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# Improved resolution of inorganic anions in capillary electrophoresis by modification of the reversed electroosmotic flow and the anion mobility with mixed surfactants

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## Abstract

The standard method for analysis of inorganic anions by capillary electrophoresis involves adding tetradecyltrimethylammonium bromide (TTAB) to the buffer to reverse the electroosmotic flow (EOF). The resolution achieved using this procedure is greatly improved by adding the zwitterionic surfactant, coco amidopropylhydroxydimethylsulfobetaine (CAS U) to lower the magnitude of the reversed EOF and alter the anion mobilities. In a mixed surfactant system, varying the ratio of TTAB to CAS U allows monotonic alteration of the EOF from fully reversed (TTAB alone) to near zero (CAS U alone). The total surfactant concentration (if greater than the critical micelle concentration) and buffer pH have minimal effect on the EOF. In addition, the anion mobilities can be altered to a minor degree by varying the ratio of TTAB to CAS U, which contributes to the improved anion resolution. The effect on the EOF of other surfactant systems involving CAS U and other cationic or anionic surfactants is also studied. © 1998 Elsevier Science B.V.

**Keywords:** Electroosmotic flow; Buffer composition; Inorganic anions; Tetradecyltrimethylammonium bromide; Coco amidopropylhydroxydimethylsulfobetaine; Surfactants

## 1. Introduction

Capillary electrophoresis (CE) provides rapid separations of small organic and inorganic anions compared to conventional ion chromatography (IC) techniques [1]. The high efficiency of CE also yields much greater peak capacity than IC techniques. CE separations are based on the intrinsic electrophoretic mobility of the anions, i.e., how fast the ions move under an applied electric field.

Anions migrate in the opposite direction as the electroosmotic flow (EOF) in an untreated silica capillary. This complicates the analysis of small inorganic and organic anions since some of the

smaller anions migrate more rapidly than the EOF. This means that under normal EOF, some anion's net migration is towards the anode, while others are swept to the cathode by the EOF. One solution to this problem is to reverse the direction of the EOF using buffer additives such as cationic surfactants or polyamines. Under reversed EOF conditions, anions co-migrate with the EOF. This results in rapid separations. However in some cases, the reversed EOF sweeps the anions through the capillary too quickly to allow sufficient time to fully develop separation.

The resolution ( $R$ ) of two components in an electrophoretic separation is described by [2]:

$$R = \frac{\sqrt{N}}{4} \left( \frac{\Delta\mu_{\text{app}}}{\bar{\mu}_{\text{app}}} \right) \quad (1)$$

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where  $N$  is the efficiency of the separation,  $\Delta\mu_{\text{app}}$  is the difference in the apparent mobilities of the two components, and  $\bar{\mu}_{\text{app}}$  is the average apparent mobility. Note that  $N$  is also a function of the apparent mobility,  $\mu_{\text{app}}$ . The apparent mobility ( $\mu_{\text{app}}$ ) is the sum of the analyte's intrinsic electrophoretic mobility ( $\mu_{\text{ep}}$ ) and the electroosmotic mobility ( $\mu_{\text{EOF}}$ ):

$$\mu_{\text{app}} = \mu_{\text{ep}} + \mu_{\text{EOF}} \quad (2)$$

Eqs. (1) and (2) predict that the resolution of a separation can also be improved by altering  $\Delta\mu_{\text{app}}$  and/or  $\mu_{\text{EOF}}$ . This is the approach that will be explored in this work.

The most common procedure for small inorganic ion analysis in CE uses the cationic surfactant tetradecyltrimethylammonium bromide (TTAB) [3] to reverse the EOF. Alternatively other cationic surfactants [4–9], alkylamines [10], diamines [11] or polyamines [12] and references therein) have been used. Two phenomena occur upon the addition of cationic surfactants. First, cationic surfactants adsorb onto the bare silica walls of the capillary to form a hemimicelle dynamic layer. This makes the wall cationic, thus causing the direction of the EOF to reverse [13]. The magnitude of the reversed EOF is effectively independent of the surfactant concentration and the buffer pH [13]. Second, cationic surfactants exist as micelles in solution and provide a media for ion-exchange with some anionic analytes. As a consequence, the electrophoretic mobility of these anions may be altered by the concentration of EOF modifier [1,14]. The goal of this work is to improve the existing CE procedure for anion analysis by enabling alteration of the magnitude of the reversed EOF and/or alteration of the anion mobilities, which will in turn improve the resolution of the separation.

