Interfacial Tension

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2.2.1 Interfacial tension

- Let us consider two immiscible liquids in contact with each other as shown in Fig. 2.2.1.

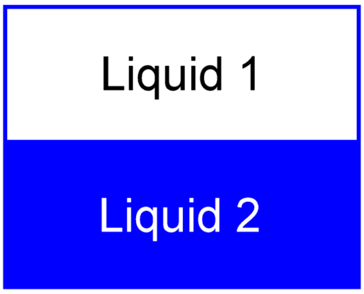


Fig. 2.2.1 Interface between two liquids.

- The molecules at the surface of both of these liquids experience unbalanced forces of attraction. These unbalanced forces at the surface of separation between the two immiscible liquids (i.e., at the interface) give rise to interfacial tension. It can be defined in the same way as the surface tension.
- Antonoff's rule predicts that the interfacial tension (γ_{AB}) between two liquids A and B will be equal to the difference between the respective surface tensions (i.e., γ_A and γ_B).

$$\gamma_{AB} = |\gamma_A - \gamma_B| \tag{2.2.1}$$

Therefore, it can be anticipated from this rule that the interfacial tension should lie between the surface tensions of the two liquids. This is indeed found to be true for many liquids (e.g., water and carbon tetrachloride). However, for many liquids, this prediction does not hold (e.g., water and cyclohexanol).

2.2.2 Girifalco–Good correlation

- Girifalco and Good (1957) incorporated the effects of the free energies of cohesion of the two phases and the free energy of adhesion on interfacial tension. They proposed the following equation for interfacial tension.

$$\gamma_{AB} = \gamma_A + \gamma_B - 2\Phi\sqrt{\gamma_A \gamma_B} \tag{2.2.2}$$

where Φ is a constant, which is defined as,

$$\Phi = -\frac{\Delta G_{AB}^{a}}{\sqrt{\Delta G_A^c \Delta G_B^c}} \tag{2.2.3}$$

where ΔG_{AB}^{a} is the free energy of adhesion for the interface between the phases A and B, ΔG_A^c is the free energy of cohesion for phase A, and ΔG_B^c is the free energy of cohesion for phase B.

- For many liquid–liquid systems, Φ lies between 0.5 and 1.2. It has been observed that for non-associated liquids, Φ lies between 0.5 and 0.8, and for the associated liquids, the value of Φ is higher.
- If the two phases are composed of spherical or nearly-spherical molecules, Φ can be related to the molar volumes of the liquids by the following equation.

$$\Phi = \frac{4\left(\bar{v}_A \bar{v}_B\right)^{1/3}}{\left[\left(\bar{v}_A\right)^{1/3} + \left(\bar{v}_B\right)^{1/3}\right]^2} \tag{2.2.4}$$

- The values of interfacial tension for several organic liquids with water at 293 K, and the values of Φ are presented in Table 2.2.1.

Table 2.2.1 Interfacial tension between water and immiscible organic liquids

Organic Liquid	Interfacial Tension (mN/m)	Φ
Aniline	5.8	0.98
Benzaldehyde	15.5	0.90
Benzene	35.0	0.72
Bromobenzene	38.1	0.69
Carbon disulfide	48.4	0.58
Carbon tetrachloride	45.0	0.61
Chlorobenzene	37.4	0.70
Chloroform	31.6	0.76
Cyclohexane	50.2	0.55

Cyclohexanol	3.9	1.04
Decalin	51.4	0.55
Dichloromethane	28.3	0.80
Ethyl acetate	6.8	1.08
Ethyl bromide	31.2	0.78
Iodobenzene	41.8	0.66
Isoamyl alcohol	4.8	1.11
Isobutyl alcohol	2.0	1.15
Isopentane	48.7	0.59
Mesitylene	38.7	0.67
m-Nitrotoluene	27.7	0.79
m-Xylene	37.9	0.69
n-Amyl alcohol	4.4	1.09
n-Butyl acetate	14.5	0.97
n-Butyl alcohol	1.8	1.13
n-Decane	51.2	0.55
n-Heptane	50.2	0.55
n-Hexane	51.1	0.55
Nitrobenzene	25.7	0.81
n-Octane	50.8	0.55
n-Pentane	49.0	0.58
Octanoic acid	8.5	1.03
o-Nitrotoluene	27.2	0.79
o-Xylene	36.1	0.71
p-Xylene	37.8	0.69
Tetrachloroethylene	47.5	0.59
Toluene	36.1	0.71

2.2.3 Fowkes correlation

- An improvement of the correlation of Girifalco and Good was proposed by Fowkes (1964). He suggested that in strongly polar or metallic liquids (such as mercury) the nonpolar dispersion forces must be treated differently from the hydrogen bonding or metallic forces.

