



# Impact of acetazolamide on the double layer parameters at the R-AgLAFe/chlorates(VII) interface in the presence of ionic surfactants

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## Abstract

Adsorption of acetazolamide (ACT) and formation of a mixed adsorption layers of acetazolamide (ACT)—sodium 1-decane-sulfonate (SDS) and acetazolamide—hexadecyltrimethylammonium bromide (CTAB) at the R-AgLAFe/chlorates(VII) interface are described. The systems were characterized by the measurements of differential capacity, potential of zero charge, and surface tension at this potential. The adsorption parameters determined in the studied systems indicate the SDS domination in the adsorption equilibria and the competitive adsorption between the ACT—SDS or mixed micelles. However, acetazolamide dominates at adsorption equilibria of the ACT—CTAB mixture.

**Keywords** Adsorption · Mixed adsorption · Double layer parameters · Ionic surfactants · Acetazolamide

## 1 Introduction

Explanation of the mixed adsorption layers formation is significant importance as it is the basis for the action of corrosion inhibitors [1, 10, 11, 18, 22, 32]. The mechanism of the corrosion inhibitor action may differ depending on the type of metal, the character of environment and the structure of the inhibitor [3, 4, 14, 15, 26, 29, 32]. One possible mechanism involves adsorption of the inhibitor, which blocks the metal surface and prevents the corrosion process. The use of corrosion inhibitors is to prevent the anodic and cathodic processes that may be triggered by an increase in the overvoltage or to block the active metal surface. Organic corrosion inhibitors belong to the same type of compounds as those preventing metal etching in acids or shine-formers [3, 4, 7, 14, 30]. Their effectiveness depends largely on their adsorption properties, as adsorption of such compounds changes the metal ability to resist corrosion. The process of

adsorption usually involves electrostatic or covalent interactions between the atoms of the inhibitor and the metal surface. The adsorbed organic molecules form a protective layer on the surface that prevents or reduces the rate of metal dissolution.

Thanks to their great effectiveness, ease of production, relatively low price and, above all, no threat to the environment, ionic and nonionic surfactants have been generally appreciated corrosion inhibitors for steel, copper, aluminum and iron [2, 19, 21, 26, 32]. The use of mixtures of these compounds can significantly improve the corrosion prevention effectiveness [3, 12, 27, 31]. Thus, the adsorption mechanism is mainly related to competitive adsorption [5, 8, 9, 13, 24]. The degree of competitiveness of adsorption depends on the potential of the electrode, its charge as well as the interactions in the adsorbate layer and those at the adsorbate–metal (electrode) interface.

The adsorption of acetazolamide and formation of mixed adsorption layers of acetazolamide—sodium 1-decane-sulfonate and acetazolamide—hexadecyltrimethylammonium bromide was studied. Experimental data indispensable for the determination of adsorptive properties of acetazolamide and mixed layers were obtained from measurements of differential capacity of the double layer, the potential of zero charge and the surface tension with this potential.

Earlier adsorption studies indicated a change in the structure and properties of the adsorption layer at the R-AgLAFe/

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aqueous chlorates(VII) solution interface in the presence of mixtures of an organic substance and a surfactant, which resulted in the mixed adsorption layer formation [5, 8, 9, 13, 24].

## 2 Experimental

### 2.1 Materials and methods

A cyclically renewable liquid silver amalgam film electrode (R-AgLAFe) was prepared by reproduction of the liquid amalgam layer prior to the series of measurements. The 1% liquid amalgam was prepared by immersion of the pieces of silver wire (of 0.5 mm diameter) in the 0.5 ml mercury for 2 weeks [25].

The automatic cell stand (Mtm Anko) included an electrode cell containing: a R-AgLAFe electrode along with the automatized steering system as well as the stable reproducible electrode surface (17.25 mm<sup>2</sup>), as a working electrode; an Ag/AgCl (3 M saturated solution of NaCl), as a reference electrode and a platinum spiral, as an auxiliary electrode at 298 K.

More detailed description of the R-AgLAFe electrode refreshment before each measurement, the operating principles, and the design are presented in the paper [25].

Acetazolamide (Sigma Aldrich) used in the study was of analytical purity. CTAB (Fluka) and SDS (Fluka) were used without further purification. The solutions were made using newly doubly-distilled water purified in a Millipore Milli-Q system. Before measurements, the solutions were deaerated using high-purity nitrogen (assay 99.999%, Air Liquid). Nitrogen was passed over the solution during the measurements.

