



Measuring contact angles inside of capillary tubes with a tensiometer



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ABSTRACT

We describe a new tensiometry method that allows for determination of wetting inside small diameter tubes or hollow fibers, where the maximum force from the ultimate rise height of liquid is used to estimate advancing contact angles. The technique was first validated with transparent tubes of glass, poly(carbonate) (PC) and poly(tetrafluoroethylene) (PTFE) using four liquids: isopropanol, silicone oil, ethylene glycol and water. Advancing contact angles measured with the tensiometer agreed well with those estimated from final rise height. As this tensiometry technique does not require a view of the liquid, it can be used to measure the wettability inside opaque tubes. We demonstrated this with poly(ether ether ketone) (PEEK) tubes.

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1. Introduction

Contact angle measurements are used widely to assess wettability, cleanliness, surface chemistry and the potential for creating a high strength adhesive bond [1]. The most common method of measuring contact angles involves the use of sessile drops on flat surfaces [2,3]. Alternatively, tensiometers are frequently used to evaluate curved solids, such as rods and fibers. Traditionally in tensiometry, the solid is immersed or withdrawn from a liquid while measuring force [4]. Proper analysis of the interplay between capillary forces and buoyancy allows for indirect estimation of contact angles [5,6].

Tubes and fibers produced for industrial applications may have different inner and outer surfaces. For example, during extrusion of plastic tube, molten polymer usually passes through a liquid cooling tank where its outer surface is rapidly quenched while in contact with various sizing fixtures; in contrast, its inner surface cools slowly in the presence of a gas without further mechanical manipulation. Consequently, the inner and outer surfaces of extruded tubes and hollow fibers may have distinct morphologies. Moreover, the chemistry of the inner surface may be intentionally modified to improve hydrophobicity and/or the outer surface may be rendered more hydrophilic to improve adhesion.

In recent work, we developed a new tensiometry technique for evaluating the wetting inside capillary tubes and hollow fibers [7]. Rather than immersing, capillary tubes of diameter D were brought into contact with a liquid of surface tension γ and density of ρ , such

that their bottoms just touched the surface of the liquid, Fig. 1. A meniscus formed around the inner and outer diameter, wetting the inside of the tube with a contact angle of θ . If θ was $<90^\circ$, liquid rose upward (z -direction), reaching a final height of h . We isolated the forces associated with liquid rising inside the tubes and then used those values to estimate work, viscous dissipation and potential energy change. Here, we show how our experimental technique can be used to estimate contact angle inside of capillary tubes or hollow fibers.

2. Theory

Capillary rise generates forces that can be measured with a tensiometer. The force inside the tube (f_i) exhibits a maximum value ($f_{i,max}$) when the liquid reaches its ultimate height (h). The maximum force can be estimated from the product of the hydrostatic pressure of the liquid column and the cross-sectional area of the capillary tube,

$$f_{i,max} = \rho gh \cdot \frac{\pi}{4} D^2, \quad (1)$$

where g is the acceleration due to gravity. The final rise height is determined by balancing the forces associated with the hydrostatic pressure in the liquid column and the capillary forces that arise from interactions in the vicinity of the contact line [8],

$$h = \frac{4\gamma}{\rho g D} \cos \theta. \quad (2)$$

Combining Eqs. (1) and (2) yields an expression that allows for estimation of contact angle inside a capillary tube from the maximum force, the tube diameter and the surface tension of the liquid,

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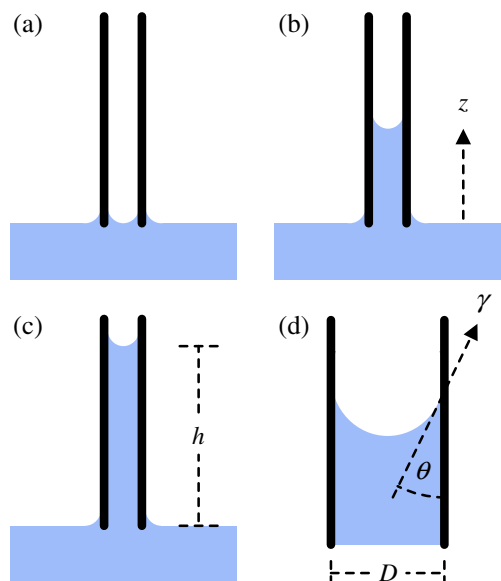


Fig. 1. A depiction of capillary rise in a small diameter (D) tube. (a) The tube contacts the liquid. (b) Liquid with a surface tension and density of γ and ρ rises vertically inside the tube. (c) The liquid stops at a final height of h . (d) A close-up view of the meniscus.

$$\theta = \text{ArcCos} \left(\frac{f_{i,\max}}{\pi D \gamma} \right). \quad (3)$$

3. Experimental details

The liquids used in our experiments were isopropanol (IPA, Brenntag, 99%), silicone oil (Aldrich, AP 100, polyphenylmethylsiloxane), ethylene glycol (Sigma-Aldrich, anhydrous 99.8%) and de-ionized (DI) water. The surface tension (γ), density (ρ) and viscosity (μ) of the liquids are given in Table 1. Surface tension was measured with a DCAT 11EC tensiometer by default Du Nuoy ring method; error was ± 1 mN/m. Measured γ values generally agreed with published data [1,2,9]. Densities and viscosities of the liquids were taken from the suppliers' data sheets and the open literature [9].

