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The development of Langmuir's adsorption isotherms for glucose and fructose and the separation via continuous parametric pumping

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DEVELOPMENT OF LANGMUIR'S ADSORPTION ISOTHERMS

FOR GLUCOSE AND FRUCTOSE

AND

THE SEPARATION VIA CONTINUOUS

PARAMETRIC PUMPING

BY

JAFIR ABBAS JAFERI

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ABSTRACT

The adsorption equilibria and adsorption rates of
fructose and of glucose on aqueous suspensions of
fullers earth were determined at various temperatures.
The Langmuir adsorption equations were fitted to the
equilibrium data and the Langmuir constants were deter-
mined so that isotherms could be developed for any pair
of parametric pumping operating temperatures.

This glucose+fructose+water system was then subj-
ected to continuous thermal parametric pumping for the
separation of glucose and fructose. The operating
conditions required for the separation were determined
from the experimental adsorption rates and equilibrium
isotherms. Samples taken from the continuous top and
bottom streams were analyzed by polarimetry for fructo-
se content and by evaporation for total sugar concent-
ration. The results compared favourably with those
predicted by the equations of Chen and Hill.[1] Although
the separation was successful, the cycle times required
were very long due to the time required to reach
adsorption equilibrium.

PREFACE

This research was done for a Master's thesis, and is presented in partial fulfillment of the requirements for Master of Science in Chemical Egineering degree at Newark College of Engineering, Newark, New Jersey.

The literature review depicts the lack of Langmuir's adsorption isotherms for glucose and fructose with fullers earth as solid adsorbent. Although some experimental work had been done by Kniaseff, Valsily[9] and B.P.Gyani[4] about the adsorption of glucose and fructose on charcoal and fullers earth, yet the effect of temperature on the adsorption remained uninvetigated.

The purpose of this research work is, to develop Langmuir's adsorption isotherms for glucose and fructose using fullers earth as adsorbent. Hence the adsorption data at any optimum temperature may be calculated through Langmuir's equations.

The system, glucose + fructose + water, was subjected to parametric pumping operation to separate glucose from fructose. Using the information from adsorption isotherms, operating conditions were selected such as to operate thermal parametric pumps in Region (1) for glucose and water system and in Region (2) for fructose and water system. This would accomplish the proposed separation.

Theoretical aspects of Langmuir's adsorption isotherms and thermal parametric pumping separation technique have been

discussed briefly in the text of the thesis. The experimental procedures for adsorption isotherms and parametric pumping have also been written in the subsequent pages. Bibliographic references have been brought up-to-date and in alphabetical order. All the tables and graphs, showing experimental data and calculations are placed at the end, before the appendix.

I am pleased to acknowledge the invaluable suggestions, continued encouragement and kind guidance of Dr.H.T.Chen, in this research work. I would not hesitate to submit that his dedication to the research work was always a source of constant inspiration to me and imparted confidence, patience and perseverance in me. I am thankful to Mr.J.D.Stokes, for his help in solving practical problems, faced during the research work. I appreciate the kind co-operation of Dr.J.J.Salamone,in allowing prompt use of Chemical Engineering Research Laboratories during vacations. My special thanks go to Dr.D.Hanesian and Dr.A.J.Perna, for assistance in the correction of the proofs and in the preparation of the manuscript.

At the end I must thank my brother Dr.G.A.Jaferi, whose financial support enabled me to complete graduate studies as a full-time student at NCE Newark, New Jersey.

Newark College of Engineering   Jaferi, J.A.

April, 1972.

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INTRODUCTION

The major part of this thesis consists of Langmuir's adsorption isotherms developement for glucose and fructose using fullers earth as adsorbent. The adsorption equilibria and adsorption rates are determined at several temperatures. Then the adsorption data is fitted to the Langmuir's Equations and the various constants are estimated for the adsorption isotherms of glucose and fructose separately.

Then glucose + fructose + water system is subjected to continuous parametric pumping technique for the separation of fructose. The necessary operating conditions for the separation are predicted from the experimental adsorption rates and the equilibrium isotherms. Comparison is made between the experimental data and the theoretical prediction by an equilibrium theory presented by Chen and Hill[1].

Theory and the experimental procedures for adsorption isotherms as well as for parametric pumping have been briefly in the text of this thesis. All the experimental results, calculations ,graphs and figures are shown at the end. The detailed procedures for the experiments, analysis and calculations are given in the Appendix.

SCOPE

According to the equilibrium theory as extended by Chen and Hill[1] to the separation of components by continuous thermal parametric pumping, it should be possible to separate fructose from glucose in sugar solution. To accomplish this purpose, adsorption isotherms were experimentally determined at several temperatures for the glucose-water and the fructose-water binary systems and the data were fitted to the Langmuir's equations. The equations were then used to extend the data to any selected pair of parametric pumping operating temperatures.

The thermal parametric pump was operated with continuous feed and product take off using the conditions predicted from the isotherms and also the experimental observations of the time required to reach equilibrium. The experimental results agree reasonably with the results predicted from the equations of Chen and Hill[1].

CONCLUSION

Langmuir's adsorption isotherms have been developed for glucose and fructose using fullers earth as an adsorbent. The effect of temperature on the rates of adsorption of glucose and fructose is a new addition to the previous works in this field of research. These equations would be useful in obtaining adsorption data for glucose and fructose at any suitable temperature. It is important to mention that the validity of the equations is limited to the use of powdered form of fullers earth. As fullers earth is a natural product so there are various kinds of it whose physical properties vary from one another partly because of their different source of origin and mainly because of their methods of treatment. The powdered form of fullers earth manufactured by S.B.Penick Co.N.Y., has shown one hour as the minimum time required to reach equilibrium and the amount of glucose adsorbed is about three times greater than that of fructose. While the course grade 30/60 mesh RVM fullers earth manufactured by Engle-hard Minerals Edison, N.J., has shown two hours as the minimum time required to reach equilibrium and the relative adsorption strength of glucose is about twice as great as that of fructose under the same conditions.

A new process, for the manufacture of fructose is developed, through parametric pumping technique. This is the first attempt in separating fructose from sugar solution

via continuous parametric pumping. The difference in adsorption for glucose and fructose at $20^\circ C$ and $40^\circ C$ indicated probable separation. Thermal parametric pumps were operated under a set of conditions,[*] which worked well in getting separation. The half cycle time 3 hrs., was long, based on adsorption data. It approximates the instantaneous equilibrium inside the column and complete mixing in the reservior liquid.

An automatic polarameter was used to analyse fructose contents in the top and bottom samples. The rotation of glucose is almost constant at all temperatures but that of fructose varies with temperature. It is observed that the increase in fructose rotation for 1.0gm.fructose/100 ml solution is 0.2667°. This is the priciple for obtaining fructose concentration in sugar solution. The total amount of sugar was determined gravimetrically. The concentration of glucose was obtained by difference. The results obtained were plotted in graph (12), which indicated that the glucose-water system was operated in Region (1) and fructose - water system in Region (2) simultaneously. This is in accordance with the theoretical prediction by Chen and Hill.[1]

(* suggested by Dr. H.T.Chen.)

PREVIOUS WORKS

Nothing was found in the literature regarding the application of the Langmuir Equations to the adsorption isotherms of glucose and fructose.

Vasily Kniaseff[9] presented experimental data showing the relative adsorption of different sugars on charcoal and fullers earth. He also showed the effect of pH and contact time on the adsorption but did not include the effect of temperature, the main variable in the Langmuir's equations. B.P.Gyani[4] presented data on the adsorption of various sugars on charcoal at $30^{\circ}C$, applied the data to the Freundlich equation, but again the effect of temperature was not determined. Therefore, it was necessary to determine experimentally adsorption isotherms at several temperatures and fit the data to the Langmuir's equations. The isotherms for any pair of operating temperatures could then be calculated and the results used to predict the separation of glucose and fructose by the parametric pumping technique.

Other methods of separating glucose from fructose have been employed. Dubrunfaut,[3] who was the first to separate fructose by chemical means, precipitated the sugar as Calcium fructosate and regenerated fructose with carbondioxide or with sulfuric or oxalic acid. Harding[5] surveyed some of the other chemical methods which have been proposed, and since then other chemical

methods have been published.[16],[17]

But physical methods of separation should be more economically attractive.... Harding[6] was pioneer in this field . He separated fructose from invert sugar by fractional crystallization from acetic acid and obtained 50% of the fructose and 75% of the glucose in a crude crystalline form. However, Harding regarded this method as suitable only for the laboratory.

Mowery[7] was among the first to consider adsorptive techniques for the industrial separation of glucose and fructose, and he used fullers earth as the adsorbent. More recently ion exchange techniques have been used[8] and appear to be industrially attractive.[12],[13] Also the separation of glucose and fructose by liquid liquid extraction has been accomplished by Hatt and Triffett.[7]

The work presented in this paper represents a separation by parametric pumping and may prove to be an interesting alternative to the physical and chemical methods previously devised.

THEORY

(a)- Langmuir Adsorption Isotherms

Langmuir[11] proposed a theory which assumes that
forces acting in adsorption are similar in kind to
those involved in chemical combination. From the view
point of the electronic conception of the atom, the
forces acting in ordinary chemical combination would
be due to very strong deviations of orbits of outer
electrons in the atomic structure, while the forces
acting in adsorption would be due to comparatively
small deviations. He assumed that the adsorbed layer
would be only one molecule thick, thus the forces bet-
ween the atoms of the surface of the adsorbent and the
atoms of the adsorbed substances decrease rapidly with
distance between atoms. Langmuir proposed a relation
between the concentration in the solution and the ads-
orbed quantity based on the assumption that equilibrium
is established between the rate of adsorption and the
rate of desorption i.e. the rate at which molecules st-
rike the surface and remain for an appreciable length
of time, will be exactly balanced by the rate at which
molecules detach themselves from the surface. The rate
of adsorption will be equal to the number of collisions
n_c , of gas molecules with the surface per second, mult-
iplied by a factor, F, representing the fraction of
colliding molecules that adhere. At a fixed temperature,

the number of collisions would be proportional to the pressure, p, of the gas and the fraction F, would be constant. Here the rate of adsorption per unit of bare surface would be, n_cF. This is equal to, kp where k is constant involving the fraction F and the proportionality between n_c and p.

Since the adsorption is limited to a monomolecular layer, the surface may be divided into two parts, the fraction θ covered by the monomolecular layer of adsorbed molecules and the fraction $(1-\theta)$ which is bare. Since only those molecules striking the uncovered part of the surface can be adsorbed, the rate of adsorption per unit of total surface would be proportional to $(1-\theta)$, that is,

$$r_a = kp(1-\theta) \qquad (1)$$

The rate of adsorption would be proportional to the fraction O of covered surface and is given by expression.

$$r_d = k\theta. \qquad (2)$$

Equating the two rates of equilibrium and solving for the fraction of surface that is covered ,

$$\theta = \frac{kp}{k + kp} = \frac{k_a p}{1 + k_a p} \qquad (3)$$

where, $k_a = k/k =$ adsorption equilibrium constant. For solid-liquid system, using concentration adsorbed in moles, the above equation simplifies to

$$X = \frac{NKY}{1 + KY} \qquad (4)$$

Where N is constant independent of temperature but
K is a function of temperature and is given as

$$K = K^o . T^{-0.5} . e^{-(\Delta H / RT)} \qquad (5)$$

The purpose of this research is to separate fructose , from glucose – fructose solution through thermal parametric pumping technique. To accomplish this separation a suitable adsorbent is required which should adsorb glucose more strongly than fructose. It is indicated in Kaniseff[9] and B.P.Gyani's[4] experimental work that charcoal is a good adsorbent for sugars but it adsorbs glucose as well as fructose to the same extent. On the other hand Fullers earth adsorbs glucose more than fructose . Therefore, fullers earth was selected as adsorbent for this purpose.

The solvent appears to have a profound effect upon the relative adsorptive strength on fullers earth. Wolfrom[20] reports fructose is more strongly adsorbed than glucose from 95 % Ethanol (H_2O) solution where as Kaniseff reports glucose is more strongly adsorbed than fructose from water solution. The results of present work using water as the solvent, support the observations of the latter investigator.

THEORY

(b) Thermal Perametric pumping

Thermal parametric pumping is a separation tech-
nique which comprises alternating axial displacement of a
fluid mixture in a column of adsorptive particles upon
which a cycling temperature is imposed synchronously.

Parametric pumping equilibrium theory presented
by Chen and Hill[1] consists of the characteristics of
batch, semi-continuous and continuous equilibrium para-
metric pumps. Then latter on Chen, et al,[2] have support-
ed this theory by experimental work. The experimental
investigations by Wilhelm and Sweed[17] have shown great
separative capability (10^5: 1) of batch parametric
pumping technique, while Chen, et al have obtained
infinite separation by continous and semi-continuous para-
metric pumps, with feed to the top of column.

In principle the technique is applicable to any
fluid mixture that adsorbs on a second phase, the extent
of separation however, depending mainly on the system
and on the operating conditions of thermal parametric
pumps. In fact, the ease of separation depends upon the
adsorption behavior of components on solid phase in the
column. Only the least adsorbable component can be
separated from the mixture, as a bottom product by con-
tinuous parametric pumping technique. At the same time, the
top product will be a mixture of fluids, richer than the feed.

