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Polymer / Surfactant Mixtures at the Cyclohexane-Water Interface

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Abstract

Measurements of interfacial tension of polymer/surfactant mixtures at oil-water interface are scarce. Here we report on measurements of interfacial tension of Sodium polystyrene sulfonate (NaPSS, anionic polyelectrolyte)/Benzyldimethylhexadecylammonium Chloride (BAC, cationic surfactant), Polyallylamine hydrochloride (PAACl, cationic polyelectrolyte)/sodium dodecyl benzene sulfonate (SDBS, anionic surfactant) and Polyvinyl alcohol (PVA, neutral water soluble polymer)/sodium dodecyl benzene sulfonate (SDBS) at the Cyclohexane-water interface. Individual polyelectrolytes NaPSS and PAACl are interfacial inactive. Polyvinyl Alcohol shows interfacial activity comparable to surface active agents due to its H-bonding forming capacity. For the system SDBS/PAACl, a hump in the interfacial tension was observed with a maxima in the interfacial tension. This is explained by changes of the amount of different polyelectrolyte/surfactant complex structures at the interface and their competition with complexes in the bulk. No hump nor maxima in the interfacial tension was observed for the system BAC/NaPSS but surprisingly an increase in the interfacial tension was observed. This increase is due to insufficient BAC molecules to neutralize the charges at the repeating units of NaPSS. To the best of our knowledge the finding here for the oppositely charge polymer/surfactant systems studied at the cyclohexane interface are reported for the first time.

Keywords: Polyelectrolytes; Surfactants; Polyelectrolytes/surfactant complexes.

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1. Introduction

Surfactants are chemical compounds that have dual chemical nature. They consist of two parts. One part is a carbon chain that is oil loving and the other part is a water loving one. The latter can carry a negative (anionic surfactant) or positive charge (cationic surfactant) or can be neutral. Another type of surfactants carries both negative and positive charges and are called amphoteric surfactants [1]. Surfactants are used in industrial formulations to reduce the interfacial tension between two immiscible phases, these can be gas/liquid, two liquids (e.g. water and hexane) or surface free energy between a liquid and a solid (e.g. water drop on top of a plastic plate). When a surfactant is added to water in small amounts, it migrates to the surface and accumulates there leading to a large reduction of the water surface tension. When oil is mixed with water, the oil separates itself from water and floats on it (if the density of the oil phase is less than water) forming two layers. If a surfactant is added to the two layer it accumulates at the interface between water and oil causing the interfacial tension to decrease. The surface tension of water or the interfacial tension between water and oil stops decreasing when the bulk concentration of surfactant reaches a critical concentration. This concentration is called critical micelle concentration (CMC). At this concentration two things happen, the surface tension becomes almost constant and micelles start to form. Reduction of surface and interfacial tension between immiscible phases is of great important for many industrial applications such as detergents [2], cosmetics [3], enhanced oil recovery [4], food industry Reference [5].

Polymers are chemical compounds that consists of many repeating units and have large molecular mass. When a polymer carries a charge on its repeating unit it is called polyelectrolyte. Polymers and polyelectrolytes (here we are talking about water soluble polymers) are also used in many industrial formulations. One such formulation is polymer (or polyelectrolyte) / surfactant mixtures. There are different combinations of these mixtures. The polymer can be neutral carrying no charge and the surfactant can be charged or both can be charged (oppositely charged polyelectrolyte/surfactant mixture). These latter systems display interesting behavior. They form different interesting structures [6-9] such as micelle-like aggregates, gels, complexes and coacervates. These formulations and structures are important for the manufacturing of cosmetic products, coatings and drug delivery [10-13]. They are also used to stabilize dispersed systems like foams and emulsions [14-16]. The behavior of these complex mixtures is determined by the interaction between polymer (polyelectrolytes) chains and surfactant molecules or micelle [17-20]. There are many aspects and details on the nature of the complex interactions and the parameters that control the interactions in these systems that cannot be discussed in details in this article and the reader is referred to the excellent review by Guzmán *and his colleagues* [21]. Polymer (polyelectrolyte) / surfactant mixtures have been studied by many authors, both the solution properties [22-24] and to a less degree the surface properties [25-27]. The latter studies concentrated on the weakly interacting systems where one of the component is neutral. For strongly interacting mixtures where both the polymer and the surfactant are oppositely charged studies are scarce Reference [21]. One of the systems in the strong interaction category that has been studied is Poly(dimethyldiallylammonium chloride / Sodium Dodecyl Sulfate system (SDS) [28, 29]. In this latter system a cliff edge, maxima and minima, in the plot of surface tension versus the logarithm of SDS concentration was found [30, 31]. This behavior was attributed to the formation of highly surface active species at different surfactant concentrations. These studies were performed at the air/aqueous solution interface. Studies of polyelectrolytes / surfactants mixtures at the oil/aqueous solution interface are lacking.

In this report we present data on the surface tension as a function of surfactant concentration of Sodium polystyrene sulfonate (NaPSS, anionic polyelectrolyte)/Benzyldimethylhexadecylammonium Chloride (BAC, cationic surfactant), Polyallylamine hydrochloride (PAACl, cationic polyelectrolyte)/sodium dodecyl benzene sulfonate (SDBS, anionic surfactant) and Polyvinyl alcohol (PVA, neutral water soluble polymer)/sodium dodecyl benzene sulfonate (SDBS) at the Cyclohexane / water interface. These type of measurements are more difficult than those at the air/water interface that we have reported recently [32] and are presented for the first time for these systems. The data obtained in this work would definitely make a humble contribution in the field of interest.

