Effect of the Interfacial Viscosities upon Displacement

A capillary rise technique was developed for measuring the effect of the surface viscosities upon the displacement of air by liquids in glass capillaries. Measurements were made in two tubes with optically measured radii of 37.5 and 48.6 μm.

No surface viscous effects were observed with doubly distilled water or with octane, in good agreement with the Washburn equation. The behavior of a dilute aqueous solution of dodecyl sodium sulfate was easily distinguished from that corresponding to zero surface viscosities. The surface dilatational viscosity was found to be two orders of magnitude greater than the surface shear viscosity. Previous qualitative (Slattery, 1974, 1979) and quantitative (Giordano and Slattery, 1983) theories for the effect of the interfacial viscosities upon displacement are fully supported by this experimental study.

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SCOPE

Our capability for optimally designing an enhanced oil recovery process depends upon our ability to simulate it. A reliable simulation requires accurate correlations for the relative permeabilities and the capillary pressure as well as an understanding of the impact of these correlations on the stability of a displacement. Correlations for the relative permeabilities and the capillary pressure in turn can be enhanced by a more detailed appreciation for the displacement and coalescence of phase interfaces within individual pores. The displacement and coalescence of interfaces within individual pores depends upon the interfacial viscosities, interfacial tension, wetting, and contact angle hysteresis.

Slattery (1974, 1979) developed a qualitative theory for the relative effects of the interfacial tension, interfacial viscosities, and wetting upon the displacement of residual oil. The predictions of this theory were fully supported by Giordano and Slattery's (1983) quantitative analysis that generalized Washburn's (1921) result to include the effects of the interfacial viscosities upon displacement in a single cylindrical pore. They predicted that the effect of the interfacial viscosities is to increase the resistance to displacement regardless of the wetting conditions. Sen and Slattery (1983) further observed that, of the two interfacial viscosities, the interfacial dilatational viscosity may play the more important role.

Our objective here is to report an experimental test of these conclusions for displacement of air by a dilute aqueous solution of dodecyl sodium sulfate in vertical capillaries. Measurements of the interfacial shear viscosity in a variety of interfacial viscometers have been reported previously. The optically measured radii of the capillaries were 37.5 and 48.6 μm.

CONCLUSIONS AND SIGNIFICANCE

No interfacial viscous effects were observed in the displacement of water-air and octane-air interfaces, the results for which were in good agreement with the analysis given by Washburn (1921).

The behavior of the solution-air interface was easily distinguished from that corresponding to zero interfacial viscosities. Experiments using both capillaries were in excellent agreement for the sum of the two interfacial viscosities. Given the interfacial shear viscosity from prior measurements, we conclude that the interfacial dilatational viscosity is two orders of magnitude greater.

This experimental study fully supports both the qualitative conclusions of Slattery (1974) and the quantitative analysis of Giordano and Slattery (1983).

A capillary-rise experiment, such as the one used here, together with a separate measurement of the interfacial shear viscosity may prove to be an effective technique for determining the difficult-to-measure interfacial dilatational viscosity.

INTRODUCTION

Hagen (1845) was apparently the first to suggest that the "viscosity" of the interfacial region differs from those of the adjoining phases. Plateau (1869) conducted some simple experiments using a floating needle rotating in a magnetic field. His interpretation of his results in terms of a "viscosity" of the liquid-gas interfacial region is almost certainly in error as the result of surface tension gradients developed in the interface.

Boussinesq (1913; Ericksen, 1952; Oldroyd, 1955; Scriven, 1960) correctly stated for the first time a linear relationship between the interfacial stress tensor and the interfacial rate of defromation tensor involving three physical parameters: the interfacial tension, the interfacial shear viscosity, and the interfacial dilatational visocity. Although numerous measurements of the interfacial shear viscosity of increasing quality have appeared in the literature (Jiang et al., 1983, for a review), there have been private as well as published (Levich, 1962) doubts about the reality and practical significance of the interfacial viscosities. In order to begin to allay these doubts, it has been clear that three types of studies would be required.

First, it would be necessary to demonstrate that, for a given interface, the same value for the interfacial shear visocity could be measured in a variety of interfacial viscometers. Jiang et al. (1983) have now successfully carried out such a series of experiment.

