# Spectrophotometric Study of the Effect of Univalent Electrolytes on Critical Micelle Concentrations of [1-(Ethoxycarbonyl)pentadecyl]trimethylammonium, 1-Hexadecylpyridinium, and Dimethylbenzyldodecylammonium Bromides

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Critical micelle concentrations of three antimicrobial cationic surfactants, *i.e.* quaternary ammonium salts [1-(ethoxycarbonyl)pentadecyl]trimethylammonium bromide, 1-hexadecylpyridinium bromide, and dimethylbenzyldodecylammonium bromide were determined by UV spectrophotometry in aqueous solutions. In the case of the first two compounds, the effect of addition of strong electrolytes KF, KCI, KBr, KI, KSCN, and LiCI, respectively, on the decrease of CMC was also followed. The results were processed in the form of a logarithmic dependence of CMC on the total concentration and the effect of both anions and cations of the added electrolyte was discussed. The UV spectrophotometry appeared to be a useful tool for the determination of CMC's of the order 10<sup>-3</sup> mol dm<sup>-3</sup> or smaller, if the micellizing surfactant cations or at least the corresponding counterions (in this work Br<sup>-</sup> and I<sup>-</sup>) exhibited convenient spectral parameters.

Aqueous solutions of ionogenic surfactants are usually sensitive to the addition of another electrolyte, presumably because of the interaction of the counterions with the charged micelles of the amphiphilic ions of the surfactant [1]. Thus the size and shape of the micelles of the cationic surfactants are influenced by the nature of anions of an inorganic salt added to the solution [2-6]. It has been recognized that the often observed decrease of the critical micelle concentration (CMC) of the ionogenic surfactant on the admixture of another electrolyte indicates enhanced micellization due to the binding of the counterions to the ionic micelles [7, 8]. Differences in counterion binding seem to be also responsible for the mutual differences in the CMC values of the cationic surfactant salts with the same amphiphilic cation but various inorganic anions [9].

Many ionogenic surfactants are characterized by a pronounced decrease of the concentration of the free surfactant ions in their micellar solutions at overall concentrations above CMC [1]. As an important consequence, CMC then approximately represents the maximum attainable concentration of the corresponding surfactant ion in its free monomeric (unmicellized) form. In our previous works we also found this type of behaviour of the antimicrobially active cationic surfactants [1-(ethoxycarbonyl)pentadecyl]trimethylammonium bromide (SEBr) [10, 11],

1-hexadecylpyridinium bromide (CPBr) [11], and dimethylbenzyldodecylammonium bromide (AJBr) [12] and it initiated our systematic study of the effect of the addition of various electrolytes on the decrease of CMC of the mentioned quaternary surfactant salts in aqueous solutions. We have already followed CMC of the solutions of SEBr and CPBr with the admixtures of potassium salts with a number of various univalent anions [11] by conductometry and potentiometry. We used the same methods for the determination of CMC of AJBr at the respective presence of several salts with various anions and cations [13] and pointed out an unexpected difference of the effect of the univalent cations Li<sup>+</sup>, Na<sup>+</sup>, and K<sup>+</sup> on CMC of the cationic surfactant.

However, the mentioned electrochemical methods of the CMC determination become less advantageous at a greater excess of the added electrolyte and, besides, a comparison of the CMC's measured by various methods is generally recommended. Thus the aim of this report was to work out the UV spectrophotometric determination of CMC's of SEBr, CPBr, and AJBr in aqueous solutions, and with SEBr and CPBr we also succeeded to determine the dependence of CMC on the admixture of some univalent strong electrolytes. With respect to our previous results [13], we added salts with various anions and cations.

Other authors also mentioned spectrophotometric determination of CMC of some surfactants [14], in-

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cluding CPBr [15], and SEBr was used in spectrophotometric studies as a cationic surfactant influencing the complexation equilibria of metal chelates [16]. SEBr, CPBr and its analogous quaternary alkylpyridinium salts notoriously affect the dissociation and optical properties of dves and colour indicators [16-19]. Some authors presented spectrophotometric evidences of a specific chargetransfer interaction of the micelles of alkylpyridinium cations (1-dodecylpyridinium [20], 1-methyl-4-dodecylpyridinium [21]) with iodide counterions. Micelles of CPBr also accelerate the oxidative reaction of Ce(IV) [22]. The shift of the acid-base and complexation equilibria of dyes and colour complexes by the surfactants is in general affected by the eventual presence of a neutral electrolyte [16-18], which may be regarded as a further motivation of our spectrophotometric study of the dependence of CMC on the added electrolyte.

