

**STUDY OF ADSORPTION OF OXALIC ACID
AT DIFFERENT CONCENTRATIONS ON
CHARCOAL/RICE HUSK ASH AND
HENCE TO VERIFY FREUNDLICH'S
ADSORPTION ISOTHERM**

**PROJECT REPORT
(2009-10)**

By

C.Masharing & D.L.Buam

**Department of Chemistry,
Shillong College**

Submitted

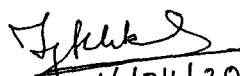
to

**Research Sub-Committee,
Shillong College,
Shillong-3**

CERTIFICATE

This is to certify that C.Masharing and D.L.Buam, Department of Chemistry, Shillong College have completed the Project titled, 'Study of Adsorption of oxalic acid at different concentrations on Charcoal/Rice Husk Ash and hence to verify Freundlich's Adsorption Isotherm' for the year 2009-10.

The Project report is now being submitted to the Research Sub-Committee, Shillong College, Shillong on the 10th April 2010 .


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T.J.Kharbhih

Head, Department of Chemistry,
Shillong College, Shillong- 3

Study of Adsorption of oxalic acid at different concentrations on Charcoal/Rice Husk Ash and hence to verify Freundlich's Adsorption Isotherm

C.Masharing & D.L.Buam
Department of Chemistry,
Shillong College

Abstract:

Adsorption is a well-known surface phenomenon where one substance getting adsorbed is termed as adsorbate and the other substance on whose surface adsorption takes place is termed as adsorbent. Adsorbate can be liquid or gas whereas adsorbent is always a solid substance. The adsorptive behavior of activated charcoal and rice husk ash for oxalic acid from aqueous solutions has been investigated as a function of appropriate equilibrium time, amount of adsorbent, concentration of adsorbate. Studies showed that with decreasing concentration adsorption decreases. The maximum adsorption was about 36.0 and 62.8% for activated charcoal and rice husk ash respectively at pH 6, contact time 180 min and initial concentration of 0.05 N. The oxalic acid adsorption obeyed the Freundlich's Adsorption isotherm. The present study showed that rice husk ash was more favorable than activated charcoal in removing oxalic acid and thus is a better adsorbent.

Keywords: Oxalic acid, activated charcoal, rice husk ash, adsorption, aqueous system

Introduction:

Adsorption is a phenomenon in which particles adhere to the surface of solids, the particles instead of penetrating inside remain relatively more concentrated near or on the surface. In other words, we can say the concentration of one component in the vicinity of the surface is different from that in the bulk. Adsorption occurs because of the unsaturation of the surface or the excess free energy of the surface. Based on the nature of interaction of the adsorbate molecules with the surface of adsorbent, adsorption is classified into two types- Physical Adsorption or Physisorption and Chemical Adsorption or Chemisorption. Physical adsorption occurs when the interaction is vander Waals type, and Chemical adsorption occurs when the interaction is chemical in nature. Generally physical adsorption takes place at low temperature on a surface i.e it is exothermic process while on the same surface, chemisorption is favoured at a higher temperature. The substance on the surface of which

adsorption occurs is the adsorbent and the substance that gets adsorbed is termed adsorbate. The magnitude of adsorption depends upon i) the nature of the adsorbent and its state of subdivision ii) the nature of the adsorbate iii) its concentration or pressure and iv) the temperature.

Known Practical applications of Adsorption

There are innumerable industrial processes starting from the synthesis of ammonia to the manufacture of alcohol or synthesis of petrol, or heterogeneously catalysed reactions which involve the process of adsorption. Most of these catalytic reactions are effective through adsorption of reactants on solid surfaces. The stabilization of colloid through adsorption of ions on the particles is of considerable interest both from the practical and theoretical stand point.

The function of gas mask is based on the preferential adsorption of harmful gases present in the atmosphere by the use of suitable adsorbents in order to purify the air from inhalation. The role of charcoal in the recovery of iodine and many dyes from their dilute solution, or its role in the clarification of syrups in sugar industry, etc is based on its adsorption capacity.

Adsorption is widely used now a day in separating and analyzing valuable and minute quantities of component from a mixture. A suitable adsorbent, often used is finely powdered aluminium oxide, magnesium oxide or charcoal. The principle involved in the process is known as chromatographic method. Another application of adsorption is found in the use of ion exchangers. One such example is water softening by using Zeolites which has the capacity to exchange some of the cations they contain for others in solution.

