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CONTACT ANGLES OF PENDANT DROPS ON ROUGH SURFACES

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ABSTRACT

Study of contact angles and shapes of pendant drops on a substrate are important for the understanding of many engineering processes. Imaging, analysis and simulation of pendant glycerin drops on physically textured surfaces have been performed in this work for varying surface inclinations. The effects of surface roughness, plate inclination, drop volume and substrate material on the pendant drop shapes and advancing and receding angles have been discussed. Plate roughness generated using the process of hand lapping is without any pattern. Two types of contact angles have been reported. In one approach they are measured using a tangent method by curve fitting the data close to the wall. In the second, the Young–Laplace equation is first solved. The numerical drop shape is fitted to the experimental drop profile using an inverse method. The Young–Laplace equation has been numerically solved by Surface Evolver, an open source software. Here, a variational method wherein the overall potential energy of the three-dimensional pendant drop is minimized is made use of. The inverse method developed in this work calculates the advancing and receding angles by minimizing the error between numerical and experimental drop shapes. The following results have been obtained. Inclining the surface with respect to the horizontal causes a continuous reduction in receding angle of the pendant drop whereas the advancing angle remains nearly constant. The three-phase contact line is not pinned and its shape does not remain circular. Contact angles reduce as the surface roughness is increased for hydrophilic pendant drops. For a horizontal surface, contact angle is independent of drop volume but the receding angle shows dependence on inclination.

1. INTRODUCTION

Study of static contact angles and shapes of pendant drops on a substrate is important for the understanding of various engineering processes. Examples include dropwise condensation, bio-MEMS applications, drug delivery applications, lab-on-chip processes, and adhesive technology. Contact angles carry information about surface properties including wettability and surface energy. An important application in the study of drops is dropwise condensation (Sikarwar et al., 2011). Dropwise condensation is favored over the filmwise owing to its high heat transfer coefficient and control of the condensation rate. Here, the knowledge of the three-phase contact angle distribution of the liquid condensed on the substrate is essential. Contact angles, in turn, govern interfacial and body forces acting on sessile and pendant drops.

The surface chemistry of solid-liquid combinations is an active subject of research and contact angles of various solid-liquid combinations have been reported (Lee, 1999). Much of the data is applicable to sessile drops owing to the ease of experimentation. Although there is an abundance of data on contact angles, there exists a gap in the knowledge of contact angles of pendant drops on inclined surfaces. Pendant drops are difficult to deal with in experiments, especially when volumes of the order of microliters are concerned. Cheng et al., (1990) performed experiments to measure contact angles of pendant axisymmetric glycerin and water drops.

When the surface on which the pendant drop is deposited is inclined with respect to the horizontal, the advancing angle is greater than the receding angle. These angles are a function of plate inclination. Overall, the drop is deformed and becomes non-axisymmetric. Brown et al. (1980), ElSherbini and Jacobi (2004) and Annapragada et al. (2012) have discussed the effect of plate inclination on the advancing and receding angles of sessile drops. The present study examines the effect of substrate inclination on advancing and receding angles of pendant drops.

Factors such as surface energy, surface roughness, drop size, substrate inclination and substrate vibration affect contact angles. Temperature is also an independent variable though it can be accounted for through fluid properties. In the present work, the effect of surface roughness, inclination and drop volume on contact angles of pendant drops is examined. The difference in advancing and receding angles is the contact angle hysteresis (CAH). One method of altering the wettability of a surface is to treat it chemically by grafting or adsorbing molecules with specific wetting characteristics. The effect of chemical texturing has been studied by various authors. Lee (1999) used physical vapor deposition to place a monolayer of stearic acid on a glass substrate to alter its wettability. In applications where the liquid is highly corrosive, for example, metal vapor condensation, the chemically textured surface has a short lifespan. The chemical monolayer is leeched away by the liquid and needs to be replaced often. This shortcoming has created a need to alter the surface properties by introducing surface roughness (Quéré, 2002; Barthlott and Neinhuis, 1997). Abdelsalam et al. (2005) used electrodeposition to produce structured gold-plated surfaces. Bhushan and Jung (2011) deposited carbon nanotubes on a surface to develop nanoscale roughness patterns. In the present work, a process of hand lapping is used to produce surfaces with random roughness patterns, instead of chemical texturing. Experiments have been performed on physically textured aluminum and copper surfaces to measure contact angles and shapes of deformed pendant drops.