2. Materials and Methods

Our experimental method was described in details elsewhere [32]. Briefly, all glassware used were cleaned by boiling in water bath for 90 min and then rinsed with distilled water to avoid any possible contamination. Surfactants, polyelectrolytes and salts used in this work were supplied by Sigma-Aldrich and of high purity. The polyanion NaPSS has a weight average molar mass of M_w = 70,000 g/mol. The polycation PAACl has an average weight average molar mass of M_w = 17,500 g/mol. Successive dilution method was used to prepare all solutions. All solutions were left in dark for 24 hours before measurements.

Interfacial tension was measured using an advanced K100 – MK3 tensiometer from (Krüss, Germany) using the plate method. This tensiometer is fully automatic and computerized, with high resolution (± 0.01 mN/m). It is fitted with a force sensor that is highly accurate and does not need to be recalibrated. The plate was heated with flame until it turned red for several seconds before every measurement. The surface tension of pure water was measured with an average value of 71.92 mN/m which agrees well with the reported value of ref. [1]. All measurements were conducted at 20^{o}C.

3. Results and Discussion

The bare cyclohexane-water interfacial tension was measured to be 49.6 mN/m which is in good agreement with the value of 48.92 mN/m reported by Viviana *and his colleagues* [33]. Figure 1 shows the aqueous solution of SDBS-air and the aqueous solution of SDBS-cyclohexane interfacial tension at 293 K.

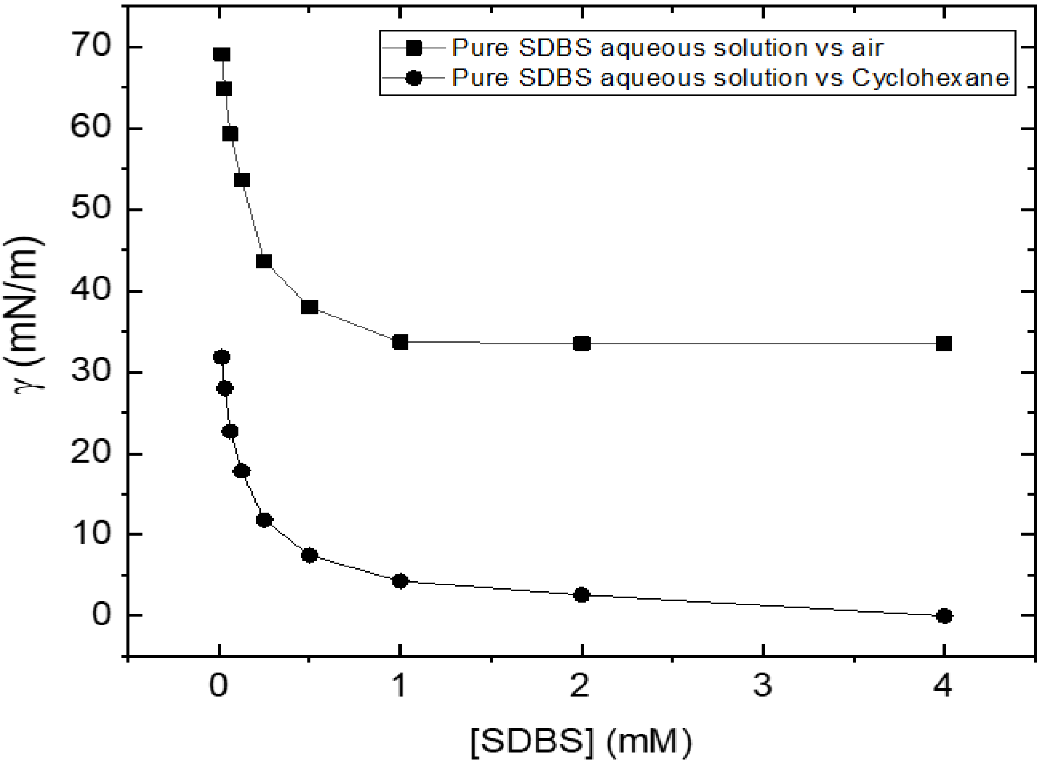

Figure 1: Aqueous solution of SDBS-air (solid squares) and aqueous solution-cyclohexane of SDBS (solid circles) interfacial tension at 293 K. The lines are drawn to guide the eye

As is well known, the surface and the interfacial tension decreases with increasing the anionic surfactant SDBS concentration. The critical micelle concentration of SDBS was determined to be 0.61 mM. The reported values for the CMC of SDBS are at 303 K or higher and no comparison can be made here. Aqueous solution of BAC-air (solid squares) and aqueous BAC solution-cyclohexane (solid circles) interfacial tension at 293 K are shown in figure 2. The CMC for cationic surfactant BAC was determined to be 0.