Second, a physical application would have to be developed in which it was demonstrated that the interfacial viscosities play a significant role. Slattery (1974, 1979) developed a qualitative theory

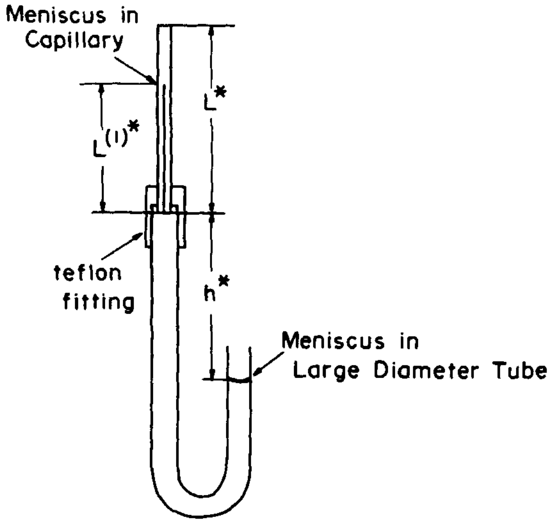

Figure 1. Vertical capillary used to study capillary rise. The height $L^{(1)*}$ of liquid in the capillary is measured as a function of time t^*. The time rate of change of $L^{(1)*}$ is determined by the value of h^* chosen.

for the relative effects of the interfacial tension, the interfacial viscosities and wetting upon the displacement of residual oil. The qualitative predictions of this theory have now been fully supported by the quantitative analysis given by Giordano and Slattery (1983) for the effects of the interfacial viscosities upon displacement in a single cylindrical pore.

Third, any physical application in which the interfacial viscosities were claimed to play a significant role would have to be demonstrated experimentally. Since difficulties were anticipated in distinguishing the effects of interfacial tension, of the interfacial viscosities, and of wetting upon displacement, an experimental test of Slattery's (1974) qualitative theory had to wait for Giordano and Slattery's (1983) quantitative analysis. In what follows, we describe an experimental test of their result for the displacement of air by a liquid in a vertical capillary.

CAPILLARY RISE

The experiment with which we are concerned here is shown schematically in Figure 1. A precision-bore, redrawn, Pyrex, capillary tube of radius R^* is mounted vertically. A length of flexible Teflon tubing is attached to the bottom by means of a Teflon fitting. For time $t^* < 0$, the equilibrium position of the liquid-gas interface in the capillary is $L_0^{(1)*}$ above the bottom of the capillary and the equilibrium position of the liquid-gas interface in the Teflon tubing is h_0^* below the bottom of the capillary tube. At time $t^* = 0$, h_0^* is reduced to h^* and the liquid-gas interface in the capillary rises to a new equilibrium position

$$L_{\text{eq}}^{(1)*} = \frac{2\gamma^* \cos\alpha}{R^*(\rho^{(1)*} - \rho^{(2)*})g^*} - h^* \tag{1}$$

in which γ^* is the interfacial tension, α the contact angle measured through the liquid phase, $\rho^{(1)*}$ the density of the displacing liquid phase, $\rho^{(2)*}$ the density of the displaced gas phase, and g^* the acceleration of gravity. [We have retained the notation of Giordano and Slattery (1983), in which a superscript . . .* is used to denote dimensional quantities.]

Giordano and Slattery (1983) made the following physical assumptions in analyzing displacement in a cylindrical capillary.

(a) Both the displacing and displaced fluids are incompressible and Newtonian.

(b) The fluid-fluid interfacial stress-deformation behavior can be represented by the linear Boussinesq surface fluid model (Boussinesq, 1913; Scriven, 1960).

(c) Inertial forces are neglected with respect to viscous forces within the bulk phases, which implies

$$N_{Re}^{(j)} \equiv \frac{\rho^{(j)*}V^*R^*}{\mu^{(j)*}} \ll 1 \tag{2}$$

where $N_{Re}^{(j)}$ $(j = 1,2)$ is the Reynolds number for phase j, V^* the speed of displacement of the phase interface in the capillary and $\mu^{(j)*}$ the viscosity of phase j.

(d) We require that the Bond number

$$N_{Bo} \equiv \frac{|\rho^{(1)*} - \rho^{(2)*}|g^*R^{*2}}{\gamma^*} \ll 1 \tag{3}$$

which means that the effect of gravity upon the configuration of the interface can be neglected.

