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STUDIES OF ADSORPTION FROM
NON-AQUEOUS BINARY AND
TERNARY SYSTEMS

Thesis for the Degree of M. S.
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Ruth Frances Hill

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STUDIES OF ADSORPTION
FROM
NON-AQUEOUS BINARY AND TERNARY
SYSTEMS.

by
Ruth Frances Hill

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I. INTRODUCTION

Within the past fifteen years, a great deal of work has been done on Adsorption from binary liquid systems. Various workers have attempted to show a correlation between the selective Adsorption of one constituent and some physical or chemical characteristic of the system. Although different workers have stressed the importance of different properties, certain facts were observed by all.

Thus, as Kane and Jathar¹ point out, the preferential adsorption of either constituent, depends to a very large degree upon the nature and activity of the adsorbent. In studying the system, toluene-acetic acid, they found that toluene is adsorbed at all concentrations, on sugar charcoal, whereas acetic acid is adsorbed in this manner on silica gel. For animal charcoal, however, they obtained S-curves, showing that the adsorption of either component depends upon its relative concentration.

Similar results were obtained by K.S. Rao and B.S. Rao² in studying adsorption on gels of silica, alumina and ferrous oxide. These workers found that alcohol is selectively adsorbed on both aluminum oxide and ferrous oxide, over the entire concentration range, from mixtures of alcohol and benzene, and alcohol and carbon tetrachloride.

Many workers have stressed the importance of Adhesion Tension as a determining factor. Bartell and Sloan³, in

studying the systems benzene-ethyl alcohol, alpha bromonaphthalene-ethyl alcohol and ethyl carbonate-ethyl alcohol, using a charcoal adsorbent, found that, in dilute solutions, the solutes with the greatest Adhesion Tension against carbon, are adsorbed most. They agree, however, on the importance of relative concentration, since, if the component of lower Adhesion Tension is present in very slight amount, it is preferentially adsorbed, anyway. In each case, they obtained S-curves over the entire range of concentration.

E. Heymann and E. Boye⁴ summarize these observations by classifying the adsorption curves of binary liquid systems into two types. In the first, we have positive adsorption of one component over the whole range, with a maximum at some point. This is characteristic of mixtures in which one component has a high Work of Adhesion and also of mixtures in which the Surface Tension of one constituent is markedly lowered by the addition of the other. In the second, we have an S-curve which passes through a point of equal adsorption of both and moreover, shows an apparent negative adsorption of one component over a definite concentration range. This is characteristic of systems, whose components have a small Work of Adhesion.

These workers also make the important generalization that, in mixtures of aliphatic and aromatic compounds,

the aromatic compound tends to be adsorbed best.

Work along these lines has been extended to the case of a solute in a mixture of solvents. The dominant factor, here, appears to be the polarity of the solvents. Thus Ermolenko and Levina⁵ in studying the adsorption of salicylic and butyric acid from the mixtures, $\text{CCl}_4\text{-CHCl}_3$, $\text{C}_6\text{H}_6\text{-C}_2\text{H}_5\text{OH}$, $\text{C}_2\text{H}_5\text{OH-H}_2\text{O}$ and $\text{CHCl}_3\text{-C}_2\text{H}_5\text{OH}$, make these observations:

1. From mixed solvents of similar polarities or similar chemical nature, Adsorption is absolutely independent of the relative concentrations of the solvents.

2. From mixed solvents of the polar-non polar types, the Adsorption of salicylic acid increases with an increase in the concentration of the non-polar component. However, for butyric acid, other factors must be considered.

In an earlier paper⁶, these same workers studied the adsorption on charcoal of anthranilic acid from binary solvents, and made these observations: in a mixture of two non-polar components or one polar and one non-polar or two strongly polar, Adsorption is inversely proportional to Solubility. In a mixture of similar chemical nature, changes in Adsorption nearly parallel changes in Solubility. Here, Solubility is introduced as an important factor to be considered.

In general then, the important factors influencing adsorption from a two-component liquid system, appear to be

the nature and activity of the adsorbent used, the relative concentration of the components, the Adhesion Tensions against the adsorbent, the chemical nature of the components. In the case of three-component systems, containing a solute in a binary mixture of solvents, three additional factors must be included--the nature of the solute, its solubility in both components and the polarities of the components. For any particular system, one or more of these factors may predominate, although, at the present state of investigation, just which ones, seems to be a matter of experimental observation rather than theoretical prediction.