The basic electrolyte contained 1 mol·dm<sup>-3</sup> chlorates(VII) solution (mixed in the proportions 0.5 mol·dm<sup>-3</sup> NaClO<sub>4</sub> (Fluka) + 0.5 mol·dm<sup>-3</sup> HClO<sub>4</sub> (Fluka)), made of the reagents of analytical purity. Acetazolamide solutions were prepared directly before the measurements. Their concentration was in the interval from 1·10<sup>-5</sup> to 1·10<sup>-3</sup> mol·dm<sup>-3</sup>, but the concentration of CTAB ranged from 1.5·10<sup>-6</sup> to 1.5·10<sup>-5</sup> mol·dm<sup>-3</sup>, and that of SDS ranged from 1·10<sup>-5</sup> to 9·10<sup>-5</sup> mol·dm<sup>-3</sup>.

The double layer capacity ( $C_d$ ) was measured using the AC impedance technique at several frequencies with the amplitude of 5 mV, using an electrochemical analyzer Autolab controlled by the GPES software (Version 4.9) (Eco Chemie, Utrecht Netherlands). The reproducibility of the average capacity measurements was  $\pm 0.5\%$ .

The double layer capacity ( $C_d$ ) was measured using the impedance technique at several frequencies with the amplitude of 5 mV. For the whole polarization range, the capacity dispersion was tested at different frequencies

changed between 200 and 1000 Hz. Adsorption equilibrium was not preserved in any of the systems. Therefore, to obtain the equilibrium values of differential capacity, the linear dependence of capacity on frequency was extrapolated to the zero frequency [23]. The reproducibility of the average capacity measurements was  $\pm 0.5\%$ .

The potential of zero charge ( $E_z$ ) was measured for each solution by the method of streaming mercury electrode [24], (the accuracy of  $\pm 0.1$  mV).

The surface tension at the potential of zero charge ( $\gamma_z$ ) was determined using the method of the highest pressure inside the mercury drop [24], (the accuracy of  $\pm 0.2$  mNm<sup>-1</sup>).

Viscosity measurements were performed using a rotational CVO 50 rheometer with the “double gap”; measuring system (Bohlin Instruments).

Applying the viscosity method [using a rotational CVO 50 rheometer (Bohlin Instruments)] [24] the critical micelle concentration (CMC) was determined. The measurements were made in the 1 mol·dm<sup>-3</sup> chlorates(VII)—CTAB, 1 mol·dm<sup>-3</sup> chlorates(VII)—SDS and in the presence of acetazolamide system.

## 3 Results and discussion

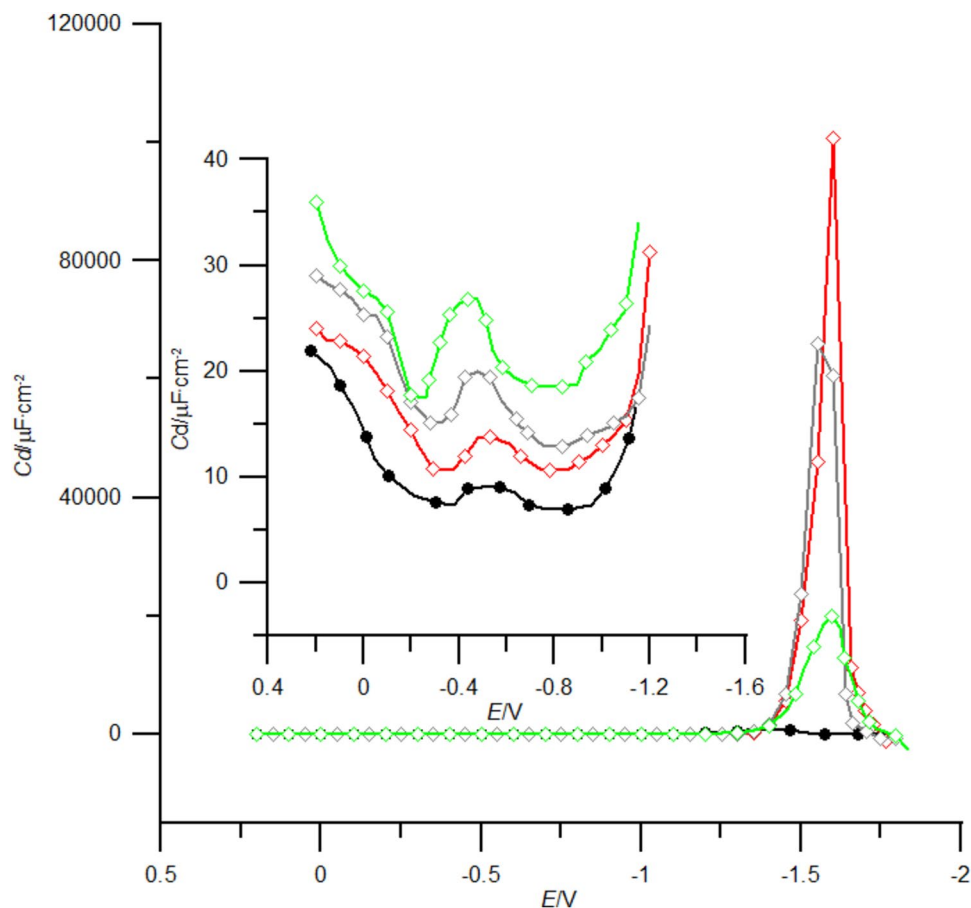
The evident changes in the differential capacity curves in the presence of acetazolamide indicate the adsorption of the substance at the R-AgLAFe/1 mol·dm<sup>-3</sup> chlorates(VII) interface (Fig. 1). The possible partial charge transfer between the sulfur atom and the electrode due to  $\pi$  electrons suggests specific adsorption [5, 17, 28].