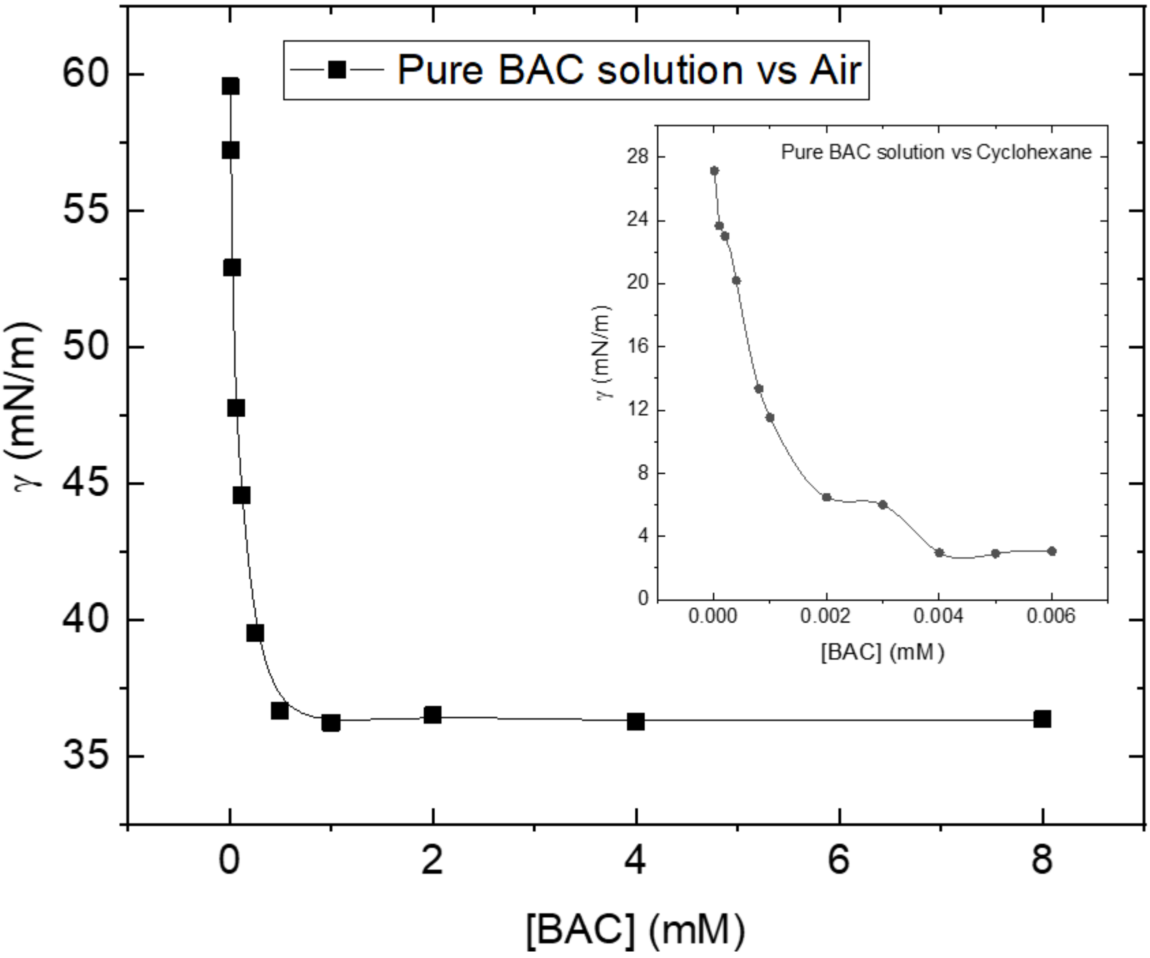

Figure 2: Aqueous solution of BAC-air (solid squares) and (inset) aqueous BAC solution-cyclohexane (solid circles) interfacial tension at 293 K. The lines are drawn to guide the eye

The value of CMC determined by us is in reasonable agreement with that of George *and his colleagues* [34]. In the inset figure, note that the concentration of BAC is very low, which implies that the cationic surfactant shows interfacial activity at very low concentration. This is good for practical applications since a tiny amount is needed to obtain surface activity. Another hint is that the saturation of the cyclohexane-water interfacial tension occurs two times at two different concentrations. Note that these experiments were repeated more than one time and the same behavior was found.

Figure 3 shows the cyclohexane-water interfacial tension with added either NaPSS (solid squares) or added PAACl (solid circles). The outcome of these measurements is that there is very weak activity of the individual polyelectrolytes at the interface studied.

