

# Experiment 9

## Adsorption from Solution

### **Purpose :**

To investigate the adsorption isotherms from aqueous solutions of acetic acid on activated charcoal.

### **Principle :**

The type of interaction between the adsorbed molecule and the solid surface varies over a wide range from weak non-polar **van der Waals'** forces to strong chemical bonding. Examples of adsorption where ionic or covalent bonding occurs are the adsorption of chloride ions on silver chloride (ionic) or of oxygen gas on metals where oxygen-metal bonds are formed (covalent). In these cases the process is called chemisorption, and it is generally characterized by high heats of adsorption (from 10 to 100 Kcal per mole of gas adsorbed). Chemisorption is highly specific in nature and depends on the chemical properties of both the surface molecules and the adsorbed molecules. Adsorption arising from the weaker **van der Waals'** and dipole forces is not so specific in character and can take place in any system at low or moderate temperatures. This type of adsorption is called physical adsorption and is usually associated with low heats of adsorption (less than about 10 Kcal/mole).

The amount of solute adsorbed by a given quantity of adsorbent increases with the concentration of the solution. In some cases the layer of adsorbed molecules is only one molecule deep, and further adsorption stops when the surface of the crystal lattice is covered. The equilibrium between the dissolved solute and the material adsorbed also depends upon the nature of the solvent and the temperature, the amount adsorbed increasing at lower temperatures.

From measurements at constant temperature, one can obtain a plot of  $x/m$ , the number of grams adsorbed per gram of adsorbent, vs.  $c$ , the equilibrium solute concentration. This is called an adsorption isotherm.

### **(1) Freundlich isotherm :**

$$\frac{x}{m} = kc^n \quad (1)$$

$x$  = weight of material, in grams, adsorbed by  $m$  grams of adsorbing material

$c$  = concentration in solution, mole/liter

$n$  = constant ranging from 0.1 to 0.5

$k$  = another constant

Although  $k$  varies considerably with the temperature and nature of the adsorbent, the ratio of  $k$  values for two different adsorbents is constant for different solutions.

By taking the logarithm of Eq. (1) we obtain

$$\log \frac{x}{m} = n \log c + \log k \quad (2)$$

According to this equation a plot of  $\log(x/m)$  versus  $\log c$  is a straight line, and the constants may be evaluated from the slope  $n$  and the intercept  $\log k$

**(2) Langmuir isotherm :**

$$\frac{x}{m} = \frac{\alpha c}{1 + \beta c} \quad \text{or} \quad \frac{c}{x/m} = \frac{1}{\alpha} + \frac{\beta}{\alpha} c \quad (3)$$

$\alpha$  and  $\beta$  are constants. For cases in which this equation represents the data,  $c/(x/m)$  may be plotted as a linear function of  $c$ , and the constants evaluated from the slope  $\beta/\alpha$  and intercept  $1/\alpha$ .

However, Eq. (1) fails to predict the behavior usually observed at low and high concentrations. At low concentration,  $x$  is often directly proportional to  $c$ ; at high concentration,  $x$  usually approaches a constant limiting value which is independent of  $c$ . The **Langmuir** equation differs from the **Freundlich** equation in that the adsorption approaches a finite limit as the concentration is increased.

### ***Apparatus and Chemicals :***

Three *100-ml* volumetric flasks; fourteen *250-ml* Erlenmeyer flasks; *50-ml* pipette; *25-ml* pipette; *10-ml* pipette; *5-ml* pipette; *2-ml* pipette; safety bulb; three *250-ml* beakers; two funnels; Büchner funnel; filtering flask; filter paper; two *50-ml* burettes.

*0.5N* HOAc; activated charcoal; *0.25N* NaOH; KHP; phenolphthalein.

### ***Procedures :***

- (1) Prepare six different concentrations of acetic acid solutions by pipetting separately *2, 5, 10, 20, 50,* and *100 ml* of *0.5N* acetic acid into several volumetric flasks and diluting to *100 ml* with distilled water.
- (2) Each solution is transferred to a *250-ml* Erlenmeyer flask, and *2 g* of activated charcoal (weighed accurately to the nearest *milligram*) is then added to each flask.
- (3) Blank determination (*100 ml* distilled water + *2 g* of activated charcoal) should be made.
- (4) Shake the Erlenmeyer flasks periodically for a period of *30 min* and then allow it to stand at least *30 min* for equilibrium.
- (5) After equilibrium has been reached, each solution is filtered using suction filter. Pipette *25 ml* solution from the clear supernatant solution and titrate with *0.25N* NaOH using phenolphthalein as an indicator.
- (6) Standardize *0.25N* NaOH solution: Weigh accurately *0.5 g* KHP into distilled water and titrate with NaOH, using phenolphthalein as an indicator.

- (7) Standardize 0.5N acetic acid: Pipette 20 ml 0.5N acetic acid into an Erlenmeyer flask and titrate with 0.25N NaOH that has been standardized

**Calculations :**

- (1) For each flask, calculate
- (a)  $c$ , the final equilibrium concentration of the absorbate (acetic acid);
  - (b)  $x$ , the weight of acetic acid removed by absorbent;
  - (c)  $x/m$ , the amount of adsorption per  $m$  grams of activated charcoal.
- (2) Plot  $x/m$  against  $c$ .
- (3) Test the applicability of the **Freundlich** isotherm by plotting  $\log(x/m)$  against  $\log c$ . Calculate constants  $k$  and  $n$  in Eq. (2).
- (4) Test the applicability of the **Langmuir** isotherm by plotting  $c/(x/m)$  against  $c$ . Calculate constants  $\alpha$  and  $\beta$  in Eq. (3).

**References :**

- (1) **P. W. Atkins and J. de Paula**, "Physical Chemistry," 9th ed., pp. 889-894, Oxford University Press, U.S.A (2010).
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- (3) **D. P. Shoemaker and C. W. Garland**, "Experiments in Physical Chemistry," 2nd ed., pp. 258-262, McGraw-Hill, U.S.A. (1967).
- (4) **F. Daniels** and others, "Experimental Physical Chemistry," 6th ed., pp. 328-333, Europe-Asia book company, Taiwan (1956).