Ministry of Higher
Education and Scientific
Research

Al-Mustaqbal University College

Chemical engineering and petroleum industries

laboratory manual
of
physical chemistry

second year

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Experiment No. 1

VISCOSITY

A liquid moving through a tube may be considered as composed of concentric layers moving with different velocities. Layers adhering to the sides of the tube are considered to be stationary, and the velocity increases as the middle of the tube is approached. Hence there will be a velocity gradient between the layers due to fric

Fig. 3. tional forces acting between the different layers of the liquid. These forces are responsible for the so called viscosity of the liquid (Fig. 3).

It has been shown experimentally that the tangential force, f, required to maintain a constant difference between the velocities of the parallel layers of liquid moving in the same direction varies directly with the difference in velocity U, and the area A, of the surface of contact of the two layers, and inversely as the distance, d, between the layers. That is,

where n is a proportionality factor known as the coefficient of viscosity. The unit of viscosity is the poise. This is defined as the force necessary to move a layer of liquid of area 1 cm.? with a velocity of 1 cm. per second, past another layer at distance of one centimeter.' The viscosity of a liquid is generally measured by observing the time required for a definite volume of liquid to flow through a standardised capillary tube under a known difference of pressure. The apparatus commonly used in the laboratory is the Ostwald's Viscometer (Fig. 4). The law governing the flow of liquids through capillary tubes was discovered by Poiseuille, and is given by the relation :

I T Pret

- 8 VI in which y denotes the volume of a liquid of viscosity flowing through a capillary tube of length 1 and radius r in time t and under the pressure P.

If the times of flow of equal volumes of two liquids through the same capillary are measured under the same experimental conditions, it follows that:

na Pit de ti

12 P2 ta d ta where n and no denote the coefficient of viscosity of the two liquids, d, and d, their densities and t, and t, their times of flow. This equation is used to calculate the so-called relative viscosity. Water is quite generally accepted as the standard of reference in determinations of relative viscosity. If the absolute viscosity of water is known, that of the liquid can be calculated.

Experiment :

Determination of the relative and absolute viscosities of a liquid.

Procedure :

(1) The viscometer is cleaned and dried in the same way as described for the pyknometer.

Fig. 4. (2) The viscometer is clamped vertically in a water thermostat. It must be dipped in the thermostat down to the mark (a).

(3) A definite volume of the liquid is introduced in the wide tube (limb C) of the viscometer and is left until it acquires the required temperature of the bath. The liquid is then forced up through the capillary tube by suction through a rubber tube attached to the end (a) of the viscometer until the liquid fills the bulb (E) and rises slightly above the mark (a). The volume of the liquid introduced must be sufficient to fill the bulb (E), the lower bend (F) and exter slightly into the wider tube (C), otherwise air bubbles will form in the capillary and affect the time of flow.

. (4) The liquid is then allowed to flow back through the capillary tube and a stop watch is started when the meniscus passes by the upper mark (a) and 'is stopped when the meniscus passes by the lower mark (b) Repeat twice.

(5) Repeat the above steps on freshly boiled then cooled distilled water, using exactly the same volume as that taken of the liquid.

6) Determine the density of the liquid by the pyknometer, as described before.

Calculations :

Let the time of flow of the liquid be tı, its density d, and its absolute viscosity na, the time of flow of water bet, , its density d, and its absolute 'viscosity 12.

Then,

11 - td The t, d

Experiment No.2

SURFACE TENSION

A liquid surface tends to contract to the minimum area as a result of the unbalanced forces of molecular attraction acting at the surface (Fig. 5). The molecules at the surface are attracted into the body of the liquid because the attraction of the underlying molecules is greater than the attraction by the vapour molecules on the other side of the surface. The surface tension of a liquid, y, is the force per centimeter on the surface which opposes the expansion of its area. Surface tension is responsible for the formation of spherical droplets of liquid, the rise of water in capillary tubes, and the movement of a liquid through a porous solid.