A liquid droplet can sit on a solid surface in two distinct configurations. It is said to be in Wenzel state when it is conformal with the topography and Wenzel's equation can be used to compute the apparent contact angle. The other state in which a droplet can rest on the surface is the Fakir state where it touches the peaks of the protrusions on the surface. Textured surfaces have been synthesized to control surface energy gradients to obtain very large contact angles and low CAH (Bico et al., 1999). Such surfaces have a contact angle of greater than 150° and CAH less than 10° and are termed as super-hydrophobic. Lotus leaf has a hierarchical roughness distribution and has been characterized by many authors including Barthlott and Neinhuis (1997), Bico et al. (1999) and Bhushan and Jung (2011). Surfaces with designed energy gradients can be useful in controlling the flow of drops for low or no plate inclination and is an area of extensive research (Chaudhury and Whitesides, 1992; Shastry et al., 2006).

Besides measuring the contact angles at the base of a drop, it is important to obtain the shape of the three-phase contact line. The contact line is actually in quasi-static equilibrium and changes with plate inclination angle (Berejnov and Thorne, 2007). In earlier studies Wolfram and Faust (1978) and Brown et al. (1980) assumed the contact line for a static drop to be pinned everywhere and circular in shape for horizontal and inclined plate configurations. ElSherbini and Jacobi (2004) showed that the three-phase contact line for a static drop does change with plate inclination and fitted the base contour by two ellipses sharing the same minor axis. Extrand and Kumagai (1995) measured the aspect ratios of base contours for water and ethylene glycol drops on polymers. Most of the work on contact lines is for sessile drops while the present study is for contact lines of pendant drops.

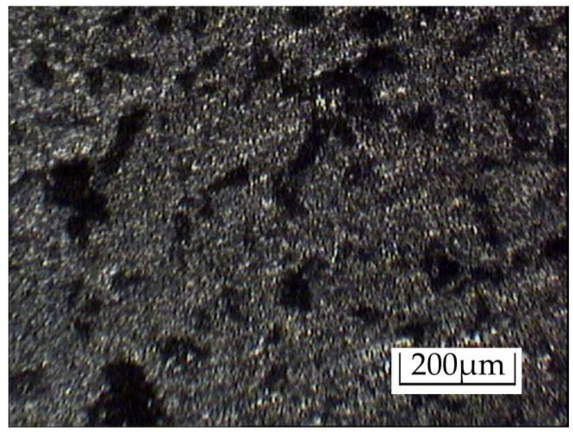
In the light of the discussions given above, experiments have been performed to tabulate advancing and receding angles of pendant drops for various plate inclinations, substrate roughness and drop volumes. Glycerin is chosen as the working fluid due to its low vapor pressure and volatility and high viscosity. Aluminum and copper are used as substrate materials. These surfaces are lapped to different roughness levels to study the effect of surface texture on contact angle. Computer programs to measure contact angles and drop profiles from optical images have been developed.

2. EXPERIMENTS

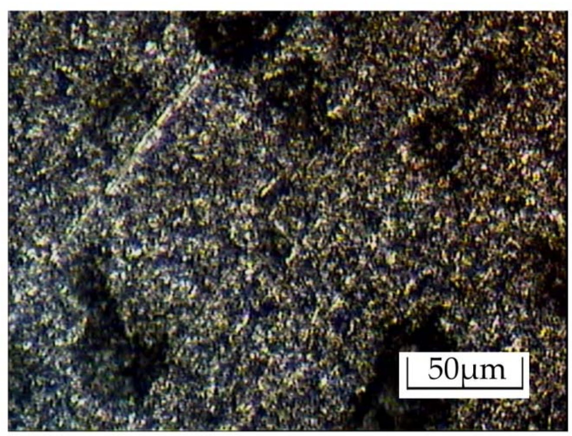
A contact angle measurement apparatus, developed as a part of this work, has been used to image pendant drops on inclined, physically textured surfaces. This apparatus can tilt the plate in steps of 2º. The pendant drop is imaged from a direction normal to the vertical plane of tilt, namely the plane of symmetry of the drop. A microliter syringe with 100 µL capacity and a least count of 2 µL has been used to deposit pendant drops on the underside of the substrate.

