

## Determination of Phenol from wastewater by Cloud Point Extraction method

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**Abstract :** A simple, low cost and sensitive method based on cloud point extraction (CPE) used for separation/preconcentration of phenol has been described. After extraction into surfactant phase followed determination of the phenol by visible spectrophotometry method. The analyte are quantitatively extracted to the phase rich in Triton X-114 following centrifugation. The parameters which affecting the separation phase and detection process, were performed. Calibration graph showed linear trend in rang of 1.0-14.0 mg l<sup>-1</sup> for the analyte. Phenol was determined in petrochemical waste water sample which gave satisfactory results.

**Key words:** Cloud point extraction, phenol, spectrophotometry method.

### Introduction

Many industrial wastes contain organics which are difficult or impossible to remove by conventional biological treatment process. Phenols as a class of organics are similar in structure to the more common herbicides and insecticides in that they are resistant to biodegradation. Phenols are widely used for the commercial production of a wide variety of resins including phenolic resins, which are used as construction materials for epoxy resins, adhesives and polyamide for various applications<sup>1-7</sup>.

The cloud point extraction (CPE) is an interesting and efficient alternative because reduces the consumption of and exposure to solvents, the disposal costs and the extraction time, and surfactants are not toxic, not volatile and not easily flammable. The cloud point extraction (CPE) has been used for the extraction and pre-concentration of some analytes after the formation of sparingly water-soluble specieses and then determined in the surfactant-rich phase by different spectrometric technique. In the present work, a study of the CPE for simple and selective preconcentration of phenol after extraction in surfactant reach phase and later analysis by visible spectrophotometric method. The proposed method is also applied to the determination of the phenol in some environmental samples such as petrochemical waste water, which shown satisfactory results.

## Material and Method

### Reagents and solutions

All solutions were prepared with ultra pure water (obtained from HAMILTON, England) Laboratory glass was kept overnight in a 10% (V/V) HNO<sub>3</sub> solution and then rinsed with deionized water. Phenol, K<sub>3</sub>Fe(CN)<sub>6</sub>, 4-aminoantipyrine, ammonia solution (25%), ammonium chloride, were prepared from Merck company.

The stock phenol solution (100 mg l<sup>-1</sup>) were prepared by Merck standard solutions.

The nonionic surfactant Triton X-114, 5.0% (W/V) prepared by dissolving 5.0 gr surfactant Triton X-114 made up to 100 ml with distilled water. A solution of 8.0 g L<sup>-1</sup> of K<sub>3</sub>Fe(CN)<sub>6</sub> was prepared by dissolving 8.0 gr of this reagent in 100 ml water. A solution of 2.0 g L<sup>-1</sup> of 4- aminoantipyrine was prepared by dissolving 2.0 gr of this reagent in 100 ml water. A buffer pH=10 was prepared by ammonia/ammonium chloride (0.1 M) in 500 mL volumetric flask.

### Apparatus .

The spectrophotometric measurements were carried out with a (perkinelmer lambda25) UV/Vis spectrometer. A pH meter (691 metrohm, herisau Switzerland), a balance (Sartorius, BL 210S, d=0.1mg), an oven (memmert) were used. A centrifuge (Hettich) was used to accelerate the phase separation process. A thermostated water bath (Memert) was employed to maintain the experimental temperature.

### Procedure

For the cloud point extraction, an aliquots of 10 ml of a solution containing phenol, and 1 ml of buffer solution pH=10, 2 ml Triton X-114 5.0% (W/V), 0.2 ml of K<sub>3</sub>Fe(CN)<sub>6</sub>, 8.0 g l<sup>-1</sup> and 0.2 mL of 2.0 g mL<sup>-1</sup> 4-aminoantipyrine were placed in a centrifuge tube and heated in a thermostat water bath at 50°C for 20 min. After heated, the resultant solution was subjected to centrifugation at 4000 rpm for 15 min for phase separation, and then cooled in an ice-water for 10 min in order to increase the viscosity of the surfactant-rich phase. The supernatant aqueous phase was easily decanted by simply inverting the tube. The residue of phenol concentration in the supernatant solution after dilution to 3 mL methanol was determined by spectrophotometric method based on ASTM D1783-01<sup>8</sup> at  $\lambda_{\max} = 510$  nm. In this test method phenol form a reddish complex with 4-aminoantipyrine and K<sub>3</sub>Fe(CN)<sub>6</sub> in the presence of ammonia/ammonium chloride buffer solution. A blank solution was also run without adding the analyte.

