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Conference Paper

Polymers at Interfaces — Studies on Contact Wetting of Polymers*

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A review is given of recent work in the author's laboratory concerning *(1)* phenomena of contact wetting in a four phase system: solid/liquid/vapor/solid, and the formation of liquid bridges between solid surfaces, and *(2)* contact wetting of polymer surfaces with surfactant solutions.

INTRODUCTION

The knowledge of wetting properties of macromolecular solids is of primary importance for understanding some phenomena occuring in inhomogeneous or multiphase polymer systems as well as understanding the physical principles of practical applications connected with them. Hence, the investigations of the wettability of polymers can give insight into technical adhesion, reinforcement, structure and properties of composite materials, compatibility, crystallization *etc.* In addition, it represents a relatively simple experimental way to get general information on molecular interactions at the solid/liquid interface.

Solid-phase polymers have low-energy surfaces which means that common liquids generally do not spread over such solids and that a contact angle configuration occurs in the polymer/liquid/second fluid three-phase system. This contact angle configuration will be called contact wetting.

It is not intended to give here a general survey on the whole topic for excellent review articles and monographs are available[1-4]. Also our earlier results have recently been summarized in a book[5]. The purpose of the present paper is to report on some unpublished work recently carried out in our laboratory in two directions: first, the phenomenon of contact wetting in solid/liquid/vapor/solid four-phase systems, or more precisely the formation and rupture of liquid bridges between two solid surfaces, and second, the contact wetting of polymers with surfactant solutions.

Liquid Bridges

Very little attention has been paid so far to adhesional wetting involving one liquid and two solids, *i. e.* to the phenomenon where adhesion between two solid surfaces is brought about by a liquid bridge. The main interest was the calculation, on a theoretical basis, of the capillary force acting between solid spheres held together by a liquid bridge[6-8]. Recent works of Padday[9,10]

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deal with the shape of the liquid menisci between a solid plane and a sphere, but no experimental data are available as to the conditions of the formation and rupture of the liquid bridges between solids.

In order to continue our previous work[11,12], the purpose of the present study was to carry out experiments with simple model systems involving different types of solids and liquids. Both high-energy and low-energy solid surfaces as well as pure liquids and aqueous solutions of surfactants have been used.

EXPERIMENTAL

When a small drop of a non-spreading liquid L is placed on a horizontal low-energy solid surface S_1 and a second solid surface S_2 of any kind is brougth into contact with the apex of the drop, a liquid bridge is formed with a capillary surface which reaches its equilibrium shape in some hundredths of a second, as shown in Fig. 1 (a).

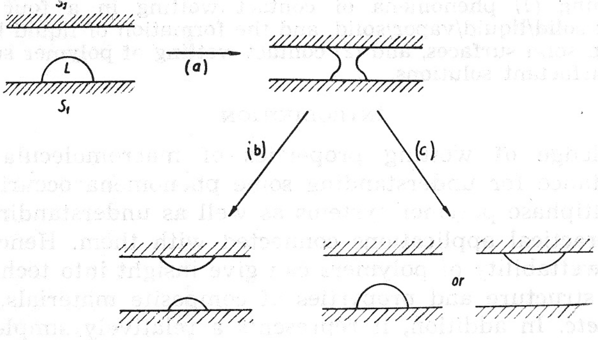


Fig. 1. Formation of a liquid bridge *(a)* and subsequent separation of the solid surface with *(b)* and without *(c)* drop distribution (schematically).

When the solid surfaces are separated again, the bridge becomes thinner, having at each distance a well-defined shape. At a certain distance, which depends on the nature of the system and the drop mass, either rupture occurs, resulting in a distribution of the liquid between the two solid surfaces, [see Fig. 1 (b)], or the whole mass of the drop remains adhering to one of the solids, [see Fig. 1 (c)]. As shown earlier[12], a very small droplet is always formed from the liquid filament, appearing immediately before rupture takes place.

Because of its general occurrence, we were mainly interested in that case of rupture which is followed by distribution. We have determined *drop distribution curves* representing the mass of L adhering to S_2 after rupture (m_2) as a function of the total drop mass involved (m). The experimental technique was described previously[12]. In some cases, the capillary force exerted by the liquid bridge against the separation of the solid surfaces was also measured as a function of the distance between the solids. For this purpose, a torsion balance and a cathetometer were used. Liquid surface tension was measured with the pendant drop method and contact angles were determined by means of a goniometric device described elsewhere[13].