Aluminum and copper plates were hand lapped with lapping pastes of three different grades to produce RMS surface roughness of 0.5, 1.5 and 3.5 µm. Figure 1 shows an enlarged view of the lapped aluminum surface under a microscope for magnifications of 50 and 200. Microstructures are clearly visible with dark patches representing the valleys in a shiny metal pool. These microstructures show that the roughness created on the surfaces is randomly distributed with no definite pattern. Roughness of the lapped substrates was measured using a stylus based Mitutoyo's *Surftest SJ-301* machine. Figure 2 shows the roughness of a substrate measured by this apparatus. Out of the three roughness parameters reported by the device, RMS roughness (R_q) is used to characterize roughness of substrates in the work.

A color CCD camera, Basler A202k, used to image drops is a high resolution, progressive scan camera with a high signal-to-noise ratio. It is fitted with a macro lens to take enlarged pictures of the drop from a close distance without any wide-angle distortion. A dual channel frame grabber card is used to grab frames from the camera to be stored into the hard drive. Post processing of the images has been performed by programs written in WiT (Teledyne Dalsa) and MATLAB. Image processing programs have been developed to obtain contact angles and drop profiles from raw images. Color images are converted to grayscale and thresholded. A pixel-wise scanning algorithm extracts drop profiles and contact angles are determined using a tangent method.



(a)



(b)

Figure 1: Microstructures of lapped aluminum plate (roughness = 1.5 µm) viewed under a magnification of (a) 50 and (b) 200

Figure 2: Roughness profile measured by the roughness measurement instrument. Ra (average), Rz (maximum peak-to-valley distance) and Rq (RMS roughness) are the three roughness parameters for the surface. Rq has been used to report the roughness values

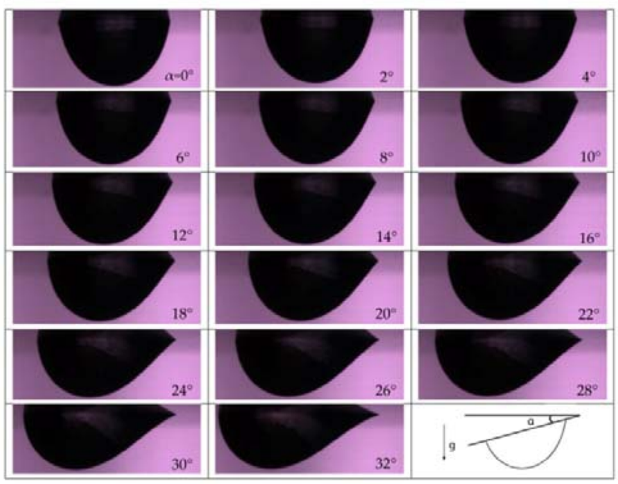


Figure 3: Pendant glycerin drop imaged as a function of plate inclination (α); substrate material = aluminum, plate roughness = 1.46 µm (RMS), drop volume = 30 µL

Drop volume, surface inclination, substrate roughness and substrate material are four parameters varied in experiments. Drop volume ranges from 5 to 30 µL, surface inclination from 0 to 44º, substrate roughness from 0.5 to 4 µm while the substrate materials used are aluminum and copper. The working fluid is glycerin, density ρ =1260 kg/m^3, surface tension γ =63.4 mN/m and viscosity μ =1.069 Pa-s. During experiments, the room temperature was maintained at 20±1°C. Figure 3 is a collection of pendant drop images of a 30 µL glycerin drop on an aluminum surface of 1.46 µm roughness. Change in the drop shape and contact angles with increasing plate inclination can be seen in this figure.

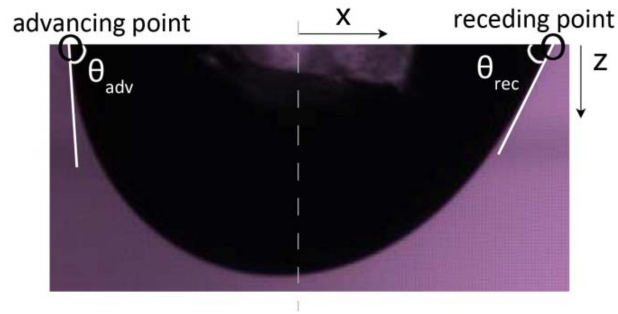


Figure 4: Image of a pendant drop with advancing and receding angles shown on an inclined plate

3. RESULTS AND DISCUSSIONS

Advancing and receding angles have been measured from images of pendant drops for a range of plate inclinations. The liquid considered is glycerin. The effect of volume and surface roughness are considered. Figure 4 shows schematically the advancing (θ_{adv}) and receding (θ_{rec}) angles for a pendant drop. The liquid drop is taken to be stationary in this work and instability and drop motion form the scope of future work.

