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Determination of phenols in soil by supercritical fluid extraction–capillary electrochromatography ☆

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Abstract

A new analytical procedure is developed to couple supercritical fluid extraction with capillary electrochromatography (SFE–CEC) to extract and determine phenols in soil. Ten phenols consisting of phenol, methylphenols (*p*-cresol and *o*-cresol), dimethylphenols (3,5-xylenol, 3,4-xylenol and 2,6-xylenol), trimethylphenol, ethylphenols (*p*-ethylphenol and *o*-ethylphenol), and *o*-isopropylphenol are investigated. The use of supercritical CO_2 with 10% methanol as the organic modifier was found to give satisfactory extraction of alkylphenols from soil at 1200 p.s.i. and 50°C for 45 min under a total extractant flow-rate of 0.2 ml/min (1 p.s.i.=6894.76 Pa). Baseline resolution was achieved for the 10 selected phenols under optimised CEC conditions at 20 kV in a mobile phase of acetonitrile–4 m*M* Tris, pH 7.0 (35:65) in a 45 cm (25 cm packed with 3 μm ODS)×75 μm I.D. fused-silica capillary column. Using SFE with a 10-fold preconcentration factor, all alkyl-substituted phenols in soil can be determined with detection limits ranging from 0.0032 to 0.014 mg/kg and working range from 0.019 to 2.72 mg/kg. The SFE–CEC procedure developed has been applied successfully to determine phenols extracted from real soil sample contaminated with medical disinfectant. It will provide a rapid method for the direct determination of phenol and alkyl-substituted phenol in soils, with capability for confirmation of unknown peaks.

Keywords: Soil; Supercritical fluid extraction; Electrochromatography; Phenols; Alkylphenols

1. Introduction

Phenol and alkyl-substituted phenols occur naturally in the polar fraction of crude oil with significant effect on both the refining and stability of the crude and refined products [1]. Due to their widespread use as germicides, medical disinfectants, paint strippers and solvents for cleaning and dissolution, and as ingredients in fuel, they are well known pollutants frequently found in the effluents discharged into the environment at industrial sites, petroleum stations, depots or other storage areas. As many phenolic compounds are impacting bad taste and undesirable odour to the water bodies and some of them are toxic and hazardous to human health [2], their levels in water and soil samples are regularly monitored to determine whether or not they are exceeding the action limits imposed by governments worldwide for environmental protection. Due to the large difference in toxicity amongst different phenols, the analysis of individual phenols are required in addition to the

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determination of total phenols in environmental samples.

The problems for the analysis of phenols in the environment are the low concentration of phenols at the μg/kg level to be quantified, the strong retention of phenols in soils, the complexity of sample matrix and the presence of a large number of phenols and substituted phenols in petrochemicals with similar structure and chemical properties, though with differing health impact. Thus, a clean-up procedure is required prior to the use of a highly efficient separation method and a sensitive mode of detection for the quantitation of individual phenols in environmental samples. The determination of residual individual phenols as indicators for the clean-up of contaminated soils from spillage of petrochemicals is at present a demanding analytical task requiring the use of new analytical methods.

Various sample pretreatment methods have been developed for phenol analysis such as distillation [1], membrane extraction [3–5], Soxhlet liquid–liquid extraction [6,7], solid-phase extraction [8], microwave-assisted extraction [9,10], ultrasonication and supercritical fluid extraction (SFE) [10–16]. Distillation and membrane extraction are only suitable for phenols present as major component in the samples and solid-phase extraction useful only for extracting phenols from water samples. The extraction of phenols from soil by Soxhlet liquid extraction was time consuming and low recovery was observed using sonification for extraction [10]. For microwave-assisted extraction, there are problems for extracting soil samples containing material with a high absorption of microwave energy [10]. SFE [13–16] offers a promising method for extracting polar and high-molecular-mass phenols from soil samples within a short extraction time. Good recovery of strongly retained phenols had been reported using SFE for extraction and its use led to reduced consumption of organic solvent in the environmental laboratory [10,13–16].

For the separation of individual phenols, various techniques have been used such as gas chromatography (GC) [10,14–19], high-performance liquid chromatography (HPLC) [20–23], micellar electrokinetic chromatography (MEKC) [24] and capillary electrochromatography (CEC) [25]. GC can only separate volatile compounds and derivatization is necessary prior to analysis. Separation of phenols by HPLC is time consuming and often with insufficient selectivity for separating alkyl-substituted phenols with similar structures. For coupling the above separation methods with SFE, only GC procedures had been reported in the literature [10,14–16] for quantitation of individual phenols extracted from soil samples and the studies were restricted to the separation of a few volatile phenols.

Recently, various rapid separation techniques have been developed based on capillary electrophoresis (CE) due to its high separation efficiency, short separation time and good sensitivity. These will provide promising techniques for quantitation of phenols extracted from soil samples. Although separation of 14 phenols had been achieved by Terabe et al. [24] using MEKC, the buffer is not compatible with the use of mass spectrometry which can provide important information for confirming unknown eluted peaks. The use of CEC, combining the advantages of both CE and HPLC, offers a suitable technique for rapid and efficient separation with capability of interfacing with mass spectrometry for peak confirmation. Although the application of CEC for the analysis of mono- and dihydroxyphenols in tobacco smoke had been reported [25], no paper has been found in the literature on the separation of phenol and alkyl-substituted phenols by CEC and this initiates the present investigation.

In this paper, phenol and nine alkyl-substituted phenols have been selected for the investigation of their extraction from soil by SFE prior to their separation and quantitative determination by CEC. The optimisation of the working conditions for SFE extraction and CEC separation will be reported. The advantages and limitations of the SFE–CEC technique developed for phenol determination in soil samples will be given and discussed.