RESULTS AND DISCUSSION

(a) Drop Distribution

Drop distribution curves for pure liquids can be seen in Figs. 2 and 3, and those for solutions in Figs. 4 and 5. The characteristic shapes of two types of such curves are shown in Fig. 6, one of them corresponding to a drop-mass-dependent distribution, and the other one to a drop-mass-independent distribution. The meaning of this distinction is obvious from Fig. 6.

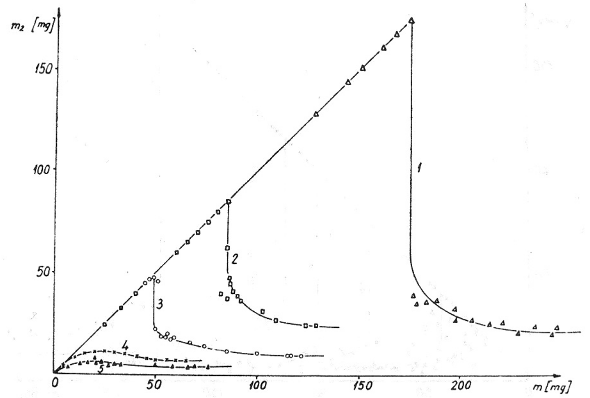


Fig. 2. Drop distribution curves for pure liquids between paraffin (S_1) and silica (S_2). *1,* water, *2,* glycerol, *3,* ethyleneglycol, *4,* aniline, *5,* methylene iodide.

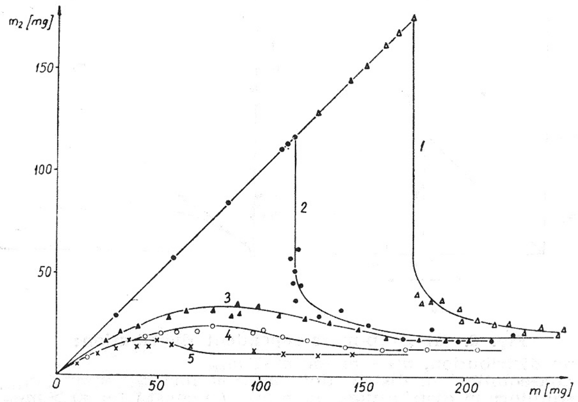


Fig. 3. Drop distribution curves for water between low-energy surfaces (S_1) and silica (S_2). *1,* paraffin, *2,* poly(tetrafluoro-ethylene), *3,* polyethylene, *4,* poly(vinyl-chloride), *5,* poly(methylmethacrylate).

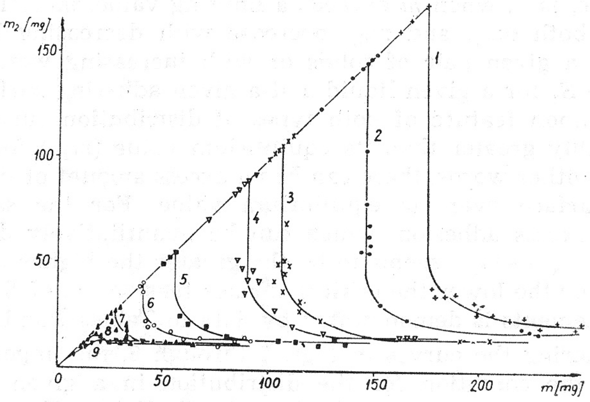


Fig. 4. Drop distribution curves for aqueous *n*-butyl alcohol solutions of concentration *c* (mmole/l) between paraffin (S_1) and silica (S_2). *1,* c = 0, *2,* c = 17, *3,* c = 34, *4,* c = 68, *5,* c = 135, *6,* c = 270, *7,* c = 405, *8,* c = 540, *9,* c = 677.