3.1 Physical Texturing

Figure 5 shows the contact angle variation with surface inclination for pendant glycerin drops of volumes 5 and 30 µL. Advancing and receding angles are presented for three substrate roughness values, namely 0.55, 1.46 and 3.98 µm for an aluminum plate. It can be seen that increasing the roughness leads to a reduction in contact angle. The entire band of advancing and receding angles shifts down by an amount proportional to roughness and the band lines remain nearly parallel.

The extent of reduction in contact angle with increasing roughness of a horizontal surface can be seen from the three images in Figure 6. As per Wenzel's model:

$$\cos\theta^{*} = r'\cos\theta_E \tag{1}$$

where θ^{*} is Wenzel's contact angle and θ_E is Young's contact angle. For a horizontal plate, the contact angle for a perfectly smooth aluminum surface in Figure 6 is less than 90º $\left(\theta_E < 90°\right)$. Wenzel's equation predicts that if roughness $r' > 1$,

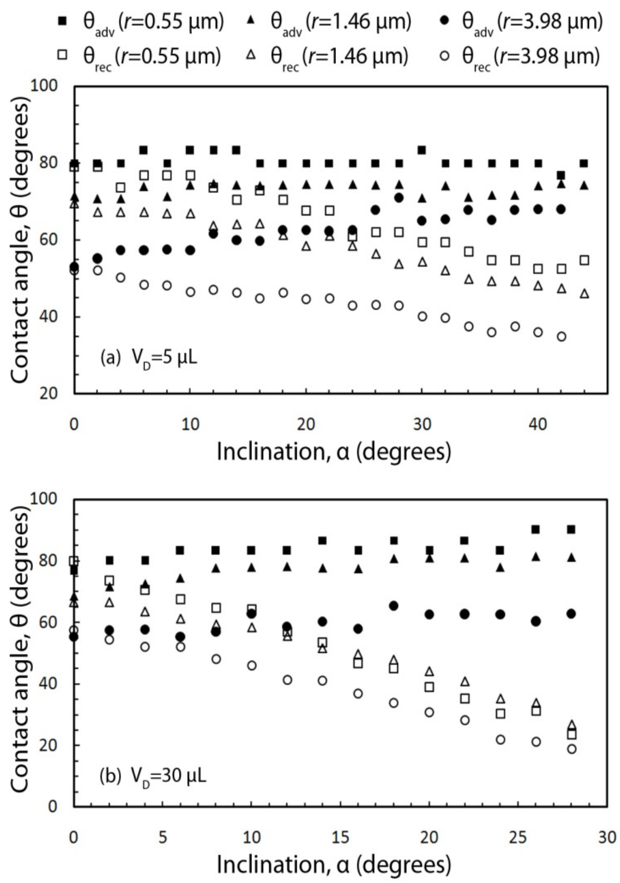


Figure 5: Contact angle variation of pendant glycerin drops on inclined aluminum substrates. Advancing and receding angles are plotted against inclination angle for various substrate roughness values: 0.55, 1.46 and 3.98 µm for drops of volumes (V_D) (a) 5 µL and (b) 30 µL

then $\theta^* < \theta_E$, that is, the apparent contact angle will be less than Young's contact angle. Experimental data presented in Figure 5 agree with this theory. If $\theta_E > 90°$, an increase in surface roughness will lead to an increase in the apparent contact angle.

3.2 Plate Inclination

The effect of plate inclination on advancing and receding angles is of major concern in engineering applications. Figure 7(a) presents the variation of advancing and receding angles with Bond number (Bo). Figure 7(b) is a plot of contact angles with plate inclination. Here, Bond number is given as:

$$Bo = \frac{\rho g D^2 \sin\alpha}{\gamma} \tag{2}$$

with the usual nomenclature. It represents a ratio of the gravity force to the surface tension force.

