UNITED STATES
DEPARTMENT OF THE INTERIOR

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MASSACHUSETTS INSTITUTE OF TECHNOLOGY
CAMBRIDGE, MASSACHUSETTS

OFFICE OF SALINE WATER

RESEARCH AND DEVELOPMENT PROGRESS REPORT NO. 128

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DEPARTMENT OF THE INTERIOR

Stewart L. Udall, Secretary

Kenneth Holum, Assistant Secretary
for Water and Power Development

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WASHING OF BRINE FROM ICE CRYSTALS

by

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MASSACHUSETTS INSTITUTE OF TECHNOLOGY

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for

OFFICE OF SALINE WATER

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Created in 1849, the Department of the Interior--America's Department of Natural Resources--is concerned with the management, conservation, and development of the Nation's water, wildlife, mineral, forest, and park and recreational resources. It also has major responsibilities for Indian and Territorial affairs.

As the Nation's principal conservation agency, the Department of the Interior works to assure that nonrenewable resources are developed and used wisely, that park and recreational resources are conserved for the future, and that renewable resources make their full contribution to the progress, prosperity, and security of the United States--now and in the future.

This is the one hundred and twenty-eighth of a series of reports designed to present accounts of progress in saline water conversion with the expectation that the exchange of such data will contribute to the long-range development of economical processes applicable to large-scale, low-cost demineralization of sea or other saline water.

Except for minor editing, the data herein are as contained in the reports submitted by Massachusetts Institute of Technology, under Grant No. 14-01-0001-295, covering research carried out through August 1964. The data and conclusions given in this report are essentially those of the Grantee and are not necessarily endorsed by the Department of the Interior.

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Abstract

Experimental results are reported for the washing of brine from beds of small plastic and glass beads, and from beds of ice particles prepared by partial freezing of brine. The data are analyzed in terms of a longitudinal-dispersion model which describes the blending of water and brine at the moving wash-water "front". The results lend strong support to the validity of the model as a basis for correlating and interpreting ice-washing data.

The data show that washing is accomplished with the least loss of fresh water if long columns are used, and with no agitation of the bed. Washing performance improves slightly as wash rate is reduced. Washing is much more effective in flooded than in drained beds.

Calculations based on the dispersion model and literature data on washing of sand (13) are used to illustrate the manner in which fresh water wasted varies with the principle design and operating variables for flooded and non-agitated ice-washing columns. These calculations, summarized in Figures 58-61, show that the required washing column height increases sharply as the ice crystal size is increased. However, column capacity increases with increased crystal size, and the wash column volume requirements will probably be lower for larger crystals. Furthermore, brine drains more readily from beds of large particles, and it may prove advantageous to produce large ice particles and to drain the bed prior to washing.

I. FOREWORD

Freezing continues to show promise as a method of producing low-cost fresh water from sea water. By partially freezing salt water, a slush of pure ice crystals suspended in brine can be obtained. The crystals are washed and the ice melted to form pure water.

The object of this report is to present the results of an experimental and theoretical study of the removal of brine from ice-brine "slush" by countercurrent washing. Two separate investigations were carried out: one to study the washing of brine from both glass and plastic beads; and a second to study the batch washing of brine from a mass of small ice crystals.

This work was done under Grant 14-01-0001-295 made to the Massachusetts Institute of Technology by the Office of Saline Water in 1962. The program under the grant included the study of salt build-up at phase boundaries and a basic study of the effect of additives on crystallization of super-cooled water, in addition to studies of the removal of brine from ice brine "slush" by countercurrent washing. Work on washing started in February of 1963. Experimental work was completed by the end of the summer. The ice washing experiments were made by White (14). The results were outlined in the annual progress report under Grant 14-01-0001-295 dated September 30, 1963, by T. K. Sherwood and P. L. T. Brian. This report includes a detailed presentation of the results outlined in the annual report, as well as the results of a comprehensive theoretical study.

II. SUMMARY

This report presents the results of an experimental and
theoretical study of the removal of brine from ice-brine
"slush" by countercurrent washing in a downflow column
operated batchwise. Two separate investigations were carried
out: one to study the washing of brine from inert beads;
and a second to study the batch washing of brine from a mass
of small ice crystals. A primary purpose of this study was
to determine whether the extensive data on longitudinal
mixing of fluids in packed beds obtained in the last 10-15
years by residence-time distribution studies could be directly
applied to the problem of washing brine from ice crystals.
The experiments showed that the wealth of information in the
literature could be directly applied, and that it could be of
great value as a basis for the design of a commercial wash
column.

In most past studies of the washing of brine from ice
crystals, attempts were made to determine mass transfer
coefficients or the height of a transfer unit. Rather than
use this approach, it was felt that the theory of longitudinal
dispersion in packed beds provided a more promising basis for
the interpreparation and correlation of data on washing. The
washing studies herein reported were carried out by "batch" op-
eration, that is, by washing brine from a fixed bed of ice-
brine mixture. This is experimentally simpler than counter-
current washing with continuous supply and removal of ice,

and provides the same basic information of interest.

Classical theory considers a packed bed of uniform cross section and length L, having a void volume V_o. The voids are filled with brine having a salt concentration c_o. A flow of pure water at a volumetric flow rate v and a linear velocity u is started at time $\theta = 0$, and the effluent concentration c is monitored. Four possible concentration profiles are shown in Figure 1. Clearly piston flow (Figure 1a) is the ideal case, since all the wash water can be recovered with the melted ice; however, piston flow never occurs since there is always some longitudinal mixing. Previous studies (7, 8, 9, 11, 12, 13, 17) of flow of fluids through packed beds of spheres indicate that the pattern is usually similar to Figure 1b, and that no stagnation would occur as shown in Figure 1d. Many of the previous studies showed that longitudinal mixing could be treated like diffusion, that the mixing could be characterized by a dispersion coefficient D, and that a Fick's Law type of expression could be used to describe the behavior. The solution to the differential equation describing axial diffusion in a fluid moving at constant flow rate is
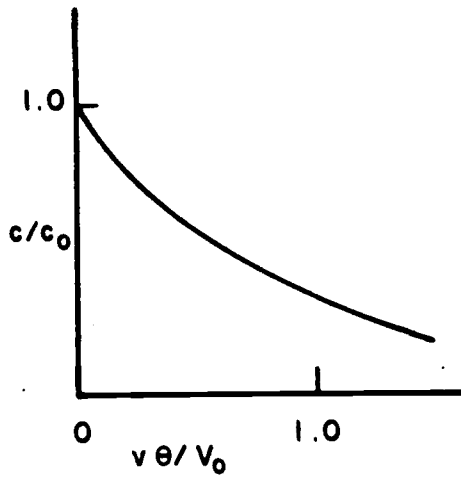
$$\frac{c}{c_o} = \frac{1}{2} + \frac{1}{2} \, erf\left[\frac{1 - v\theta/V_o}{2\sqrt{\frac{v\theta}{V_o} \cdot \frac{D}{Lu}}}\right] \qquad (1)$$
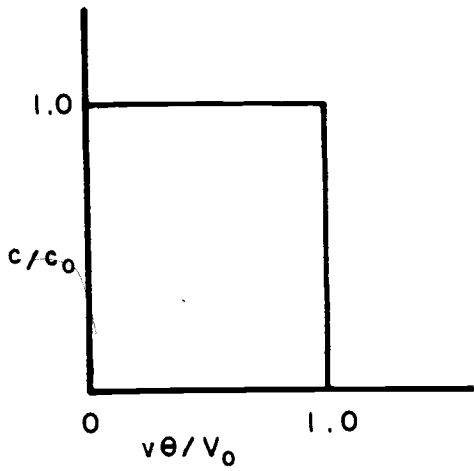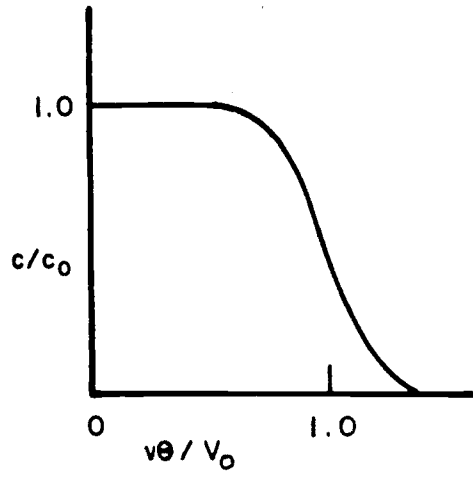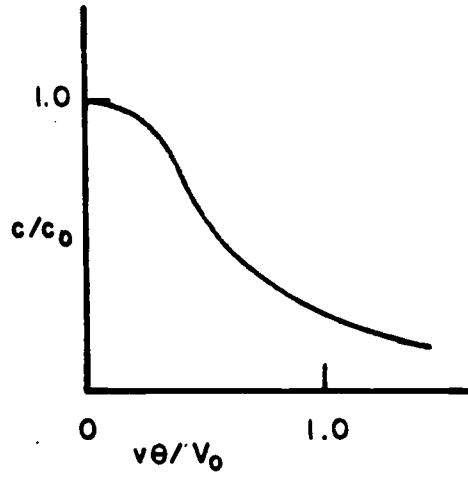
Fig.1 : CONCENTRATION PROFILES

In previous residence-time distribution studies, the data were successfully correlated by describing the Peclet number, N_{Pe}, as a function of the Reynolds number, N_{Re}. The Peclet number is defined as

$$N_{Pe} = \frac{d_p u}{D} \tag{14}$$

and the Reynolds number, N_{Re}, is defined as

$$N_{Re} = \frac{d_p u \rho}{\mu} \tag{15}$$

where d_p is the particle diameter, u is the linear velocity of the fluid past the particle, ρ is the fluid density, and μ its viscosity. The Peclet number was found to be only a weak function of Reynolds number, either being independent of Reynolds number, or varying inversely to a fractional power. It was recognized that quantitative prediction of the performance of a wash column would be possible if it could be shown that the voluminous data on longitudinal mixing in packed beds could be applied to packed or floating ice crystals. Thus the primary objective of current study was a fairly limited one -- to test the applicability of the extensive existing data to ice washing.