2. Experimental

2.1. Reagents and standards

All phenol standards (between 97.5 and 99.5% purity) were prepared from the highest purity grade chemicals purchased from Aldrich (Milwaukee, WI, USA). Methanol and acetonitrile were HPLC grade

and purchased from J.T. Baker (Phillipsburg, NJ, USA). The phenol stock solutions were prepared by dissolving individual phenols to the concentration of 10 m*M* in 10 ml methanol and storing at 4°C prior to use.

The mobile phase used in the CEC runs was prepared daily by mixing acetonitrile with 4 m*M* Tris buffer in a volume ratio of 35:65 prior to adjusting pH to 7.0 with 0.01 *M* HCl. Both the acetonitrile and Tris buffer were filtered through a 0.45-μm nylon membrane and the mobile phase was thoroughly degassed by ultrasonication for 20 min before use.

2.2. Instrumentation

The SFE system was comprised of a syringe pump (Model 100 DX, ISCO), a pump controller (Series D, ISCO), a programmable isocratic pump (Model 305, Gibson) and a 5-ml hand-tight SFE vessel (Keystone). The CEC set-up consisted of a high-voltage power supply (Spellman, Model CZE1000R) to provide the running voltage, a fused-silica capillary column of 45 cm (25 cm length packed with 3 μm ODS particles)×75 μm I.D. (Unimicro Technologies, USA) for separation, a variable-wavelength UV–visible detector (CE Resources, Model UV-1) for detection, and an integrator (Hewlett-Packard, HP3396A) for data acquisition.

2.3. Extraction procedure by supercritical CO_2

The soil sample was weighed accurately to 5 g, placed in a 5-ml hand-tight SFE vessel and mixed thoroughly with glass beads (diameter 4 mm) to fill up the entire space. The glass beads were pre-cleaned by washing with acetone, chloroform and supercritical CO_2 before use. The packed SFE vessel was put into the oven maintained at 50°C. Organic modifier was then pumped at a flow-rate of 0.02 ml/min while the supercritical CO_2 flow-rate was kept at 0.18 ml/min. The liquid extractant formed by mixing methanol and supercritical CO_2 was then passed through the vessel at a specified flow-rate and pressure as regulated by the controller of the syringe pump for a given extraction time. The temperature of the restrictor was kept at 200°C and the extract was collected into a 2 ml methanol solution inside a 3-ml vial. For studying the recovery of individual phenols, 5 g of blank soil sample pre-cleaned twice with acetone, chloroform, and supercritical CO_2 was spiked with 10 μl of a 10 m*M* target phenol solution prior to performing the above extraction procedure.

2.4. Separation procedure by CEC

The fused-silica capillary with dimensions of 45 cm (25 cm packed with 3 μm ODS)×75 μm I.D. was conditioned overnight washing with the degassed mobile phase using a spring-loaded syringe pump. After the column was installed in the CEC system, the voltage was set at 5 kV for 30 min. It was then increased slowly to the separation voltage and kept for 30 min before the injection of the samples.

The volume of the SFE extract in the vial was reduced slowly from 1.8 ml to about 10 μl by bubbling nitrogen via a capillary tube through the sample vial at the rate of 20 ml/min with exact preconcentration factor determined gravimetrically. The solution was then injected electrokinetically at 10 kV for 2 s. The electrochromatogram was obtained by applying a running voltage of 20 kV to the degassed mobile phase (acetonitrile–Tris buffer) and the eluted phenols were detected by UV at 270 nm.

3. Results and discussion

3.1. Choice of phenols and soil

Most of the work on phenol analysis is concentrating on separating chlorophenols and nitrophenols, as they are more toxic and have differing polarities. The separation of alkyl-substituted phenols demands the use of a highly efficient separation as provided by CE. The 10 phenols selected in the present study consist of phenol, methylphenols (*p*-cresol and *o*-cresol), dimethylphenols (3,5-xylenol, 3,4-xylenol and 2,6-xylenol), trimethylphenol, ethylphenols (*p*-ethylphenol and *o*-ethylphenol), and *o*-isopropylphenol. They belong to a group of alkyl-substituted phenols commonly found in fuel and petrochemical products with similar chemical properties [5].

To produce a blank soil for recovery studies, soil samples were collected from topsoil near a contami-

nated site and extracted twice with supercritical CO_2 until no signal was detected by the CEC system. The blank soil was spiked by a mixture of 10 alkyl-substituted phenols for recovery test of phenols in the optimisation of the SFE extraction procedure.

3.2. Optimising supercritical fluid for extraction of phenols

In order to maintain the state of supercritical fluid for CO_2, the temperature must be kept above 32°C and pressure above 1072 p.s.i. (1 p.s.i.=6894.76 Pa). The effect of temperature and pressure of supercritical CO_2 on the extraction of selected phenols from soils are shown in Figs. 1 and 2. The results obtained indicated that higher phenol recovery from soils was obtained using lower extraction temperature and higher pressure. In order to maintain the supercritical fluid state during extraction and to reduce the leakage of CO_2 at high pressure and temperature, 50°C and 1200 p.s.i. were used in the supercritical CO_2 extraction.

