (1:1 v/v) was chosen as the optimum eluent.

Effect of sample volume on the SPE efficiency

Breakthrough volume is another parameter that influences the preconcentration performance. For this purpose, 50, 75, 100 and 250 mL of the sample solution containing 10 mg L⁻¹ of phenol after addition of reagents were passed through the column at the optimum conditions. According to the results, the extraction efficiency

increased when the volume of sample was increased to 250 mL.

Evaluation of method performance

Quantitative parameters of the proposed SPE method were calculated under the optimized conditions described in the previous sections (sorbent mass: 0.025 g PS NF, sample flow rate: 2.5 mL min⁻¹, eluent: 4 mL mixture of acetone: NaOH 0.1 M (1:1 v/v)). The calibration curve was obtained by plotting the measured absorbance against the concentration of phenol in the sample prior to derivatization reaction. Under the optimum conditions, the calibration curve was obtained by passing 50 mL of spiked standard solutions (15 stock standard solutions) in the concentration range of 1-10000 µg L⁻¹ of phenol. The reagents were added to these stock standard solutions and the solution containing colored product was passed through the sorbent in the SPE column. After desorption, the eluted product was measured by UV-Vis spectrophotometer against the blank solution that was prepared using the solutions containing reagents without phenol and passed through the PS SPE column. The linear dynamic range (LDR) of the proposed method was obtained in the range of 15-2500 µg L⁻¹ with correlation coefficient (r^2) of 0.9901. The limit of quantification (LOQ) of method was obtained as 15 µg L⁻¹ and limit of detection (LOD) based on $C_{LOD}=3 S_b/m$ was calculated as 10 µg L⁻¹ where m is the slope of the calibration graph after preconcentration and S_b is the standard deviation of five replicates of the blank measurement. The precision of the method was evaluated using extraction from five replicate preconcentration of solutions containing 100 µg L⁻¹ of phenol and the RSD (%) values were lower than 7.6%.

Determination of phenol in real samples

The applicability and accuracy of the proposed SPE method for the extraction and determination of phenol was investigated in the different water samples including sea water (collected from Caspian Sea) and three industrial wastewaters all located in Iran. At first, the real water samples were filtered through 0.45 mm micropore membrane (Tianjin Automatic Science Instrument Co., Ltd., China) prior to use, to remove suspended particles. Then, the phenol

content of each sample was determined under the optimized conditions.

In order to validate the accuracy and the performance of the proposed method, the samples were spiked with $100 \mu\text{g L}^{-1}$ of phenol and after addition of reagents (according to section 2.4), the recovery values were calculated by extracting from 50 mL of the spiked solution *via* dividing the found values to the sum of real and added values. The results are presented in Table 1.

Table 1. Determination of phenol in different water samples by applying the proposed method

Sample	Real ($\mu\text{g L}^{-1}$)	Added ($\mu\text{g L}^{-1}$)	Found ($\mu\text{g L}^{-1}$)	Recovery (%)
Wastewater 1	100.0 ± 14.2	100	192.2 ± 25.7	96.1
Wastewater 2	1300.0 ± 60.1	100	1420.0 ± 36.2	101.4
Wastewater 3	333.3 ± 21.4	100	450.6 ± 39.1	104.0
Sea water	ND	100	100.0 ± 2.7	100

As could be seen, the relative recoveries for the spiked samples are in the range of 96.1-104% that are in satisfactory agreement with the spiked amounts.

Comparison of the proposed method with other methods

The proposed method was compared with different methods reported in the

literature for determination of phenol and the results are summarized in Table 2. As observed, the method offers wide dynamic linear range and acceptable values of LOD and RSD in comparison with the other techniques. If the UV-Vis detection method could be replaced with other sensitive methods such as HPLC, the better LOD and LOQ values will be obtained.

Table 2. Comparison of the proposed method with the other techniques for determination of phenol

Analytical method	Detection	Detection limit	Linear range	RSD (%)	Reference
IL-UA-EME ^a	UV-Vis	0.07 ($\mu\text{g L}^{-1}$)	0.2-25 ($\mu\text{g L}^{-1}$)	2.6	[24]
Kinetic spectrophotometric	UV-Vis	0.60 (mg L^{-1})	1.52-19.76 (mg L^{-1})	-	[25]
Pervaporation-flow injection	UV-Vis	0.9 (mg L^{-1})	1-50 (mg L^{-1})	1-4	[26]
HF-LPME ^b	UV-Vis	1.5 ($\mu\text{g L}^{-1}$)	5-500 ($\mu\text{g L}^{-1}$)	4.9	[27]
SPE ^c	GC-MS	0.3 ($\mu\text{g L}^{-1}$)	1-160 ($\mu\text{g L}^{-1}$)	5.5	[28]
SBSE ^d	GC-MS	0.3 ($\mu\text{g L}^{-1}$)	---	13	[29]
LPME-BE- HPLC ^e	HPLC	0.5-2.5 ($\mu\text{g L}^{-1}$)	---	6.3	[30]
HF-LPME-CE ^f	CE	2 ($\mu\text{g L}^{-1}$)	10-4000 ($\mu\text{g L}^{-1}$)	2.7-7.6	[31]
EPS-NFs SPE ^g	UV-Vis	10 ($\mu\text{g L}^{-1}$)	15-2500 ($\mu\text{g L}^{-1}$)	7.6	This work

a Ionic Liquid based Ultrasound-Assisted Emulsification Microextraction

b Hollow fiber liquid phase microextraction

c Solid phase extraction

d Stir bar sorptive extraction

e Liquid phase microextraction –back extraction high performance liquid chromatography

f Hollow fiber liquid phase microextraction - Capillary electrophoresis

g Electrospun synthesized polystyrene nanofibers- solid phase extraction

Conclusion

Electrospun nanofibers have unique properties as compared to other conventional fibrous structures because of their high surface area to mass ratio. This makes nanofibers ideal materials for use as sorbent for extraction applications. In the present study, SPE method was developed for the preconcentration of phenol in different water samples using PS NFs as sorbent. The method was successfully applied to determine phenol in the environmental samples and the recovery percentages for different samples were obtained as more than 96%.

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