

An experimental study about the imbibition of aqueous solutions of low concentration of a non-adsorbable surfactant in a hydrophilic porous medium

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Received 10 March 2006; accepted 27 April 2006

Available online 5 June 2006

Abstract

The imbibition of aqueous solutions of Triton X-100 in calcium fluoride columns has been studied in order to determine the influence of the interfacial adsorption of the surfactant in the capillary rise of the solutions. This system has been chosen because this surfactant behaves as non-adsorbable at the surface of this solid when it is in aqueous solution. The experiments have consisted of the measurement of the increase in the weight of the porous columns caused by the capillary rise of the solutions. The analysis of the results has been made through a modified expression of Washburn's equation that takes into account that the experimental increase in the weight is caused by the imbibition as well as by the development of a liquid meniscus around the bottom base of the columns. From this analysis, it has been deduced that the surfactant concentration does not influence on the imbibition rate, it being equal to the observed for water. However, it has been also proved that the contact angle depends on the surfactant concentration, taking decreasing values as the surface tension of the solutions decreases. In order to justify these findings, a study about the influence of the interfacial adsorption on the imbibition has been carried out. By means of them, it has been proved that the absence of adsorption at the solid–liquid interface is the reason that explains both the independence of the imbibition rate from the surfactant concentration and the decrease of the contact angle. Moreover, this fact indicates that the depletion of the surfactant molecules from the advancing meniscus, which has been normally adduced as the phenomenon causing the observed behaviour, has to be ruled out as the physical cause that justifies the behaviour found from the analysis of the imbibition experiments. As a corollary, it has been also stated that only if the adsorption at the solid interfaces happened, the imbibition of aqueous solution of surfactant in hydrophilic media could be influenced by the surfactant concentration.

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Keywords: Imbibition; Surfactant; Porous media; Adsorption; Free energy; Contact angle

1. Introduction

Experimental techniques based on capillary rise of pure liquids in porous substrates have been broadly utilised to carry out the thermodynamic characterisation of these solids through the deduction of their surface free energy [1–7]. This is why a considerable group of studies has been developed in order to go deeply in the knowledge of the imbibition phenomenon. From them, the surface tension and the contact angle have been found as the driving quantities for the movement of the liquid inside the pores of the medium [8–10]. However, the researches related to the imbibition of aqueous solutions of surfactant are

rather limited [11–22] despite the well-known fact that these molecules are able to alter the surface tension of the solvent as well as its contact angle on the surface of the solid [23]. As pointed elsewhere [11,13–18,20–22], it would be expected that the possible adsorption of the surfactant at the interfaces that take part in the capillary rise played a decisive role in the imbibition kinetic. On the one hand, it is well known that the adsorption at the liquid–vapour interface decreases the surface tension of the liquids [23]. On the other hand, the probable adsorption at the solid interfaces during the imbibition would cause an additional change of the surface tension with regard to equilibrium value because of the depletion of the surfactant molecules from the liquid–vapour interface [11,13,15,17,18,20–22]. Besides, these adsorptions would give rise to the alteration of the energetic characteristics of the interfaces near the

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three phase contact line, and hence, the contact angle at the advancing meniscus could also change with regard to the value corresponding to the pure solvent [11,13,15,19–22].

All the features just described above seem to indicate that the possible simultaneous adsorption of the surfactant at the mentioned interfaces may change the characteristic quantities of the imbibition of the solvent, surface tension and contact angle, these ones acquiring a value compatible with the surfactant concentration. As a consequence, the higher the concentration of the solution is, the more affected the imbibition velocity should be [11–22]. Even so, despite the efforts made, the role of the surfactant on the imbibition dynamics is not clear enough at present.

