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Short communication

An extraction technique for analytical sample preparation in aqueous solution based on controlling dispersion of ionic surfactant assemblies in isotachophoretic migration

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ABSTRACT

An extraction technique for analytical sample preparation in aqueous solution has been developed based on controlling dispersion of ionic surfactant assemblies. An extraction technique was realized based on controlling dispersion of the ionic surfactant assemblies in their isotachophoretic migration during the extraction by arranging the solutions of leading electrolyte, ionic surfactant and terminating electrolyte in order and applying voltage. Potential of the technique for analytical sample preparation in aqueous solution has been demonstrated by extracting a model sample of four alkylphenones, which were analyzed by HPLC after the extraction. The extraction showed concentration effects on all the four alkylphenones of butyrophenone, valerophenone, hexanophenone and heptanophenone in the model sample. The enrichment factors were 5.29, 7.70, 7.25 and 7.49 for the four alkylphenones of butyrophenone, valerophenone, hexanophenone and heptanophenone, respectively. Linear relationship was obtained with all the four alkylphenones between their chromatographic peak areas before and after the extraction in the range of concentration from 0.05 ppm to 1.5 ppm. The RSD of the chromatographic peak areas in triplicate extractions was 7.97%, 3.75%, 2.91% and 4.45% for butyrophenone, valerophenone, hexanophenone and heptanophenone, respectively. Advantages of the extraction technique developed include ease of operation, low reagent cost, no consumption of organic solvents and no requirement for additional phase separation.

1. Introduction

Although instrumental analysis has become an important subject in chemistry, biochemistry, materials science, as well as pharmaceutical, biological, and environmental fields, the preliminary step of sample preparation should not be overlooked in order to obtain accurate and quick analytical information in carrying out modern analytical tasks. As commented by Grob, a worldwide well-known chromatographer, sample preparation is the most error-prone and labor-intensive task in the analytical laboratory [1]. In general, sample preparation serves as two functions. One is to enrich analytes of low concentration to adequate levels of detection or quantification; the other is to isolate the desired components from sample matrices, which the instruments cannot handle directly. Because of its impact on nearly all subsequent steps, sample preparation is an essential step in an analytical process. A successful sample preparation step can improve quality of final analytical results. On the contrary, an inappropriate sample preparation step will render all efforts in vain in the later analytical steps. Hence, there has been a strong research trend of developing and improving sample preparation techniques in the field of analytical chemistry recently [2–10].

The sample preparation step in an analytical process typically involves an extraction procedure, which results in the isolation and enrichment of target analytes from a sample matrix [11]. Liquid–liquid extraction is a classical and common technique used for sample preparation of organic compounds from aqueous samples prior to chromatographic or electrophoretic analysis [12–14]. However, the main drawback of liquid–liquid extraction is that it is a time- and labor-intensive procedure and requires large amounts of high-purity solvents, which are expensive and toxic. Reviews have been published on the recent developments in non-traditional extraction technologies to address the problems mentioned above [15,16]. Solid-phase extraction (SPE) has been developed to overcome the drawbacks of classical liquid–liquid extraction and has been widely used in analytical sample preparation. Advantages of SPE include easier to automate, shorter processing times, low solvent consumption, attainable to remove matrix interferences and possible to extract polar analytes. However, SPE techniques have their own problems. The surface chemistry, and therefore sorption properties, of solid phases are not as reproducible as solvent properties. The mixed retention mechanism occurring sometimes can

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interfere with analyte recovery since elution solvents is ineffective for displacing ionically bound analytes to residual silanol groups of the silica substrate. Solid phases tend to have a higher level of contamination by manufacturing and packaging materials than is the case for solvents. Sample-processing problems in SPE related to the limited sorption capacity of sorbents and analyte displacement or plugging of sorbent pores by matrix components easily pass unnoticed, resulting in changes in analyte recovery [17]. The need to pre-filter the real-life samples to avoid clogging and the steps of cleaning and elution in SPE may lead to analyte loss and contamination.