When a capillary tube is wetted by a liquid, the liquid rises in the tube because this change is in the direction of a decreased sur sce area. It continues to rise until the force due to surface tension tending to pull the liquid

Fig. 5. upwards is counterbalanced by the force of gravity pulling it downwards. The downward force is equal to 1 r2 hdg where g is the acceleration of gravity, d the density of the liquid and r the radius of the capillary tube. The upward force is equal to 2 st r » cos , where @ is the angle of contact. At equilibrium the upward and downward forces are equal, hence

2 - ry cos

= r ra h dg hdgr 2 cos

For many liquids including water against glass the angle of contact is very small and cos is practically equal to 1. The above relation reduces to

Y = 1 hd gr

Wlien h, r, and g are expressed in c.g.s, units, the unit of per centimeter.

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Experiment :

Determination of the surface tension of a liquid by the capillary rise method.

(a) Determination of the radius of the capillary tube Procedure :

(1) Clean the capillary tube and dry it in the usual way.

(2) Suck carefully an amount of clean dry mercury in the capillary tube.

(3) Measure the length of the mercury thread using the sliding microscope.

(4) Weigh the mercury after transferring it into a clean weighed watch glass.

Calculations :

The radius of the capillary tube is obtained using the relation :

W.

V Lda where r is radius of the tube; W is the weight of the mercury thread, L its length and d density of mercury.

(b) Determination of the rise of the liquid in the capillary tube :

Procedure :

Thermometer

Ruder Tube

(1) Set up the apparatus and place it in a thermostat until it acquires its temperature (Fig. 6).

Stari

HIcepeat

- Tube

Measurement of surface tension of a liquid by capillary rise method

Objective: To determine of the surface tension of a liquid by capillary rise method .

Theory:

Surface tension is the tendency of liquid surfaces to shrink into the minimum surface area possible. Surface tension allows insects (e.g. water striders), usually denser than water, to float and slide on a water surface.

A liquid surface tends to contract to the minimum area as a result of the unbalanced forces of molecular attraction acting at the surface. The molecules at the surface are attracted into the body of liquid because the attraction of the underlying molecules is greater than the attraction by the vapor molecules on the other side of the surface .the surface tension of a liquid (T) is the force per centimeter on the surface which opposes the expansion of its area. Surface tension is responsible for the formation of spherical droplets of liquid, the rise of water in capillary tubes, and the movement of a liquid through a porous solid.

When a capillary tube is wetted by a liquid, the liquid rises in the tube because this change in the direction of a decreased surface area. It continues to rise until the force

due to surface tension tending to pull the liquid up words is counterbalanced by the force of gravity pulling it downwards.

Surface tension has the dimension of force per unit length, or of energy per unit area. The two are equivalent, but when referring to energy per unit of area, it is common to use the term surface energy, which is a more general term in the sense that it applies also to solids.

The surface tension γ is defined as the magnitude F of the force exerted tangential to the surface of a liquid divided by the length l of the line over which the force acts in order to maintain the liquid film.

$\gamma = F/l$ (1)

In this experiment we will determine the surface tension of water by capillary rise method. Capillarity is the combined effect of cohesive and adhesive forces that cause liquids to rise in tubes of very small diameter. In case of water in a capillary tube, the adhesive force draws it up along the sides of the glass tube to form a meniscus. The cohesive force also acts at the same time to minimize the distance between the water molecules by pulling the bottom of the meniscus up against the force of gravity.

Consider the situation depicted in Fig. 1, in which the end of a capillary tube of radius, r, is immersed in a liquid of density ρ. For sufficiently small capillaries, one observes a substantial rise of liquid to height, h, in the capillary, because of the force exerted on the liquid due to surface tension. Equilibrium occurs when the force of gravity on the volume of liquid balances the force due to surface tension. The balance point can be used to measure the surface tension. Thus, at equilibrium force of gravity is given as

where is the acceleration due to gravity. Force due to surface tension (see Fig. 2) is along the perimeter of the liquid. Let θ be the angle of contact of the liquid on glass. The vertical component of the force (upward) at equilibrium is given as

Assuming θ to be very small and neglecting the curvature of liquid surface at the boundaries, one can obtain surface tension by equating Eqs. 2 and 3 as follows:

It should also be noted that surface tension of a liquid depends very markedly upon the presence of impurities in the liquid and upon temperature. The SI unit for surface tension is N/m.