The effect of extraction time and the addition of methanol on the extraction efficiency are shown in Figs. 3 and 4. A longer extraction time and a higher methanol content were found to give a more complete extraction. The extraction time was selected at 45 min to reduce the sample preparation time while keeping a constant phenol recovery. The percentage of methanol-to-supercritical CO_2 must be kept at 10% or above in order to achieve recovery close to 90%. In order to maintain a constant ratio of methanol in the extractant, the flow ratio of methanol-to-supercritical CO_2 was kept at 1:9 by careful control of the HPLC pump and the syringe pump during extraction. The effect of total extractant flow-rate on phenol recovery is shown in Fig. 5. The total extractant flow-rate should be kept below 0.20 ml/min. in order to achieve a high recovery and this can be done using a heated variable restrictor. Under the optimised SFE conditions with the addition of 10% (v/v) methanol as the organic modifier in the extractant and maintaining constant pressure and temperature at 1200 p.s.i. and 50°C for 45 min at a total extractant flow-rate of 0.2 ml/min, a 10-fold preconcentration factor was obtained and the procedure was shown to provide a satisfactory method for the extraction of alkylphenols from soil.

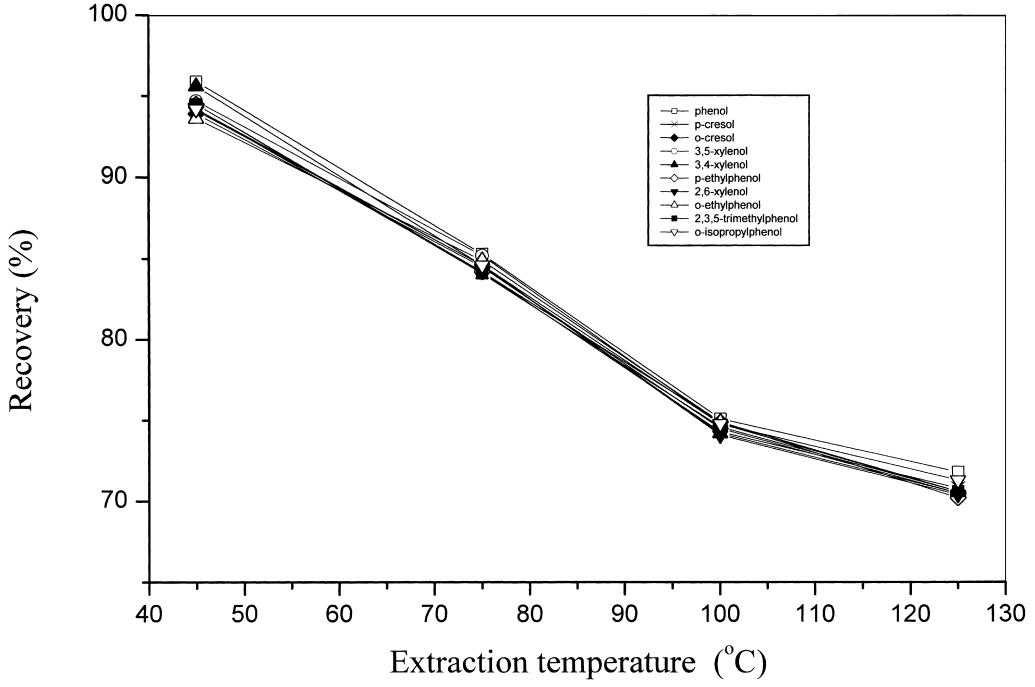

Fig. 1. The effect of extraction temperature on the recovery of phenols from soil extract. [Phenol]: 1 m*M*/each; extraction pressure: 1200 p.s.i., extraction time: 45 min; supercritical CO_2 flow-rate: 0.18 ml/min; methanol flow-rate: 0.02 ml/min; total flow-rate: 0.20 ml/min.

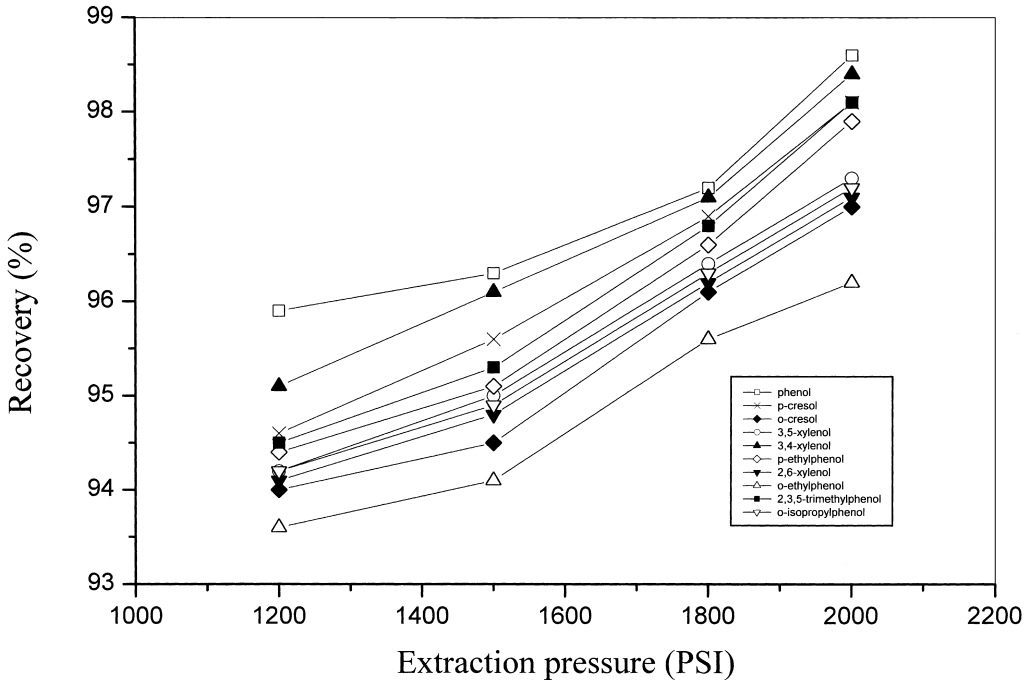

Fig. 2. The effect of extraction pressure on the recovery of phenols from soil extract. [Phenol]: 1 m*M*/each; extraction temperature: 50°C; extraction time: 45 min; supercritical CO_2 flow-rate: 0.18 ml/min; methanol flow-rate: 0.02 ml/min; total flow-rate: 0.20 ml/min.