In the region of the hump potentials ( $\approx -200$  –  $-800$  mV), after the addition of ACT to the solution, the height of the hump increases. In the region of  $\approx 100$  mV potential, small adsorption peaks appear in the presence of ACT (Fig. 1), however, in the area of the most negative potentials about  $-1600$  mV a desorption peak appears. It should be noted that the increase in ACT concentration in the basic electrolyte results in the evident decrease in the desorption peaks.

The addition of the surfactant to the basic electrolyte solution containing ACT (Fig. 2) changes the character of the capacity curves, particularly for the 5·10<sup>-5</sup> mol·dm<sup>-3</sup> (CMC) concentration. It should be noted that the increase in the SDS concentration in the basic electrolyte results in the evident increase in adsorption and decrease in the desorption peaks. In turn, the appearance of a clear adsorption peak is definitely associated with the competitive adsorption between the ACT and SDS molecules or mixed micelles [5, 8, 9, 13, 24]. The surfactant molecules dominate in the formulation [13, 16, 24].

However, the interactions of ACT molecules and the surfactant resulting in the formation of a more or less compact

**Fig. 1** Differential capacity—potential curves of the double layer interface R-AgLAFe/1 mol·dm<sup>-3</sup> chlorates(VII) (●) with various concentrations of ACT (1·10<sup>-5</sup> mol·dm<sup>-3</sup> (Red ◇); 1·10<sup>-4</sup> mol·dm<sup>-3</sup> (Grey ◇); 1·10<sup>-3</sup> mol·dm<sup>-3</sup> (Green ◇)). Capacitance curves obtained after extrapolation to the zero frequency (Color figure online)



structure of mixed adsorption layers cannot be excluded [13, 16, 24].