Parametric pumping involves a synchronism between periodically reversing displacements between fluid and adsorbent phases, and a cycling of the adsorptive equilibrium.

In the present research work, it is of interest to separate fructose from sugar solution through parametric pumping. The adsorbent selected for this purpose is fullers earth, which adsorbs glucose more strongly than fructose, the essential feature for separating fructose as bottom product. It is simple to show adsorption and desorption effects in the column during cold and hot cycles respectively. The column packings are filled with feed at ambient temperature before starting parametric pumping experiment. The first half cycle is hot up flow because hot water is circulated through the jacket of packed column and the flow of fluid is from bottom reservior to the top reservior, during this hot cycle, there will be practically no adsorption on solid phase so the average concentrations of top and bottom product samples will be the same as that of feed. The feed (glucose+fructose+water) is always fed from the top of the column continuously. Then during second half of the cycle, cold water is switched to the jacket and simultaneously reservior pump reverses the direction of flow through the column. At cold temperature adsorbent will restrict more molecules of glucose from going down then those of fructose due to their difference in

adsorption. Therefore, during down flow, bottom product will deplete glucose molecules more as compared with fructose molecules and as a result bottom product sample will show an increase in fructose concentration. In the second half cycle, the hot water is again circulated and the direction of flow is also reversed simultaneously. The absorbent will desorb molecules of glucose which have been adsorbed during last cold cycle. Therefore, the top product will slightlyrich in glucose concentration. Glucose molecules are adsorbed more strongly during cold cycle and desorbed during hot cycle. By repeating hot and cold cycles periodically, the amount of glucose will go on depleting in the bottom product and pure fructose will be obtained eventually. But in the top product there will be a mixture of glucose and fructose richer than the feed in any case.

According to parametric pumping theory by Chen and Hill[1] it is possible to obtain separation by operating the pump continuously in the region (1) for glucose-water system and in region (2) for fructose water system simultaneously.

The operating conditions of the parametric pumping experiment have been selected such as to accomplish the above idea. The result shown in graph (12) is in accordance with the theoretical prediction by Chen and Hill.[1]

EXPERIMENTAL

(a) Langmuir Adsorption Isotherms

Aqueous solutions of 2.5,5,10 and 15 percent concentration, by weight of glucose were prepared and 50 ml of each solution were added in 125 ml conical flask. 1.00 gm of fullers earth was added to each of these flasks and the flasks were immersed in a constant temperature water bath with mechanical shacker for two hrs. An other flask containing 50 ml of 10 % glucose solution without fullers earth was also immersed in the bath to get a blank reading for purpose of comparision. After 2 hrs of constant shacking at 25°C, the slurries were filtered and the filtrate was analysed gravimetrically. The amount of glucose adsorbed per gm. of solid fullers earth was calcuted for each of 2.5, 5, 10, and 15 % concentration solutions originally used for test.

This procedure was repeated for temperatures of 10°C, 20°C, 30°C and 40°C. The adsorption rates determined at all these temperatures are shown in Table (1).

Similarly, adsorption rates for fructose were obtained experimentally at 10°C, 25°C and 40°C. The results are shown in Table (2).

From the equilibrium data thus obtained, the values of X, Y and Y/X for glucose and fructose were calculated at all temperatures (Tables 3&4) and applied to the Langmuir's equations.

EXPERIMENTAL

(b) Parametric Pumping Experiment

Therman parametric pumping apparatus is shown in Fig.(1) schematically. The top of the column was removed and dry granular fullers earth 30/60 mesh RVM was packed in the column. The top connection of the column was reconnected. The top and bottom product sample lines were closed and glucose - fructose feed solution was added in the packed column and in all the lines, by means of the top reservior syringe. The feed solution contained 10 gms. glucose, 10 gms. fructose and 80 gms. of water. The two feed pump syringes were filled up to 24 ml with feed solution. After eliminating all of the air from the system, the bottom reservior still held 43 ml of solution (feed) and the top reservior held 3 ml of solution. The micro-switch stops were adjusted to reverse the action of the reservior pumps after 40 ml of piston displacement in reservior syringe. The temperature of the hot bath water was kept at $40^{\circ}C$ and that of the cold water bath at $20^{\circ}C$. The reservior pump speed was set to deliver 0.233 ml/minute and that of feed pump was set to deliver 0.0808 ml of feed per minute.

To start the run, the feed and reservior pumps were switched on and the timer was activated. The first half cycle was hot up flow, during which bottom reservior syringe pushed the fluid into the bottom of the column and the timer switched the soliniod to supply hot water

at $40^{\circ}C$ to the jacket. During this half cycle 7 ml of the bottom product and 8 ml of the top product solution were collected in receivers. At the end of three hours half cycle, the timer switched the solenoids to supply cold water at $20^{\circ}C$ to the jacket and the micro-switch simultaneously reversed the reservior pump movement. This was the second half of the first cycle, called the cold down flow. At the end of every half cycle, the position of the reservior pistons and the feed syringes were recorded as well as the volume of top and bottom products collected. When the feed pump syringes were nearly empty, they were be refilled at

the end of half cycle. Dow Corning grease (insoluble in water) was used to lubricate the syringes. Two small magnetic stirrers were used in the reservior syringes to assure perfect mixing. At the end of the cold half cycle the second cycle was started and the procedure was repeated till six cycles were completed.

The analysis of all cold down flow samples were performed gravimetrically for total sugar and by polar-imetrically for fructose concentration. The concentration of glucose was obtained by difference. The results of the analysis are shown in Tables (10) and (11).

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DISCUSSION

Fullers earth has been selected as an adsorbent for glucose and fructose solution, because previous experimental investigations related to this field of research indicate that it is the only adsorbent which adsorbs sugar selectively, although charcoal has greater capacity to adsorb sugars but it adsorbs glucose and fructose approximately to the same extent.

A lot of experimental work was performed to determine adsorption rates at different temperatures after estimating the contact time and the adequate dosage of fullers earth in preliminary experiments. These two conditions were kept constant in all the experiments. The adsorption rates of glucose and fructose were estimated at various temperatures. At every temperature three or four adsorption rates were determined, using 2.5, 5, 10 and 15 % concentration by weight of sugars solution. The analysis of the solutions was done gravimetrically, which was explained in Appendix (II). The adsorption data was fitted reasonably good to Langmuir's Equations.

It is obvious that the powdered fullers earth used to develope Langmuir's adsorption isotherms is not suitable for packing the column in parametric pumping experiment. Therefore, the course grade 30/60 mesh RVM fullers earth was also tested under the same conditions and the adsorption results shown in graph(11) concluded that its

(16)

adsorption behavior was more or less the same as that of
the powdered form but it required longer time(2hrs.) to
reach equilibrium.It was estimated to be one hour for the
powdered form. In fact, the equilibrium time was needed
to set half cycle time in subsequent parametric pumping
experiment. Thremal parametric pumping column was packed
with 30/60 mesh RVM fullers earth, which worked well till
six complete cycles but it became difficult to operate
the pumps beyond that, due to breakdown of fullers earth
particles. The number of cycles would have exceeded this
limit, if the experiment had not been interupted. The total
exposure time is probably, the greatest factor in break-
down of particles. The flow in the column is laminar,hence
the possibility of turbulence in the column, is certainly
not a factor in breakdown. The change in temperature could
be a factor but probably not as important as time.

Anyhow the results obtained in all six cycles were
plotted in graph(12), which shows successful separation.

RECOMMENDATIONS

The cycle time 6 hrs, is too lengthy to be economical in practice. Hence it is quite important to reduce the cycle time and make the process attractive to industries.

The following recommendations are suggested to improve the operating conditions of thermal parametric pumping.

1: Other types of fullers earth, like 30/60 mesh LVM or the mixture of LVM and RVM could be tried to get shorter equilibrium time.

2: A specially treated fullers earth should be used in the colunm, so that it should retain its structure during thermal parametric pumping experiment.

3: The temperature range could be expanded (e.g. $10^{o}C$ - $60^{o}C$) to get separation in relatively less number of cycles.

4: In case,if it is not possible to shorten cycle time through any of the above mentioned suggestions then it would be necessary to look for another adsorbent e.g. ionexchange resins, specially treated charcoal etc.

For a new adsorbent except ionexchange resins, the relative adsorption strength of glucose and fructose should be estimated experimentally. And the minimum time required to reach equilibrium should also be determined before starting thermal parametric pumping experiment.

NOTATION

b = dimentionless equilibrium parameter.

$C_1 = \dfrac{V_T}{Q}$, dimensionless.

$C_2 = V_B/Q$, dimensionless.

ΔH = heat of adsorption, g-cal/gm.mole solute adsorbed.

h = column height, m.

L = penetration distances (see ref. 2)

K = adsorption equilibrium parameter defined by Eq(5).

K^0 = adsorbtion equilibrium constant.

M = equilibrium constant.

m_0 = equilibrium constant, dimentionless (see ref. 1).

N = Langmuir equation constant.

n = number of cycles of pump operation.

p_1, p_2 = defined by Eq.(3) and (8) in ref.(1).

Q = reservior displacement rate, $cm^3/$ sec.

q_1, q_2 = defined by Eq.(3) and (8) in ref.(1).

R = gas constant, 1.987 g-cal/g-mole.K^0.

T = temperature in K^0.

v_0 = interstitial velocity, m/sec.

V_T = top reservior volume, $cm.^3$

V_B = bottom reservior volume, $cm.^3$

X = g-moles of solute adsorbed/ gm.solid adsorbent.

Y = concentration of solute in the liquid phase,

in g-moles solute / g-mole solution.

$\langle \ \rangle$ = average value.

Greek Letters

ϕ = product volumetric flow rate/reservior displacement, dimensionless.

$\dfrac{\pi}{\omega}$ = duration of half cycle, sec.

Subscripts

O = initial condition.

1 = upflow.

2 = downflow.

BP = bottom product.

TP = top product.

B = stream from or to bottom of the column.

T = stream from or to top of column.

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TABLE 1 : Experimental Results of Glucose Adsorption

Temp.	B	C	V	D
10°C	$\dfrac{0.1143 \text{ gm.}}{5\text{c.c.soln.}}$	$\dfrac{0.1100 \text{ gm.}}{5\text{c.c.soln.}}$	98 c.c.	0.08430 gm.
	$\dfrac{0.25550}{5\text{c.c.soln.}}$	$\dfrac{0.24150}{5\text{c.c.soln.}}$	54	0.15120
	$\dfrac{0.10420}{1\text{c.c.soln.}}$	$\dfrac{0.0940}{1\text{c.c.soln.}}$	24	0.24500
	$\dfrac{0.14650}{1\text{c.c.soln.}}$	$\dfrac{0.1330}{1\text{c.c.soln.}}$	20	0.27000
20°C	$\dfrac{0.1262625}{5\text{c.c.soln.}}$	$\dfrac{0.11800}{5\text{c.c.soln.}}$	39.6	0.0654388
	$\dfrac{0.255102}{5\text{c.c.soln.}}$	$\dfrac{0.24400}{5\text{c.c.soln,}}$	49	0.1087996
	$\dfrac{0.208334}{2\text{c.c.soln.}}$	$\dfrac{0.20000}{2\text{c.c.soln.}}$	48	0.200016
25°C	$\dfrac{0.1262625}{5\text{c.c.soln,}}$	$\dfrac{0.1207625}{5\text{c.c.soln.}}$	39	0.044560
	$\dfrac{0.255102}{5\text{c.c.soln.}}$	$\dfrac{0.24150}{5\text{c.c.soln.}}$	49	0.097934
	$\dfrac{0.208334}{2\text{c.c.soln.}}$	$\dfrac{0.20200}{2\text{c.c.soln.}}$	48	0.152016
	$\dfrac{0.161400}{1\text{c.c.soln.}}$	$\dfrac{0.15500}{1\text{c.c.soln.}}$	31	0.19840

TABLE 1: —Continued.

Temp.	B	C	V	D
30°C	$\dfrac{0.12315 \text{ gm.}}{5 \text{ c.c. soln.}}$	$\dfrac{0.1190 \text{ gm.}}{5 \text{ c.c. soln.}}$	40.6	0.033698 gm.
	$\dfrac{0.255102}{5 \text{ c.c. soln.}}$	$\dfrac{0.24850}{5 \text{ c.c. soln.}}$	49	0.06468
	$\dfrac{0.208334}{2 \text{ c.c. soln.}}$	$\dfrac{0.203134}{2 \text{ c.c. soln.}}$	48	0.12480
40°C	$\dfrac{0.10200}{5 \text{ c.c. soln.}}$	$\dfrac{0.10000}{5 \text{ c.c. soln.}}$	50	0.02000
	$\dfrac{0.11350}{2 \text{ c.c. soln.}}$	$\dfrac{0.11140}{2 \text{ c.c. soln.}}$	43	0.04515
	$\dfrac{0.20840}{2 \text{ c.c. soln.}}$	$\dfrac{0.20480}{2 \text{ c.c. soln.}}$	45	0.08100

B = Initial concentration of glucose solution.

C = Equilibrium concentration of glucose soln.

V = Total volume of glucose solution used.

D = Amount of glucose adsorbed per gm. Adsorbent.

Time of contact for all these experiments was 2hr.