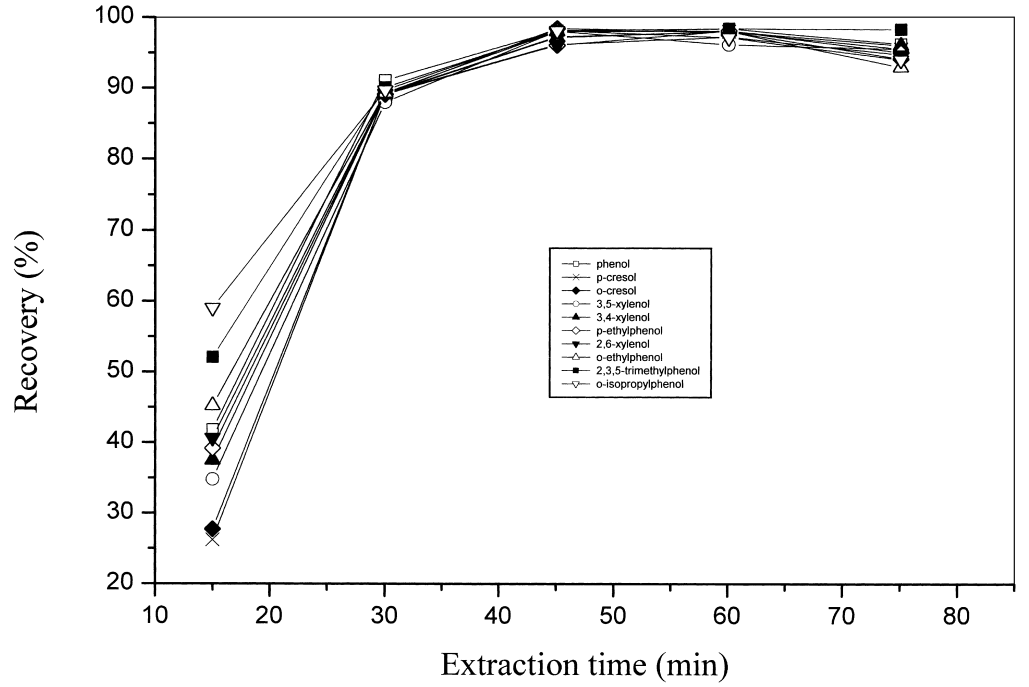

Fig. 3. The effect of extraction time on the recovery of phenols from soil extract. [Phenol]: 1 m*M*/each; extraction temperature: 50°C; extraction pressure: 1200 p.s.i.; flow-rate of supercritical CO_2: 0.18 ml/min; flow-rate of methanol: 0.02 ml/min.

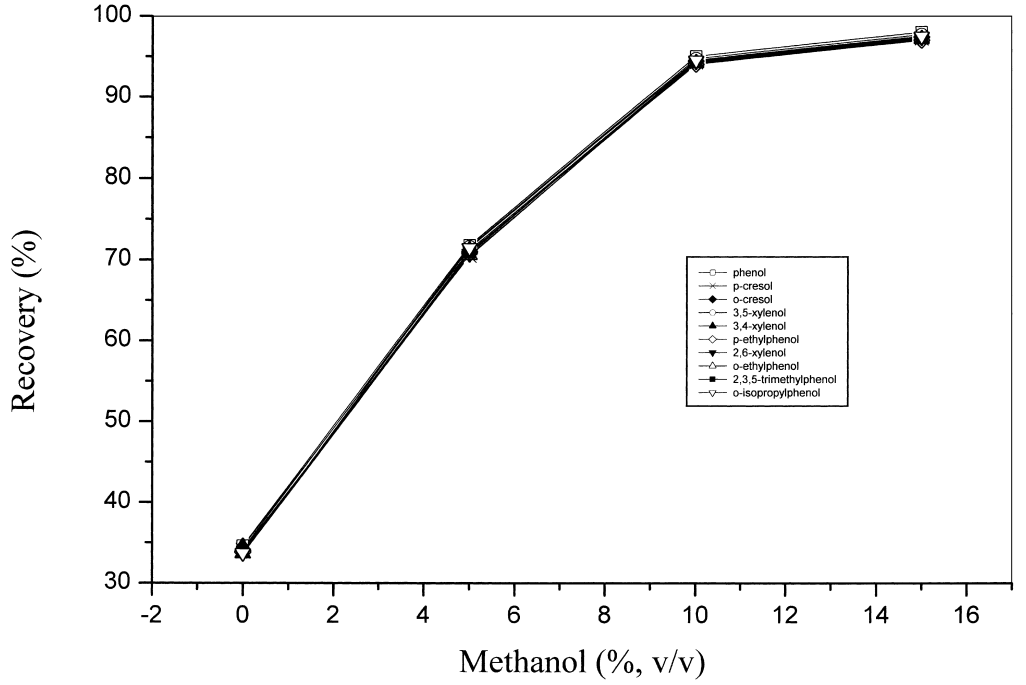

Fig. 4. The effect of the percentage of methanol in supercritical CO_2 on the recovery of phenols from soil extract. [Phenol]: 1 m*M*/each; extraction temperature: 50°C; extraction pressure: 1200 p.s.i.; extraction time: 45 min; total extractant flow-rate: 0.20 ml/min.

