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## **Influence of adsorption of n-alkyltrimethylammonium bromides (C<sub>8</sub>, C<sub>12</sub>, C<sub>16</sub>) and bubble motion on kinetics of bubble attachment to mica surface**

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Abstract. Influence of adsorption of n-alkyltrimethylammonium bromides (C<sub>8</sub>, C<sub>12</sub>, C<sub>16</sub>) and formation of motion induced dynamic architecture of adsorption layer (DAL) over surface of the colliding bubble on kinetics of three-phase contact (TPC) formation at mica surface was studied. The dynamic phenomena occurring during the bubble collisions were monitored using a high-speed camera of frequency 1040 Hz. The effect of solution concentration and the DAL formation, due to the bubble motion, was determined. It was showed that stability of the wetting film formed between the colliding bubble and mica surface was governed by the electrostatic interactions between the film interfaces. It was found that when the distance covered by the bubble (i.e. the distance between the capillary and the mica surface) was L=3 mm (location "close") then the time of the three phase contact formation ( $t_{\text{TPC}}$ ), was significantly shorter than for the L=100 mm (location "far"). The differences between the  $t_{\text{TPC}}$  for the locations "close" and "far" were the largest at lowest concentration. The mechanism responsible for significant differences in the  $t_{\text{TPC}}$  values for the location "close" and "far" is described.

*keywords: three phase contact, cationic surfactant, bubble collision, thin liquid film, electrostatic interactions, charge reversal*

### 1. Introduction

Flotation separation is a dynamic process, where motion and collisions of bubbles and grains leading to formation of stable bubble-grain aggregates are of crucial importance. The collision time of the gas bubble and grain is rather short (milliseconds) and the three phase contact (TPC) and formation of needs to occur during the collision. For facilitation the TPC and stable bubble-gas aggregates various flotation reagents are used. The flotation reagents (surface active substances) are added mainly to modify properties of the gas/liquid and liquid/solid interfaces. The addition of these substances can change the hydrophobic/hydrophilic properties of solid/liquid interface and/or electrostatic properties of both solid/liquid and gas/liquid interfaces and facilitates the TPC formation (Zawala et al., 2008). For the TPC formation a liquid film separating the colliding bubble and grain needs to be ruptured.

Stability of the liquid films is determined, according to the DLVO theory, by interrelation between electrostatic interactions of the electrical double layers of two interfaces (long-range, 1-100 nm), and the van der Waals interactions (short, 1-10 nm) (Scheludko, 1967). Thus, when a wetting film is formed during the bubble collision with solid surface then the electrostatic interactions start to operate when the draining film reaches locally a thickness ca. 100nm. When the film interfaces are similarly charged, the repulsive interactions between the surfaces stabilize the film. Otherwise, that is, when the film interfaces are oppositely charged, the attractive electrostatic forces act as a destabilizing factor leading to the film rupture. Thus, a preferential adsorption of ionic surfactant on one of the film interfaces can cause a reversal of the surface electrical charge. As a result the electrical interactions can be changed from repulsive to attractive, leading to the film rupture and the TPC formation.

Adsorption of surface active substances (SAS) can also affect strongly the bubble motion (Levich, 1962, Dukhin et al., 1959, 1995, Malysa et al., 2005, Zhang et al., 2001, Krzan et al., 2007). Velocity of the bubble can be strongly lowered in solutions of surface active substances (SAS) and as a result the time of contact between the colliding bubble and grain is prolonged. When the bubble formed in SAS solution is motionless then adsorption coverage over its surface is uniform. As a consequence of viscous drag exerted by continuous medium on the rising bubble interface the uneven distribution of adsorption coverage, due to surface advection flow from the bubble top to the bottom stagnation point, is induced (Frumkin, 1947, Levich, 1962). This non-uniform bubble adsorption coverage leads to inducement of surface tension gradient and causes a tangential 'Marangoni' stress opposing the flow shear stress. The uneven distribution of the surfactant molecules over the bubble surface, with practically no adsorption coverage at the top bubble pole, is called dynamic adsorption layer (DAL). As a consequence of the DAL formation the bubble interface mobility is retarded and its velocity can be lowered even by over 50% (Malysa et al., 2005; Krzan, et al., 2007).