## 2. Experimental

### 2.1. Apparatus

All experiments were performed using a P/ACE 2100 equipped with UV detection (Beckman Instruments, Fullerton, CA, USA). Untreated silica capil-

laries (Polymicro Technologies, Phoenix, AZ, USA) with an inner diameter of 75  $\mu\text{m}$  and an outer diameter of 365  $\mu\text{m}$  were used. The total capillary length was 47 cm (40 cm to the detector) for the  $\mu_{\text{EOF}}$  measurement, and was 57 cm (50 cm to the detector) for the anion analysis. Data acquisition using System Gold software (Beckman, version 8.10) was performed on a 386-based microcomputer. For the  $\mu_{\text{EOF}}$  measurements, a data collection rate of 5 Hz and detector rise time of 0.5 s were used, whereas 20 Hz data collection and 0.1 s rise time were used for the anion analyses.

### 2.2. Reagents

All solutions were prepared in Nanopure ultrapure water (Barnstead). Buffers were prepared from orthophosphoric acid (BDH) with sodium hydroxide (BDH) to adjust the pH, or potassium chromate (BDH) with phosphoric acid to adjust the pH. Tetradecyltrimethylammonium bromide (TTAB, Aldrich), cetyltrimethylammonium bromide (CTAB, Aldrich), sodium dodecyl sulfate (SDS, Aldrich) and Rewoteric AM CAS U (Witco) were used as received. The structure of Rewoteric AM CAS U is given in Fig. 1. The "R-" group represents an alkyl chain with variable chain length from  $\text{C}_8$  to  $\text{C}_{18}$ . The molecular mass of CAS U was estimated to be 450 g/M based on the result of a gas chromatographic analysis of the acid hydrolysis products of CAS U [15]. Samples of 5 ppm anion solutions were prepared from potassium bromide (BDH), potassium chloride (BDH), potassium sulfate (Fisher Scientific), sodium nitrite (Fisher Scientific), potassium nitrate (Fisher Scientific) and potassium oxalate (Matheson, Cincinnati, OH, USA) without further purification. A 5 mM mesityl oxide (Aldrich) solution in water was used as the EOF marker.

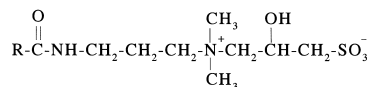


Fig. 1. Chemical structure of the Rewoteric AM CAS U: coco amidopropylhydroxydimethylsulfobetaine (CAS U).  $\text{R}=\text{C}_8$  to  $\text{C}_{18}$ .

### 2.3. Procedures

Each new capillary was pretreated with 0.1 M NaOH for 10 min. Before each run, the capillary was rinsed at high pressure (20 p.s.i.) with 0.1 M NaOH for 2 min, distilled water for 1 min and buffer for 2 min (1 p.s.i. = 6894.76 Pa). In all experiments the capillary was thermostatted to 25°C.

#### 2.3.1. Electroosmotic mobility measurement

Mesityl oxide was introduced as an EOF marker using low pressure hydrodynamic injection for 1 s. Constant voltage (−15 kV) was applied to induce the EOF, and direct UV detection at 254 nm was used. Measurements of  $\mu_{\text{EOF}}$  were performed in two ways as described previously [16]. When the magnitude of the EOF was high ( $>2.0 \cdot 10^{-4} \text{ cm}^2/\text{V s}$ ), it was determined by the migration time of mesityl oxide under constant voltage. When the electroosmotic mobility was low ( $<2.0 \cdot 10^{-4} \text{ cm}^2/\text{V s}$ ), the direct approach would take too long. As a quicker alternative, the sequential injection method of Williams and Vigh [17] was used. The ionic strength of all electrophoretic buffers used in this  $\mu_{\text{EOF}}$  study was adjusted to 50 mM by varying the concentration of phosphate.

