The University of Jordan

School of Engineering

Chemical Engineering Department

Chemical Engineering Laboratory (3)

Experiment #7

Adsorption of Dye Solution on Activated Carbon

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Abstract

Adsorption is a separation process widely used in various industries, and the design of processes involving adsorption requires, much like many other separation processes, both equilibrium and kinetics data.

This experiment was conducted with the purpose of finding the concentration-time curve that describes the adsorption of dye solution by activated carbon (kinetics data), as well as finding the equilibrium isotherm for the adsorption of the dye on activated carbon (equilibrium data). The effects of agitation speed and initial dye concentration on the concentration-time curves were also to be investigated. Batch adsorption was studied. The Freundlich isotherm model was used to describe the behavior of the system.

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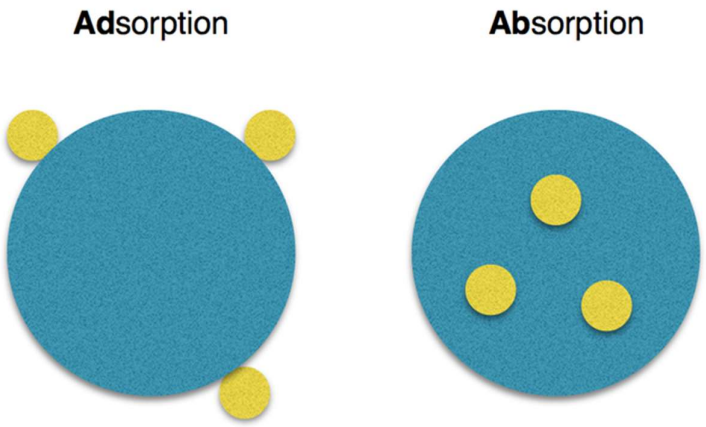
Introduction

According to Professor Dąbrowski, the technological, environmental, and biological importance of adsorption can hardly be overstated. The process of adsorption has found practical application in industry and in environmental protection, and it has proven effective in solving problems related to the purification of water, sewage, air, and soil. Examples of the aforementioned industrial applications include heterogeneous catalysts, activated carbon, and the capturing and usage of waste heat.

The study of adsorption requires an interdisciplinary approach—the process lies in the proverbial area where physics, chemistry, biology and engineering meet. IUPAC defines adsorption as 'an increase in the concentration of a substance at the interface of a condensed and a liquid or gaseous layer owing to the operation of surface forces.'

As the name implies, it is similar to absorption, but different in several important ways. The image below, courtesy of the Daily Garden, illustrates the most basic and crucial difference between the two phenomena:



As the definition states and the picture above illustrates, adsorption is a surface phenomenon, and like surface tension it is a consequence of surface energy.

The experiment we conducted places particular emphasis on *activated carbon.* Activated carbon is an adsorbent that has many applications and is highly versatile. It is purified and powdered charcoal, treated to generate microfissures so as to increase its surface area. The surface area per unit mass of activated carbon can be as high as 2000 m^2/g. Activated carbon is used in the purification of water and various pollutant gases, as well as for solvent recovery and the retention of toxic gases. It can also be used for the control of krypton and xenon

radionuclides from nuclear power plants. It is used in the production of food and beverages as well as the chemicals and pharmaceuticals industries.

A lot of emphasis was also placed on Safranin-O. According to Florida State University, safranin O is a stain used in microscopy that appears, at room temperature, as dark red to brown crystals. It must be handled with care. According to both FSU and Lab Chem, safranin O can cause skin irritation and in certain cases serious eye damage.