- Fowkes assumed that the interaction between the immiscible liquid phases *A* and *B* involve only dispersion forces. The interfacial tension is given by the following equation.

$$\gamma_{AB} = \gamma_A + \gamma_B - 2\sqrt{\gamma_A^d \gamma_B^d} \tag{2.2.5}$$

  where γ^d is the contribution from dispersion forces to the surface tension of the pure liquid. For a nonpolar liquid, $\gamma = \gamma^d$. Therefore, this provides a method to determine the unknown γ^d.

- The interfacial tension between the strongly polar (or metallic) liquid and a nonpolar liquid is measured to calculate γ^d using Eq. (2.2.5). Nonpolar hydrocarbons can be used for this purpose.

Example 2.2.1: For mercury and water, the values of γ^d are 200 mN/m and 22 mN/m, respectively at 293 K. Using these values, compute the interfacial tension between water and mercury at the same temperature. Compare your result with the experimental value of 426 mN/m.

Solution: The surface tensions of water and mercury at 293 K are,

$$\gamma_{\mathrm{W}} = 72.8 \text{ mN/m}$$

$$\gamma_{\mathrm{Hg}} = 476 \text{ mN/m}$$

Given that,

$$\gamma_{\mathrm{Hg}}^d = 200 \text{ mN/m}$$

$$\gamma_{\mathrm{W}}^d = 22 \text{ mN/m}$$

$$\gamma = \gamma_{\mathrm{W}} + \gamma_{\mathrm{Hg}} - 2\sqrt{\gamma_{\mathrm{W}}^d \gamma_{\mathrm{Hg}}^d} = 72.8 + 476 - 2\sqrt{22 \times 200} = 416.1 \text{ mN/m}$$

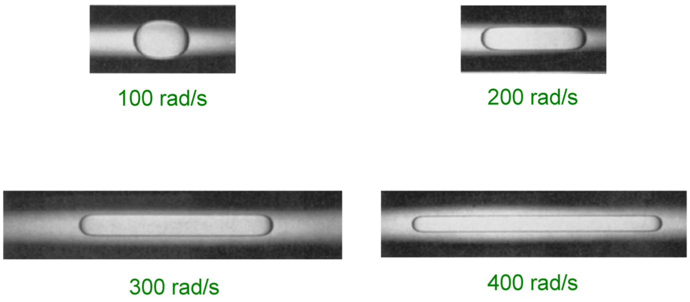
Therefore, this value compares well with the experimental value.

2.2.4 Measurement of interfacial tension

- The drop-weight method and the du Noüy ring method are extensively used for measuring the interfacial tension.
- In the drop-weight method, the drop of one liquid is formed inside the other liquid.
- In the du Noüy ring method, the ring pierces the liquid–liquid interface.
- A very useful technique for measuring the interfacial tension is the spinning-drop method. It is especially suitable for measuring ultra-low interfacial tensions in presence of surfactant mixtures, which are used in microemulsions.
- Interfacial tension can also be measured from the analysis of drop shape, which we will discuss later. In this Lecture, we will discuss the spinning-drop method.

2.2.4.1 Spinning-drop method

- In this method, a small drop is placed in a denser liquid enclosed in a glass tube which is subjected to rotation at a high angular velocity (say, 1000 rad/s) about its horizontal axis.
- The method is based upon the principle of gyrostatic equilibrium, which is the state of uniform rotation in which every bit of the fluid inside the tube is at rest with respect to the wall of tube. Gyrostatic equilibrium is achieved at high angular velocities when the gravitational force perpendicular to the axis of rotation is negligible as compared with the centrifugal force.
- When the tube rotates with high velocity, the drop migrates to the axis of rotation and assumes a cylindrical shape with hemispherical ends. For each angular velocity, the drop comes to an equilibrium shape which is characteristic of that velocity.
- Some of the shapes of the drop are shown in Fig. 2.2.2 at different angular velocities.



