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ORIGINAL CONTRIBUTION

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An absolute droplet pressure interfacial tensiometer and its application to bituminous systems of vanishing density contrast

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Abstract Experimental problems preclude or limit measurements of interfacial tension in bitumen or extra-heavy crude oil-containing systems when there exists a vanishing density difference between the phases. We describe a novel droplet pressure method that allows such measurements to be made. This method is based on a liquid/liquid adaptation of the capillary displacement differential maximum bubble pressure surface tension method of Schramm and Green [29]. In this method, interfacial tension is calculated from the difference between maximum droplet pressures reached at capillaries of differing internal radii, immersed to slightly different depths. The elimination of the influence of liquid densities allows the measurement of interfacial tensions without independently determining the liquid densities, and in particular, permits measurements in systems for which the density difference is vanishingly small. The absolute measuring technique is illustrated for several systems of pure and practical liquids.

Key words Interfacial tensiometer · Bituminous systems · Vanishing density contrast · Droplet pressure

List of symbols and abbreviations

b	radius of curvature at the bubble/droplet apex, cm
g	gravitational constant = 981 cm s^{-2}
l	immersion depth to the bubble/droplet apex, cm
MBPM	maximum bubble pressure method
MDPM	maximum droplet pressure method
p^h	pressure due to immersion, mPa
p^s	pressure due to surface forces, mPa
p^t	total pressure in capillary tube, mPa
Δp	differential maximum bubble/droplet pressure, mPa
r	capillary tip radius, cm
t	capillary tip immersion depth, cm
Δt	capillary tip displacement, cm
z	immersion depth between capillary tip and bubble/droplet apex, cm
$\Delta\rho$	density difference between the phases, g cm^{-3}
γ	surface tension, mN/m

Subscripts 1,2 designate properties at capillary tubes 1 and 2

Introduction

The behaviour at the transition region of contact between two phases may be very important for diverse reasons ranging from explaining interactions at a molecular level, to determining the influence of synthetic and natural surfactants on capillary numbers and enhanced oil recovery [1], to calculating parameters such as spreading and entering coefficients, or lamella numbers, to making predictions about the destabilising effects of oils on foams [2, 3].

Among the traditional methods for measuring interfacial tensions, the du Noüy ring and drop weight or volume methods are not appropriate for dynamic measurements while the methods of capillary rise, du Noüy ring, and Wilhelmy plate are generally not appropriate for interfacial tension measurements when other than pure liquids are involved [4, 5]. For crude oil/aqueous systems the conventional techniques that may be appropriate are pendant and sessile drop, spinning drop, and surface laser light scattering [6, 7].

The spinning drop technique has been adapted for use at elevated temperatures and low pressures (at the equilibrium vapour pressure of water at the temperature of measurement) and applied to crude oil/aqueous interfacial tension determinations [8, 9]. The surface laser light scattering technique (SLLS) has been successfully applied to gas/crude oil surface tensions [10, 11], but has apparently not been successfully applied to aqueous/crude oil systems, possibly because crude oils are strongly photoabsorbing [10, 11] and because SLLS is not capable of accurate interfacial tensions for viscous fluids [12]. The pendant and sessile drop techniques are usually applicable to dynamic or equilibrium conditions, are readily adapted to elevated temperatures and pressures [7, 13–15], but are generally limited to interfacial tensions greater than about 1–10 mN/m. The captive drop method, an adaptation of the sessile drop technique, extends the range of dynamic, high temperature and pressure interfacial tension measurements down to values as low as 0.003 mN/m in crude oil/natural surfactant-cosurfactant systems [16]. Each of these techniques requires an accurate and precise knowledge or measurement of the densities of each liquid phase. Although this is normally only an inconvenience of time and labour, it does present a significant experimental difficulty when the density contrast between the phases begins to vanish. For example, in systems containing an aqueous phase and Athabasca bitumen the density difference can range from extremely small to zero, for system temperatures in the range 40–120 °C, while remaining immiscible. For such systems the measurement of sufficiently accurate density differences is either extremely difficult or impossible.