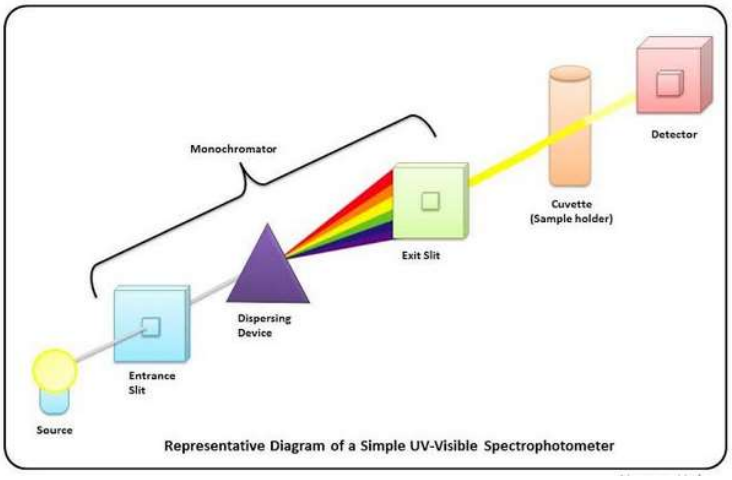
In short, adsorption has many applications, and like all other important separation processes, it is paramount that we have quality data that can be used for unit and process design. Both equilibrium and kinetics data matter here—the engineer tasked with designing a process involving adsorption must be aware of the theoretical limits of the process (equilibrium) as well as optimal unit size and flow rates (kinetics). As such in this experiment we will produce and present both concentration-time curves as well as equilibrium curves.

Apparatus

As stated in the manual, the experiment requires the use of 3 two-liter beakers, 3 variable motors, 50 small bottles and a visible spectrophotometer. We will only describe the spectrophotometer and the variable, as the rest are basic, unremarkable equipment.

The picture below, courtesy of LaboratoryInfo, shows the basic structure of a spectrophotometer.

A spectrophotometer is a device used in many fields for the identification and quantifying of microscopic samples, as well as the to check for the quality of a product. It works like this: a light is shined through a given sample, and the monochromator splits the light into individual wavelengths. The light is then read and interpreted as shown in the diagram below (also courtesy of LabratoryInfo):



There are many kinds of spectrophotometers. The one we require for this experiment is a visible light spectrophotometer, which means that it uses visible light, typically from a tungsten lamp.

As for variable speed motors, they are simply motors for which the number revolutions per minute (RPM) is variable. They are, intuitively enough, typically

used to test the effect of motor speed on a given output variable. The speed is usually controlled via a dial similar to the one shown below:

Theory

We will be using the Freundlich equation, otherwise known as the Freundlich adsorption isotherm, to model the system at hand. The Freundlich equation is an empirical relationship between the quantity of either a gas or a liquid solute adsorbed into a solid surface and either the pressure of the gas or the concentration of the solute, respectively.

The Freudlich equation is expressed as (for gasses):

$$log\frac{x}{m} = logK + \frac{1}{n}logP$$

Or, equivalently:

$$\frac{x}{m} = K \cdot P^{\frac{1}{n}}$$

For liquids, the equation takes the form:

$$\frac{x}{m} = K \cdot C^{\frac{1}{n}}$$

Where:

- x is the mass of the adsorbate,
- m is the mass of the adsorbent,
- P is the equilibrium pressure of the gaseous adsorbate,
- C is the equilibrium concentration of the adsorbate,
- K and n are empirical parameters.

The math tells us that as the concentration of solute increases in a liquid solution, so too does the mass adsorbed by the adsorbent. The same logic applies for gas-phase adsorption: as the pressure of a given gas increases, the mass of that gas which is adsorbed increases too.

Procedure

Isotherms

1. Dissolve one gram of dye in two liters of water.
2. Weigh out varying amounts of carbon in the small bottles.

Batch Studies

1. Put 1.7 liters of dye solution into each of the three two-liter beakers.
2. Adjust the speed of the agitator to three different values in the beakers (100, 200, 300rpm).
3. Add 0.085g of carbon into each beaker. Take samples from each beaker after (1, 2, 3, 4, 5, 9, 15, 25, 35, 49, 64, and 80 minutes).
4. Repeat steps 1 and 2, but with the amount of carbon increased to 1.2 g.