100 rad/s 200 rad/s

300 rad/s 400 rad/s

Fig. 2.2.2 Photographs of a heptane drop in glycerol at different angular velocities (source: H. M. Princen, I. Y. Z. Zia, and S. G. Mason, *J. Colloid Interface Sci*., **23**, 99, 1967; reproduced by permission from Elsevier Ltd., © 1967).

- The drop cannot elongate indefinitely. When the interfacial tension everywhere balances the centrifugal force that produces the pressure difference across the interface, the elongation ceases. The equilibrium condition exists because the force due to the rotating field tending to elongate the drop is proportional to the fourth power of the radius of the drop whereas the opposing interfacial tension force is proportional to the first power of the radius. The shape of the drop during rotation is schematically shown in Fig. 2.2.3.

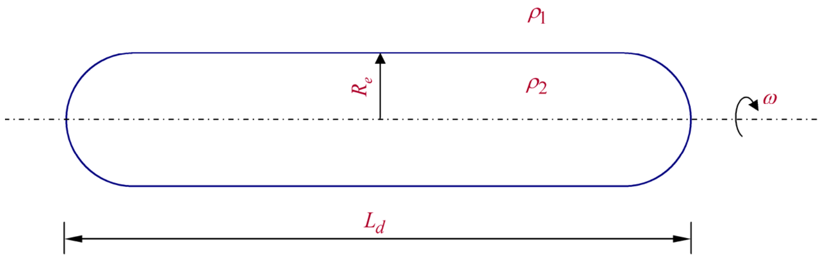


Fig. 2.2.3 Schematic diagram of drop under rotation.

- If the densities of the heavier and lighter liquids are ρ_1 and ρ_2, angular velocity is ω, and the equatorial radius of the cylindrical drop is R_e, the following formula given by Vonnegut (1942) can be used to calculate the interfacial tension.

$$\gamma = \frac{(\rho_1 - \rho_2)\omega^2 R_e^3}{4} \tag{2.2.6}$$

- If the ratio of the length (L_d) and the diameter of the drop exceeds 4, Eq. (2.2.6) can be used to determine interfacial tension with reasonable accuracy.
- The advantage of the spinning drop method is that it can be used to measure very low interfacial tensions (~10^{-6} mN/m). Such low interfacial tensions are encountered in applications such as microemulsions.
- The spinning drop method has also been used in systems such as polymer melts, bitumen, crude oil and other organic solvents.
- In modern spinning drop tensiometers, the interfacial tension range is about $10^{-6}-50$ mN/m. The angular velocity can be as high as 1500 rad/s. Typical radius of the rotating tube is 0.1–0.2 cm.

Example 2.2.2: The interfacial tension between an oil and water is 50 mN/m. The density of oil is 850 kg/m^3. If the angular velocity is 1050 rad/s, calculate the radius of the cylindrical drop.

Solution: Here, $\gamma = 0.05$ N/m, $\Delta\rho = 150$ kg/m^3, and $\omega = 1050$ rad/s. From Vonnegut equation, the equatorial radius of the drop is given by,

$$R_e = \left[\frac{4\gamma}{\Delta\rho\omega^2}\right]^{1/3} = \left[\frac{4\times 0.05}{150\times(1050)^2}\right]^{1/3} = 1.065\times 10^{-3} \text{ m} = 1.065 \text{ mm}$$

2.2.5 Contact angle and wetting

- If a small drop of liquid is placed on a uniform flat solid surface it will, in general, not spread completely over the surface. However, its edge will make an angle (θ) with the solid.
- The angle between the tangent to the liquid surface at the point of contact and the solid surface inside the liquid is called *contact angle* for that liquid–solid combination. It strongly depends upon the nature of the liquid and the solid, and can have values between zero and π rad.

- For water on a hydrophilic surface, the contact angle is less than $\pi/2$ rad. On the other hand, if the solid surface is hydrophobic, the contact angle will be greater than $\pi/2$ rad. This is illustrated in Fig. 2.2.4.