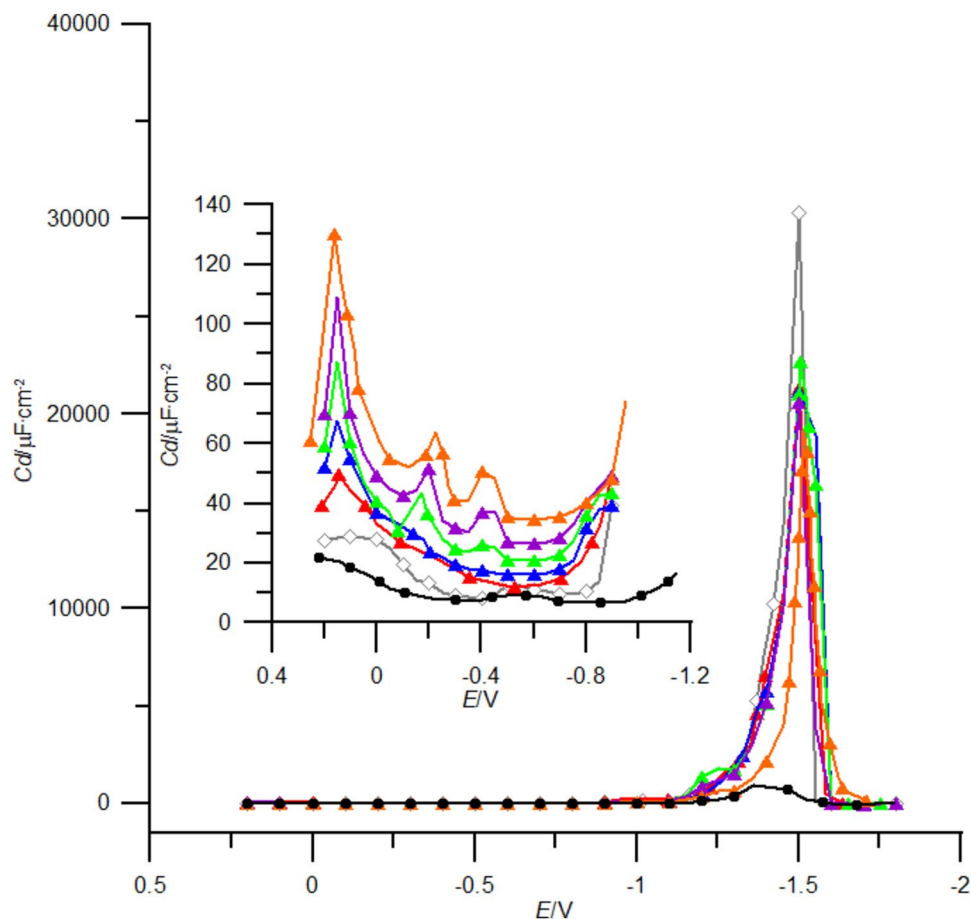
Moreover, the presence of CTAB in the basic electrolyte solution containing  $1 \cdot 10^{-4}$  mol·dm<sup>-3</sup> of ACT, does not change the character of capacity curves (Fig. 3). It should be noted that with increasing CTAB concentration, the height of the desorption peaks does not change. As CTAB is a cationic molecule, one can expect its different arrangements on the electrode surface. It should be mentioned that in the range of the studied concentrations in the mixture of ACT and cationic detergent, the critical micelle concentration CMC was not observed [20]. Definitely one can observe a change in the structure and properties of the adsorption layer at the R-AgLAFe/aqueous chlorates(VII) solution in the presence of ACT and CTAB solution, which results in the mixed adsorption layer formation. However, the predominance of acetazolamide in formation of adsorption equilibria of acetazolamide-CTAB should be pointed out [17, 24, 28]. However, such a predominance was not observed for acetazolamide-SDS.

As follows from Table 1, the addition of ACT in concentrations increasing to  $1 \cdot 10^{-4}$  mol·dm<sup>-3</sup>, to a 1 mol·dm<sup>-3</sup> chlorates(VII) solution causes a shift of  $E_z$  towards more positive potentials.

A further increase in the ACT concentration results in a change in the  $E_z$  value toward more negative potentials, which may suggest a change in the position of the ACT molecule on the electrode surface [13, 16, 24]. The trend of the value change indicates definitely the change in the orientation of the molecule from the positive to the negative direction relative to the electrode surface [6]. At lower concentrations, ACT molecules assume a more oblique orientation towards the electrode surface, so that their interaction with the electrode surface is weaker, making them more accessible to the electrode surface. At higher concentrations, on the other hand, the interaction becomes much stronger as a result of the transition of the molecule into a planar orientation, facilitating the interaction of the  $\pi$  electrons of sulfur present in the aromatic ring with the electrode surface [5, 17, 28] (Scheme 1).

The addition of SDS results in a shift of  $E_z$  towards the positive potentials. However, it should be noted that for the CMC concentration of  $5 \cdot 10^{-5}$  mol·dm<sup>-3</sup> these changes are definitely significant (the displacement effect is much pronounced) and may indicate the aggregation phenomena at the Hg–aqueous electrolyte solution interface in the presence of surfactant (Table 2) [20].

**Fig. 2** Differential capacity—potential curves of the double layer interface R-AgLAFc/1 mol·dm<sup>-3</sup> chlorates(VII) (●) with 1·10<sup>-4</sup> mol·dm<sup>-3</sup> ACT (Grey ◇) and with various concentrations of SDS (1.5·10<sup>-5</sup> mol·dm<sup>-3</sup> (Red ▲); 3·10<sup>-5</sup> mol·dm<sup>-3</sup> (Blue ▲); 5·10<sup>-5</sup> mol·dm<sup>-3</sup> (Green ▲); 7.5·10<sup>-5</sup> mol·dm<sup>-3</sup> (Purple ▲); 9·10<sup>-5</sup> mol·dm<sup>-3</sup> (Yellow ▲)). Capacitance curves obtained after extrapolation to the zero frequency (Color figure online)



The increase in CMC value in the presence of ACT (CMC for SDS = 3·10<sup>-5</sup> mol dm<sup>-3</sup>) and in the presence of ACT (CMC = 5·10<sup>-5</sup> mol dm<sup>-3</sup>) should be associated with competitive adsorption but also between the mixed micelles [5, 8, 9, 13, 24]. A synergistic effect should not be excluded [5, 8, 9, 13, 24]. This observation is consistent with the conclusions resulting from the differential capacitance curves analysis (Fig. 2).