### **EXPERIMENTAL**

[1-(Ethoxycarbonyl)pentadecyl]trimethylammonium bromide (synon, carbethopendecinium bromide, Septonex, SEBr) and dimethylbenzyldodecylammonium bromide (synon, benzododecinium bromide, Ajatin, AJBr), purity after PhBS 4, were products of Slovakofarma, The Slovak Republic. 1-Hexadecylpyridinium bromide monohydrate (synon, 1-cetylpyridinium bromide, CPBr), purum, was a product of Lachema, The Czech Republic. AJBr was used from the original aqueous solution Ajatin 10 % and standardized by a potentiometric titration as described previously [12, 13]. Other chemicals, KF, KCl, KBr, KI, KSCN, and LiCl, were of reagent grade purity. They were used without purification but dried properly before the solutions were prepared in redistilled water.

Absorbances of the solutions were measured with UV VIS spectrophotometer Hewlett—Packard 8452A (diode array) in the 0.5 cm and 1 cm quartz cells at  $ca.\ 25\ ^{\circ}\text{C}$ . The resulting CMC's were evaluated from the spectrophotometric measurements in the range of wavelengths  $\lambda$  = 200—260 nm.

### **Determination of CMC**

CMC's of the studied surfactants without the admixture of another electrolyte were evaluated from the absorbances of the series of usually 12 aqueous solutions, measured in the following concentration ranges ( $c/(\text{mol dm}^{-3})$ ): (SEBr) 1—12 × 10<sup>-4</sup>, (CPBr) 1—10 × 10<sup>-4</sup>, and (AJBr) 1—10 × 10<sup>-3</sup>. At first, CMC was always evaluated as a break on the curve  $A = f(c_{SBr})$ , where A is the absorbance at an

appropriately selected constant wavelength and  $c_{\rm SBr}$  is the concentration of the corresponding surfactant. If a concentrational shift of the wavelength  $\lambda_{\rm max}$  of the absorption maximum was observed, the second style of the CMC evaluation was from the break on the curves of the dependences  $\lambda_{\rm max} = f(c_{\rm SBr})$ , or  $\Delta\lambda_{\rm max} = f(c_{\rm SBr})$ , where  $\Delta\lambda_{\rm max} = \lambda_{\rm max,k} - \lambda_{\rm max,i}$  is the shift of the absorption band maximum,  $\lambda_{\rm max,k}$  and  $\lambda_{\rm max,i}$  are the wavelengths of the absorption band maximum, measured at the highest and lower surfactant concentrations, respectively.

Determination of CMC of the surfactants SEBr and CPBr in the presence of a strong electrolyte MeX (KF, KCl, etc.) was done by measuring absorbances of several series of aqueous solutions of the surfactant with added MeX. Concentrations of the both solutes were variable but all the solutions in one series had a constant total concentration  $c_t$ 

$$c_{t} = c_{SBr} + c_{MeX} \tag{1}$$

where  $c_{\rm SBr}$  is the surfactant concentration (SEBr or CPBr) and  $c_{\rm MeX}$  is the concentration of MeX.

Each series again consisted usually of 12 solutions and when precipitation did not occur, 5 series were measured with each MeX, up to  $c_t = 0.007$  mol dm<sup>-3</sup> or 0.008 mol dm<sup>-3</sup>. With respect to the decreasing CMC, the range of  $c_{\rm SBr}$  was set up somewhat lower than that without MeX. CMC at the given  $c_t$  was usually evaluated from the absorbance dependence  $A = f(c_{\rm SBr})$  in the corresponding series. In the solutions of the two solutes, the wavelength shift of the maximum was less convenient for the evaluation.