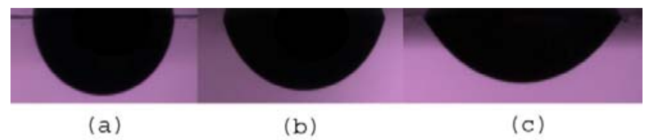


Figure 6: 5 µL pendant glycerin drops on aluminum substrates for roughness values of: (a) 0.5 µm (b) 1.46 µm (c) 3.98 µm. The increase in wettability with increasing surface roughness is evident from this image

Figure 7(b) shows that inclining the surface causes a monotonic reduction in receding angle. The advancing angle remains nearly constant for the range of plate inclinations (0–44º) used in the experiments. Receding angle normalized with the advancing angle can be considered as a function of the Bond number. The functionality has a correlation coefficient of 0.99 for a linear fit and the regression equation is obtained as:

$$\frac{\theta_{rec}}{\theta_{adv}} = 1 - 0.394Bo \tag{3}$$

Annapragada et al. (2012) performed a similar analysis for sessile drops and obtained

$$\frac{\theta_{rec}}{\theta_{adv}} = 1 - 0.298Bo \tag{4}$$

The advancing angle in the present study does not change significantly with plate inclination and hence

$$\frac{\theta_{adv}}{\theta_o} \approx 1 \tag{5}$$

for the plate inclination angles studied. Here θ_0 is the horizontal plate contact angle. Berejnov and Thorne (2007) performed inclination experiments with sessile drops and their advancing angles remained nearly constant with changing plate inclination whereas the receding angles showed a linear reduction. The present results for pendant drops are in agreement with the literature.

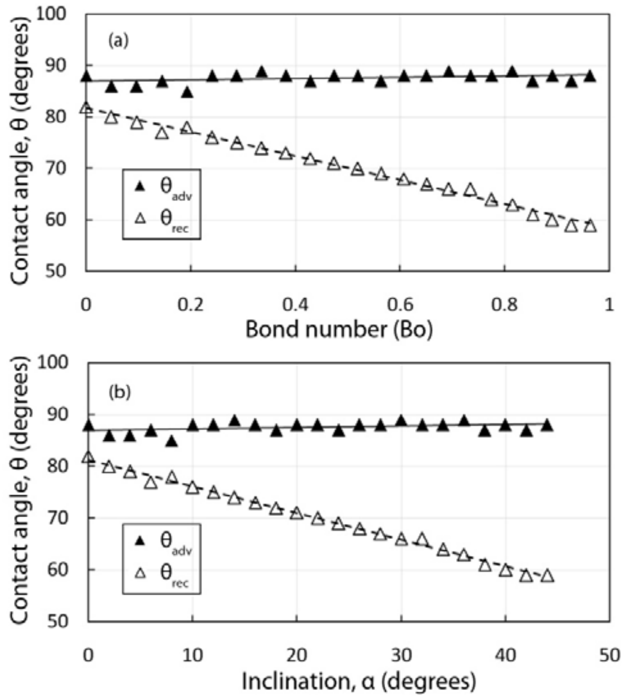


Figure 7: Advancing and receding angles plotted against (a) Bond number and (b) plate inclination, for a 5 μL pendant glycerin drop on an aluminum substrate (0.55 μm RMS roughness)

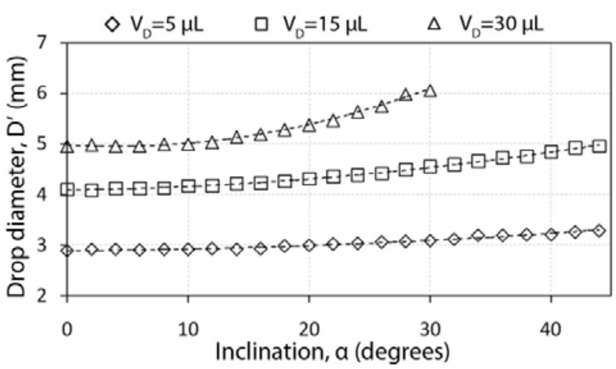


Figure 8: Variation of pendant drop diameter with plate inclination angle for three drop volumes (5, 15 and 30 μL). The plot is for a glycerin drop on an aluminum substrate (1.46 μm RMS roughness)