Moreover, in the presence of CTAB in the basic electrolyte solution containing 1·10<sup>-4</sup> mol dm<sup>-3</sup> of ACT,  $E_z$  is shifted towards more positive potentials (Table 3). No evident anomalies were observed in the changing values.

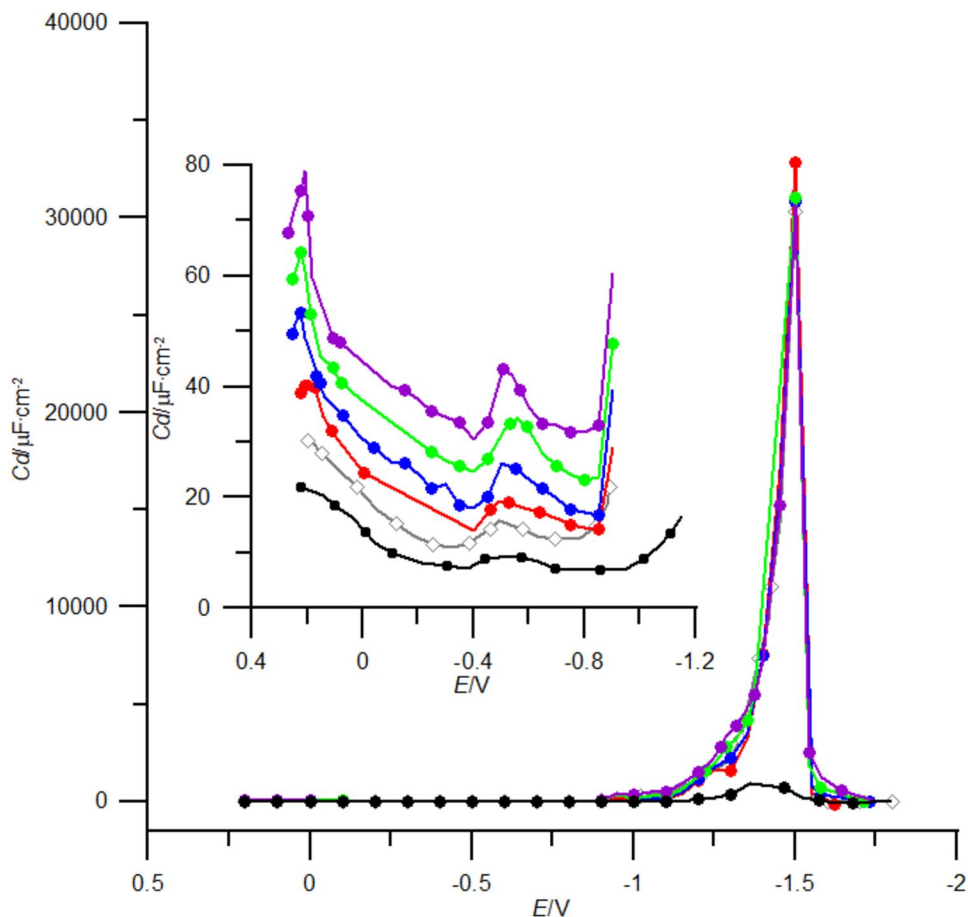
The values of surface tension  $\gamma_z$  (Tables 1, 2, 3) decrease for all studied systems as proved by the adsorption phenomenon.

## 4 Conclusions

The results presented in this paper show specific adsorption of ACT at the R-AgLAFc/1 mol·dm<sup>-3</sup> chlorates(VII) interface. Changes in  $E_z$  with increasing ACT concentration suggest a change in the orientation of the ACT molecule on the electrode surface. Formation of the mixed adsorption layers of acetazolamide and an ionic surfactant at the R-AgLAFc/chlorates(VII) interface was proved. In the systems containing ACT and SDS, the surfactant dominance and competitive adsorption [appearance of a well-developed adsorption peak (Fig. 2)] or the formation of mixed micelles should be considered.

