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Influence of head resistance force and viscous friction on dynamic contact angle measurement in Wilhelmy plate method

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A R T I C L E I N F O

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A B S T R A C T

Wilhelmy plate method measures and calculates dynamic contact angle based on force balance equation of the test plate (or fiber). The equation doesn't consider the resistance force which may result in a measurement error at high testing speed or for plate with non-negligible thickness. In this work, the influences of two ignored resistance forces, i.e. head resistance force and viscous friction, on dynamic contact angle were investigated both theoretically and experimentally. An auxiliary optical facility was designed and added to the tensiometer in order to get dynamic contact angle by direct optical method to verify the validity of the force balance method's result. A series of experiments for silicone oils with various viscosities on quartz plates with different thicknesses were done and proved that the two above forces cannot be ignored when *Ca* got larger or the plate got thicker. A new force balance equation was established, in which the fitting results of both head resistance force coefficient and viscous friction coefficient were nearly constant for all the testing twelve systems. By using new equation, the accuracy of dynamic contact angle testing results were obviously improved especially at high *Ca*.

1. Introduction

The Wilhelmy plate method is widely used to study the static and dynamic contact angle of fluids on solid substrates [1–3]. When a plate (or a fiber) is partially immersed into a pool of liquid with a certain speed, the total forces keeping the plate balance can be related to surface tension force, buoyancy force, and the detected lifting force acting on the plate by microbalance, in which the weight of the test solid substrate is zeroed before the test. This technique does not involve a direct measurement of the contact angle, but determine the contact angle by the force balance equation [4–7]

$$F = L\sigma_{LV}\cos\theta_D - \rho g S Z \tag{1}$$

where F is the detected lifting force, L is the wetting length, θ_D is advancing contact angle, σ_{LV} is the surface tension, ρ is the liquid density, g is the gravitational acceleration, S is the cross-sectional area, and Z is the length of the test solid below the liquid, i.e., immersion depth. The first term and second term on the right represents surface tension force and buoyancy force respectively. F vs Z wetting curve showing in Fig. 1 can be recorded during the immersion process by the microbalance, and Eq. (1) allows one to obtain θ_D using the intercept of the curve when the surface tension of the liquid is already known [8,9].

Owing to the very high sensitivity of the microbalance, Wilhelmy plate method is expected to measure a more accurate contact angle than other experimental method that is currently available, for example, sessile drop method [10–14]. However, the overall solid/liquid interaction is not completely controlled by the forces in Eq. (1), there are still two neglected forces which may result in test error for θ_D. But the error is usually negligible in low-viscosity and low velocity conditions [15,16]. Thus the test plate's thickness is required to be much smaller than the other two dimensions [17] and also, the test velocity is limited to a low value. However, for some special materials (such as wood), the test plate cannot be made to be very thin and dynamic contact angle at high speed is also important [18]. Thus, a systematic analysis is essential to understand the influence of the ignored forces on θ_D.

One ignored force in Eq. (1) is a resistance force acting on the bottom surface which is greatly influenced by the smallest dimension of the plate. This force does not include the buoyancy forces and is caused by the relative motion between solid and liquid. It is more like an impact force which may be related to the impact velocity, density of the liquid and so on [19]. It is defined as head resistance force in this work. Some researchers believed that this force does exist, but is small and

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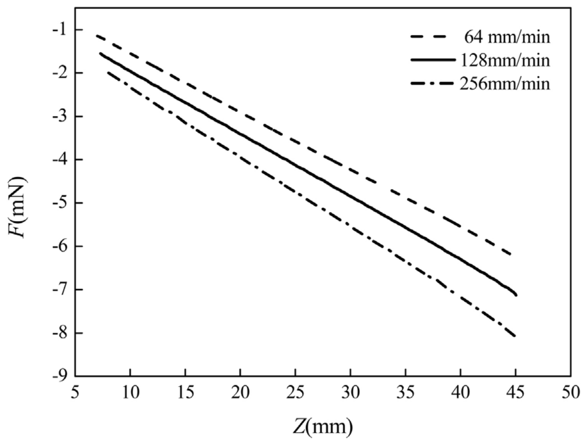

Fig 1. Detected force (F) changes with immersion depth (Z) for silicone oil/quartz plate wetting system.

can be neglected when the cross-sectional area of the test plates are small enough or the impact velocity is low enough [16,17,20,21]. But until now, there is still no systematic research on how the force influence dynamic contact angle's measurement in Wilhelmy plate method, and no quantitative analysis can be offered to verify the validity of ignoring the head resistance force so far.

Another ignored force acting on the solid substrate when the plate moves into or out of the fluid is the viscous friction on the sides of the plate [16,17]. The *F-Z* curves under different contact velocity are expected to be parallel lines according to Eq. (1) accounted by constant buoyancy at a certain *Z*. But actually, *F-Z* line becomes steeper as the contact velocity increase as shown in Fig. 1, which had been reported due to the viscous friction, and the viscous friction becomes important when the speed or viscosity increase [16]. Theoretical analysis on the viscous effect at low capillary number had been progressed [17], but there is still no clear conclusion on how viscous friction influence the test result of θ_D, especially at high testing speed.