II. NATURE OF THE PRESENT STUDY

This paper will be devoted chiefly to a study of the chromatographic and ordinary Adsorption from mixed solvent systems, containing cholesterol as the solute. The original purpose was to investigate the possibility of controlling the Adsorption and Desorption of cholesterol on a Twissett column, by varying the nature and concentrations of the mixed solvents. Throughout, the adsorbent used is activated bentonite. The cholesterol, in percolates and filtrates, was determined, colorimetrically, using the Liebermann-Burchard reaction. Special care had to be taken, often by means of preliminary runs, to use proper dilutions and concentrations to bring the developed colors within a readable range.

The last section is concerned with a possible theoretical approach to the observed phenomena and constitutes a starting point for further theoretical investigation.

III. ADSORPTION ISOTHERMS FOR CHOLESTEROL

Preliminary work on the adsorption of cholesterol had consisted of sending solutions of cholesterol dissolved in benzene, through Twestt columns, 2 cm. long. The developing agent, which also eluted the column, was a mixture of benzene and Skelly Solve, of varying concentration. Although the quantity of cholesterol, appearing in the percolates, increased with an increase in benzene concentration, anomalously high results led to the question of the possible negative adsorption of cholesterol or positive adsorption of benzene. The concentration used in this work was 1.1 mg. per ml.

With this question in mind, an adsorption isotherm for cholesterol in benzene, was determined over a range in concentration from 0.197 mg. per ml. to 19.73 mg. per ml. Each sample was mixed with 1.1500 grams of adsorbent. The flasks were swirled frequently and one hour allowed for equilibrium to be reached. The samples were then rapidly filtered through paper, previously moistened with benzene; The first 2-3 ml. were discarded and the concentration of cholesterol determined in the remainder.

The accompanying graph and Table I give the results. The nature of the S-curve obtained, indicates that we can

TABLE 1

CONCENTRATION BEFORE ADSORPTION	CONCENTRATION AFTER ADSORPTION	DIFFERENCE $\times 20 = X$	X/M IN MGN. / MGN.
.197 MGN./ML	.215	+ .360 MGN.	- .0003
.49	.54	+ 1.00	- .0008
.98	1.30	+ 6.40	- .0056
1.48	1.39	- 1.80	+ .0016
1.97	1.71	- 5.20	+ .0045
2.46	2.06	- 8.00	+ .0069
4.93	4.30	- 12.60	+ .0110
9.87	7.88	- 39.80	+ .0340
19.73	17.10	- 52.60	+ .0460

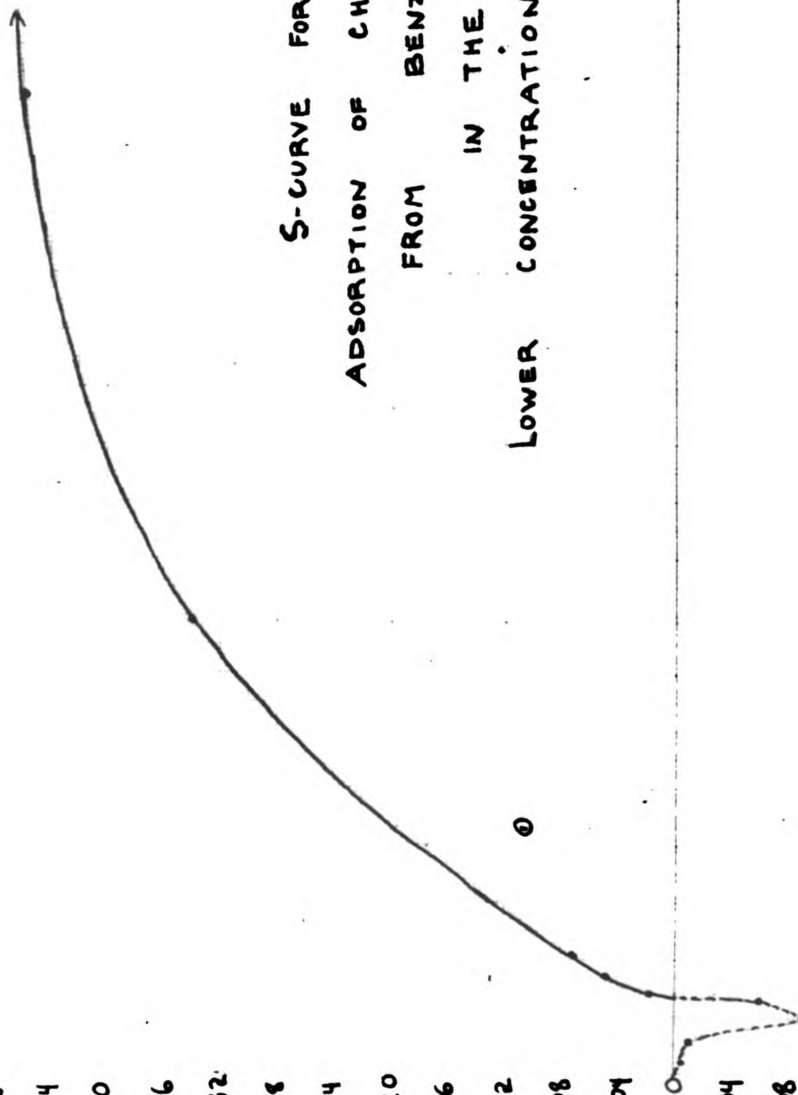
M = 1.1500 GR. OR
11500 MGN OF ACTIVATED
BENTONITE,