However, acetazolamide dominates in the formation of adsorption equilibria of the ACT-CTAB mixture. The absence of a well-developed adsorption peak in this system (Fig. 3) suggests the co-adsorption of ACT and CTAB, with the CTAB interactions with the electrode taking place through the adsorbed molecule, which can be partially inclined in these conditions. In both mixed systems interactions the role of ClO<sub>4</sub><sup>-</sup> ions should be neglected.

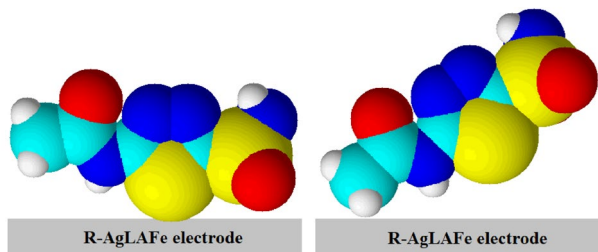
**Fig. 3** Differential capacity—potential curves of the double layer interface. R-AgLAFe/ $1 \text{ mol}\cdot\text{dm}^{-3}$  chlorates(VII) (●) with  $1\cdot 10^{-4} \text{ mol}\cdot\text{dm}^{-3}$  ACT (Grey ◇) and with various concentrations of CTAB ( $1.5\cdot 10^{-6} \text{ mol}\cdot\text{dm}^{-3}$  (Red ●);  $3\cdot 10^{-6} \text{ mol}\cdot\text{dm}^{-3}$  (Blue ●);  $6\cdot 10^{-6} \text{ mol}\cdot\text{dm}^{-3}$  (Green ●);  $1.5\cdot 10^{-5} \text{ mol}\cdot\text{dm}^{-3}$  (Purple ●). Capacitance curves obtained after extrapolation to the zero frequency (Color figure online)



**Table 1** The values of the zero charge potentials  $E_z$  vs. Ag/AgCl electrode and surface tension  $\gamma_z$  for  $E_z$  for the studied systems ( $1 \text{ mol}\cdot\text{dm}^{-3}$  chlorates(VII)+ different ACT)

$10^4 c_{\text{ACT}}/\text{mol}\cdot\text{dm}^{-3}$	$-E_z/\text{V}$	$\gamma_z/\text{m N}\cdot\text{m}^{-1}$
0.00	0.466	423.0
0.10	0.454	421.0
0.30	0.447	420.7
0.50	0.440	420.3
1.00	0.435	419.6
3.00	0.446	420.2
5.00	0.452	420.8
10.0	0.463	421.4

to  $1\cdot 10^{-4} \text{ mol}\cdot\text{dm}^{-3}$  ACT      up  $1\cdot 10^{-4} \text{ mol}\cdot\text{dm}^{-3}$  ACT



**Scheme 1** Scheme of acetazolamide orientation on the R-AgLAFe electrode surface depending from concentration of ACT



**Table 2** The values of the zero charge potentials  $E_z$  vs. Ag/AgCl electrode and surface tension  $\gamma_z$  for  $E_z$  for the studied systems (1 mol dm<sup>-3</sup> chlorates(VII)+1•10<sup>-4</sup> mol•dm<sup>-3</sup> ACT+different SDS surfactant)

10 <sup>5</sup> c <sub>SDS</sub> /mol dm <sup>-3</sup>	- E <sub>z</sub> /V	γ <sub>z</sub> /m N m <sup>-1</sup>
0.0	0.435	419.6
1.5	0.428	460.1
3.0	0.411	459.3
5.0	0.368	457.5
7.5	0.352	455.8
9.0	0.335	453.9

**Table 3** The values of the zero charge potentials  $E_z$  vs. Ag/AgCl electrode and surface tension  $\gamma_z$  for  $E_z$  for the studied systems (1 mol dm<sup>-3</sup> chlorates(VII)+1•10<sup>-4</sup> mol dm<sup>-3</sup> ACT+different CTAB surfactant)

10 <sup>5</sup> c <sub>CTAB</sub> /mol dm <sup>-3</sup>	- E <sub>z</sub> /V	γ <sub>z</sub> /m N m <sup>-1</sup>
0.0	0.435	419.6
0.15	0.402	387.5
0.30	0.398	384.7
0.60	0.395	382.5
1.50	0.394	383.2

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**Data availability** At the author of correspondence.

## Declarations

**Conflict of interest** The authors declare that they have no conflict of interest.

**Ethical approval** Not applicable.

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