The paper presents results of experiments on kinetics of the TPC formation by the bubble colliding with mica surface in solutions of three n-alkyltrimethylammonium bromides, having 8, 12 and 16 carbon atoms in alkyl chain (OTAB, DDTAB and CTAB, respectively). The effect of (i) preferential adsorption of SAS at the gas/liquid interface, and (ii) the DAL formation at the bubble surface was investigated. It is showed that stability of the wetting film (time of the TPC formation) vary with the n-alkyltrimethylammonium bromides concentration and depends on the molecule chain-length. The TPC formation at mica surface is governed by the attractive electrostatic interactions and the time of the TPC formation is affected by the DAL formation induced by the bubble motion - wetting film ruptures when the adsorption coverage at the bubble surface is high enough to change the sign of the bubble surface from negative to positive.

## 2. Experimental

Experiments were carried out using experimental set-up presented schematically in Fig. 1. It consisted of: (i) a square glass column (cross section 50x50mm), (ii) glass capillary of inner diameter 0.075 at the bottom, (iii) syringe pump with gas tight high precision glass syringes (Hamilton), enabling well-controlled air supply, (iv) high-speed camera (SpeedCam MacroVis), (v) PC with image analysis software. Collisions of the single bubbles with mica plates were recorded at the camera frequency 1040 Hz and analyzed using the WinAnalyze 3D software. The movies were also transformed into BMP pictures and analyzed using the SigmaScan Pro 5.0 Image Analysis Software. The bubble velocity variations were determined from measurements of the bubble bottom pole positions on subsequent frames of the camera recording.

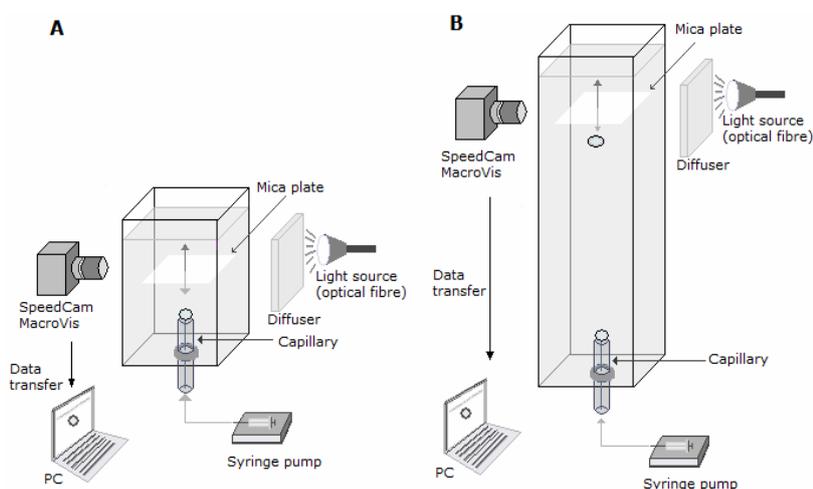


Fig. 1. Schematic of the experimental set-up - A) mica plate at location “close” ( $L=3\text{mm}$ ), B) mica plate at location “far” ( $L=100\text{mm}$ )

Prior to each experiment the gas flow was adjusted carefully to get the time interval of ca. 30 s between two subsequent bubbles detaching from the capillary. The mica plates, freshly cleaved from sheets of layered mica sample, were positioned horizontally inside the column, just below the solution surface. Two series of experiments were carried out in the solutions studied to find if and how the motion induced dynamic (non-equilibrium) architecture of adsorption layer (DAL) affects an outcome of the collisions and time of the bubble attachment to mica surface. In the first series the distance between the bubble formation point and the surface of mica plate ( $L$ ) was ca. 3 mm (see Fig. 1A). This location of mica plate will be further called the location “close” to underline the fact that the bubble collided with mica plate immediately after detachment from the capillary and therefore the adsorption coverage over its surface was uniform. In the second series (see Fig. 1B) the mica plated was

fixed at the distance  $L=100$  mm (location “far”) and at this distance, as showed previously (Krzan et al., 2007), the DAL was formed over the bubble. To get reliable data the experiments were repeated 20-30 times for each solution concentration and the mica plate location.