## Preparation of Real Samples

### Waste water samples

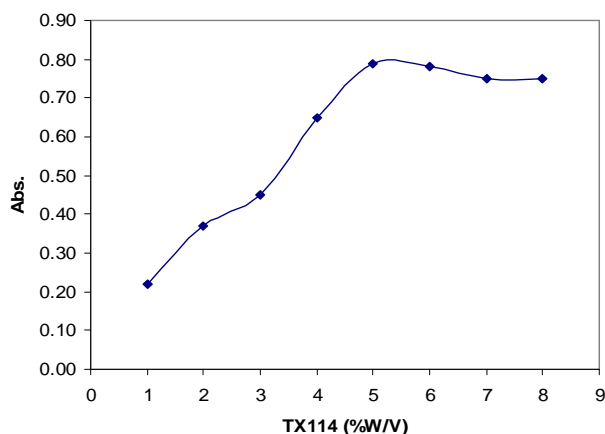
Waste waters were collected from some Mahshar petrochemical industry (Mahshar city is the important city in Khozestane province- IRAN). They were filtered through Watman No. 42 and stored in glass bottles at 4°C. Then the samples were ready for purposed procedure.

## Results and Discussion

### Effect of Triton X-114 concentration

A successful cloud point extraction should maximize the extraction efficiency by minimizing the phase volume ratio (V<sub>org</sub>/V<sub>aq</sub>), thus improving the concentration factor.

Fig. 1 has shown the effect of TritonX-114 concentration on the CPE pre-concentration of the phenol. The signals are increased as the Triton X-114 concentration increased from 1.0- 5.0% (W/V) when, Triton X-114 concentration was increased larger than 5.0% (W/V), the signal was decreased. Hence a low concentration of Triton X-114, 5.0% (W/V), was employed for the rest of this work.



**Fig. 1.** Effect of the Triton X-114 concentrations on the extraction recovery of the analyte by proposed method.

### Practical parameter

The dependence of extraction efficiency on equilibration temperature and time were studied within a range of 30 – 70 °C and 5–25 min, respectively. The results showed that an equilibration temperature of 50 °C and an equilibration time of 20 min are enough to get quantitative extraction of the phenol. The effect of the centrifugation time on the extraction efficiency was also studied within a range of 5–25 min. A centrifugation time of 15 min at 4000 rpm was selected for the entire procedure, since analytes extraction in this time is almost quantitative.

### Figures of merit

Calibration graph was obtained in optimum conditions and for phenol was linear from 1.0-14.0 mg L<sup>-1</sup> with a correlation coefficients of 0.9887. The detection limit which obtained by the equation of  $DL=3S_b/m$  is 0.28 mg L<sup>-1</sup>. The relative standard deviation (RSD, n=5) for 5.0 mg L<sup>-1</sup> concentration of phenol was 2.5%.

### Determination of phenol in petrochemical waste water

In order to test the reliability of the proposed methodology suitable for the assaying of analyte in waste water samples a series of petrochemical waste water sample were used. For this purpose, 10ml of each of the samples were pre-concentrated with 5.0% (w/v) Triton X- 114 and, following the proposed procedure. The results are shown in **Table 1**.

**Table 1.** Determination of phenol in petrochemical waste water samples.

Sample	Phenol (mg L <sup>-1</sup> ) <sup>a</sup>		% Recovery
	Added	Found	
Waste water 1	-	ND	-
	2.0	2.2	110
	5.0	4.9	98
Waste water 2	-	ND	-
	2.0	2.1	105
	5.0	5.0	100
Waste water 3	-	ND	-
	2.0	1.9	95
	5.0	5.2	104
Waste water 4	-	ND	-
	2.0	2.1	105
	5.0	5.2	104
Waste water 5	-	ND	-
	2.0	1.9	95
	5.0	5.3	106

a) After appropriate dilution

## Conclusion

This paper proposes a simple and effective method for the preconcentration and determination of trace levels of phenol by CPE in waste water samples for their determination by spectrophotometric method. The methodology offers a reliable method for determination of phenol in petrochemical industries.

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