At first there seemed reasons to doubt that floating ice crystals would behave in the same manner as a packed bed. It was feared that certain parts of the fluid would short circuit the main body of crystals. It was also feared that

significant quantities of brine would be trapped within irregular ice particles, or within clumps of crystals, and that "tailing" would result. Such behavior would result in curves similar to Figure 1d, and the simple dispersion model would not apply. It was believed that this difficulty could be overcome in a column with a stirrer designed to give a maximum of radial mixing with a minimum of longitudinal mixing.

The washing of brine from inert beads was studied first. Use of the inert beads permitted an investigation of the basic factors involved without attendant complications due to freezing and melting of ice. The results demonstrated that a close approach to piston flow could be accomplished in displacing the brine from beds of beads. In general, the salt concentration in the effluent remained exactly the same as that originally in the bed till nearly 90 percent of the original volume had been displaced. At this point the concentration dropped rapidly and was nearly zero by the time 25 percent more than the original brine volume was displaced.

Almost all of the runs with inert beads were made with agitation. Although nearly all of the information of a basic nature was obtained in subsequent ice-washing experiments, these preliminary experiments were of value in developing experimental techniques, and in showing that agitation was undesirable. As a result of these experiments, emphasis in the ice-washing runs was placed on obtaining data with no agitation.

Probably the most significant finding in the ice-washing studies was that the process can indeed by characterized by a model involving a simple conbination of piston flow and dispersion at the water-brine interface. The data closely followed the relation predicted by Equation 1. The data were tested using other mathematical techniques, and there was no indication of channeling or stagnation, thus fears that significant quantities of brine would be trapped with ice particles were unfounded.

Although the data within any one run were consistent, there was a noticeable scatter when comparing data of different runs. This scatter was probably caused by variation in the size of crystals between runs. Despite this difficulty, it was clearly shown that agitation had a deleterious effect, whether it was from a rotating stirrer, or applied pulsations. The washing efficiency was relatively insensitive to flow rate, but decreased slightly as flow rate was increased. The washing efficiency decreased as the column height was decreased. Results in a drained bed were far inferior to those in a flooded bed.

Using an approximate effective particle diameter of 0.5mm, Peclet numbers in the range 0.25-0.85 were obtained. The data showed a downward trend in Peclet number as Reynolds number was increased. The Peclet number did not seem to be a function of column height. Thus all the data supports the conclusion that the behavior can be predicted by the simple

dispersion model, and that the wealth of existing data on
flow of fluids through packed beds can be directly applied
to washing ice crystals.

If the ice crystals were spherical and of uniform size,
the work of previous investigations in packed beds could be
used with confidence to predict the dispersion coefficient.
Because of the non-uniform size distribution and non-
spherical shape of ice particles, it is as yet not possible to
predict the dispersion coefficient D, except from correla-
tions of experimental data. Even though it is not possible
to calculate the dispersion coefficient D for any set of
conditions, the effect of changes in operating conditions can
be calculated if D/Lu is known for one set of conditions.
The results of the calculations are shown in Figures 57-60.
The conditions of the base case are listed in Table 3 and
indicated by a crosshatched mark on the graph.

As can be seen from Figure 58, wash water wasted increases
with wash rate; however, rather drastic changes in wash rate
do not seriously increase the amount of water wasted.
Figure 61 shows that the amount of wash water wasted depends
strongly on column height. The curve also shows that ex-
cellent performance may be obtained at very moderate heights.
Figures 59 and 60 show the effect of particle size. At any
given flow rate, the wash column height required for a given
amount of wash water wasted should increase almost linearly
as particle size is increased. Figure 60 does not necessarily

indicate that small crystals are desirable, since in a flooded
column with floating particles the wash rates operable without
ice entrainment in the brine effluent decrease drastically as
size of the particles is decreased. Furthermore, since brine
drains readily from beds of quite large particles, it may
prove to be both practical and desirable to produce large ice
particles and drain the brine prior to washing with piston-
flow of wash water.

In conclusion, emphasis was placed on a model using
piston flow with superimposed dispersion. It was demonstrated
that no stagnation occurred in washing ice crystals, and that
the model was valid. Thus the wealth of existing information
on fluid flow through packed beds was shown to be directly
applicable to washing ice crystals.

III. <u>INTRODUCTION</u>

<u>The Freezing Process</u>

A general discussion of the freezing process for de-
salination of water is given in this section. Alternate
methods of removing brine from ice crystals are discussed.
Previous work on brine removal from ice crystals is reviewed
briefly. The principal emphasis in this section is on the
application of existing data on longitudinal mixing in beds
of solids to the current problem of removing brine from ice
crystals. The mechanisms of various types of mixing are
discussed, and the mathematical treatment required for the
prediction of dispersion coefficients is developed in some
detail. The data of previous investigations of longitudinal
mixing of both gases and liquids in packed beds is reviewed,
and its direct application to washing of ice crystals is made
clear.

Water is an exceedingly cheap commodity, and it seems
unlikely that relatively expensive metal heat transfer surfaces
can be employed to remove the heat of fusion of ice in practical
freezing processes for desalination. Such metal surfaces
are avoided if ice is formed by evaporation from brine at
low pressures, or if an immiscible refrigerant such as
isobutane is dispersed in the brine and allowed to evaporate.
These processes produce ice in the form of an ice-brine slurry,
from which the brine must be removed before the ice can be
melted to yield the demineralized water product.

Brine removal and ice washing can be accomplished on a centrifuge, but this operation is expensive. A more promising procedure is to displace the brine and wash the crystals by countercurrent flow of wash water and ice-brine slurry in a "wash column". The slurry rises in the column, moving against a downward flow of water supplied at the top. Salt is removed by two mechanisms: the brine is displaced by the piston-like action of the descending water, and brine adhering to the crystals is diluted by mixing with and diffusion to the descending liquid stream.

Two different situations may prevail in such a wash column. If the brine has been partially drained from the ice by gravity, the descending water will not fill all the voids in the bed of ice crystals; many of the void spaces will be filled with air. There will be little motion of ice crystals with respect to the moving bed. On the other hand, if the liquid level is held at the top of the column, the voids will be entirely filled with liquid, and the ice packing density may be anything between that of the drained bed and that of a thin slurry. Motion of ice crystals within the bed is to be expected if the ice packing density is not too great. The two situations are referred to as "drained beds" and "flooded beds".

The washing studies here reported were carried out by batch operation, that is by washing brine from a fixed bed of ice-brine mixture. This is simpler to do than countercurrent washing with continuous supply or removal of ice, and it provides the same basic information of interest.

Previous Ice Washing Studies

The Carrier Corporation has extensively studied the washing of ice-brine slurries (1, 2). A large part of their work was carried out in a vertical column operated continuously. The brine level was held at various points, so that the column was a hybrid column -- a combination of a flooded bed and drained bed. Their results indicated that the net wash water lost to the brine need not be greater than 10 percent of the ice production to obtain adequate washing of the ice. They reported that washing above the brine level is much better than below, because a larger amount of liquid adheres to the ice in the latter case.

A primary object of the Carrier studies was to assess the ability of a separation column to remove brine from ice, by getting quantitative data on temperature, pressure, and salt concentration at various points; and by determining its capacity to handle ice and wash water. All of these data were correlated by what was termed "simple extraction theory". The process was modeled by comparing it to a countercurrent extraction process with wash water as the extracting agent, brine as the original solvent, and salt as the common solute. The height of a transfer unit was determined for various combinations of operating variables.

Struthers Wells - Scientific Design also studied desalination of sea water by freezing, with particular emphasis on the production of large, uniform-size ice crystals (3).

Studies were made of the removal of excess brine from ice crystals by free-draining filtration, vacuum filtration, and by centrifugal filtration. Their results indicated that complete removal of mother liquor by various drainage techniques was impossible. According to their report, the alternative was displacement of the entrained mother liquor with fresh water; a process stated to require large and uniform crystals with low specific surface. Accordingly, their efforts were directed toward a process in which large ice crystals were made so that subsequent centrifugation would be simplified.