TABLE 2 : Experimental Results of Fructose Adsorption

Temp.	B	C	V	D
10°C	$\dfrac{0.1280\ gm.}{5c.c.soln.}$	$\dfrac{0.1255\ gm.}{5c.c.soln.}$	48.8 c.c.	0.024400 gm.
	$\dfrac{0.113600}{2c.c.soln.}$	$\dfrac{0.10900}{2c.c.soln.}$	22.0	0.050600
	$\dfrac{0.208400}{2c.c.soln.}$	$\dfrac{0.20200}{2c.c.soln.}$	24.0	0.076800
	$\dfrac{0.155000}{1c.c.soln.}$	$\dfrac{0.15140}{1c.c.soln.}$	25.0	0.088000
25°C	$\dfrac{0.1262625}{5c.c.soln.}$	$\dfrac{0.12400}{5c.c.soln.}$	39.6	0.018100
	$\dfrac{0.1530600}{3c.c.soln.}$	$\dfrac{0.15080}{3c.c.soln.}$	43.0	0.032540
	$\dfrac{0.2083340}{2c.c.soln.}$	$\dfrac{0.205834}{2c.c.soln.}$	48.0	0.057600
40°C	$\dfrac{0.126250}{5c.c.soln.}$	$\dfrac{0.124250}{5c.c.soln.}$	39.6	0.015940
	$\dfrac{0.25510}{5c.c.soln.}$	$\dfrac{0.252500}{5c.c.soln.}$	49.0	0.025480
	$\dfrac{0.208334}{2c.c.soln.}$	$\dfrac{0.20640}{2c.c.soln.}$	48.0	0.046416

TABLE 3: Experimental Results of Glucose Adsorption

Temp.	X	C	E	Y	Y/X
10°C	0.000468	0.0220	1.0085	0.002223	4.75
	0.000840	0.0483	1.0185	0.004930	5.87
	0.001360	0.0940	1.0370	0.009880	7.27
	0.001500	0.1465	1.0610	0.015780	10.50
20°C	0.0003635	0.0236	1.0087	0.002395	6.59
	0.0006040	0.0488	1.0185	0.005020	8.31
	0.0011100	0.1000	1.0395	0.010670	9.60
25°C	0.0002475	0.02415	1.0090	0.002450	9.90
	0.0005450	0.04830	1.0185	0.004960	9.10
	0.0008450	0.10416	1.0415	0.010960	12.97
	0.0011000	0.16140	1.0675	0.017530	15.93
30°C	0.0001870	0.02380	1.0087	0.002410	12.89
	0.0003600	0.04970	1.0190	0.005110	14.20
	0.0006930	0.10156	1.0400	0.010730	15.50
40°C	0.0001110	0.02000	1.0072	0.002025	18.22
	0.0002520	0.05570	1.0210	0.005730	22.74
	0.0004500	0.10240	1.0410	0.010800	24.00

X = gm.mol.glucose/gm. adsorbent.

Y = gm.mol.glucose/gm.mol.solution.

C = gm.glucose/c.c.solution left after the test.

E = Density of solution in gm.soln./c.c.soln.

TABLE 4: Experimental Results of Fructose Adsorption

Temp.	X	C	E	Y	Y/X
10°C	0.0001355	0.02510	1.010	0.002540	18.70
	0.0002810	0.05450	1.021	0.005610	20.00
	0.0004270	0.10100	1.039	0.010650	25.00
	0.0004720	0.15140	1.065	0.016560	35.00
25°C	0.0001005	0.02480	1.010	0.002515	25.10
	0.0001810	0.05027	1.020	0.005190	28.70
	0.0003140	0.10292	1.040	0.010870	34.60
40°C	0.0000885	0.02485	1.010	0.002520	28.70
	0.0001416	0.05050	1.020	0.005140	36.30
	0.0002580	0.10320	1.040	0.010900	42.25

X = gm.mol.fructose/gm. adsorbent.

Y = gm.mol.fructose/gm.mol.solution.

C = gm.fructose/c.c.solution left after the test.

E = Density of solution in gm.soln./c.c.soln.

(27)

TABLE 5: Effect of Adsorbent Amount Used for Test Using Powdered Fullers Earth[1]

Concentration of glucose solution = 5 %

Volume of glucose solution used = 49 c.cs

Contact time = 2 hrs.

Temperature = 25^0C

A = Amount of adsorbent used in gms.

B = Glucose in 5c.cs of solution before test.

C = Glucose in 5c.cs of solution after test.

D = Total amount of glucose adsorbed.

Test No.	A	B	C	D
1	0.50	0.255102	0.25110	0.0784
2	1.00	0.255102	0.24510	0.0980
3	2.00	0.255102	0.23740	0.0868
4	5.00	0.255102	0.21300	0.0825

1 : Manufactured by S.B. Penick & Co. N.Y.

(28)

TABLE 6: Equilibrium Time for Glucose Adsorption

(a) Using Powdered Fullers Earth (From S.B.Penick)

Amount of fullers earth used = 1.0 gm.

Other experimental conditions are same as

given in Table(5)

No.	Contact Time	B	C	D
1	30 Minutes	0.255102	0.24800	0.079599
2	60 "	0.255102	0.24510	0.098000
3	90 "	0.255102	0.24520	0.097200
4	120 "	0.255102	0,24510	0.098000

(b) Using RVM 30-60Mesh Fullers Earth

Concentration of glucose solution = 10 %

Total volume of solution used   = 48 c.cs

B & C represent the amout of glucose in 2c.cs

of solution before and after the test respectiv-

ely. Other experimental conditions are same.

No.	Contact Time	B	C	D
1	55 Minutes	0.208334	0.20400	0.104016
2	120 "	0.208334	0.20300	0.134480
3	180 "	0.208334	0.20210	0.152000
4	240 "	0.208334	0.20280	0.132816

TABLE 7 : Equilibrium Time for Fructose Adsorption
Using RVM 30-60 Mesh Fullers Earth

Concentration of fructose solution = 10 %

Total volume of fructose solution = 48 c.cs

Amount of fullers earth used = 1.0 gm.

Temperature = $25^{o}C$

B & C represent the amount of glucose in 2c.cs
of solution before and after the test respect-
ively,

No.	Contact Time	B	C	D
1	60 Minutes	0.208334	0.206084	0.054000
2	120 "	0.208334	0.206100	0.053616
3	180 "	0.208334	0.206274	0.049440

TABLE (8-a)

Calculation of Dimensionless Equilibrium
Parameter "b"

Parameters	Glucose	Fructose
M_{20}	0.0800	0.0327
m_{20}	4.5120	1.8450
M_{40}	0.0400	0.0220
m_{40}	2.2560	1.2400
m_o	3.3840	1.5425
a	1.1280	0.3025
b	0.2570	0.1190

Necessary Operating Conditions for
Continuous Parametric Pumping.

Cold cycle Temp. $T_c = 293°K$,   Hot cycle Temp. $T_h = 313°K$

$Q \doteq 0.222$ c.c/min. $\frac{\pi}{\omega} = 180$ min.

$Q\frac{\pi}{\omega} = 40.000$ c.c. $F = 0.0896$ c.c/min.

$\phi_B = 0.200$ $P_B = 0.0443$ c.c/min.

$\phi_T = 0.204$ $P_T = 0.0453$ c.c/min.

TABLE 8-b: Parametric Pumping Experiment Observations

Pump Type   Continuous Top Feed.

System   (Glucose + Fructose + Water)

$Q(\frac{\pi}{\omega})$ =   40.00 c.c.

π/ω =   180 Minutes.

Temperatures   Cold $20^{o}C$ & Hot $40^{o}C$.

Cycle No. n	Position of Top Reserv.	Position of Bot. Reserv.	P_B c.c.	P_T c.c.	Feed F_1	Pumps F_2
0	3.0 c.c.	43.0 c.c.	–	–	23 c.c	24 c.c
1-Up	43.0	3.0	7.0	8.0	14.5	15.5
1-Down	7.0	40.0	7.5	9.5	5.5	6.5
Feed syringes were refilled with feed.					32.0	32.0
2-Up	45.0	3.0	7.8	9.0	23.0	23.0
2-Down	5.0	42.5	7.0	10.2	14.5	14.5
3-Up	43.0	3.0	8.0	10.6	6.0	6.0
Feed syringes were filled with feed					15.0	15.0
3-Down	10.0	40.0	7.0	11.0	4.5	4.5
Feed syringes were filled with feed					32.0	32.0
4-Up	43.0	3.0	8.8	10.8	22.0	22.0
4-Down	12.0	38.0	7.0	11.0	11.0	11.0
5-Up	43.0	3.0	8.5	10.0	1.0	1.0

TABLE 8-b:- Continued -

CycleNo.	Position of Top Reserv.	Position of Bot.Reserv.	P_B c.c.	P_T c.c.	Feed Pumps F_1	F_2
Feed syringes were refilled with feed					15.0	15.0
5-Down	14.0c.c.	36.0 c.c.	7.0	11.0	4.0	4.0
Feed syringes were refilled with feed					30.0	30.0
6-Up	42.0	4.0	7.0	10.0	22.0	22.0
6-Down	22.0	29.0	7.0	11.4	10.0	10.0

F_1 & F_2 represent c.c. of feed solution in the syringes. Due to leakage in feed syringes there will be a small difference between the total volume collected as products and the feed volume pumped.

TABLE 9: Analysis of Fructose by Polarameter

Feed solution (10gm.Gluc.+10gm.Fruc.+80gm.Water)

Total volume of feed solution $\qquad$ = 92.0 c.c.

Amount of frucose in 100 c.c. of soln. = 10.869 gm.

Increase in rotation for $\dfrac{1.0gm.fructose}{100 \ c.c. \ soln.}$ = 2.90/10.869 = 0.2667^{o}

Sample	Rotation at 22°C	Rotation at 70°C	Increase in rotation	Fructose in 100c.c.soln.
Feed	-4.030^{o}	-1.130^{o}	2.90^{o}	10.8690
1TopDown	-3.900	-1.060	2.840	10.6486
2TopDown	-3.935	-1.005	2.930	10.9861
3TopDown	-3.916	-1.004	2.912	10.9186
4TopDown	-4.050	-1.100	2.950	11.0611
5TopDown	-4.188	-1.212	2.976	11.1586
6TopDown	-4.206	-1.267	2.939	11.0198
1BotDown	-3.580	-0.910	2.670	10.0078
2BotDown	-3.625	-0.925	2.700	10.1237
3BotDown	-3.500	-0.880	2.620	9.82370
4BotDown	-3.745	-0.901	2.844	10.6636
5BotDown	-3.830	-0.986	2.844	10.6636
6BotDown	-3.660	-0.922	2.738	10.2662

TABLE 10: Results of Fructose Samples Analysis

S.No.	Gluc.+Fruc.in 1c.c.soln.	Fructose in 1c.c.soln.	$\dfrac{\langle Y_{TP2}\rangle n}{Y_{OF}}$
	gms.	gms.	
Feed	0.21730	0.108695	1.000
1TopDown	0.21530	0.106486	0.979677
2TopDown	0.22030	0.109861	1.010727
3TopDown	0.22230	0.109186	1.0045172
4TopDown	0.22300	0.110611	1.017630
5TopDown	0.22450	0.111586	1.026597
6TopDown	0.22370	0.110198	1.013828
			$\dfrac{\langle Y_{BP2}\rangle n}{Y_{OF}}$
1Bot.Dwn	0.21900	0.100078	0.920723
2Bot.Dwn	0.21000	0.101237	0.931386
3Bot.Dwn	0.21800	0.098237	0.903786
4Bot.Dwn	0.20150	0.106636	0.981057
5Bot.Dwn	0.19700	0.106636	0.981057
6Bot.Dwn	0.18850	0.102662	0.085838

(35)

TABLE 11: Results of Glucose Samples Analysis

S.No.	Glucose in 1c.c.soln. gms.	$\dfrac{\langle Y_{TP2} \rangle n}{Y_{OG}}$
Feed	0.108695	1.000000
1 TopDown	0.108814	1.001095
2 TopDown	0.110439	1.016045
3 TopDown	0.113114	1.040655
4 TopDown	0.112389	1.033985
5 TopDown	0.112914	1.038815
6 TopDown	0.113512	1.044317

		$\dfrac{\langle Y_{BP2} \rangle n}{Y_{OG}}$
1 Bot.Dwn.	0.118922	1.094089
2 Bot.Dwn.	0.108763	1.000626
3 Bot.Dwn.	0.119763	1.101826
4 Bot.Dwn.	0.094864	0.872754
5 Bot.Dwn.	0.090364	0.831354
6 Bot.Dwn.	0.085838	0.789714

TABLE (12)   Calculation of Langmuir's Adsorption
Isotherm Constants Graphically

(a)   Constants for Glucose Isotherms

Slope of all isotherms lines = 1/N = 1/0.0025

Temperature	N	$\frac{1}{NK}$	K
10°C	0,0025	3.50	114.300
20°C	0.0025	5.72	70.000
25°C	0.0025	7.90	50.650
30°C	0.0025	11.80	33.900
40°C	0.0025	19.20	20.820

(b)   Constants for Fructose Isotherms

Slope of isotherm lines =(1/N)=1310

Temperature	N	$\frac{1}{NK}$	K
10°C	0.000764	12.50	105.00
25°C	0.000764	21.50	60.90
40°C	0.000764	29.50	44.40