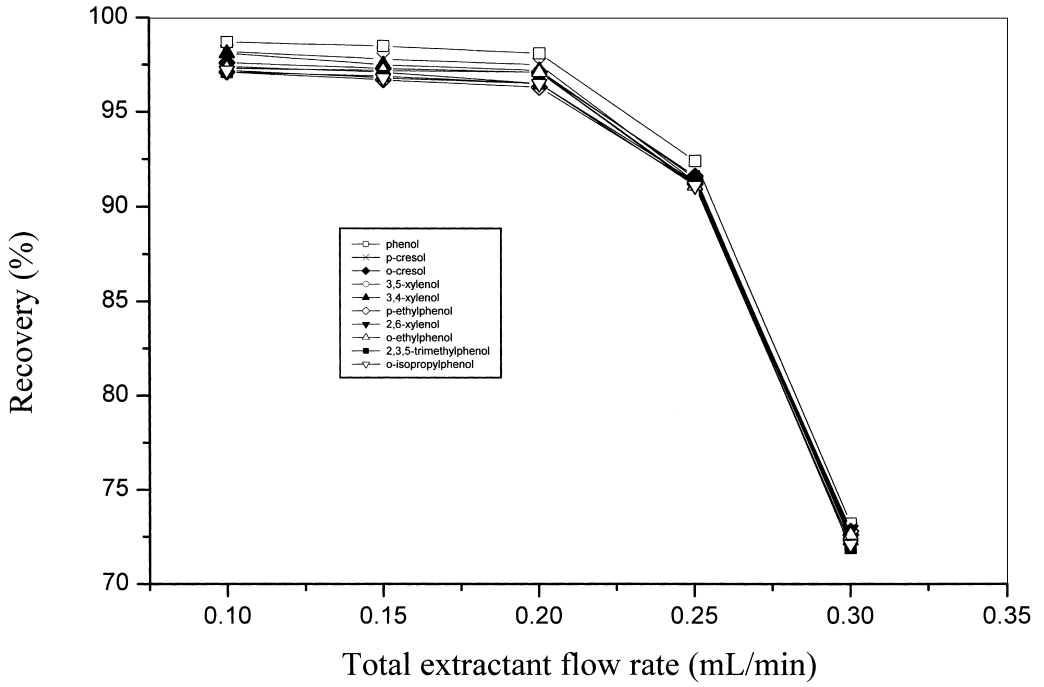

Fig. 5. The effect of total extractant flow-rate on the recovery of phenols from soil extract. [Phenol]: 1 m*M*/each; extraction temperature: 50°C; extraction pressure: 1200 p.s.i., flow ratio of methanol-to-supercritical CO_2: 1:9; extraction time: 45 min.

3.3. Optimising CEC for separation of alkylphenols

The effects of buffer pH and composition on the separation of alkylphenols are shown in Figs. 6 and 7. Use of a lower buffer pH was found to give a slightly improved separation as shown by the retention time difference of the alkylphenols investigated. Thus, pH 7 was selected as the Tris buffer pH. The major change in the buffer composition is the addition of acetonitrile. Its effect is shown in Fig. 7. In general, the lower the acetonitrile content, the better will be the separation amongst different phenols, though it suffers the disadvantage of having a longer CEC run time. Thus, the volume ratio of acetonitrile was kept at 35% in the Tris buffer.

The effect of applied voltage on phenol separation is shown in Fig. 8. In general, the differential in retention time for different phenols were found to increase using a lower applied voltage. Thus, 20 kV was selected as the applied voltage. The optimised operational parameters are summarised in Table 1 and the analytical parameters obtained under the optimised conditions are shown in Table 2. The results obtained indicate that very low detection limits were achieved for the 10 alkylphenols investigated, all showing working ranges with high linearity in concentrations covering three orders of magnitude and satisfactory repeatability within 10% relative standard deviation (RSD) except *o*-isopropylphenol with RSD 12%.

3.4. Application for analysis of phenols in soil

The detection limits and working ranges for the determination of alkylphenols in soils using the SFE–CEC method developed are shown in Table 3. The results indicate detection limits ranging from 0.0032 to 0.014 mg/kg soil and working ranges covering three orders of magnitude for all 10 phenols investigated. The values obtained are much lower in comparison to the levels of total phenol requiring remedial action as published by the US Environmental Protection Agency (EPA) [26] of less than 0.5 mg/kg soil.

The capillary electrochromatogram showing the separation of 10 phenols is given in Fig. 9. Two system peaks are identified and all 10 phenols are baseline resolved. Moreover, the order of elution follows the increasing complexity of the phenols