#### 2.3.2. Anion analysis

Anions were separated using 5 mM potassium chromate at pH 8.0 and monitored using indirect detection at 254 nm [3,18]. TTAB and CAS U were used to modify the EOF. Solutions of 5 ppm anion mixture (bromide, chloride, sulfate, nitrite, nitrate and oxalate) were introduced onto the capillary using low pressure (0.5 p.s.i.) hydrodynamic injection for 10.0 s (ca. 50 nl). Constant voltage (−20 kV) was applied.

## 3. Results and discussion

### 3.1. EOF alteration by mixed surfactant systems

The standard procedure for anion analysis in CE utilizes TTAB to reverse the EOF [3,18]. TTAB, like other cationic surfactants, forms a hemimicelle layer on the walls of the capillary. This hemimicelle layer is a micellar phenomenon, and so is dynamic in

nature and only occurs at surfactant concentrations above the critical micelle concentration (CMC). The charge density of the hemimicelle layer, and thus of the capillary wall, is a function of the electrostatic repulsion between the head-groups of the surfactant monomers. Previous studies have demonstrated that factors such as buffer pH and concentration have little effect on the resultant EOF [13].

To alter the EOF resulting from the hemimicelle layer, electrostatic properties of the layer must be altered. One way to achieve this is to add another surfactant possessing a different charge, i.e., create a mixed surfactant system. This has been successfully demonstrated by Hult et al. [19] for protein separations using mixtures of cationic and anionic fluoro-surfactants. In our work, we explore the use of mixed surfactants to modify the standard CE anion procedure involving TTAB [3,18].

The first approach attempted was to mix a cationic hydrocarbon surfactant, such as TTAB or CTAB, with an anionic hydrocarbon surfactant, SDS. Precipitation occurred when these surfactants were mixed. A second approach was to mix a cationic surfactant, CTAB, with a nonionic surfactant, such as hexanol and octanol. No significant change in EOF was observed when 0.5 mM hexanol or octanol was added to 0.5 mM CTAB. It is believed that the nonionic surfactant partitions into the micelle, without changing the electrostatic properties of the hemimicelle.

Our final, and successful, approach to modifying the reversed EOF was to add a zwitterionic surfactant to the cationic surfactant. Our previous studies showed that the zwitterionic surfactant CAS U (Fig. 1) itself adsorbs onto the capillary wall to form a hemimicelle. The resultant EOF remains in the normal-forward direction, but is suppressed to 10% of its original value over a wide range of pH [16]. Based on the manufacturer's literature, CAS U is compatible with cationic, anionic and nonionic surfactants [20]. Below we explore the use of mixed TTAB–CAS U surfactant systems to modify the reversed EOF.

### 3.2. Effect of TTAB and CAS U on EOF

Fig. 2 shows the effect of varying the TTAB–CAS U surfactant ratio on the EOF under constant

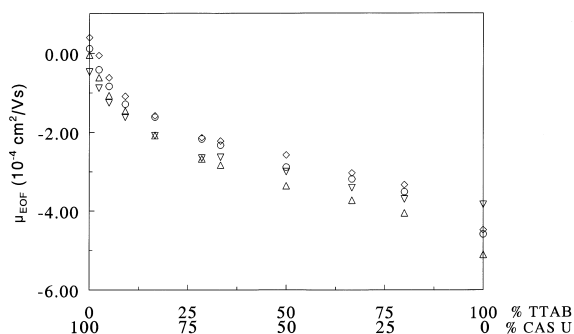


Fig. 2. Effect of TTAB–CAS U on the EOF at:  $\nabla$ , pH 3.0;  $\triangle$ , pH 6.0;  $\circ$ , pH 8.0 and  $\diamond$ , pH 11.0. Experimental conditions: applied voltage,  $-15$  kV; capillary length, 47 cm; length to detector 40 cm; detection, 254 nm; buffer, sodium phosphate at a constant ionic strength of 50 mM; total surfactant concentration, 2.0–4.0 mM.