TABLE (13)

(a) Calculations for Glucose Isotherm

T in K°	$T^{0.5}$	K	$KT^{0.5}$	$\frac{1}{T}$
283°	16.8000	114.30	1920	3.534×10^{-3}
293°	17.1172	70.00	1198	3.412×10^{-3}
298°	17.2626	50.65	824	3.355×10^{-3}
303°	17.4069	33.90	590	3.300×10^{-3}
313°	17.6918	20.82	368	3.195×10^{-3}

(b) Calculations for Fructose Isotherm

T in K°	$T^{0.5}$	K	$KT^{0.5}$	$\frac{1}{T}$
283°	16.8000	105.00	1764.0	3.340×10^{-3}
298°	17.2626	60.90	1050.0	3.355×10^{-3}
313°	17.6918	44.40	785.0	3.195×10^{-3}

(38)

TABLE (14)   Langmuir's Adsorption Isotherms

Constants

$$X = \frac{NKY}{1 + KY} \quad ; \quad K = K^O \cdot T^{-0.5} \cdot e^{(-\Delta H/RT)}$$

Where   N and K^O are indipendent constants
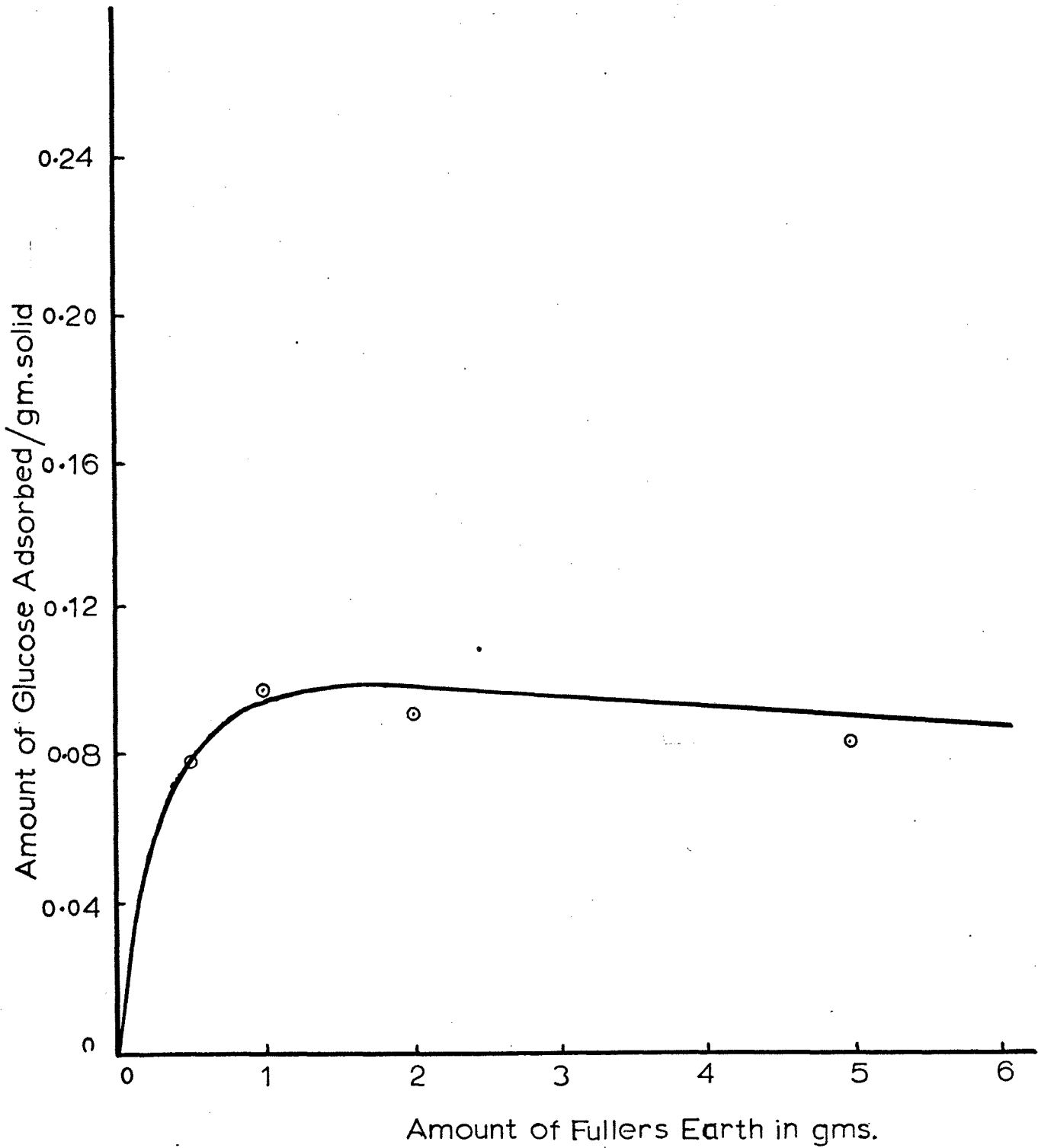
(a) Constants for Glucose Isotherms

N	K^O	$-\Delta H/R$	ΔH
0.0025	0.494×10^{-4}	4960.0	-9820 cal/gmol

(b) Constants for Fructose Isotherms

N	K^O	$-\Delta H/R$	ΔH
0.764×10^{-3}	0.2387	2510.0	-4972 cal/gmol

Graph(1)

Effect of Adsorbent Amount Used for Test

using powdered fullers earth (S.B.Penick)

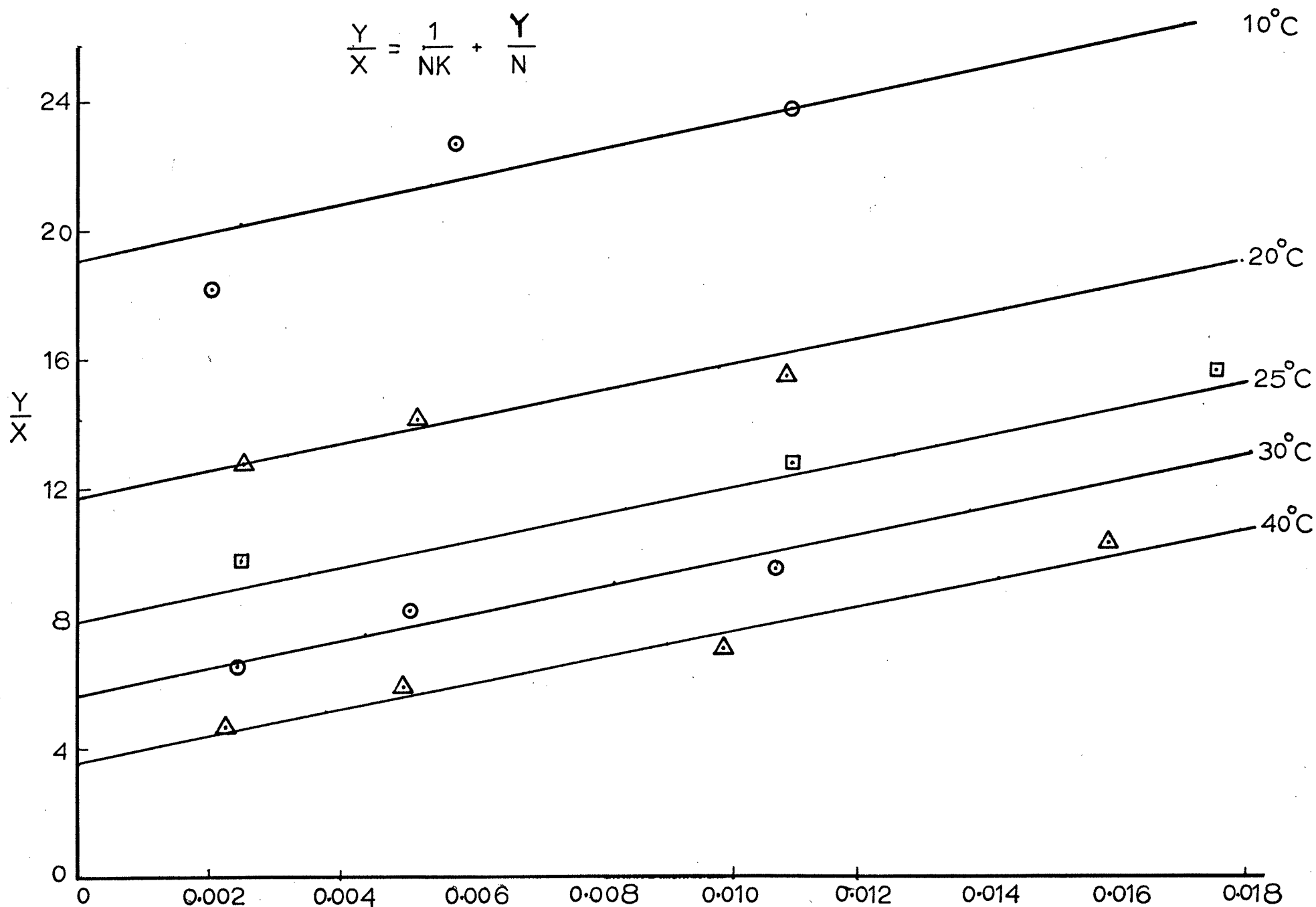Amount of Fullers Earth in gms.

(40)

Graph(2)