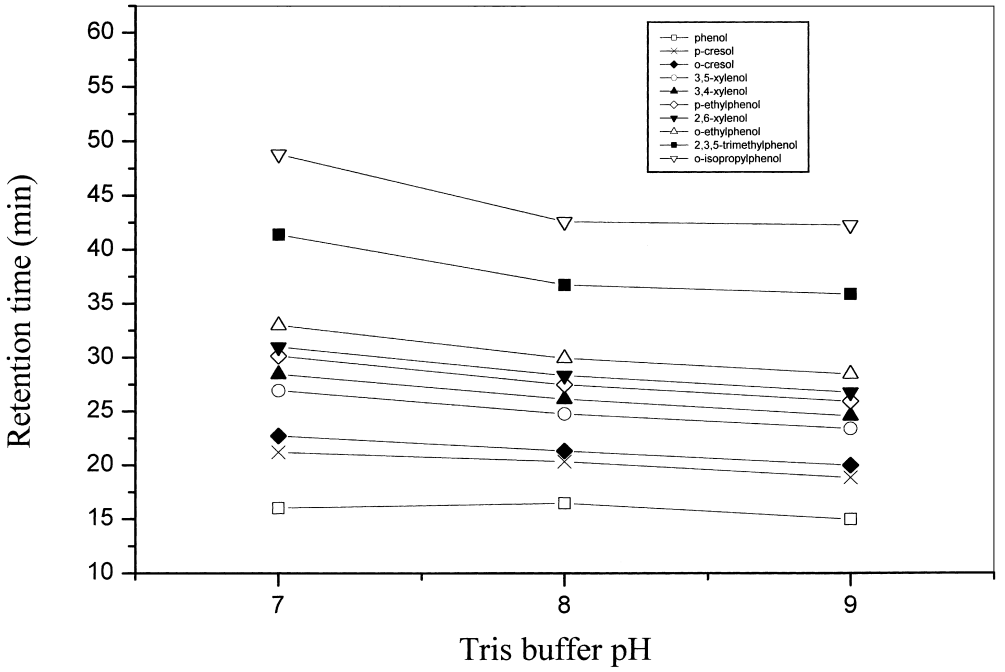

Fig. 6. The effect of Tris buffer pH on the retention time of phenols.

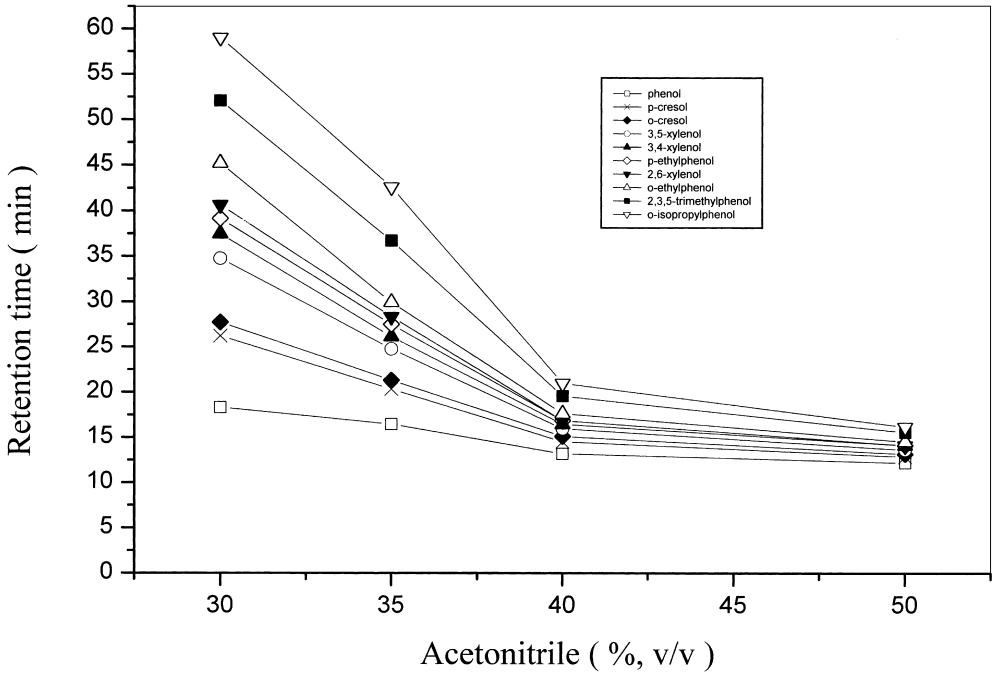

Fig. 7. The effect of acetonitrile on the retention time of phenols.

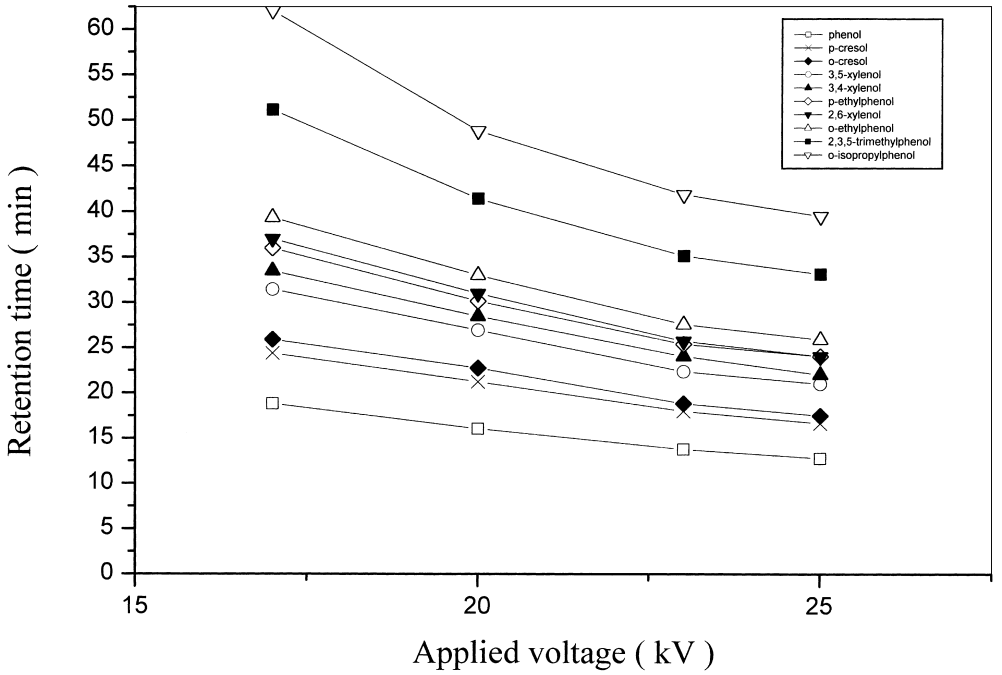

Fig. 8. The effect of applied voltage on the retention time of phenols.