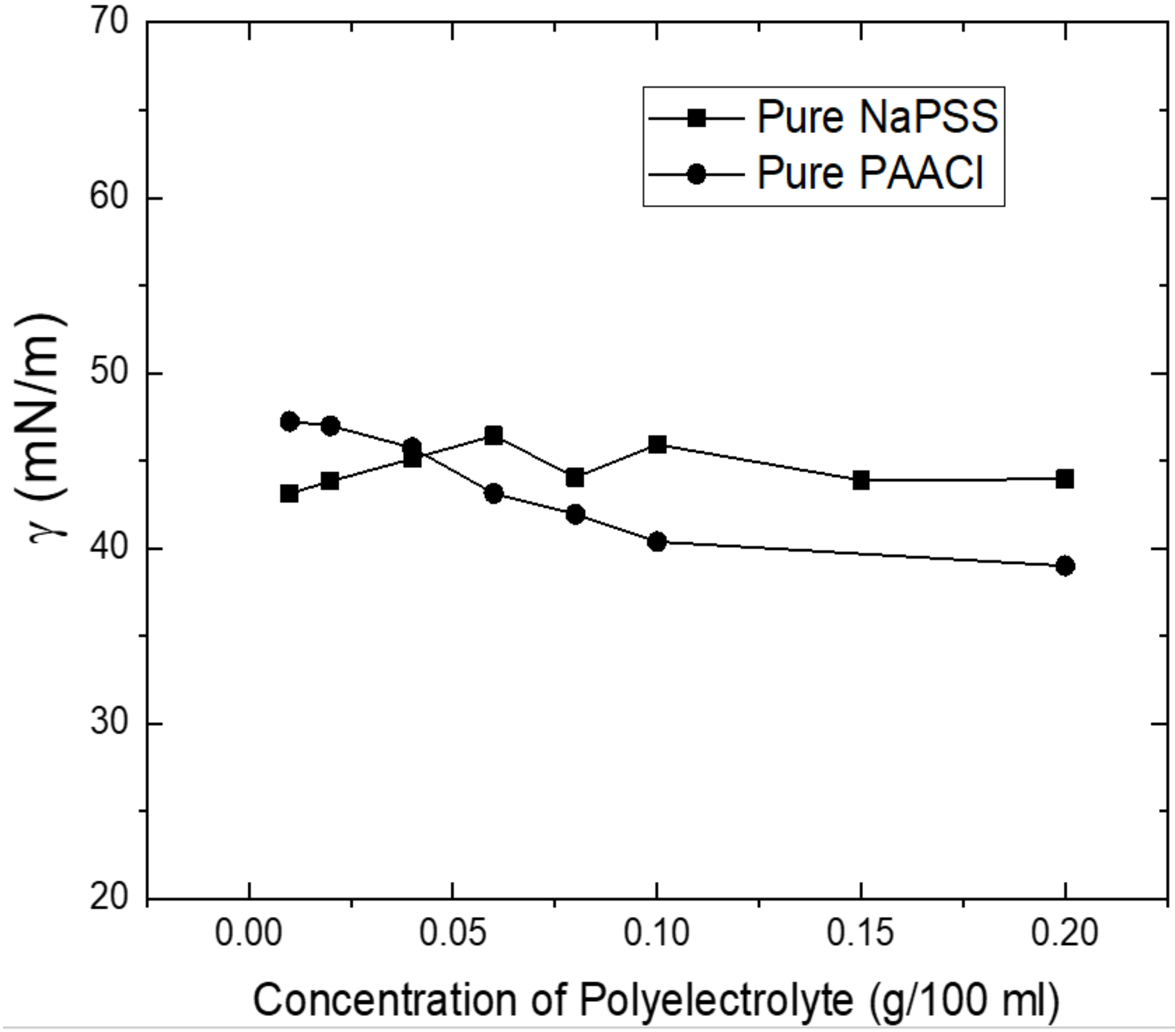

Figure 3: Cyclohexane-water interfacial tension with added either NaPSS (solid squares) or PAACl (circles). The solid lines are drawn to guide the eye

Figure 4 shows the surface tension of PVA at the air-water interface (solid squares) and at the cyclohexane-water interface (solid circles) . As is clear form figure 4, this water soluble polymer displays surface activity similar to that of surface active agents. At the air-water interface, measurements of the surface tension of this neutral polymer are well known and reported [35]. However, at the cyclohexane-water interface measurements are lacking. The surface activity of this water soluble polymer (solute) stems from the hydroxyl group (-OH) on its repeating unit. This functional group is capable of forming hydrogen bonding with water molecules (the solvent). Both water and PVA make mutual interaction and migration to the surface. Now because of their different structures, PVA

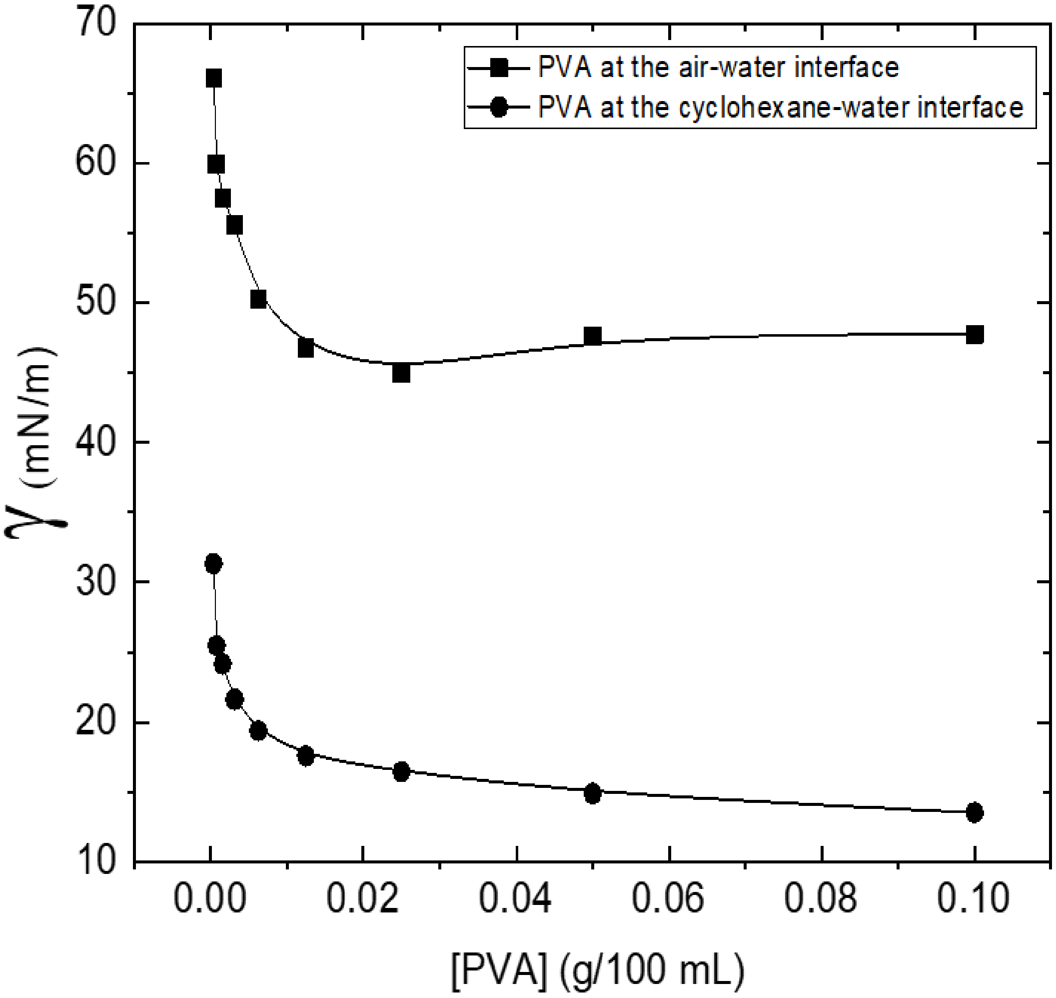

Figure 4: Surface tension PVA aqueous solution at the air-water (solid squares) and at the cyclohexane interface (solid circles). The lines are drawn to guide the eye