# **RESULTS AND DISCUSSION**

The measured absorption spectra of the aqueous solutions of the surfactants SEBr and CPBr are in Figs. 1 and 2. They enabled us to evaluate CMC both from the concentration dependences of the absorbance at a constant wavelength and from the wavelength shift of an absorption maximum. Spectrum of SEBr exhibits only one rather marginal absorption band, and, after a comparison with the spectrum of the KBr solution, it is obviously a band of the bromide counterions with a small contribution from the micellizing quaternary cations of SEBr. The maximum of this band exhibits a bathochromic shift on increasing concentration, in the measured concentration range  $\lambda_{max}$  = 201.9—209.2 nm. After tables, the absorption maximum of bromides is at a wavelength 196 nm in the aqueous KBr solution [23].

For SEBr, the dependence  $A = f(c_{SEBr})$  at a constant wavelength 209.2 nm gave the value of CMC  $c^* = (7.8 \pm 0.1) \times 10^{-4}$  mol dm<sup>-3</sup> and from the concentration dependence of the shift of the absorption

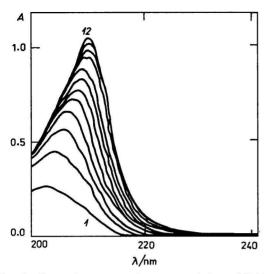


Fig. 1. Absorption spectra of aqueous solutions of SEBr. Curves 1-12 correspond to the concentrations  $c_{SEBr} = (1-12) \times 10^{-4}$  mol dm<sup>-3</sup> in the 0.5 cm cell.

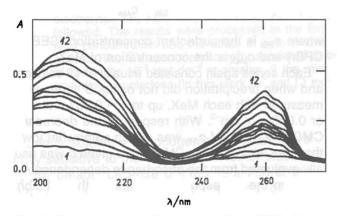


Fig. 2. Absorption spectra of aqueous solutions of CPBr. Curves 1-12 correspond to the concentrations  $c_{\text{CPBr}} = (1-10) \times 10^{-4} \text{ mol dm}^{-3}$  in the 0.5 cm cell.

maximum, only a slightly different value of CMC  $c^*$ =  $(7.9 \pm 0.1) \times 10^{-4}$  mol dm<sup>-3</sup> resulted (Fig. 3). Our spectrophotometrically determined CMC is in a good agreement with the results of other methods [10, 24], although the light absorption by the micellizing quaternary cation of SEBr is quite insufficient. The point is we utilize the spectral parameters of the bromide counterions, bound in part to the formed cationic micelles.

Absorption spectrum of CPBr is more convenient for the CMC determination since, besides of the marginal band near the wavelength of 200 nm, there is also another band with a maximum at 260.4 nm (Fig. 2). The marginal band again corresponds predominantly to the absorption of bromide counterions and its maximum exhibits analogous bathochromic shift from 201.1 nm to 208.3 nm on increasing concentration. On the other hand, the band at 260.4 nm may be assigned to the micellizing quaternary 1-hexadecylpyridinium cations and its maximum does not exhibit a substantial concentrational shift, only a

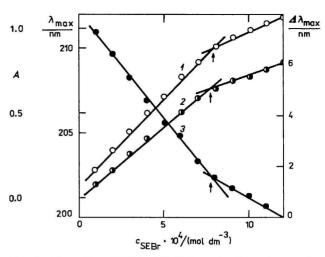


Fig. 3. Evaluation of CMC of SEBr from the dependences  $A = f(c_{\text{SEBr}})$  at  $\lambda = 209.2$  nm (1),  $\lambda_{\text{max}} = f(c_{\text{SEBr}})$  (2), or from  $\Delta\lambda_{\text{max}} = f(c_{\text{SEBr}})$  (3).

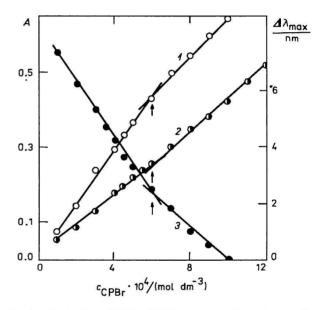


Fig. 4. Evaluation of CMC of CPBr from the dependences  $A = f(c_{\text{CPBr}})$  at  $\lambda = 208.3$  nm (1),  $\lambda = 260.4$  nm (2), and from  $\Delta\lambda_{\text{max}} = f(c_{\text{CPBr}})$  at  $\lambda_{\text{max}} = 201.1$ —208.3 nm (3).

change of the absorbance. Three ways of the evaluation of CMC of CPBr are seen in Fig. 4, two from absorbance dependences  $A = f(c_{CPBr})$  at 208.3 nm and 260.4 nm (at the respective maxima) and the third from the concentrational shift,  $\Delta \lambda_{\max} = f(c_{CPBr})$ , of the first band. All the three resulting CMC values of CPBr are practically equal,  $c^* = (6.0 \pm 0.1) \times 10^{-4}$  mol dm<sup>-3</sup>, and this average value is in a good agreement with the conductometric and potentiometric CMC [11].