Table 1
Optimised operational parameters for the separation of phenols by CEC

Column	Fused-silica capillary: 45 cm (25 cm packed with 3 μm ODS particles)×75 μm I.D.
Mobile phase	Acetonitrile–Tris buffer (4 m*M*) (35:65, v/v)
Tris buffer pH	7.0
Applied voltage	20 kV
Injection	10 kV/2 s
Temperature	25°C
Detection	UV at 270 nm

Table 2
The analytical parameters for the determination of phenols in methanol by CEC

Phenol	Detection limit[a] (m*M*)	Working range (m*M*)	Correlation coefficient	Repeatability (RSD, %)[b]
Phenol	0.0034	0.02–2.0	0.9984	5.6
p-Cresol	0.0042	0.02–1.8	0.9998	2.1
o-Cresol	0.0050	0.02–1.8	0.9995	6.3
3,5-Xylenol	0.0086	0.01–2.0	0.9994	5.4
3,4-Xylenol	0.0085	0.02–1.6	0.9983	4.2
p-Ethylphenol	0.0100	0.02–1.8	0.9991	4.9
2,6-Xylenol	0.0091	0.02–1.5	0.9926	5.3
o-Ethylphenol	0.0085	0.02–2.0	0.9995	7.4
2,3,5-Trimethylphenol	0.0100	0.02–2.0	0.9995	9.3
o-Isopropylphenol	0.0100	0.02–1.2	0.9945	12.1

[a] Detection limit based on: $S/N=2$.
[b] $n=3$.

investigated. Mono-, di- and trimethylphenols are eluted at longer times indicating a trend of increasing retention time for those with more alkyl-substituted groups. The increase of the carbon chain from methyl- to ethyl- and from aliphatic to branched isopropylphenols was found to give higher retention times. Thus, the retention time obtained from unknown samples could be used to predict the type of alkylphenols separated by CEC.

For a real soil sample obtained from the topsoil collected at a site contaminated by medical disinfectant, the SFE procedure developed was used to

Table 3
The detection limit and working range for the determination of phenols in soil using the SFE–CEC method developed

Phenol	Detection limit[a] (mg/kg)	Working range[a] (mg/kg)
Phenol	0.0032	0.019–1.88
p-Cresol	0.0045	0.022–1.95
o-Cresol	0.0054	0.022–2.16
3,5-Xylenol	0.011	0.012–2.44
3,4-Xylenol	0.010	0.024–1.95
p-Ethylphenol	0.012	0.024–2.20
2,6-Xylenol	0.011	0.024–1.83
o-Ethylphenol	0.010	0.024–2.44
2,3,5-Trimethylphenol	0.014	0.027–2.72
o-Isopropylphenol	0.014	0.027–1.63

[a] Soil sample: 5 g; total phenol extract: 2 ml; preconcentration factor: 10.

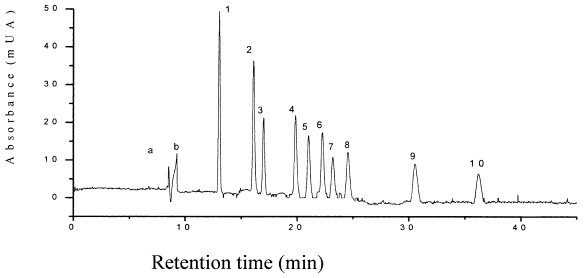

Fig. 9. Electrochromatogram showing the separation of 10 phenols using 3 μm ODS particles. Column dimensions: 45 cm (packed length)×75 μm I.D.; mobile phase: 35% acetonitrile in 4 m*M* Tris solution; applied voltage: 20 kV. Peaks (0.1 m*M* of each compound): (1) phenol; (2) *p*-cresol; (3) *o*-cresol; (4) 3,5-xylenol; (5) 3,4-xylenol; (6) *p*-ethylphenol; (7) 2,6-xylenol; (8) *o*-ethylphenol; (9) 2,3,5-trimethylphenol; (10) *p*-isopropylphenol; a, b: system peaks.

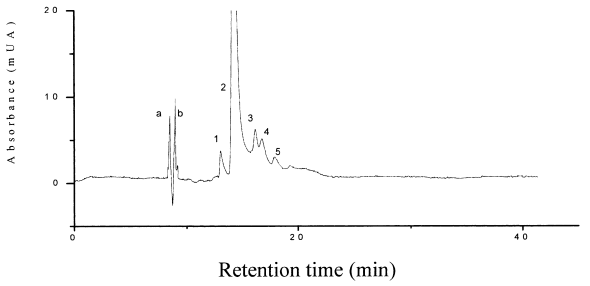

Fig. 10. Capillary electrochromatogram of phenols extracted from soil contaminated by medical disinfectant. Peaks: (1) phenol; (3) *p*-cresol; (4) *o*-cresol. 2, 5, Unknown peaks; a, b: system peaks. CEC conditions as in Fig. 9.

extract phenols out from the contaminated soil sample prior to separation by the CEC procedure developed. The capillary electrochromatogram obtained is shown in Fig. 10. Three phenols (phenol, *p*-cresol and *o*-cresol) were separated from the sample matrix amongst a huge unknown peak (peak 2). The high resolution obtained from the CEC column is shown able to separate trace levels of phenol from the presence of a large amount of other organic compounds. The concentrations of phenol, *p*-cresol and *o*-cresol determined by SFE–CEC are 0.12, 0.37 and 0.53 mg/kg, respectively.

