Novel approach to the analysis and use of fullerenes in capillary electrophoresis[☆]

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Abstract

The analysis and use of fullerenes in capillary electrophoresis (CE) was investigated. Sodium dodecyl sulfate (SDS) was used to solubilize fullerenes C_{60}, C_{70}, and a mixture of C_{60} and C_{70} in water. The behavior of the solutions of the C_{60}– and C_{70}–SDS complexes was examined by CE with on-line UV–Vis diode array detection. This study included the use of a C_{60}–SDS complex as a new method of micellar electrokinetic chromatography (MEKC) for the separation of polycyclic aromatic hydrocarbons (PAHs) using CE with uniwavelength detection. Since SDS micelles act as a pseudostationary phase in which the PAH compounds partition with their hydrophobic interior, the addition of C_{60} within the micelles enhanced separation of the PAHs. The preliminary results using C_{60}-MEKC with SDS were compared to those obtained with MEKC with SDS. The capillary electrophoretic separations were performed in 10 mM borate–phospate buffer with 100 mM SDS at pH 9.5.   © 2000 Elsevier Science B.V. All rights reserved.

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1. Introduction

Since the discovery of the buckminsterfullerene (C_{60}) produced from the vaporization of graphite [1], several methods for the synthesis [2–6], purification [7–10], and material science applications [11–15] of fullerenes and fullerene derivatives of various sizes have evolved. One application of C_{60} and C_{70} fullerenes and fullerene derivatives that is of major interest is their use as biologically active compounds in medicinal chemistry [16–21]. In particular, re-search efforts include investigating water-soluble fullerene complexes as virucidal agents [17,19,20], for membrane transport [18], and in studies of their behavior in drug transport mechanisms [16].

Because the solubility of fullerenes has been limited to nonaqueous solvents, such as hexane, toluene and dichloromethane, fullerenes could not be used directly for medicinal purposes. However, researchers have discovered ways of dissolving fullerenes in aqueous environments to circumvent their insolubility in aqueous solvents and their toxicity caused by organic solvents for physiological purposes. It has been found that fullerenes, such as C_{60} and C_{70}, can be dissolved in aqueous media through derivatization [25–29] or complexation with liposomes, micelles, or other aggregate systems [16,22–24]. These fullerene complexes have been

studied for drug transport in various pharmaceutical applications [16,21].

In the past 5 years the physical and chemical properties of fullerene complexes dissolved in water in the presence of complexing agents have been studied. Agents such as γ-cyclodextrin [22,24], lipids [16,23], and surfactants [16], which aggregate in water, have been investigated. The aggregate has a hydrophobic interior or core and a hydrophilic exterior. The fullerene favorably associates with the hydrophobic interior of the aggregate. Simultaneously, the complexation of C_{60} and/or C_{70} with the aggregate is exposed to an aqueous environment due to the hydrophilic exterior, making the fullerene soluble. Fig. 1 displays an illustration of C_{60} associated γ-cyclodextrin, lipids, and surfactants.

High-performance liquid chromatography (HPLC) has been the technique of choice for the qualitative and quantitative analysis of individual fullerenes and fullerene mixtures. A reversed-phase separation is the HPLC method most frequently used that involves
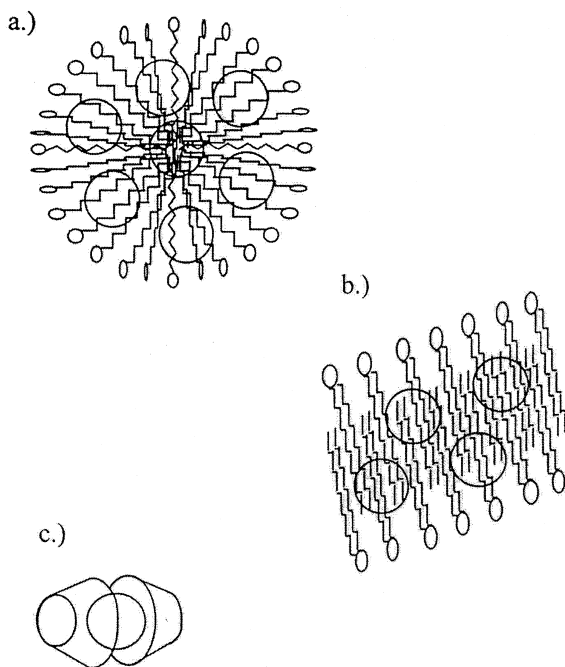
monomeric or polymeric C_{18} stationary phases [30–39]. Other stationary phases include γ-cyclodextrin [7], tetraphenylporphyrin [8], porphyrin [9], and heavy atom [10]. Although CE techniques have been used to separate fullerenes, namely capillary electrochromatography (CEC) [40] and non-aqueous capillary electrophoresis (NACE) [41], both techniques used non-aqueous solvent systems. A CEC separation method for fullerenes using a non-aqueous solvent was developed by Whitaker and Sepaniak [40]. In addition, Wan et al. [41] developed an NACE method using the solvophobic interaction of tetraalkylammonium ions to separate C_{60} and C_{70}.