In this study, a new force balance equation considering head resistance force and viscous friction is proposed. A series of experiments were done to quantitatively study the influence of the two new added forces on dynamic contact angle measurement. The test liquids were silicone oils with different viscosities and the solid substrates were quartz plates with different thickness. The contact velocity changes from 0.5 mm/min to 500 mm/min whose Capillary number (*Ca*) are wider than most of previous experimental studies using Wilhelmy plate technique (velocity and *Ca* are usually smaller than 150 mm/min and 1 in previous works) [6,22–28]. Wilhelmy plate tensiometer is used to get dynamic contact angle $\theta_{D,force}$ by force balance method. An auxiliary optical facility is added to get dynamic contact angle $\theta_{D,optical}$ by a direct image processing simultaneously. The comparison between $\theta_{D,force}$ and $\theta_{D,optical}$ proved that two extra terms must be added to Eq. (1) in order to account for the contribution of the head resistance force and viscous friction on the plate surface especially at high contact velocity. Two resistance coefficients were regressed from the experimental data. And the applicability of the original force balance equation was analyzed in the last section.

2. Materials and methods

2.1. Instrumentation

A widely used commercial equipment, K100MK2 tensiometer (Krüss GmbH, Hamburg, Germany) based on Wilhelmy plate method was used in this work. A schematic diagram of the equipment is presented in the dotted box in Fig. 2. The test plate was attached to the microbalance with a clamp, and the liquid reservoir on the mobile platform could rise up or down in order to immerse or withdraw the solid plate. The *F-Z* data was measured by the microbalance and recorded by a built in software. The software adopted the original Wilhelmy plate method, and use Eq. (1) to calculate $\theta_{D,force}$.

In order to verify the validity of $\theta_{D,force}$, an extra optical auxiliary system is designed and added to the tensiometer to record a video of the immersing process, and directly measure $\theta_{D,optical}$ from the snapshots of the video. Thus, the modified equipment can get $\theta_{D,force}$ and $\theta_{D,optical}$ at the same time.

The optical auxiliary system is consisted by a CCD (TOSHIBA Teli TK5573AO ROHS), a camera lens(Computar MLM-10X), an adjustable parallel light source (CCS PD3-3024-3-PI) and a self-made light board, which is shown in the solid box in Fig. 2. This optical auxiliary system moves up and down with the liquid reservoir. The CCD record the whole wetting process. Fig. 3 shows the snapshots of the menisci formed on test plate during immersion process in silicone oil at different immersion velocity. The sharpness of the image near the contact line, the stability of the contact line position, and the invariance of the interface shape indicate that the contact line is stable and measureable. The dynamic contact angle is measured directly by protractor. This optical auxiliary system can only measure contact angle from 0 to 90 degree because the CCD cannot get a reliable image when the menisci is below the liquid level.

The surface tension of test liquids was also tested on the K100MK2 tensiometer. Plate roughness was measured by AFM (Veeco Nanoscope IIIa Scanning Probe Microscope).

2.2. Materials

The test solid substrates are four quartz plates with different thickness and similar width, the geometry parameters of which are listed in Table 1. The roughness of quartz plates (1.16 nm) is much smaller than 0.1um, so roughness has little influence on the dynamic contact angle measurement [29–32]. The test fluids are silicone oils (Sigma–Aldrich) with different viscosity, whose physical properties are listed in Table 2.

2.3. Operation procedure

The experiments were done at room temperature (25 ± 3 °C) with a humidity of 40 ± 5%. Clean quartz glass is obtained by a sets of cleaning process by detergent, ultrapure water, 99.7% ethanol, acetone via ultrasonication. After cleaned, each plates were dried by compressed nitrogen before operation.

3. Theory

In this study, the test plates were assumed to be rigid body, so the free vibration and deformation force of the plate are not considered. A new force balance equation based on Eq. (1) which considers head resistance force F_h and viscous friction F_v is expressed as following

$$F = L\cdot\sigma_{LV}\cdot\cos\theta_D - \rho gSZ - F_v - F_h \quad (2)$$

And the schematic force diagram of the plate shows in Fig. 4.

3.1. Head resistance force F_h

When a plate is partially immersed into a liquid pool, the relative velocity of liquid and is decreased greatly due to fluid's viscosity near the bottom surface of the plate where a high pressure region is formed. Thus, the pressure between the bottom surface of the plate and the ambient causes the head resistance force putting on the plate. Early investigation can be traced back to the pioneering work of Th. von Karman [19]. For a flat-bottomed float, it is deduced that the peak pressure of the bottom (p) is related to the speed of sound in the fluid (c), the water density (ρ), contact velocity (U), that is $p = c\rho U$ [19,33]. But Chuang's experimental work demonstrated that the maximum of p