(e) In the dividing surface, interfacial viscous forces dominate bulk viscous forces,

$$N_{\kappa+\epsilon} \equiv \frac{\kappa^* + \epsilon^*}{\mu^{(2)*}R^*} \gg 1 \tag{4}$$

Here κ^* is the interfacial dilatational viscosity and ϵ^* the interfacial shear viscosity.

(f) The capillary number N_{ca} is small,

$$N_{ca} \equiv \frac{\mu^{(2)*}V^*}{\gamma} \ll 1 \tag{5}$$

(g) The speed of displacement V^* is sufficiently small that surfactant concentration in the dividing surface may be considered nearly independent of position and the interfacial tension and the two interfacial viscosities may be treated as constants. [See Giordano and Slattery (1982), Appendix A.]

(h) In forming the integral mechanical energy balance for the system consisting of all of the liquid and gas in the capillary, the rate of viscous dissipation of mechanical energy within the bulk phases has been estimated as though these phases were in Poiseuille flow everywhere.

In addition to a few technical assumptions, Giordano and Slattery (1983) also chose to neglect gravity for the sake of simplification. When we take gravity into account in the manner that they indicated, we find

$$\begin{aligned}\frac{dL^{(1)*}}{dt^*} &= V^* \\ &= \frac{1}{\mu^{(2)*}}\left[\Delta\mathcal{P}^* - (\rho^{(1)*} - \rho^{(2)*})(L^{(1)*} + h^*)g^* + \frac{2\gamma^*}{R^*}\cos\alpha\right] \\ &\qquad \left[\frac{8}{R^*}\left(N_\mu \frac{L^{(1)*}}{R^*} + \frac{L^* - L^{(1)*}}{R^*}\right) + \frac{2}{R^*}N_{\kappa+\epsilon}A^2G\right]^{-1}\end{aligned} \tag{6}$$

Here

$$N_\mu \equiv \mu^{(1)*}/\mu^{(2)*} \tag{7}$$

$$\begin{aligned}A \equiv -\ &\{N_\mu(\alpha\cos\alpha - \sin\alpha)[\sin^2\alpha - (\alpha - \pi)^2] \\ &+ [(\alpha - \pi)\cos\alpha - \sin\alpha](\alpha^2 - \sin^2\alpha)\} \\ &\{N_\mu(\sin\alpha\cos\alpha - \alpha)[(\alpha - \pi)^2 - \sin^2\alpha] \\ &+ (\alpha - \pi - \sin\alpha\cos\alpha)(\alpha^2 - \sin^2\alpha)\}^{-1}\end{aligned} \tag{8}$$

$$G \equiv (1 - \nu)\frac{P_{\nu-1}^{-1}(\sin\alpha)}{P_\nu^{-1}(\sin\alpha)} + (1 - \nu^2)\sin\alpha \tag{9}$$

in which $P_{\nu-1}^{-1}$ is the associated Legendre function of the first kind (Abramowitz and Stegun, 1965) and ν satisfies

$$\nu(\nu + 1) = \frac{2\epsilon^*}{\kappa^* + \epsilon^*} \tag{10}$$

By $\Delta\mathcal{P}^*$ we mean the pressure difference (including hydrostatic effects) between the open end of the capillary and the open end of the Teflon tubing, which in this case is zero. Integrating Eq. 6, we conclude

$$\begin{aligned}&\frac{t^*R^*}{\mu^{(2)*}}(\rho^{(1)*} - \rho^{(2)*})g^* + \frac{8}{R^*}(N_\mu - 1)(L^{(1)*} - L_0^{(1)*}) \\ &= \left[\frac{8L^*}{R^*} + 2N_{\kappa+\epsilon}A^2G + \frac{8}{R^*}(N_\mu - 1)L_{\text{eq}}^{(1)*}\right] \\ &\qquad \ln\left(\frac{L_{\text{eg}}^{(1)*} - L_0^{(1)*}}{L_{\text{eg}}^{(1)*} - L^{(1)*}}\right)\end{aligned} \tag{11}$$