Equilibrium Time for Glucose Adsorption
using powdered fuller's earth (s.B.penick)
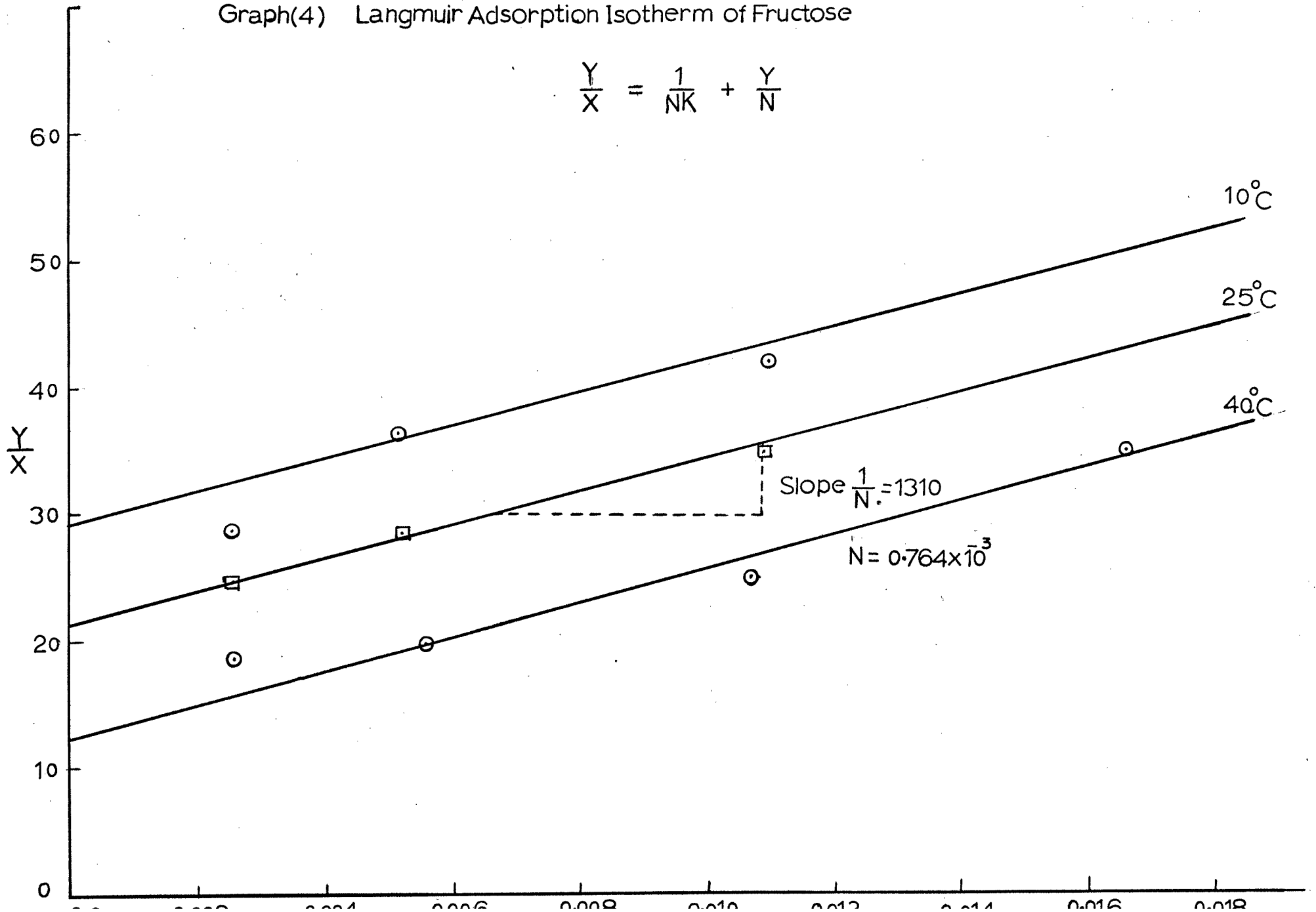
(41)
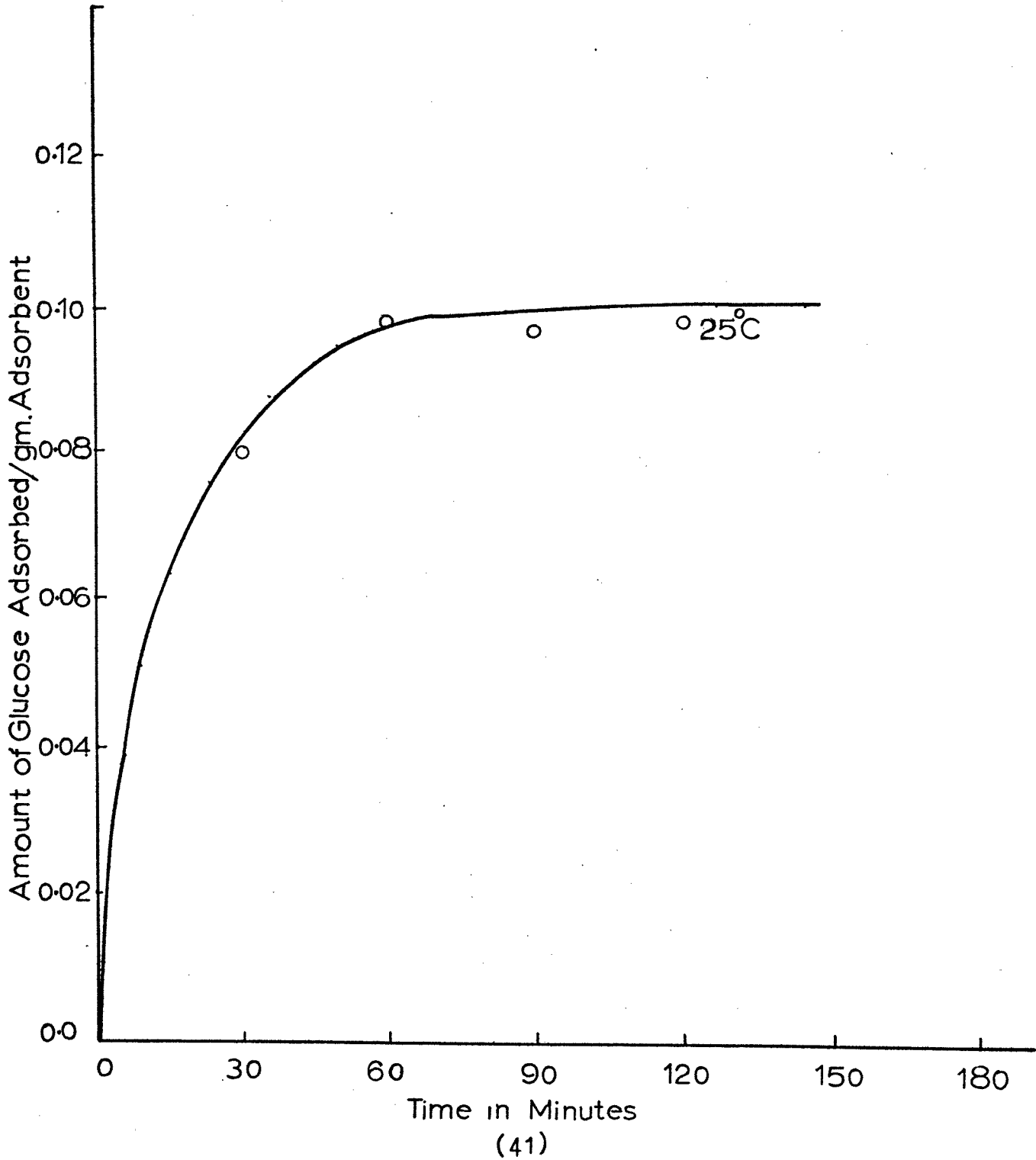
Graph(3)   Langmuir Adsorption Isotherm of Glucose
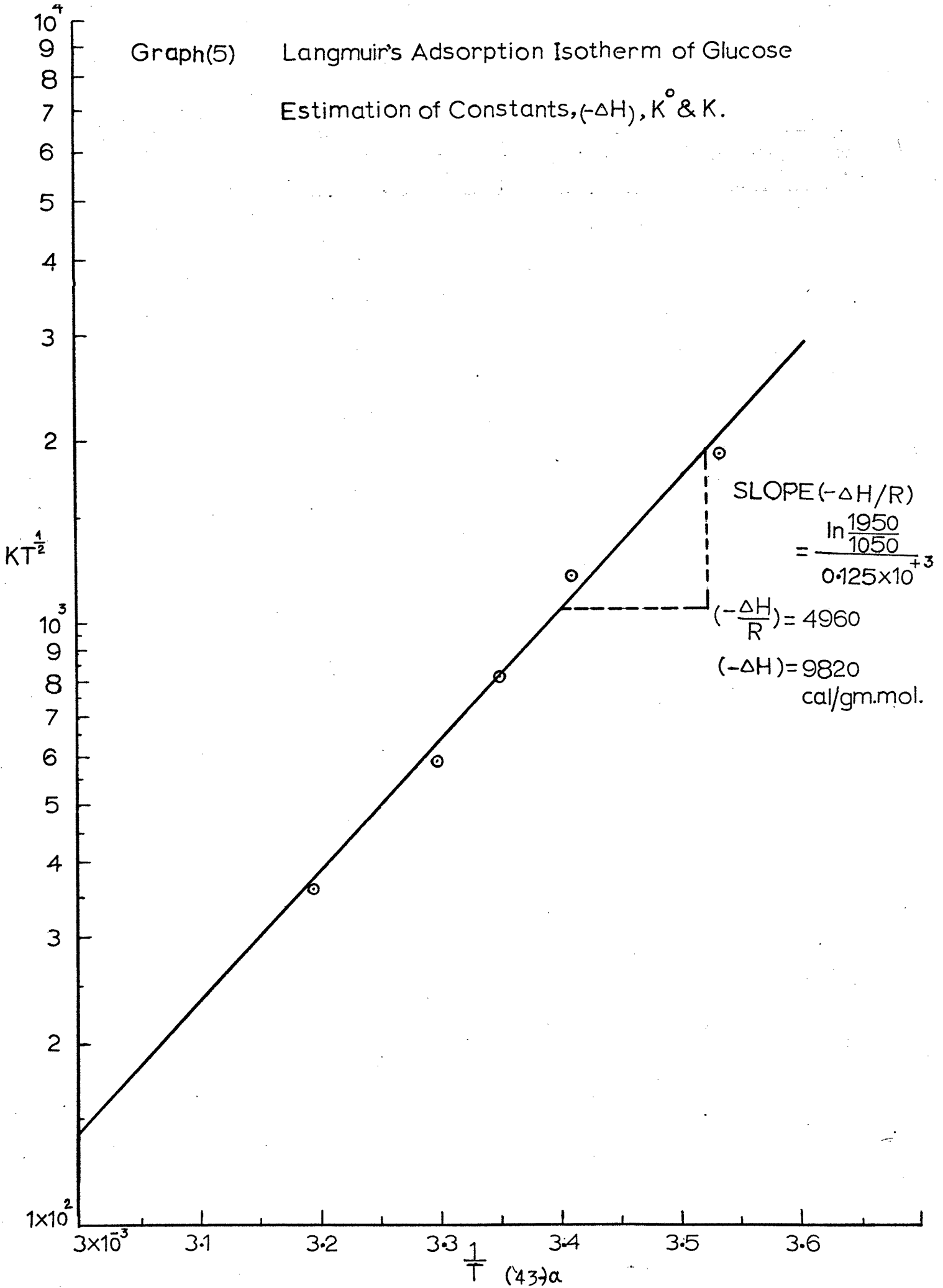
$$\frac{Y}{X} = \frac{1}{NK} + \frac{Y}{N}$$

Graph(4)   Langmuir Adsorption Isotherm of Fructose
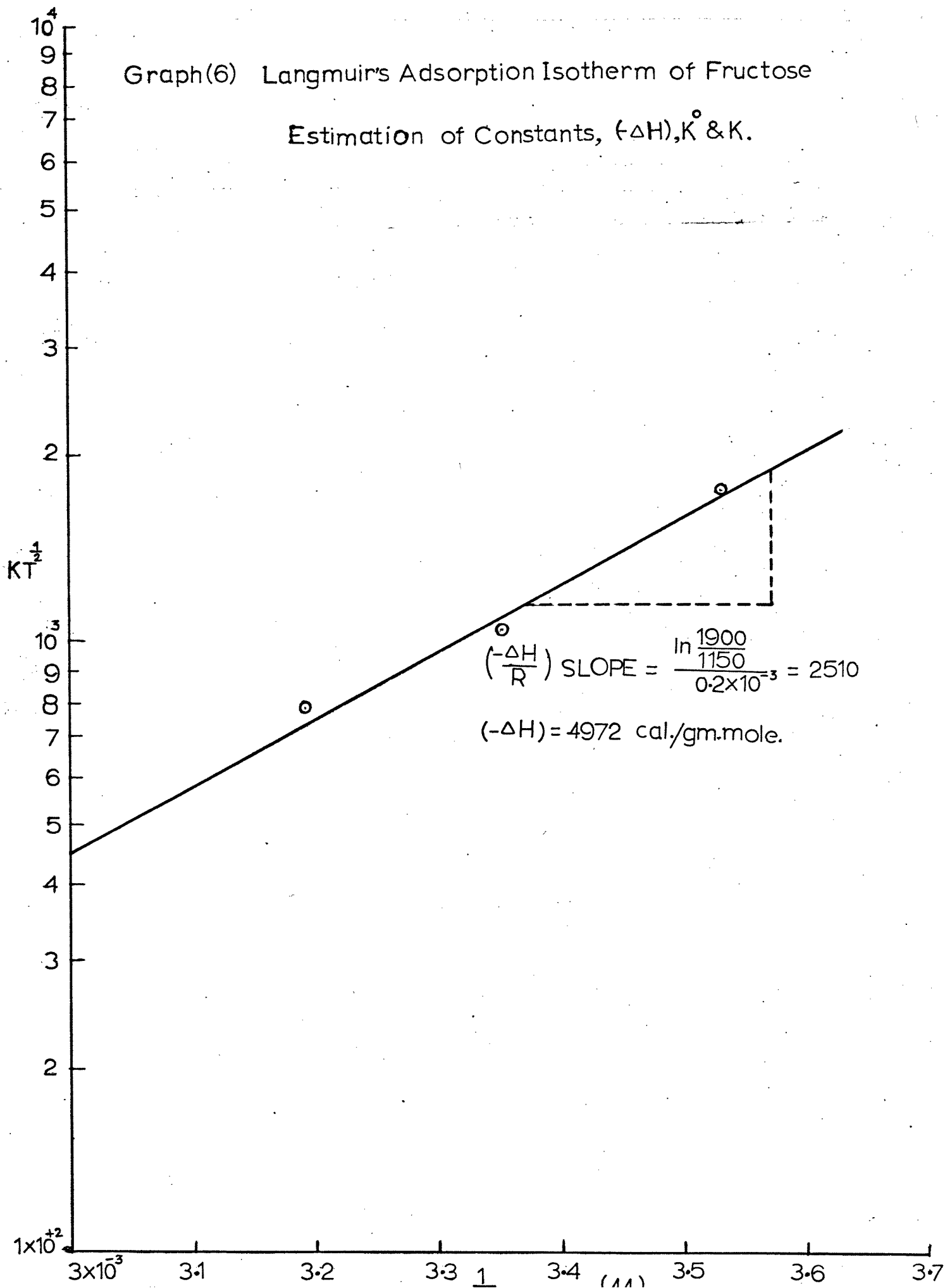
$$\frac{Y}{X} = \frac{1}{NK} + \frac{Y}{N}$$

Slope $\frac{1}{N} = 1310$

$N = 0.764 \times 10^{3}$

$10°C$

$25°C$

$40°C$

Graph(5)   Langmuir's Adsorption Isotherm of Glucose

Estimation of Constants, $(-\Delta H)$, $K°$ & K.

$KT^{\frac{1}{2}}$ (y-axis)

$\frac{1}{T}$ ('43)a (x-axis)

SLOPE $(-\Delta H/R)$

$$= \frac{\ln \frac{1950}{1050}}{0.125 \times 10^{+3}}$$

$\left(-\dfrac{\Delta H}{R}\right) = 4960$

$(-\Delta H) = 9820$ cal/gm.mol.