Surfactants have been extensively used in various analytical techniques [18,19]. Among many others, micellar electrokinetic chromatography (MEKC) [20] together with its on-line concentration scheme of sweeping [21] is a good example, of successful application of surfactants in analytical chemistry. Surfactant mediated extractions are environmentally friendly and cost effective [22–24]. In a surfactant mediated extraction, either phase separation after the extraction or control of dispersion of the surfactant assemblies during the extraction can be employed in principle. Generally, phase separation after extraction are realized by evoking a chemical or physical perturbation in surfactant mediated extraction systems [25,26]. For example, phase separation in cloud point extraction is typically made by heating above the cloud point temperature. A nonionic surfactant micelle solution will separate after a certain time into two phases: a surfactant-rich layer and a bulk aqueous phase [27]. Phase separation in coacervation of ionic surfactant assemblies can be achieved by changing pH [28,29] or adding concentrated aqueous ionic salt solution and introducing organic solvents [30]. In this short communication, we explore potential of the extraction based on controlling dispersion of ionic surfactant assemblies for sample preparation of neutral compounds in aqueous solution. Controlling dispersion of the ionic surfactant assemblies in their isotachophoretic migration during the extraction was realized by arranging the solutions of leading electrolyte, ionic surfactant and terminating electrolyte in order and applying voltage. Comparing with the conventional organic-solvent-based liquid–liquid extraction, advantages of the sample preparation technique presented in this communication like other surfactant mediated extractions include: reduction in costs associated with organic solvent purchase, storage, and disposal, as well as the associated worries regarding toxicity or hazards such as fire and explosion; the capacity to concentrate a plethora of analytes with almost quantitative recoveries; the preconcentration factors to be comparable or superior to other schemes, and adjustable by varying the amount of surfactant [24].

2. Experimental

2.1. Chemicals

Butyrophenone, valerophenone, hexanophenone and heptanophenone were obtained from TCI (Tokyo, Japan). Sudan I was a product of Dr. Ehrenstorfer (Augsburg, Germany). The other chemicals were purchased from Sinopharm Chemical Reagent Co. (Shanghai, China). Sodium chloride, phosphoric acid, sodium dodecyl sulfate (SDS), sodium octane sulfonate and sodium gluconate (NaGlu) were of analytical grade. Acetonitrile, methanol and ethanol were of HPLC-grade. Distilled water was used throughout the experiments.

2.2. Standard solutions and samples

Stock solutions (1.00 mg/mL) of individual alkylphenones were prepared in ethanol and were stored at 4 °C in a refrigerator when not in use. Working solutions containing mixtures of the alkylphenones were prepared by mixing appropriate quantities of the stock solutions and diluting to desired concentrations with 100 mM H_3PO_4.

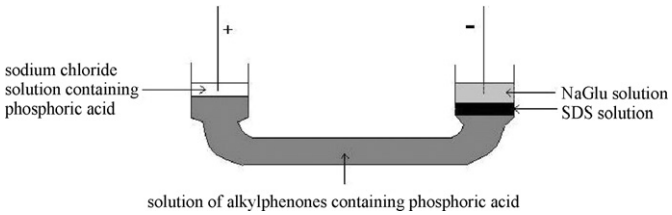

Fig. 1. Schematic of the extraction device.

2.3. Apparatus

A d.c. power supply used was a model of ES 0300–0.45 from Delta Power Supplies (Delta Electronika, Zierikzee, The Netherlands) with programmable voltage in the range of 0–300 V, providing currents in the range of 0–450 mA. A laboratory-built electrolytic cell consisted of two 1.5 mL glass vials connected with a 24 cm U-shaped glass tube of an internal diameter of 13.8 mm. Platinum wires were used for both the anode and cathode. The HPLC system used in this work consisted of an LC-10 AT pump (Shimadzu Kyoto, Japan) and a Linear UVIS 200 ultraviolet/visible (UV/vis) detector (Alltech, USA). The HPLC separation was performed on a 250 mm × 4.6 mm I.D., 5 μm, VP–ODS column (Shimadzu, Kyoto, Japan).

2.4. Procedure of the surfactant extraction

The following solutions were filled sequentially into the electrolytic cell in order with medical syringes (as shown in Fig. 1): 0.2 mL 1400 mM sodium chloride solution containing 100 mM phosphoric acid as leading electrolyte; 2.5 mL mixed solution of the alkylphenones containing 100 mM phosphoric acid as sample; 0.2 mL (equal to zone length of 33 mm) 100 mM SDS, and 0.5 mL 800 mM NaGlu as terminating electrolyte. After the solutions were filled into the electrolytic cell, a voltage of 300 V was immediately applied to the cell with the anode in contact with the leading electrolyte solution and the cathode in contact with the terminating electrolyte solution. After applying voltage for 35 min, a surfactant-rich zone of about 2 mm in length was collected into a centrifuge tube with a syringe and diluted with the same volume of acetonitrile for the HPLC analysis (acetonitrile was added to destroy the surfactant assemblies and to facilitate the HPLC analysis). Then, the power supply was turned off.