Glass, polycarbonate (PC), polyetheretherketone (PEEK) and polytetrafluoroethylene (PTFE) capillary tubes of constant diameter and wall thickness were used in the experiments. The PEEK tubes were opaque; the others were transparent. The glass tubes were purchased from CTech Glass Company (Part #s CT95-02, CT95-03, CT95-04), PC tubes from Paradigm Optics, Inc. (Part # CTPC562-750-5), PEEK tubes from IDEX Health and Science (Part # 1568-12x, Tub PEEK Nat 1/32x015x12/10pk) and PTFE tubes from Zeus Inc. (Part # 17536). Nominal inner diameters (D) ranged between 0.38 and 1.0 mm, corresponding outer diameters (D_o) between 0.7 and 1.3 mm. Tubes were chosen such that D_o was within $\pm 1\%$ of the nominal value, reducing the error in D to ± 0.01 mm. Tubes were broken or cut to fit the instrument. Glass tubes were cleaned immediately before use with oxygen plasma.

Table 1
Properties of the liquids.

Liquid	γ (mN/m)	ρ (kg/m ³)	μ (mPa s)
Isopropanol	22	786	2.0
Silicone oil	27	1060	100
Ethylene glycol	48	1110	20
Water	72	998	1.0

Our tensiometer (DataPhysics DCAT 11EC) has a load cell with a full scale of 210 g (2 N) and 0.1 mg (1 μ N) resolution. The sampling rate was set at 1–30 Hz, depending on the time scale of rise. For the capillary rise experiments, tubes were attached to the load cell, the load cell was zeroed and then the liquid reservoir was raised at 0.1 mm/s speed until the bottom of the capillary just touched the surface of the liquid. Time (t) and mass of the rising liquid were measured using the resident software. Masses were converted into forces by multiplying by $g = 9.81$ m/s². For sufficiently transparent tubes, the final height (h) was measured after each experiment using a ruler. We independently measured the contact angles (θ) of our tubes from the final rise height and the properties of liquid [8],

$$\theta = \text{ArcCos} \left(\frac{\rho g h D}{4\gamma} \right). \quad (4)$$

All measurements were made at $(20 \pm 1)^\circ\text{C}$.

We estimate the uncertainty in our parameters to be ± 0.01 mm for D , ± 1 mN/m for γ , ± 2 kg/m³ for ρ , ± 1 μ N for $f_{i,\max}$ and ± 0.5 mm for h . As indirect estimates of θ from h and $f_{i,\max}$ are functions of cosine, uncertainty in both is greater for near-zero θ values – roughly $\pm 5^\circ$ where $\theta < 10\text{--}15^\circ$ – and $\pm 2^\circ$ for greater values.

4. Results and discussion

To indirectly estimate contact angles inside of a capillary tubes using a tensiometer, we must extract the forces associated with the rising liquid. Fig. 2 shows an example of force (f_m) versus time (t) curves for ethylene glycol rising inside a lyophilic glass tube with $D = 0.8$ mm. Two curves are depicted, where $f_m = f_b$ or f_o . The data for the curve denoted f_b was generated by raising the liquid reservoir of the tensiometer until the bottom of the empty tube just touched the surface of the liquid. As ethylene glycol rose, the total force (f_b) was measured over time. The total force, f_b , shown in Fig. 2 is the sum of the force from liquid rise inside the capillary tube (f_i) and the force of the meniscus (f_o) that formed around the outer diameter of the tube.

To isolate f_i , additional experimental steps were performed [7]. First, the liquid reservoir was lowered to detach the capillary from the liquid. This step removes the outer meniscus from the base of the tube, but does not significantly change the height of the liquid inside the tube. The tensiometer was zeroed and then the liquid reservoir was raised again to contact the bottom of the filled tube. The outer meniscus reformed around the base of the tube. The resulting outer meniscus force (f_o) is also shown in Fig. 2. By subtracting the final or maximum values of outer meniscus force from

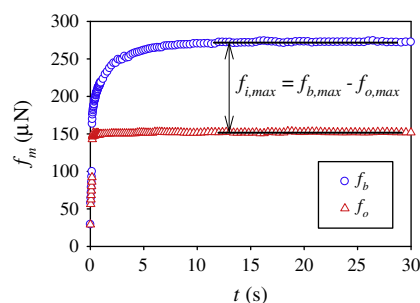


Fig. 2. Force (f_m) versus time (t) for the transient rise of ethylene glycol in and around a lyophilic glass tube with an inner diameter of $D = 0.8$ mm, where $m = b$ for the initial contact of the empty capillary to the surface of the liquid and $m = o$ for second contact of the capillary containing a liquid column. Thus, f_b is the sum of the forces from rise inside the tube and meniscus formation on the outer diameter of the tube; f_o is the outer meniscus.

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