pH and ionic strength conditions. A positive  $\mu_{\text{EOF}}$  represents a normal (forward) EOF while a negative  $\mu_{\text{EOF}}$  represents a reversed EOF. The composition of the surfactant mixture is expressed as % TTAB and % CAS U. As the EOF is dependent on the surfactant ratio, and not the surfactant concentration (Section 3.2.2), it was most convenient to collect the data for Fig. 2 in the following manner. First, data for % TTAB < 50% were determined by varying the TTAB concentration from 0 to 2 mM with a constant CAS U concentration of 2 mM. Then, data for % TTAB > 50% were determined by varying the CAS U concentration from 0 to 2 mM with a constant TTAB concentration of 2 mM.

As the % TTAB increases in Fig. 2, the EOF changes gradually from near-zero to fully reversed. The gradual transition in the EOF suggests that the composition of the adsorbed hemimicelle layer is proportional to the ratio of the zwitterionic and cationic surfactants in solution. When two compatible surfactants are present at concentrations above their CMC, the surfactant mixture will co-micellize. In the TTAB–CAS U system, the mixed-micelle is positively charged overall. Thus, the hemimicelle formed at the capillary wall remains cationic, and a reversed EOF is observed. However each zwitterionic surfactant incorporated into the hemimicelle displaces a cationic surfactant, thus reducing the net charge at the hemimicelle surface.

### 3.2.1. Effect of pH on EOF modification

Fig. 2 contains data obtained over a range of

buffer pH. The effect of pH was relatively minor compared to that caused by the surfactant ratio. At a given surfactant ratio, the EOF decreased in magnitude with pH over the range of 6 to 11. This trend results from the EOF dependence on the buffer counter-ion [13,21] caused by ion-exchange of the counter-ion with the wall charge. As the pH increases, phosphate shifts from predominately  $\text{H}_2\text{PO}_4^-$  to  $\text{HPO}_4^{2-}$ .  $\text{HPO}_4^{2-}$  ion-exchanges with the positive wall charge more strongly than  $\text{H}_2\text{PO}_4^-$ . As a result buffers containing more  $\text{HPO}_4^{2-}$  (i.e., higher pH) display slower EOF.

The EOF observed for pH 3 (Fig. 2) does not follow the trend described above. At pH 3 the wall charge on the capillary is very low. This low wall charge results in incomplete hemimicelle formation [13], which in turn minimizes the effect of the mixed surfactant on the EOF.

### 3.2.2. The effect of surfactant concentration on the EOF

The effect of surfactant concentration on the EOF is shown in Fig. 3. The concentrations of each of TTAB and CAS U was varied from 0.01 to 5.0 mM with the surfactant ratio held at a constant 1:1. The  $\mu_{\text{EOF}}$  measured (ca.  $3 \cdot 10^{-4}$   $\text{cm}^2/\text{V s}$ ) were essentially constant for surfactant concentrations greater than 1.0 mM. Below 1.0 mM the  $\mu_{\text{EOF}}$  decreases toward near-zero. The behavior in Fig. 3 results from the EOF modification being due to the formation of a hemimicelle at the capillary wall. Modification of  $\mu_{\text{EOF}}$  only occurs at surfactant concentrations above

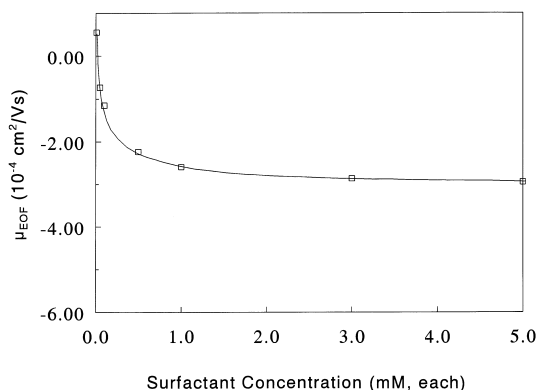


Fig. 3. Effect of surfactant concentration on the EOF. Experimental conditions as in Fig. 2 except: pH, 8.0; and TTAB–CAS U ratio, 1:1.