Apparatus:

(i) Capillary tubes of different radii, (ii) Experimental liquid (water)(iii) Beaker, (v) Glass plate to fix the tubes, (vi) A needle, (vii) Laboratory Jack/support base to keep the beaker, (viii) Support stands and clamps.

Procedure:

1. Fix the supplied capillary tubes on the glass plate parallel to each other. The lower ends of the tubes, which are to be immersed in water, should be nearly at the same level. Fix the tubes as close as possible.

2. Clamp the glass plate to the support stand and check that the tubes remain perfectly vertical.

3. Keep the beaker filled with water on the support base. Bring the clamp stand near the beaker. Let the tubes immerse in water.

4. Determine the vernier constant of the travelling microscope to be used.

5. Focus the travelling microscope so that you can see the inverted (convex) meniscus of water. Adjust the horizontal crosswire to be tangential to the convex liquid surface. Note down the readings (say R1) on the vertical scale.

6. Turn the microscope screws in horizontal direction to view the next capillary tube and follow the above step to note the position of liquid surface.

7. After noting the positions of liquid surface for all the tubes, move the microscope further horizontally and focus to the needle. Now move the microscope vertically and let the lower tip of the needle be focused at the point of intersection of the two cross wires. Note down the readings on the vertical scale (say R).

8. Thus the height of the liquid can be calculated from the difference of the two readings noted above, e.g. (R1-R).

9. Now to find the radius of the tube, lower the height of the support base and remove the beaker. Carefully rotate the glass plate with the tubes so that the immersed lower ends face towards you. 3

10. Focus one of the tubes using travelling microscope to clearly see the inner walls of the tube.

11. Turn the microscope screws in horizontal direction to view the next capillary tubes and follow the above step to find the radius of each tube. 12. Finally calculate the surface in your experiment. Report the result at the noted room temperature.

Questions for discussion:

1. **What does surface tightening of liquids mean?**
2. **What is capillary rise method?**
3. **What is the surface tension of water?**
4. **Why do we measure surface tension?**
5. **How does surface tension change with temperature?**

NOTE:

g = 9.8m/s2

density for water(ρ)= 1g/cm3 = 1000Kg/m3

Surface tension (γ)= N/m

1Newton(N) =1 Kg*m/s2

Experiment No. 3

PHYSICAL PROPERTIES OF LIQUIS AND SOLUTIONS

DENSITY The absolute density of a liquid or solution is the mass of unit volume of the substance. The relative density at a given temperature is the density relative to that of a standard substance (water). It is easily determined by means of a vessel of accurately definite volume, called Ostwald's pyknometer (fig. 2), or by hyaluronicter.

Experiment

Determination of the relative and absoluta densities of á Hquid or sclation

Procedure:

(1) Wash the pyknometer with

chromic acid, water, and

distilled water. (2) For drying the pykno

meter wash it with alcohol, followed by ether, then pass a current of dry air through it by attaching HO one of its ends to a water 50 on suction pump. (The pyknometer must not be dried in the oven).

Fig. 2 (3) Weigh the pyknometer empty with its caps on. (4) Fill the pýknometer with freshly boiled and cooled distilled

water by sucking gently through a rubber tube fitted to the end of limb (b), while the other end of limb (a) dips in the

water.

(5) Adjust the thermostat at the required temperature to within

0.1°C. as shown by a thermometer immersed in the bath. (6) Suspend the pyknometer in the thermostat and leave it to

acquire the temperature of the bath (15-20 minutes). The amount of water in the pyknometer must be adjusted so that

it fills it from the end (a) up to the mark on the limb (b). If there is too little water it is completed by touching the end by a glass rod or a tube carrying a drop of water. If too much water, a filter paper is used to absorb the excess. These adjustments must be done while the pyknometer is in the thermostat. It must be noted that air bubbles should not be present within the pyknometer from the end (a) up to the mark on (b).