$\frac{x}{A}$ IN MG/MG

.056
.052
.048
.044
.040
.036
.032
.028
.024
.020
.016
.012
+ .008
+ .004

-.004
-.008
-.012

S-CURVE FOR THE
ADSORPTION OF CHOLESTEROL
FROM BENZENE
IN THE
LOWER CONCENTRATION RANGE.



1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24
C IN MG/ML

expect negative adsorption of cholesterol in benzene, from concentrations less than 1.3 mg. per ml.

In all the succeeding work, the major solvent for cholesterol was changed to benzine, a hexane fraction of petroleum. It is hoped that confusion will not arise due to the similarity in names.

Since benzine gives a turbid solution with the concentrated sulphuric acid, used in the Liebermann-Burchard test, all filtrates and percolates had to be evaporated to dryness and the residues picked up in benzene (C_6H_6), before the concentrations of cholesterol could be determined. Moreover, the solubility of cholesterol in benzine is far less than in benzene so that concentrations exceeding 10 mg. per ml. could not be employed. Solution required heating in a water bath at 35° - $40^{\circ}C$.

Four isotherms were determined. The first was for cholesterol in pure benzine. In the others, solvent mixtures were used, made up to contain 20 ml. of benzine and 3 ml. of benzene, absolute alcohol and ethyl ether, respectively. The relative concentrations were thus 87% benzine to 13% of the minor component.

Since the filtrates had to be evaporated to dryness and taken up in varying quantities of benzene for colorimetric determination, the equilibrium concentration C , could not be expressed in mg. per ml. and is given, therefore, as total weight of cholesterol unadsorbed. The

initial concentration before adsorption is also given as total weight. The calculation of x/m is unaffected.

The graph and Tables II, III, IV, and V are highly illuminating. Cholesterol is very highly adsorbed from pure benzene alone. From the solvent mixtures, this is markedly reduced. In order of decreasing adsorbability, we have ethyl ether, absolute alcohol and benzene. The curves for ether and alcohol have maxima, indicating that the adsorption of cholesterol increases with increasing concentration up to a certain point, beyond which any further increase in concentration is accompanied by a decrease in adsorption. For all three minor constituents, the quantity adsorbed is always less than from benzene alone.

IV. CHROMATOGRAPHIC ADSORPTION OF CHOLESTEROL

The results of the previous study were next extended to the case of adsorption on a Twett column. With benzene as the major constituent of the solvent mixture, the effects of various minor components were observed. The substances used were absolute alcohol, ethyl ether and benzene, as before, and in addition, isopropyl alcohol, glacial acetic acid and chloroform.

