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ADSORPTION OF STRYCHNINE SULPHATE BY VARIOUS
CHARCOALS AND BY LLOYD'S REAGENT

by

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PREFACE

The author wishes to thank Dr. John F. Suchy for his help and assistance in the development of this problem, and to acknowledge the donation of charcoals by Merck and Co., Industrial Chemical Sales Co., and the Pfanstiehl Chemical Company.

INTRODUCTION

This problem suggested itself during the course of an analysis in which charcoal was used as a decolorizing agent. In various phases of industry this same medium ranging from the crude technical product to the highly activated form finds much practical application.

The pharmacist in his field is often called upon to use charcoal either as a filtering or as an adsorbent medium and as a rule but little consideration is given to the amount of active medicinal ingredient which may be lost in the process. It is also of interest to know relatively the adsorbing power of these filtering media.

The present research was undertaken to gain some information as to the diminution of potency of strychnine sulphate solutions when brought in contact with a number of commercial charcoals and with Lloyd's Reagent. The results obtained show relatively the adsorbing powers of these media with respect to almost any dissolved substance.¹

A thorough search of the literature showed that very little work had been done on the adsorption of strychnine sulphate by charcoal. The only record found relating to this subject is pharmacological in nature,⁴ that is, F. Horst in 1921 used the action of strychnine sulphate solutions on animals following contact with various charcoals as the criterion of the quantity of alkaloid adsorbed. The commercial

charcoals used in the following experiments were not used in Horst's work.

THEORY

Adsorption⁶ is a term used to denote the formation of layers of molecules attached to or attracted by a solid surface of some other material. It is a species of chemical reaction but is somewhat looser than that of the usual chemical reaction.

Adsorption takes place to satisfy free atomic attractions present at the surface of all solids. In a solid body molecules lose their identity as such and each atom's attraction is split equally among the surrounding atoms. At the surface of a solid the atoms are not completely surrounded so their attraction in the outer direction must either be bent in some interior direction and there satisfied or this attraction must exist unsatisfied. This leaves a definite chemical attraction extending out into space.

The latter view appears to be the correct one since a crystal of some substance suspended in its saturated solution will attach more layers to it and thus grow in size.

Since adsorption is due to unsatisfied atomic attractions, the forces holding adsorbed molecules to a solid must be the same as those holding the atoms of a crystal together. From this it follows that whether adsorption takes place and if so to what extent, depends upon the magnitude of these forces, that is, upon residual valences in the atoms of the solid surface and in the adsorbed molecule.

The extent of adsorption³ is also dependent upon several other factors, namely: the extent of surface exposed, the pressure or concentration of the adsorbed substance, the temperature, the time of contact, the specific nature of the substances involved, and the surface tension of the solvent.

Thus the effect of variation of any one of these conditions may be studied by holding the others constant. A study of this type in which concentration was taken as the variable led Freundlich to the formation of his empirical equation to represent experimental facts. He plotted concentrations against quantities adsorbed and obtained curves which he called "adsorption isotherms." His equation, however, holds for these curves only within narrow limits of temperature and concentration.

Letting (x) equal the weight of substance adsorbed, (m) the weight of adsorbent and (c) the original concentration of the solution, Freundlich gave his equation as:

$$\left(\frac{x}{m}\right)^n = kc$$

where n and k are constants to be determined by experiment for each temperature.

Langmuir⁵ likewise developed an equation for adsorption which fits experimental data better over wider ranges of concentration and temperature than does that of Freundlich. Langmuir obtained his equation after a study of the adsorption of gases. He found that gas molecules impinging against

a solid or liquid surface do not rebound as expected but condense on the surface, being held there by the fields of force of the atoms of the solid or liquid. A relatively strong surface force then causes the formation of a layer of gas molecules one molecule thick, a second layer forming only thru the attraction of adsorbed molecules for more molecules of the same kind.