The Phillips Petroleum Company has developed a process for washing crystals in flooded beds (4, 5). This process was originally developed for purification of organic materials by fractional crystallization, but the technology is now being applied to the concentration of beverage and food products (6). In all of the Phillips processes, concentration is accomplished by freezing. Pure crystals are formed and subsequently washed by displacement with liquid, in turn obtained by remelting crystals formed during freezing. The technology for this process is well developed, and the process is similar in many respects to that contemplated for the washing of brine from ice crystals.

Use of Existing Data on Longitudinal Mixing in Beds

To the knowledge of the authors, no studies have been made to relate to the current problem the extensive data on longitudinal mixing in packed beds and in fluidized beds obtained in the last 10-15 years by residence-time distribution studies. As discussed earlier, data on washing brine from ice crystals have been correlated on the basis of height of a transfer unit (HTU). A primary purpose of this study is to apply the voluminous literature on longitudinal mixing of fluids in packed beds and fluidized beds to the washing of brine from ice-brine slurries. It appears that much of the diffusion theory already developed in residence-time distribution studies can be applied. Hopefully, the present studies will enable a better basic understanding of the factors involved in washing ice crystals, and will enable much of the empiracism to be eliminated in the design of wash columns.

A large number of chemical engineering operations involve the flow of a fluid through packed or unpacked vessels. Many studies have been made to determine the extent of longitudinal mixing because of the great practical importance of the problem. For example, a knowledge of the degree of mixing is important when two petroleum fractions are pumped one after another in a pipeline. A knowledge of the extent of mixing is also essential for the prediction of the degree of approach to plug flow in catalytic converters. Improved knowledge of longitudinal mixing has lead to a better basic understanding of the performance of fluidized beds. Significant progress

has been made in the last decade in the analysis and under-
standing of longitudinal mixing through residence-time
distribution studies. The purpose of this section is to
discuss theories of longitudinal mixing, and to show how this
theory can be applied to washing of ice-brine slurries.

In batch washing of ice-brine slurries with fresh water,
brine is displaced and the crystals are washed by the downward
flow of wash water. In the ideal case, a piston-like front
of descending wash water completely displaces the brine and no
mixing occurs at the wash water-brine interface. In the actual
case, some mixing does occur. This mixing is undesirable, as
it leads to a "smearing" of the water-brine interface, with
dilution and consequent waste of part of the wash water.
Much of the current study has been directed to an investigation
of the factors influencing mixing as the wash water front
moves down the column.

Types of Longitudinal Mixing -- Longitudinal mixing in a
packed bed can take place by several different mechanisms. As
pointed out by Danckwerts (7), the longitudinal velocity of any
element of fluid in a wash column relative to the imaginary
moving boundary of the water-brine "interface" will fluctuate
irregularly. At times the element will be close to a solid surface
and viscous forces will slow it down, while at other times it will
be near the center of a channel and moving at a velocity greater
than the mean; part of the time may be spent in regions of turbulence

where the element will undergo rapid and irregular fluctuations
in velocity. If the packing is quite random, without any
channeling, and is non-porous, each element of fluid will
travel at the same average or statistical velocity and will
experience fluctuations of the same magnitude and frequency.
It is clear that the ordinary random-walk theory can be
applied to such behavior, and that a probability distribution
will be followed. For this reason, longitudinal mixing of
this type can be treated like diffusion. Thus, in many cases
longitudinal mixing can be characterized by a dispersion
coefficient D, and a Fick's Law-type of expression can be
used to describe the behavior. In general, the longitudinal
dispersion coefficient is determined by eddy diffusion and the
coefficient D is not to be interpreted as bearing any relation
to the molecular diffusion coefficient. Throughout this report,
dispersion will be discussed extensively. It should be clearly
recognized that the term dispersion applies to a type of
mixing characterized by statistical variations. As will be
seen later, this is by far the most important type of mixing
that occurs in washing ice crystals.

Mixing can also occur by channeling or short circuiting.
In this type of mixing, certain parts of the fluid short
circuit sections of the bed, whereas other parts of the fluid
move slowly through relatively stagnant parts of the bed.
The portions that short circuit the main part of the bed
remain in the vessel a relatively short time -- the slow-moving

stagnant stream remains in it a long time. Thus a uniform
front does not move through the bed and longitudinal mixing
occurs. Such a deviation from piston flow is often serious
in large fluidized beds where part of the gas passes through
in large bubbles. As will be seen later, this type of mixing
is probably relatively unimportant in washing ice crystals
with downflow of wash water; however, it might be important in
upflow washing.

A third type of mixing can occur when a fluid displaces
another fluid in a bed of porous packing. Here the main body
of wash water may pass through a near piston-like flow, but
"tailing" results as the fluid in the porous packing slowly
diffuses into the wash liquid. If significant quantities of
brine are trapped within irregular ice crystals, or in clumps
of crystals clustered together, this type of mixing can be
important. As will be seen later, this type of mixing is also
probably not predominant in washing ice-brine slurries.

Mixing in Uniform Flow Through Packed Beds -- The bulk of
this section is concerned with mixing that can be characterized
by a dispersion coefficient D. It should be realized,
however, that the mathematical treatment is only as good as
the model, and that it will only apply where random-type
fluctuations apply. This behavior often applies in flow
through packed beds.

Consider a packed bed of length, L, having a void volume, V_o. The voids are filled with brine having a salt concentration c_o. A flow of pure water at a volumetric flow rate v is started at time $\theta = 0$, and the effluent concentration, c, is monitored. If no longitudinal dispersion of salt occurs, the salt concentration in the effluent will drop suddenly from c_o to 0 at the instant the water "front" reaches the outlet, that is at $\theta = V_o/v$ (or $v\theta/V_o = 1$), as shown in Figure 1a. This "piston flow" is the ideal case, since all of the wash water can be recovered with the melted ice.

Ideal piston flow (Figure 1a) never occurs, since there is always some longitudinal mixing due to viscous effects and to molecular or eddy diffusion. Figure 1b illustrates the departure from ideal piston flow caused by longitudinal mixing. Figure 1c illustrates an extreme case -- complete mixing of the column contents, so that the effluent has the same concentration as the contents of the wash vessel. Figure 1d illustrates still another possibility -- non-uniform flow with channeling in which stagnation occurs.

Danckwerts (7) gives an approximate solution to the general problem of displacement of one fluid by another in a packed bed. In the mathematical treatment presented by Danckwerts, it was assumed that the physical situation could be taken as a combination of ideal piston flow with longitudinal dispersion at the "interface" of the two fluids. He used a set of axes moving at the average velocity of the

fluid. With slight modifications and with the proper choice of boundary conditions that closely approximate the actual situation, the solution is

$$\frac{c}{c_o} = \frac{1}{2} + \frac{1}{2} \text{ erf} \left[\frac{1 - v\theta/V_o}{2\sqrt{\frac{v\theta}{V_o} \frac{D}{Lu}}} \right] \tag{1}$$

D is a "dispersion coefficient" which must be determined empirically; it will presumably depend on the viscosity, density, and velocity of the liquid, and on the size and shape of the packing; u is the velocity at which the water "front" moves through the bed, and is equal to u_o/ϵ , where u_o is the mean or superficial velocity, and ϵ the void fraction. According to Danckwerts, the approximate solution will be valid if $D/Lu \ll 1$ -- that is, provided mixing is not too effective or the tube too short.

Let Z_1 equal the expression within the brackets on the right hand side of Equation 1

$$\frac{c}{c_o} = \frac{1}{2} + \frac{1}{2} \text{ erf } Z_1 \tag{2}$$

where

$$Z_1 = \frac{1 - v\theta/V_o}{2\sqrt{\frac{v\theta}{V_o} \frac{D}{Lu}}} \tag{3}$$

Clearly Z_1 can be determined from measured values of the effluent

concentration by Equation 2, and the value of D calculated by Equation 3; thus D can be determined from data of the type shown in Figure 1.

Levenspiel and Smith ($\underline{8}$) showed that when D/Lu is very small the concentration curve, which is normal in shape at any instant, does not change appreciably in the time interval required for a small element of the fluid to pass by the recording point at the outlet. However, when D/Lu is of the order of 0.01 or greater, the concentration curve changes significantly as the outlet fluid passes the recording point, and a skewed, non-symmetrical curve results.

Examination of Equation 1 indicates that the curve of c/c_o versus $v\theta/V_o$ is uniquely determined by the value of D/Lu. With small values of D/Lu, the curves will approximate that for ideal piston flow (Figure 1a); with large values of D/Lu, there will be significant smearing at the interface and the flow pattern will cause the effluent concentration to vary with through-put as indicated by Figure 1b.