However, there have been no reports of the analysis of C_{60} or C_{70} in an aqueous buffer by CE. Since fullerenes are soluble in water when complexed with a surfactant or other additive, capillary electrophoresis could be used as a technique to separate fullerenes by charge-to-size ratio via complexation. Fig. 2 illustrates the possible mechanism for the separation of C_{60}–sodium dodecyl sulfate (SDS) from C_{70}–SDS by CE. In addition, we propose that fullerenes, such as C_{60}, suspended in buffer solution as a SDS complex could separate other compounds by CE. The following preliminary investigation of the CE analysis of C_{60} and C_{70}
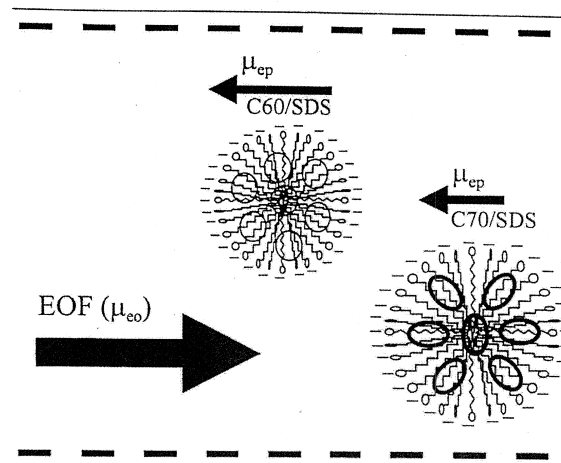

Fig. 1. Illustration of the incorporation of C_{60} and C_{70} within the aggregation of (a) surfactants, (b) lipids and (c) γ-cyclodextrin.

Fig. 2. Illustration demonstrating the separation of C_{60}–SDS and C_{70}–SDS in capillary electrophoresis (CE) using buffer. EOF, electroosmotic flow; μ_{ep}, electrophoretic mobility.

fullerene complexes with SDS and the use of the C_{60} complex for the separation of polyaromatic hydrocarbons (PAHs) in C_{60}-MEKC are described.

2. Experimental

2.1. Chemicals and reagents

A C_{60}–C_{70} mixture and C_{60} (99.5%) were purchased from Southern Chemical Group (Tucker, GA, USA). Yuri Gorfinkl of Southern Chemical Group donated C_{70} (98%). SDS was purchased from Fluka (Milwaukee, WI, USA). Sodium tetraborate, sodium phosphate tribasic and HPLC-grade toluene were purchased from Sigma (St. Louis, MO, USA).

2.2. Capillary electrophoresis instruments

The capillary electrophoresis instrument with on-line diode array detection was a Hewlett-Packard 3D/CE system (Palo Alto, CA, USA). The signals were acquired by using the Chemstation software package (Hewlett-Packard). On-line ultraviolet–visible (UV–Vis) spectra were detected within a range of 190–600 nm. Electropherograms were obtained using the single detection wavelength of 254 nm, unless specified otherwise. The capillary cassette temperature was set at 20°C. Pressure injection at 20 mbar was employed when introducing the samples.

The Waters Quanta 4000E capillary electrophoresis instrument (Milford, MA, USA) with uniwavelength detection was used for the separation of polyaromatic hydrocarbons by SDS and SDS–C_{60} complex buffer systems. The wavelength of detection was performed at 254 nm. Injections were gravity fed with a height displacement of 10 cm. The separations were recorded using a Spectra-Physics SP4270 model integrator from Thermo Separation Products (San Jose, CA, USA).

Fused-silica capillary tubing of 75 μm internal diameter (I.D.) and 375 μm outer diameter (O.D.) was purchased from Polymicro Technologies (Phoenix, AZ, USA). The total length of the capillary was 35 cm with an effective length of 28 cm.

2.3. UV–Vis spectrometer

Off-line UV–Vis spectra were obtained with a Beckman DU 7500 Spectroplot (Fullerton, CA, USA). The spectra were observed through the use of Grams 386 software.

All solutions were degassed in an ultrasonic sonicator. A Corning model pH meter (Corning, NY, USA) was employed to measure the pH of the buffer being used.

2.4. Procedure

2.4.1. SDS buffer
A stock solution of the SDS buffer was prepared by dissolving the appropriate amount of SDS in 10 mM borate–10 mM phosphate buffer to make a 100 mM SDS buffer solution. The buffer was then sonicated and degassed for approximately 1 h and filtered using a nylon 0.45-μm acradisc filter before use.

2.4.2. Samples
All samples involving C_{60}, C_{70}, and the mixture of C_{60} and C_{70} dissolved in 100 mM SDS solution were prepared in the following manner. First, approximately 1 mg of the fullerene was weighed and placed into a solution containing 100 mM SDS in deionized water. This solution was then refluxed for 6 h above 100°C under constant stirring conditions. This procedure was followed to provide maximum concentration of the fullerenes in solution. The saturated fullerene–SDS solutions were then allowed to stand at room temperature to allow for precipitation of any remaining fullerenes left undissolved in solution. The samples were sonicated and degassed after dissolution.