In the adsorption of gases on solid surfaces the forces involved are much stronger than the attractive forces between molecules of a liquid. The term liquid is applicable since the formation of a second layer of gaseous molecules on the solid is in effect the condensation of the gas as a liquid upon the solid surface. This condensation takes place only under proper conditions, namely a pressure and temperature quite close to the condensation temperature and pressure of the gas. Thus the formation of a second layer of gas molecules upon a solid surface takes place only under very restricted conditions.

Langmuir carried out his experiments at such a temperature and pressure that the formation of any second layer was quite improbable. Therefore, in the development of his equation he assumed a unimolecular layer of molecules on a surface, which amounts to a saturation limit for adsorption. He also assumed a plane surface of adsorbent in which all atoms in this surface are capable of adsorbing gas molecules.

This is perfectly legitimate since in the usual case where there is a surface which is not homogeneous and all molecules are not capable of adsorption, the portion of the surface capable of adsorption takes the place of the plane surface assumed.

If θ is the fraction of the surface occupied by adsorbed molecules, then $1 - \theta$ is the fraction unoccupied. The rate of condensation of gas molecules is proportional to the gas pressure, p , and the fraction of the surface still unoccupied, therefore,

$$\text{Rate of condensation} = k_1 p(1-\theta).$$

Adsorbed molecules will from time to time evaporate from the surface, and the rate of evaporation will be directly proportional to the amount of gas already on the surface, therefore,

$$\text{Rate of evaporation} = k_2 \theta.$$

At equilibrium these two processes will be equal, so,

$$k_2 \theta = k_1 p(1-\theta)$$

$$\text{or } \theta = \frac{k_1 p}{k_2 + k_1 p}$$

Using different constants, a and b , the weight of adsorbed material Q is given by:

$$Q = \frac{abp}{1 + ap}$$

The concentration C may be substituted for the pressure in this equation. It may then be written:

$$Q = \frac{abC}{1 + aC}$$

$$\text{or } \frac{C}{Q} = \frac{1}{ab} + \frac{C}{b}$$

If the quotient C/Q is plotted against C , a straight line should result with a slope of $1/b$ and a Y-intercept of $1/ab$. In this way the constants of Langmuir's equation may be determined.

In the present research the above values were plotted and the constants determined. These data are shown in the results and calculations which follow.

Experimental Procedure

Identical procedures were followed in all cases to show the variation of adsorption of strychnine sulphate by the various adsorbing media. Groups of from five to ten samples were run together, each sample representing a different concentration of strychnine sulphate.

The following general procedure was employed:

Exactly one gram of adsorbent was weighed out and a calculated quantity of strychnine sulphate added. To this was added exactly 100 cc. of distilled water and the mixture tightly stoppered and warmed on a steam bath until solution of the alkaloidal salt was affected. Each flask was then cooled to 25°C. and allowed to stand for 24 hours in a wall case where the variation of temperature was not over two degrees. The contents were shaken frequently to aid in reaching equilibrium. After the proper time interval, the mixtures were filtered and a 25 cc. portion of each was pipetted into a separatory funnel. The solution in each case was made alkaline with 5 cc. of 10 per cent ammonia and allowed to stand a short time until completion of precipitation. The strychnine was then entirely extracted with chloroform, the latter being added in small portions. Each chloroformic solution was filtered into a tared beaker which was then placed in a 50 degree oven where the solvent was evaporated. The residue was finally dried in another oven

at 100°C. for about 30 minutes, weighed and calculated as the sulphate. The difference between this result and the original weight of the sulphate gave the amount of the latter adsorbed per gram of charcoal.

Each sample of the strychnine salt used in this study was assayed by dissolving a carefully weighed quantity in water, making the solution alkaline with ammonia, and extracting the resulting alkaloid with chloroform. The strychnine thus obtained was calculated as the sulphate. Any deviation from the pure salt was taken into account in all subsequent determinations.

In order to obtain an idea of the amount of strychnine sulphate adsorbed by the filter paper used in the process, a series of blank determinations was run, the charcoal being omitted. Other details of the procedures were identical. The results obtained showed such extremely small deviations from no adsorption that any loss due to this factor was neglected.