An approach to this problem involves an adaptation of the classical bubble pressure method [17, 18]. The pressure needed to grow a bubble at the tip of a capillary tube immersed in a liquid increases to a maximum before the bubble is released and a new cycle begun. In the classical maximum bubble pressure method (MBPM) by measuring this maximum pressure and knowing the density of the liquid, the size and depth of immersion of the capillary, the surface tension of the liquid can be calculated [4, 5]. Although not as widely adopted as for example the du Noüy ring, Wilhelmy plate or drop-weight methods, the method has advantages in that the influence of surface impurities is small and a wide variety of liquids can be accommodated, ranging from simple hydrocarbon liquids to molten metals. Recently there has been somewhat of a resurgence of interest in its use owing largely to the availability of improved means to measure conveniently small pressures and a number of commercial and individual research laboratory bubble pressure surface tensiometers reported, operating on either the original Sugden principle, or the differential method [19–26]. Several adaptations of the method are now available, including one that allows measurement of the differential maximum bubble pressures from two capillaries placed at equal depths in a liquid of interest. This method almost, but not quite, eliminates the need to measure the capillary immersion depth and the liquid density [27]. A better method is the capillary displacement differential MBPM which almost entirely eliminates density influences [28, 29]. This paper describes a density-independent, absolute interfacial tensiometer using the capillary displacement differential maximum droplet pressure method (MDPM) which is based on a liquid/liquid adaptation of the capillary displacement differential MBPM. For convenience we will refer to the new method as the droplet pressure method.

Theory

The relationship between differential maximum droplet pressure and interfacial tension comes from the theory for maximum bubble pressure in a single capillary. Figure 1 shows bubbles being formed at the tips of two

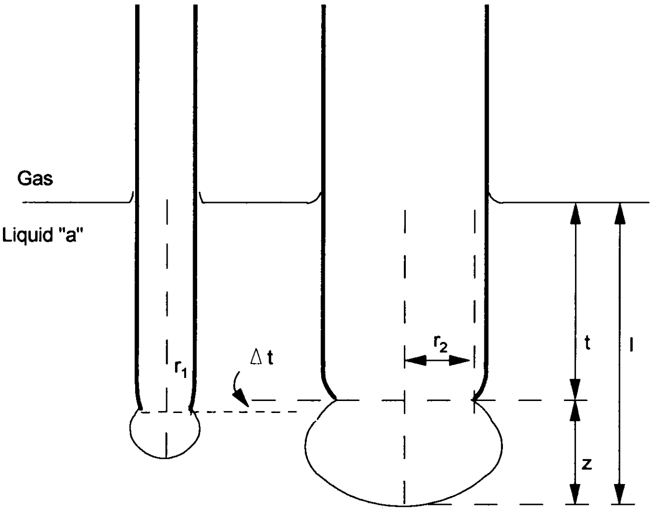

Fig. 1 Illustration of bubble formation at the tips of displaced capillaries in a liquid (Liquid "a"). For droplet formation the gas would be replaced by a second liquid (Liquid "b")

immersed capillary tubes. For either tube, the pressure in the tube, p^t, is the sum of the pressure due to surface forces described by the equation of Young and Laplace, p^s, and the hydrostatic pressure due to the immersion of the capillary tip and bubble below the bulk liquid surface, p^h. Thus,

$$p^t = p^s + p^h = (2\gamma)/b + \Delta\rho g l \tag{1}$$

where b is the radius of curvature at the apex of the bubble, $\Delta\rho$ is the density difference between the phases, and l is the immersion depth to the apex of the bubble. During the growth of a bubble at the capillary tip the internal pressure will go through a maximum value which is uniquely related to the surface tension and which is readily measured. If the maximum internal pressure, densities and immersion depth are known then the surface tension can be obtained from an evaluation of the drop shape.