Rifai, Kaufman, and Todd ($\underline{9}$) have presented the exact solution to the displacement of one fluid by another in flow through a packed bed. Rather than use a moving set of axes, they set up the differential equation for dispersion with respect to stationary axes fixed at the column inlet. With slight modifications to fit the present case, their exact

solution was

$$\frac{c}{c_o} = \frac{1}{2} + \frac{1}{2} \text{ erf } Z_1 - \frac{1}{2} e^{Lu/D} (1 - \text{erf } Z_2) \qquad (4)$$

where Z_1 is given by Equation 3, and Z_2 is given by

$$Z_2 = \frac{1 + v\theta/V_o}{2\sqrt{\frac{v\theta}{V_o}\frac{D}{Lu}}} \qquad (5)$$

As pointed out by these investigators, the term $e^{Lu/D}$ is extremely large when the term $(1 - \text{erf } Z_2)$ is extremely small. As a matter of fact, the term Z_2 required values of the error function larger than those commonly tabulated. For large values of Z_2 the following expression is valid:

$$\text{erf } Z_2 = 1 - \frac{e^{-Z_2^2}}{Z_2\sqrt{\pi}}\left(1 - \frac{1}{2Z_2^2} + \frac{1.3}{(2Z_2)^2} - \frac{1.3.5}{(2Z_2)^3} + \cdots\right) \quad (6)$$

If all terms after the first term with the brackets are dropped, Equation 4 becomes

$$\frac{c}{c_o} = \frac{1}{2} + \text{ erf } Z_1 - \frac{e^{-Z_1^2}}{2Z_2\sqrt{\pi}} \qquad (7)$$

The approximations involved in deriving Equation 7 are such as to cause a negligible error; thus Equation 7 will be termed the "exact solution".

A. Ogata and R. B. Banks (10) have shown that the third term of Equation 4 can be neglected when D/Lu is less than 0.0075 with an error of less than five percent. Except in runs with

agitation, values of D/Lu reported in this investigation are less than 0.0075. It can be seen that when D/Lu is small, Equation 7 approaches Equation 1. Thus the third term on the right hand side of Equation 7 is a correction term to be applied to the approximate solution given by Danckwerts.

For a given flow rate and depth of packing, the dispersion coefficient D calculated directly from Equations 2 and 3 should be constant for all values of $v\theta/V_o$ if Equation 1 is obeyed. A serious disadvantage in the direct use of Equations 2 and 3 is that the method lacks precision as the value of $v\theta/V_o$ approaches unity. A second disadvantage is that the value of the dispersion coefficient is very sensitive to accurate determination of the value of the void volume V_o. Danckwerts (7) suggests that the value of D is more easily determined from the slope of the c/c_o curve at $v\theta/V_o = 1$, by using the relation

$$\left[\frac{d\left(\frac{c}{c_o}\right)}{d\left(\frac{v\theta}{V}\right)}\right]_{\frac{v\theta}{V_o}=1} = -\frac{1}{2}\sqrt{\frac{Lu}{\pi D}} \tag{8}$$

Because there is an inflection point in the curve at exactly this spot, however, this slope is very difficult to determine from the experimental curves.

A better method is to plot Z_1 versus $\dfrac{1 - v\theta/V_o}{2\sqrt{v\theta/V_o}}$. As can be seen from Equation 3, this plot should yield a straight line

passing through the origin with a slope equal to $\dfrac{1}{\sqrt{D/Lu}}$. An

important advantage of this method is that small errors in
the determination of the void volume V_o do not lead to corre-
spondingly large errors in the dispersion coefficient D. An
unexpected advantage of this method of determining the dis-
persion coefficient is that values of D will be close to those
given by Equation 4, even though the values of D/Lu exceed
0.0075 by a substantial extent. Although values of the dispersion
coefficient D determined directly by substitution in Equation 1
can be seriously in error, those determined from the slope
method are very close to the correct value.

Hold-back and Segregation -- The systems illustrated in
Figure 1 show varying degrees of "hold-back". In a system
with piston flow, there is no hold-back; that is, all elements
of the fluid spend the same time in the vessel. In a system
with hold-back, some elements of fluid spend more, others
less, than the average time in the vessel. Quantitatively,
the magnitude of the hold-back, denoted by H, can be conveni-
ently defined as the area above the curve of c/c_o between
$v\theta/V_o = 0$, and $v\theta/V_o = 1$

$$H = 1 - \int_0^1 \frac{c}{c_o}\, d\left(\frac{v\theta}{V_o}\right) \tag{9}$$

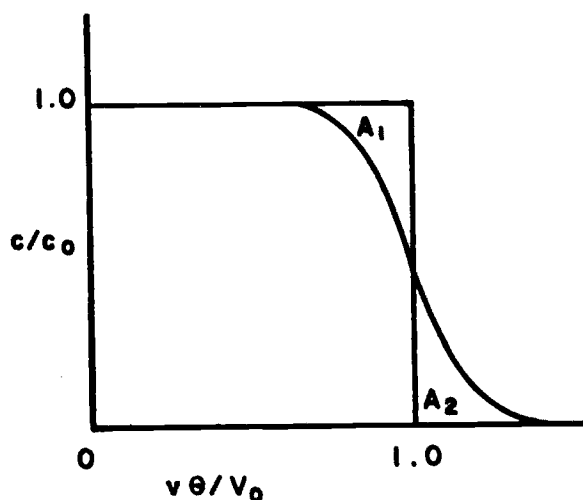This area is denoted by A_1 in Figure 2 below.

Fig. 2 : HOLD - BACK IN A
WASH COLUMN

The magnitude of H is a measure of the deviation from piston

flow.

In a completely random system obeying the mathematics

upon which Equation 7 is based, the value of H depends solely

on the value of D/Lu. Since H is a unique function of D/Lu,

it is possible to plot H versus D/Lu and obtain a single curve.

Thus an alternate method of evaluating D is to obtain H

(by measurement of Area A_1, Figure 2) and to use the known

relations between H and D/Lu. Both this method and the slope

method (involving the use of Equations 2 and 3) were used in

this study. In most cases the slope method was more reliable,

but in some cases the scatter of the data was such that the

method based on the factor H was more convenient.

The Intensity Function -- Hold-back can be due to "smearing"
at the wash-water front caused by random mixing. There is no
assurance that this type mixing will occur. It is thus of
importance to know whether random-type mixing occurs, or
whether the mixing is due to channeling or other non-uniform
flow. Naor and Shinnar (15) recently presented a method of
evaluating residence-time distribution based on the use of the
so-called intensity function I. Physically $I(\theta)$ is a measure
of the probability of escape for a particle which has stayed
in the system for a period θ. Naor and Shinnar showed that
the intensity function is:

$$I(\theta) = - \frac{d}{d\theta} \ln \frac{c(\theta)}{c_o} \qquad (10)$$

Thus $I(\theta)$ $d\theta$ is the probability that a particle in the system
for a time θ will leave during the period θ to $\theta + d\theta$. Usually
the intensity function and time are plotted in normalized form
by plotting $I/\bar{\theta}$ vs $v\theta/V_o$, where $\bar{\theta}$ is the average residence
time V_o/v. The quantity of $I/\bar{\theta}$ is readily obtained by taking
the slope of the curve of $\ln c/c_o$ vs. $v\theta/V_o$ at any point (See
Appendix I). This normalized intensity function often gives
information difficult to see on the usual plot of c/c_o vs
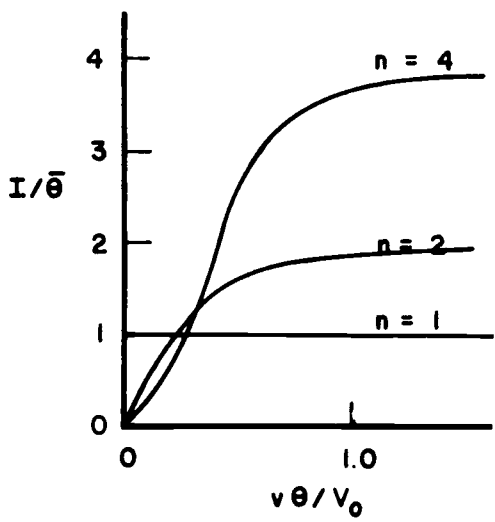$v\theta/V_o$, as can be seen in Figure 3.