There are several methods to treat the metal contaminated effluent such as precipitation, ion exchange and adsorption etc, but the selection of the treatment methods is based on the concentration of waste and the cost of treatment. Carbon has been the most used adsorbent; nevertheless it is relatively expensive. Cost is an important parameter for comparing the sorbent materials. However, cost information is seldom reported and the expense of individual sorbents varies depending on the degree of processing required and local availability. In general, a sorbent can be assumed as low cost if it requires little processing, is abundant in nature, or is a by-product or waste material from another industry. Activated carbon from cheap and readily available sources such as coal, coke, peat, wood,

rice husk may be successfully employed for the study of adsorption of oxalic acid in aqueous solution. In the last few years, adsorption has been shown to be an economically feasible alternative method, the study on the adsorption of gaseous liquid metal has been carried out using rice husk ash (henceforth abbreviated as RHA) as adsorbent. In many cases, RHA shows excellent adsorption capacity compared to other known adsorbents. The objective of this study was to explore the feasibility of rice husk ash as an adsorbent as well as to compare the adsorption capacity between rice husk ash against charcoal for the removal of oxalic acid from aqueous solution. Moreover it will help the student in understanding better how different materials are adsorbed differently. The type of adsorption that takes place is mainly the physical adsorption or physisorption. The parameters that influence adsorption such as initial oxalic acid concentration, agitation time were investigated.

Source of Adsorbent :

The rice husk used was obtained from one of the rice mills situated in Ri Bhoi district of Meghalaya. The rice husk is kept and burnt in an open space for 6 hours, it is crushed and thoroughly washed with distilled water to remove all dirt and then dried at 100°C to be of constant weight. The dried husks is sieved in order to obtain in powder form and finally stored in desiccator until it is used.

Apparatus Required:

Electrical shaker, Beakers, Burette, Pipette, Charcoal, RHA, Oxalic acid solutions.

Methodology:

Freundlich's adsorption isotherm is an empirical relation showing that the amount of adsorbate per unit mass of adsorbent depends on the concentration or pressure of the adsorbate as shown by the equation,

$$x/m = kC^{1/n} \dots\dots\dots(1)$$

where x = mass of oxalic acid = $(C_0 - C)MV$,

m = mass of Charcoal/RHA

M = molecular weight of Oxalic Acid

V = Total volume of solution

Taking log of the above expression,



$$\log (x/m) = \log k + 1/n \log C_0 \quad \dots\dots\dots(2)$$

Experimental Procedure:

1. Stock Solutions of 0.05 N Oxalic Acid and 0.02 N NaOH are prepared
2. NaOH solution is standardized with standard solution of oxalic acid using phenolphthalein indicator.
3. Oxalic acid solution is taken in four labelled-reagent bottles as follows :

Bottle No	Volume of Oxalic Acid (ml)	Volume of Water (ml)
1	100	0
2	50	50
3	25	75
4	12.5	87.5

4. In each reagent bottle, 2 gm of charcoal is added and then put in the shaker for at least 2 hours until equilibrium is attained.
5. The bottles are then removed from the shaker, allowed to stand and then filtered.
6. Pipette out 10 ml of the filtrate into the conical flask, add one drop of phenolphthalein indicator and titrate against 0.02 N NaOH solution from the burette till the colour changes to light pink.
7. The titration is repeated for at least twice for each bottle to get two consecutive readings same.
8. The experiment is repeated all over using Rice Husk Ash (RHA) in place of charcoal
9. A graph is then plotted with log x/m as ordinate and C₀ as abscissa when a straight line is obtained making an intercept with the ordinate. The intercept made by the line is measured wherefrom the value of k is calculated. The slope of the line gives the value of n.

Results and Discussions:

The adsorption of oxalic acid in aqueous solution on charcoal and rice husk ash (RHA), were examined by optimizing various physicochemical parameters such as contact time, concentration amount of adsorbent and adsorbate.

Table I & II a) show the data obtained using Charcoal as adsorbent while Tables I & II b) show the data obtained using RHA as adsorbent and the discussions to

compare and contrast the results using the two types of adsorbents are of interest in making a choice of selecting the adsorbent for practical purposes.