The capillary displacement differential version of the method involves placing two capillary tubes of differing internal radii in the liquid so that both orifices are slightly displaced in terms of immersion depth. The differential maximum bubble pressure between bubbles at each tube is described as $\Delta p_{1,2}$ for tubes numbered 1 and 2 by

$$\Delta p_{1,2} = p_1^t - p_2^t \tag{2}$$

For this situation, Cuny and Wolf [28] derived the following equation relating the differential maximum bubble pressure to surface tension:

$$\frac{0.005\Delta p_{1,2} + 981\Delta\rho((r_2 - r_1)/3 - 1/2\Delta t) + 4.0098\left(r_2^3 - r_1^3\right)\Delta\rho^2/\gamma}{1/r - 1/r_2} \tag{3}$$

where r_1 and r_2 are the capillary tip radii and Δt is the capillary displacement (Fig. 1). In this equation the constant 0.005 is exact, the gravitational constant 981 is in cm s^{-2}, and the constant 4.0098 is in cm^2 s^{-4}. For $\Delta t = 0$, Eq. (3) reduces almost exactly to the two-capillary Schrödinger equation for equally submersed capillaries, which is accurate to a part per thousand for small enough capillary radii [30]. In practice, one measures the capillary radii, calculates the value of Δt for which the second term of Eq. (3) would become zero, and then adjusts the capillaries so that the difference between their immersion depths is equal to that value of Δt [29]. If an accuracy of about 0.4% in the surface tension is sufficient then the third term can be dropped. Two significant advantages of the capillary displacement, differential method are that the hydrostatic pressure due to capillary immersion depth and the liquid density do not need to be measured. The Cuny and Wolf approach was reduced to practice for gas/liquid systems by Schramm and Green [29]. In the present work we have developed a liquid/liquid droplet pressure method, based on the principles of capillary displacement and differential maximum droplet pressures, retaining the independence from liquid densities.

Experimental

Materials

The water used was doubly-deionized by a Millipore water purification system and then thoroughly degassed. All liquids used were chromatography or certified ACS grade unless otherwise stated. Sodium dioctylsulfosuccinate (Aerosol OT, Sigma-Aldrich, 98%) was used as received. Coker-feed Athabasca bitumen was obtained from Syncrude Canada Ltd.

Apparatus

The apparatus shares some common features with the analogous bubble pressure instrument we developed previously [29]. In the present apparatus, pressures were measured using a precision differential pressure gauge (Rosemount C1151HP5E22B1E6). The droplet phase was delivered by pumping aqueous sample phase from a floating piston transfer vessel driven by pump oil from a precision HPLC pump (ISCO Model 314). The larger radius was stainless-steel, machined to a smooth 45° knife edge, mounted with Teflon ferrules designed to allow vertical alignment in the supporting block (Fig. 2). The smaller radius capillary was stainless-steel, blunt-tipped, and similarly mounted. To facilitate temperature control, levelling and adjusting the capillary displacement, the capillary-holding block was mounted on an environmental chamber (Ramé-Hart Model 100-07) from an NRL contact angle goniometer (Ramé-Hart Model 100-00). To hold the continuous phase liquid samples and allow viewing without optical distortions, a 44 × 24 mm rectangular spectrophotometer cuvette was placed inside the environmental chamber. Bitumen samples were placed in a Teflon cup to eliminate water separation along the walls from such samples. The environmental chamber was attached to a travelling linear scale (Model 192-106, Mitutoyo Mfg. Co.,