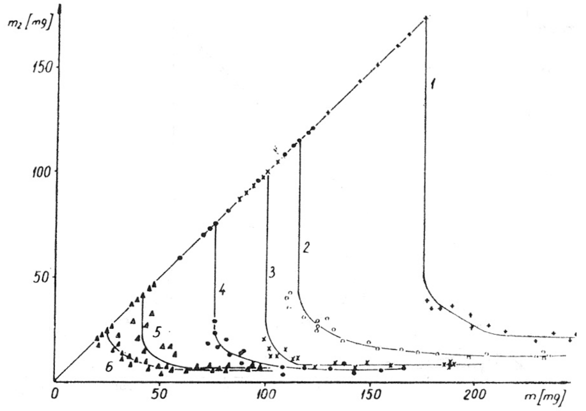
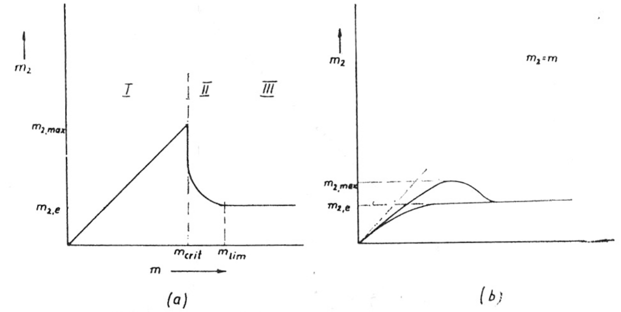


Fig. 5. Drop distribution curves for aqueous sodium dodecyl sulfate solution of concentration c (mmole/l) between paraffin (S_1) and silica (S_2) *1,* c = 0, *2,* c = 1, *3,* c = 2.5, *4,* c = 4.7, *5.* c = 6, *6,* c = 8.



(a) (b)

Fig. 6. (a) Drop-mass-dependent distribution:
I no distribution, $m_2 = m$ for $m < m_{crit}$
II non-equilibrium distribution, $m_2 > m$ for $m_{crit} < m < m_{lim}$
III equilibrium distribution, $m_2 = m_{2,e}$ (= *const.*) for $m > m_{lim}$.
(b) Drop-mass-independent distribution: $m_2 < m$ for all values of m.

No matter of what type of distribution takes place, m_2 reaches a constant equilibrium value, $m_{2,e}$, when m exceeds a limiting value, m_{lim}. The measuring data show that both $m_{2,e}$, and m_{lim} decrease with decreasing liquid surface tension, γ_{LV}, for a given pair of solids or with increasing wettability of the substrate surface S_1 for a given liquid and a given adhering surface S_2.

Another common feature of both types of distribution curves is that m_2 can be considerably greater than its equilibrium value ($m_{2,e}$) for values of m less than m_{lim}. In other words, there can be an excess amount of liquid adhering to the upper surface over its equilibrium value. For the same adhering surface S_2, this excess adhesion, which can be quantitatively described with the difference $m_{2,max} - m_{2,e}$, seems to be the greater the higher the liquid surface tension and/or the lower the critical surface tension, γ_c, of S_1. The validity of the above statements is demonstrated by data in Tables I and II.

When considering the curves in Figs. 2 through 6, the important question arises: What is the condition for the distribution in a given system to be drop-mass-dependent or drop-mass-independent? Taking into account the competitive character of the phenomenon, *i. e.* the fact that the adhesion

TABLE I

Some Characteristic Data of Drop-Distribution Curves for Different Liquids

S_1 = paraffin; S_2 = silica

System No	Liquid	λ_{LV} dyne/cm	$m_{2,e}$ mg	$m_{\lim}$ mg	$m_{2,\max}—m_{2,e}$ mg
1	Water	72,5	21	240	155
2	Glycerol	66,6	24	130	61
3	Ethylene glycol	47,8	10	110	38
4	Methylene iodide	52,0	4	60	2
5	Aniline	42,9	7	40	4

TABLE II

Some Characteristic Data of Drop-Distribution for Different Substrate Surfaces

L = water; S_2 = silica

System No	Substrate solid (S_1)	γ_c of S_1 dyne/cm	$m_{2,\max}—m_{2,e}$ mg
6	Polytetrafluoroethylene	18	155
7	Paraffin	25	98
8	Polyethylene	31	14
9	Polyvinyl chloride	39	11
10	Polymethyl methacrylate	43	6

energy of any liquid to any solid must always have a finite value according to the Young and Dupré equation, it is obvious that in our case the difference of the two adhesion energies rather than the individual ones involved must be responsible for both the nature (or type) of the distribution and the characteristic values of m_2 as discussed in detail above.