Table I a) : Concentration of oxalic acid before and after adsorption on Charcoal

Bottle No.	Volume in ml		Concentration of Oxalic acid (N)	
	Oxalic acid	NaOH	before	after
1	10	16.0	0.05	0.0320
2	10	6.6	0.025	0.0132
3	10	2.6	0.0125	0.0052
4	10	1.2	0.00625	0.0011

Table II a) : Amount of adsorbate per mass of adsorbent (x/m) using Charcoal

Bottle No.	Weight of charcoal (g)	Concentration change of oxalic acid (C_0-C)	$x=(C_0-C)MV$	x/m	logx/m
1	2	0.018	226.8	113.4	2.050
2	2	0.0118	148.6	74.34	1.870
3	2	0.0073	91.98	45.99	1.660
4	2	0.00514	64.8	32.40	1.510

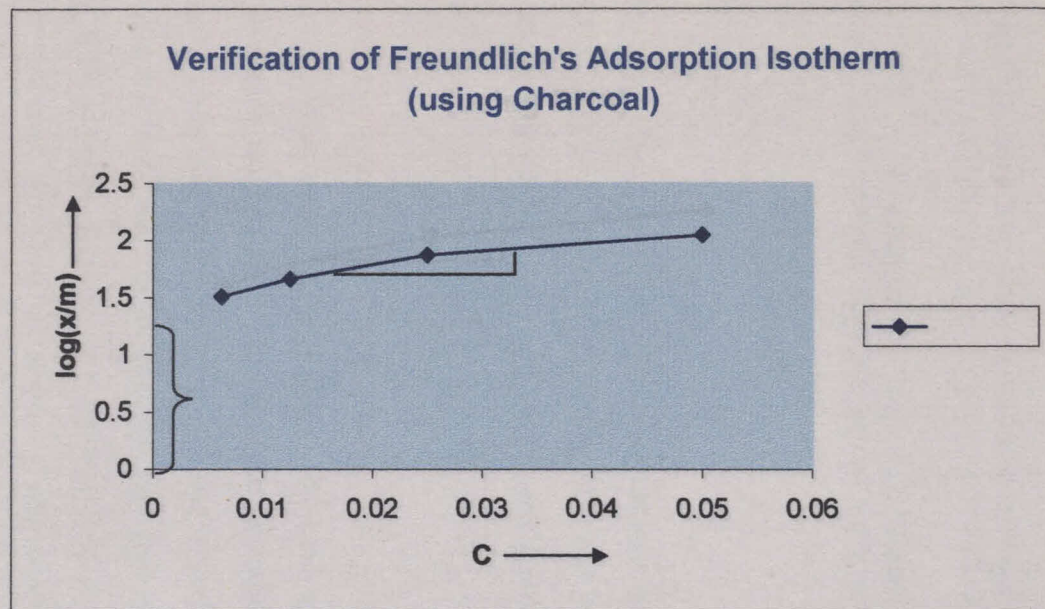
Table I b) : Concentration of oxalic acid before and after adsorption using RHA

Bottle No.	Volume in ml		Concentration of Oxalic acid (N)	
	Oxalic acid	NaOH	before	after
1	10	9.3	0.05	0.0186
2	10	4.1	0.025	0.0082
3	10	1.0	0.0125	0.0020
4	10	0.8	0.00625	0.00002

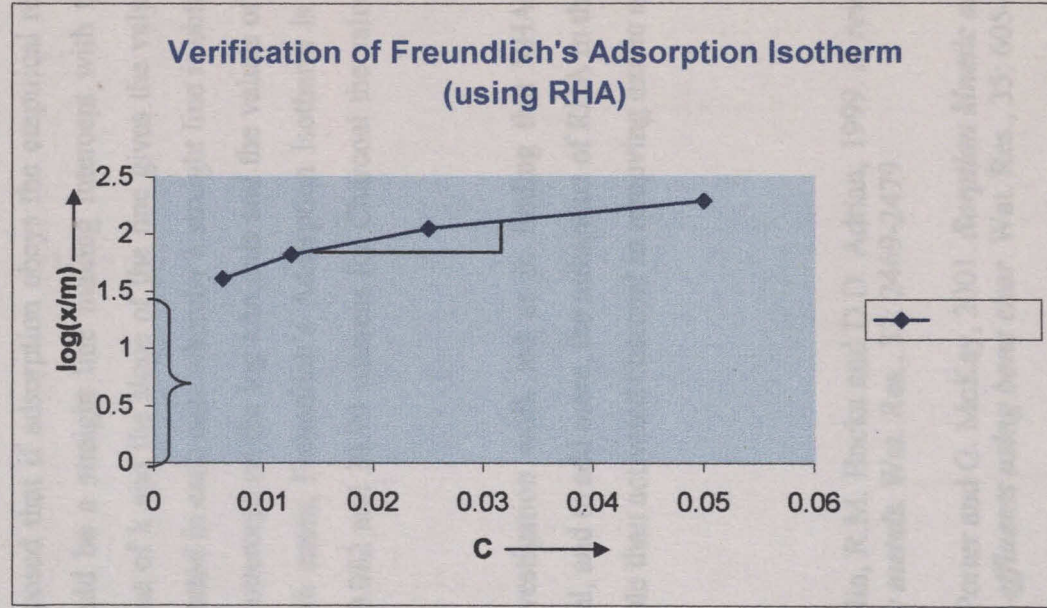
Table II b) : Amount of adsorbate per mass of adsorbent (x/m) using RHA