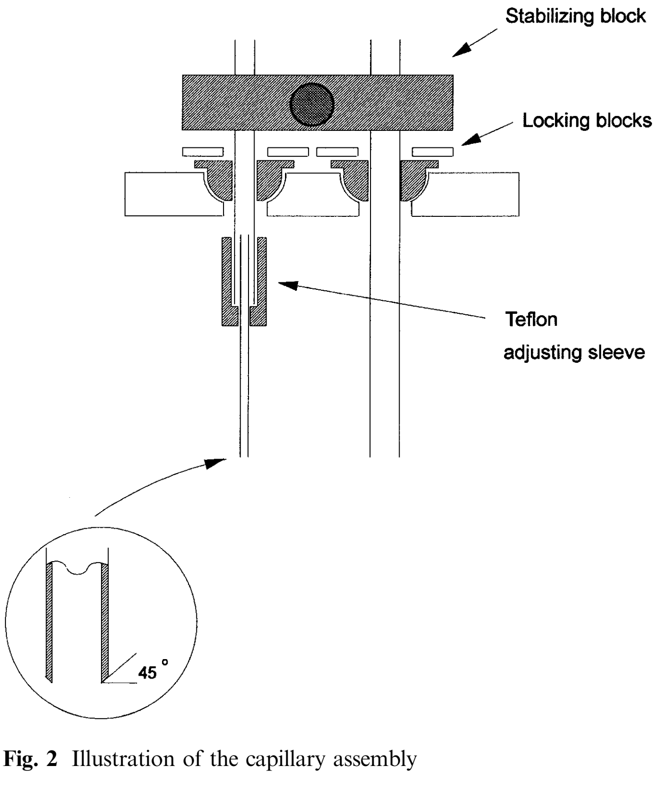

Fig. 2 Illustration of the capillary assembly

Tokyo, Japan) which in turn was mounted on an optical table. The travelling scale was used to measure the capillary displacement, aligning the planes of each capillary orifice with the cross-hairs in a rack and pinion microscope (Model 101A, Gaertner Sci. Corp., Chicago, Ill.). A schematic drawing of the apparatus is shown in Fig. 3.

The HPLC pump and transfer cylinder delivery arrangement allowed for continuously variable, stable droplet-phase liquid flow rates capable of producing droplet periods from less than a few seconds to greater than 5 min. A minimum 30 s interval to reach maximum droplet pressure, measured from the time of dislodgement of the previous drop, was found to be the most reliable. To ensure this, we found that total droplet periods of about 2–4 min worked best.

Operation was initiated by closing off one of the capillaries and causing droplet formation at the other capillary. Measurements were made by carefully increasing pressure so that a droplet would begin to grow at the capillary tip, and continue to grow through a complete cycle to break-off. Throughout the cycle the pressure was monitored and the maximum pressure reached was recorded. The procedure was repeated for more droplets and then for droplets using the other capillary until sufficient maximum droplet pressure readings were obtained to provide averages for each capillary. From these the differential maximum droplet pressure was calculated.

Other

Densities were measured using the vibrating reed principle (Anton Paar, Graz, Austria, Model DMA 46). Drop volume interfacial tension measurements were made following the procedure and recommendations of Padday [31]. Captive drop interfacial tension measurements were performed using the apparatus and method of Schramm et al. [32].