2.4.3. C_{60}–SDS buffer
The C_{60}–SDS separation buffer for the polyaromatic hydrocarbon analysis was prepared by combining two methods mentioned above. The 100 mM SDS, 10 mM borate phosphate buffer was refluxed with C_{60} under the appropriate conditions. The maximum concentration of C_{60} was dissolved in enough volume (~100 ml) of SDS borate–phosphate

buffer to be used for the CE runs and for replenishing the buffer reservoirs.

2.4.4. Dilutions

Where there is an indication that a series of dilutions were performed an Oxford Benchmate micropipet (200–1000 μl) was used. The appropriate aliquots of the saturated fullerene solutions were taken and diluted with 100 mM SDS solution.

3. Results and discussion

3.1. C_{60}–SDS sample

After C_{60} was saturated in an aqueous solution of 100 mM SDS, a yellowish–brown color resulted. Since C_{60} alone is insoluble in water, the observed color change is an indicator that solubility has occurred [23]. An off-line UV–Vis spectra of the C_{60}–SDS complex is shown in Fig. 3. The maxima

of 219.9, 263.8, 341.4, and 443.3 nm are observed. The spectra bands in the region of 210, 260, 330, and 450 nm are characteristic of C_{60} in similar micellar environments such as Triton X-100 and Triton X-100 R-S [16].

The C_{60}–SDS sample was then analyzed by capillary electrophoresis using a 10 mM phosphate–borate buffer with 100 mM SDS at a pH of 9.5. Replicate injections were performed with reproducible results (0.1% RSD). The electropherogram in Fig. 4a shows the migration of a C_{60}–SDS sample. The electrophoretic mobility was calculated to be -5.88×10^{-4} cm^2/V s. The major signal at 19.8 min in Fig. 4a is the C_{60}–SDS complex. The identity of the complex is confirmed by the on-line UV–Vis spectra shown in Fig. 4b, because it is similar to the off-line UV–Vis spectra of C_{60}–SDS in Fig. 3. The maxima were 216.5, 260.4, and 340.5 nm.

Based on work conducted by Bensasson et al., the relative absorbance ratios within the spectral data reveal that C_{60} molecules form aggregates within the
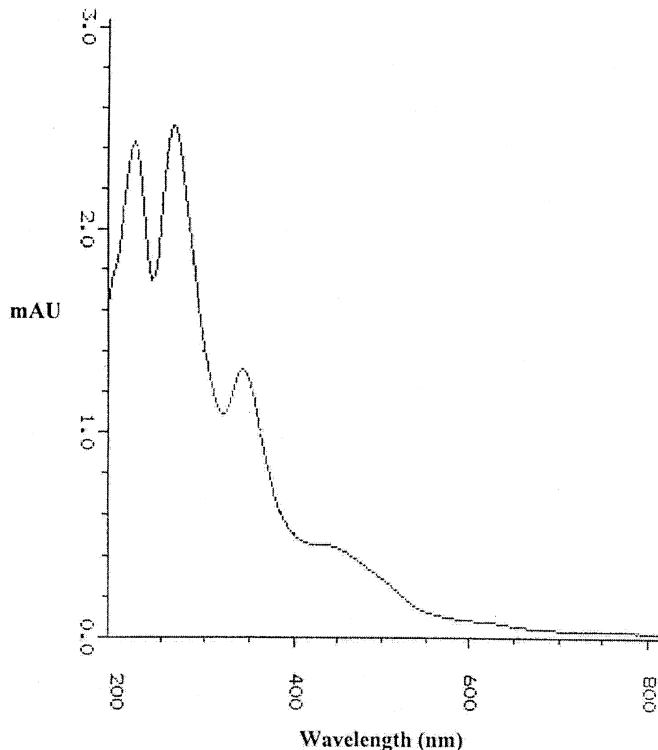