Results

Part 1

Table 1: Change of concentration of dye with time for activated carbon mass = 5g

Run 1					
V. of Solu.	500ml	Time	(Absor. or Trans.)	C(f)(ppm)	C(f)/C0
m. of Carbon	5g	1	27	6.95	0.993
SP. Agitator	250rpm	2	30	6.4	0.914
C0(ppm)	7	3	32	6.1	0.871
		4	37	5.2	0.743
		6	39	4.9	0.700
		9	46	3.9	0.557
		15	55	2.9	0.414
		25	69	1.2	0.171
		35	79	0.8	0.114
		49	86	0.5	0.071
		64	90	0.3	0.043
		80	92	0.2	0.029

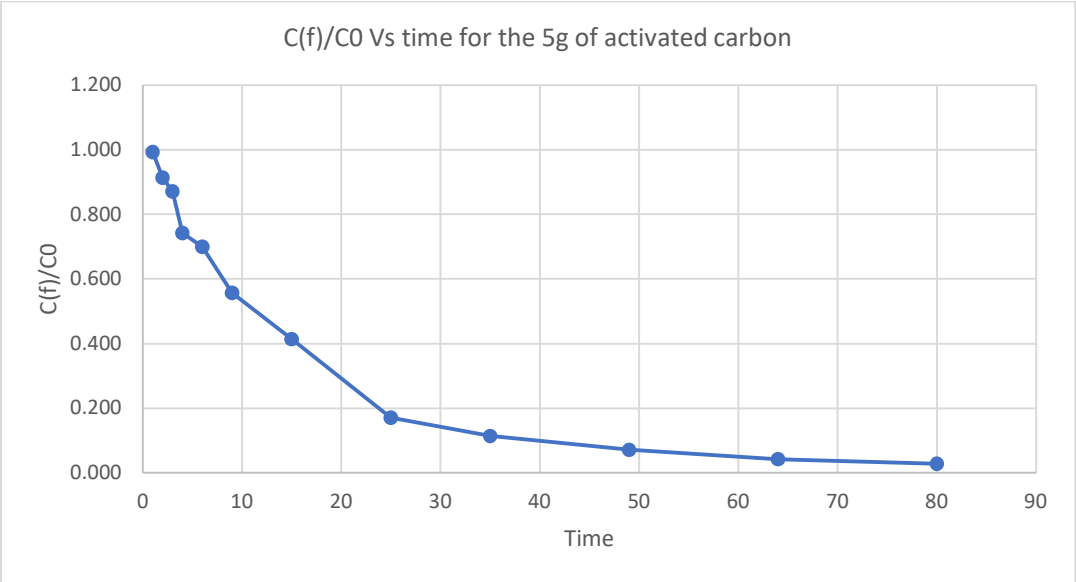


Figure 1: Plot of Cf/Co Vs time at constant activated carbon mass = 5g

Table 2: Change of concentration of dye with time for Activated carbon mass = 4g

2					
V. of Solu.	500ml	Time	(Absor. or Trans.)	C(f)(ppm)	C(f)/C0
m. of Carbon	4g	1	26	7	1.000
SP. Agitator	250rpm	2	28	6.8	0.971
C0(ppm)	7	3	29	6.5	0.929
		4	30	6.3	0.900
		6	33	5.8	0.829
		9	40	4.8	0.686
		15	44	4.2	0.600
		25	62	2	0.286
		35	66	1.7	0.243
		49	82	0.7	0.100
		64	89	0.4	0.057
		80	91	0.3	0.043