For the purpose of more clearly identifying surface viscous effects, *it will be convenient* to rearrange Eq. 11 as

$$Z = S \ln X \qquad (12)$$

where

$$Z \equiv \frac{t^*R^{*2}}{\mu^{(2)*}} (\rho^{(1)*} - \rho^{(2)*})g^* + 8(N_\mu - 1)(L^{(1)*} - L_0^{(1)*}) - [8L^* + 8(N_\mu - 1)L_{eq}^{(1)*} \ln X \qquad (13)$$

$$S \equiv 2R^*N_{\kappa+\epsilon}A^2G \qquad (14)$$

$$X \equiv \frac{L_{eq}^{(1)*} - L_0^{(1)*}}{L_{eq}^{(1)*} - L^{(1)*}} \qquad (15)$$

In the absence of interfacial viscous effects, we will find it helpful to express Eq. 6 as

$$Y = R^{*2}t^* \qquad (16)$$

in which we have introduced

$$Y \equiv \frac{\mu^{(2)*}}{(\rho^{(1)*} - \rho^{(2)*})g^*} \{[8L^* + 8(N_\mu - 1)L_{eq}^{(1)*}]\ln X - 8(N_\mu - 1)(L^{(1)*} - L_0^{(1)*})\} \qquad (17)$$

EXPERIMENTAL

We observed the capillary rise of two clean interfaces at room temperature (23 ± 1°C): doubly distilled water-air and octane-air. Values for the interfacial tensions, densities and viscosities were taken from the literature.

We also studied the displacement of the interface between air and an aqueous solution of 0.1 wt. % dodecyl sodium sulfate at room temperature (23 ± 1°C). The solution was prepared by gently mixing dodecyl sodium sulfate (catalog no. 5967, lot no. B3x, Eastman Kodak Co., Rochester, NY) and doubly distilled water for about two hours. The equilibrium interfacial tension ($\gamma = 21.3$ mN/m) was achieved 12–24 hours after the interface was formed and remained constant for at least a week. Jiang et al. (1983) measured the interfacial shear viscosity ϵ^* for this same interface in a variety of interfacial viscometers. They found that ϵ was weakly dependent upon the rate of deformation of the interface, approaching ~1 mN·s/m in the limit as the magnitude of the rate of deformation approached zero. The density and viscosity of this very dilute solution were assumed to be the same as water.

The borosilicate glass (Pyrex) capillary tubes were cleaned by a process suggested by Tichane (1963; see also Bernett and Zisman, 1968). The last step in the cleaning procedure was to evacuate each tube and place it in an oven to dry for one hour at 250°C, in order to remove any macroscopic water film.

Once the liquid under study was siphoned into the flexible tubing in Figure 1, the capillary was inserted into the Teflon fitting as follows. The lower leg in Figure 1 was raised until h^* was very close to zero, causing the liquid to form a protruding bulb in the mouth of the fitting. The capillary, at a small angle with respect to vertical in order to eliminate the formation of bubbles during the subsequent rapid uptake of fluid, was inserted into the fitting, the fitting tightened, and the capillary mounted vertically. The value of h^* was quickly adjusted to trap the meniscus in the capillary and the maximum height reached by the meniscus noted. The interface was then allowed to fall back to an equilibrum position.

For the octane-air and solution-air interfaces, 72 hours passed before experiments were begun. We waited until no deviations were observed as the interface passed from the prewet to the virgin portion of the capillary. Apparently 72 hours were required for any macroscopic film formed to drain and evaporate.

In the case of the solution-air interface, the 72 hour waiting period had an additional purpose. Jiang et al. (1983) observed that 24–48 hours were required after the formation of a fresh interface before equilibrium was established.

In the case of the water-air interface, two experiments were run in the dry portion of the tubing above the maximum height achieved during loading. The third was run over a prewet portion. No difference in behavior was observed among the three experiments. Very little time was required for any macroscopic film formed to drain and evaporate. The higher surface tension of the water-air interface may have allowed the meniscus to retreat without forming any appreciable macroscopic film.

As described above, an experiment was begun with a step change in h^* shown in Figure 1. The resulting advance in the capillary of the liquid-gas interface was observed using a cathetometer, incremental changes in its position being timed with a stopwatch. The value of h^* was monitored to confirm that it remained constant. In the case of the solution-air interface, measured speeds of displacement were always less than 3.5 μm/s (1 ft/d), in order to prevent the formation of concentration gradients and interfacial tension gradients in the interface (Giordano and Slattery, 1983). The estimated uncertainty in length measurements was ±0.002 cm; for time measurements, ±0.25 s.