In all cases, except that of glacial acetic acid, the mixture consisted of 20 ml. of benzene to 3 ml. of the other component. In the case of glacial acetic acid, only 0.5 ml. were used, since previous work had shown that its desorbing

TABLE II BENZINE SOLVENT

WEIGHT OF CHOLESTEROL BEFORE ADS.	WEIGHT AFTER ADS. = C-	DIFFERENCE = X	x/m
100.2 mg	5.00	-95.2	+ .083
80.2	1.76	-78.4	+ .068
60.0	6.45	-53.6	+ .046
40.0	.28	-39.7	+ .034
25.0	.80	-24.2	+ .021
15.0	.35	-14.7	+ .013
10.0	0	-10.0	+ .008
5.0	0	-5.0	+ .004

TABLE III BENZINE - ABS. ALCOHOL

WEIGHT BEFORE ADS.	WEIGHT AFTER ADS.	DIFFERENCE = X	x/m
100.00	76.20	-23.8	+ .021
80	53.20	-26.8	+ .023
60	43.44	-16.6	+ .019
40	26.64	-13.4	+ .012
25	17.22	-7.8	+ .007
15	11.20	-3.8	+ .003
10	7.00	-3.0	+ .002
5	4.10	-0.9	+ .0007

TABLE IV BENZINE - ETHYL ETHER

WEIGHT BEFORE ADS.	WEIGHT AFTER ADS.	DIFFERENCE = X	X/M
100 MG	45.20	- 4.8	+ .004
80	63.52	- 16.5	+ .014
60	44.04	- 16.0	+ .013
40	29.28	- 10.7	+ .009
25	18.06	- 6.9	+ .006
15	10.80	- 4.2	+ .004
10	6.96	- 3.0	+ .003
5	3.77	- 1.2	+ .001

TABLE V BENZINE - BENZENE

WEIGHT BEFORE	WEIGHT AFTER	DIFFERENCE = X	X/M
100.0	29.0	- 71.0	+ .061
80	19.0	- 61.0	+ .053
60	12.7	- 47.3	+ .041
40	7.0	- 33.0	+ .028
25	3.3	- 21.7	+ .018
15	2.6	- 12.4	+ .010
10	1.5	- 8.5	+ .007
5	.7	- 4.3	+ .003