2.5. HPLC analysis

Mobile phase consisting of methanol and water (80%:20%, v/v) was used. Flow rate of the mobile phase was set at 1.0 mL/min. Sample introduction was carried out using a Rheodyne six-port switching valve with a 20 μL loop. Detection was made at a wavelength of 245 nm. Chromatographic data were collected and recorded using CSW (Chromatography Station for Windows) (DataApex, Prague, Czech Republic).

3. Result and discussion

3.1. Dependence of dispersion of the ionic surfactant assemblies on the concentration of the leading electrolyte

As long ago as 1897, the theoretical groundwork for isotachophoresis was laid down by Kohlrausch [31]. Fundamentals of

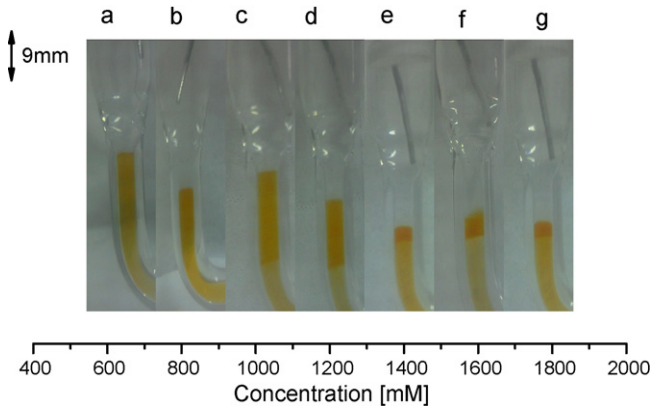

Fig. 2. Visualized dependence of dispersion of the ionic surfactant assemblies on concentration of the leading electrolyte. Leading electrolyte (at anodic side): sodium chloride solution containing 100 mM phosphoric acid; terminating electrolyte (at cathodic side): 800 mM sodium gluconate solution; initial surfactant solution: 100 mM SDS containing trace of Sudan I. Sample solution: 2.5 mL mixture of butyrophenone, valerophenone, hexanophenone and heptanophenone (1 ppm each) in 100 mM phosphoric acid solution. Concentrations of sodium chloride were 600 mM, 800 mM, 1000 mM, 1200 mM, 1400 mM, 1600 mM and 1800 mM from a to g. Voltage: 300 V.

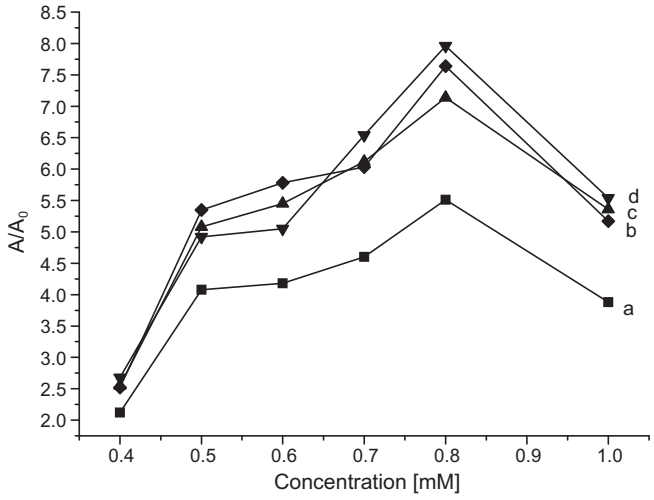

Fig. 3. Effect of concentration of sodium gluconate on enrichment of the alkylphenones. Leading electrolyte: 1400 mM sodium chloride solution containing 100 mM phosphoric acid; terminating electrolyte: 400–1000 mM sodium gluconate solution; initial surfactant solution: 100 mM SDS; sample: a 2.5 mL mixture of butyrophenone, valerophenone, hexanophenone and heptanophenone (1 ppm each) in 100 mM phosphoric acid solution. Voltage: 300 V. a: (■) butyrophenone; b: (♦) valerophenone; c: (▲) hexanophenone; d: (▼) heptanophenone.