(7) Remove the pyknometer from the bath, dry it well with a

filter paper and weigh it. (8) Remove the water from the pyknometer, and rinse with the

liquid or solution, then repeat steps (4), (5), (6) and (7) using the given unknown.

Calculations :

· Let W, be the weight of liquid (or solution) that fills the pyknometer ; its density d,, and let w, and d, be the weight and density of water.

If V is the volume of the pyknometer, then

and

d, =

The ratio d/d, is known as the relative density, i.e. the density of the liquid with respect to water at the given temperature. If d, is known, the absolute density d, can be calculated

(2) Suck slowly through the rubber tube attached to the capillary tube. The liquid will rise in the capillary. When sucking is stopped, the liquid will fall to a certain height.

(3) Measure the height of the liquid in the capillary tube by means of the sliding microscope (Fig. 7).

(4) Using a pyknometer, determine the density of the liquid.

Calculations :

Calculate the surface tension from the relation :

$Y = jrdhg$

Measurement of surface tension of a liquid by capillary rise method

Objective: To determine of the surface tension of a liquid by capillary rise method

Theory: Surface tension is the tendency of liquid surfaces to shrink into the minimum surface area possible. Surface tension allows insects (e.g. water striders), usually denser than water, to float and slide on a water surface.

A liquid surface tends to contract to the minimum area as a result of the unbalanced forces of molecular attraction acting at the surface. The molecules at the surface are attracted into the body of liquid because the attraction of the underlying molecules is greater than the attraction by the vapor molecules on the other side of the surface the surface tension of a liquid (T) is the force per centimeter on the surface which opposes the expansion of its area. Surface tension is responsible for the formation of spherical droplets of liquid, the rise of water in capillary tubes, and the movement of a liquid through a porous solid. When a capillary tube is wetted by a liquid, the liquid rises in the tube because this change in the direction of a decreased surface area. It continues to rise until the force due to surface tension tending to pull the liquid up words is counterbalanced by the force of gravity pulling it downwards. Surface tension has the dimension of force per unit length, or of energy per unit area. The two are equivalent, but when referring to energy per unit of area, it is common to use the term surface energy, which is a more general term in the sense that it applies also to solids.

The surface tension y is defined as the magnitude F of the force exerted tangential to the surface of a liquid divided by

the length 1 of the line over which the force acts in order to maintain the liquid film.

$y =F/1$(١)

In this experiment we will determine the surface tension of water by capillary rise method. Capillarity is the combined effect of cohesive and adhesive forces that cause liquids to rise in tubes of very small diameter. In case of water in a capillary tube, the adhesive force draws it up along the sides of the glass tube to form a meniscus. The cohesive force also acts at the same time to minimize the distance between the water molecules by pulling the bottom of the meniscus up against the force of gravity.

Consider the situation depicted in Fig. 1, in which the end of a capillary tube of radius, r, is immersed in a liquid of density p. For sufficiently small capillaries, one observes a substantial rise of liquid to height, h, in the capillary, because of the force exerted on the liquid due to surface tension. Equilibrium occurs when the force of gravity on the volume of liquid balances the force due to surface tension. The balance point can be

used to measure the surface tension. Thus, at equilibrium force of gravity is given as

$Fg=ph(nr?)g$

(٢)

where is the acceleration due to gravity. Force due to surface tension (see Fig. 2) is along the perimeter of the liquid. Let o be the angle of contact of the liquid on glass. The vertical component of the force (upward) at equilibrium is given as

$F=y x 21 x cose$(٣)

Assuming 0 to be very small and neglecting the curvature of liquid surface at the boundaries, one can obtain surface tension by equating Eqs. 2 and 3 as follows:

v=;pgrh(٤)

It should also be noted that surface tension of a liquid depends very markedly upon the presence of impurities in the liquid and upon temperature. The SI unit for surface tension is N/m.