Spectrum of AJBr with several absorption maxima (Fig. 5) appears to be less favourable for evaluation of CMC. Near to the 200 nm wavelength, the reliable estimation of CMC is impossible because of the inconvenient summation of the absorbances of the bromide counterions and the micellizing quaternary

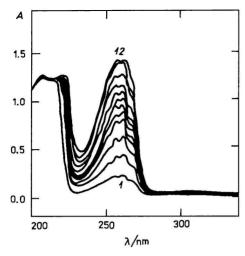
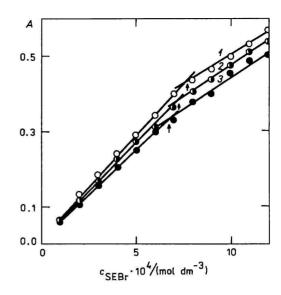


Fig. 5. Absorption spectra of aqueous solutions of AJBr. Curves 1-12 correspond to the concentrations  $c_{AJBr} = (1-10) \times 10^{-3}$  mol dm<sup>-3</sup> in the 0.5 cm cell.

cations. The absorption band in the region  $\lambda=230$ —270 nm also reflects the micellization only slightly. With some complications the average CMC of AJBr,  $c^*=(4.9\pm0.2)\times10^{-3}\,\mathrm{mol}\;\mathrm{dm}^{-3}$ , was evaluated from two absorbance curves,  $A=f(c_{\mathrm{AJBr}})$ , measured at two nearby maxima at 258 nm and 262 nm, respectively. The found value is in a good agreement with recent conductometric and potentiometric results [13] and ca. twofold of our less reliable older value [12], the reasons were already discussed [13]. Because of the unfavourable conditions, the spectrophotometric measurements of AJBr with admixture of another electrolyte were not carried out.

CMC's of the aqueous solutions of SEBr or CPBr with the admixture of a strong electrolyte MeX were evaluated mostly from the curves of absorbances.  $A = f(c_{SEBr})$  or  $A = f(c_{CPBr})$ , at constant wavelengths. At the used concentrations, absorbances of the added KF, KCI, NaCI, and LiCI were practically negligible, except of the marginal area nearby 200 nm. For this reason, the measurements with the admixtures of the mentioned salts, and also with the admixture of KBr into the solution of CPBr, were carried out at the same wavelengths as with the solutions of pure surfactants. The evaluation of CMC in three series (different  $c_t$ ) of solutions of SEBr with the admixture of KF is shown in Fig. 6. However, measurements of CMC of SEBr with the admixture of KBr were not feasible, since both the absorbance changes and the shifts of the maximum of the above described absorption band became relatively too small at the excess of bromides. Because of the precipitation, the effect of KSCN on CMC of SEBr was measurable only in a narrow concentration span. while CPBr precipitated even by a small addition of KSCN which prevented any measurements.

Spectrophotometric measurements of CMC in the presence of KI are also limited by the surfactant pre-



**Fig. 6.** Evaluation of CMC of three series of the solutions of SEBr + KF from the dependences  $A = f(c_{SEBr})$ . 1.  $c_t = 0.0008 \text{ mol dm}^{-3}$ ; 2.  $c_t = 0.0012 \text{ mol dm}^{-3}$ ; 3.  $c_t = 0.0020 \text{ mol dm}^{-3}$ .

cipitation, and another problem is the UV light absorption of iodide anions, since the aqueous KI solutions exhibit two absorption maxima at the respective wavelengths 194 nm and 226 nm [23]. The measured spectra of a series of solutions of SEBr + KI with variable  $c_{\rm SEBr}$  but constant  $c_{\rm t}$ , i.e. with a constant sum of bromides and iodides, are seen in Fig. 7. The smaller band near to 200 nm represents the sum of absorbances of the bromide and iodide anions, thus it is nearly independent of  $c_{\rm SEBr}$  and unsuitable for the CMC estimation. A new outstanding band with found  $\lambda_{\rm max}$  = 227 nm represents nearly exclusively the light absorption of iodide anions. Still, it enables