isotachophoresis were comprehensively discussed in a monograph authored by Everaerts et al. [32]. According to Kohlrausch's regulating function [31], dispersion and zone length of the ionic surfactant assemblies can be effectively controlled in isotachophoretic migration. As stated in Section 2, solutions of the leading electrolyte of sodium chloride, sample of alkylphenones, the surfactant of SDS and the terminating electrolyte of NaGlu were arranged in order in the electrolytic cell to meet the requirements of isotachophoretic migration. Hence, zone length of the SDS assemblies could be controlled by manipulating concentration of the leading electrolyte of sodium chloride. The dependence of zone length of the SDS assemblies on concentration of the leading electrolyte of sodium chloride was investigated. During the investigation, 0.2 mL (equal to zone length of 33 mm) of 100 mM SDS and 0.5 mL of terminating electrolyte of 800 mM NaGlu were used initially and constantly, but concentration of sodium chloride was varied in the range of 600–2000 mM. After applying voltage for 35 min, no surfactant-rich zone was observed when concentration of the leading electrolyte of sodium chloride was 800 mM or lower. When concentration of the leading electrolyte of sodium chloride was 1000 mM or higher, a surfactant-rich zone was observed clearly. The resultant surfactant-rich zones were photographed as shown in Fig. 2. Sudan I was used as a tracer to enhance visualization effect in the figure. However, the surfactant-rich zone could be seen in the absence of it. Length of the surfactant-rich zones was about 2 mm and nearly constant when concentration of sodium chloride was in the range of 1400–1800 mM. With the initial surfactant zone length of about 33 mm and the final length of about 2 mm of the surfactant-rich zone, the surfactant zone was contracted by a factor of about 16.5 fold. When concentration of sodium chloride was 1400 mM or higher, further contraction of the surfactant-rich zone was not observed. The nearly constant length of the surfactant-rich zones was likely a result of counterbalanced convection caused by Joule heating. During the experiments, it was found that current increased with concentration of the leading electrolyte of sodium chloride. When concentration of sodium chloride reached 2000 mM, current exceeded 25 mA.

3.2. Evaluation on the extraction for sample preparation

Since the extraction was carried out under the conditions of isotachophoretic migration of the ionic surfactant assemblies, the effective mobility, effective charge and concentration of the terminating ion all may have influences on the results of the extraction of the alkylphenones [33,34]. Octane sulfonate and gluconate of low mobilities were examined as the terminating ions. When octane sulfonate of 500 mM was used as the terminating ion, no surfactant-rich zone was observed. Possible reasons might be that the effective mobility of octane sulfonate was not low enough or/and formation of mixed micelles of octane sulfonate and dodecyl sulfate due to their structural similarity. The critical micelle concentration (CMC) of sodium octane sulfonate was reported to be 130 mM [35]. Surprisingly, when gluconate was used as the terminating ion, a surfactant-rich zone was clearly seen. This might be due to high hydrophilicity of gluconate, its incorporation into the SDS assemblies was not expected. The effect of concentration of sodium gluconate on enrichment of the alkylphenones was investigated. Fig. 3 shows area ratio of chromatographic peaks of the extracted alkylphenones to those originally present in the samples under the same chromatographic conditions, where A represents chromatographic peak area of the alkylphenones finally collected in the surfactant-rich zone and A_0 represents peak area of the alkylphenones originally present in the sample in Fig. 3. Concentration of sodium gluconate was examined in the range of 400–1000 mM. It can be noted that the maximum area ratio of the chromatographic peaks for the alkylphenones were obtained in Fig. 3 with concentration of 800 mM of the terminating ion of gluconate. Therefore, 800 mM of sodium gluconate was used in the following experiments in this work.

The dependence of enrichment factors of the alkylphenones on concentration of the leading electrolyte of sodium chloride was examined in the range of 800–1800 mM. The results are shown in Fig. 4. The enrichment factors increased significantly with concentration of the leading electrolyte up to 1400 mM and then increased slightly when concentration of the leading electrolyte was higher than 1400 mM. The enrichment factors were related to some extent with length of the surfactant-rich zone. The longer the length of the surfactant-rich zone, the lower was the enrichment factor. This can be evidenced by Figs. 2 and 4. Difference in length of the surfactant-rich zones implied different mole fraction of the surfactant in the surfactant-rich zones, that is, a surfactant-rich zone of shorter length had greater mole fraction of the surfactant. Conse-