RESULTS AND CALCULATIONS

I

Solvent - Water

Time - 24 Hours

Seven different charcoals and Lloyd's reagent were used in this investigation. The values obtained are shown in the following tables.

Table I

Merck's Medicinal Charcoal		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
1.0000	0.0000	0.1000
2.0000	0.0000	0.2000.
3.0000	0.0000	0.3000
4.0000	0.0000	0.4000
5.0000	0.0115	0.4885
6.0000	0.0660	0.5340
7.0000	0.1550	0.5450
8.0000	0.2472	0.5518
10.0000	0.4385	0.5615
15.0000	0.9237	0.5763

Table II

Nuchar		
Concentration - Gms. per liter	Wt. of residue	Amount adsorbed per Gm. of charcoal
1.0000	0.0000	0.1000
2.0000	0.0110	0.1890
2.5000	0.0349	0.2151
4.0000	0.1495	0.2505
7.0000	0.4278	0.2722
9.0000	0.6192	0.2808
12.0000	0.8893	0.3107

Table III

Carbex T		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
0.5000	0.0000	0.0500
1.0000	0.0000	0.1000
1.5000	0.0000	0.1500
2.0000	0.0220	0.1780
2.5000	0.0596	0.1904
3.5000	0.1472	0.2028
4.0000	0.1922	0.2078
6.0000	0.3885	0.2115
9.0000	0.6843	0.2157
15.0000	1.2649	0.2351

Table IV

Carbex E *		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
0.9164	0.0000	0.0916
1.3746	0.0000	0.1375
1.8328	0.0000	0.1833
2.7492	0.0459	0.2290
3.6556	0.0991	0.2665
4.5820	0.1647	0.2935
5.4984	0.2399	0.3099
7.3312	0.4202	0.3129
9.1640	0.6025	0.3139
10.9968	0.7836	0.3161
12.8296	0.9640	0.3190

* Results of Dr. John F. Suchy

Table V

Pfanstiehl Decolorizing Carbon *		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
0.4564	0.0000	0.0456
0.9128	0.0036	0.0877
1.8256	0.0383	0.1443
3.6512	0.1544	0.2107
4.5640	0.2241	0.2323
5.4768	0.3003	0.2474
6.3896	0.3670	0.2620
7.3024	0.4556	0.2746
8.2152	0.5391	0.2824
9.1280	0.6295	0.2833
10.9536	0.8111	0.2843
13.6920	1.0783	0.2909

Table VI

Bone Black *		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
0.9235	0.0563	0.0361
1.8470	0.1184	0.0663
2.7705	0.1757	0.1014
3.6940	0.2433	0.1261
4.6175	0.3048	0.1570
5.5410	0.3742	0.1799
7.3880	0.5237	0.2151
9.2350	0.6841	0.2394
11.0820	0.8485	0.2597
12.9290	1.0532	0.2597

* Results of Dr. John F. Suchy

Table VII

Willow Charcoal *		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
0.3974	0.0396	0.0001
0.7947	0.0784	0.0011
1.5894	0.1468	0.0121
2.3841	0.2144	0.0240
3.1788	0.2864	0.0315
3.9735	0.3684	0.0290
4.7682	0.4456	0.0312
9.5360	0.9220	0.0316

Table VIII

Lloyd's Reagent		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
1.0000	0.0000	0.1000
2.0000	0.0335	0.1665
3.0000	0.0738	0.2262
4.0000	0.1390	0.2610
5.0000	0.2224	0.2776
6.0000	0.3059	0.2941
8.0000	0.4852	0.3148
10.0000	0.6678	0.3322
12.0000	0.8609	0.3391

* Results of Dr. John F. Suchy

By plotting the original concentration of strychnine sulphate against the amount adsorbed per gram of charcoal, an isotherm was obtained in each case as is shown in Figures 1 and 2.