All experiments were carried out at room temperature. N-octyltrimethylammonium (OTABr), n-dodecyltrimethylammonium (DDTABr) and n-hexadecyltrimethylammonium (CTABr) bromides were commercial reagents (Fluka) of high purity ( $\geq 99\%$ ). High purity distilled water was used in the experiments.

### 3. Results and discussion

#### 3.1. Uniform adsorption coverage over surface of the colliding bubble

Adsorption coverage of surface active molecules over surface of the motionless bubble, i.e. when the bubble is growing at the capillary orifice, is uniform. Thus, when the mica plate is located in close vicinity of the capillary then the adsorption coverage over the colliding bubble is still almost uniform. Figure 2 presents the series of photos illustrating the bubble collision with mica plate at the location “close” ( $L=3$ mm) in distilled water and OTABr and CTABr solutions of concentration  $1 \cdot 10^{-2}$  and  $5 \cdot 10^{-6}$  M, respectively. For the sake of convenience the moment of the bubble first collision was denoted as  $t=0$ . It is seen in Fig. 2 that in all cases the bubble bounced from the mica surface after the first collision and the amplitude of the bounce was the largest one in distilled water. In the OTABr and DDTABr solutions the bouncing distance was shorter due to smaller impact velocity of the colliding bubble resulting from presence of adsorption layer over the bubble surface. Note please, in the last sequence of each of the photo series (Fig. 2), that the TPC was not formed at mica surface in distilled water but was formed in the OTABr and CTABr solutions. After a complete dissipation of the kinetic energy, that is, when the bubble was almost motionless, the bubble stayed captured beneath the mica plate without the TPC formation in distilled water. As there was no TPC formation even after long time (bubble was monitored for 15min) so it means that the wetting film formed was stable and did not rupture. Otherwise occurred in OTABr and CTABr solutions - the wetting film was unstable and the TPC was formed on mica surface after the film rupture (see the marked photos in Fig. 2). The time of TPC formation ( $t_{\text{TPC}}$ ), determined as the time interval between the bubble first collisions till the moment of bubble attachment, was  $55 \pm 19$  and  $138 \pm 23$  ms for these OTABr and CTABr solutions, respectively.

Figure 3 presents a comparison of the velocity variations during the bubble collisions in distilled water,  $1 \cdot 10^{-2}$  M OTABr and in  $5 \cdot 10^{-6}$  M CTABr solutions with the mica plates at location “close”. The bubble velocity variations are presented as a function of time and there are also marked moments of the bubble attachment (TPC formation) to the mica surface. It is clearly seen in Fig. 3 that at location “close” the

bubbles were at the acceleration stage of their motion and the impact velocity of the bubble collision was the highest (ca. 27cm/s) in distilled water. In the OTABr and CTABr solutions the bubble impact velocity was lower - ca. 14.5 cm/s and 13 cm/s, respectively. It needs to be added here that the velocity was determined in respect to the bubble bottom pole. Thus, moment of the TPC formation means a rapid jump in position of the bubble bottom pole and therefore is clearly noticeable when the bubble stays motionless. after dissipation of the kinetic energy associated with its motion (see Figs. 3 and 4).