Fig. 3. UV–Vis spectra of C_{60} dissolved in 100 mM SDS in water. The four wavelength maxima are 220, 264, 342, and 443 nm.
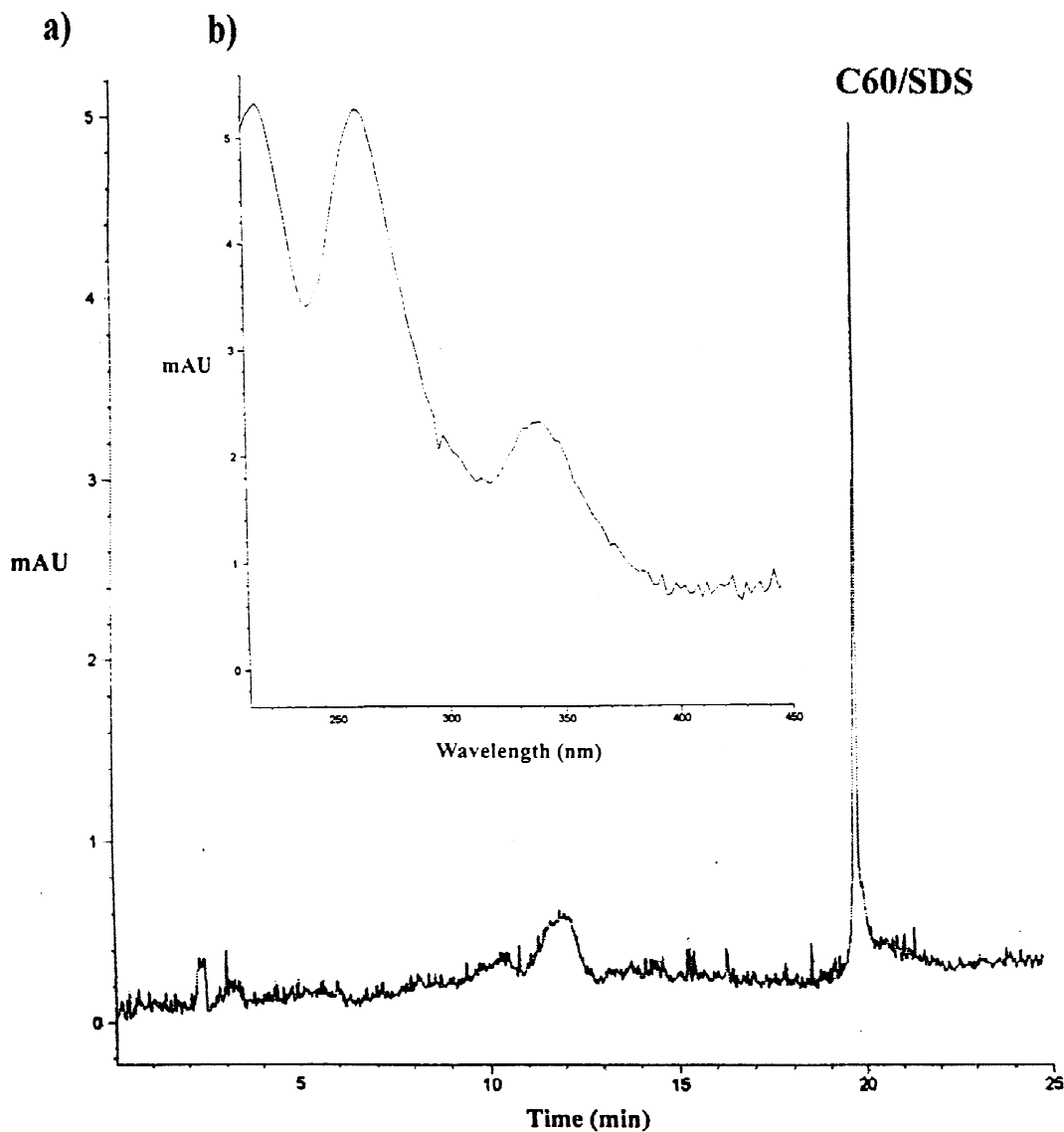
Fig. 4. (a) Electropherogram of C_{60}–SDS complex. Conditions were 100 mM SDS in 10 mM phosphate buffer at a pH 9.5 using an applied voltage of 10 kV and 254 nm detection. (b) On-line UV–Vis spectra of the C_{60}–SDS complex.

SDS micelles [16]. Also, the difference in the maxima for the on-line versus the off-line UV–Vis spectra are due to the buffer salts affecting the micellar aggregation number by a salting out effect [42]. The negative electrophoretic mobility and the UV–Vis spectra confirm that the SDS surfactant is associated with the C_{60} fullerene.

The electropherogram in Fig. 4a shows a broad, low intensity peak at the migration time of 12 min. This peak is due to the sample background and is

observed throughout the CE analysis of the SDS–fullerene samples.

3.2. C_{60}–C_{70}–SDS sample

A mixture of 70% C_{60} and 20% C_{70} was refluxed in 100 mM SDS using the same conditions for dissolving C_{60} in SDS. After heating, the solution changed to a dark red–brown color indicating the solubility of C_{70} or a mixture of C_{70} with C_{60} in the

SDS solution. An off-line UV–Vis spectra of the mixture was taken. In Fig. 5 the UV–Vis spectra for the fullerene mixture (C_{60}–C_{70}–SDS) is compared with the spectra of the C_{60}–SDS solution. The major spectral bands for the mixture were 225, 266, and 339 nm. Two shoulders appear within the C_{60}–C_{70}–SDS spectra. One shoulder appears at 387 nm and a very broad shoulder appears at 510 nm. C_{60}–SDS is blue shifted from the C_{60}–C_{70}–SDS mixture within the region of 210 nm, whereas in the 330-nm region the C_{60}–SDS is slightly red shifted from the C_{60}–C_{70}–SDS mixture. There are obvious spectral differences in the two fullerene solutions.

The C_{60}–C_{70}–SDS sample was then analyzed by CE. Fig. 6a shows the electropherogram of the C_{60}–C_{70}–SDS sample. The first major signal at the migration time of 10 min had an average electrophoretic mobility of -5.3×10^{-4} cm^2/V s (0.1% RSD). The sharp signal at the migration time of 17

min had an average electrophoretic mobility of -5.91×10^{-4} cm^2/V s (0.8% RSD).