Bottle No.	Weight of RHA (g)	Concentration change of oxalic acid (C_0-C)	$x=(C_0-C)MV$	x/m	logx/m
1	2	0.0314	395.64	197.82	2.290
2	2	0.0168	211.68	105.84	2.024
3	2	0.0105	132.30	66.15	1.820
4	2	0.00648	81.60	40.80	1.61

C	log(x/m)
0.05	2.05
0.025	1.87
0.0125	1.66
0.00625	1.51



C	log(x/m)
0.05	2.29
0.025	2.05
0.0125	1.82
0.00625	1.61



The above results obtained give interesting observations which are of importance for discussion. Table I a) for all sets using charcoal as adsorbent clearly indicates that there is not much of difference between the concentration of oxalic acid before and after adsorption and in contrast to Table I b) for all sets using RHA as adsorbent showing a large difference in the concentration of oxalic acid before and after adsorption. One point to interpret here is that more amount of oxalic acid is getting adsorbed on to the RHA than to Charcoal surface. Another observation is that the amount of adsorbate adsorbed per mass of adsorbent is more in the case of RHA than of Charcoal as shown by the different values of x/m for all the four different sets.

Freundlich proposed that if adsorption obeys the empirical relation then the plot of $\log (x/m)$ Vs C_0 should be a straight line making intercept with the $\log (x/m)$ axis, the intercept gives the value of k and the slope of the line gives the value of n . A graph of $\log x/m$ Vs C_0 has been plotted in each case whereby a straight line is obtained in both cases, the straight line makes an intercept on the $\log x/m$ axis and the values of k and n are calculated from the graph. In both cases, Freundlich's Adsorption Isotherm is verified, for RHA the values of n and k are 0.082 and 30.90 whereas for Charcoal the values are 0.029 and 19.95 respectively.

Conclusion The investigation work led us in finding that RHA would make a better adsorbent than Charcoal, and to add more, the utilization of RHA in the process of adsorption would be more favorable than activated charcoal in removing oxalic acid and thus is a better adsorbent.

References

1. Bailey, S.E., T.J. Colin, R.M. Bncka and D.D. Adrian, 1999. *A review of potentially low-cost sorbents for heavy metals*. Wat. Res., 33: 2469-2479.
2. Cheung, C.W., J.F. Porter and G. McKay, 2001. *Sorption kinetic analysis for the removal of cadmium ions from effluents using bone char*. Wat. Res., 35: 605-621.
3. Dezuane, J., 1990. *Handbook of Drinking Water Quality Standards and Controls*. Van Nostrand Reinhold. New York, pp: 64-69.
4. Petemele, W.S., A.A. Winkler-Hechenleitner and E.A. GomezPmeda, 1999. *Adsorption of Cd (II) and Pb (II) on to functionalized formic lignin from sugar cane bagasse*. Bioresource. Technol., 68: 95-100.

5. Ajmal, M., R.A. Rao, S. Anwar, J. Ahmad and R. Ahmad, 2003. *Adsorption studies on rice husk: Removal and recovery of Cd (II) from wastewater*. Bioresource. Technol, 86: 147-149.
6. Gabaldon, C, P. Marzal and A. Seco, 1996. *Cadmium and Zinc adsorption on to activated carbon: influence of temperature, pH and metal/carbon ratio*. J. Chem. Technol. Biotechnol., 66: 279-285

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