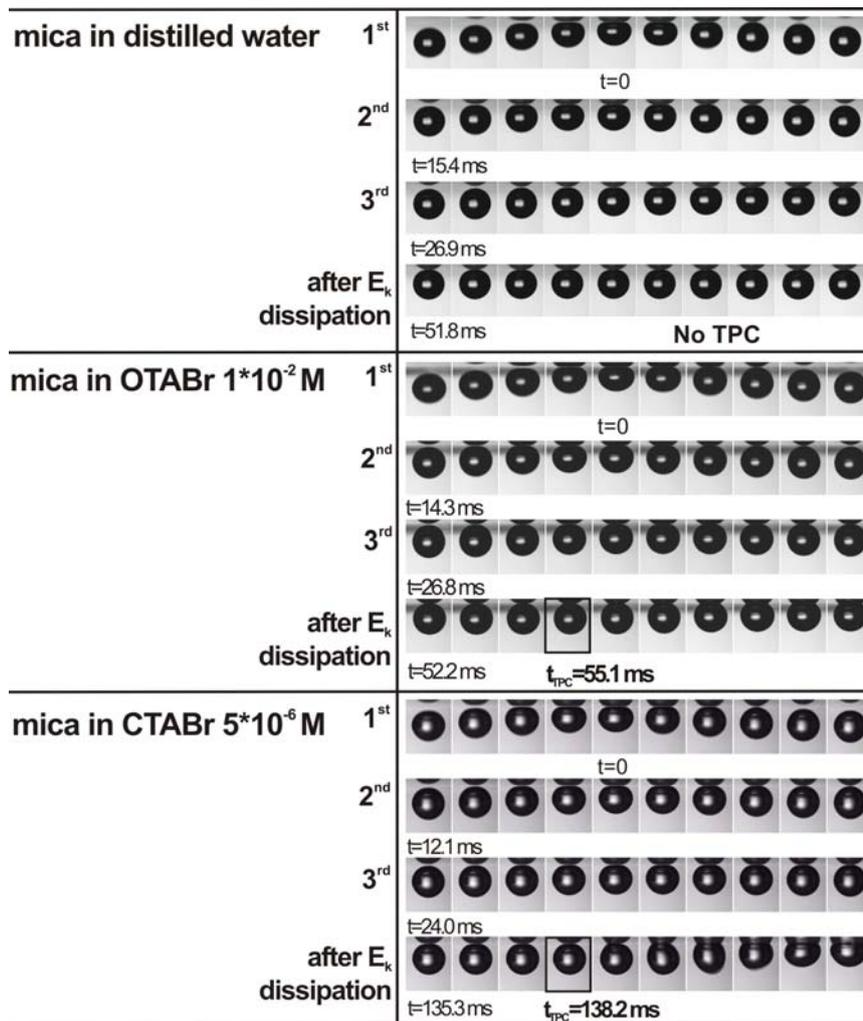


Fig. 2. Sequences of photos illustrating the bubble collision with mica plate (location “close”) in distilled water and OTABr and CTABr solutions of concentration  $1 \cdot 10^{-2}$  and  $5 \cdot 10^{-6}$  M, respectively. Time interval between subsequent photos  $\Delta t = 0.96$  ms - when not marked

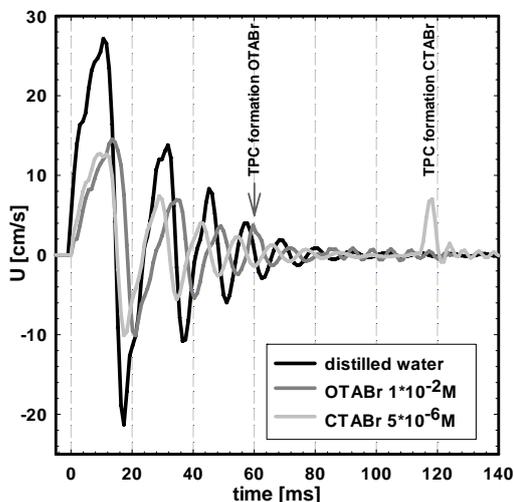


Fig. 3. Velocity variations during the bubble collisions in distilled water  $1 \cdot 10^{-2}$  M OTABr and  $5 \cdot 10^{-6}$  M CTABr solutions with mica plate located at the distance  $L=3$  mm from the capillary orifice