Merck's Medicinal Charcoal (Curve No. 1) proved by far the best adsorbent of the group, adsorption being complete up to a concentration of nearly 5 grams of alkaloidal salt per liter. None of the other charcoals used showed total adsorption beyond concentrations of 2 grams per liter. On the other hand, willow charcoal, the type used in most pharmaceutical operations, (Curve No. 7) proved by far the poorest adsorbent of the group, taking up only 11 per cent of the salt when the original concentration was but 1 gram per liter.

Bone Black (Curve No. 6) showed a surprisingly high adsorbing power. Like the willow charcoal there was no finite concentration at which total adsorption took place, but the percentage of adsorption dropped much less rapidly with increasing concentrations.

The other carbons used all proved intermediate in their affinity for the alkaloidal salt. Nuchar (Curve No. 2) and Carbox E (Curve No. 4) gave total adsorption in concentrations to almost 2 grams per liter, while Carbox T (Curve No. 3) and Pfanstiehl Decolorizing Carbon (Curve No. 5) adsorbed completely up to about 1 gram per liter. Lloyd's Reagent (Curve No. 8) also proved an intermediate adsorbent but

Figure 1

Figure 2

differed from the others in that the percentage adsorbed at higher concentrations fell much less rapidly.

In order to make a direct comparison of the adsorption efficiency of each charcoal in the different concentrations, the results were tabulated to show the per cent of strychnine sulphate adsorbed. These results are shown in table IX.

Table IX

Per Cent of Strychnine Sulphate Adsorbed										
Adsorbent	Concentration - Grams per Liter									
	1	2	3	4	5	6	7	8	10	12
Merck's Medicinal Charcoal	100	100	100	100	97.7	89.0	77.9	69.0	56.1	47.1
Nuchar	100	94.5	76.3	62.6	52.0	44.2	38.9	34.4	28.8	25.9
Carbex E	100	95.5	80.4	69.3	60.1	51.7	44.7	39.2	31.5	26.4
Carbex T	100	89.0	66.3	51.9	41.9	35.2	30.3	26.5	21.7	18.5
Pfanstiehl Decolorizing Carbon	94.0	75.6	63.3	54.5	48.0	42.8	38.4	34.6	28.6	24.0
Bone Black	36.3	36.0	35.4	34.7	33.5	31.7	29.8	27.9	24.7	21.6
Lloyd's Reagent	100	83.2	75.4	65.2	55.5	49.0	43.7	39.3	33.2	28.3
Willow Charcoal	11.0	10.3	10.0	7.8	6.3	5.3	4.5	3.9	3.2	2.6

II

Langmuir's Equation

It was stated in the preceding theoretical discussion that Langmuir's equation⁵ holds more nearly true over wider ranges of concentration than does Freundlich's equation. By plotting logarithms of the values of x/m and c of Freundlich's equation a straight line results if the original curve obtained by plotting x/m against c proves to be a parabola. As can be seen by inspection of the isotherms of Figures 1 and 2, none of these curves are by any means perfect parabolas, hence Freundlich's equation is not suitable for the expression of these isotherms.

On the other hand the plotting of C/Q against C of Langmuir's equation gives a near straight line in most of the cases tried. The plots of these values are shown in Figures 3 and 4. It was found that the sample of willow charcoal used yielded such extremely high values for C/Q that the plotting of its curve in these figures was impossible. That the isotherm for this adsorbing medium (Curve 7 - Figure 2) is not well represented by Langmuir's equation, can be seen by the large variation of points given in table XVI. All the other curves as shown by the figures conform quite well to the equation. (See Figures 3 and 4. Curve 1, Merck's Medicinal Charcoal; Curve 2, Nuchar; Curve 3, Carbox T; Curve 4, Carbox E; Curve 5, Pfanstiehl Decolorizing Carbon; Curve 6, Bone Black; Curve 8, Lloyd's Reagent.)