$\frac{x}{H}$ IN MG/GM

.125

.120

.115

.110

.105

.100

.095

.090

.085

.080

.075

.070

.065

.060

.055

.050

.045

.040

.035

.030

.025

.020

.015

.010

.005

.000

ADSORPTION ISOTHERMS FOR CHOLESTEROL IN BENZINE SOLUTION

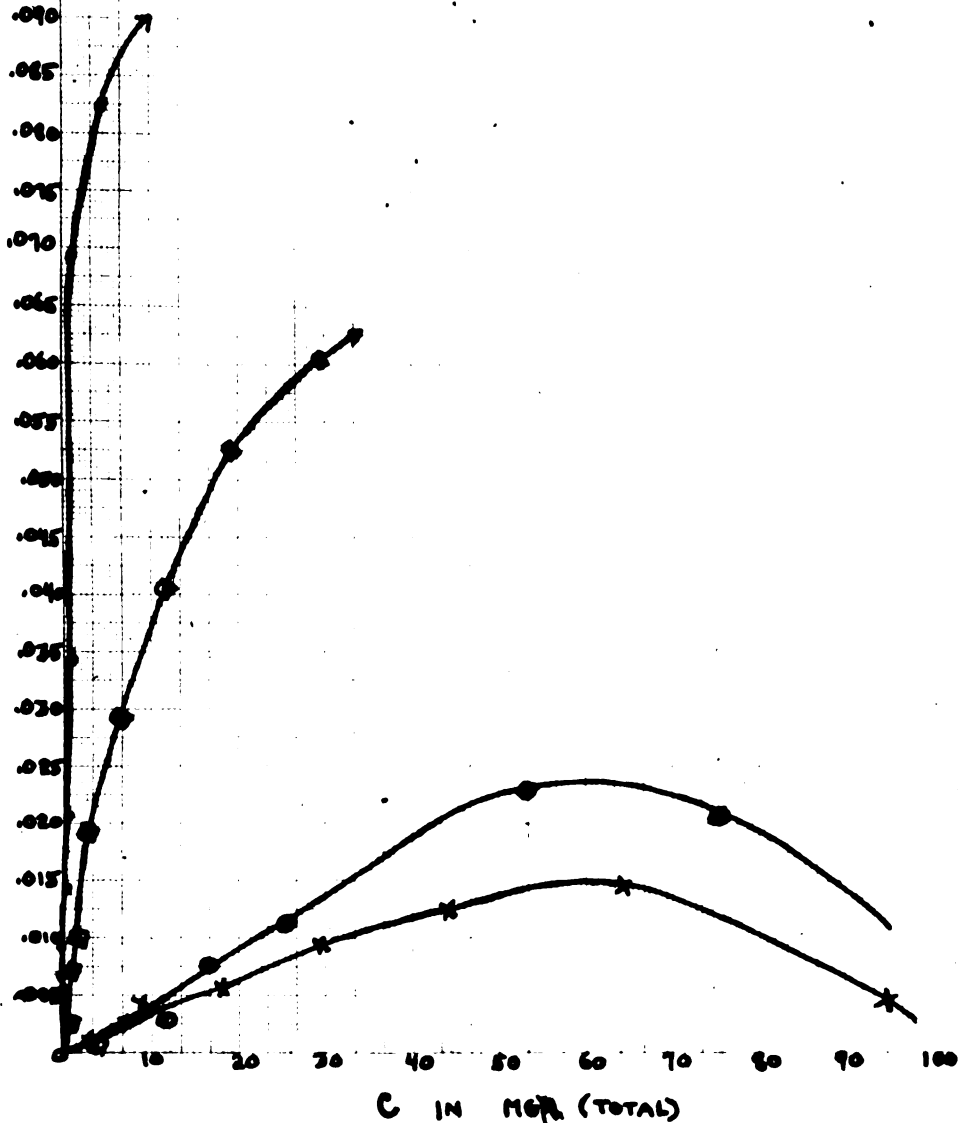
● BENZINE

■ BENZINE + 3 ML. BENZENE (C_6H_6)

○ BENZINE + 3 ML. ABS. ALCOHOL

x BENZINE + 3 ML. ETHYL ETHER

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ability is very high. The 20 ml. of benzene contained 2 mg. per ml. of dissolved cholesterol. The columns were first moistened with the solvent mixture and then the samples, containing also 4 drops of a solution of Sudan III, were allowed to percolate through, under slight suction; the percolates were collected and the columns then eluted with 20 ml. of the same solvent mixture. Both percolates were evaporated to dryness and taken up in sufficient benzene, with dilution, if necessary, to insure a readable color reaction.

The purpose of the Sudan III was to serve as a possible marker for the chromatograph but repeated trial failed to produce good banding. Moreover, the use of the dye must be restricted to an extremely dilute solution since, up to very high dilutions, it will give a positive Liebermann-Burchard reaction.

The results are summarized in Table VI.

*The high results in the case of absolute alcohol are due to the fact that the dye came through the column in the first percolate.

The results for the first three mixtures check very well with the adsorption isotherms for these same mixtures. Reference to the graph for this reveals that benzene is not as efficient in desorbing cholesterol, when present to the same extent as ether and alcohol.

The desorbing power of isopropyl alcohol does not

TABLE VI

SOLVENT MIXTURE	WEIGHT OF CHOLESTEROL IN PERCOLATE 1	WEIGHT IN PERCOLATE 2	TOTAL RECOVERY OUT OF 40 MGR
BENZINE + BENZENE	0	0.6	0.6
BENZINE + ETHYL ETHER	8.60	27.2	35.8
BENZINE + ABS. ALCOHOL	37.80	5.6	43.4 *
BENZINE + ISOPROPYL ALC.	33.90	5.2	39.1
BENZINE + GLACIAL ACETIC	18.00	17.6	35.6
BENZINE + CHLOROFORM	0	0	0

differ much from that of ethyl alcohol. Also in both cases, most of the cholesterol is carried down in the first percolate. The reverse is true for ether and benzene, while the distribution for glacial acetic acid is approximately 50-50.

Chloroform presents the most interesting case of all for it is the one substance that does not desorb the cholesterol. Apparently, the addition of chloroform to the solvent mixture retards the removal of cholesterol from the adsorbent. The reason for this is definitely not a question of solubility since cholesterol is very soluble in chloroform as it is in glacial acetic, ether and benzene.