Let us discuss reasons of differences in stability of the wetting films formed at mica surface in distilled water and solutions of the n-alkyltrimethylammonium bromides studied. Mica is a model hydrophilic surface with water contact angle equal zero. As was demonstrated (see Figs 2-3) there was no the TPC formation and the bubble attachment when the rising bubble collided with mica surface in water because the water wetting film formed at mica surface was stable. Mica surface immersed in distilled water is negatively charged and its zeta potential value is reported to be in the range between -80 and -120 mV (Scales et al., 1992, Debacher and Ottewill, 1992; Zembala et al., 2001, 2003; Zembala and Adamczyk, 2000). In distilled water the bubble surface is also negatively charged and zeta potential values reported in literature varied from -35 mV (Stockelhuber, 2003) to -65mV (Graciaa et al., 1995; Lee and Li, 2006). Thus, in distilled water both the air/water and water/mica interfaces of the wetting films, formed by the colliding bubble, are negatively charged. These repulsive electrostatic interactions assure stability of the wetting film and prevent formation of the TPC in distilled water. In the case of n-alkyltrimethylammonium bromides (cationic surfactants) solutions the situation was different – the film was unstable and TPC formation was observed. It was due to the fact that the cationic surfactant molecules were preferentially adsorbed and caused a reversal of the bubble surface electrostatic charge from negative to positive. As a consequence of the charge reversal there were attractions interactions between oppositely charged interfaces of the wetting film formed in solutions of n-alkyltrimethylammonium bromides studied.

### 3.2. Non-uniform adsorption coverage over the bubbles – DAL is formed

Motion leads, as described above, to formation a dynamic architecture of the adsorption layer with significantly diminished adsorption coverage at the bubble top

pole. The bubble velocity variations during its collisions with mica plate located at  $L=$ “close” and “far” in  $1 \cdot 10^{-6}$  M DDTABr solution are presented in Fig. 4. It is seen that due to different distances covered by the bubble before the collision, the bubble impact velocity was different for these two interface locations. In the case of  $L=100$  mm the bubble impact velocity was 34 cm/s, while for  $L=3$  mm it was 12.8 cm/s, only. This difference in the impact velocity is the reason that longer time was needed for dissipation of the bubble kinetic energy when the mica plate was at location “far”, but this time difference was ca. 40 ms, only. For the both mica plate locations the TPC was formed in  $1 \cdot 10^{-6}$  M DDTABr solution, after the kinetic energy dissipation, i.e. when the bubble was motionless beneath the mica surface. Note please that the time of the TPC formation was much longer when the mica was located at the distance  $L=100$  mm. In the case of  $L=3$  mm the  $t_{\text{TPC}}=542 \pm 79$  ms while for  $L=100$  mm was elongated to  $1837 \pm 440$  ms.

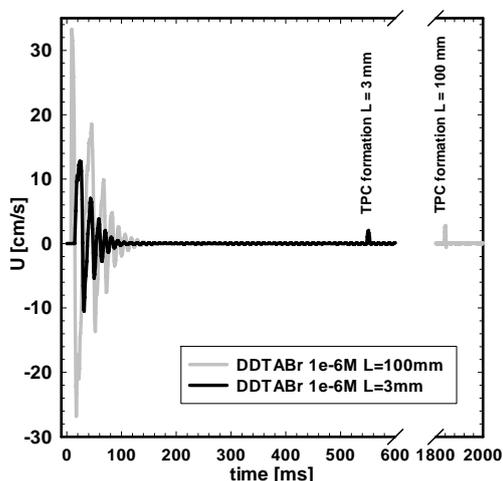


Fig. 4. Variation of the bubble local velocity during collisions with mica plate located “close” ( $L=3$ mm) and “far” ( $L=100$  mm) in  $1 \cdot 10^{-6}$ M DDTABr