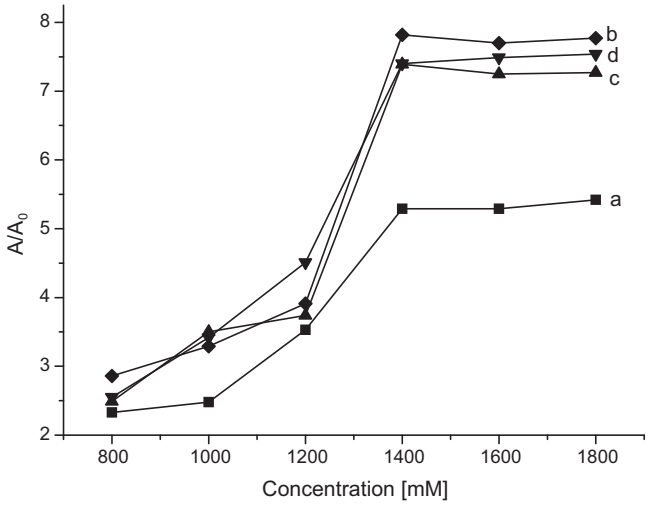

Fig. 4. Effect of concentration of sodium chloride on enrichment of alkylphenones. Leading electrolyte: 800–1800 mM sodium chloride solution containing 100 mM phosphoric acid; terminating electrolyte: 800 mM sodium gluconate solution. Other conditions as those in Fig. 3. a: (■) butyrophenone; b: (♦) valerophenone; c: (▲) hexanophenone; d: (▼) heptanophenone.

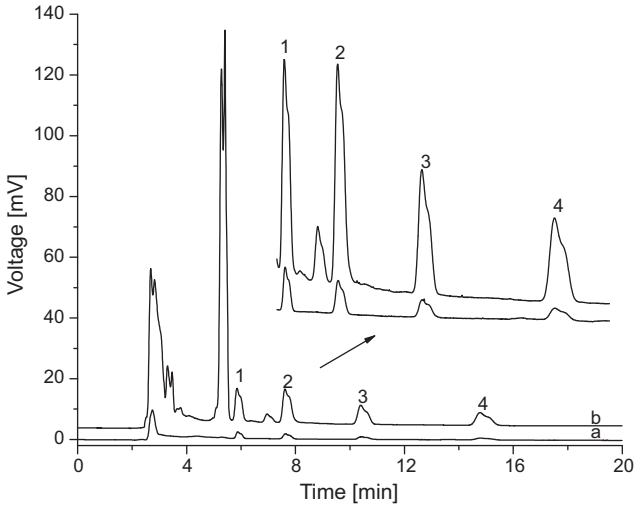

Fig. 5. Comparison of HPLC chromatograms obtained with samples of the alkylphenones before and after the extraction. Chromatographic conditions as described in Section 2. Extraction conditions: leading electrolyte: 1400 mM sodium chloride solution containing 100 mM phosphoric acid; terminating electrolyte: 800 mM sodium gluconate solution; initial surfactant solution: 100 mM SDS. Sample: a 2.5 mL mixture of butyrophenone, valerophenone, hexanophenone and heptanophenone (1 ppm each) in 100 mM phosphoric acid solution. Voltage: 300 V. (a) Before extraction. (b) After extraction. Peaks identification: 1, butyrophenone; 2, valerophenone; 3, hexanophenone; 4, heptanophenone.

quently, variation in size and shape of the surfactant assemblies and in the partition coefficient of the solutes might occurred [36,37].

A mechanism of the surfactant extraction of the alkylphenones was proposed. Once voltage was applied across the electrolytic cell for the extraction, the leading ion of chloride, surfactant assemblies of SDS and the terminating ion of gluconate migrated towards the anode in this order at the same velocity. Dispersion of the SDS assemblies was controlled as the result of meeting the requirement of Kohlrausch's regulating function. While the SDS assemblies were migrating, there was partition of the alkylphenones between the SDS assemblies and the aqueous sample matrices. When the SDS assemblies passed through the whole sample zone, the surfactant extraction of the alkylphenones was completed. Enrichment of the alkylphenones in the surfactant-rich zone was a function of the partition coefficients of alkylphenones between the SDS assemblies and the aqueous sample matrix.