The normalized intensity function for perfect piston flow
is shown in Figure 3a. At a short time θ (θ less than the
average residence time $\bar{\theta}$), the probability is $I(\theta)$ $d\theta$ that a
particle which has been in the system for a time θ will escape
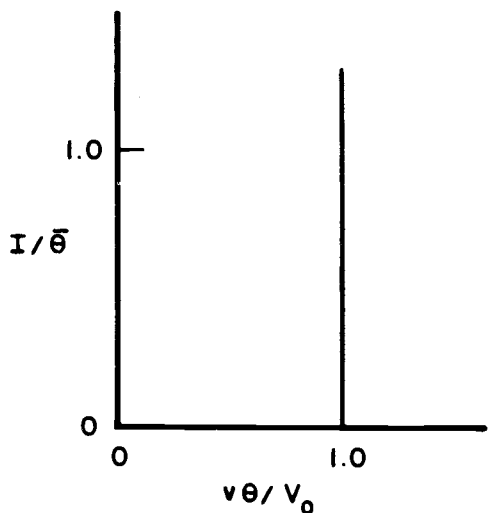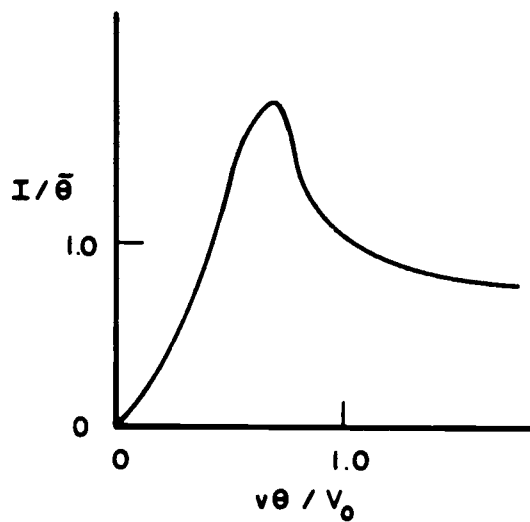
(a) PISTON FLOW
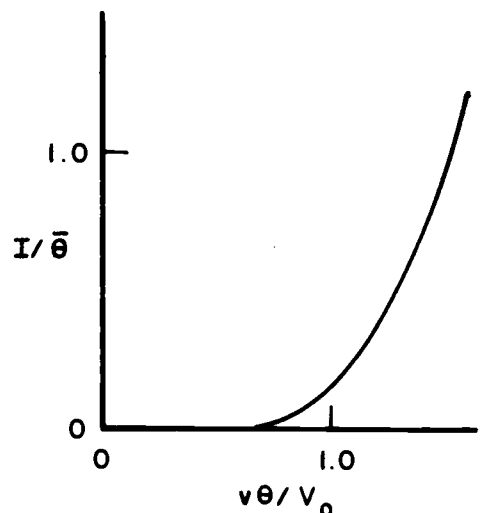
(b) PISTON FLOW WITH AXIAL MIXING

(c) n COMPLETELY MIXED VESSELS IN SERIES

(d) FLUIDIZED BED

Fig. 3: NORMALIZED INTENSITY FUNCTION

in the period dθ, and this probability is zero. The intensity function is zero for all times except a time equal to the average residence time, at which time I(θ) becomes infinite.

The intensity function for a single perfectly mixed vessel is constant (I/$\bar{\theta}$ = 1), a straight line parallel to the abscissa. The reason for this is that the chance of escape is independent of past history. The intensity function for two or more identical, ideally mixed vessels in series starts from the origin and increases to an asymptotic value (I(θ)/$\bar{\theta}$ $\rightarrow$ n) which is equal to the number of vessels.

Another typical case is that of piston flow with super-imposed dispersion of a completely random type (See Figure 3c). This case is characterized by the lack of an asymptotic value for the intensity function as θ is increased. Instead, the intensity function grows without bounds.

The intensity function is particularly useful in deter-mining whether or not stagnancy exists. Consider a system in which a considerable fraction of the fluid moves in near plug flow, the remainder being absorbed into a stagnant zone which later joins the main stream. An element of fluid which has been in the system a time slightly less than the average residence time has a high escape probability and a consequently high intensity function. Any particle which has remained in the vessel for a time exceeding the average residence time has a high chance of being in the stagnant phase and therefore a low escape probability and a low intensity function.

Figure 3d shows the intensity function for gas flow in a fluidized bed, as calculated by Naor and Shinnar from the data of Gilliland and Mason (16). Here, part of the gas apparently flows through in near piston flow as large bubbles, and part moves through as in a perfectly mixed vessel.

According to Naor and Shinnar, the use of the intensity function allows an unequivocal definition of stagnancy. A system with stagnancy is one in which the escape probability (or the intensity function) decreases in time over some intervals. If one is specifically interested in stagnancy, they recommend examination of a plot of log $\frac{c}{c_0}$ vs. $v\theta/V_0$. Any inflection point or region of decreasing slope will indicate stagnancy.

To make full use of the method, accurate data are needed. The method was used in this study to decide with assurance which of the three types of mixing actually occurs in washing brine from ice crystals.

Properties of Concentration Curves -- Curves of c/c_0 vs. $v\theta/V_0$ have several interesting properties. Referring to Figure 2, one important relation is that Area A_1 must always be the same as Area A_2. This identity is a consequence of the material balance on salt. If all the salt in the column is removed by washing, all the salt originally present in the column will be contained in the washings, that is

$$c_o V_o = \int_0^\infty c \ d(v\theta) \tag{11}$$

$$= c_o V_o \int_0^\infty \frac{c}{c_o} \ d(\frac{v\theta}{V_o}) \tag{12}$$

Thus

$$\int_0^\infty \frac{c}{c_o} \ d(\frac{v\theta}{V_o}) = 1 \tag{13}$$

Since the area of the square (perfect piston flow) in Figure 2 is unity, identity of A_1 and A_2 is a necessary consequence of Equation 13.

Although area A_1 must equal A_2 for all types of mixing, it does not follow that there must be symmetry about the line $v\theta/V_o = 1.0$. Even if Equation 1 applies, it can be seen that the term $v\theta$ in the denominator will lead to an unsymmetrical distribution.

Equation 1 indicates that the curve of c/c_o vs. $v\theta/V_o$ will cross the line $v\theta/V_o = 1.0$ at a value of $c/c_o = 0.50$, since erf (0) = 0. Actually the curve always intersects at a value of c/c_o somewhat less than 0.50, as indicated by Equation 7. The amount of this deviation is a function of the size of the term D/Lu. Thus Equation 1 always gives high values of c/c_o.

Results of Previous Investigators -- The subject of longitudinal dispersion in packed beds has received considerable study. Most investigators have correlated their data by

expressing the Peclet number (N_{Pe}) as a function of the Reynolds number (N_{Re}). The Peclet number is defined as

$$N_{Pe} = \frac{d_p u}{D} \tag{14}$$

and the Reynolds number, N_{Re}, is defined as

$$N_{Re} = \frac{d_p u \rho}{\mu} \tag{15}$$

where d_p is the particle diameter, u is the linear velocity (superficial velocity divided by void fraction) of the fluid past the particle, ρ is the density, and μ is the viscosity.

A number of studies have been made of the degree of longitudinal dispersion of gases flowing in packed beds; fewer have been made for the flow of liquids through packed beds. Very generally, it has been found that varying the flow velocity has only a slight effect on the Peclet number.

Danckwerts ([7]) studied the longitudinal mixing of water flowing through a bed 1.9 inches in diameter and 4.6 feet long that was packed with 3/8-inch raschig rings. A Peclet number of 0.49 can be calculated from these data. Kramers and Alberda ([11]), investigated the longitudinal mixing of water in a column 2.9 inches in diameter and 13.4 inches long, packed with 3/8-inch raschig rings. Their data indicate a value of 0.86 for the Peclet number for the two water velocities investigated (Reynolds numbers 85 and 155).

Ebach and White (12) obtained extensive data for liquids
in a 2-inch glass pipe filled with glass spheres, raschig
rings, berl saddles, and Intalox saddles. Peclet numbers
varied from 0.3 to 0.8 for a range of Reynolds numbers from
0.01 to 150.

Rifai, Kaufman, and Todd (9) studied longitudinal dis-
persion of sodium chloride solutions in laminar flow through
various types of sand. They concluded that the longitudinal
dispersion can be represented by a simple error function
expression (Equation 1), and that the coefficient of longi-
tudinal dispersion is directly proportional to the average
velocity of flow and independent of the length of flow. The
non-uniform particle size precluded calculation of a Peclet
number.

Cairns and Prausnitz (17) studied the longitudinal mixing
of water flowing through 2- and 4-inch tubes packed with
spheres 1.0 - 3.2 mm in diameter. They correlated all their
data in terms of a hydraulic radius that depended on both tube
diameter and particle diameter. However, if the particle
diameter is used as the characteristic dimension, their data
can be directly compared with those of other investigators.
They found that the Peclet number varied from 0.6 - 2.4 over
a range of Reynolds numbers of from 9 - 6,000. Thus over a
700-fold range in Reynolds numbers, the Peclet number varied
only by a factor of 4, again demonstrating that the Peclet
number is but a weak function of Reynolds number. Their flow