Figure 5 presents the  $t_{\text{TPC}}$  values as a function of the OTABr, DDTABr and CTABr concentration. As there are given the  $t_{\text{TPC}}$  values for the mica plate locations “close” and “far” so a few important features can be noticed there: i) the differences in the time of the TPC formation are the largest at lowest concentrations, ii) differences in the  $t_{\text{TPC}}$  values between both mica locations depend on the surfactant type, iii) these differences are tending to zero at high concentrations, and iv) regions of the  $t_{\text{TPC}}$  variations are shifted towards higher concentrations with decreasing *n*-alkyltrimethylammonium bromide surface activity, that is, when the hydrocarbon chain is shorter. As described above the TPC formation at mica in OTABr, DDTABr and CTABr solutions is due, in our opinion, to a charge reversal of the bubble surface charge, from positive to negative, resulting from a preferential adsorption of these cationic surfactant molecules at solution/gas interface. However, possibility of the mica surface charge reversal and/or hydrophobization due to adsorption of cationic

surfactant on negatively charged surface needs to be taken also into considerations and its potential importance should be evaluated. Influence of n-alkyltrimethylammonium bromides on the mica zeta potential was studied by Debacher and Ottewill (1992) and Fa et al. (2005).

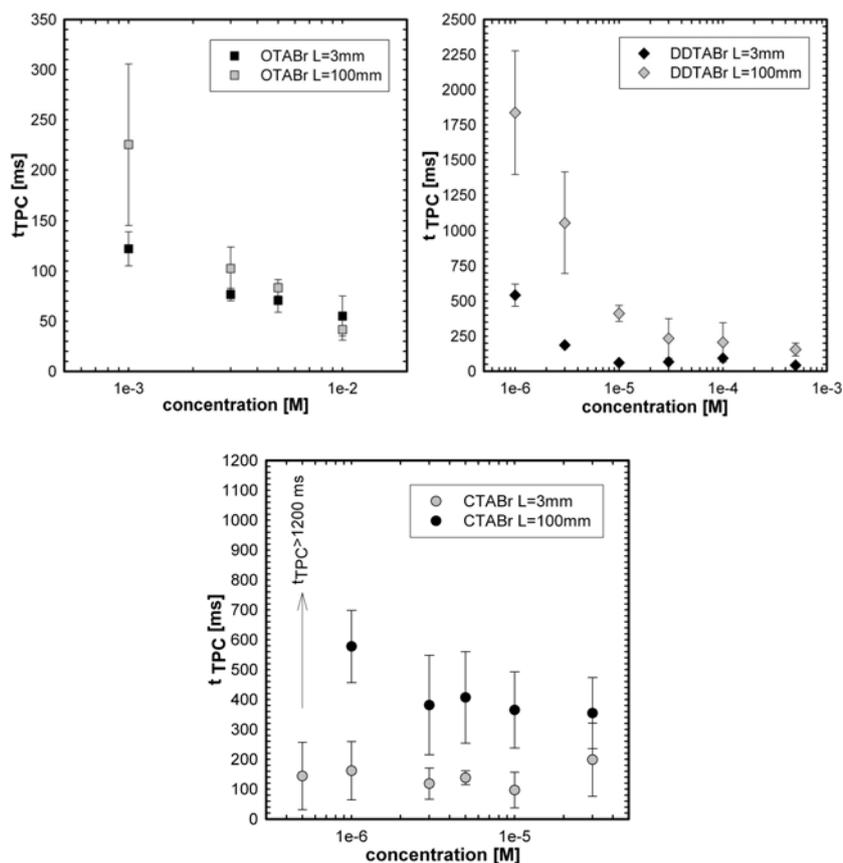


Fig. 5 Time of the TPC formation as a function of OTABr, DDTABr and CTABr concentration for the mica plate location "close" and "far"