It is obvious that when computing this difference the advancing contact angle of the liquid on the adhering surface, $\overrightarrow{\Theta}_2$, and the receding contact angle of it on the substrate surface, $\overleftarrow{\Theta}_1$, have to be taken into account. The net work of adhesion, W_{2L1}, (»sticking-up energy«) is thus defined as

$$W_{2L1} = \gamma_{LV} (\cos \overrightarrow{\Theta}_2 - \cos \overleftarrow{\Theta}_1). \tag{1}$$

The calculated values for W_{2L1} as shown in Table III for the systems corresponding to the curves in Figs. 2 and 3 clearly demonstrate that there is a limiting value of the sticking-up energy at which a change in type of the distribution occurs. This value has been found for both groups of systems to be about 58 ergs per cm^2 showing the predominant role played by the adhering upper surface (which was the same in the above mentioned cases) in the competition.

Not only the type of distribution is governed by the value of W_{2L1} but also the excess adhesion and the equilibrium adhesion are dependent on it.

TABLE III

Sticking-up Energy (W_{2L1}) *and Type of Distribution for the systems 1 to 10 of Tables I and II*

System No	W_{2L1} ergs/cm^2	Type of distribution*
1	78.1	a
2	58.4	a
3	25.8	b
4	15.7	b
5	13.6	b
6	57.2	a
7	78.1	a
8	47.1	b
9	26.2	b
10	10.0	b

* a = drop-mass dependent; b = drop-mass-independent.

The latter relation is demontrated in Fig. 7 showing that $m_{2,e}$ increases continuously with increasing W_{2L1} and seems to tend toward a plateau above a certain value of W_{2L1}, the latter being approximately in the same range which corresponds to the chage-in-type of the distribution.

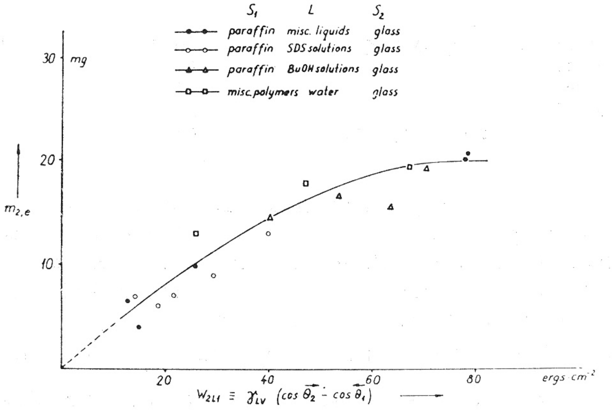


Fig. 7. Dependence of $m_{2,e}$ upon W_{2L1} (see text).

It is, however, to be stressed that in a more correct treatment the kinetic character of the phenomenon must be taken into account, too. As some preliminary results obtained when using a high-speed kinematografic technique showed, both the advancing and the receding contact angles can, at the moment of the rupture of the liquid bridge, have dynamic values which depend upon the rate of separation and are completely different from those measured under static conditions. In addition to that, the actual dynamic values of the liquid surface tension itself have to be considered instead of the static ones. So far, the static values for both the contact angles and the liquid surface tensions had been used only when calculating the sticking-up energy according to Eq. (1). The use of static values is essentially incorrect but can still work as a first approximation when pure liquids and non-adsorbing solids are involved. For solutions of surface active substances this approximation can be used only when the rate of separation is very small. Therefore we have carried out the measurements under this condition.

(b) Capillary Force Measurements

Force measurements data could evidently give information on energy relations only before the rupturing of the liquid bridge but they cannot be, at least in a simple manner, correlated to W_{2L1}. In the case of a still existing liquid bridge the variation of the pulling force with the separation distance is in connection with the force acting at the solid/liquid contact line, *i. e.* with the wetting tension, $\gamma_{SV} - \gamma_{SL}$. In order to establish experimental conditions as simply as possible, only systems consisting of a pure liquid and two solids of the same nature were investigated. Even in this case, reproducible results were obtained only with solids of neither too high nor too low-energy surfaces, *e. g.* with polar polymers, such as poly-(methyl-methacrylate), PMM, or polyamides.