the CMC [13]. The CMC of TTAB is ca. 1 mM at ionic strength of 50 mM [22] (interpolated from the CMC of tetradecyltrimethylammonium chloride, 1.36 mM; TTAB, 1.3 mM; and tetradecyltrimethylammonium sulfate, 0.89 mM). Hence one would expect the effect of TTAB in a mixed surfactant system to diminish at concentrations below 1.0 mM. The CMC of CAS U is however much lower ( $<0.01$  mM), thus its effect sustains at concentrations below 1.0 mM. As a consequence, the EOF obtained with TTAB–CAS U at a concentration below 1.0 mM would approach that obtained with CAS U alone (near-zero). When the surfactant concentration exceeds the CMC, the  $\mu_{\text{EOF}}$  depends on the surfactant ratio and does not vary with the actual surfactant concentration.

### 3.3. Anion analysis

The mixed TTAB–CAS U surfactant system was applied to the standard anion analysis procedure to improve its resolution. The separation of six anions: bromide, chloride, sulfate, nitrite, nitrate and oxalate was performed in a 5.0 mM chromate buffer at pH 8.0 with indirect UV detection at 254 nm. Fig. 4 shows electropherograms performed at three TTAB–CAS U compositions. In Fig. 4A, the separation is

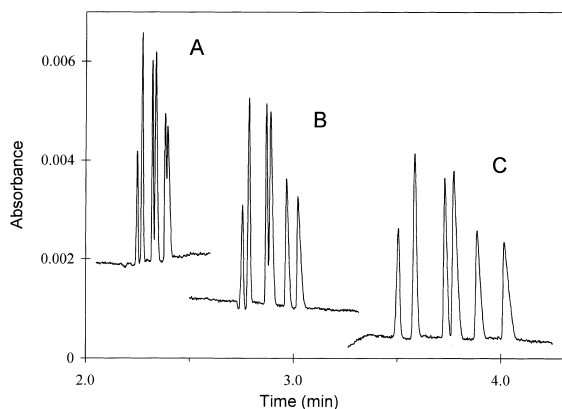


Fig. 4. Separation of six inorganic ions with various amounts of TTAB–CAS U as additives: (A) 0.5 mM TTAB; (B) 0.5 mM TTAB and 0.5 mM CAS U and (C) 0.5 mM CAS U. Experimental conditions: applied voltage  $-20$  kV; capillary length, 57 cm; length to detector, 50 cm; detection, indirect at 254 nm; buffer, 5.0 mM potassium chromate with pH adjusted to 8.0 using phosphoric acid; sample, 5 ppm mixed anion solution. Anion migration order: bromide, chloride, sulfate, nitrite, nitrate and oxalate.

performed under standard conditions that use only TTAB (0.5 mM) as the EOF modifier [3,18]. Baseline resolution is achieved between bromide and chloride, near baseline resolution between sulfate and nitrite, but only partial separation between nitrate and oxalate. In Fig. 4B, 0.5 mM of CAS U is added to the 0.5 mM TTAB giving a 1:1 TTAB–CAS U buffer. This reduces the EOF to about 2/3 of that in TTAB alone. The separation between nitrate and oxalate is now improved to baseline resolution (Fig. 4B). Finally if only CAS U (0.5 mM) is used as the additive (Fig. 4C), the  $\mu_{\text{EOF}}$  is reduced effectively to zero. Baseline resolution is observed between all components.