At the conclusion of all of the experiments, the tubes were broken into short lengths and the diameters measured with a micrometer microscope. The estimated uncertainty in the measured values was ±0.5 μm. Deviations in radius over the length of each capillary were found to be negligible. The radius of tube 1 was 37.5 μm; its length was 13.920 cm. The radius of tube 2 was 48.6 μm; its length was 12.050 cm.

In order to minimize the uncertainty in X, defined by Eq. 15, we restricted our attention to runs for which the overall path travelled $(L_{eq}^{(1)*} - L_0^{(1)*}) \geq 1.0$ cm. Only that portion of the data for which $(L_{eq}^{(1)*} - L^{(1)*}) \geq 0.2$ cm was analyzed.

Many more details as well as all of the original data are given by Stoodt (1982).

RESULTS

The data for three runs, in which a doubly distilled water-air interface is displaced through tube 1, are plotted in Figure 2 as suggested by Eq. 16. Figure 3 is a similar plot of data for two runs, in which an octane-air interface is displaced through tube 2. These data plots confirm both that our optically measured values for the tube radii were correct and that the surface viscosities for these two interfaces were either zero or below the sensitivity of this experiment, as we would expect for relatively clean interfaces.

As another check on our experimental technique, we compared the equilibrium heights measured in these experiments with those predicted by Eq. 1, using the optically measured tube radii and assuming $\alpha = 0$. For the doubly distilled water-air interface in tube 1, the measured value of $L_{eq}^{(1)*}$ was 0.387 compared with 0.395 from Eq. 1. For the octane-air interface in tube 2, we observed 0.123 compared with 0.129 from Eq. 1.

Surface viscous effects were expected and observed for the interface between air and an aqueous solution of 0.1 wt. % dodecyl sodium sulfate. Figure 4 displays for a selected run the deviation from the Washburn (1921) Eq. 6 with $N_{\kappa+\epsilon} = 0$.

A least squares fit of Eq. 12 to the data for two runs in tube 1 is

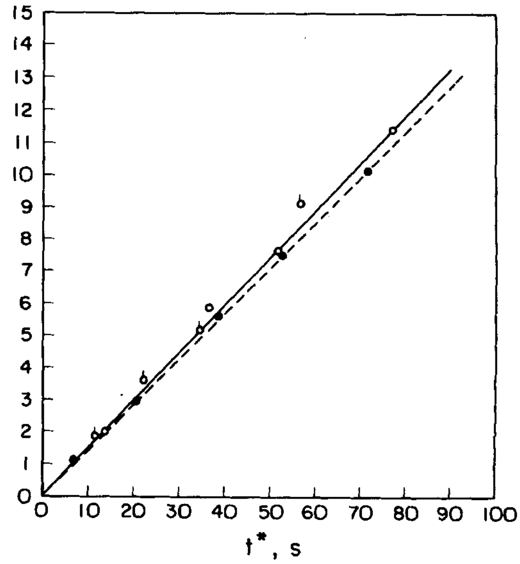

Figure 2. Data for three runs in which a doubly distilled water-air interface is displaced through tube 1, plotted as suggested by Eq. 16. The dashed line corresponds to the optically measured $R^* = 37.5$ μm; the solid line is the least squares fit of Eq. 16 to the data, suggesting $R^* = 38.5$ μm.