$N_{Pe} = d_p u / D$

$N_{Re} = d_p u \rho / \mu$

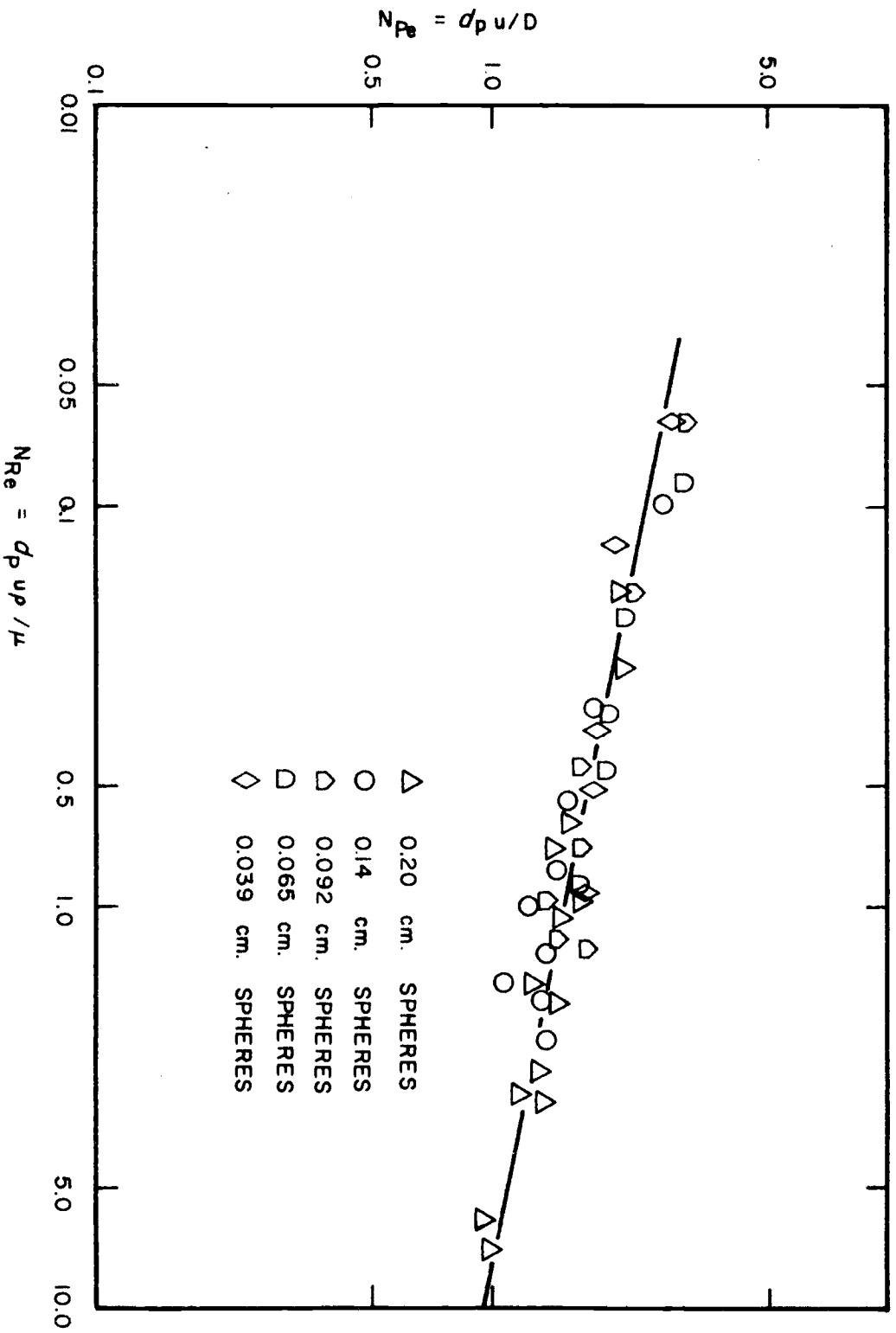| △ | 0.20 cm. | SPHERES |
○	0.14 cm.	SPHERES
◁	0.092 cm.	SPHERES
□	0.065 cm.	SPHERES
◇	0.039 cm.	SPHERES

Fig. 4 : DISPERSION OF SALT BRINE IN BEDS OF SPHERES (13)

rates were far higher than those investigated in the current study, and the results are not of direct applicability.

Harleman, Mehlhorn, and Rumer (13) made a comprehensive study of the displacement of fresh water by brine in beds of spheres from 0.039 - 0.20 cm in diameter. Their data also indicate that the Peclet number is relatively insensitive to variations in the Reynolds number; thus for a 100-fold change in Reynolds number there was only a three-fold change in the Peclet number. Values computed from their data indicate that N_{Pe} varied inversely as $(N_{Re})^{0.2}$. A plot of their data is given in Figure 4. The data of Harleman, Mehlhorn, and Rumer on washing brine from stationary beds are particularly pertinent to the current studies of the washing of ice-brine slurries because ice crystals are commonly in the size range studied by these authors, and because their flow rates were in the range investigated in the current study.

The foregoing investigations can be summarized by stating that (a) data generally can be correlated by plotting the Peclet number versus the Reynolds number; (b) the Peclet number is not a strong function of Reynolds number, being either independent of Reynolds number or varying inversely to a fractional power; (c) the Peclet number lies generally in the range 0.3 to 3.

Because of the success of previous investigators in correlating longitudinal mixing by equations based on a dispersion model, this method was used rather than methods

involving the determination of HTU. Whether or not a bed of floating ice crystals should behave in the same manner as a stationary bed of spheres was a question investigated in the studies to be described.

IV. EXPERIMENTAL PROCEDURE

Two separate investigations were carried out: one to study the washing of brine from inert glass and plastic beads; a second to study the batch washing of brine from a mass of small ice crystals. Use of the inert beads permitted an investigation of the basic factors involved without attendant complications due to freezing and melting of ice; in the second study the washing of brine from ice was investigated under typical conditions. Details regarding apparatus and experimental procedures are given in this section, both for the studies with inert beads and for the studies on washing of ice crystals. Results and their significance are given in Section V.

Washing Brine From Beds of Solid Beads

All tests were made batch-wise. A vertical column containing the beads was filled with salt water. Brine was removed at the bottom at a fixed rate, and wash water was added at the top at a rate to hold the level constant. The salt concentration at the exit was monitored during the run. In the first series of runs, polyethylene beads 0.3-0.4 cm in diameter were used. These beads had a sp. gr. less than 1.0 and floated. In the second series of runs, beads with a diameter of 0.02-0.04 cm were used. Polyethylene beads in this size range were not available and it was necessary to use "Scotchlite" glass beads. These beads had a sp. gr. greater than 1.0 and did not float.

Fig. 5   APPARATUS FOR WASHING INERT BEADS

RESERVOIRS

WASH-WATER
DISTRIBUTOR

WASH COLUMN

EFFLUENT
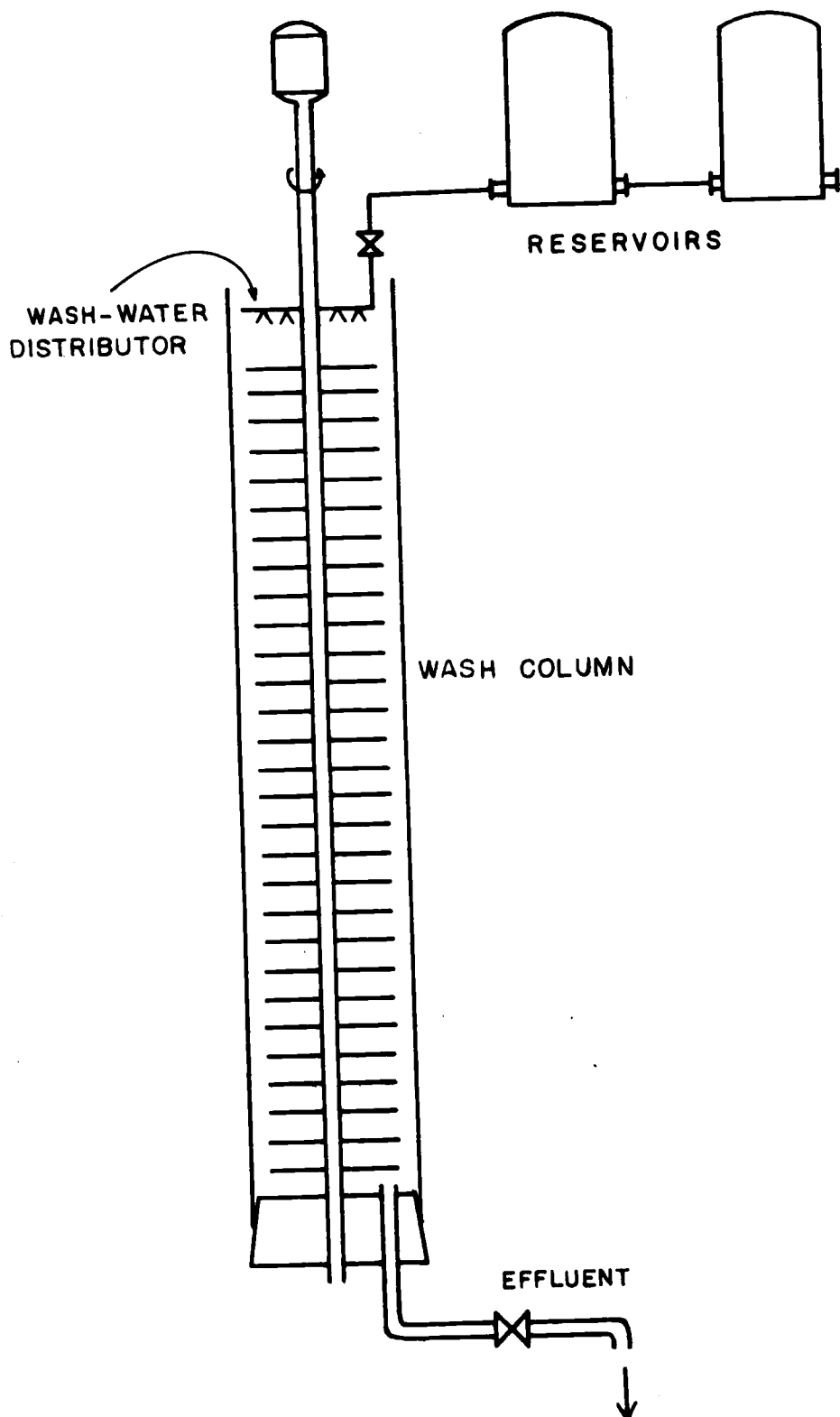
The beads were contained in a 2.5-inch ID glass column 22.5 inches high (see Figure 5). A stirrer provided a small amount of radial mixing with a minimum of axial mixing. The shaft (1/4-inch diameter rod) was supported by bearings at each end of the column. Short, straight rods were soldered perpendicular to the shaft. These extended nearly to the glass wall and were placed along the entire length of the shaft at intervals of one inch. The beads were supported by a large rubber stopper at the bottom of the column. A screen was placed over the stopper to prevent beads from dropping through the small outlet hole.