Debacher and Ottewill (1992) reported that mica surface charge was reversed at CTABr, DDTABr and OTABr concentrations higher than ca.  $6 \cdot 10^{-6}$ ,  $6 \cdot 10^{-4}$  and  $8 \cdot 10^{-2}$  M, respectively. Fa et al. (2005), who determined so called "point of zeta reversal" (PZR) reported that the PZR values were  $2 \cdot 10^{-5}$  M for CTABr and  $1 \cdot 10^{-3}$  M for DDTABr solutions, i.e. the similar but not identical to values found by Debacher and Ottewill (1992). Despite some discrepancy regarding exact values of the n-alkylammonium bromides concentrations causing the mica surface charge reversal these literature data indicate that in our experiments there was no charge reversal of the mica plates because the upper limits of the concentrations used (see Fig. 5) were

smaller or similar as the PZR values. This comparison indicates that the TPC formation in our experiments was indeed due to the charge reversal at the bubble surface. This conclusion is supported also by the experiments (Zawala et al., 2008) in which the mica plate was immersed in OTABr solution ( $1 \cdot 10^{-3}$  M) for 30 minutes prior to measurements of the TPC formation by the colliding bubble in distilled water. As there was TPC formation so it confirms that adsorption of OTABr molecules at mica surface did not cause either electrical charge reversal or the mica surface hydrophobization, high enough to cause instability of the wetting film formed by the colliding bubble.

The results presented in Fig. 5 show clearly that the  $t_{\text{TPC}}$  values were dependent, especially at lowest solution concentrations, on location of the mica plate. Let us discuss and explain the reasons of the differences between the times of TPC formation at mica surface located "close" and "far". Locations "close" and "far" mean that the bubble covered different distances prior to the collision with mica surface and, as a consequence, the state of the adsorption layer at the bubble surface at the moment of collision was also different – this is presented schematically in Fig. 6. When the bubble was formed at the capillary orifice in surfactants solution studied there was adsorption layer formation at the growing gas/liquid interface. At the moment of the bubble detachment the adsorption coverage was uniform. As described above (Introduction) the bubble motion induces non-uniform distribution of the surfactant molecules over surface of the rising bubble and there is needed some distance for establishment of the DAL (Krzan et al., 2007). Therefore, the adsorption coverage over top poles of the bubble colliding with mica plate located "close" and "far" was different (see Fig. 6). At the location "close" the adsorption coverage was still uniform and at the moment of collision the bottom interface of the wetting film formed was positively charged. The attractive interactions between negatively charged mica surface and positively charged bubble top pole caused rupture of the wetting film formed and the TPC formation. When the mica was at the location "far" the distance covered by the bubble was long enough for the DAL formation. As a consequence, when the wetting film was formed, the bubble top pole was practically devoid of surface active molecules (see Fig. 6) Thus, at the very beginning of the film existence, the electrical charge of the bubble top pole was still negative - there were electrostatic repulsions between interfaces and the film was stabilized. When the bubble stayed motionless beneath the mica plate then the diffusion processes tended to re-establish uniform and equilibrium adsorption coverage of the cationic surfactant over entire bubble surface. The longer time of TPC formation in the case of the location "far" was caused by the fact that an additional time was needed to restore such adsorption coverage, over top pole of the bubble forming the bottom interface of the wetting film, which ensures the charge reversal from negative to positive. As diffusion kinetics depends strongly on surfactant concentration so the differences between the  $t_{\text{TPC}}$  values were the largest ones at lowest concentrations of CTABr, DDTABr OTABr solutions.

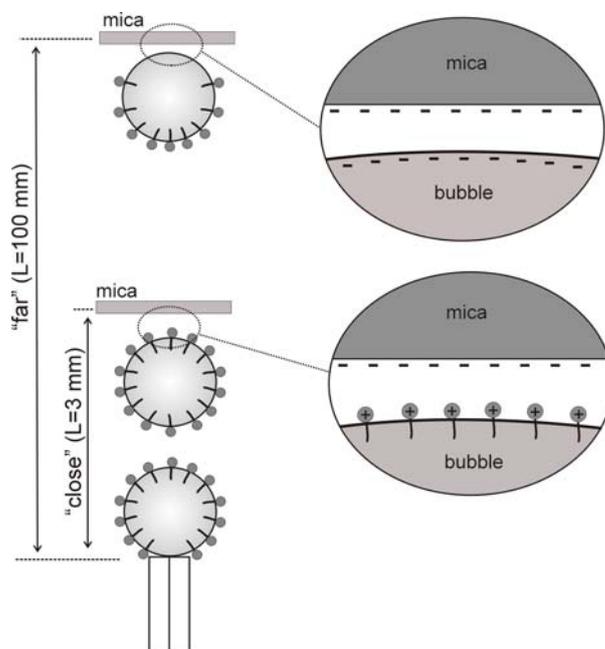


Fig. 6. Schematic presentation of the DAL formation and state of adsorption layer at solution/air interface of wetting film formed by the bubble colliding with mica plate located "close" and "far"