The following picture served as a starting point for calculating the wetting tension from force *(F) vs.* separation distance *(a)* data. When increasing the distance from a_1 to a_2, the mechanical work W is done on the system, as given by

$$W = \int_{a_1}^{a_2} F \, da \tag{2}$$

As a result, both the potential energy (E_{pot}) and the surface energy (E_{surf}) of the system will be changed. In equilibrium the relation

$$W = \Delta E_{pot} + \Delta E_{surf} \tag{3}$$

must be obeyed, where Δ denotes the corresponding changes.

It is clear that

$$\Delta E_{pot} = m \, g \, (h_2 - h_1) \tag{4}$$

where m is the mass of the liquid bridge, g is the acceleration due to gravity; h_2 and h_1, resp. are the height coordinates of the mass center of the system at the corresponding separation distances.

For the second term of Eq. (3) it can be written that

$$\Delta E_{surf} = \gamma_{SV} \, \Delta A_{SV} - \gamma_{SL} \, \Delta A_{SL} + \gamma_{LV} \, \Delta A_{LV}. \tag{5}$$

Here ΔA denotes the change in the interfacial area and γ the interfacial energy between the corresponding phases as shown by the indices S (solid), L (liquid) and V (vapour). Again, the changes of A refer to different separation distances.

When comparing eqs. (2) through (5) and taking into account that obviously $\Delta A_{SV} = \Delta A_{SL}$, the obtained relation for the wetting tension is

$$\gamma_{SV} - \gamma_{SL} = \frac{W - [\gamma_{LV} \, \Delta A_{LV} + m \, g \, (h_2 - h_1)]}{\Delta A_{SL}}. \tag{6}$$

It can be seen that all quantities on the right side of eq. (6) are experimentally available and thus the value obtained for the wetting tension from eq. (6) can be compared with that given by the Young equation, using measured γ_{LV} and Θ data. Because eq. (6) does not contain any contact angle data, the coincidence of the values as obtained in these two ways can be considered as a direct experimental proof for the validity of the classical Young relation.

The basic principle of the calculation is as follows. *W* can be obtained by the graphical integration of the *F*, *a*-curves which were found to fit a straight-line relationship for the systems so far investigated. Also A_{SL} was easy to estimate from enlarged photos by taking into account the cylindrical symmetry of the liquid bridge which results in a circular shape of the solid/liquid/vapour contact line. The values of ΔA_{LV} as well as those of *h* were calculated in a slightly more complicated manner, namely, by approximating the liquid bridge as being composed of a great number of truncated cones lying parallel to each other. The calculation was made by a computer.

The usefulness of the method outlined is demonstrated by the data of Table IV obtained for a water bridge between two poly(methyl-methacrylate) surfaces. It can be seen that the calculated contact angle values are practically independent of the actual values of *W* obtained from the data of the *F*, *a*-curves.

TABLE IV

Comparison of Contact Angle Values Measured (Θ_{exp}) *and Calculated* (Θ_{calc}) *from* F, a = *Data Using Eq. (6)*

Separation distance		W	$\gamma_{SV}-\gamma_{SL}$	Θ_{calc}	Θ_{exp} advancing	receding
a_1	a_2					
mm		erg	erg/cm²	degree	degree	
1.80	3.84	33.0	25.2	69.6	79	52
2.26	3.66	21.6	25.1	69.7		
2.26	3.84	23.2	29.9	66.3		
1.80	3.66	31.3	20.6	73.4		
1.80	3.26	26.3	25.7	69.9		
			Mean value:	69.7	65.5	

In addition, there is reasonable agreement of the calculated contact angle with the arithmetic mean of the experimental values measured under advancing and receding conditions. The same result has been found for other systems not discussed here, too. All data so far available show that the model applied for the treatment of solid/liquid/solid wetting systems is likely to be close to being correct, although refinements of the model will be needed in future work.