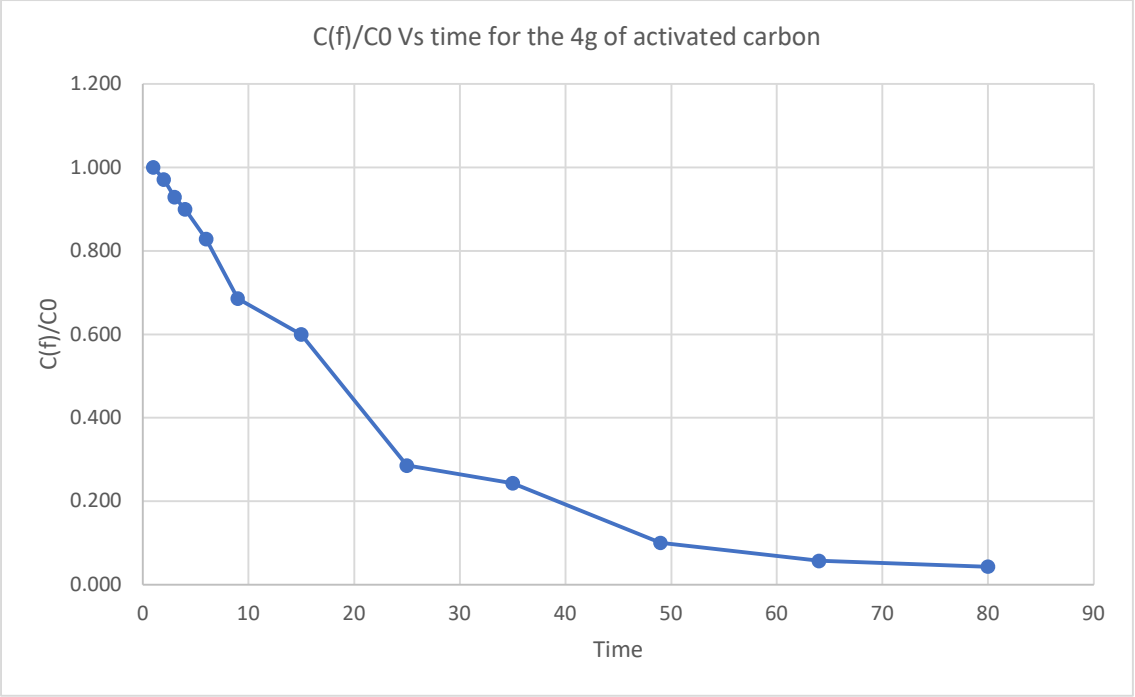


Figure 2: Plot of Cf/Co Vs time at constant activated carbon mass = 4g

Table 3: Change of concentration of dye with time for Activated carbon mass = 3g

3					
V. of Solu.	500ml	Time	(Absor. or Trans.)	C(f)(ppm)	C(f)/C0
m. of Carbon	3g	1	24	7.5	1.071
SP. Agitator	250rpm	2	26	7	1.000
C0(ppm)	7	3	27	6.9	0.986
		4	28	6.8	0.971
		6	31	6.15	0.879
		9	35	5.5	0.786
		15	42	4.45	0.636
		25	51	3.3	0.471
		35	60	2.2	0.314
		49	66	1.7	0.243
		64	71	1.1	0.157
		80	77	0.9	0.129