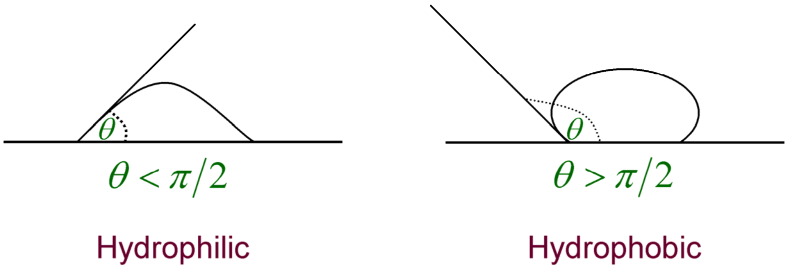


Fig. 2.2.4 Contact angle on solid surfaces.

- The contact angle for water and glass is $\sim \pi/10$ rad, and the same for mercury and glass is $\sim 7\pi/9$ rad.
- If the liquid is very strongly attracted to the solid surface (e.g., water on a strongly hydrophilic solid) the droplet will almost completely spread out on the surface. In that case, the contact angle will be close to zero.
- If the surface of the solid is less hydrophilic, the contact angle may be close to $\pi/2$ rad.
- On highly hydrophobic surfaces in contact with water, the contact angle can be as high as $5\pi/6$ rad or even higher. On these surfaces, a water droplet will simply rest without actually wetting it to any significant extent (see Fig. 2.2.5).

Fig. 2.2.5 Water drops on a hydrophobic leaf.

2.2.5.1 Young–Dupré equation

- Let us consider the thermodynamic equilibrium between three phases,viz. air, liquid and solid as depicted in Fig. 2.2.6.

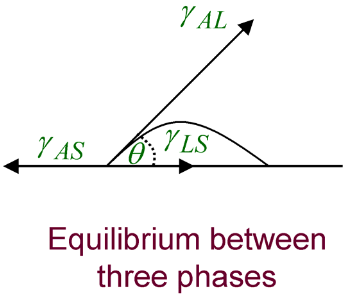


Fig. 2.2.6 Illustration of Young–Dupré equation.

- Here, γ_{AL}, γ_{AS} and γ_{LS} are the interfacial energies (or tensions) at the air–liquid, air–solid and liquid–solid interfaces, respectively. At equilibrium, we have,

$$\gamma_{AS} = \gamma_{LS} + \gamma_{AL} \cos\theta \tag{2.2.7}$$

Therefore,

$$\cos\theta = \frac{\gamma_{AS} - \gamma_{LS}}{\gamma_{AL}} \tag{2.2.8}$$

Equation (2.2.8) is known as *Young–Dupré equation*. If $\gamma_{AS} > \gamma_{LS}$, then $\theta < \pi/2$ rad (e.g., water on a hydrophilic surface). If $\gamma_{AS} < \gamma_{LS}$, then $\theta > \pi/2$ rad (e.g., water on a hydrophobic surface).

- If another liquid is present instead of air, the contact angle is defined in a similar manner.

2.2.5.2 Applications of contact angle

- Knowledge of contact angle is very important in the study of wetting and adhesion. For example, how well a polymer coating will repel solvents is important to the coating manufacturers.

- The wetting of the biological fluids of the drug powders is important in pharmaceutical industry.
- A good insecticide should wet the waxy leaves of the plants to protect them from the insects and disease.
- Wetting of textured surfaces, and porous media have very important emerging industrial applications.

2.2.5.3 Measurement of contact angle

- Two most widely used methods for measuring the contact angle are the plate method and the sessile drop method.
- The Wilhelmy plate method can be used to measure the contact angle of symmetric plate-shaped solids (i.e., plates whose both sides are identical) if it is dipped in a liquid of known surface tension. The contact angle can be determined from the following equation.

$$\theta = \cos^{-1}\left[\frac{F}{2\gamma(l+t)}\right] \tag{2.2.9}$$

  where l is the horizontal length (when it is suspended from the balance) of the plate and t is its thickness. The wetted perimeter is $2(l+t)$.
- The surface tension of the liquid is first measured using the platinum Wilhelmy plate. When the sample advances into the liquid, the contact angle measured from the force recorded by the balance is termed *advancing contact angle*. The sample is immersed to a certain depth and then the process is reversed. As the sample retreats from the liquid, the contact angle measured is termed *receding contact angle*. These are illustrated in Fig. 2.2.7.
- When the sample is above the liquid, the force is set to zero. As the sample is immersed into the liquid, the buoyant force increases causing a decrease in the force recorded by the balance. During this process, the forces are measured for the advancing angle.
- After reaching the desired depth, the sample is pulled out of the liquid. During this process, forces are measured for the receding angle.