One kind of research works carried out so far deals with the imbibition of aqueous surfactant solutions in hydrophilic porous solid [13,14,18,21,22]. These media are characterised by the spontaneous rise of water in their pores. When the surfactant is added to the solution, its adsorption at the liquid–vapour interface causes an alteration of the surface tension of the solution. On the other hand, the adsorption at the solid–liquid interface gives rise to a change of the energetic features of the solid surface that modifies the contact angle in relation to the value corresponding to water, it increasing or decreasing depending on the orientation of the adsorbed surfactant molecules. This fact could yield to a higher capillary pressure that should accelerate the rise of the solutions in the pores of the solid [21,22]. However this adsorption of the surfactant at the solid–liquid interface would cause a decrease in the surfactant concentration at the advancing meniscus that would modify again the values of the surface tension and the contact angle in relation to their equilibrium values. In consequence, the imbibition should be also affected by the new change of these parameters. The latest theoretical model proposed to describe this capillary rise process predicts that the imbibition of the solutions will be developed in the same way as the imbibition of water whenever the adsorption of the surfactant at the solid–liquid interface is strong enough as to be able to remove the whole of the surfactant molecules from the advancing meniscus [21,22]. In order to avoid the depletion of the surfactant molecules from the advancing meniscus due to the adsorption on the solid, some authors suggest the adsorption to be carried out before the imbibition experiments [16]. Thus, the maximum number of surfactant molecules compatible with the concentration of the solution would be always placed at the liquid–vapour interface during the imbibition. Under this experimental condition, the velocity of the capillary rise should show a marked dependence on the surfactant concentration. To check experimentally this situation is precisely the purpose of this work. However, instead of carrying out the adsorption of the surfactant on the solid surface before the imbibition, we have employed a solid material, calcium fluoride, unable to adsorb on its surface the surfactant, Triton X-100, selected to perform the capillary rise experiments [24]. Thus, it is sure that the solid–surfactant solutions systems verify the wanted experimental condition. We would like to throw into relief the novelty of this approach, since, until we know, no similar experimental work has been developed yet.

2. Experimental

Imbibition experiments have been carried out using glass tubes of the same diameter (4.95 ± 0.05 mm) and length (73.30 ± 0.05 mm). Prior to use, the tubes were acid-cleaned, washed with water several times and, once dried, one of their ends sealed by a filter paper disc. The glass tubes were filled with identical amounts (1.0005 ± 0.0005 g) of powdered sintered calcium fluoride (Merck, purity 99.5%) introduced inside it by hand. Previously, calcium fluoride had been dried into an oven at 150°C overnight. To obtain a good experimental reproducibility, the filled glass tubes were mechanically tapped by an automatic controlled-frequency device on their bottom base. To assure the uniform packing throughout the columns, a massive metallic rod was placed on the top of the powdered solid inside the tube. This tapping was performed continuously until the solid reached the same length in every glass column (35.3 ± 0.3 mm).

The liquids employed in the capillary rise experiments were water, distilled and deionised from a Milli-Q system, and aqueous solutions of the non-ionic surfactant Triton X-100 (Fluka, purity 99.0%). Triton X-100 consists of a 1,1,3,3-tetramethylbutyl group, a phenyl group, and a $-(\text{OCH}_2\text{CH}_2)_{9-10}\text{OH}$ chain grafted to it. The average number of oxyethylene groups is assumed to be 9.5. Its critical micelle concentration in water at 20°C is 3.0×10^{-4} M [25]. The concentration of the surfactant solutions was comprised between 10^{-4} and 10^{-9} M. Their surface tensions were measured by means of an automatic tensiometer, the du Noüy ring technique being employed. Viscosities have been considered, as elsewhere [13], equal as for water due to the low surfactant concentration.

The increase in the weight of the porous columns caused by the imbibition was measured with the aid of an automatic measurement device previously described [26–31], similar to those used by other authors [4,14]. Basically, it was a digital balance (Mettler AE240) with the porous column hung in the below-the-balance weighing facility. The glass receptacle with the liquid used in the experiment was placed on a mobile stage. This platform was lifted up by means of an Oriel motor computer controlled just until the free surface of the liquid contacted with the bottom of the glass tube. Weight–time data acquisition by the computer started before contact between the liquid and the porous column was achieved in order to avoid the loss of information at the very beginning of the experiment. It finished when the porous column was saturated with liquid, indicated by a constant weight value. Finally, the liquid mass inside the porous media was determined as the weight after breaking the contact between the column and the free surface of the liquid. The measurements were carried out at $20 \pm 0.5^\circ\text{C}$. For each surfactant concentration, imbibition experiments were repeated done at least four times.

3. Analysis of results and discussion

The capillary rise experiments have consisted of the measurement of the increase in the weight, w_{exp} , versus time, t , as a result of the imbibition of water and aqueous solutions of

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