The on-line UV–Vis spectra for the two SDS–fullerene complexes are shown in Fig. 6b. The spectra corresponding to the SDS–fullerene complex at a migration time of 10 min is distinctly different from the spectra shown for the SDS–fullerene complex at the migration time of 17 min.

A close study of the mobility and the on-line UV–Vis spectra at 17 min suggests that the SDS–fullerene complex closely resembles C_{60}–SDS. Although the electrophoretic mobilities are the same, the UV–Vis spectra are shifted in comparison to C_{60}–SDS alone (Fig. 3). In Fig. 6b, the wavelength maxima were 214.5, 258.5, and 334.3 nm. All three major bands in Fig. 6b were blue shifted from the C_{60}–SDS sample analyzed on-line. The shifts may be explained by a decrease in the aggregation of C_{60} fullerenes encapsulated by SDS in the mixture.
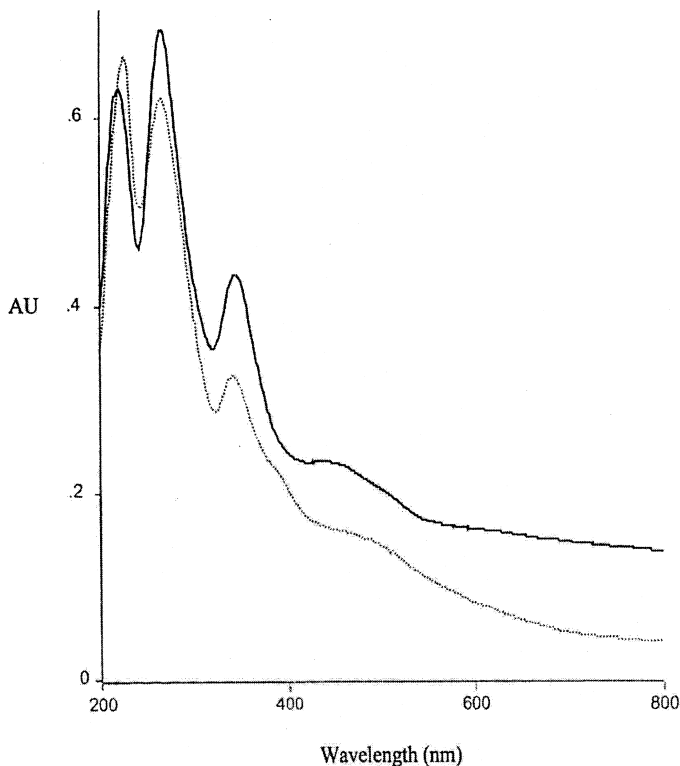

Fig. 5. Comparison of the UV–Vis spectra for the C_{60}–C_{70}–SDS solution ($\cdots$) versus the C_{60}–SDS solution (—).
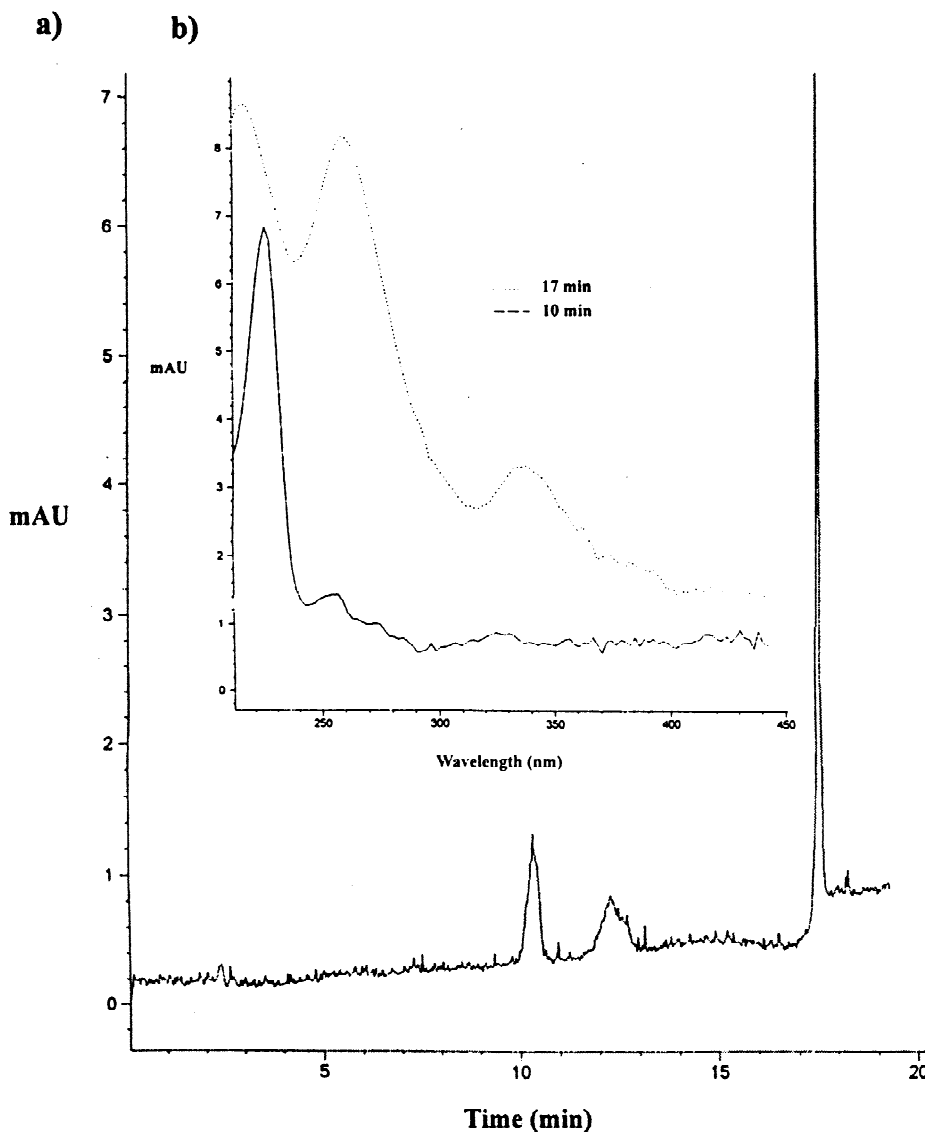
Fig. 6. (a) Electropherogram of C_{60}–C_{70}–SDS sample. Conditions the same as in Fig. 4. (b) UV–Vis spectra of the SDS–fullerene complexes at the migration times of 10 and 17 min.