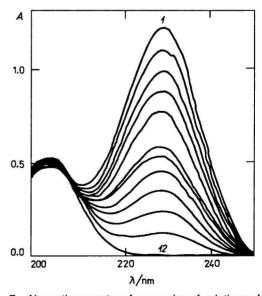


Fig. 7. Absorption spectra of one series of solutions of SEBr + KI with  $c_{\rm t}$  = 0.0012 mol dm<sup>-3</sup>. Characteristic band of iodides with  $\lambda_{\rm max}$  = 227 nm.

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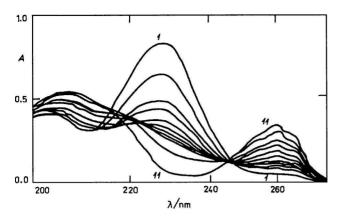


Fig. 8. Absorption spectra of one series of solutions of CPBr + KI with  $c_1$  = 0.0008 mol dm<sup>-3</sup>. Characteristic band of iodides with  $\lambda_{\rm max}$  = 229 nm.

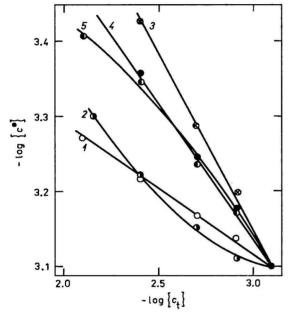


Fig. 9. CMC (c\*) of solutions of SEBr with the addition of strong electrolytes, as - log {c\*} = f(- log {c₁}). 1. KF; 2. KCl; 3. KSCN; 4. NaCl; 5. LiCl.

at least an approximate estimation of CMC from the break on the absorbance curves  $A = f(c_{SEBr})$ , measured in its maximum, apparently because of the competitive binding of the iodide anions on the formed cationic micelles.

A similar strong band with  $\lambda_{\rm max} = 229$  nm is also seen in the spectrum of solutions of CPBr + KI in Fig. 8. Here the light absorption of the iodides is summed up with a certain contribution from the 1-hexadecylpyridinium cations, but CMC may be estimated by the same way as in the case of SEBr. In both the cases CMC cannot be determined at a greater excess of KI because of the formation of the colloidal turbidity and the subsequent precipitation of the surfactant iodide. In the solutions of SEBr + KI with the total concentrations  $c_{\rm t} = 0.0012$  mol dm<sup>-3</sup> and 0.002 mol dm<sup>-3</sup> the determined CMC were 7.0 ×

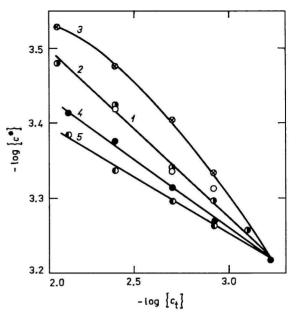


Fig.10. CMC (c\*) of solutions of CPBr with the addition of strong electrolytes, as - log {c\*} = f(- log {c₁}). 1. KF; 2. KCl; 3. KBr; 4. NaCl; 5. LiCl. The curves 1 and 2 merge.

 $10^{-4}$  mol dm<sup>-3</sup> and  $5.9 \times 10^{-4}$  mol dm<sup>-3</sup>, respectively. In the solutions of CPBr + KI with  $c_{\rm t}$  = 0.0008 mol dm<sup>-3</sup> and 0.0012 mol dm<sup>-3</sup> the respective CMC values were  $5.0 \times 10^{-4}$  mol dm<sup>-3</sup> and  $3.9 \times 10^{-4}$  mol dm<sup>-3</sup>. In the light of this work, we regard our previous electrochemical measurements of CMC with greater excess of KI, *i.e.* with higher  $c_{\rm t}$ , as unreliable [11], since it is difficult to spot visually the slight colloidal turbidity.