Figure 3

Figure 4

The constants a and b of Langmuir's equation were calculated for each carbon by plotting the curves of Figures 3 and 4 on quite a large scale in order to obtain the slope of the lines and their Y-intercepts more accurately. Values of the terms of the equation in each case are given in Tables X to XVII.

Table X

Merck's Medicinal Charcoal			
C/Q	C	a	b
10.24	5.00	1.154	0.610
11.24	6.00		
12.84	7.00		
14.50	8.00		
17.81	10.00		
26.03	15.00		

Table XI

Nuchar			
C/Q	C	a	b
10.58	2.00	0.144	0.336
11.63	2.50		
15.97	4.00		
25.72	7.00		
32.05	9.00		
38.62	12.00		

Table XII

Carbex T			
C/Q	C	a	b
11.24	2.00	1.969	0.231
13.13	2.50		
17.26	3.50		
19.25	4.00		
28.37	6.00		
41.72	9.00		
63.80	15.00		

Table XIII

Carbex E			
C/Q	C	a	b
10.00	0.916	0.329	0.483
10.00	1.375		
10.00	1.833		
12.00	2.749		
13.72	3.656		
15.61	4.582		
17.74	5.498		
23.43	7.331		
29.19	9.164		
34.79	10.997		
41.22	12.830		

Table XIV

Pfanstiehl Decolorizing Carbon			
C/Q	C	a	b
10.00	0.456	0.318	0.393
10.41	0.913		
12.65	1.826		
17.33	3.651		
19.65	4.564		
22.14	5.477		
24.39	6.390		
26.59	7.302		
29.09	8.215		
32.22	9.128		
38.53	10.954		
47.07	13.692		

Table XV

Bone Black			
c/q	C	a	b
25.60	0.924	0.062	0.734
27.84	1.847		
27.32	2.771		
29.30	3.694		
29.41	4.618		
36.19	5.541		
35.77	6.465		
34.35	7.388		
38.58	9.235		
42.67	11.082		
49.79	12.929		

Table XVI

Willow Charcoal			
c/q	C	a	b
3974.0	0.397	Not determined	Not determined
722.5	0.795		
130.9	1.589		
116.9	2.384		
101.0	3.179		
137.3	3.974		
152.7	4.768		
301.8	9.536		

Table XVII

Lloyd's Reagent			
c/q	C	a	b
12.01	2.00	0.455	0.400
13.26	3.00		
15.33	4.00		
18.01	5.00		
20.40	6.00		
25.41	8.00		
30.10	10.00		
35.39	12.00		

III

Solvent - water

Time - 48 hours

The same procedure employed in the preceding 24 hour determinations was followed in this case, with the exception that the period of contact was prolonged to 48 hours. Isotherms were determined for Merck's Medicinal Charcoal and for Carbox T.

Merck's Medicinal Charcoal gave an isotherm identical with the one obtained in the shorter period of contact, as shown by comparison of the values of tables I and XVII. This means that equilibrium must have been reached between the adsorbent and the strychnine sulphate solution within the 24 hours of contact and that this equilibrium was permanent for at least an additional 24 hours.

Carbox T on the other hand adsorbed more alkaloid when left in contact with the solution 48 hours than it did in the shorter time period. These results indicate that it takes more than 24 hours for the Carbox T and strychnine sulphate solution to reach adsorption equilibrium. The 48 hour curve in this instance probably represents the equilibrium but no determination involving a longer period of time was run to prove this presumption. Curve 1 of Figure 5 is the 48-hour isotherm obtained by plotting values of Table XIX, while Curve 2 is the result of values obtained in the shorter period of contact.

figure 5

Table XVIII

Merck's Charcoal		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
3.0000	0.0000	0.3000
4.0000	0.0000	0.4000
5.0000	0.0115	0.4885
6.0000	0.0656	0.5344
6.5000	0.1064	0.5436
8.0000	0.2412	0.5588
10.0000	0.4357	0.5643

Table XIX

Carbex T		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
1.0000	0.0000	0.1000
1.5000	0.0000	0.1500
2.0000	0.0174	0.1826
2.5000	0.0459	0.2041
3.0000	0.0876	0.2124
4.0000	0.1766	0.2234
5.0000	0.2752	0.2248
7.0000	0.4706	0.2294
9.0000	0.6696	0.2304
10.0000	0.7645	0.2355

IV

Solvent - alcohol

Time - 24 hours

The amount of adsorption³ is dependent upon the surface tension of the solvent, hence the use of alcohol in place of water should give a different isotherm when all other factors are the same.

