Experiment 1. Adsorption Isotherm

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Abstract. Adsorption results from the attractive interactions between the surface of a solid and a fluid. This interactions may occur due to van der Waals forces (physisorption) or formation of bonds such as covalent (chemisorption). The objective of this experiment was to determine the specific area of activated charcoal using the Langmuir adsorption isotherm model. We obtained the amount of acetic acid absorbed per gram of activated carbon for different concentrations and this was used to generate graphs which showed a clear asymptotic behavior. Results were found to be in agreement with those in literature.

I. INTRODUCTION

Adsorption is the adhesion of a substance to a surface. This phenomena is usually described through isotherms, which describes the amount of adsorbate on the adsorbent as a function of its pressure (if gas) or concentration (if liquid) at constant temperature. [1]

Irving Langmuir was the first to derive a scientifically based adsorption isotherm in 1918. This model (which bears his name) is the most common isotherm equation to use due to its simplicity and its ability to fit a variety of adsorption data. [2] It is based on four assumptions:

1. All of the adsorption sites are equivalent and each site can only accommodate one molecule.

2. The surface is energetically homogeneous and adsorbed molecules do not interact.

3. There are no phase transitions.

4. At the maximum adsorption, only a monolayer is formed. Adsorption only occurs on localized sites on the surface, not with other adsorbates.

Which are all idealizations [4].

In this model, the adsorption takes place through a simple mechanism of

$$A + S \rightleftharpoons AS \tag{1}$$

where **A** is a gas or a liquid molecule, **S** is an adsorption site and **AS** represents the molecule bound to the site.

Considering that the adsorbent has a limited amount of sites to cover, the fraction of those already covered (θ) is given by

$$\theta = \frac{[AS]}{[S_T]} = \frac{[AS]}{[S] + [AS]} \tag{2}$$

where $[A]$, $[S]$, $[AS]$ and $[S_T]$ represent concentrations. Plus S_T is the total of surface sites

$$[S_T] = [S] + [AS] \tag{3}$$

Now, the reaction equilibrium is represented by

$$K_{ads} = \frac{[AS]}{[A][S]} \tag{4}$$

This allows θ to be rewritten as

$$\theta = \frac{K_{ads} \cdot [A]}{1 + K_{ads}} \tag{5}$$

which yields the Langmuir adsorption isotherm.

Rearranging the last equation we get

$$\frac{[A]}{W_A} = \frac{1}{K_{ads}W_{A-max}} + \frac{[A]}{W_{A-max}} \tag{6}$$

where $W_A \equiv [AS]$ and $W_{A-max} \equiv [A]$. W_A is interpreted as the moles of A adsorbed per gram of adsorbed. W_{A-max} represents the maximum capacity of A that one gram of adsorbent can hold.

Finally, the specific area of the adsorbent (σ_S) is determined by

$$\sigma_S = W_{A-max} \cdot N \cdot \omega_A \tag{7}$$

where ω_A is the cross section of the adsorbate.

The final intention of this experiment is to give an educated guess for the values W_{A-max} and σ_S of activated charcoal "Norit". Acetic acid will be the adsorbate. This will be done through the measurement of W_A for different concentrations of acetic acid and then via lineal regression will be possible to get W_{A-max}. σ_S is then gotten directly by equation 7.

II. EXPERIMENTAL PROCEDURE

The materials needed for this experiment are 6 funnels, 1 special stand for funnels, 50 mL Burette 50 (1), 50 mL volumetric flask (1), 1 clamp for burette, 10 mL volumetric pipette (1), filter paper, 1 retort stand, 125 mL Erlenmeyer flasks (12), 6 rubber stopper No. 4, Activated Charcoal "Norite", Acetic Acid 0.4 M, Sodium Hydroxide 0.1 M, and Phenolphthalein.

The scale was set to 0 after setting each of them on top. After calibrating the scale, 1(+-0.001) grams of activated charcoal were added and then put into 250 mL flasks.

Six Erlenmeyer flasks were rinsed with water to prevent contamination and dried out. Acetic acid solution was placed to each flask with the concentrations as shown in table 1.

TABLE I: Elements used for solutions prepared with 50 mL volumetric flasks

Solution #	Water(mL)	Acetic Acid(mL)
1	0	50
2	25	25
3	35	15
4	42.5	7.5
5	46	4
6	48	2

Erlenmeyer flasks (covered) were set on the shakers using clamps and left to shake for 1 hour. Using 6 more clean Erlenmeyer flasks, 6 funnels, and 6 pieces of filter paper, mixtures were filtered and flasks labeled.

Once the 6 solutions were filtered and stored on Erlenmeyer flasks, 2 samples of 10 mL are taken from flask 1 and 2, 2 samples of 20 mL from flasks 3 and 4, and 1 sample of 40 mL for flasks 5 and 6, for a total of 10 samples.