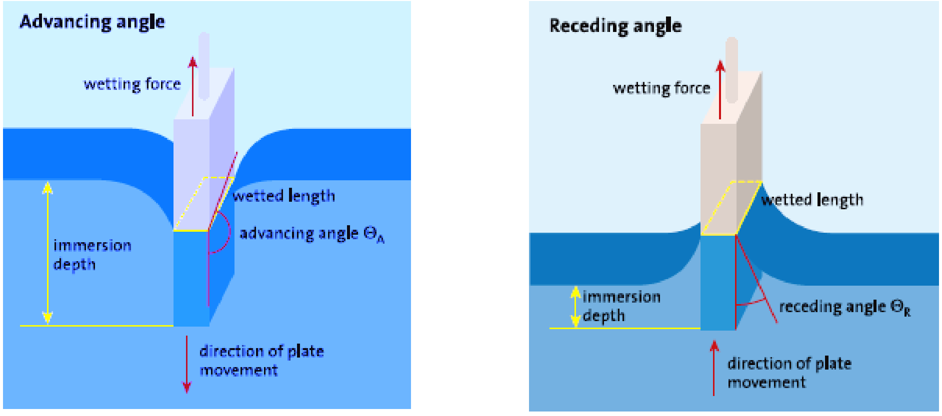


Fig. 2.2.7 Advancing and receding contact angles (courtesy: M/S DataPhysics Instruments GmbH).

- The contact angle measured in this way is termed *dynamic contact angle* (DCA). The dynamic contact angle can be measured at various velocities. Dynamic contact angle measured at low velocities is generally expected to be close to the static (equilibrium) contact angle.
- The sessile drop method is a frequently used method for measurement of contact angle. The traditional manually-operated equipment is known as *contact goniometer*. It is a simple contact angle apparatus. A protractor is used to align a tangent along the three phase (i.e., solid, liquid and vapor) contact point to determine the contact angle of liquid–solid systems. The error associated with using this instrument can be quite large by the present standards, and it varies depending on the expertise of the user.
- Modern instruments use digitization of the drop profile along with numerical integration of the Young–Laplace equation to generate the best-fit curve. It is a versatile, repeatable and accurate technique for determining the interfacial tension and contact angle. An optical contact angle measurement equipment is shown in Fig. 2.2.8.

Fig. 2.2.8 Optical contact angle measuring equipment (courtesy: M/S DataPhysics Instruments GmbH).

- To measure the advancing and receding contact angles by the sessile drop method, drops with advanced and receded edges can be generated. Drops can be made to have advanced edges by addition of liquid, and the receded edges may be produced by allowing sufficient evaporation or by withdrawing liquid from the drop. Alternatively, both advanced and receded edges can be produced when the stage on which the solid is held is tilted to the point of incipient motion. Using an instrument with high-speed image capture capabilities, the shapes of drops can be analyzed.

2.2.5.4 Advantages and disadvantages of the contact angle measurement techniques

- The biggest advantage of the goniometer method is that it can be used to measure contact angle between any liquid and any non-porous solid. If the size of the surface is large, this method is good for studying surface heterogeneity. The droplets can be placed at different locations on the surface and the contact angles can be measured at those locations. Another advantage of this method is that the contact angle can be measured quickly and easily.
- The method has a few disadvantages as well. For conventional goniometers, determination of the contact angle is dependent upon the subjective placement of two lines: one characterizing the edge of the drop and the other characterizing the plane of contact between the drop and the solid surface. The reproducibility in the measurement depends on the criteria used to place these lines. With computerized goniometers, these criteria are based on the analysis of the image of the drop.

