Practical Work N°4 :

ADSORPTION OF AN ORGANIC POLLUTANT ON MICROPOROUS ACTIVATED CARBON

I-1 AIM OF THE EXPERIMENT:

To establish the adsorption isotherm for acetic acid on activated carbon at room temperature.

I-2 THEORETICAL BACKGROUND

I- 2.1 Definition

Adsorption is a treatment process which is well suited to eliminating a wide variety of toxic compounds from our environment. It is mainly used for water and air treatment. During this process, the molecules of a fluid (gas or liquid), called an adsorbate, attach themselves to the surface of a solid, called an adsorbent (Figure 1). This process defines the property of certain materials to bind molecules (gases, metal ions, organic molecules, etc.) to their surface.

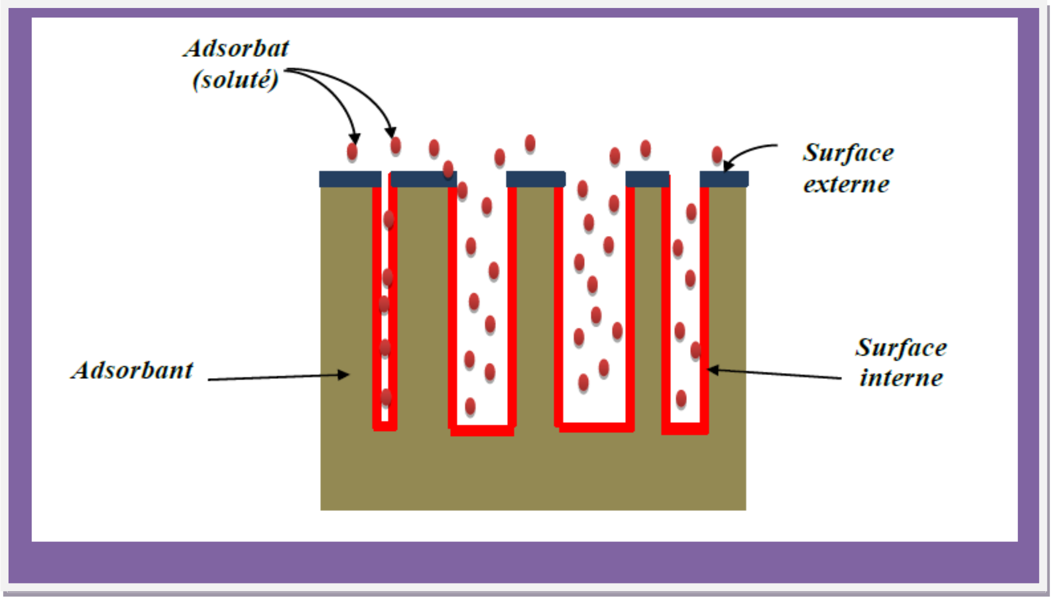

Figure .1: Adsorption process

Depending on the nature of the bonds between the substrate and the adsorbed particles, the forces responsible for the adsorption phenomenon may be physical or chemical, leading to two types of adsorption: physical adsorption (physisorption) and chemical adsorption (chemisorption). The adsorbents most commonly used in industry are:

zeolites, activated carbons, silica gels and clays.
Others include resins, polymers, etc.

Activated carbons are products obtained by high-temperature heating of carbon-rich natural raw materials, a process called carbonization followed by a second process called activation.An essential characteristic of activated carbons is their highly-developed network of micropores. which are responsible for their high adsorption capacity.

Activated carbons are highly interesting substances; they are used to separate mineral and organic compounds, or to purify liquid solutions by extracting pollutants. Industrial applications are numerous: water treatment, sugar refining, precious metals recovery, etc.

Figure 2: Granulated and powdered activated carbon

II- MATHEMATICAL MODELS APPLIED TO ADSORPTION

Adsorption isotherms enable the study of an equilibrium between two phases (adsorbate and adsorbent), by plotting the quantity of solute adsorbed as a function of the equilibrium concentration at constant temperature. They can be simulated by a number of mathematical models available in the literature, with two in particular mentioned in this PW:

II.1-Freundlich model

This is an empirical model (Freundlich, 1906), which assumes that the heat of adsorption decreases exponentially with the rate of saturation of the solid surface. In the case of solute adsorption, the equation is given by :

$$\boxed{q_e = K_F\ C_e^{1/n}}$$

Where ;

- q_e(mol/g): quantity adsorbed at equilibrium.
- C_e(mol/l): equilibrium concentration.
- K_F(l/g): Freundlich constant, dependent on the total number of adsorption sites,
- **n** : is a function of the adsorption energy distribution.

