EXPERIMENT 4: ADSORPTION

Before the experiment: *Read the booklet carefully. Be aware of the safety issues.*

Object

To study the adsorption of acetic acid by charcoal and to determine the constants of the Freundlich adsorption isotherm.

Theory

Solids have the property of holding molecules at their surfaces, and this property is quite marked in the case of porous and finely divided material. Adsorption is the attachment of molecules to the surface of a solid, and it is frequently quite specific, so that one solute may be adsorbed selectively from a mixture.

The adsorbate refers to the substance that is adsorbed whereas the underlying material or substrate refers to the adsorbent. The amount of adsorbate for 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 ceases when the surface of the crystal lattice is covered. The adsorbed and free reactant are in dynamic equilibrium during the adsorption process, and the surface coverage depends upon surface composition and structure of the adsorbent, the nature and concentration of the solvent as well as temperature and pressure.

The amount or pressure of reactant adsorbed for a given temperature is described by adsorption isotherms. The adsorption of dissolved substances in liquid solutions by solids may be formulated (within certain concentration limits) by the equation for the adsorption isotherm proposed by Freundlich [1]:

$$x/m = ky^n \tag{1}$$

where, x/m represents the mass of material adsorbed per unit mass of adsorbent,

      y is the concentration in the solution, at equilibrium,

      k and n are the constants under the given conditions.

Definite masses of the adsorbent is brought into contact with a series of solutions of varying initial concentrations of the reagent to be adsorbed, after equilibrium has been established, liquids are filtered and analyzed to determine the degree of adsorption in each case. The amount adsorbed in each case is equal to the difference of the initial and final concentration. The equation for the adsorption isotherm may be written in the logarithmic form as follows [1]:

$$\log x/m = \log k + n \log y \qquad (2)$$

According to this equation, a plot of log x/m against log y yields a straight line. The constants k and n are evaluated from the intercept and the slope of the line.

Experimental Work

Apparatus and Chemicals: Four stoppered 250-ml. flasks, four 125-ml. Erlenmeyer flasks, four 250-ml. Büchner flasks, two Büchner funnels with rubber disks, two rubber tubes, two vacuum pumps (aspirator type), filter paper, one 500-ml. beaker, burette and holder, and two 10-ml. pipettes, charcoal, 0.4 N acetic acid, 0.1 N NaOH, phenolphthalein indicator solution.

Procedure

1. Exactly 5 g. of charcoal is placed into each of the stoppered flasks, labeled 1 to 4.

2. 50 ml. of acetic acid solution made up in the volumetric flask is placed as follows:

No.1 50 ml stock solution of acetic acid (0.4 N)

No.2 25 ml. acetic acid + 25 ml. water

No.3 15 ml. acetic acid + 35 ml. water

No.4 7.5 ml. acetic acid + 42.5 ml. water

The volumetric flask is rinsed with water each time.

3. Each sample is shaken for 5 minutes and kept at the same room temperature. Then the samples are allowed to settle and reach equilibrium.

4. The solutions are filtered and the filtrates are collected in properly labeled flasks.

5. The solutions are analyzed for acetic acid, by titration with NaOH. The given volume is pipetted from the solutions:

No.1 10 ml. sample	No.2 10 ml. sample
No.3 20 ml. sample	No.4 20 ml. Sample

Safety Issues:

Acetic acid (CH3COOH) [2]: Flammable. Colorless. Easily soluble in cold water, hot water.

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.

Very hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation. Hazardous in case of skin contact (corrosive, permeator), of eye contact (corrosive).

First Aid Measures:

Eye Contact: Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

Skin Contact: In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient. Cold water may be used. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

Inhalation: If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Sodium Hydroxide (NaOH) [3]: Non-flammable. Odorless. Solid. White. Easily soluble in cold water.

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.

Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion, of inhalation.

First Aid Measures:

Eye Contact: Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

Skin Contact: In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient. Cold water may be used. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

Inhalation: If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Charcoal (C) [4]: Flammable. Odorless. Solid. Black. Insoluble in cold water, hot water.

Routes of Entry: Inhalation. Ingestion. Slightly hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation.

First Aid Measures:

Eye Contact: Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention if irritation occurs.

Skin Contact: Wash with soap and water. Cover the irritated skin with an emollient. Get medical attention if irritation develops.

Inhalation: If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.

Calculations

1. Calculate the concentration of each solution (mol/L), which is the initial concentration.

2. Calculate the concentration of each filtrate using titration data (mol/L). This is the final concentration.

3. The difference between initial and final concentration is used for the calculation of the amount adsorbed. Convert this molar quantity to x/m by multiplying it with the volume of initial solution and the molecular weight of the adsorbed material as well as dividing by the mass of adsorbent.

4. Plot y against x/m and draw the adsorption isotherm.

5. Plot log y against log x/m and ascertain the values of k and n from the curve obtained.

Note: Explain the difference between Freundlich and Langmuir isotherms in your report. Discuss your results considering the nature of Freundlich isotherm.

References

[1] Atkins, P. and J. De Paula, J., *Physical Chemistry*, 9th Edition, W. H. Freeman, New York, 2009.

[2] Acetic Acid MSDS, http://www.sciencelab.com/msds.php?msdsId=9922769 (Retrieved February, 2014).

[3] Sodium Hydroxide MSDS, http://www.sciencelab.com/msds.php?msdsId=9924998 (Retrieved February, 2014).

[4] Charcoal MSDS, http://www.sciencelab.com/msds.php?msdsId=9923389 (Retrieved February, 2014).