Wash water was fed from two 8-liter glass bottles in series located on a shelf 18 inches above the top of the wash column. The wash water was distributed to the top of the column through a coiled piece of 1/4-inch copper tubing drilled with a number of small holes. The wash water required (generally about one liter) was only a small fraction of the capacity of the reservoirs, so the level in the wash-water reservoirs did not change greatly during a run. The wash rate was set by adjusting the valve at the bottom of the column. The water feed to the top of the column was adjusted so that the level remained constant. Before the start of a run, the valves were adjusted to give steady-flow conditions with water being fed at the desired flow rate.

At the start of the run, water in the column was allowed to drain. A quantity of brine (approximately 600 ml)

containing 5.00 percent salt was added to bring the liquid level
up to a mark at the top of the column. The location of this
mark was such that in the first series of runs the beads were
just barely covered. The amount of brine required to bring the
level to the mark was recorded. The contents were then stirred
for one-half hour to ensure uniform salt concentration
throughout the column. A sample of salt solution was withdrawn
by a pipette shortly before the start of the run. The salt
concentration in the column was always less than that of the
brine added because of dilution with fresh water that had not
completely drained from the column. The concentration at the
start of a run was about 4.5 percent.

The run was started by opening pinch clamps at the top and
bottom of the column. The drainings were collected in a large
graduate. At intervals throughout the run, 10-ml samples
were removed at carefully noted times. The salt concentration
remained constant till a quantity of wash water equivalent to
0.7-0.9 times the void volume V_o had been used, at which time
it dropped rapidly and asymptotically approached zero. No
readings of the amount of drainings were taken until the end
of the run. Instead, the exact time at which samples were taken
was noted. The quantity of drainings at any time was computed
from a knowledge of the average wash rate and the time at
which samples were taken. Several demonstration runs were made
to prove that flow rates actually did remain constant during
the time required to complete a run. The void volume was

determined by multiplying (volume of brine added to fill the column) by the ratio (concentration of brine added ÷ brine concentration at start of washing). Most runs took from 7-20 minutes to complete.

The salt content of samples was determined by means of a conductivity cell submerged in a constant temperature bath held at 25°C. The conductivity cell was calibrated against a number of samples of known concentration. Because of improved accuracy at concentrations below one percent, most samples were diluted with distilled water before analysis.

Washing Brine From Ice Crystals

An ice-brine slurry was made before each run. This slurry was prepared in a large glass vessel by passing liquid isobutane under the surface of the brine. The slurry was then transferred to a vertical wash column, the operation of which was similar to that used for washing inert beads. Wash water was fed to the top of the column and drainings were removed from the bottom. Salt concentration was monitored during the run.

Preparation of Ice-Brine Slurries -- The ice-brine slurry was made in an insulated glass vessel 6 inches in diameter and 24 inches high. The vessel was initially filled with a 3-percent solution of sodium chloride that had previously been cooled to 40°F. Liquid isobutane (technical grade, 95 percent isobutane) was fed near the bottom of the vessel through a
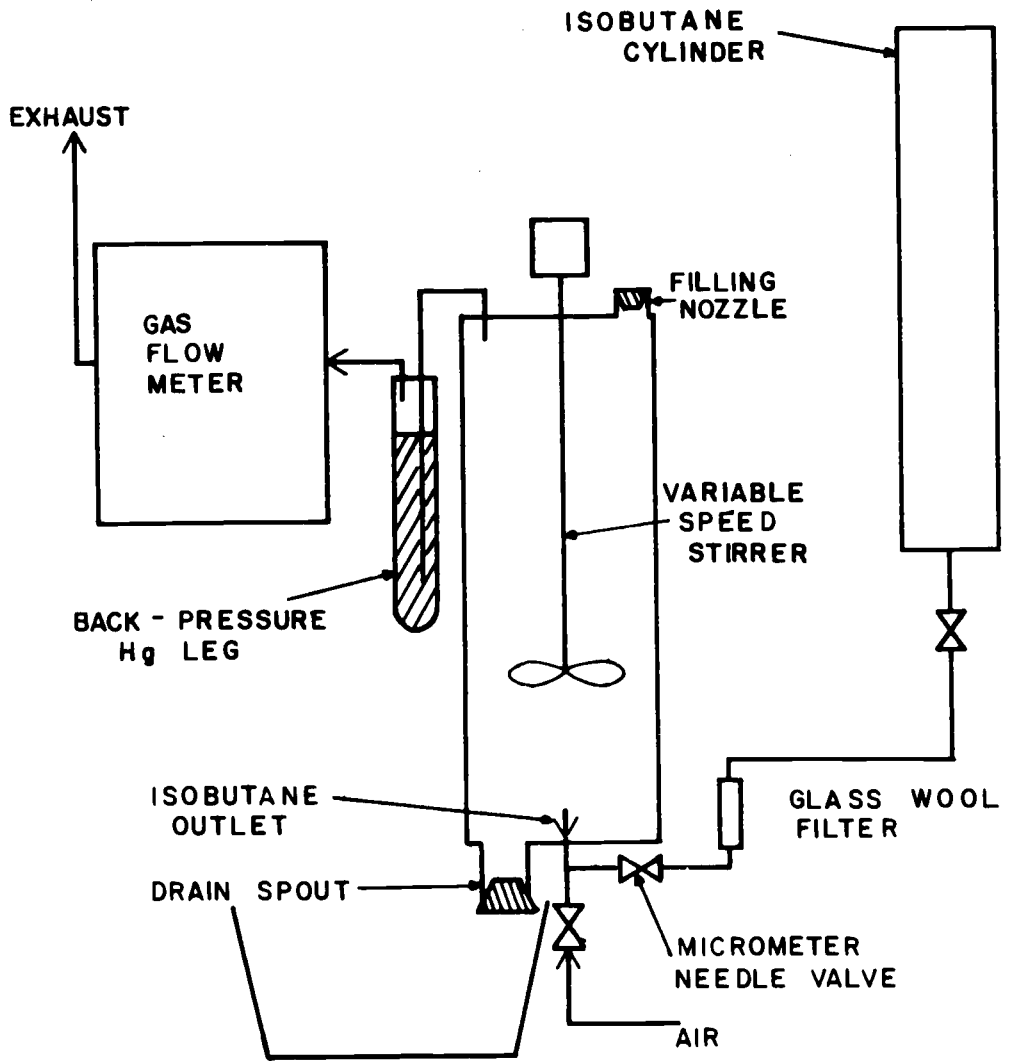
Fig. 6 :   ICE - MAKING EQUIPMENT

piece of rubber tubing perforated with a number of small holes. The contents of the vessel were stirred vigorously. A diagram of the ice-making equipment is shown in Figure 6. Ice began to form almost immediately in the insulated vessel. After about 90 minutes, about 2.5 kg of ice had formed. At this time, the vessel was approximately 3/4 full of ice, and the brine concentration had increased to about 4 percent. The flow of isobutane was discontinued, and air was sparged through the slurry for 15 minutes to remove dissolved hydrocarbons. The latter step was necessary to prevent foaming during ice washing operations. The ice-brine slurry was then removed and charged to the ice-washing equipment.

The equipment for preparing the ice-brine slurry was initially operated at atmospheric pressure. Some ice formed as separate crystals but most formed as a large ball at the bottom of the vessel. Probably the reason for this behavior was that too large a temperature difference existed between the boiling isobutane and the brine, since isobutane has an atmospheric boiling point of -10°C and a 3-percent salt solution freezes at about -2°C. A back-pressure regulator was subsequently installed to hold a positive pressure of 7 inches of mercury upon the equipment. This increase in pressure lowered the temperature differential from 8°C to 3°C. After this change, all the ice was formed as discrete particles and ball formation ceased.

<u>Ice-Washing</u> -- The ice-brine slurry was washed in a
stainless steel column 4 inches in diameter and 30 inches
high. The column was provided with a removable variable speed
stirrer similar in design to that used in the experiments with
inert beads. The stirrer was installed so that it oscillated
at a fixed speed through a 100° arc. The wash water was fed
with a variable-speed positive-displacement pump through a
distributor at the top of the column. The wash water reservoir
was calibrated to àllow measurement of the volume of wash
water. The entire assembly was located in a refrigerated
room. A diagram of the apparatus is given in Figure 7.
Provision was also made to permit pulsing of the entire column.
(Pulsing equipment is not shown.) Pulsing was accomplished
by alternately inflating and deflating a small rubber balloon
which was placed in the bottom of the wash column. As the
balloon expanded, the entire contents of the column moved
upwards. The alternate inflation and deflation was accomplished
by squeezing a polyethylene bottle, which was connected to the
balloon by tubing.