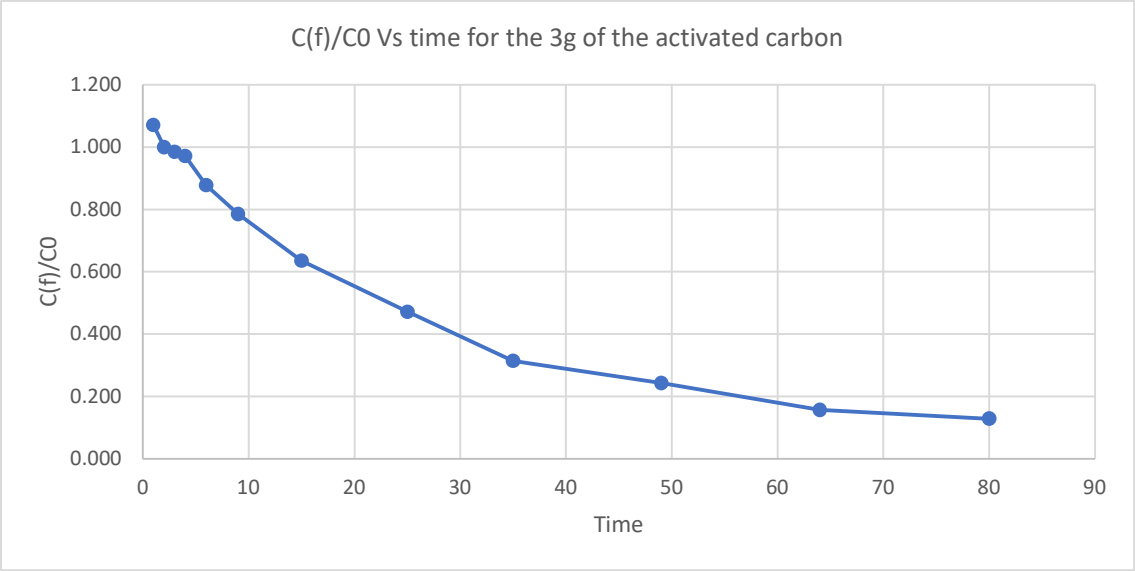


Figure 3: Plot of Cf/Co Vs time at constant activated carbon mass = 3g

Part 2

Table 4 : Freundlich Isotherm relation results

m(g)	Transmittance Reading	C(f)	$m\_absorbate$	$m\_absorbent$	q	log(q)	log(C)
0.001	12	11	0.014	0.033	0.42	-0.377	1.041
0.002	13.5	10.7	0.0143	0.067	0.2145	-0.669	1.029
0.004	20	8.5	0.0165	0.133	0.12375	-0.907	0.929
0.008	55	2.8	0.0222	0.267	0.08325	-1.080	0.447
0.012	80	0.8	0.0242	0.400	0.0605	-1.218	-0.097
0.016	87	0.6	0.0244	0.533	0.04575	-1.340	-0.222
0.02	89	0.45	0.02455	0.667	0.036825	-1.434	-0.347
0.03	92	0.2	0.0248	1.000	0.0248	-1.606	-0.699
0.04	93	0.1	0.0249	1.333	0.018675	-1.729	-1.000
0.08	95	0.05	0.02495	2.667	0.00935625	-2.029	-1.301
0.1	98	0.003	0.024997	3.333	0.0074991	-2.125	-2.523
0.15	99	0.005	0.024995	5.000	0.004999	-2.301	-2.301

V(ml)	C0(ppm)	log(K)	K	n
30	25	-1.1921	0.06425	0.4367

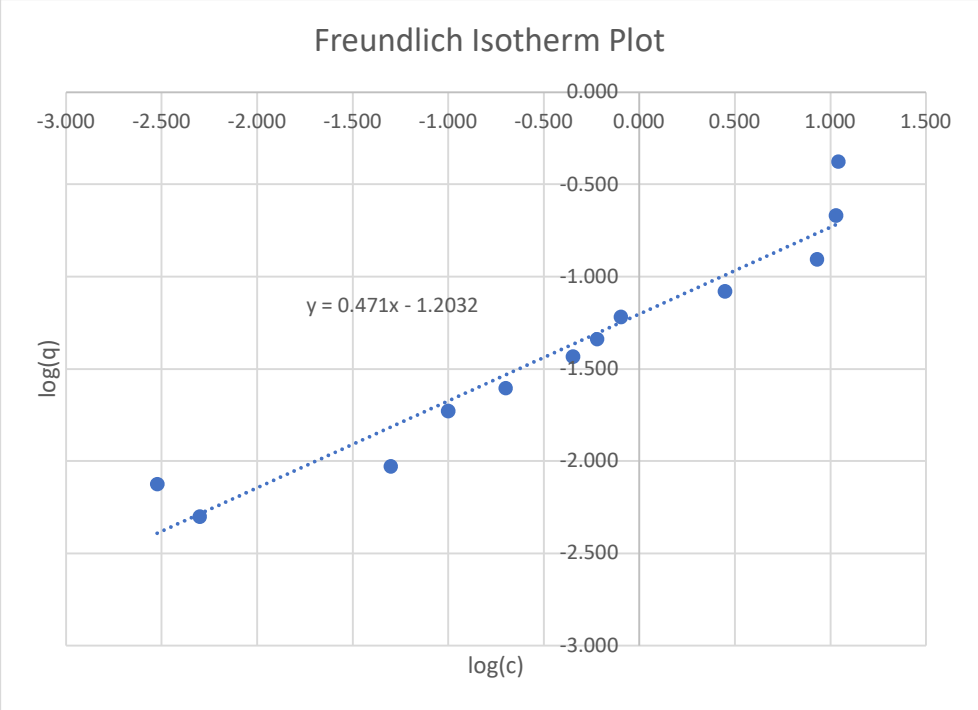


Figure 4: Freundlich Isotherm Plot

Discussion of results

Adsorption is a purification or separation process where one or more components of a gas or liquid stream are adsorbed on the surface of a solid adsorbent due to the forces of attraction (like Van der Waals forces) where the solute molecules go to surface and connected with adsorbent until equilibrium happens where no net change of the concentration of solute on the surface of adsorbent

The chosen adsorbent has to meet certain requirements for an efficient separation:

- Porosity and large surface area that results in high capacity.
- Selectivity.
- Low solubility in the carrier solvent.
- Low cost.