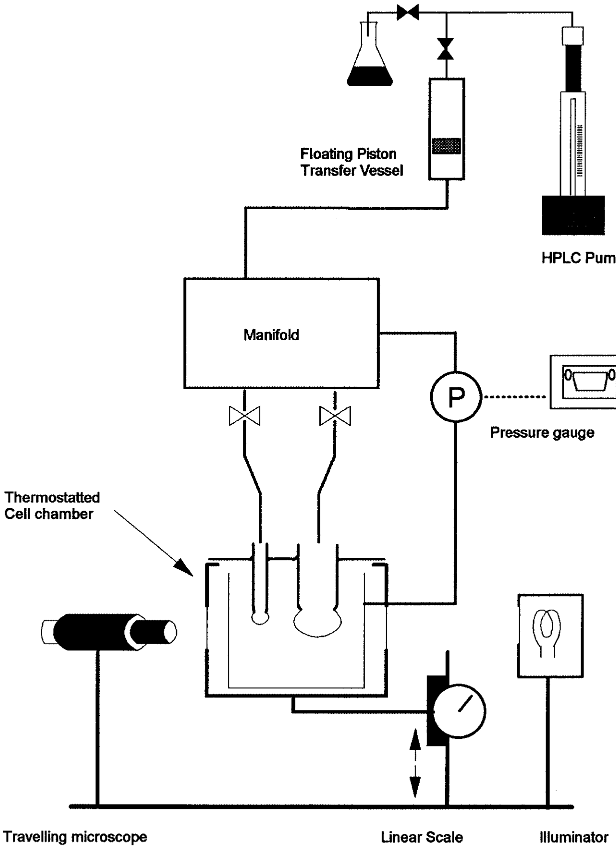

Fig. 3 Schematic illustration of the apparatus

Results

Interfacial tension of pure liquid pairs

Equation (3) is accurate for capillary radii of not more than 1.8 mm [28], setting an upper bound on r_2. The smaller capillary should be as small as practical to minimise the errors in measuring $\Delta p_{1,2}$, Δt and r_1. In gas/liquid work [29] we found that it was difficult to control the bubble period at the smaller capillary below a certain radius, and that a good practical compromise was achieved by employing radii values of $r_1 = 0.344 \pm 0.002$ mm and $r_2 = 1.545 \pm 0.007$ mm. In the case of liquid/liquid system measurements, such a large r_2 capillary size resulted in extremely small pressures and we found it advantageous to employ smaller capillaries. After trying various combinations of sizes we found that good results could be obtained with capillary tip radii of $r_1 = 0.155$ mm and $r_2 = 0.344$ mm. For these values, the second term in Eq. (3) will be zero if $\Delta t = 2(r_2 - r_1)/3$. The capillary displacement was set to this value. Furthermore, for an accuracy of 0.5% the third term can be neglected. Therefore,

$$\gamma = 1/2\Delta P_{1,2}/(1/r_1 - 1/r_2) \qquad (4)$$

Equation (4) can be used for absolute measurement if the r values are carefully measured, otherwise the slope can be determined by calibration with a series of reference liquid/liquid pairs.

Using a series of solutions of varying interfacial tensions and densities (Table 1), the absolute calibration is indicated in Table 1. The mean error in the experimentally determined surface tensions was 0.6 mN/m absolute. This is quite good for an absolute interfacial tension method that is independent of liquid densities, and is certainly acceptable for our interest in applying the technique to liquid/liquid systems of vanishing density difference.

Table 1 Interfacial tensions determined using the droplet pressure method, involving capillary displacement and differential maximum droplet pressures (23.5 °C)

System	Density difference ($g\ cm^{-3}$)	Interfacial tension (mN/m)	
		Reference	Present work
Ethyl acetate/water	0.0972	6.8	6.6
Methylisobutyl ketone/water	0.1997	9.8	10.7
Toluene/water	0.1336	36.1	37.3
Decane/water	0.2670	51.2	51.7

Dynamic interfacial tension

Dynamic interfacial tensions are exhibited by solutions which equilibrate slowly at the liquid/liquid interface. A number of surfactant solutions exhibit such dynamic surface tensions. This property is of interest where a certain time value of interfacial tension is needed or where the shape of the surface tension vs time curve is needed, e.g. to calculate interfacial elasticity. Finally, when comparing interfacial tension results obtained from different measuring techniques, the possible influence of dynamic character must be considered.

In the present instrument it was possible to adjust either of the capillaries to yield droplet periods from less than a few seconds to greater than 5 min. The shorter period was limited mainly by the response time of the pressure sensor while the longer period was limited by instabilities in the growing droplets. By varying the droplet periods, the interfacial age corresponding to each interfacial tension measurement is changed, although for each measurement the appropriate regions of the droplet growth cycles at each of the two capillaries must be equal [27].

Interfacial tensions of bituminous systems of vanishing density difference

Research into interfacial tension relationships in bitumen/aqueous systems is complicated by a rather unique problem. That is, at some temperatures the density difference between bitumen and water is either negligible or vanishes completely, yet almost all interfacial tension techniques rely on having a significant density difference between the phases. Figure 4 shows the densities for bitumen (interpolation of measured values, present work) and pure water (single curve representing numerous values from the literature) as a function of temperature. It can be seen that the density differences can become vanishingly small, and even invert, at temperatures between 50 °C and 85 °C. It is this temperature range that is of practical interest for the hot water flotation of bitumen from oil sands slurries in commercial surface mining and processing operations; see [33–35]. For such practical samples the density differences can also be made small by the presence in samples of small amounts of highly dispersed fine solids or bubbles that may persist after treatment. In order to determine interfacial tensions using conventional techniques for such systems of vanishing density, the individual liquid densities must be measured with great precision and accuracy in order to calculate sufficiently accurate density differences. Not only is this laborious, but at some point the densities of the liquids concerned may become so close to each other that density measuring errors exceed the actual density difference. It is for such cases that the present technique is particularly well-suited.