Apparatus:

(i) Capillary tubes of different radii, (ii) Experimental liquid (water)(iii) Beaker, (v) Glass plate to fix the tubes, (vi) A needle, (vii) Laboratory Jack/support base to keep the beaker, (viii) Support stands and clamps.

Procedure:

1 .Fix the supplied capillary tubes on the glass plate parallel to each other. The lower ends of the tubes, which are to be immersed in water, should be nearly at the same level. Fix the tubes as close as possible.

2 .Clamp the glass plate to the support stand and check that the tubes remain perfectly vertical.

3 .Keep the beaker filled with water on the support base. Bring the clamp stand near the beaker. Let the tubes immerse in water.

4 .Determine the vernier constant of the travelling microscope to be used.

5 .Focus the travelling microscope so that you can see the inverted (convex) meniscus of water. Adjust the horizontal crosswire to be tangential to the convex liquid surface. Note down the readings (say R1) on the vertical scale.

6 .Turn the microscope screws in horizontal direction to view the next capillary tube and follow the above step to note the position of liquid surface.

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lower tip of the needle be focused at the point of intersection of the two cross wires. Note down the readings on the vertical scale (say R).

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Questions for discussion:

1 .What does surface tightening of liquids mean? 2. What is capillary rise method? 3. What is the surface tension of water? 4. Why do we measure surface tension? 5. How does surface tension change with temperature?

NOTE:

g=9.8m/s

density for water(p)= 1g/cm3 = 1000Kg/m3

Surface tension (Y)=N/m

1 Newton(N) =1 Kg*m/s2

Experiment No. 4

REFRACTIVE IDEX BY THE ABBÉ BUFRACTOMETER

It is well known that when light passes from one medium into another, as from air to water, it suffers refraction, i.e., a change of direction. The refractive indes which is a measure of the refraction of light is used to determine the concentration of solutions, to identify chemical compounds, and to determine the molecular structure. The refractive index depends on the temperature, and on the wave length of light. It is given by the relation

sin i N

LAE Denise(١)

Media sin rn where i is the angle of incidence, r the angle of refraction, and n and N are the indices of refraction of the less dense and more dense media respectively. When i increases, r also in

Fig. 10. creases and reaches its maximum when i is equal to 90°, i.e. when the ray just grazes the surface between the media. In this case the refractive index of the less dense medium is given by n= N sin r.

It was established theoretically by Lorentz and Lorentz that the specific refraction, R, given by the relation

n'-1 1 R, -(-) =

n+2'd' where n is the refractive index and d the density, is a characteristic property of a substance independent of its physical state and of the temperature. The inolar refraction is equal to the specific refraction multiplied by the molecular weight of the substance;

n

١-

M

MR = 172+2'd

M

The presence of the factor - shows that the molar refraction is a

type of molar volume which, like other forms of molar volumes (e.g., the parachor), is an additive property and also partly a constitutive property. From a study of the molar refractions of a large number

of compounds it was possible to obtain the atomic refractions of the different elements, and the different bond contributions. Molar refractions have been used to decide between alternative structures of some organic compounds; the correct formula being that which gives a calculated molar refraction in best agreement with the experimental value.

In the Abbé refractometer the more dense medium consists of a right-angled prism ABC, and the less dense medium consists of a layer of the liquid under investigation placed in contact with the hypotenuse face AB. A ray of monochromatic light passing through the liquid and entering the prism, will emerge from the face AC perpendicular to the surface if $n = N \sin A$ (i.e., when $r = A$). For any other value of n, however, the ray of light will emerge at an angle, a, less than the right angle (Fig. 11). In order that the ray may be brought parallel to the axis of the fixed telescope (T), the prism must be rotated through an angle a. The refractive index of the liquid n can be calculated from the relation

$n = \sin A$

$N2 – \sin a - \cos A \sin a$(٢)

The scale in Abbé refractometer is not graduated in values of a; but directly in re

Fig. 11. fractive indices, calculated as shown in equation (2).