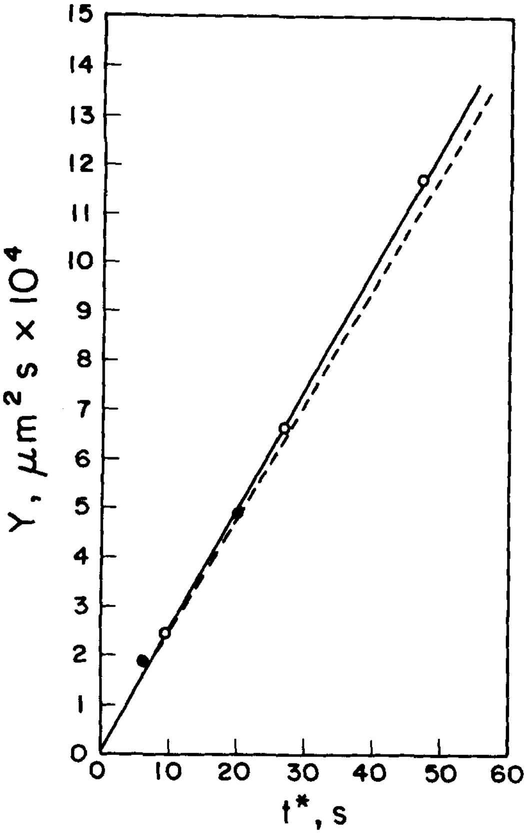

Figure 3. Data for two runs in which an octane-air interface is displaced through tube 2, plotted as suggested by Eq. 16. The dashed line corresponds to the optically measured R^* = 48.6 µm; the solid line is the least squares fit of Eq. 16 to the data, suggesting R^* = 49.7 µm.

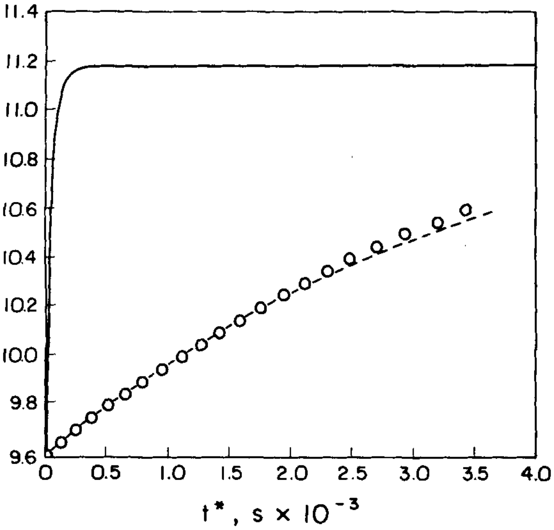

Figure 4. Displacement of solution-air interface through tube 1, as observed in run II. The solid line corresponds to the Washburn (1921) Eq. 6 with $N_{\kappa+\epsilon} = 0$; the dashed line is Eq. 6 with $\kappa^* + \epsilon^*$ = 101 mN·s/m. In both cases, the contact angle α is assumed to be zero.

shown in Figure 5. The conclusion is that with 95% confidence limits

$$A^2G(\kappa^* + \epsilon^*) = 25.3 \pm 0.8 \text{ mN} \cdot \text{s/m}$$

A similar least squares fit of Eq. 12 to the data for two runs in tube 2 is given in Figure 6, with the conclusion that

$$A^2G(\kappa^* + \epsilon^*) = 27.0 \pm 0.4 \text{ mN} \cdot \text{s/m}$$

For this system, $N_\mu = 50.8$. With $0° \leq \alpha \leq 30°$, Eq. 8 requires $A = 0.5$. (See also Giordano and Slattery, 1983, Figure 4.) For the same system, Jiang et al. (1983) have measured $\epsilon^* \sim 1$ mN·s/m. From Eq. 9, we see that

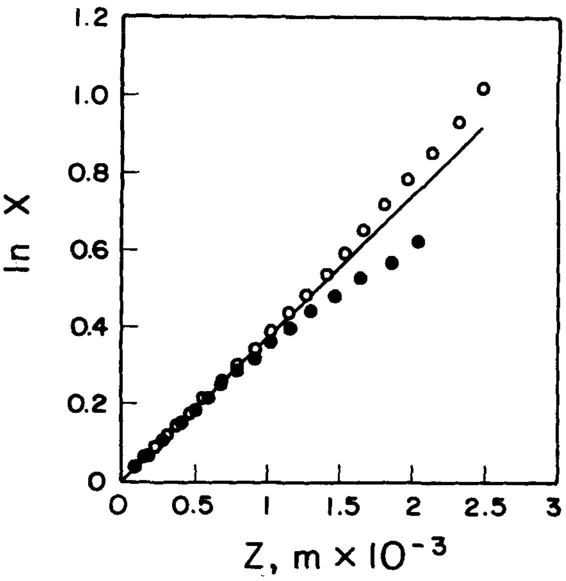

Figure 5. Data for two runs in which a solution-air interface is displaced through tube 1, plotted as suggested by Eq. 12. The solid line is the least squares fit of Eq. 12 to the data, suggesting $\kappa^* + \epsilon^*$ = 101 ± 3 mN·s/m.