Graph(6) Langmuir's Adsorption Isotherm of Fructose

Estimation of Constants, $(-\Delta H)$, K° & K.

$$\left(\frac{-\Delta H}{R}\right) \text{SLOPE} = \frac{\ln \frac{1900}{1150}}{0.2 \times 10^{-3}} = 2510$$

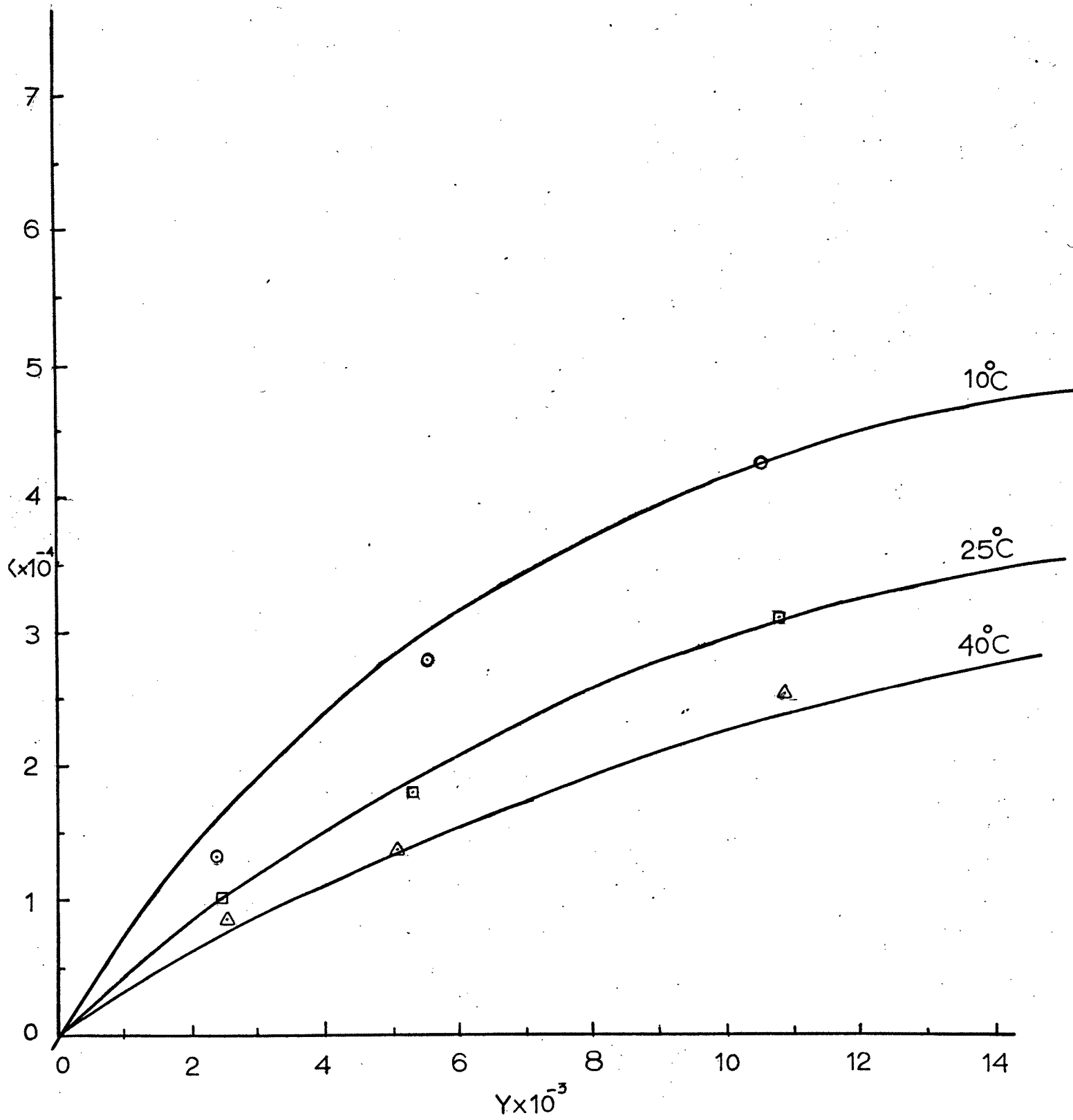$$(-\Delta H) = 4972 \text{ cal./gm.mole.}$$

Graph(7)

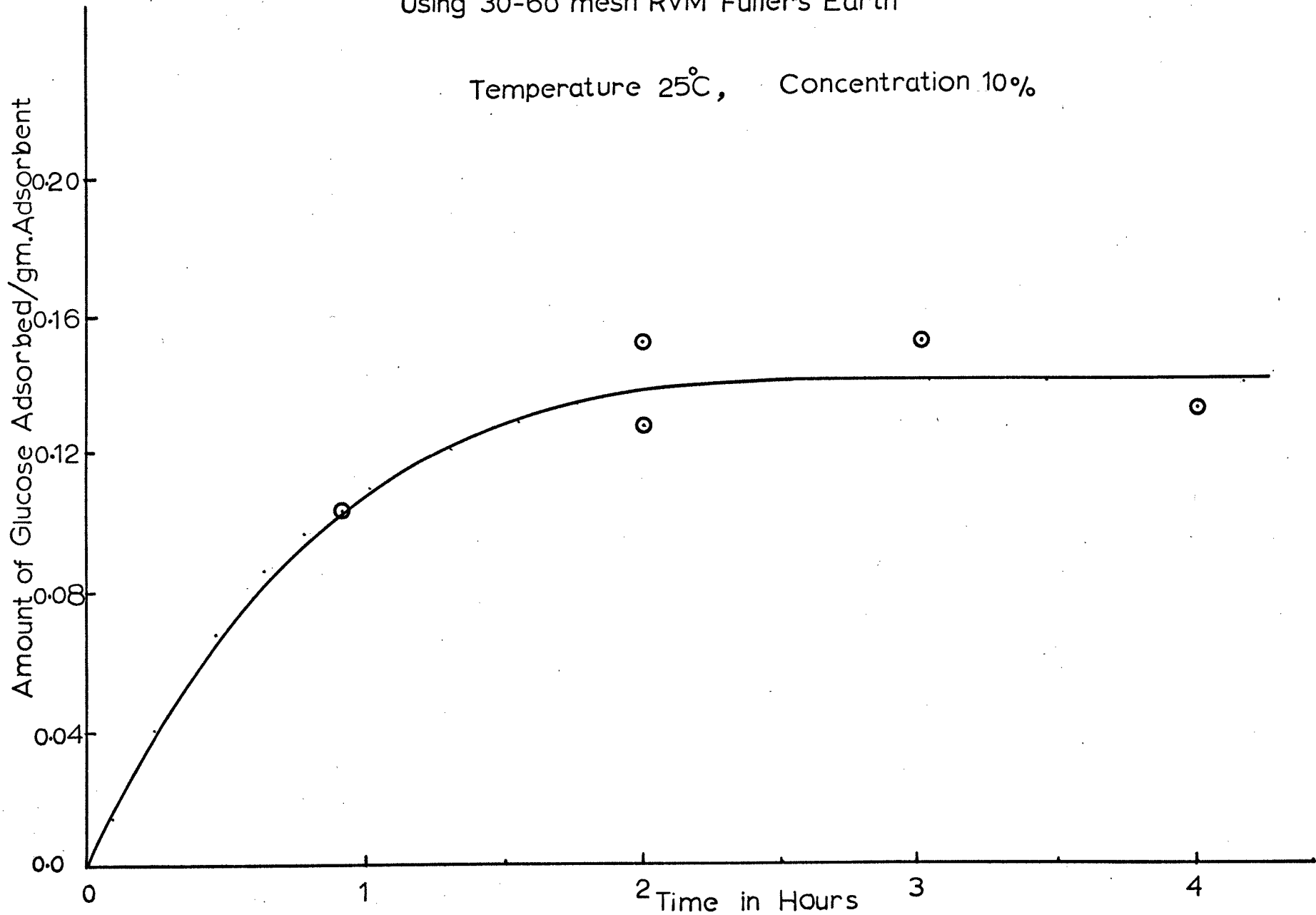Adsorption Isotherms of Glucose
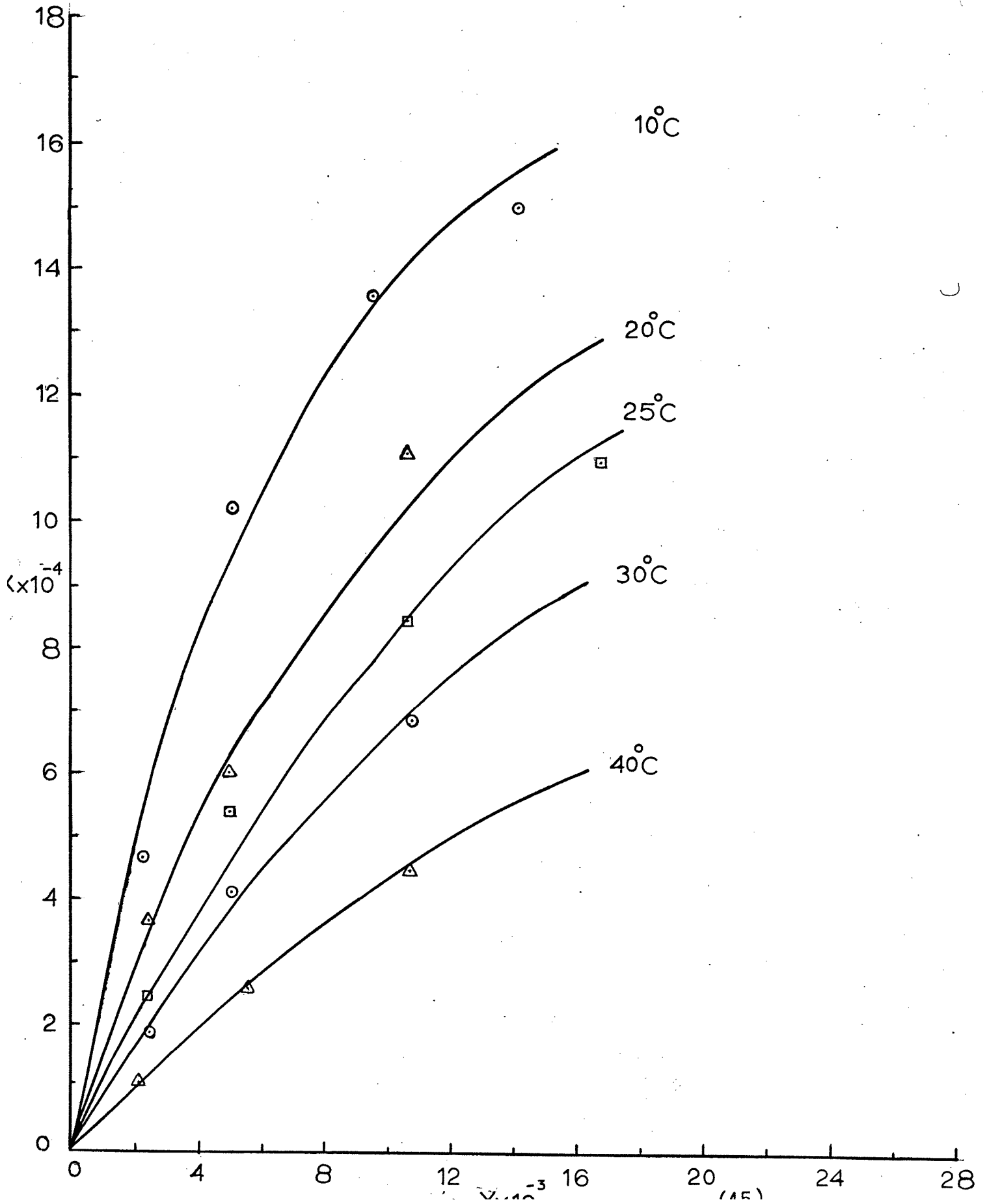
Graph(8)

Adsorption Isotherms of Fructose

(46)

Graph(9)   Equilibrium Time for Glucose Adsorption
Using 30-60 mesh RVM Fuller's Earth