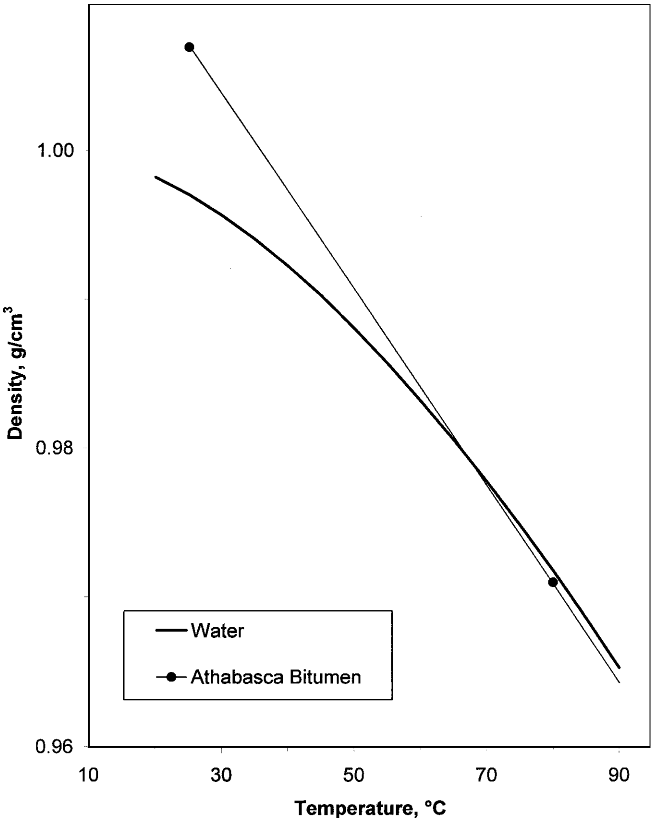

Fig. 4 Variations in Athabasca bitumen and water densities with temperature

The instrument was next tested with bitumen/aqueous systems at 80 °C, at which temperature the densities of the phases were 0.9839 g cm^{-3} and 0.9718 g cm^{-3} respectively, yielding a density difference of only 0.0121 g cm^{-3}. Drop periods up to about 8 min were used if the drop reached its maximum pressure early in its growth detachment cycle [27]. This was usual for the larger radius capillary. For the smaller radius capillary, the maximum pressure was usually reached closer to detachment, and due to flow rate constraints of the pumping system, drop cycles of 20–40 sec were commonly used. These droplet periods were adjusted to ensure an approximate equality in the drop surface ages at the time of maximum pressure. The droplet pressure measurement result is shown in Table 2, together with a comparison value from extremely careful drop volume measurements.

A third comparison measurement was made in which the density difference was artificially enhanced. Previous researchers have reduced bitumen densities by adding toluene, but toluene is such a good solvent for asphaltenes that the risk of changing the interfacial properties is very high. Instead we selected decane,

Table 2 Interfacial tensions for coker-feed bitumen/water at 80 °C

Decane dilution (%)	Interfacial tension (mN m^{-1})	Method
0	19.9 ± 1.6	Drop volume
31.0	20.6 ± 1.7	Captive drop
0	20.5 ± 1.3	Droplet pressure

which has long been used in enhanced oil recovery research to reduce the viscosity of medium-heavy crude oils (to simulate gas-saturated oil) without significantly influencing oil/aqueous interfacial tensions. Comparing various additions showed that the decane addition did not significantly alter the interfacial tension. A 30 mass% solution of decane in bitumen was found to reduce significantly the density, to 0.8828 g ml^{-1} at 80 °C. The near-equilibrium (interfacial age of >30 min) result obtained using the captive drop method is shown in Table 2.

The captive drop, drop volume, and droplet pressure measurements of about 20.5 mN/m for coker-feed bitumen/water agree well with each other, and also with the early results reported by Bowman [36] and may represent the situation for nearly surfactant-free bitumen.