The receding point remains pinned whereas the advancing point starts moving at small plate inclinations. Although the pendant drop is stable, it is in a state of quasi-static equilibrium with its contact line changing continuously with plate inclination. Figure 4 shows the receding and advancing points on the contact line. An important observation is that the contact line of the glycerin drop does not remain pinned on textured aluminum and copper surfaces. Figure 8 shows the plot of the drop diameter D' with plate inclination angle for pendant glycerin drops of three volumes, namely 5, 15 and 30μL. The variation of drop diameter with plate inclination shows that the contact line is not pinned everywhere. The term drop diameter should be cautiously used as the base contour does not remain circular for non-zero plate inclinations, contrary to the assumptions of Wolfram and Faust (1978) and Brown et al. (1980). In Figure 8, the drop span along the plate inclination is termed the drop diameter (shown later in Figure 11Figure 11). A quadratic fit that presents a good fit to the drop diameter variation with plate inclination angle is

$$D' = 0.001\alpha^2 - 0.013\alpha + 4.977 \text{ for } V_D=30\ \mu L \quad (6)$$

$$D' = 0\alpha^2 + 0.003\alpha + 4.094 \text{ for } V_D=15\ \mu L \quad (7)$$

$$D' = 0\alpha^2 + 0.001\alpha + 2.897 \text{ for } V_D=5\ \mu L \quad (8)$$

Here D' is the drop diameter in mm, α is the plate inclination in degrees and V_D is the drop volume.

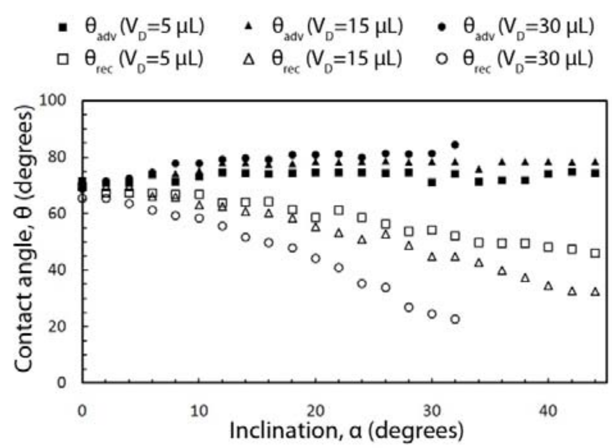


Figure 9: Advancing and receding angles of pendant glycerin drops on an aluminum substrate. Three drop volumes considered are (5, 15 and 30 μL) for a RMS surface roughness of 1.46 μm

3.3 Effect of Drop Volume

Figure 9 shows the contact angle plots for three drop volumes of 5, 15 and 30 μL. The plots clearly show that as the drop volume increases, the sensitivity of the receding angle with plate inclination increases. This observation can be interpreted in the following manner: Higher volumes show greater dependence on inclination in terms of a change in the receding angle. These results are similar to the sessile drop contact angles obtained by Berejnov and Thorne (2007). The horizontal plate contact angles are independent of drop volume, Figure 9, as long as the plate material and roughness are fixed.

4. NUMERICAL SHAPES OF DROPS

Young–Laplace equation represents force equilibrium for a static drop and is given in three dimensions as (Pozrikidis, 2009):

$$2\kappa_m = \frac{\Delta\rho}{\gamma}\vec{g}.\vec{z} + B \tag{9}$$

Here κ_m is the mean curvature of the liquid-air interface; $\Delta\rho = \rho_{liq} - \rho_{air}$; γ is the surface tension of the liquid; $B = \Delta p/\gamma$ where Δp is the excess pressure in the liquid contained within the drop. For a three-dimensional drop surface $z = f(x, y)$, the curvature of the drop is given by:

$$\kappa_m = \frac{1}{2}\frac{\left(1+f_x^{\,2}\right)f_{yy} - 2f_x f_y f_{xy} + \left(1+f_y^{\,2}\right)f_{xx}}{\left(1+f_x^{\,2}+f_y^{\,2}\right)^{3/2}} \tag{10}$$

Here x and y are the coordinates in the plane $z = 0$ of three-phase drop contact line as shown in Figure 4 Substituting Eq. (10) in Eq. (9) gives the governing equation for the shape of a non-symmetric drop in terms of the function *f(x,y)*. The governing equation is subject to the following boundary conditions:

$f(S) = 0$ where $S(x, y) = 0$ is the contact line of the drop (11)

$$\nabla f.\hat{n} = \left|\nabla f\right|\sin\theta \quad \text{on } S(x, y) = 0 \tag{12}$$

Here, $\hat{n}$ is a normal to the plane $z = 0$, θ is the contact angle which varies along the contact line.