#### 4. Concluding remarks

It was shown that stability of the wetting film, formed at mica surface by the colliding bubble, is governed by the electrostatic interactions between the film interfaces. Both bubble and mica surface are negatively charged in distilled water. In solutions of the cationic surfactants studied (OTABr, DDTABr and CTABr) a preferential adsorption at solution/gas interface caused reversal of the bubble electrical charge from negative to positive and the wetting film formed was destabilized. It was found that time of the three-phase contact formation ( $t_{\text{TPC}}$ ) at mica surface in cationic surfactants solutions strongly depends on: (i) solution concentration and (ii) state of the adsorption layer at the bubble surface. When the distance from the capillary orifice (bubble formation point) to the mica plate was  $L=3\text{ mm}$  (location "close") then the time of the three phase contact formation ( $t_{\text{TPC}}$ ) was significantly shorter than for the location "far" ( $L=100\text{ mm}$ ). The differences between the  $t_{\text{TPC}}$  for the locations "close" and "far" were the largest at lowest concentration. Significant difference in  $t_{\text{TPC}}$  for location "close" and "far" was caused by formation of the dynamic adsorption layer (DAL) at the bubble surface. For location "far", where the adsorption coverage at the top pole of the colliding bubble was almost zero an additional time was needed to restore such adsorption coverage which caused the charge reversal from negative to positive and the film destabilization.

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**A. Niecikowska, J. Zawala, K. Malysa**, *Wpływ adsorpcji bromków n-alkilotrimeetyloamoniowych ( $C_8$ ,  $C_{12}$ ,  $C_{16}$ ) i ruchu pęcherzyka na kinetykę przyłączenia pęcherzyka do powierzchni miki*, Physicochem. Probl. Miner. Process., 47 (2011) 237-248, (w jęz. ang.)

Badano wpływ adsorpcji n-alkilotrimeetyloamoniowych bromków ( $C_8$ ,  $C_{12}$ ,  $C_{16}$ ) oraz utworzenia na powierzchni pęcherzyka, ruchem indukowanej dynamicznej architektury warstwy adsorpcyjnej (DAL), na kinetykę powstawania kontaktu trójfazowego (TPC) na powierzchni miki. Zjawiska zachodzące podczas kolizji pęcherzyka były rejestrowane przy użyciu szybkiej kamery o częstotliwości 1040 Hz. Określono wpływ stężenia roztworów i utworzenia DAL na kinetykę powstawania TPC. Wykazano, że stabilność ciekłego filmu, powstającego w trakcie kolizji pomiędzy pęcherzykiem a powierzchnią miki, jest determinowana przez siły elektrostatyczne pomiędzy granicami faz, które tworzą ciekły film. Kiedy pęcherzyk pokonywał odległość (od kapilary do powierzchni miki)  $L=3$  mm (lokalizacja „blisko”) czas powstawania kontaktu trójfazowego był znacznie krótszy, w porównaniu z odległością  $L=100$  mm („daleko”). Różnice obserwowane dla  $L=3$  mm i  $L=100$  mm wzrastały wraz ze zmniejszeniem stężenia. Przedstawiono mechanizm wyjaśniający znaczące różnice w czasie powstawania kontaktu trójfazowego dla położenia „blisko” i „daleko”.

słowa kluczowe: kontakt trójfazowy, kationowy surfaktant, zderzenia pęcherzyka, ciekły film, oddziaływania elektrostatyczne, zmiana ładunku