CMC's determined in the presence of other added electrolytes are plotted as  $\log c^* vs$ .  $\log c_t$ , for the solutions of SEBr + MeX in Fig. 9 and for CPBr + MeX in Fig. 10. The logarithmic plot is recommended by other authors [1, 5, 7, 8, 20] on a theoretical basis but, contrary to expectations, some of the curves deflect from linearity. Thus the results were also processed in the form of an extended logarithmic function

$$\log \{c^*\} = p_0 + p_1 \log \{c_t\} + p_2 \log^2 \{c_t\}$$
 (2)

In eqn (2),  $\{c^*\} = c^*/(\text{mol dm}^{-3})$  is the corresponding CMC and  $\{c_t\} = c_t/(\text{mol dm}^{-3})$  is the total concentration after eqn (1). The empirical coefficients  $p_0$ ,  $p_1$ ,  $p_2$ , calculated by the least-squares method, are in Table 1 for solutions of SEBr + MeX and in Table 2 for CPBr + MeX. Two statistical parameters and the maximum  $c_t$  values, limiting the reliability of the measurements, are also listed in Tables 1 and 2. Some electrochemical results [11] are included for the sake of comparison, the differences between them and the spectrophotometric values are mostly within the range of the given standard deviations.

In our previous work [13] we pointed out that the decrease of CMC of the cationic surfactant AJBr

**Table 1.** Coefficients of Eqn (2) Expressing the Logarithmic Dependence of CMC (c\*) on c, for SEBr

Added MeX	p <sub>0</sub>	<b>p</b> <sub>1</sub>	<b>p</b> <sub>2</sub>	±δ	r	Up to
KF	- 3.652 - 3.742 <sup>a</sup>	- 0.180 - 0.208 <sup>a</sup>		0.007 0.009 <sup>a</sup>	0.996 0.995°	8.0 8.0 <sup>a</sup>
KCI	- 4.722 - 4.546 <sup>a</sup>	- 0.966 - 0.760 <sup>a</sup>	- 0.142 - 0.095 <sup>a</sup>	0.006 0.025 <sup>a</sup>	0.998 0.968 <sup>a</sup>	7.0 8.0 <sup>a</sup>
KSCN	- 4.568	- 0.474		0.012	0.997	4.0
NaCl	- 4.238	- 0.370		0.016	0.995	8.0
LiCI	- 3.341	0.268	0.112	0.012	0.997	8.0

a) Conductometric and potentiometric results [11] .  $\delta$  is the standard deviation of the measured log  $\{c^*\}$  values from eqn (2), r is the index of correlation,  $\{c_1\} = c_1/(\text{mol dm}^{-3})$ .

**Table 2.** Coefficients of Eqn (2) Expressing the Logarithmic Dependence of CMC (c\*) on c, for CPBr

Added MeX	$\rho_0$	P <sub>1</sub>	<i>p</i> <sub>2</sub>	±δ	<i>r</i> {	Up to c <sub>t</sub> } · 10 <sup>3</sup>
KF	- 3.979 - 4.050 <sup>a</sup>	- 0.234 - 0.252ª		0.008 0.016°	0.995 0.989°	4.0 8.0 <sup>a</sup>
KĊI		- 0.230 - 0.253 <sup>a</sup>		0.017 0.016 <sup>a</sup>	0.987 0.990 <sup>a</sup>	8.0 8.0 <sup>a</sup>
KBr	- 3.216 - 4.009 <sup>a</sup>	0.423 - 0.242 <sup>a</sup>	0.131	0.004 0.012 <sup>a</sup>	0.999 0.992 <sup>a</sup>	8.0 7.0°
NaCl	- 3.822	- 0.187		0.007	0.997	7.0
LiCI	- 3.704	- 0.150		0.005	0.998	7.0

a) Conductometric and potentiometric results [11].

caused by the presence of another electrolyte depended not only on the nature of anions but also cations of the added electrolyte. The results presented in Figs. 9 and 10 show similar behaviour of the studied cationic surfactants SEBr and CPBr, when a strong univalent electrolyte is added into their aqueous solutions. Besides, the effect of the cations Li<sup>+</sup>, Na<sup>+</sup>, and K<sup>+</sup> on the CMC lowering of CPBr increases regularly in this order (Fig. 10). On the other hand, the order of the effect of these cations on CMC of SEBr is rather irregular (Fig. 9), in a similar way observed with various anions [11, 13].

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