In our experiment the adsorbent was activated carbon and dye was adsorbed on the carbon surface from the dye solution.
Factors that affect our experiment were: the initial concentration of the dye, the speed of agitation of the motor and the weight of activated carbon that used.
For the batch studies part we did 3 runs at constant initial concentration of the dye *and* speed of agitation but at different weight of activated carbon and the results for each run were recorded in table 1,2 and 3. for run 1,2 and 3 where the weight of activated carbon 5, 4, and 3g and the final concentration of dye in the solution 0.2, 0.3 and 0.9 ppm respectively
From those results we can notice that as the weight of activated carbon increases the final concentration of dye in the solution (Cf) become smaller which was expected due to increase in the surface area that available for adsorption. In addition, from Figure 1,2 and 3 which show *the concentration-time curve*, it is clearly that as the weight of activated carbon increases, the time needed to adsorb the same amount of dye on the surface decreases.

For the isotherm part, we found the equilibrium isotherm for the adsorption by using 30 ml of 25 ppm initial concentration and leave it for a long time until reach equilibrium. After that we need to test our data in isotherm model and for our case we used Freundlich adsorption isotherm. From Figure 4 it is clearly that the Freundlich adsorption isotherm does not describe our system because the relationship is not linear.

Conclusion

- Two procedures are studied; isotherm and kinetics.
- Equilibrium isotherm represents the change of mass transferred with equilibrium concentration until it becomes constant due to saturation of the active site which don't allow further adsorption to take place.
- Physical adsorption is caused mainly by van der Waals forces and electrostatic forces between adsorbate molecules and the atoms which compose the adsorbent surface.
- A large specific surface area is preferable for providing large adsorption capacity
- As the weight of activated carbon increases the final concentration of the dye in the solution decreases
- Isotherm data give a good estimation about the capacity of the adsorbent; (How much solute "dye" can be adsorbed per unit mass of adsorbent)
- There were some errors in our data and that because of
    - Personal errors: in the preparation of the solution, determining the time.
    - Experimental error: the accuracy of spectrophotometry

References

1. *Skoog; et al. (2007). Principles of Instrumental Analysis (6th ed.). Belmont, CA: Thomson Brooks/Cole. pp. 169–173. ISBN 9780495012016.*

2. *John K. Brennan, Kendall T. Thomson, and Keith E.Gubbins,Adsorption of Water in Activated Carbons: Effects of Pore Blocking and Connectivity, 2002 American Chemical Society, Published on Web 06/13/2002.*

3. *Competitive Adsorption of dyes (Congo red, methylene blue, malachite green) on Activated Carbon. Under the guidance of Prof. S. Mishra.*

4. *Treybal, "Mass Transfer Operations".3rd.ed. McGraw Hill.*
5. https://www.sciencedirect.com/topics/agricultural-and-biological-sciences/activated-carbon
6. https://www.sciencedirect.com/science/article/abs/pii/S0001868600000828
7. https://www.labchem.com/tools/msds/msds/75583.pdf
8. https://micro.magnet.fsu.edu/primer/techniques/polarized/gallery/pages/safraninosmall.html
9. https://laboratoryinfo.com/spectrophotometer/

10. *Laboratory Manual for "Chemical Engineering Laboratory (3)" by the University of Jordan*

Notation

symbol	Description
C_f	Concentration of dye at any given time
Co	Initial concentration of dye in the solution
K , n	Freundlich isotherm constants
q	Mass of absorbate over the mass of absorbent
V	Volume of the solution
m	Mass of the activated carbon

Appendices

Sample of calculation

Part a

***Freundlich isotherm**:*

$$q = \frac{m_{absorbate}}{m_{absorbent}} = K * {C_f}^n$$

$$Log(q) = Log(K) + n * Log(C_f)$$

Taking the 1st point as our sample of calculation

$V_{solution}$ (ml)	C_0(ppm)	m(g)
30	25	0.001

1st we found the transmittance reading using the calibration curve shown in figure 2 at m=0.001 ---- > *transmittance reading =12*