In general, the use of benzine in the solvent mixture causes bad breaks in the column. This effect is very much worse when absolute alcohol is also present so that the column must be packed very tightly before use. Even so, the percolation is very slow. 20 ml. of the mixture containing ether came through in 15 minutes whereas, 20 ml. of the mixture containing absolute alcohol required 65 minutes or about four times as long.

In the running of the adsorption isotherm, it was noticed that swirling of the flasks containing benzine and absolute alcohol had caused the adsorbent to appear finer and whiter and to require more time to settle. The significance of these observations will be discussed later.

Although most of this study was restricted to non-

aqueous solutions, the effect of water in the solvent mixture was included. Its presence was compared with absolute alcohol. Two samples were run, using the same method. The first contained 20 ml. of the benzine solution of cholesterol, 1 ml. of alcohol and 8 drops of water while the second had the same components but no water. In both cases, most of the cholesterol appeared in the first percolate, 39.6 mg. being the total recovery, using water and 34.6 mg. without water. An attempt to use just benzine and water failed to desorb any cholesterol, due to the fact that the water did not mix with the benzine. Benzine alone does not desorb cholesterol when 40 ml. are run through the column.

However, the observation made in the case of the mixture containing benzine, alcohol and water was that the presence of the water greatly slowed up the percolation. The affinity of activated bentonite for water is apparently much greater than for any other substance so that as soon as the mixture is added to the column, a layer of water can be seen to form on the surface of the adsorbent. This clogs up the pores so that the other constituents have difficulty getting through. Once the film of water comes through, the rate of percolation markedly increases.

V. THEORETICAL ASPECT

A. Nature of the Problem.

The entire subject of adsorption from two and three-component systems is fraught with many questions, some of

which have been only partially answered and some not at all. Just why is a solute adsorbed from one solvent and not from another? Is the adsorbent itself, of prime importance and if so, why are different effects observable on charcoal, silica gel, aluminum oxide, ferrous oxide, bentonite etc.? Can we infer some chemical interaction between the adsorbent and one or more components of the solution? How far does solubility go in explaining observable effects? Why does Adhesion Tension cease to be a major factor in the case where the component of low tension is present in high dilution? How much credence are we to give explanations for a given system, based on Polarity?

It is with an attempt to simplify some of this, that this last section is concerned.

B. Method of Attack

The system cholesterol-benzine-absolute alcohol was selected as a starting point. Let us suppose, for the moment, that the explanation for the observed desorption of cholesterol from the solvent mixture of 87% benzine and 13% alcohol, can be based on Adhesion Tensions. If we assume at this point, that the tension of benzine against bentonite is very low, then we might expect cholesterol to be highly adsorbed from pure benzine solution, providing that some sort of attraction exists between the solid adsorbent and the solute. Actually, cholesterol is highly

adsorbed from pure benzine. Now what happens when alcohol is added to the solution? A small amount removes cholesterol from the bentonite. This can not be due to the greater solubility of cholesterol in alcohol, since chloroform does not give the same effect although cholesterol is very soluble in it.

We might assume that the alcohol increased the Adhesion Tension of the benzine, thereby causing benzine molecules to displace cholesterol molecules. But what of the alcohol molecules? Are they adsorbed too? Are cholesterol molecules displaced by both alcohol and benzine or by just one of these? This question resolves itself into the simpler problem of determining whether or not there is any change in the binary mixture, benzine-alcohol, after contact with the adsorbent. The method used was the determination of density before and after adsorption and comparison with the densities of the pure components. The entire concentration range was covered.

Density measurements were made by the Pycnometer method. All mixtures were determined at 25°C. Great care had to be taken to prevent evaporation since benzine is extremely volatile. The flasks containing adsorbent and solvent mixture were carefully stoppered and wrapped in tinfoil. Rapidity of manipulation and weighing and repetition of determinations served to minimize the error.

C. Results

A definite change in density after adsorption was detected. Table VII and the accompanying graph give the magnitude and direction of the differences. The density of benzene at 25°C is 0.6299 while that of absolute alcohol is 0.7866.