exerts different field of attractive forces. In this situation there will be a state of high free energy in the bulk and the PVA will migrate to the surface where there is a lower state of free energy comparing to the bulk. This is the main cause of its surface activity. Figure 5 shows the interfacial tension of 0.02 g PVA in solution as a function of SDBS concentration (solid circles) and 0.2 g PVA in solution (solid triangles) at the cyclohexane-water interface plotted together with the interfacial tension of pure SDBS solution at the same interface (solid squares) for comparison. The solid lines are drawn to guide the eye. Clearly the presence of the anionic surfactant SDBS lower the interfacial tension of solution at low SDBS concentration from 35 mN/m to about 12 mN/m for 0.02 g PVA to 9 mN/m for 0.2 g PVA. However, at higher SDBS concentration (above the CMC of SDBS) there is almost no further decrease in the interfacial tension. Note that PVA is neutral polymer and does not make strong interaction with SDBS (Weak interaction).

The interracial tension of different PAACl amounts in solution of 0.001 g/100ml (solid circles), 0.003 g / 100 ml (solid triangles) and 0.005 g/100 ml (solid stars) as a function of SDBS concentration at the cyclohexane-water interface, plotted together with free SDBS solution (solid squares) is shown in figure 6. The solid lines are drawn to guide the eye. In figure 6, the interfacial tension of SDBS/PAACl system displays a hump as a function of

SDBS concentration. The maximum of the hump shifts to higher SDBS concentration as the amount of PAACl increases in solution. Staples and his colleagues [31] studied the system sodium dodecyl sulfate (SDS)/poly(dimethyldiallyammonium chloride) at the water-air interface and observed a hump in the surface tension plot against SDS concentration. Their system is anionic surfactant/cationic polyelectrolyte meaning it is an oppositely charges surfactant/polymer system. Unfortunately, there are no studies at the oil/water interface of this complex system to compare with. It seems that a hump in the surface tension behavior in these systems is not a rare phenomenon. To explain the origin of a hump in our studied complex system we have to rely on neutron reflection study of [36] where they studied the adsorption of the cationic surfactant alkyltrimethylammonium bromide (CTAB)/sodium polystyrene system and found a hump in the surface tension behavior as a function of CTAB concentration and chain length. From their reflectivity data they resolved three different complex structures of their polyelectrolyte/surfactant systems.

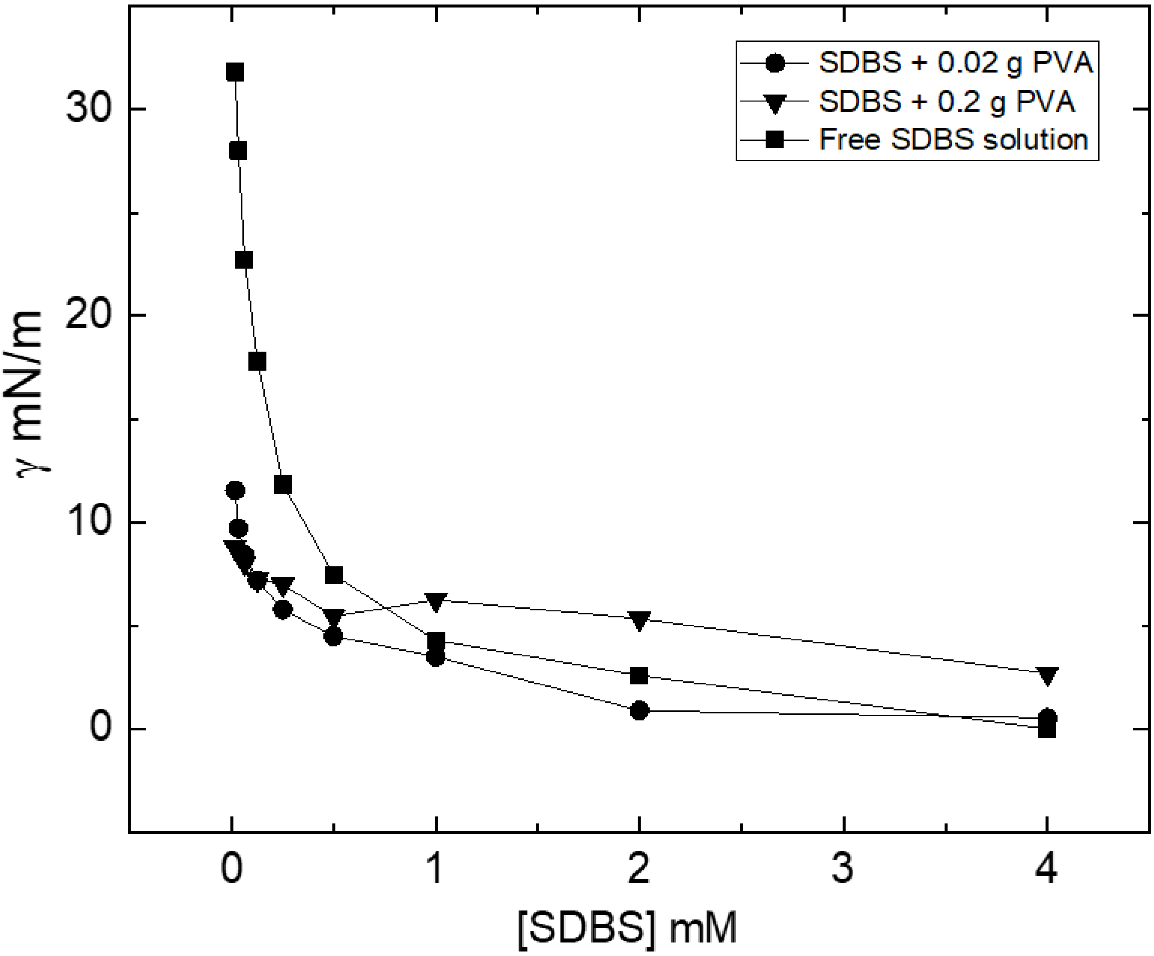

Figure 5: Interfacial tension of 0.02 g PVA in solution a function of SDBS concentration at the cyclohexane-water interface (solid circle) and 0.2 g PVA in solution (solid triangles) plotted together with the interfacial tension of free SDBS solution for comparison