- Factors such as illumination intensity, focus, contrast, refractive indices of the materials and the reflectance of the solid surface affect the measurement. In practice, the non-reflective surfaces pose the biggest measurement problems because the placement of the solid–liquid contact line becomes somewhat nebulous in such a situation. For small contact angles, the error can be as large as 0.1 rad.
- The Wilhelmy plate method is easy to use with the modern computer controlled tensiometers. This method is easier to apply for small contact angles since the measured parameter in this method is force, and the force decreases with increasing contact angle. The Wilhelmy method is free from the subjectivity of the goniometer method because no contact line needs to be set. In the Wilhelmy method, the measured contact angle is the average over the entire wetted length of the sample. This inherent averaging process makes the contact angle measurements more reproducible than the data obtained from goniometer.
- However, this method is not suitable for studying the heterogeneity of the surface simultaneously. The main disadvantage of the Wilhelmy plate method is the sample preparation. The sample must have well-defined cross-section and its wetted length must be known precisely to apply Eq. (2.2.9).
- Another important disadvantage is that the two surfaces of the plate must be identical. Therefore, it may be difficult to study the samples with one side coated (e.g., asymmetric membranes). If the two surfaces are different, the results will reflect some average contact angle representative of both the surfaces. Sometimes, the sample is folded or bonded back-to-back so that only one type of surface is exposed to the liquid. Such preparations of the sample may prove to be cumbersome at times.

2.2.5.5 Washburn method

- The wetting of powders and porous solids has many important industrial applications. Measurement of contact angle in these systems is complicated by the presence of the porous architecture.
- Although various methods are employed to characterize this architecture, contact angle is the primary parameters used to characterize wetting.
- The Washburn method is a well-known method for measuring contact angle in porous materials. According to this method, when a porous solid is brought into contact with a liquid, the rise of the liquid into the pores of the solid obeys the following relationship.

$$x = \left(\frac{r_e \gamma \cos\theta}{2\mu}\right)^{1/2} t^{1/2} = kt^{1/2} \qquad (2.2.10)$$

where *x* is the liquid rise (or "wicking distance"), *t* is the time after contact, μ is the viscosity of the liquid, r_e is the wicking equivalent radius (that accounts for the non-uniform irregular shape of the pores), γ is the surface tension of the liquid, and θ is the contact angle. Equation (2.2.10) is known as the *Lucas–Washburn equation*. The rise of liquid in the porous solid is schematically illustrated in Fig. 2.2.9.

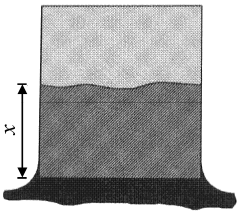


Fig. 2.2.9 Schematic representation of wicking flow in porous medium.

- There is sometimes a "non-Washburn" period right at the beginning of wicking, which is termed "wicking delay". This may be due to the time required for the meniscus to establish itself, and the flow to reach quasi-steady state. In many cases, it is negligibly small, usually 1 s, or less.

- The slope of the wicking distance versus $\sqrt{t}$ is termed the "Washburn slope", k. According to Eq. (2.2.10), it varies directly as $\sqrt{\gamma/\mu}$. Thus, the contact angle for a fibrous or a granular material may be inferred from wicking experiments by comparing the Washburn slope of the liquid of interest against the slope obtained for a liquid giving $\theta = 0$.
- For a given porous material, such as a powder packed into a tube, an experiment is performed with a liquid which is known to wet out the solid (so that for it, $\cos\theta = 1$). A good candidate is hexamethyldisiloxane. Assuming that γ and μ for this liquid is known, the measured slope will yield the wicking equivalent radius, r_e, for the material.
- The inferred contact angle is computed as,

$$\cos\theta = \frac{(\gamma/\mu)_{\text{ref}}}{(\gamma/\mu)}\left(\frac{k}{k_{\text{ref}}}\right)^2 \qquad (2.2.11)$$

  assuming that r_e of the medium to be the same for both liquids (e.g., no swelling of the particles or the fibers of the medium).
- This method has been used to obtain surface energies of carbon fibers for the manufacture of fiber-reinforced composite materials. A bundle of carbon fibers is drawn into a tube, and the liquid drawn up into the skein is recorded over time. Similar studies have been performed on various non-woven fibrous media.
- Various methods are employed to try and ensure a consistent packing, e.g., by using a reproducible force to pack a known mass of powder into a sleeve with a porous bottom (e.g., sintered glass or screen lined with filter paper), such as the sonic vibration methods.