Interfacial tensions in a surfactant-containing system

Next, the interfacial tension of bitumen/water and *n*-dodecane/water systems to which were added varying amounts of surfactant Aerosol OT, dissolved in 2-propanol, was determined. The common name Aerosol OT refers to the active component from many marine oil spill dispersant formulations [37], sodium dioctylsulfosuccinate. Aerosol OT is also of considerable research interest because its wedge-shaped molecular geometry can lend itself to the formation of reverse micelles without the addition of cosurfactant.

The interfacial tensions of water against *n*-dodecane to which 2-propanol alone was added decrease regularly as shown in Fig. 5a. However, when Aerosol OT in 2-propanol was added to water/*n*-dodecane the interfacial tensions increased to a maximum followed by a decrease as shown in Fig. 5b. It appears that low concentrations of Aerosol OT, which for pure alkanes partitions preferentially to the water phase below the critical micelle concentration (cmc) [38], act to solubilise the 2-propanol in the aqueous phase. Aerosol OT molecules have been reported to aggregate at concentrations as low as 10^{-5} mol/l in solution [39] and have been speculated to form iceberg-like assemblages with nonpolar molecules [40]. At higher Aerosol OT-2-propanol additions, above the cmc, the Aerosol OT is expected to remain partitioned primarily in the aqueous phase [38], the interfacial adsorption layer is complete, and the 2-propanol effect begins to dominate again causing the interfacial tension to decrease, Fig. 5b.

Conversely, adding 2-propanol alone to bitumen causes interfacial tension to rise (Fig. 6a), while the addition of Aerosol OT (in 2-propanol) to bitumen/water produced an interfacial tension reduction to a minimum followed by an increase[1] (Fig. 6b). It appears that 2-propanol additions to bitumen/water cause interfacial tension to increase by driving asphaltenes away from the interface [41]. In this case, low concentrations of Aerosol OT, which for crude oils partitions preferentially to the oleic phase below the cmc [38], displace asphaltenes from the interface [42], are more surface active, and reduce interfacial tension. At higher Aerosol OT-2-propanol additions, above the cmc, the Aerosol OT is expected to have shifted its partitioning preference to the aqueous phase [38] and the 2-propanol effect begins to dominate again causing the interfacial tension to increase (Fig. 6b). It is thought that the changing partitioning preferences may be accompanied by changing conformations of the Aerosol OT at the interface, such as has been described by Hossain et al. [43].

We note that the maximum IFT for Aerosol OT added to dodecane and the minimum in IFT for Aerosol OT added to the bitumen occur at essentially the same surfactant concentration which, on a fully partitioned into oil basis, is about 0.46 mmol/l. This is slightly lower than the range reported for other hydrocarbon systems, 0.49–1.1 [39, 40, 43–45], possibly due to the presence of the propanol in the oleic phases. As referenced already, Aveyard et al. [38] found that Aerosol OT partitions preferentially to the aqueous phase in pure alkane/water systems at lower than cmc concentrations, whereas in North Sea crude oil systems it partitions preferentially to the oleic phase. In both cases, at above cmc concentrations, Aerosol OT preferentially partitions to the aqueous phase. They also found that the demulsification rate increased up to the cmc (in either phase), and thereafter decreased in the case of the crude oil systems. These observations, together with the interfacial tension results above, are relevant to research into the properties and treatment of bituminous froths created by the flotation of bitumen from oil sands [33–35, 41].

Discussion and conclusion

The droplet pressure method does not require droplet detachment nor complete wetting of an immersed

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[1] The apparent oscillation evident at high surfactant concentrations in Fig. 6b was reproducible. We have, at present, no explanation for this aspect of the interfacial tension behaviour.

Fig. 5 a Effect of 2-propanol additions (as volume%) to *n*-dodecane on interfacial tension against water at 80 °C. **b** Effect of Aerosol OT (prepared as wt% active ingredient in 2-propanol) additions to *n*-dodecane on interfacial tension against water at 80 °C