2nd from the calibration curve shown in figure 1 at transmittance reading =12 ---- > C_f*=11*

$$m_{absorbate} = C_o - C_f = \frac{25 - 11}{1000} = 0.014\ \frac{g}{L}$$

$$m_{absorbent} = \frac{m}{V} = \frac{0.001 * 1000}{30} = 0.033333\ \frac{g}{L}$$

$$q = \frac{m_{absorbate}}{m_{absorbent}} = \frac{0.014}{0.033333} = 0.42$$

$$Log(q) = -0.377$$

$$Log(C_f) = 1.041$$

The same approach for other point and we then ended with the table below:

log(q)	log(C)
-0.377	1.041
-0.669	1.029
-0.907	0.929
-1.080	0.447
-1.218	-0.097
-1.340	-0.222
-1.434	-0.347
-1.606	-0.699
-1.729	-1.000
-2.301	-2.301

The best fit for plotting Log(q) Vs Log (Cf) given below:

$$Log(q) = 0.471 * Log(C_f) - 1.2032$$

$$Slope = n = 0.471$$

$$Intercepet = Log(K) = -1.2032$$

$$K = 10^{-1.2032} = 0.062633$$

Part b

We take the 1st run as sample of calculation

V. of Solu.	500ml	Time	(Absor. or Trans.)
m. of Carbon	5g	1	27
SP. Agitator	250rpm	2	30
C0(ppm)	7	3	32
		4	37
		6	39
		9	46
		15	55
		25	69
		35	79
		49	86
		64	90
		80	92

From Figure 1 we can find the concentration of the dye in solution corresponding to the transmittance reading. see the table below:

C(f)(ppm)	C(f)/C0
6.95	0.992857
6.4	0.914286
6.1	0.871429
5.2	0.742857
4.9	0.7
3.9	0.557143
2.9	0.414286
1.2	0.171429
0.8	0.114286
0.5	0.071429
0.3	0.042857
0.2	0.028571