A 50 mL Burette was set in a special stand for funnels and NaOH was poured inside. A flask with the first sample was set below the Burette and 2 drops of Phenophthalein were added. The inicial amount of NaOH was registered. While shaking lightly the Erlenmeyer flask, the Burettes knob was turned to let the NaOH drop and closed once a purple color was noticeable. The volume of NaOH in the Burette was measured one more time. This process was repeated for every sample.

III. RESULTS

TABLE II: Experimental measurements for different concentrations of Acetic acid.

Acetic Acid (ml)	Molarity	Vai	V NaOH	[A]l,2	V NaOH 2
50	0.4	10	34.2	0.342	33
25	0.2	10	13.8	0.138	14.8
15	0.12	20	9.9	0.0495	12.5
7.5	0.06	20	2.3	0.0115	2.5
4	0.032	40	1.6	0.004	x
2	0.016	40	1.5	0.00375	x

TABLE III: Experimental measurements for different concentrations of Acetic acid.

A. Acid	[A]l,2 2	Wai1	Wai2	[A]l,2 Avg	Wai Avg	A/W
50	0.33	0.029	0.035	0.336	0.032	10.5
25	0.148	0.031	0.026	0.143	0.0285	5.0175
15	0.0625	0.03525	0.02875	0.056	0.032	1.75
7.5	0.0125	0.02425	0.02375	0.012	0.024	0.5
4	0	0.014	0.2	0.002	0.107	0.01869
2	0	0.00612	0.2	0.001875	0.10306	0.01819

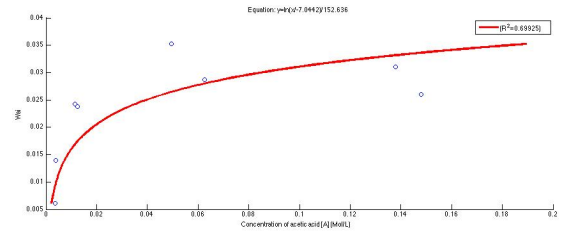A phase diagram was adapted from [3] to display the results, as shown in figure 2.

FIG. 1: Number of moles of acetic acid adsorbed by one gram of "Norit A" activated charcoal (WA) with respect to the equilibrium concentration of aqueous acetic acid solution [A], at room temperature.Measurements taken at the laboratory

Figure 2 depicts a scatter plot between the number of moles of acetic acid adsorbed and the change in the concentration [1]. Behavior of data fits onto the model. More data is neccessary to determine accurate regression coefficients.
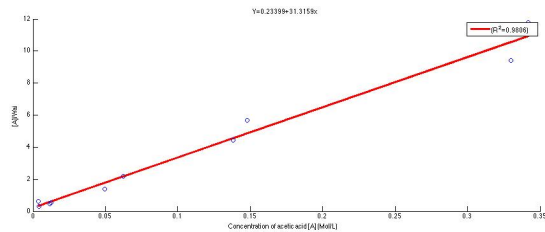
2.

FIG. 2: Number of moles of acetic acid adsorbed by one gram of "Norit A" activated charcoal (WA) with respect to the equilibrium concentration of aqueous acetic acid solution [A], at room temperature.Measurements taken at the laboratory.

IV.   DISCUSSION OF RESULTS

A logarithmic regression was used for figure 2 based in the predicted behavior of the activated charcoal reaching a saturation phase where the molecules of acetic acid are not being adsorbed by the charcoal anymore.

Figure 3 shows linear behavior between the relation [A]/Wa and [A]. This is consistent with the prediction coming from Langmuir's model (equation 6). Values for the β_0 (which is directly related to the adsorption constant) is around 0.23. Value for β_1 (related to the maximum quantity of acetic acid adsorbed by activated charcoal)[3] has a value of 31.3159 It would be neccessary to make more measurements to perform a more accurate regression model. Results may vary from other experiments due to different circumstances such as not accounting for adsorbate-adsorbent interactions or the quality of activated charcoal.[4]

Specific area was calculated once Wa and Wa-max were known, using equation (7) .Our results for specific area of activated charcoal are then $\sigma = 7.58 x 10^2$ and they compare with those reported in the literature ($\sigma = 7.08 x 10^2$) with a relative error of 7% (Using Wa1).[3]

V.   CONCLUSION

-Characterization of adsorption phenomena in materials like charcoal are relevant to the manufacturing or chemistry industry, where it is neccessary to filter substances (such as paint gases) or purify areas from odors.[7] Langmuir's adsorption model may be adequate for this type of characterization, given the fact that it is able to determine adsorption constants for values of temperature and pressure where the materials may be used. During our experiment we were able to demonstrate that this model is able to describe adsorption phenomena. Values for correlation coefficient in both relation graphs (Wai vs [A] for figure 2 and [A]/Wai vs [A] for

figure 3) show decent fitting values and are in agreement with predicted behavior based on the model (equation 6). It is neccessary to take in account different factors that may have deviated ours from the desired results. Most relevant of these factors involve lack of expertise when using phenolftalein in the measurement of the adsorbed contentration and lack of time to mare more measurements.

VI.   REFERENCES

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