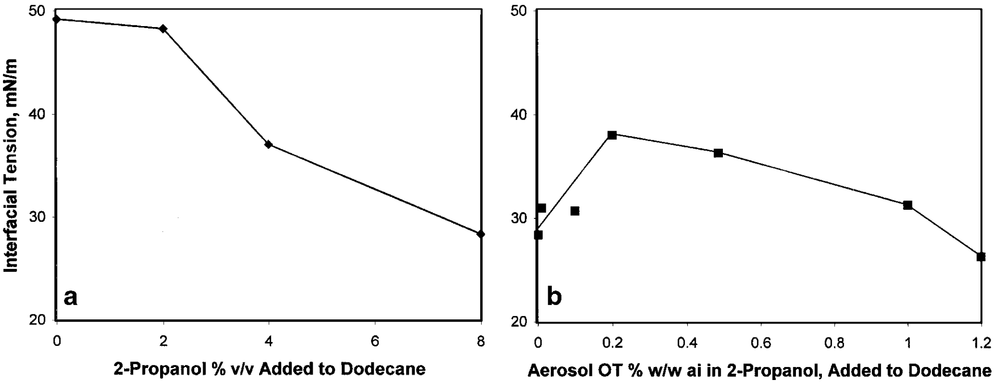
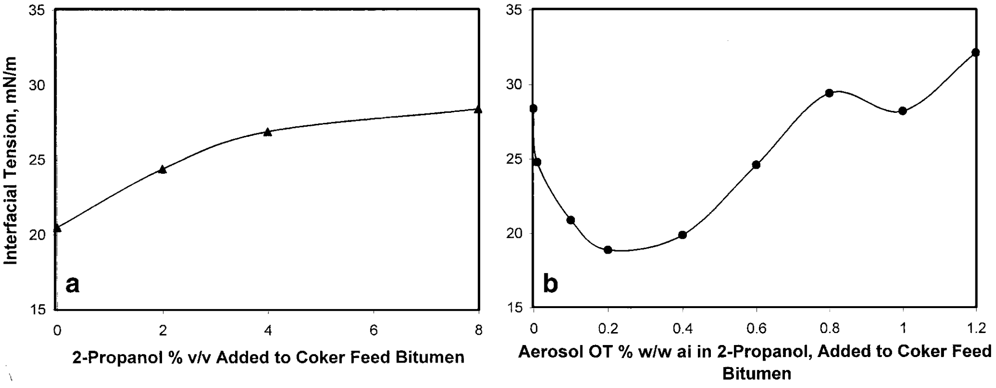


Fig. 6 a Effect of 2-propanol additions (as vol. %) to coker feed bitumen on interfacial tension against water at 80 °C. **b** Effect of Aerosol OT (prepared as wt% active ingredient in 2-propanol) additions to coker feed bitumen, at a level of 8 vol. %, on interfacial tension against water at 80 °C



surface as do the Wilhelmy plate, drop weight and drop volume methods [4, 5]. Although these latter methods can be more accurate than the droplet pressure method, they can be less convenient to use and they all require the separate determination of liquid density. Nagarajan et al. [46, 47] and Pandit et al. [48] earlier adapted the maximum bubble pressure method for use in liquid/liquid systems. In each case the system developed was a single-capillary adaptation of Sugden's method. Although the method reported by Pandit et al. was intended for systems of vanishing density difference, in fact the methods of each group require hydrostatic corrections for both the capillary and the drop at the condition of maximum droplet pressure, as discussed by Schramm [27] and Schramm and Green [29], so that the need to determine accurately the densities of both phases was not eliminated in either method.

The differential maximum droplet pressure technique is very convenient for measuring interfacial tensions of liquids of technical interest, especially for systems of vanishing density difference. An absolute droplet pressure instrument was developed using the principles of capillary displacement and differential maximum droplet pressure. The new instrument has several advantages. First, the provision of adjustable relative capillary immersion depths allows the method to be made independent of the density of the liquid samples, reducing the influence of density measurement errors and making it applicable to systems of vanishing density difference. In bituminous systems, being density independent is particularly advantageous due to the frequent presence in the bitumen of finely divided solid particles, water droplets and/or gas bubbles. Second, the present arrangement allows the technique to be used either as an absolute or relative method. Finally, with suitably chosen capillaries, a fairly wide range of droplet periods is accessible, from several tenths of a second to several minutes, allowing dynamic interfacial tension measurements to be made. Thus, for many liquid pairs, equilibrium and dynamic interfacial tensions can be measured.

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