The UV–Vis spectra for the 10-min migration time is also shown in Fig. 6b. The spectra has a pronounced maximum at 223.9 nm with weak spectral bands at 255.8, 271.0, 329.6, and 355.1 nm. Based on absorption spectra of C_{70} and C_{60} fullerenes in the literature, the UV–Vis spectra was not characteristic of any spectra reported. Therefore interpret-

ing the spectra and identifying the SDS–fullerene complex was difficult. The lower mobility compared to the C_{60}–SDS mobility suggests either the SDS complex is smaller in size, and/or fewer SDS molecules encapsulate the fullerenes. Also, the evidence of the reddish brown color and HPLC analysis (ca. 1:1 M C_{60}:C_{70}) of the C_{60}–C_{70}–SDS mixture,

suggests that the SDS complex could be an encapsulation of an aggregate of C_{70} with C_{60} in some proportion [43].

3.3. C_{70}–SDS sample

After reflux of the C_{70}–SDS solution the color of the solution became dark purple, but the fullerenes were suspended rather than dissolved. Over a period of 2 days the solution cleared up as the C_{70} precipitated out of the suspension. By UV–Vis analysis it was determined that the C_{70} fullerene was practically insoluble in the 100 mM SDS solution. Since higher

concentrations of SDS in the CE buffer produce joule heating, a concentration of 100 mM SDS was maintained [44]. As a result, an examination of the dependence of C_{70} solubility on concentration of SDS was not performed in this CE study. However, concentrations as high as 200 M SDS did show more solubility for the C_{70} fullerene. Similar results of the insolubility of C_{70} in micelle-like systems, such as complexes with p-*tert*. Bu-calix[8]arene, have been observed [43]. Our experiments have shown that C_{60} helps dissolve C_{70} in a solution of 100 mM SDS. A mixture of a 1:1 molar ratio of C_{70} to C_{60} evolved from an approximate 3.5:1 initial mass ratio prior to
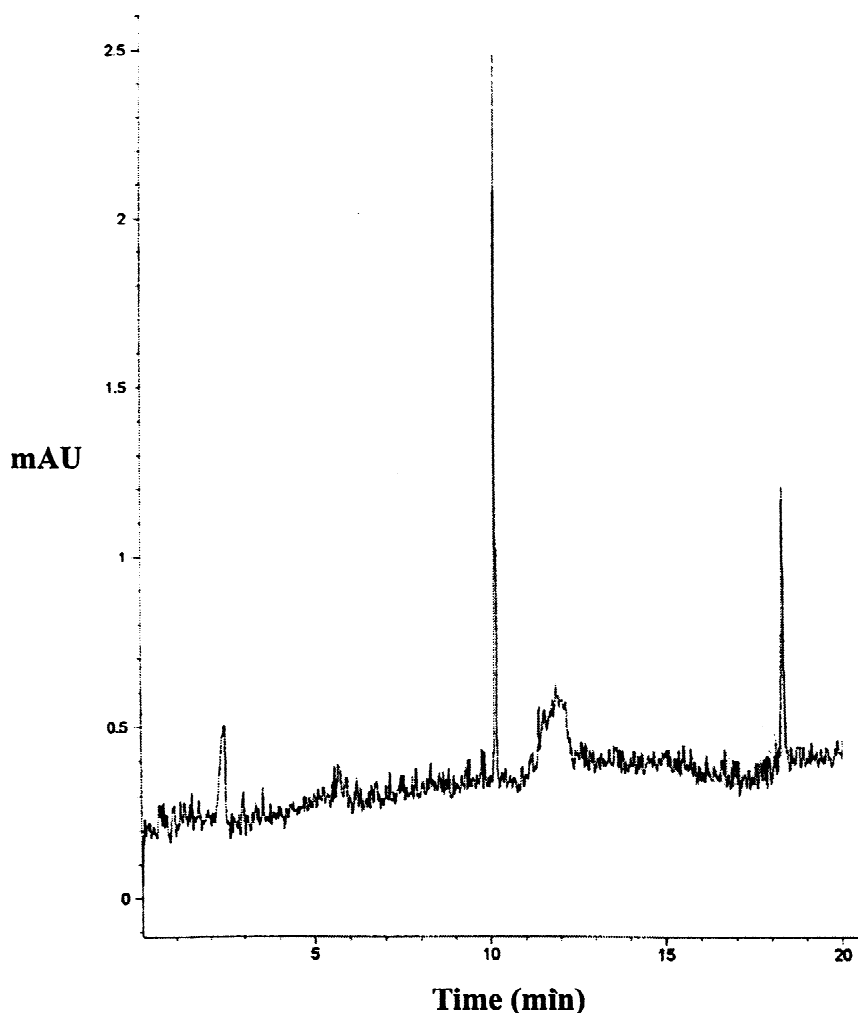