Since the excess pressure Δp ($= B\times\gamma$) is an unknown, the governing equation is solved with a volume constraint

$$\int f(x, y)dxdy = V_D \text{ , given} \tag{13}$$

Solving Eq. (9) with the boundary conditions (11) and (12) and the constraint (13) is a challenging problem. A variational approach to compute the three-dimensional drop shape can be used by minimizing the overall potential energy of the drop. An open source software, Surface Evolver (Brakke, 1992), has been used in the present study to obtain static shapes of three-dimensional pendant drops on inclined surfaces.

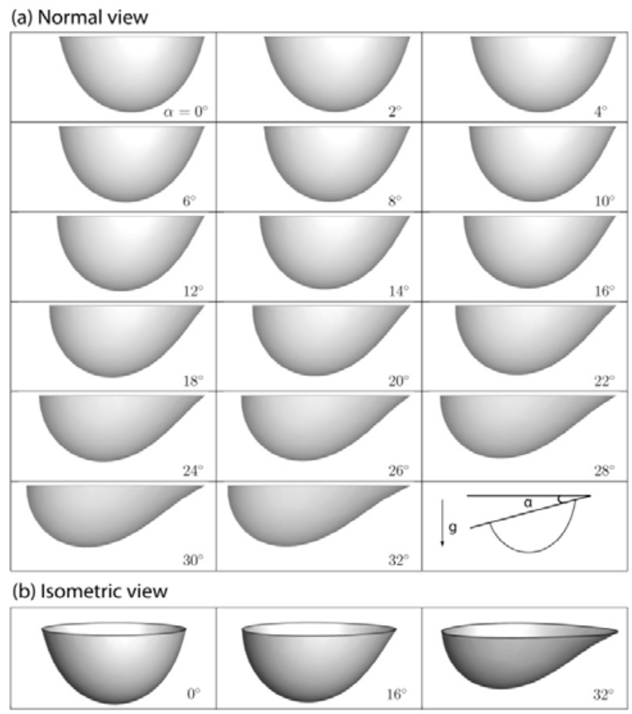


Figure 10: (a) Numerical simulation of a 30 µL pendant glycerin drop on an aluminum substrate of 1.46 µm RMS roughness for different plate inclination angles (α); (b) Isometric view of the simulated drops for three plate inclinations

Figure 10 presents three-dimensional drop images obtained from the numerical simulation of a 30 µL pendant glycerin drop for various plate inclinations. This simulation corresponds to the experimental images shown in Figure 3 for an aluminum substrate of 1.46 μm roughness. Figure 10(a) shows the simulated drops from a direction normal to the drop mid-plane and is comparable to Figure 3. Figure 10(b) is an isometric view of simulated three-dimensional drops for three plate inclinations.

Figure 11 presents the numerical analysis performed for a 15 μL pendant glycerin drop on an aluminum surface inclined at 16°. Figure 11(a) is the isometric view of the three-dimensional drop obtained from the solution of the mathematical model. Figure 11(b) presents a comparison between experimental and numerical drop shapes. Figure 11(c) is the base contour of the drop deformed under the action of gravity. It can be seen that the base contour does not remain circular as discussed in Section 3.2 and shown in greater detail in Figure 12.

Similar results have been obtained on a hand-lapped copper surface.

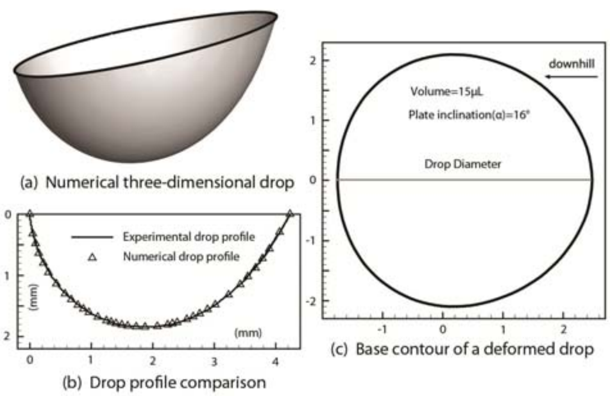


Figure 11: Numerical simulation of pendant non-symmetric drops on inclined surfaces using a variational method. (a) shows a 15 μL simulated pendant glycerin drop; (b) shows the comparison between experimental and numerical drop profiles; (c) is the three-phase contact line as obtained from the numerical drop model

5. CONCLUSIONS

Glycerin drops deposited on the underside of textured aluminum and copper substrates were imaged and analyzed for contact angle data and drop profiles. Experiments were performed by varying the plate inclination, drop volume and surface roughness. Advancing and receding angle data as a function of plate inclination was collected for static pendant drops. A numerical model based on open domain software was used to obtain three-dimensional shapes of non-symmetric drops. The following conclusions have been arrived at in the present work.