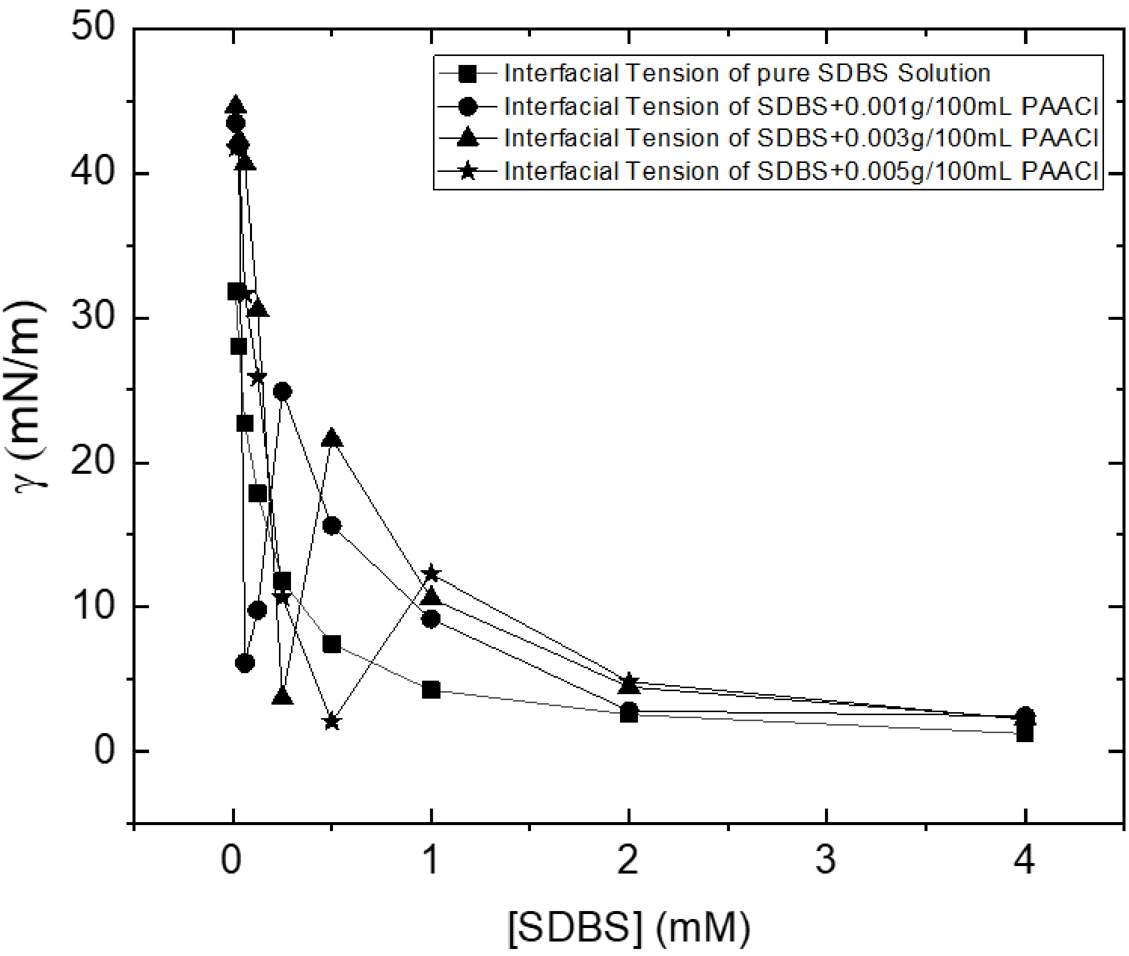

Figure 6: Interfacial tension of different PAACl amounts in solution of 0.001 g/100ml (solid circles), 0.003 g / 100 ml (solid triangles) and 0.005 g/100 ml (solid stars) as a function of SDBS concentration plotted together with free SDBS solution (solid squares). The solid lines are drawn to guide the eye

The first of these structures is a monolayer of surfactant molecules attached to the polymer backbone and complex is present all the time. The second structure is a bilayer of surfactant molecules attached to the polyelectrolyte and the third structure is surfactant aggregates with the polymer in the bulk. It seems to us that the fluctuation in our interfacial tension data and the observed hump must originate from changes of the amount of these different structures at the interface and their competition with complexes in the bulk. Guzmán [21] has mentioned that the maxima in the surface tension of these complex systems data is due to insufficient equilibration time. In our study we have equilibrated our samples for 24 hours and studied the interfacial tension as a function of time as shown in figure 7 which shows the interfacial tension of the PAACl/SDBS complex at the cyclohexane/water interface as a function of time and at two different SDBS concentration of 0.065 mM and 0.25 mM. The measurements of interfacial tension are carried out after 24 hours' equilibration time. The interfacial tension in figure 7 saturates after about 20 minutes and stays constants for about 40 minutes. This shows that the interfacial tension we have

measured is indeed an equilibrium one and the observed hump in our interfacial tension graph to be true effect and not due to insufficient time of equilibration.

Figure 8 (a) shows the change in PAACl concentration as a function of the concentration of SDBS at the minimum (critical aggregation concentration) of the interfacial tension extracted from figure 6 above. Figure 8 (b) shows the maximum of the interfacial tension as a function of PAACl concentration extracted from figure 6.