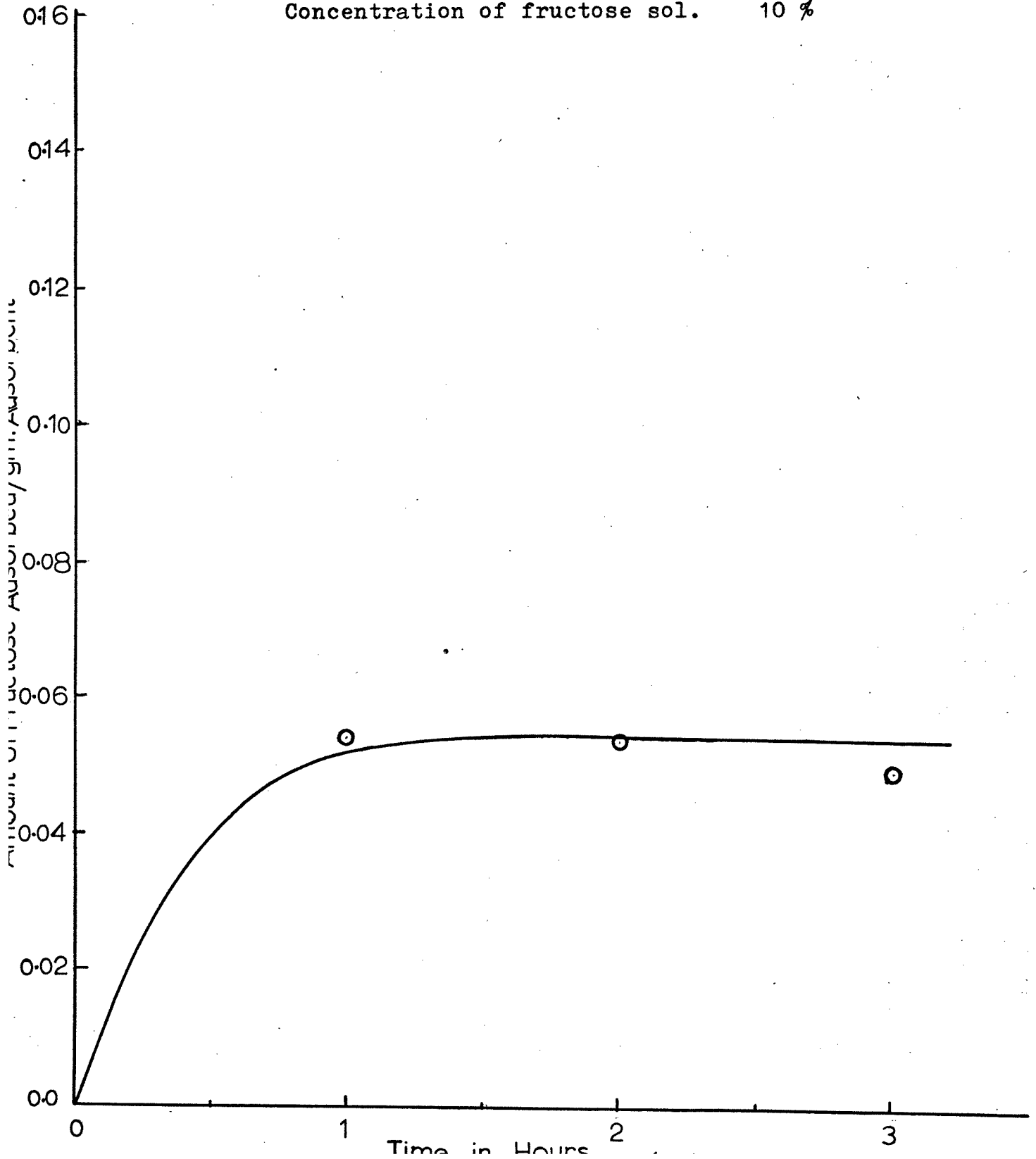Temperature 25°C,   Concentration 10%

Graph (10)

Equilibrium Time for Fructose Adsorption

Using 30-60 Mesh RVM Fullers Earth.

Temperature   25°C

Concentration of fructose sol.   10 %

Graph (11)
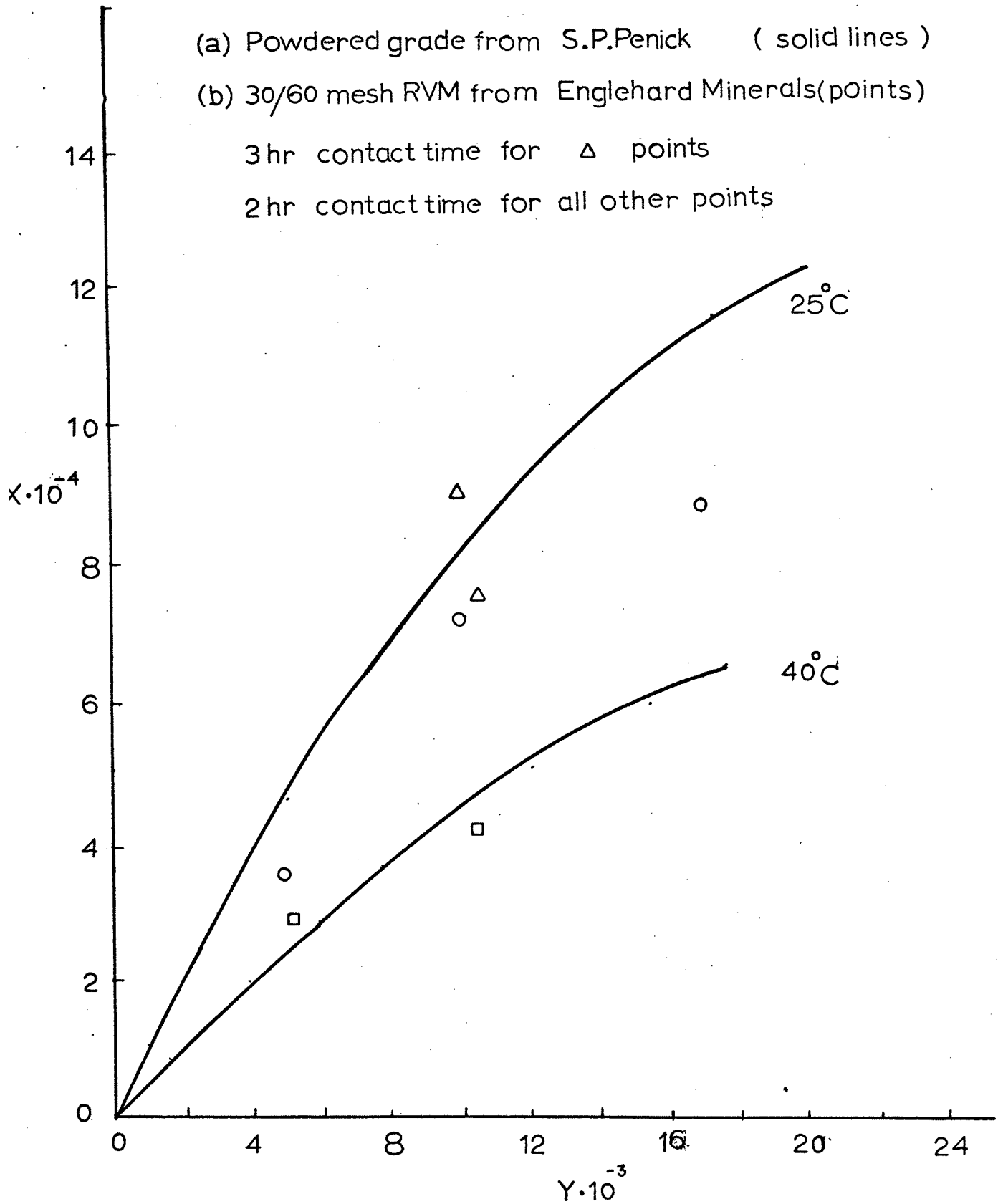Comparison of Glucose Adsorption Data
using two kinds of fullers earth

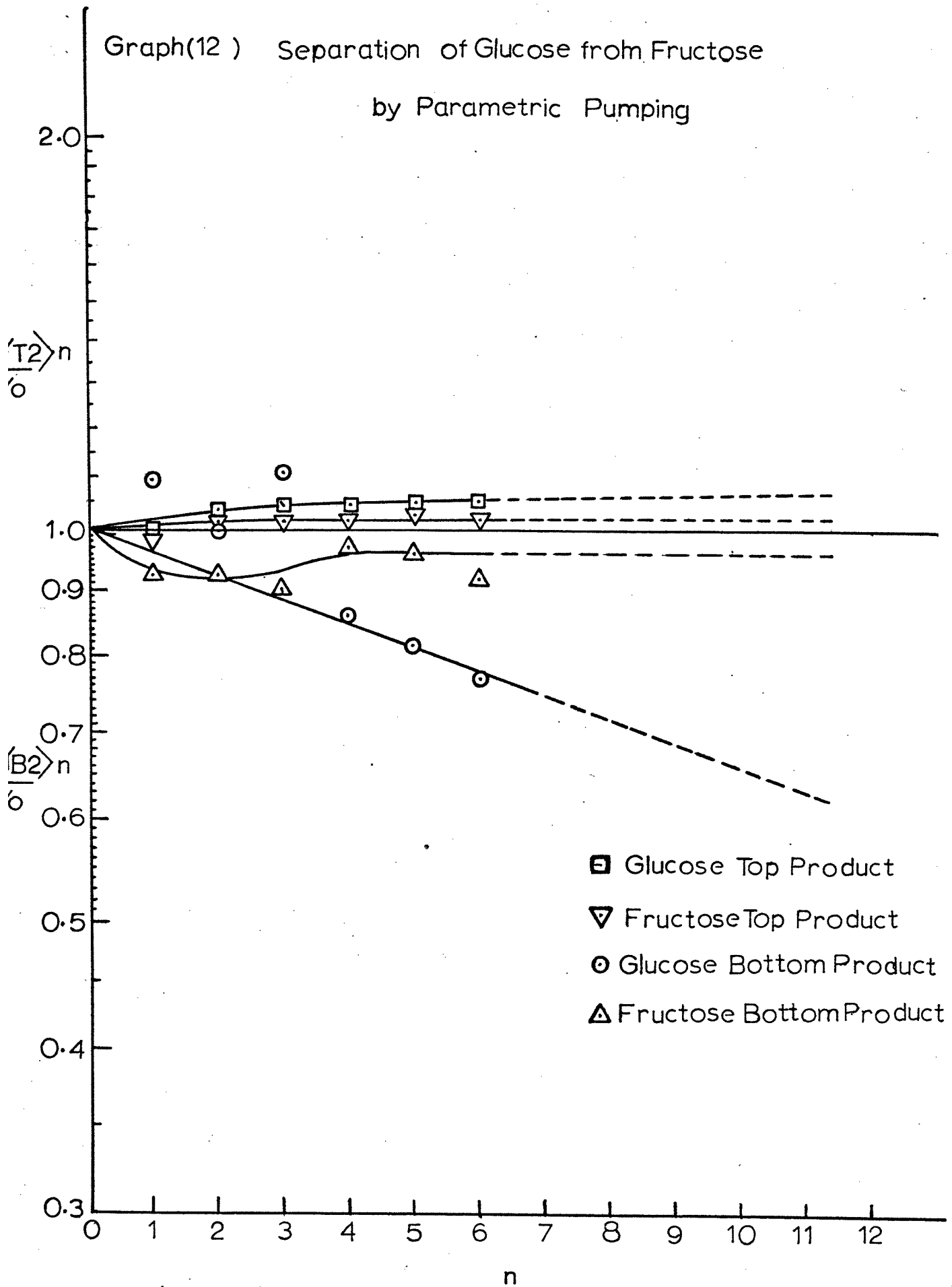(a) Powdered grade from S.P.Penick   (solid lines)

(b) 30/60 mesh RVM from Englehard Minerals (points)

3 hr contact time for △ points

2 hr contact time for all other points

(49)

Graph(12)   Separation of Glucose from Fructose
by Parametric Pumping

$_o^o\widehat{|T2\rangle}n$

$_o^o\widehat{|B2\rangle}n$

Glucose Top Product

Fructose Top Product

Glucose Bottom Product

Fructose Bottom Product

n

(50)