2.2.5.6 Contact angle hysteresis

- Basing on Young–Dupré equation [Eq. (2.2.8)], one would expect that only a single value of contact angle for a particular solid–liquid–gas system should exist. However, it has been observed in practice that θ_a and θ_r can be quite different. The difference between them is called *contact angle hysteresis*.

- This phenomenon is known for about 100 years. The reasons behind hysteresis have been attributed to surface roughness, impurities on the surface, microscopic chemical heterogeneity, molecular reorientation and the penetration of the liquid molecules into the solid surface.
- However, with the development of sophisticated instruments for testing the solid surface (e.g., atomic force microscopy), it has been observed that not only the evidently-rough and heterogeneous surfaces cause hysteresis, but different advancing and receding contact angles are observed even on molecularly-smooth surfaces.
- On smooth and low-energy surfaces such as Teflon, remarkable hysteresis of water and other liquids has been observed. The advancing and receding contact angles of some liquids on the surface of fluorocarbon FC-732 (measured by the sessile drop method) are presented in Table 2.2.2.

Table 2.2.2 Advancing and receding contact angles on FC-732

Liquid	θ_a (rad)	θ_r (rad)
Hexane	0.92	0.71
Decane	1.17	1.08
Tetradecane	1.29	1.19
Ethanol	1.22	0.71
Pentanol	1.30	0.85
Octanol	1.37	0.95
Decanol	1.39	1.22

- Modern theories suggest that hysteresis of contact angle on smooth solid surfaces for liquids such as alkanes or alcohols is due to the sorption of the liquid by the solid surface. Therefore, contact angle hysteresis is likely to depend on the molecular size of the liquid, and it occurs due to the penetration of the liquid and surface swelling.

Exercise

Exercise 2.2.1: Calculate the interfacial tension between water and aniline at 300 K using Girifalco–Good correlation. Compare your result with the experimental value.

Exercise 2.2.2: The interfacial tension between water and cyclohexanol at 298 K was measured by a du Noüy ring. The measured value was 4.2 mN/m. Calculate the correction factor by Zuidema–Waters correlation, and determine the corrected interfacial tension applying this factor. The ring-parameters are: $R_r = 0.9545$ cm and $r_w = 0.0185$ cm. Collect the necessary data from a Handbook.

Exercise 2.2.3: Calculate the contact angle of a material coated with plastic with water by the Wilhelmy plate method if the measured force is 1.2 mN. The length and thickness of the plastic sample are 1 cm and 1 mm, respectively.

Exercise 2.2.4: The contact angle of water with a porous solid is to be determined. The Washburn slope obtained with hexamethyldisiloxane (viscosity = 0.65 mm^2/s and surface tension = 16 mN/m) is 1.6, and the same with water is 2.1. Calculate the contact angle.

Exercise 2.2.5: Answer the following questions clearly.

(a) Explain the Girifalco–Good correlation for estimation of interfacial tension.
(b) Explain the Fowkes correlation for the estimation of interfacial tension.
(c) Explain what you understand by contact angle.
(d) How does the contact angle reflect the surface properties of a solid material?
(e) Write the Young–Dupré equation for contact angle. Explain how interfacial tension is measured by spinning-drop method.
(f) Write the Vonnegut equation and explain its terms. What is gyrostatic equilibrium?
(g) Under what conditions will the drop fail to detach from the wall of the tube in the spinning-drop method?

(h) Explain how the contact angle of a solid substance can be measured.

(i) What are advantages and disadvantages of the Wilhelmy plate method for measuring contact angle?

(j) Explain how a goniometer is used to measure contact angle.

(k) What are the advantages and disadvantages of goniometer for measuring the contact angle?

(l) Explain advancing and receding contact angles.

(m) What is contact angle hysteresis? Explain with two examples. Why does it occur?

Suggested reading

Textbooks

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