Fig. 7. Electropherogram of C_{70}–SDS sample. Conditions are the same as Fig. 4.

reflux. Further investigation of this phenomenon is necessary.

Despite the low solubility of C_{70} in 100 mM SDS the sample was analyzed by CE. The electrophero-gram is shown in Fig. 7. The broad peak from the sample background is observed at 12 min, along with migration times of 10.2 and 18.4 min which are similar to the signals seen with the C_{60}–C_{70}–SDS mixture. The SDS–fullerene complex at 18.4 min had an average electrophoretic mobility of $-5.85\times$

10^{-4} cm^2/V s with 0.3% RSD. The UV–Vis spectra at 18.4 min was the same as the spectra shown in Fig. 6b. However, the signal at 10 min was not reproducible. Although the C_{70} sample was 98.5% pure, based on the migration rate and UV–Vis spectra, the SDS extracted C_{60}. Further determi-nation of the fullerene(s) contained in the two SDS complexes observed in the CE separation is neces-sary.
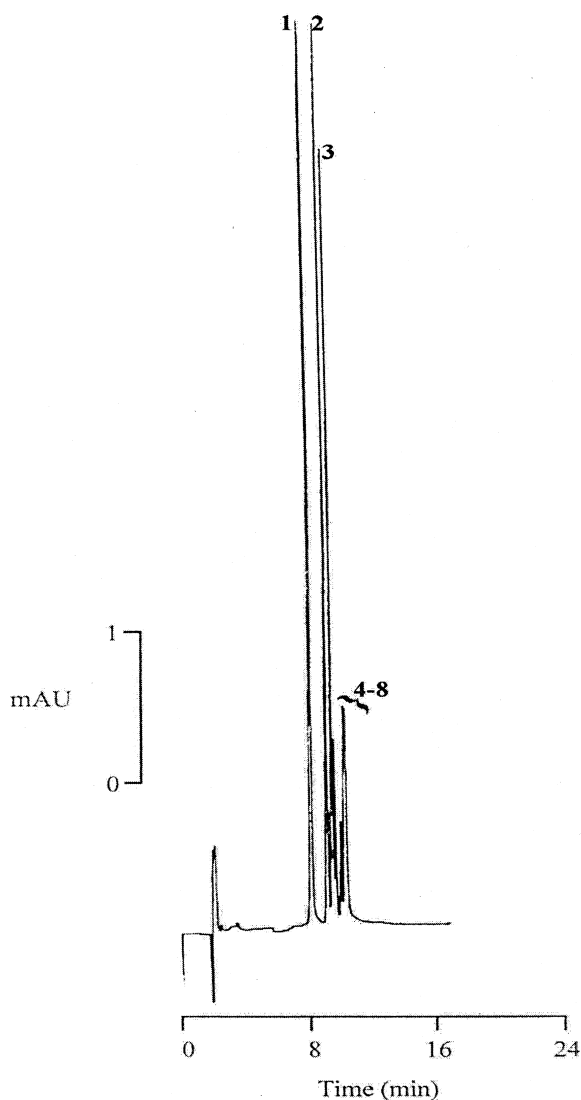