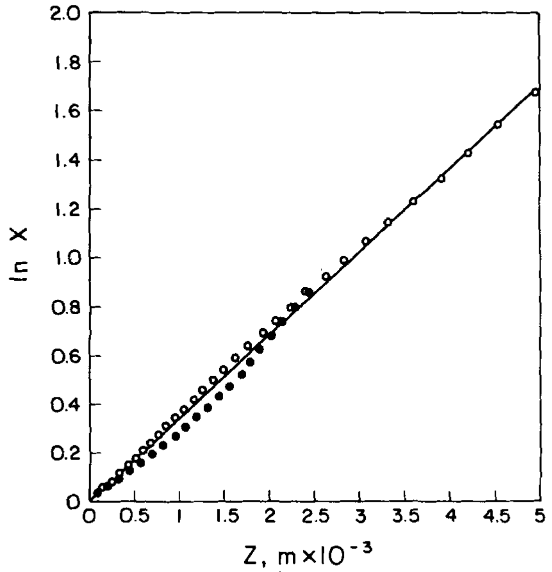

Figure 6. Data for two runs in which a solution-air interface is displaced through tube 2, plotted as suggested by Eq. 12. The solid line is the least squares fit of Eq. 12 to the data, suggesting $\kappa^* + \epsilon^*$ = 108 ± 2 mN·s/m.

$$\text{limit}\ \frac{\kappa^*}{\epsilon^*} \to \infty: \quad G = 1 + \sin\alpha \tag{18}$$

If we assume $\alpha = 0$, Eq. 18 implies that $G = 1$. With these observations, we conclude that for the two runs in tube 1

$$\kappa^* + \epsilon^* = 101 \pm 3 \text{ mN} \cdot \text{s/m}$$

and for the two runs in tube 2

$$\kappa^* + \epsilon^* = 108 \pm 2 \text{ mN} \cdot \text{s/m}$$

There is no apparent effect of tube diameter and the surface dilatational viscosity appears to be two orders of magnitude larger than the surface shear viscosity.

In the displacement of a solution-air interface, the equilibrium height $L_{eq}^{(1)*}$ was approached slowly. Comparison with Eq. 1 indicated that the equilibrium contact angle was substantially larger than the apparent dynamic contact angle.

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NOTATION

A = defined by Eq. 8
g^* = acceleration of gravity
G = defined by Eq. 9
h^* = position of liquid-gas interface in Teflon tubing for $t^* > 0$ measured with respect to the bottom of the capillary
h_0^* = position of liquid-gas interface in Teflon tubing for $t^* < 0$ measured with respect to the bottom of the capillary
L^* = total length of capillary
$L^{(1)*}$ = position of liquid-gas interface at any time t^* measured with respect to the bottom of the capillary
$L_{eq}^{(1)*}$ = equilibrium position of liquid-gas interface measured with respect to the bottom of capillary
$L_0^{(1)*}$ = initial position of liquid-gas interface measured with respect to the bottom of capillary
N_{Bo} = bond number defined by Eq. 3
$N_{Re}^{(j)}$ = Reynolds number of phase j defined by Eq. 2
$N_{\kappa+\epsilon}$ = defined by Eq. 4
N_{μ} = defined by Eq. 7
$P_{\nu-1}^{-1}$ = associated Legendre function of the first kind, where ν is determined by Eq. 10
$\Delta\mathcal{P}^*$ = pressure difference (including hydrostatic effects) between open end of capillary and open end of Teflon tubing, which in this case is zero
R^* = Radius of capillary
S = defined by Eq. 14
t^* = time
V^* = speed of displacement of liquid-gas interface
X = defined by Eq. 15
Y = defined by Eq. 17
Z = defined by Eq. 13

Greek Letters

α = contact angle measured through liquid phase
γ^* = surface tension
ϵ^* = surface shear viscosity
κ^* = surface dilatational viscosity
$\mu^{(j)}$ = viscosity of phase j
ν = determined by Eq. 10
$\rho^{(1)*}$ = density of liquid (displacing) phase
$\rho^{(2)*}$ = density of gas (displaced) phase

Others

$\ldots^{(1)}$ = superscript indicating liquid (displacing) phase
$\ldots^{(2)}$ = superscript indicating gas (displaced) phase
$\ldots^*$ = superscript indicating dimensional quantity, in referring to Giordano and Slattery (1983)

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