II.2 Langmuir model

This is a theoretical model (Langmuir, 1918). It assumes that all sites are equivalent and that there are no lateral interactions between adsorbed molecules, i.e. adsorption must be monolayer with one molecule per site at equilibrium. Langmuir's model translates into the following equation:

$$\boxed{q_e/q_m = K_L C_e / (1+ K_L C_e)}$$

- **q_e**(mg/g) : equilibrium adsorption capacity.
- **q_m**(mg/g): maximum adsorption capacity.
- **C_e**(mg/l) : equilibrium solute concentration.
- $\boldsymbol{K_L}$(l/g): is Langmuir's equilibrium constant.

III- EQUIPMENT :

- Erlenmeyer flasks - Funnels + filter paper
- Balance + stirrers + magnetic stirrers
- 50 mL flasks
- Graduated test tubes + graduated propettes
- Graduated burettes - Pissettes of distilled water.

IV- SOLUTIONS USED :

- NaOH (0.1M), Acetic acid (0.5l).
- Phenolphthalein , 30g activated carbon

Data : M(H) = 1 g/mol ; M(O) = 16 g/mol ; M(Na) = 23 g/mol , M(C) = 12 g/mol

V-OPERATING PROCEDURE

Part I:

The initial concentration of acetic acid in the starting solution (stock solution) is to be titrated with NaOH solution (0.1 mol/l), taking 20 ml test samples of the acid to determine its exact concentration.

Part 2:

a-Using two burettes (one filled with distilled water and the other with acetic acid), prepare six 25ml solutions (distilled water + acetic acid), respecting the volumes of each solution (**see Table 1**).

b-In six numbered Erlenmeyer flasks, weigh out a mass **m = 2.5 g** of activated carbon, then add **25 ml** of prepared acetic acid solution of varying concentration (respecting the erlen/solution numbering).

Table1 :

Erlenmeyers	1	2	3	4	5	6
Water (ml)	0	10	15	17,5	20,5	22,5
CH_3COOH (ml)	25	15	10	7,5	4,5	2,5
Carbon(g)	2,5	2,5	2,5	2,5	2,5	2,5
molair Concentration (Ci)						

c-These Erlenmeyer flasks are then closed, and the mixtures are regularly stirred for thirty minutes (30min) to reach adsorption equilibrium.

d- After this time, the solid is separated from the liquid by filtration.

e- To re-determine the residual acetic acid concentration, take **10 ml** of each filtrate and titrate with **0.1 mol/l** sodium hydroxide solution (**NaOH**). Add a few drops of phenolphthalein to observe the color change at equilibrium. (Note the volumes obtained - table2-).

Table 2:

V_{NaOH}						
C_e(acid)						

At the end of this step, the quantity of solute adsorbed per gram of adsorbent is calculated using the following expression:

$$\boxed{q = (C_i - C_e)\,(V/m)}$$

Where:

-q (mol/g): quantity adsorbed at equilibrium, i.e. the number of moles of acetic acid required to saturate one gram of activated carbon according to Langmuir's theory.

Langmuir theory

-Ci (mol/l): initial concentration of prepared acetic acid.

-Ce (mol/l): residual equilibrium concentration of acetic acid.

-m(g): mass of adsorbent.

-V (l): volume of solution.

6-QUESTIONS :

1-What is the fundamental characteristic that makes activated carbon?

2-What is the molar concentration of acetic acid (stock solution)?

3- Fill in table 1?

4- Give the theoretical expression for measuring Ce?

5- Calculate Ce for each solution from 1 to 6?

6- Why is stirring used?

7- Fill in table 2?

8- Measure the quantity q of acetic acid adsorbed for the six concentrations used?

9- Draw the curve q = f(Ci)? Comment?

10 -Conclusion