4. Conclusions

Ten phenols were selected in the present study: phenol, methylphenols (*p*-cresol and *o*-cresol), dimethylphenols (3,5-xylenol, 3,4-xylenol and 2,6-xylenol), trimethylphenol, ethylphenols (*p*-ethylphenol and *o*-ethylphenol), and *o*-isopropylphenol. They belongs to a group of alkyl-substituted phenols commonly found in fuel and petrochemical products with similar chemical properties. A SFE–CEC technique has been developed to extract and determine the selected phenols in soil samples. The use of supercritical CO_2 with 10% (v/v) methanol as the organic modifier was found to provide a satisfactory method for the extraction of alkylphenols from soil with extraction at 1200 p.s.i. and 50°C for 45 min under a total extractant flow-rate of 0.2 ml/min. The phenols extracted were separated by CEC. The optimised conditions using a fused-silica capillary column of 45 cm (25 cm packed with 3 μm ODS)×75 μm I.D. are: applied voltage: 20 kV; mobile phase: acetonitrile–4 m*M* Tris (35:65) (pH 7.0), electrokinetic injection: 10 kV/2 s, and UV detection at 270 nm. Baseline resolution was achieved for the 10 selected phenols, together with the system peaks. Using SFE with a 10-fold preconcentration factor, the working ranges of the 10 phenols selected using the SFE–CEC method developed for phenol determination in soils are found to vary from 0.019 to 2.72 mg/kg soil and detection limits range from 0.0032 to 0.014 mg/kg soil for the alkyl-substituted phenols. The procedure developed has been applied successfully for the determination of phenols from real soil samples contaminated with medical disinfectant. Due to the high sensitivity and good selectivity of the SFE–CEC procedure developed, the method is shown to provide a rapid method for direct determination of phenol and alkyl-substituted phenol in soils, with capability of interfacing with mass spectrometry for confirmation of unknown peaks.

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References

[1] J. Green, B. Stierwalt, P. Grizzle, Fuel 64 (1985) 1571.
[2] M. Lompart, R. Lorenz, R. Cela, J. High Resolut. Chromatogr. 19 (1996) 207.
[3] E. Rodriguez Gonzalo, J. Perez Pavon, J. Ruzicka, G. Christinan, Anal. Chim. Acta 259 (1992) 37.
[4] M. Garcia Sanchez, J. Perez Pavon, B. Moreno Cordero, J. Chromatogr. A 766 (1996) 61.
[5] D. Armentrout, J. McNeal, M. Long, Anal. Chem. 51 (1979) 1039.
[6] O. Heemken, T. Norbert, B. Wenclawiak, Anal. Chem. 69 (1997) 2171.
[7] J. Snyder, R. Grob, M. McNally, T. Ostdyk, Anal. Chem. 64 (1992) 1940.
[8] P. Bartak, L. Cap, J. Chromatogr. A 767 (1997) 171.

[9] A. Egizabal, O. Zuloaga, N. Etxebarria, L. Fernandez, J. Madariaga, Analyst 123 (1998) 1679.
[10] M.P. Llompart, R.A. Lorenzo, R. Cela, K. Li, J.M.R. Bélanger, J.R.J. Paré, J. Chromatogr. A 774 (1997) 243.
[11] M. Asharf-Khorassani, S. Gidanian, Y. Yamini, J. Chromatogr. Sci. 33 (1995) 658.
[12] F. Santos, O. Jauregui, F. Pinto, M. Galceran, J. Chromatogr. A 823 (1998) 249.
[13] R.M. Smith, J. Chromatogr. A 856 (1999) 83.
[14] M.P. Llompart, R.A. Lorenzo, R. Cela, J. Microcol. Sep. 8 (1996) 163.
[15] M.P. Llompart, R.A. Lorenzo, R. Cela, J. Chromatogr. Sci. 34 (1996) 43.
[16] M.P. Llompart, R.A. Lorenzo, R. Cela, J. Chromatogr. A 723 (1996) 123.
[17] D. Louch, S. Motlagh, J. Pawlizyn, Anal. Chem. 64 (1992) 1187.
[18] Z. Zhang, J. Pawlizyn, Anal. Chem. 67 (1993) 1843.
[19] M. Lee, Y. Yeh, W. Hsiang, B. Hwang, J. Chromatogr. A 806 (1998) 317.
[20] O. Jauregui, E. Moyano, M. Galceran, J. Chromatogr. A 823 (1998) 241.
[21] D. Puig, I. Silgoner, M. Grasserbauer, D. Barcelo, Anal. Chem. 69 (1997) 2756.
[22] O. Fiehn, M. Jekel, J. Chromatogr. A 769 (1997) 189.
[23] G. Lamprecht, J. Huber, J. Chromatogr. A 667 (1994) 47.
[24] S. Terabe, K. Otsuka, K. Ichikawa, A. Tsucgiya, T. Ando, Anal. Chem. 56 (1984) 111.
[25] M. Saeed, M. Depala, D. Craston, I. Anderson, Chromatographia 49 (1999) 391.
[26] Table 1: Target Compounds List and Contract Required Quantitation Limits for OML03.2(CRQL) as Listed in a Draft Document Entitled ‘‘Multi-Media, Multi-Concentration, Organic Analytical Service for Superfund’’, Office of Solid Waste and Emergency Response, US EPA, May 1998.