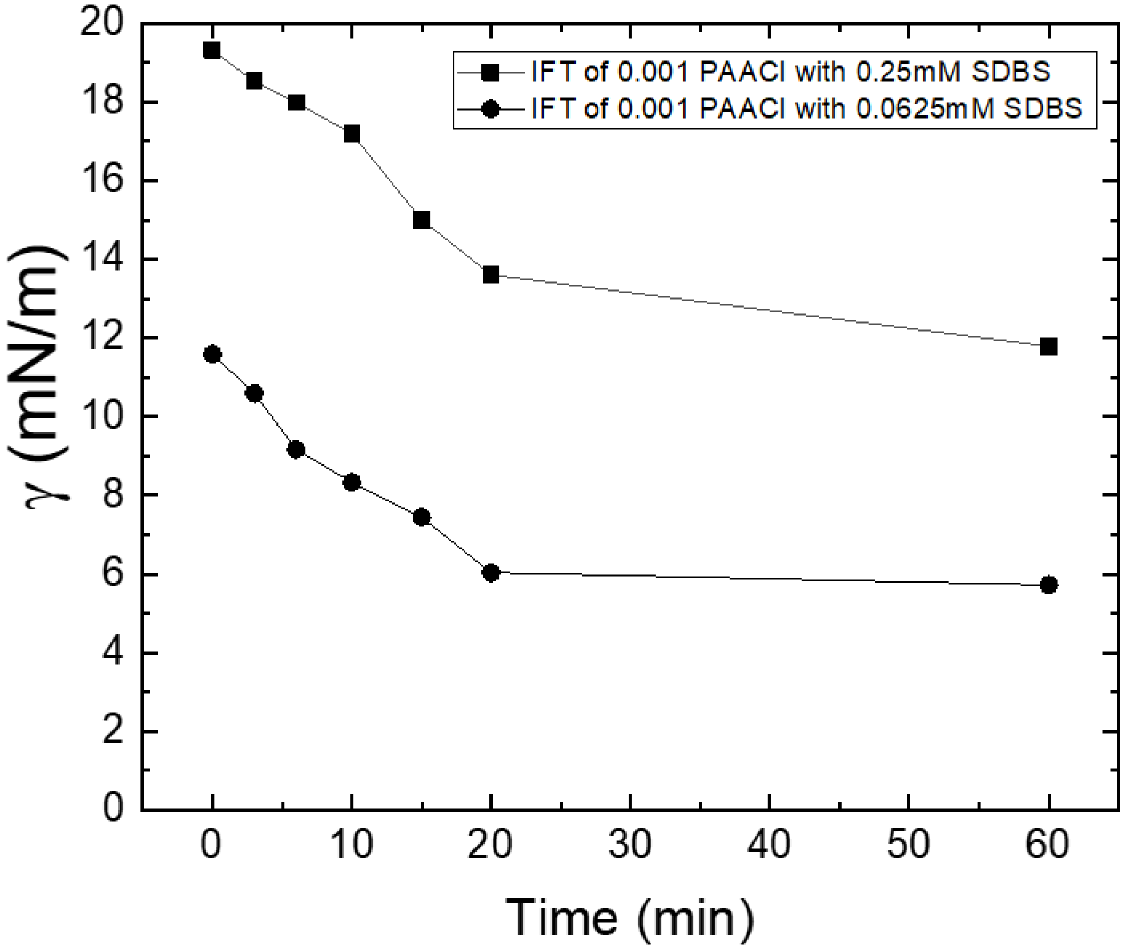

Figure 7: Interfacial tension (IFT) of the PAAl/SDBS complex at the cyclohexane interface as a function of time at two different surfactant concentration of 0.25 mM (solid squares) and 0.0625 mM (solid circles)

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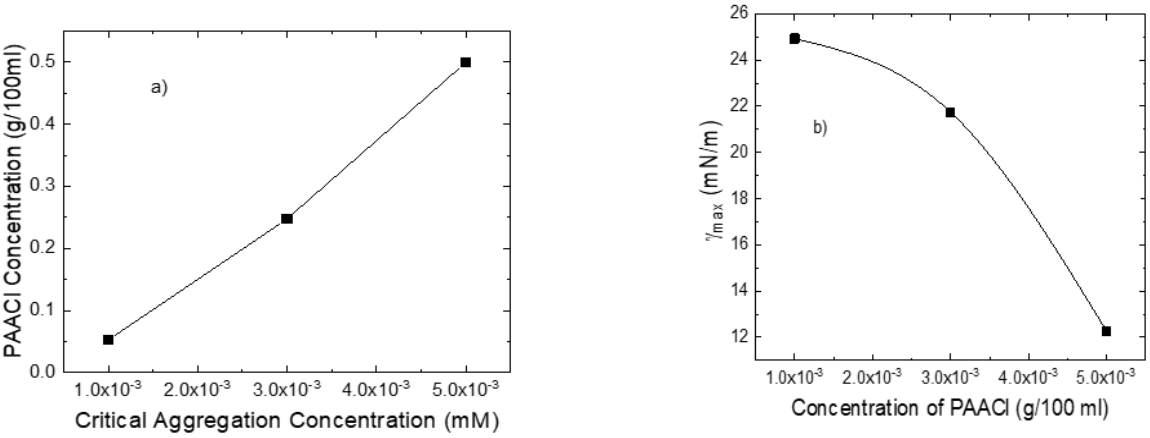

Figure 8: (a) PAACl concentration as a function of the concentration at the minimum of interfacial tension (CAC). (b) the interfacial tension at the maximum as a function of PAACl concentration

It is clear form figure 8 (a) that as the polyelectrolyte concentration increases in solution the critical aggregation concentration (CAC) increase as well because more association between the surfactant molecules and the polyelectrolyte chains occurs. The minimum in the interfacial tension at CAC is due to the presence of a surface complex (associated polyelectrolyte/surfactant molecules). It seems that this complex has high affinity to adsorb at the interface than the surfactant itself and thus impart high surface activity. In figure 8 (b), γ_{max} corresponds to a surfactant monolayer at the interface where the polyelectrolyte is associated with the surfactant micelles in the bulk. The conclusion that can be drawn here is that the polyelectrolyte/surfactant complex is more interfacial active than the surfactant itself for this particular system.