The effects of such a change were determined in the cases of Merck's Medicinal Charcoal and Nuchar using commercial

alcohol sp. gr. 0.810 at 26°C. which corresponds to about 94 per cent alcohol by volume.

Figure 6 shows the results secured by the use of Merck's Medicinal Charcoal, Curve 1 with alcohol and Curve 2 with water. A marked drop in adsorption is evidenced in the alcohol solution, showing total adsorption to a concentration of only 1.5 grams of alkaloidal salt per liter. The alcohol curve also shows a maximum of adsorption at about 9 grams per liter concentration, whereas the water curve rises slowly but continually upward after the usual sharp bend.

Values for points on the alcohol curve appear in Table XX. Table XXI gives a direct comparison of the amounts adsorbed in each case.

Table XX

Merck's Charcoal - Alcohol		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
2.00	0.0096	0.1904
3.00	0.0272	0.2728
4.00	0.0552	0.3448
5.00	0.1096	0.3904
6.00	0.1908	0.4092
7.00	0.2808	0.4192
9.00	0.4696	0.4304
10.00	0.5664	0.4262
11.00	0.6596	0.4192

Table XXI

Per Cent of Merck's Medicinal Charcoal Adsorbed									
Solvent	Concentration - Grams per Liter								
	1	2	3	4	5	6	7	8	10
Water	100	100	100	100	97.7	89.0	77.9	69.0	56.1
Alcohol	100	95.2	90.9	86.2	78.1	68.2	59.9	53.3	42.6

Figure 6

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Nuchar with alcohol (Figure 7, Curve 1) shows the same type of diminution in adsorption as does Merck's Medicinal Charcoal. The highest point of total adsorption occurs at a concentration of about 1 gram per liter. This point is somewhat lower than that of Merck's Medicinal Charcoal and the maximum of the curve occurs at about 6 grams per liter concentration in contrast to 9 grams per liter concentration in the case of Merck's Charcoal. Table XXII shows the points yielding the Nuchar-alcohol curve and Table XXIII presents a direct comparison of quantities adsorbed at different concentrations in the two solvents.

Table XXII

Nuchar - Alcohol		
Concentration - Gms. per liter	Wt. of Residue	Amount adsorbed per Gm. of charcoal
1.0000	0.0000	0.1000
1.5000	0.0176	0.1324
2.0000	0.0440	0.1560
2.5000	0.0812	0.1688
3.0000	0.1156	0.1844
3.5000	0.1576	0.1924
4.0000	0.1988	0.2012
5.0000	0.2876	0.2124
6.0000	0.3860	0.2140
8.0000	0.5900	0.2100
9.0000	0.6932	0.2068

Figure 7

Table XXIII

Per Cent of Nuchar Adsorbed									
Solvent	Concentration - Gms. per Liter								
	1	2	3	4	5	6	7	8	10
Water	100	94.5	76.3	62.6	52.0	44.2	38.9	34.4	28.8
Alcohol	100	78.0	61.5	50.3	42.5	35.7	30.4	26.3	20.2

Conclusions

1. The media studied vary widely in their ability to adsorb strychnine sulphate.
2. Merck's Charcoal reaches an equilibrium of adsorption more rapidly than does Carbox T.
3. Langmuir's equation represents the adsorption curves obtained better than does Freundlich's equation.
4. The ability of Merck's Charcoal and Nuchar to adsorb strychnine sulphate is less when the alkaloid is dissolved in alcohol than when it is dissolved in water.

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