For the determination of the refractive index of a liquid or solution by the Abbé refractometer the following steps are followed.

(1)Open the prism box and wipe the prisms with a soft cloth, care being taken not to scratch them.

(2)Place one drop or two of the liquid under investigation on the surface of the lower prism. Close and fasten the prism box again. A film of liquid will thus be enclosed between the two prisms.

(3)Adjust the mirror so that diffused light from a window in front of the instrument is sent into the telescope. If natural light is not available, place 'a thin sheet of paper before the instrument, and illuminate the sheet from behind by a powerful electric lamp.

(4)Focus the eyepiece of the telescope on the cross wires, and the reading lens on the scale.

(5)By means of the arm at the side of the apparatus, turn the prism box slowly backwards or forwards until the field of view becomes partly light and partly dark. If the border line is coloured, the compensator is rotated until the colour disappears and the bright hand shows a sharp edge. (The compensator consists of two prisms which rotate in opposite directions, so as to form a system of variable dispersion).

(6) Rotate the prism box until the sharp border line is in coincidence with the intersection of the crosswires in the telescope, and read the index of refraction on the circular scale.

(7)The temperature of the prisms should be constant by circulating thermostated water through the metal jackets surrounding the prisms.

(8)The accuracy of the scale readings may be checked by means of a small test piece of glass supplied with the instrument, whose index of refraction is engraved on it. The lower prism is slipped off its hinge, and the test piece is fixed on the surface of the upper prism by means of a drop of a substance of high refractive index, such as monobromonaphthalene. The index of refraction is then

determined as described before. The difference between the scale reading and the true refractive index of the test piece is the correction which must be added to the measured refractive indices.

Experiment:

Refractometric determination of the composition of solutions,

Procedure:

sm

en

(1) Prepare a series of solutions of known composition (binary miscible liquids, or salt solutions.(

(2) Determine the refractive indices of the solutions by the Abbt. refractometer following the above mentioned steps.

(3) Determine also the refractive index of the pure liquids in question.

Plot the refractive indices against percentage composition by weight, and join the points by a smooth curve.

(٥)Determine the refractive index of the unknown solution provided, and from the graph find out its composition.

WYA-2S ABBE Digital Refractometer

Model : WYA-2S

Characteristics

Characteristics

Measurement of refractive index np of transparent or translucent liquid and solid substances. Measurement of the Brix of sugar solution. Visual aim and LCD display Automatic correction of the effect of temperature on the Brix. • Prism made of hard glass, uneasy to be worn. • Printer interface for direct data print-out. Specifications • Measurement range: Refractive index np: 1.3000-1.7000

Dissolved Solids Brix: 0-95% • Accuracy: Refractive index np: <+0.0002 Dissolved Solids Brix:: <+0.1%
Temperature Range (Min. 0.1°C): 0-50°C Weight (net): 10kg Overall dimensions: 330mm x 180mm x 380mm

AIM: To find refractive index of the given liquid samples

APPARATUS: Abbe’s refractometer, temperature controller, light source and samples.

PROCEDURE :

1-Clean the surface of prism first with alcohol and then with avetone using cotton and allow it to dry.

2-Using a dropper put 2-3 drops of given liquid b/w prisms and press them together

3-Allow the light to fall on mirror .

4- Adjust the mirror to reflect maximum light into the prism box

5 .Rotate the prism box by moving lever until the boundary b/w shaded and bright parts appear in the field of view .

6 .If a band of colors appear in the light shade boundary make it sharp by rotating the compensator .

7 .Adjust the lever so that light shade boundary passes exactly through the centre of cross wire

.٨ Read the refractive index directly on the scale

9 .Take 3 set of readings and find the average of a

Questions for discussion:

- What is refractive index liquid?
- What is the principle of refractive index?
- What are the applications of refractive index?

- **How do you measure refractive index?**
- **Why is refractive index of oil important?**
- **What are the two types of refractive index?**
- **What is unit of refractive index?**