Wetting of Polymers with Surfactant Solutions

The wettability of polymers is often treated in terms of Zisman's critical surface tension of wetting, γ_c, which can be defined as the surface tension of a hypothetical liquid with the property to form a »zero contact angle« on the given solid. The critical surface tension has been found to be a characteristic parameter of the solid, depending primarily on the chemical constitution of the surface, but also depending to some extent on the nature of the liquids which are used for its determination by extrapolating the experimental cos Θ *vs.* γ_{LV} curves for $\cos \Theta = 1$, γ_{LV} being the liquid surface tension.

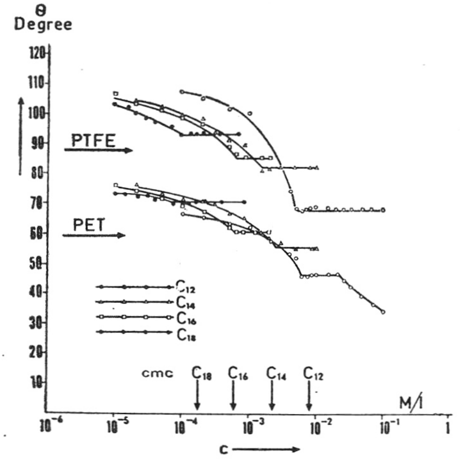
The influence of the chemical nature of the liquids on γ_c clearly shows that specific interactions can not be left out of consideration and, as it is well known, if solutions of surfactants are involved there is an oriented adsorption of amphipathic molecules at the solid/solution interface. Surprisingly enough, experimental evidence has been given[14,15] that adsorption of amphipathic solutes at the solid/vapour interface, too, has to be taken into account. It was namely found that on some low-energy surfaces, *e. g.* polyamides, polyesters, even those aqueous surfactant solutions do not spread whose surface tension is less than the γ_c of the polymer under consideration. In order to interprete this experimental result it was assumed that on the polar sites, which such surfaces undoubtedly possess, surfactant ions can be adsorbed over a microscopically narrow region of the solid/vapour interface in the immediate vicinity of the three-phase contact line. Since this interfacial microenvironment is alone responsible for a given contact angle configuration, the unexpected non-spreading behaviour in the cases mentioned above is a result of the local decrease of γ_c in a *quasi* two-dimensional area of the solid surface.

As far as aqueous solutions of surfactants or other capillary active substances are concerned, the wetting of polymers shows other peculiarities, too. As an example, we found earlier[16] that in such cases the widespreadly used Zisman plot can not be applied and instead of cos Θ, the wetting tension $\gamma_{SV} - \gamma_{SL}$ against γ_{LV} does fit a linear plot very well. The evaluation of experimental data of other authors[17-20] gave the same results and the straight-line relationship found by us was shown[5] to be in agreement with theoretical predictions as given by Fowkes[21] and Good[22] contrary to the Zisman relationship.

Furthermore, we obtained some unexpected results when investigating the dependence of the contact angle on the concentration of the anionic surfactant solutions if either the vapour phase was displaced by a nonpolar organic liquid[15], or the concentration was much higher than the c. m. c. as found for sodium dodecyl sulfate solutions on a number of polymers[23].

With this background, it seemed to be of interest to carry out systematic measurements on contact wetting of polymers of different polarity with solutions of both anionic and cationic surfactants of various chain length. Sodium alkyl sulfates from the dodecyl to the octadecyl compound were used as anionics, and alkyl trimethyl ammonium bromides in the same chain length range were the cationics investigated. Polymers were polytetrafluoroethylene (PTFE) and polyethylene terephthalate (PET), the first being practically nonpolar, and the second one having polar sites on its surface. Contact angle measurements have been performed in all cases under advancing conditions as described previously.