#### 3.3.1. Alteration of anion mobility by the mixed surfactants

In order to investigate whether the addition of CAS U to the TTAB buffer has any effect on the intrinsic mobilities of the anions, the intrinsic mobilities normalized to chloride were determined at various ratios of TTAB–CAS U (Fig. 5). As the % CAS U increased and the % TTAB decreased, the differences in mobilities among the six analytes were maximized. Although the change in mobility is small and does not involve changes in migration order, it contributes significantly to the resolution improvement. Hence the improved anion resolution is a result of both the reduction in  $\mu_{\text{EOF}}$  and the change in anion mobilities.

Given that the TTAB–CAS U ratio in the buffer affects the mobilities of the anionic analytes, similar effect may occur to the indirect detection anion–chromate. Band broadening of the anions due to

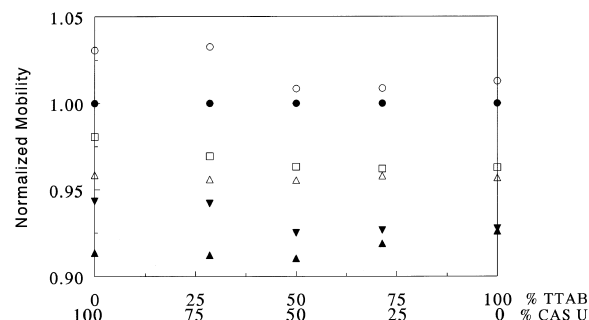


Fig. 5. The intrinsic mobility normalized to chloride at various TTAB–CAS U ratios: bromide, ○; chloride, ●; sulfate, □; nitrite, △; nitrate, ▼ and oxalate, ▲. Experimental conditions as in Fig. 2.

electrodispersion is minimized when the mobility of an anion matches with that of chromate. A change in chromate mobility thus would lead to alteration in peak shape of the anions. To determine whether the chromate mobility varies with the TTAB–CAS U ratio, we monitored the peak shape of the six anions in terms of asymmetry factor under various TTAB–CAS U ratios. The anionic analyte concentration was increased to 50 ppm to aggravate the effect of electrodispersion. The results showed no variation in peak shape as the buffer modifier was varied from 0.5 mM TTAB to 0.5 mM CAS U, which suggested that the chromate mobility is not significantly affected by the TTAB–CAS U ratio in our studies.

### 3.4. Mixed surfactant systems involving CAS U and other surfactants

As demonstrated above, the magnitude of the EOF generated by TTAB can be altered by addition of CAS U. Herein the effect of adding CAS U to other commonly used surfactants, such as CTAB and SDS is explored. Given that CAS U is compatible with other cationic and anionic surfactants CAS U should co-micellize with other surfactants, in a similar way as with TTAB [23]. A hemimicelle layer should form at the capillary wall resulting in EOF modification.

#### 3.4.1. Effect of CTAB and CAS U on the EOF

Fig. 6 shows the measured  $\mu_{\text{EOF}}$  at pH 7.2 using a CTAB–CAS U surfactant system. The  $\mu_{\text{EOF}}$  measurement is performed as described for TTAB–CAS

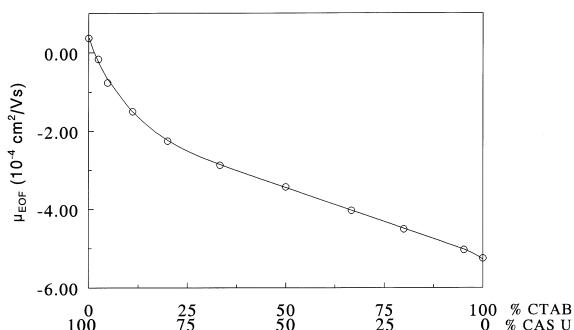


Fig. 6. Effect of CTAB–CAS U on the EOF. Experimental conditions as in Fig. 2 except: buffer, 10 mM phosphate (pH 7.2) with ionic strength adjusted to 50 mM with NaCl.