2-WAY
SOLENOID
VALVE

JACKETED
GLASS COLUMN

ABSORBENT
PACKED BED

HOT
WATER
BATH

BOTTOM
RESERVOIR

TOP
RESERVOIR

TIMER

3 WAY
SOLENOID
VALVE

MICROMETER
CAPILLARY
VALVE

COLD
WATER
BATH

BOTTOM
PRODUCT

TOP
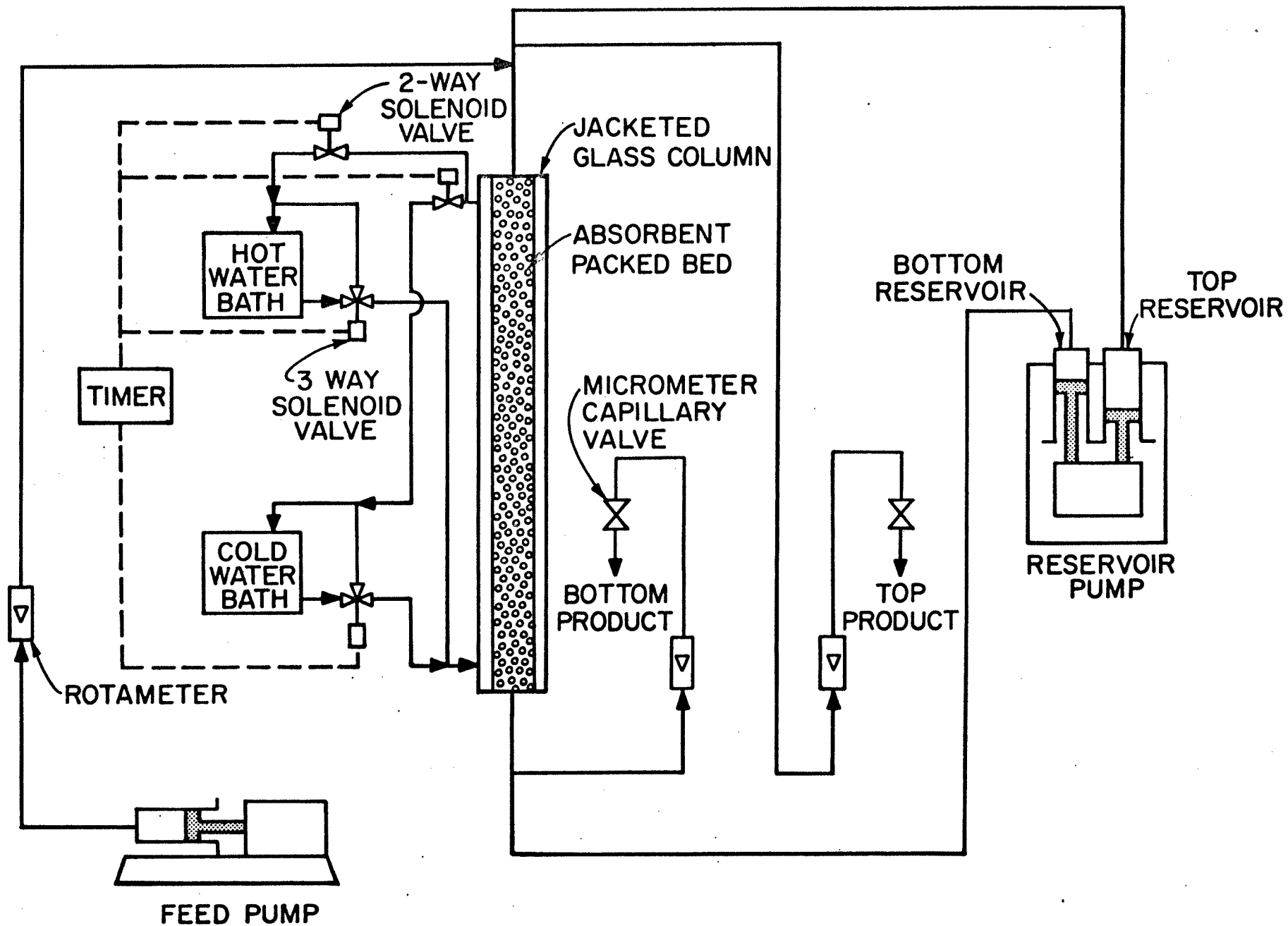PRODUCT

RESERVOIR
PUMP

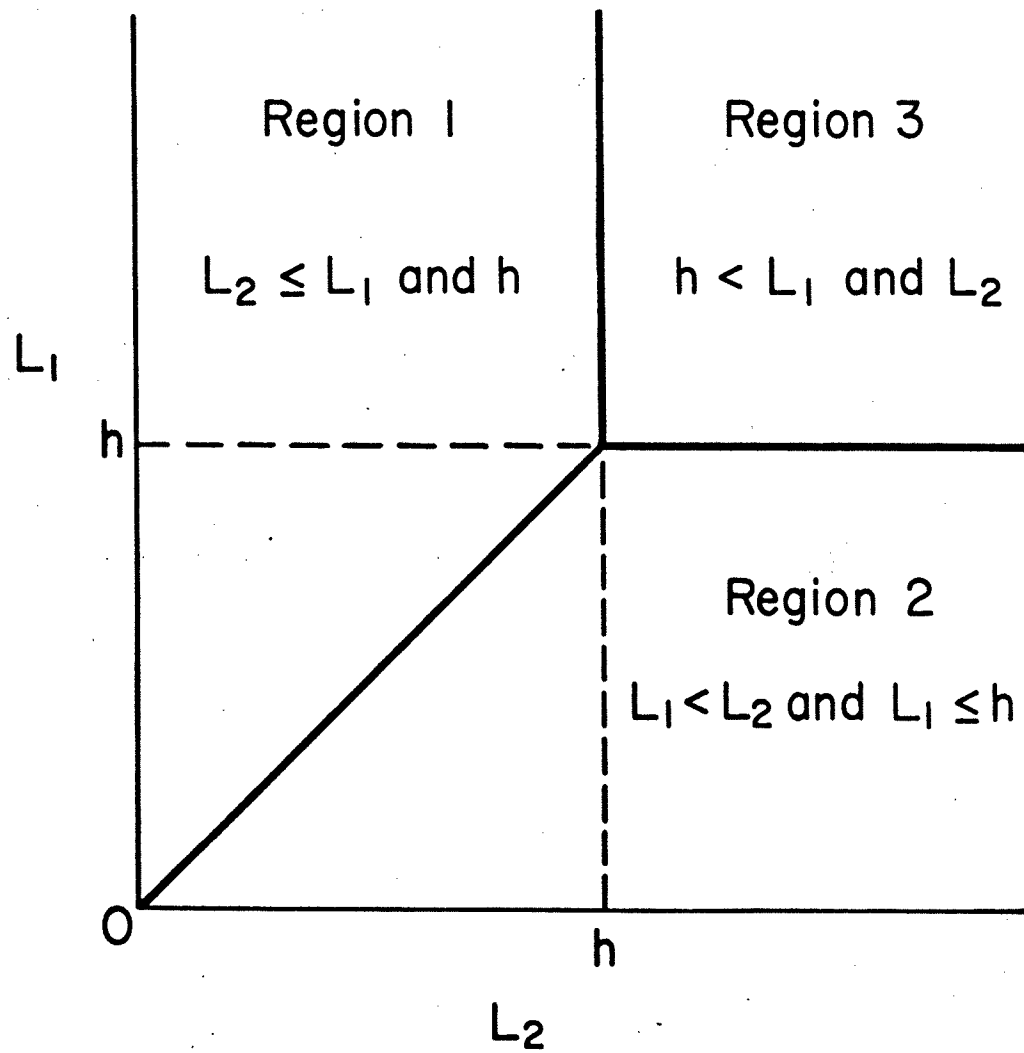ROTAMETER

FEED PUMP

Figure 1

Figure 2

APPENDIX (I)

Langmuir Adsorption Isotherms

Pulverized fullers earth was used as solid adsorbent for adsorption tests. The preliminary experimental results shown in graph (1), indicated that for the concentration ranges under consideration, a dosage of 1gm. fullers earth would be adequate to study the adsorption phenomena.

Aqueous solutions of 2.5, 5, 10, and 15% concentration by weight of glucose were prepared. 50 ml of each solution was placed in 125 ml conical flasks to which 1gm. of fullers earth was added. The flasks were clamped in a mechanical shaker constant temperature water bath, the flasks were sealed with rubber stoppers and immersed in the bath at 15 minutes intervals to allow sufficient time to filter each sample immediately after the contact time (2hrs) had elapsed. The preliminary experimental results plotted in graph (2) show that two hours is more than enough time to reach equilibrium. Another flask containing 50 ml of 10% glucose solution without fullers earth was also immersed in the bath to get a blank reading for purpose of comparision. After two hours of constant shaking at 25°C the slurries were filtered through a Buchner funnel. The filter flask was connected through a water trap flask to a water aspirator. The first few cu. cm. collected were discarded and

the remaining clear filtrate was collected for gravi-
metric analysis which was carried out as follows.

Into 125 ml, 24/40 tappered mouth flask were added
5 ml or, depending on the concentration 2 ml of the filter-
ed glucose solution. The flask was connected to the vacuum
source, the vacuum applied was kept low to avoid boiling
in the begining . But after the solution became very thick
maximum vacuum was applied. A water trap flask was used in
the vacuum line to eliminate the possibility of back suction.
From the blank reading it appeared that after one and a
half hours, the evaporation was essentially complete and
the weighings were correct to within an error of two percent.
Therefore, after one and a half hours, the evaporation was
stopped and the vacuum source was disconnected. The flask
was disconnected from the evaporator and weighed immediately
on an analytical balance. The increase in weight of the
flask gave the amount of glucose in 5 ml or 2 ml of the
equilibrium solution left after adsorption. The amount of
glucose adsorbed per gm. of adsorbent was calculated for
each of the 2.5, 5, 10 and 15% concentration solution
originally used for test. This procedure was repeated for
temperatures of $10^{o}C$, $20^{o}C$, $30^{o}C$ and $40^{o}C$. The adsorption
rates determined , at all these temperatures, were shown
in Table (1).

For fructose, the same procedure was used and the
data is shown in Table (2). However, in this case the

temperatures used were $10^{\circ}C$, $25^{\circ}C$ and $40^{\circ}C$.

From the equilibrium data thus obtained, the values of X, Y and Y/X for glucose and fructose were calculated at all temperatures and applied to the Langmuir's equa-tions.

The Langmuirs adsorption isotherm is represented mathematically as follows.

$$X = \frac{NKY}{1 + KY} \qquad \text{Eq. (4)}$$

This can also be written in the following form.

$$\frac{Y}{X} = \frac{1}{NK} + \frac{Y}{N} \qquad (6)$$

Glucose and fructose adsorption variables Y/X were plotted against Y in graphs (3) & (4) respectively. The best straight lines were fitted through the points. All the straight lines in each set of graphs should be parallel because the slope(1/N) is independent of temperature. The values of (1/NK) were estimated from the intercepts and (1/N) from the slopes. These values were then used to calculate the values of N and K for glucose and fructose adsorption isotherms which are shown in Tables (12a) & (12b) respectively. These values of K estimated at different temperatures were used in preparing Tables (13a) & (13b) for glucose and fructose respectively, showing the values of $T^{0.5}$ and $KT^{0.5}$.

From the Langmuir adsorption isotherms, the values of K is given as,

$$K = K^{o}.T^{-0.5}.e^{(-\Delta H/RT)} \qquad (7)$$

(55)

Equation (7) can be written in the following form,

$$\ln KT^{0.5} = \ln K^O + (-\Delta H/RT) \qquad (8)$$

In graphs (5) & (6), the values of $\ln KT^{0.5}$ vs $1/T$ were plotted on semi-log paper and the best straight lines through these points were fitted. The slope of these lines gave the values of $(-\Delta H/R)$ for glucose and fructose respectively. By knowing $(-\Delta H)$, K and T values the constant K^O was calculated from equation(7) for glucose and fructose. In this way the various constants for the Langmuir adsorption isotherms for glucose and fructose were determined. The values were recorded in Tables (14a) & (14b).

Using these equation constants the adsorption isotherms were calculated at all temperatures. As can be seen in graphs (7) & (8), the experimental points appear to be reasonably close to calculated isotherms.

APPENDIX (II)

Applications of Adsorption Isotherms

The operating conditions for thermal parametric pumping were partially predicted from the equilibrium adsorption isotherms obtained with the powdered fullers earth. Also required was data on rates of adsorption obtained with the same course grade 30/60 mesh RVM fullers earth used to pack the parametric pumping column

The experimental results plotted in graph(9) show that the minimum equilibrium time required for 30/60 mesh, RVM fullers earth,is two hours and the adsorption rate is very close to that of the powdered fullers earth used for adsorption isotherms. Therefore, based on the rate data,the half cycle time for thermal parametric pumping was set at 3 hrs. And based on the equilibrium data, the temperatures selected for hot and cold cycles were 40°C and 20°C respectively. The procedure used to calculate the dimensionless equilibrium parameter,(b) was that of H.T.Chen, et al.[2] The average slopes,(M_T) of the adsorption isotherms at 40°C and 20°C were determined for glucose and fructose.

Then the values of (m_T) was calculated from the following relationship.

$$m_T = \frac{(1 - \epsilon)\cdot\rho_s}{\rho_f\cdot\epsilon}(M_T) \qquad (9)$$

Where ρ_s = 2.45 gm/c.c, = 0.446 & ρ_f = 0.054

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APPENDIX (III)

Parametric Pumping Experiment

Thermal Parametric Pumping apparatus is shown schematically in Fig.(1). The equipment consists of a jacketed glass column of 0.01m inside diameter and 0.9m long, packed with 30/60 mesh RVM fullers earth. Two 50 c.c. glass syringes operated by a dual infusion-withdrawal pump, were used as reserviors at the two opposite ends of the column. To reverse automatically the movement of syringe plungers at the end of each half cycle, a microswitch with stops was wired into the pump circuit. A small magnetic stirrer was placed in each reservior syringe, to get perfact mixing.

The constant temperature hot and refrigerated baths with circulating pumps were used to supply hot and cold water to the column jacket and to recycle to the baths by solenoid valves wired to a dual timer, so that hot water supply pumped to the column jacket during up flow and cold water during down flow.

A second infusion-withdrawal pump with two 50 ml. syringes operated in parallel,was used to pump feed to the top of the column. The top and bottom product take off valves, were micrometer capillary valves,used both to regulate flow and impose a small back pressure on the system. Rotameters were used in the feed and product lines as a check against the feed and the calibrated,

product receivers.

The entire system including the interstitial column volume, the bottom reservior and the feed pump were filled with the feed mixture at ambient conditions, prior to run the experiment. The reservior syringes were set to deliver about 40 ml per half cycle with a minimum dead volume of 3 ml in each syringe.

To start the run, the feed and the reservior pumps were switched on and the timer was activated. The first half cycle is hot up flow. The bottom reservior syringe pumped the fluid into the bottom of the column and the timer switched the solenoids to supply hot water (40°C) to the jacket. At the end of the hot half cycle, the microswitch reversed the action of the reservior pump and simultaneously, the timer switched the solenoids to supply cold water (20°C) to the jacket. This second half cycle is called cold-down flow cycle. The overall procedure was repeated for six cycles.

The samples collected in top and bottom product receivers, were analysed as follows.

An automatic polarameter was used to determined the fructose concentration in sugar solution. A jacketed polarameter tube 1 dm. in length was used to determined the total rotation of sugar solution at two different temperatures, 22°C and 70°C. Hence the rotation of fructose increases with an increase of temperature while that of glucose remains constant, the difference in rotation measured at these two

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This research work was started in Spring 1971, smester, under the supervision of Dr. H.T.Chen, in parametric pumping separation research laboratory housed in Tiernan Hall, 140 High Street, Newark. The most of experimental work was done in Summer 1971, smester. The final draft of this research work was submitted in April, 1972.