The results obtained are shown in Fig. 8 and 9. For both anionic and cationic surfactants on both surfaces the contact angle decreases with increasing concentration and after reaching the c. m. c. it remains constant with the exception of sodium dodecyl sulfate on PET. In this case, as reported earlier, there is a further decrease at a concentration about four times higher than the c. m. c. The reason for the appearance of this second critical concentration is presumably a transition of the micellar structure from the spherical to the lamellar shape, which is demontrated by the occurrence of break points at the same concentration in both the specific electrical conductance and the surface tension against concentration curves. The adsorption of the lamellar micelles or, in other words, the formation of a bimolecular adsorption



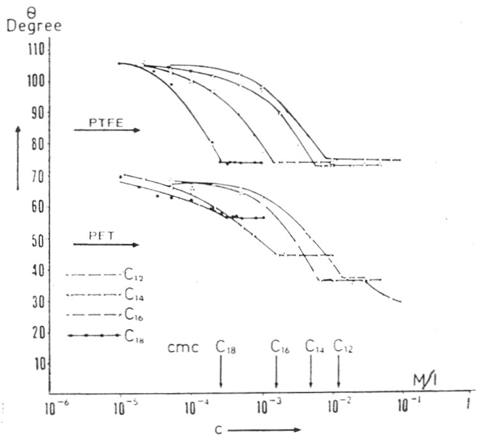


Fig. 8. Contact angle against concentration of aqueous sodium *n*-alkyl sulfate solutions on polymers.

Fig. 9. Contact angle against concentration of aqueous *n*-alkyl trimethyl ammonium bromide solutions on polymers.

layer with a normal and a reverse orientation of the molecules correspoding to the Langmuir principle, is likely to be energetically more favourable then the continued formation of the spherical associates with an increasing concentration in the bulk solution phase.

As to the influence of the chain length on how the contact angle varies with the solution concentration, there is an essential difference between the two surfactant series depending on whether the polymer surface does, or does not, have polar sites. For the polar PET, the anionics and the cationics behave very similarly, inasmuch as the contact angle decreases with increasing chain length, in the whole concentration range also covering values above the c. m. c. Although the same is true for the nonpolar PTFE when anionics are involved, there is no dependence on the chain length of the contact angle at or above the c. m. c. of cationic surfactant solutions.

For lack of a coherent theory on the contact wetting mechanism as influenced by specific molecular interactions, no quantitative analysis of the data obtained can be given. The results, however, allow a qualitative picture which is not inconsistent with present knowledge about the role adsorption plays in contact wetting. When comparing the contact angle *vs.* the surfactant concentration curves with those for the surface tension of the same sufactants (these curves, not given here, have the same minimum value at and above the c. m. c. independently of the chain length) then there is strong evidence that no specific adsorption of cationics on the non-polar Teflon surface occurs. Thus, in this case, the lowering of the contact angle with increasing concentration can unambiguously be due to the decrease of the surface tension according to the Young equation.

It can be seen from the curves shown in Fig. 8 and 9 that, at concentrations less than the c. m. c., the longer the chain the lower the contact angle, independently of the type of surfactant and of the surface. This fact shows that the adsorbability of the surfactants at the free solution surface is always greater than at the solution/solid interface even if the adsorption on the latter becomes, at and above the c. m. c., important, which is the case for all anionics on both surfaces, and especially on the polar PET.

The summarizing conclusion which can be drawn from the results is that, with the exception of cationics on the non-polar PTFE, specific interactions of a polar nature be operating, resulting in relatively strong adsorption of the surfactants at the solid/solution interface. Of what type they are is not yet clear but they certainly can not be coulombic forces. Thus the observed effects are likely to be due either to ionic-dipole or dipole-dipole interactions.

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IZVOD

Polimeri na površinama. Proučavanja kontaktnog kvašenja polimera

E. Wolfram

Prikazani su neki noviji rezultati istraživanja u laboratoriju autora, i to *(1)* o kontaktnom kvašenju u četverofaznom sistemu čvrsto/tekuće/para/čvrsto, gdje dolazi do stvaranja tekućinskih mostova između čvrstih površina, i *(2)* o kontaktnom kvašenju površina polimera otopinama površinsko aktivnih tvari.

Pokazan je izvod jednadžbe za napetost (energiju) kvašenja koja ne sadrži veličine kutova kvašenja. Na taj način jednadžba je neovisan test za klasičnu Youngovu jednadžbu.

Ukazano je da rezultati mjerenja fenomena kvašenja polimernih površina otopinama površinsko aktivnih tvari ne dozvoljavaju kvantitativne zaključke. Tome je uzrok postojanje specifičnih interakcija polarne naravi koje nisu kulonske, a koje uzrokuju jaku adsorpciju površinskoaktivnih tvari.

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