1. The three-phase contact line of pendant drops has a tendency to move and expand at even small plate inclination angles. The advancing point moved gradually, maintaining a nearly constant advancing angle, whereas the receding point remained fixed, with a linearly decreasing receding angle. This observation contradicts several drop pinning theories and agrees with the quasi-static pendant drop model.

2. Increasing surface roughness caused a reduction in contact angles. The results are in accordance with the Wenzel's model that predicts an increase in wettability for increased surface roughness values if the original smooth surface is hydrophilic.

3. The contact angle of pendant drop on a horizontal surface was not affected by volume. Receding angles fall with a greater slope for larger drop volumes, clearly showing the importance of body forces on deformation. For larger volumes, the drop became unstable at a smaller plate inclination and started moving earlier.

4. The numerical models predict the drop base contour for inclined pendant drops and provide a convenient alternative to the limitations of experimental techniques. The shape of the base contour deformed from circular as the plate was inclined.

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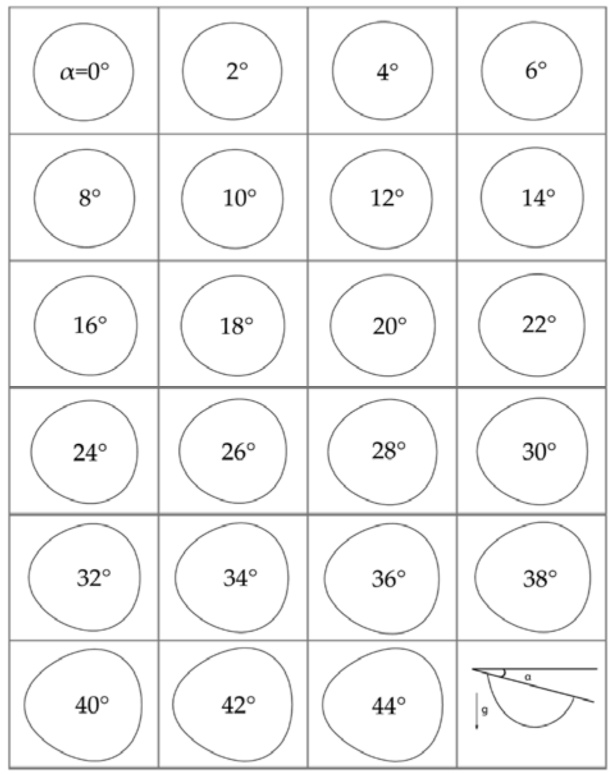

Figure 12: Numerical simulation of the three-phase contact line of a 15 μL pendant glycerin drop, on an aluminum substrate of 1.46 μm RMS roughness, for various plate inclinations (α). These contact lines have been extracted from the three-dimensional drop shapes. Drop diameter for the horizontal drop in this simulation is 4.1 mm

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NOMENCLATURE

B	Ratio of excess pressure inside the drop to the surface tension of liquid (m^{-1})
Bo	Bond Number = $\rho g D^2 \sin\alpha / \gamma$
D	Drop diameter for horizontal plate (m)
D'	Drop diameter for a drop on inclined plate (m)
$\vec{g}$	Acceleration due to gravity (m/s^2)
r	RMS roughness for a given surface (μm)
r'	Wenzel's roughness parameter = actual surface area / projected surface area
V_D	Volume of the drop (m^3)
z	*z*-coordinate, positive in the downward direction
α	Substrate inclination angle (radians)
γ	Surface tension (N/m)
Δp	Excess pressure inside the drop (Pa)
θ	Contact angle (radians)
θ_{adv}	Advancing contact angle (radians)
θ_{rec}	Receding contact angle (radians)
θ^*	Wenzel's contact angle (radians)
θ_E	Young's contact angle (radians)
θ_O	Horizontal drop contact angle (radians)
κ_m	Mean curvature of the liquid-air interface (m^{-1})
μ	Viscosity (Pa-s)
ρ_f	Density of fluid (kg/m^3)