A standard mixture of butyrophenone, valerophenone, hexanophenone and heptanophenone (1 ppm each) was tested as a model sample. The following solutions were arranged in the electrolytic cell for the extraction: 0.2 mL of 1400 mM of sodium chloride solution containing 100 mM phosphoric acid (at anodic side); 2.5 mL of 1 ppm sample solution containing 100 mM phosphoric acid; 0.2 mL of 100 mM SDS and 0.5 mL of 800 mM sodium gluconate (at cathodic side). Voltage of 300 V was applied. The surfactant assemblies migrated passing through the sample solution zone. After applying voltage for 35 min, a surfactant-rich zone about 2 mm in length could be obviously seen near the boundary between the sample solution and the leading electrolyte. The surfactant-rich zone was collected into a small vial by using a microsyringe and diluted with the same volume of acetonitrile for the HPLC analysis. Chromatograms for the standard solutions before and after the extraction are shown in Fig. 5. By comparing trace b with trace a in Fig. 5, it can be known that all the alkylphenones were concentrated after the extraction. The enrichment factors for butyrophenone, valerophenone, hexanophenone and heptanophenone were 5.29, 7.70, 7.25 and 7.49 respectively. Higher enrichment factors may be achievable by improving cell geometric design, such as a belly shaped electrolytic cell, and utilizing appropriate chemical modifiers, such as nonionic surfactants. The Log *P* values of the four alkylphenones were calculated using the ALOGPS 2.1 program of Virtual Computational Chemistry Laboratory [38]. They were 2.69, 3.15, 3.62 and 4.09 for butyrophenone, valerophenone, hexanophenone and heptanophenone, respectively. General tendency of large enrichment factors with increasing the Log *P* values was observed. Quantitative correlation of the Log *P* with the enrichment factors was not obtained. The reasons might be duo to the effect of temperature of the extraction system, the effect of different structure of the SDS assemblies from the bulky organic solvents, spatial distribution of the alkylphenones in the SDS assemblies, or other unknown factors. Clarifying the reasons requires further studies.

Instead of sequential injection of the solutions, we examined enrichment effect of the extraction by mixing SDS with the samples prior to the experiments. When 100 mM SDS was added to and mixed with the sample solutions before the extraction, large current and considerable Joule heat were generated after applying voltage. When concentration of SDS was reduced to 10 mM, the enrichment factors for the four alkylphenones in the extraction were obtained not as high as those with the sequential injection of the leading electrolyte solution, the sample, the SDS solution and the terminating electrolyte solution.

For the extraction method to be useful for sample preparation, linear relationship between original concentrations of analytes and detector responses of the extracted analytes should be held. To check such a linear relationship, we extracted and analyzed butyrophenone, valerophenone, hexanophenone and heptanophenone mixtures in the range of original concentrations from 0.05 ppm to 1.5 ppm. Good linear relationship was obtained in terms of chromatographic peak area for all the four alkylphenones before and after the extraction. The regression equations of calibration lines are given below. For butyrophenone, $y = 60.057x + 2.3713$ (R^2, 0.9914); for valerophenone, $y = 64.741x + 1.192$ (0.9905); for hexanophenone, $y = 47.57x + 2.1269$ (0.993) and for heptanophenone, $y = 42.075x + 2.5503$ (0.9922).

The relative standard deviation (RSD) of chromatographic peak area was determined for the extraction of the four alkylphenones of original concentration of 1 ppm. For triplicate extractions, the RSD was 7.97%, 3.75%, 2.91% and 4.45% for butyrophenone, valerophenone, hexanophenone and heptanophenone, respectively. It indicated acceptable precision for trace analysis in many

cases. Improvement on the precision may be obtainable using an internal standard.

In this short communication, potential of the extraction of neutral compounds from aqueous solution has been demonstrated using alkylphenones based on controlling dispersion of ionic surfactant assemblies in their isotachophoretic migration. This extraction scheme may also be useful for extraction of hydrophobic organic anions because of their solubilization in surfactant assemblies. Moreover, the extraction scheme plays a role of clean-up for impurities of anionic and hydrophilic compounds presented in sample matrices. In spite of the fact that the anionic and hydrophilic compounds could be enriched in front of or at rear of the surfactant-rich zone depending on relative magnitude of their mobilities to that of the SDS assemblies in the extraction process, they would be separated from the extracted target analytes as only the surfactant-rich phase was collected at the final stage of the extraction.

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