**Batch Adsorption**

**Theory**

Adsorption is the [adhesion](https://en.wikipedia.org/wiki/Adhesion) of [atoms](https://en.wikipedia.org/wiki/Atom), [ions](https://en.wikipedia.org/wiki/Ion), or [molecules](https://en.wikipedia.org/wiki/Molecule) from a gas, liquid, or dissolved solid to a [surface](https://en.wikipedia.org/wiki/Surface_science). This process creates a film of the adsorbate on the surface of the adsorbent. This process differs from [absorption](https://en.wikipedia.org/wiki/Absorption_(chemistry)), in which a [fluid](https://en.wikipedia.org/wiki/Fluid) (the absorbate) is [dissolved](https://en.wikipedia.org/wiki/Dissolution_(chemistry)) by or [permeates](https://en.wikipedia.org/wiki/Permeation) a liquid or solid (the absorbent), respectively. Adsorption is a surface-based process while absorption involves the whole volume of the material. Adsorption is a [surface phenomenon](https://en.wikipedia.org/wiki/Surface_phenomenon). The term [sorption](https://en.wikipedia.org/wiki/Sorption) encompasses both processes, while [desorption](https://en.wikipedia.org/wiki/Desorption) is the reverse of it.

A relation between the amount of adsorbate adsorbed on a given surface at constant temperature and the equilibrium concentration of the substrate in contact with the adsorbent is known as Adsorption Isotherm.

Some of the standard applications of adsorption are:

1. Heterogeneous Catalysis- This is probably the most important application relevant to chemical engineering. The reaction mechanism of how a reactant reacts on a catalyst surface revolves around adsorption. Therefore designing catalyst, reactors and studying them requires knowledge of adsorption
2. Separation- Adsorption is used as a separation process in many chemical as well as bio chemical industries to separate gaseous or liquid mixtures. Designing adsorption equipment like fixed bed adsorbers, gas drying, pressure swing adsorption etc. , chromatography requires knowledge of adsorption. Many experiments in the lab use adsorption as a process to calculate various parameters like surface concentration, porosity, change in surface energies, pore surface area etc.

Adsorption occurs due to the imbalance of forces at the surface of a material. This lead to formation of bonds (Covalent, ionic, Van der Waals, Hydrogen bonds etc.) between the surface molecules (adsorbents) and the molecules in the fluid phase (adsorbate).

Adsorption is usually described through adsorption isotherms that is the amount of adsorbate on the adsorbent as a function of its pressure (if gas) or concentration (if liquid) at constant temperature. The adsorption isotherm is the equilibrium relationship between the concentration in the fluid phase and the concentration in the adsorbent particles at a given temperature. The quantity adsorbed is nearly always normalized by the mass of the adsorbent to allow comparison of different materials. Some typical adsorptions are shown in the figure below



Figure 1:

**Freundlich Adsorption Isotherm**

It is an empirical relation between the amount of an adsorbate adsorbed per unit weight (x/m, mg/g) of adsorbent and the adsorbate equilibrium concentration (Ce, moles/L) in the fluid. The relation is given below



where, K and n are Freundlich coefficients. Here,

x = weight of adsorbate adsorbed on m unit weight of adsorbent and

Ce= equilibrium concentration of adsorbate.

Taking logarithm on both sides of equation 1 gives,

Hence a graph of vs gives a straight line with K as y-intercept and n as slope.

**Reference**

1. Mass Transfer Operations by Robert Treybal, McGraw Hill Publications, Ed. 3, 2012
2. NPTEL Adsorption Lectures.

**Batch Adsorption**

**Aim:** To verify Freundlich’s Isotherm for the Batch Adsorption of oxalic acid on activated charcoal.

**Apparatus required:** 5 stoppered bottles

Pipettes- 25ml, 10 ml

Conical flask- 250 ml

Burette- 50 ml

Measuring cylinder- 500 ml

Beaker- 250 ml

Standard flask- 100 ml, 250 ml, 1000 ml.

**Procedure**:

1. 6.3 g of oxalic acid crystal are weighed and transferred to 1000 ml standard flask, diluted up to mark with distilled water and shaken well.
2. The above solution in the standard flask has the strength of 0.1N
3. Weigh 0.5, 1, 2, 3, & 4 gm of activated carbon in each of the conical flasks marked 1 to 5.
4. Transfer 100 ml oxalic acid of 0.1N to the above flasks marked 1 to 5 and close the flask opening by any closure.
5. Keep the flasks for agitation in an orbital shaker for 45 mins to 1 hour.
6. Stop agitation. Collece the flasks and filter the contents of each conical flask.
7. Pipette out 10 ml of the solution from the 1st flask in a clean conical flask.
8. Titrate against standard NaOH in a burette using phenolphthalein as indicator.
9. Colour changes from colourless to pale pink.
10. Repeat the trial to get concordant values and repeat the procedure for flask 2, 3,4, 5
11. Tabulate the results and plot the graph.

**Observation and Calculations:**

# Formula to be Used :-

Where K and n are constants.

C\* = Equilibrium concentration of adsorbate.

C0 = Initial concentration of the solute in the liquid.

V = known volume of solution with a known weight of adsorbent (volume solution/mass adsorbent).

# Titration Results:-

Standardization of NaOH:

Normality of oxalic acid (N1) =0.1N (prepare from the oxalic acid powder)

Volume of oxalic acid (V1) = 10 ml

|  |  |  |  |
| --- | --- | --- | --- |
| Titration Trial | 1 | 2 | 3 |
| Initial reading of burette (ml)  Final reading of burette(ml)  Volume of NaOH run down (V2 ml) | 0  26  26 | 0  26.2  26.2 | 0  26  26 |

Average Titer value (V2) = 26ml

Normality of NaOH = = = 0.0961N

# 

# Titration Results:- Volume of supernatant liquid taken in all the 4 cases

|  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Trials: | For 1g  Charcoal | | | For 2g  Charcoal | | | For 3g  Charcoal | | | For 4g  Charcoal | |
|  | I | II | III | I | II | III | I | II | III | I | II |
| Initial reading (ml)  Final reading  (ml)  Volume of solution run down(ml) | 11.2  21  9.8 | 21  30.9  9.9 | 31  40.8  9.8 | 0  8.8  8.8 | 8.8  17.6  8.8 |  | 21.2  29.2  8 | 30  38  8 |  | 0  7.4  7.4 | 7.4  14.8  7.4 |
| Agreeing value(ml) | V1=9.8 | | | V2= 8.8 | | | V3= 8 | | | V4= 7.4 | |

# Data Tabulation:-

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Sl. No | Amount of activated carbon (g) | V in L/g | Titre Value  ml | C\* | V\*(C0-C\*) |
| 1  2  3  4 | 1  2  3  4 | 0.5  0.1  0.067  0.0.5 | 9.8  88  8  7.4 | 5.6  5.034  4.57  4.23 | 0.14  0.126  0115  0.1063 |

**Sample Calculation**

**V=**  = 0.2L/g

**C\*= = 8.6g/L**

**Co=**  = 6.3g/L

V\*·(C0-C\*) = 0.14

|  |  |
| --- | --- |
| log **C\*** | log(V\*(C0-C\*)) |
| 0.7481  0.7091  0.659  0.626 | -0.8538  0.8996  0.9384  0.9850 |

**From graph, y intercept = K = exp(-1.625) = 0.1962**

**slope = n = 1.030**

**Results:**

The Freundlich’s isotherm constants obtained are

n= 1.030

K= 0.1962

Questions on the understanding of adsorption