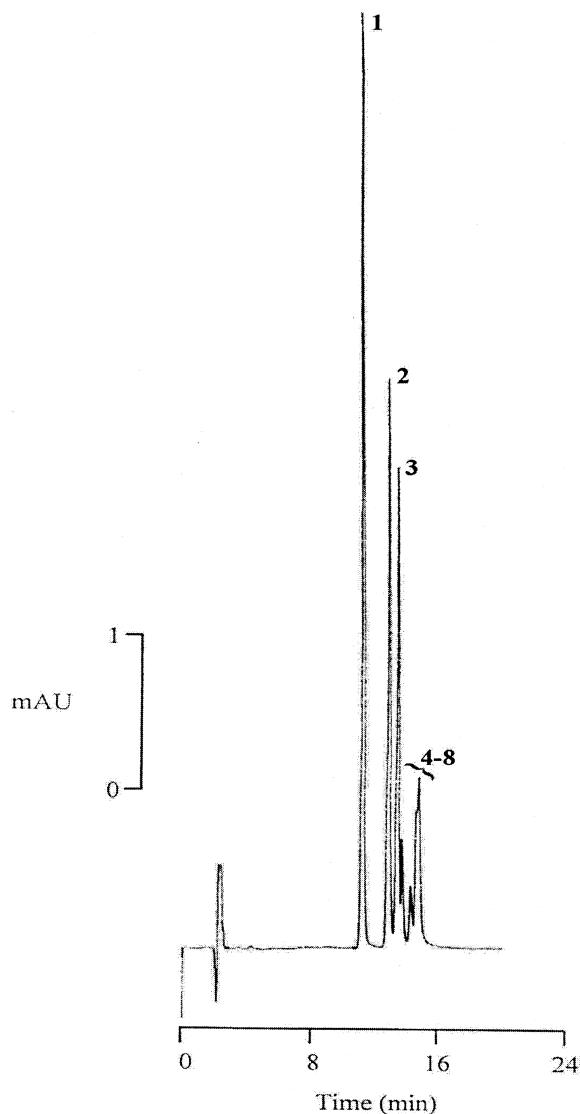Fig. 8. Separation of PAHs (1–8) using 100 mM SDS in 10 mM phosphate–borate buffer without C_{60}.

Fig. 9. Separation of PAHs (1–8) using the same sample and conditions as Fig. 8, but with C_{60}.

3.4. Use of C_{60}–SDS for separations

In preliminary studies the separation of PAHs were obtained to demonstrate the effective use of C_{60}–SDS buffer for separations. A comparison with conventional micellar electrokinetic chromatography (MEKC) using 100 mM SDS was conducted. Since the main purpose of this study was to exhibit the enhancement of the separation of PAHs no effort was made to identify the PAHs analyzed.

Fig. 8 shows a separation of a group of PAHs by CE using MEKC with SDS. The conditions were 100 mM SDS in a 10 mM borate–phosphate buffer at a pH of 9.5. The detection wavelength was 254 nm. Fig. 9 shows a separation of the same group of PAHs using the same conditions as Fig. 8, but with the addition of C_{60} in the buffer. The average electroosmotic mobility for both SDS and C_{60}–SDS buffer systems was 4.67×10^{-4} cm^2/V s (1.8% RSD). The separation of the first three peaks within the electropherograms of Figs. 8 and 9 were examined. The degree of the separation was measured by calculating the capacity factor, selectivity, and resolution. Table 1 shows these parameters for the first three PAHs separated by the SDS–MEKC buffer with and without C_{60}. One difference between the two conditions is the higher retention for the group of PAHs in C_{60}–SDS buffer. The higher retention was expected since the C_{60} fullerenes, which are encapsulated within the region of the micelles, enhance hydrophobic retention of the PAHs through π–π interactions. The capacity factors of the PAHs increase by 35% using the C_{60}–SDS buffer. Also, the resolution increases by a factor of 39 and 62% for

the first and second pair of solutes, respectively. These results show that C_{60}-MEKC can improve a conventional MEKC separation method.

4. Conclusion

In summary, this initial study has shown that CE can be used to analyze C_{60} and C_{70} fullerenes in an aqueous buffer via surfactant complexation. Based on the on-line UV–Vis diode array detection, it was possible to determine the migration of fullerene–SDS complexes. However, further verification of the fullerene complexes with the mixture of C_{60} and C_{70} in SDS is necessary. Also, more information of the aggregation and encapsulation process of C_{60} and C_{70} with SDS is required. The average number of fullerene aggregates per micelle and the various sizes in fullerenes that aggregate within the SDS micelle are questions that need to be answered. CE coupled to mass spectrometry and/or NMR are techniques that may answer some of these questions proposed.

The preliminary results of C_{60}-MEKC show promise in improving existing MEKC methods for compounds such as polyaromatic hydrocarbons, proteins, peptides, nucleotides, steroids, and vitamins. However, because there are fullerenes added to the micelle, factors known to affect MEKC separations such as pH, SDS concentration, organic modifiers, and type of surfactant used should be studied for C_{60}-MEKC. Other important factors would include C_{60} concentration and the use of other fullerenes and surfactants. One disadvantage to the use of C_{60}-MEKC may be the cost. However, in this preliminary study the maximum amount used for the 100 mM SDS buffer was approximately 10 μM. Also, lower production costs have brought the price per gram of fullerene to approximately US\$ 20 for C_{60}. Since MEKC is an important electrophoretic technique for pharmaceutical and environmental applications, further investigation of C_{60}-MEKC is worthwhile.

Table 1
Migration times and separation parameters for PAHs[a]

Peaks	Migration time (min)	k'	α	R_s
1[b]	8.65	2.47		
2[b]	9.84	2.95	1.18	50.6
3[b]	10.22	3.10	1.05	16.3
1[c]	11.08	3.34		
2[c]	12.64	3.96	1.19	45.5
3[c]	13.24	4.19	1.06	16.3

[a] Buffer: 100 mM SDS in 10 mM borate–10 mM phosphate, pH 9.5.
[b] Buffer: without C_{60}.
[c] Buffer: with C_{60}.

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