U. The general behavior observed in Fig. 6 is identical to the TTAB–CAS U system (Fig. 2). As the % CTAB increases, the magnitude of the reversed EOF gradually increases from near-zero to that of 100% CTAB. The effect of pH on the CTAB–CAS U was consistent with that shown in Fig. 2. Hence reversed EOF can be modified using CTAB–CAS U in an analogous fashion to the TTAB–CAS U surfactant system.

#### 3.4.2. Effect of SDS and CAS U on the EOF

SDS is an anionic surfactant that is often used in micellar electrokinetic chromatography (MEKC) [24]. Addition of SDS alone at concentrations above the CMC does not change the EOF beyond that due to viscosity alone. To investigate the effect of SDS–CAS U mixtures on the EOF, the  $\mu_{\text{EOF}}$  was measured at SDS concentrations from 0 to 2.0 mM with a constant 2.0 mM CAS U. As can be seen in Fig. 7, the  $\mu_{\text{EOF}}$  increases from near-zero to ca.  $4 \cdot 10^{-4} \text{ cm}^2/\text{V s}$  by 20% SDS (0.5 mM). At higher SDS concentrations the  $\mu_{\text{EOF}}$  remains constant at ca.  $4 \cdot 10^{-4} \text{ cm}^2/\text{V s}$ , which is approximately the EOF observed in the absence of surfactants.

The above observations can be explained in the following manner. Initially at 0% SDS (100% CAS U), a zwitterionic hemimicelle layer forms. As a result, the  $\mu_{\text{EOF}}$  is suppressed to near-zero. As SDS is added to the buffer it incorporates into the hemimicelle, making the hemimicelle surface anionic. Hence a cathodic EOF is generated. The greater the SDS concentration in the buffer, the more

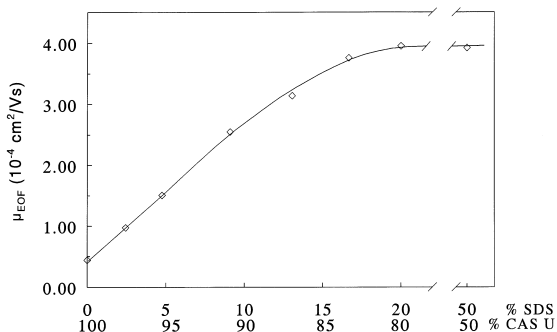


Fig. 7. Effect of SDS–CAS U on the EOF. Experimental conditions as in Fig. 2 except: applied voltage, +15 kV; buffer, 10 mM phosphate (pH 7.2) with ionic strength adjusted to 50 mM with NaCl; total surfactant concentration, 2–4 mM.

SDS incorporates into the hemimicelle and thus the greater the cathodic EOF. Nevertheless in Fig. 7 the magnitude of the EOF plateaued at % SDS greater than 20%. Also, at buffer pH from 3 to 7 (data not shown) the observed EOF for 10% SDS (90% CAS U) remained constant at ca.  $2.5 \cdot 10^{-4}$  cm<sup>2</sup>/V s. However as the pH was increased beyond pH 7, the cathodic EOF increased. It is believed that at high SDS concentrations or high buffer pH the hemimicelle is disrupted by electrostatic repulsion between the capillary wall and the negatively charged hemimicelle.

#### 4. Conclusions

In analysis of small inorganic anions the ability to modify the electrophoretic mobility of the analytes is limited. In such a case, improvements in resolution are best achieved by altering the EOF. Previous studies [13] had demonstrated that the reversed EOF generated by the cationic surfactant TTAB could not be significantly altered by buffer conditions. This work demonstrates that the reversed EOF generated by TTAB can be altered by the addition of the zwitterionic surfactant CAS U. The magnitude of the EOF depends mainly on the ratio of the two surfactants, and is independent of pH. Variation in the TTAB–CAS U ratio of the buffers improves in the resolution of inorganic anions by modifying the electroosmotic mobility and the anion intrinsic mobilities.

Similar EOF modification was also demonstrated for other surfactant systems. However, the effect of the anionic–zwitterionic (SDS–CAS U) surfactant mixtures on the EOF is not stable at high SDS concentrations or high pH.

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