The interfacial tension of the BAC/NaPSS system at the cyclohexane-water interface for two different NAPSS amount in solution is shown in figure 9 below plotted together with the interfacial tension of pure BAC solution at the same interface. This system consists of cationic surfactant and anionic polyelectrolyte. No maxima of minima of the interfacial tension is observed for this system. Note the very low concentration of the cationic BAC surfactant. This surfactant is difficult to study because it shows high interfacial activity at very low concentration. The surprising effect of this system is that the NaPSS upon association with the surfactant molecules increases the interfacial tension. This implies that the NaPSS polyelectrolyte drags the surfactant from the interface to the bulk causing less surface activity. These experiments where repeated and the same effect was found. The NaPSS is a polyanion and not interfacial active and therefore stays in the bulk. It seems that because the cationic BAC surfactant shows high surface activity at very low concentration, there is no complete association between its molecules and the NaPSS chains and thus there are not enough surfactant molecules to neutralize the negative charges at the backbone of the anionic polyelectrolyte NaPSS. No such effect was previously reported for this particular polyelectrolyte/surfactant system and thus the findings here constitute a challenge for neutron and x-ray reflectivity measurements.

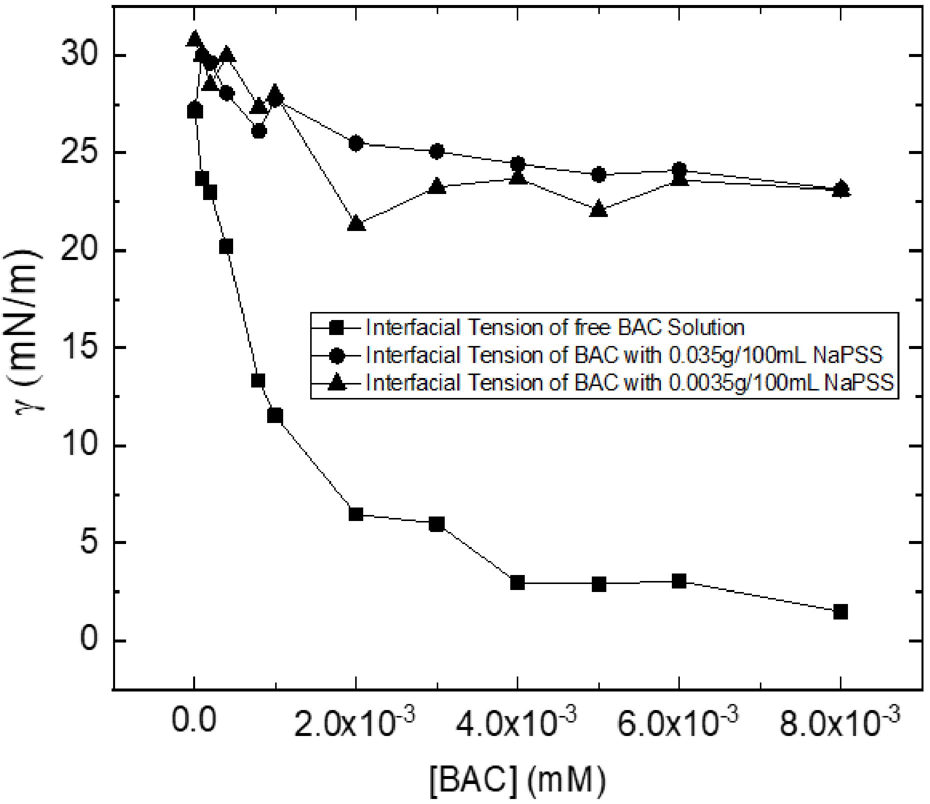

Figure 9: Interfacial tension of the BAC/NaPSS system at the cyclohexane-water interface as a function of BAC concentration for two different NaPSS amounts of 0.0035 g (solid triangles) and 0.035 g (solid circles) plotted together with the interfacial tension of pure BAC solution (solid squares). The lines are drawn to guide the eye

4.Conclusions

For the oppositely charged SDBS/PAACl (anionic surfactant/cationic polyelectrolyte) at the cyclohexane-water interface a hump was observed in the interfacial tension as a function of SDBS concentration with a minimum in interfacial tension at the critical aggregation concentration. This minimum in interfacial tension indicates the presence of a SDBS/PAACl complex at the interface with high surface activity. The maximum in the interfacial tension for this particular system corresponds to a surfactant monolayer at the interface where the PAACl molecules are engaged in the bulk solution. For the system BAC/NaPSS (cationic surfactant/anionic polyelectrolyte) there is a surprising increase in the interfacial tension comparing with that of pure BAC at the interface. This is because BAC surfactant shows high surface activity at very small concentration ~10^{-6}M which is five order of magnitude less than its CMC. This causes the interfacial inactive anionic polyelectrolyte NaPSS to drag BAC molecules to the solution leading to higher interfacial tension than that of the pure surfactant at the interface. The neutral polymer PVA shows interfacial activity at the cyclohexane interface comparable to that of surface active agents due to its hydrogen bonding formation capability. When the latter is mixed with SDBS, the onset of the interfacial tension value is reduced

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