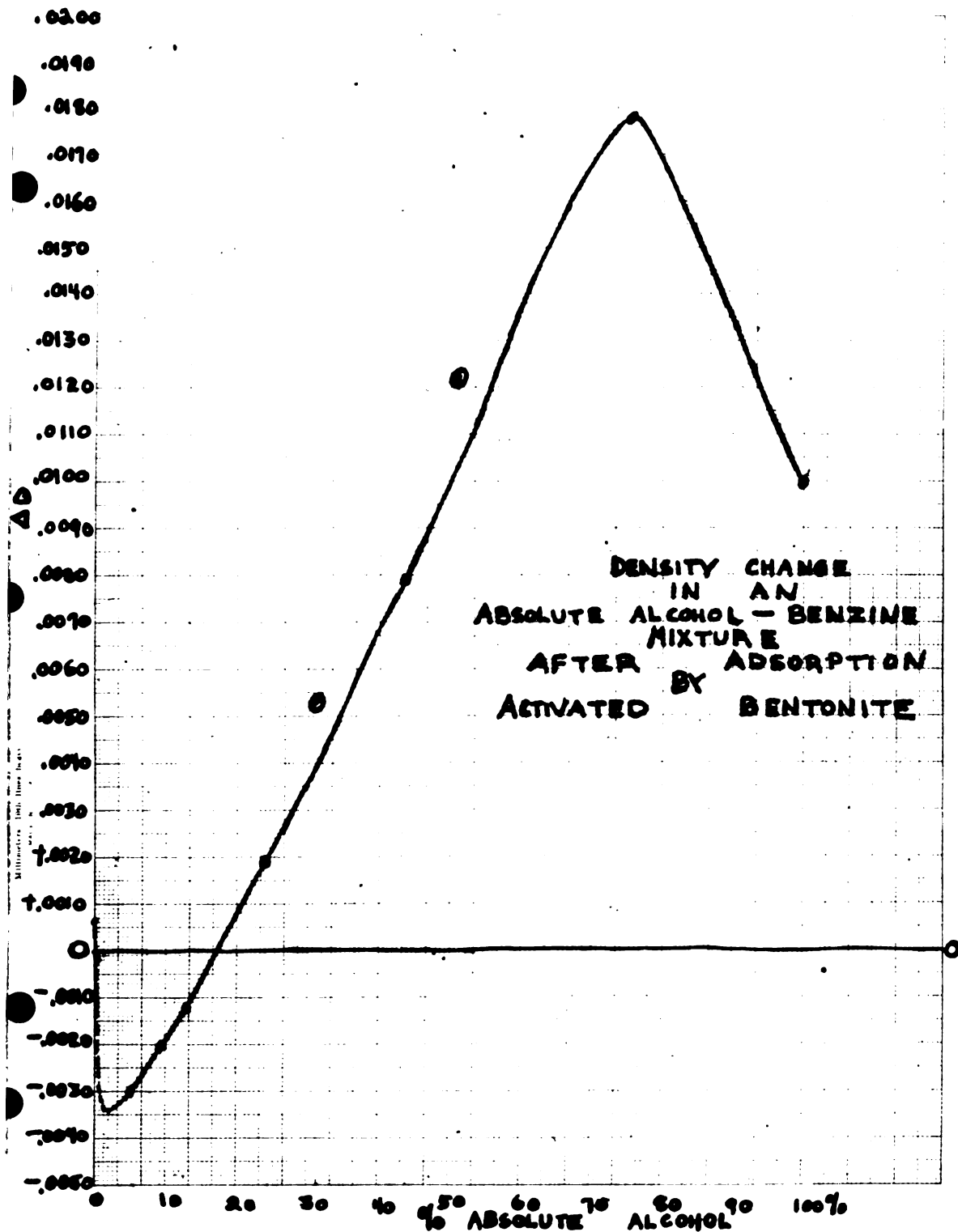
For mixtures in which the alcohol concentration is less than approximately 18%, the density after adsorption is less than before, the shift being in the direction of the pure benzene. Evidently, in such mixtures, alcohol is adsorbed to a much greater extent than benzene. At about 18% alcohol concentration, the curve crosses the x-axis, indicating that either both are adsorbed to the same extent or that neither is adsorbed. The former is the more likely possibility. Above 18% alcohol concentration, the density after adsorption increases in the direction of pure alcohol, so that we may conclude that benzene molecules are being removed to a much greater extent.