$$\frac{C_f}{C_o} = \frac{6.95}{7} = 0.992857$$

Calibration curves

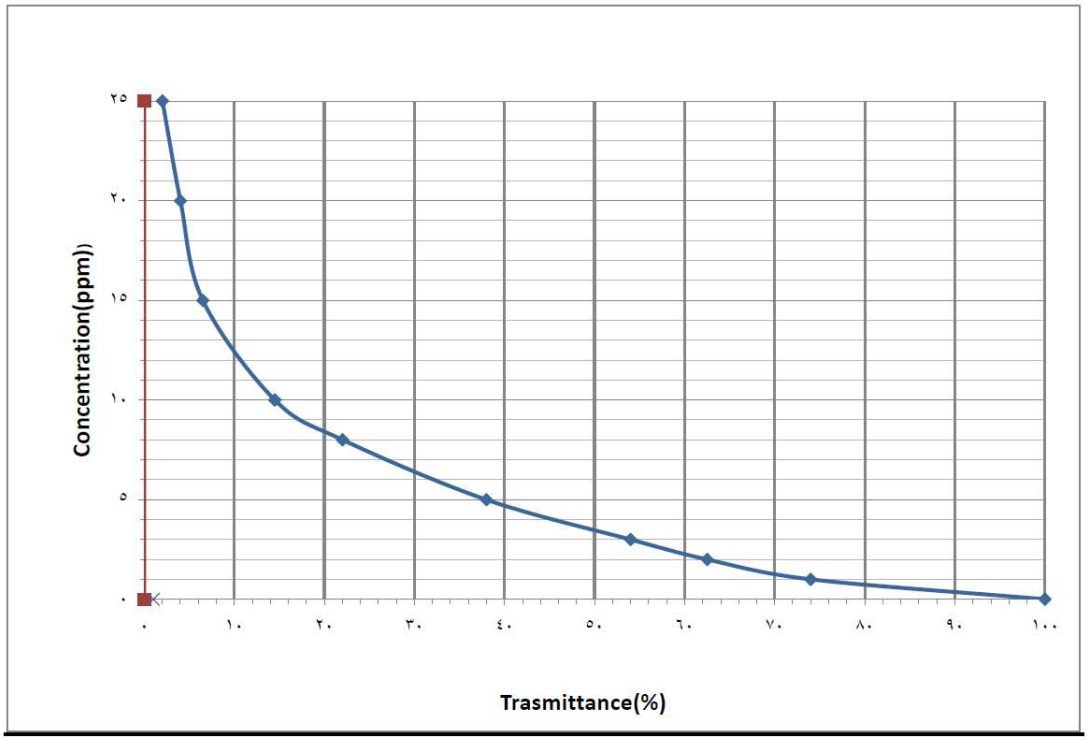


Figure 5: Calibration curves for Concentration of dye and Transmittance

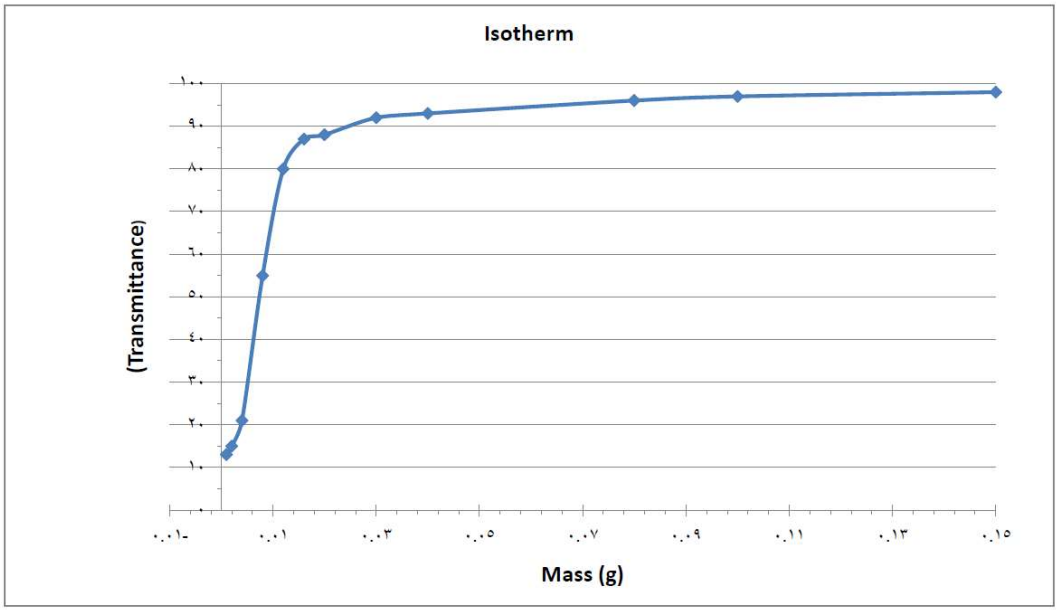


Figure 6:Calibration curves for Mass of dye and Transmittance

Data Sheet

a. Isotherms

Volume of Solution	**30ml**
Initial Concentration of Solution	**25ppm**

Mass of Carbon (g)	**Reading of (Absorbance OR Transmittance) at equilibrium**
0.001	
0.002	
0.004	
0.008	
0.012	
0.016	
0.020	
0.030	
0.040	
0.080	
0.100	
0.150	

b. Batch Studies

Run Number: _____1_____

Volume of the Solution	**500ml**
Mass of Carbon	**5g**
Speed of Agitator	**250rpm**
Initial Concentration of Solution	**7ppm**

Time	Reading of (Absorbance OR Transmittance)
1	**27**
2	**30**
3	**32**
4	**37**
6	**39**
9	**46**
15	**55**
25	**69**
35	**79**
49	**86**
64	**90**
80	**92**

Run Number: _____2_____

Volume of the Solution	**500ml**
Mass of Carbon	**4g**
Speed of Agitator	**250rpm**
Initial Concentration of Solution	**7ppm**

Time	Reading of (Absorbance OR Transmittance)
1	**26**
2	**28**
3	**29**
4	**30**
6	**33**
9	**40**
15	**44**
25	**62**
35	**66**
49	**82**
64	**89**
80	**91**

Run Number: ____3_____

Volume of the Solution	**500ml**
Mass of Carbon	**3g**
Speed of Agitator	**250rpm**
Initial Concentration of Solution	**7ppm**

Time	Reading of (Absorbance OR Transmittance)
1	**24**
2	**26**
3	**27**
4	**28**
6	**31**
9	**35**
15	**42**
25	**51**
35	**60**
49	**66**
64	**71**
80	**77**