The nature of the results shows that in the three component system, cholesterol-benzene-absolute alcohol, at the concentration previously employed, (13% alcohol to 87% benzene), alcohol molecules are adsorbed and cholesterol desorbed. Therefore, since benzene is not adsorbed either to any great extent, alcohol does not act by increasing the Adhesion Tension of benzene against Bentonite. Its behaviour is more direct. Whatever the nature of the solid-solid force of attraction between

TABLE VII

% ALCOHOL IN ORIGINAL MIXTURE	% BENZINE IN ORIGINAL MIXTURE	DENSITY BEFORE ADSORPTION	DENSITY AFTER ADSORPTION	CHANGE IN DENSITY = ΔD
0 %	100	.6299	.6306	+ .0007
4.8	95.2	.6364	.6334	- .0030
9.1	90.9	.6432	.6412	- .0020
13.0	87.0	.6507	.6495	- .0012
23.1	76.9	.6655	.6674	+ .0019
31.0	69.0	.6787	.6840	+ .0053
42.8	57.2	.6988	.7067	+ .0079
51.2	48.8	.7124	.7246	+ .0122
75.0	25.0	.7526	.7704	+ .0178
100.0	0	.7866	.7966	+ .0100

ΔD = CHANGE IN DENSITY



cholesterol and bentonite may be, this attraction is diminished and the important force becomes the Adhesion Tension of alcohol against bentonite. To complete this line of reasoning, one would have to know whether or not cholesterol is desorbed when the alcohol concentration exceeds 18%. This opens room for further study. At this point, all we know is that in such concentrations, benzine is desorbed to a much greater extent than alcohol. If additional research shows that cholesterol is desorbed throughout the entire range of concentrations in the solvent mixture, we would have to conclude that its removal depends solely upon its replacement by either or both of the other components. However, if it is found that in the upper concentration range of alcohol, cholesterol is again adsorbed, we would have to look at the phenomenon differently. In this case we would have to say that the adsorption of the cholesterol is dependent upon the simultaneous adsorption of benzine molecules, since in the upper range, benzine is definitely adsorbed. (In the lower range, where cholesterol is desorbed, benzine is not adsorbed.) The writer believes that this is actually the case and that to explain it, one would have to postulate the formation of a cholesterol-benzine complex. However, the level of this hypothesis is dependent on future research.

An interesting point to be observed about the graph is that the density change does not drop to zero at any

alcohol, alcohol, or water in it. Paradoxical as it seems, the explanation is simple. Previous work in this paper had mentioned the observation that alcohol causes bentonite to become finer and whiter and to take much longer to settle out. This agrees with the work of Ambrose and Loomis⁷ in which they state that "emulsoid colloids present in bentonite respond to changes in pH of the dispersion medium by changes in rate of settling, viscosity and swelling". As the alcohol concentration is increased, the adsorbent particles become finer. In using pure alcohol, the particles came through the pores of the filter so that the density determined, after mixture with the bentonite, is not the density of the alcohol itself but rather the alcohol plus these suspended particles. At the other concentrations used, the particles were coarse enough to be held back by the filter so that no error from this source was introduced.

The graph can be considered as starting at the origin on the left, since a density change of ± 0.0007 for pure benzene is within the limit of experimental error and can be taken as a change of zero.

VI. SUMMARY

1. An S-curve was determined for the adsorption of cholesterol on activated bentonite, from benzene solution, showing negative adsorption from concentrations less than

1.3 mg. per ml.

2. Adsorption isotherms were determined for cholesterol in benzine, benzine-benzene, benzine-absolute alcohol and benzine-ethyl ether. Cholesterol is found to be highly adsorbed from benzine but the addition of the other components, in the above order, caused increasing amounts to be desorbed.

3. The chromatographic adsorption of cholesterol was studied, using different solvent mixtures. Benzene, absolute alcohol, ethyl ether, isopropyl alcohol and glacial acetic acid are found to remove cholesterol from Tassett columns. Chloroform does not.

4. The presence of water in the benzine solution of cholesterol slows up the process of percolation due to the great attraction existing between the water and the bentonite adsorbent.

5. Density measurements were made on benzine-absolute alcohol mixtures, over the whole concentration range, before and after adsorption. Alcohol is found to be adsorbed to a greater extent from concentrations less than 13% and benzine to be adsorbed to a greater extent above 13%. Density measurements are suggested as one means of laying the foundation for theoretical explanations of adsorption from three-component systems, containing a solute dissolved in a binary mixture of solvents.

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