At the start of a run, a weighed quantity of ice-brine
slush was charged to the wash column. A sample of brine was
removed with a pipette. Wash water was fed to the distributor
at the top of the wash column. Brine was drained at such a
rate that the level in the wash column was held constant. The
drainings were collected in a large graduate. At intervals
throughout the run, 10 ml samples were removed at carefully
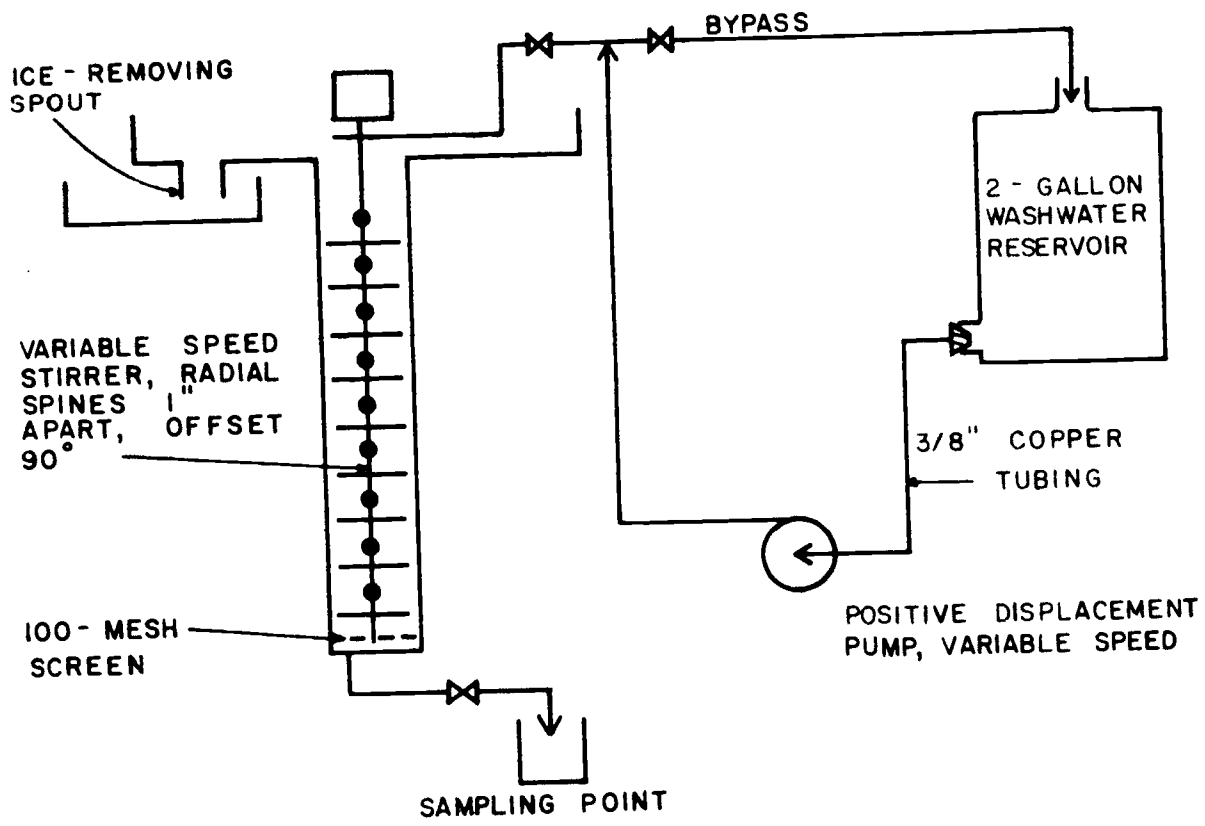
Fig. 7 :   ICE - WASHING COLUMN

noted times. The salt content of the samples was determined by analysis in a conductive cell. Experimental runs required from 20-90 minutes to complete.

The relative amounts of ice and brine in a charge were calculated by a material balance. The unknown quantity of brine times its salinity was equated to the total quantity of wash water at the end of the run times its salinity. In this calculation, it was assumed that the ice contained no salt.

The net wash water was determined in two ways. The wash water could be computed by deducting the weight of slurry charged from the total weight of drainings and washed slurry recovered at the end of the run. This value checked very closely the value obtained by reading the marks at the beginning and end of the run on the calibrated feed bottle.

Unfortunately the liquid level changed significantly during many of the runs. It was assumed that no ice melted or formed during a run. The final quantity of brine was calculated by deducting the initial quantity of ice from the total weight of slush at the end of the run.

Because of the change in liquid level in the wash column, the net quantity of wash water actually used was not truly significant. A corrected quantity was used in correlating the data. The quantity actually used was adjusted to take into account the differences between the initial and final liquid levels. Thus when the final liquid level was lower than the

initial liquid level, the corrected wash water was greater than the amount actually used. It was assumed that the liquid level changed linearly with time.

V. EXPERIMENTAL RESULTS
AND
DISCUSSION OF RESULTS

Results of all experimental runs are presented and discussed in this section. The experiments on washing brine from inert beads are described first. These results are then discussed and interpreted in terms of the dispersion theory presented in Section III. The results of experiments on washing brine from ice crystals are given next. The interpretation of these results follows. Although the results of any one of the ice-washing runs correlated well with dispersion theory, it was difficult to analyze the effect of operating variables by comparing different runs. This difficulty was probably caused by variations in the size of the ice crystals from one run to another. For this reason, a separate section is included in which the operation of the column under ideal conditions is predicted. These results are presented in Section VI. Runs with inert beads are prefixed with the letter B (e.g. Run B-10); runs with ice crystals are prefixed with the letter I.

EXPERIMENTAL STUDIES - WASHING OF INERT BEADS

The principal purpose of these studies was to determine the effect of agitator speed, wash rate, and particle size under ideal conditions not complicated by freezing and melting of ice. A total of 16 runs was made -- ten with floating polyethylene beads, and six with "Scotchlite" glass beads. Of the 16 runs, all but three were made with agitation.

Table 1

Washing of Inert Beads

Run	Type Beads	Stirrer Speed rpm	Wash Rate 16/(hr ft²)	$\frac{c}{c_o}d(v\theta/V_o)$	Hold-back, H.	D/Lu	N_{Re}	N_{Pe}
B-3	P.E.	110	174	1.13	0.112	0.0484	2.68	0.135
B-4	P.E.	110	543	1.01	0.075	0.0200	8.37	0.326
B-6	P.E.	110	1460	0.90	0.044	0.0118	22.50	0.553
B-8	P.E.	0	354	0.97	0.033	0.0050	5.46	1.32
B-9	P.E.	32	355	0.99	0.042	0.0082	5.47	0.796
B-10	P.E.	59	374	0.98	0.053	0.0162	5.77	0.404
B-11	P.E.	90	375	0.96	0.052	0.0125	5.78	0.522
B-12	P.E.	6	380	0.99	0.034	0.0050	5.86	1.30
B-13	P.E.	120	382	1.04	0.083	0.0317	5.89	0.206
B-14	P.E.	6	1510	0.90	0.040	0.0135	23.50	0.484
B-21	Scotch.	60	454	1.00	0.081	0.0185	0.64	0.0311
B-22	Scotch.	60	204	0.99	0.093	0.0185	0.29	0.0311
B-28	Scotch.	30	439	1.02	0.036	0.0040	0.62	0.144
B-29	Scotch.	90	430	1.04	0.024	0.0020	0.61	0.288
B-30	Scotch.	0	423	1.03	0.022	0.0017	0.60	0.339
B-31	Scotch.	0	856	1.02	0.047	0.0090	1.21	0.064

The results demonstrated that a close approach to "piston flow" of the wash water could be accomplished in displacing the brine from the bed of beads. The salt concentration in the effluent remained the same as that originally in the bed until 70 to 90 percent of the original volume had been displaced. The concentration then dropped rapidly and was nearly zero by the time 15-40 percent more than the original brine volume had been displaced. The degree of approach to piston flow depended strongly on the conditions of the test.

Results With Floating Polyethylene Beads

A series of runs was made at the same flow rate but with different degrees of agitation. A number of runs were made at constant agitation and varying flow rate. Only one run was made with no agitation. Figures 8-11 show the results of four typical runs. Data for all runs are given in Table 1.

Material Balances -- Material balances in the runs were only fair. As noted earlier, the value of $\int_0^\infty \frac{c}{c_o} d(\frac{v\theta}{V_o})$ must be unity, thus the area above the curve to the left of the line $\frac{v\theta}{V_o} = 1$ must be exactly equal to the area below the curve at the right of this same line. A consequence of this is that the curve should cross the line $\frac{v\theta}{V_o} = 1$ at a value of c/c_o very close to 0.5 except under conditions of relatively high longitudinal mixing. In general, the curves did not cross at a value of 0.5, and the value of the integral varied from 0.90-1.13, with an average deviation from unity of 6 percent.
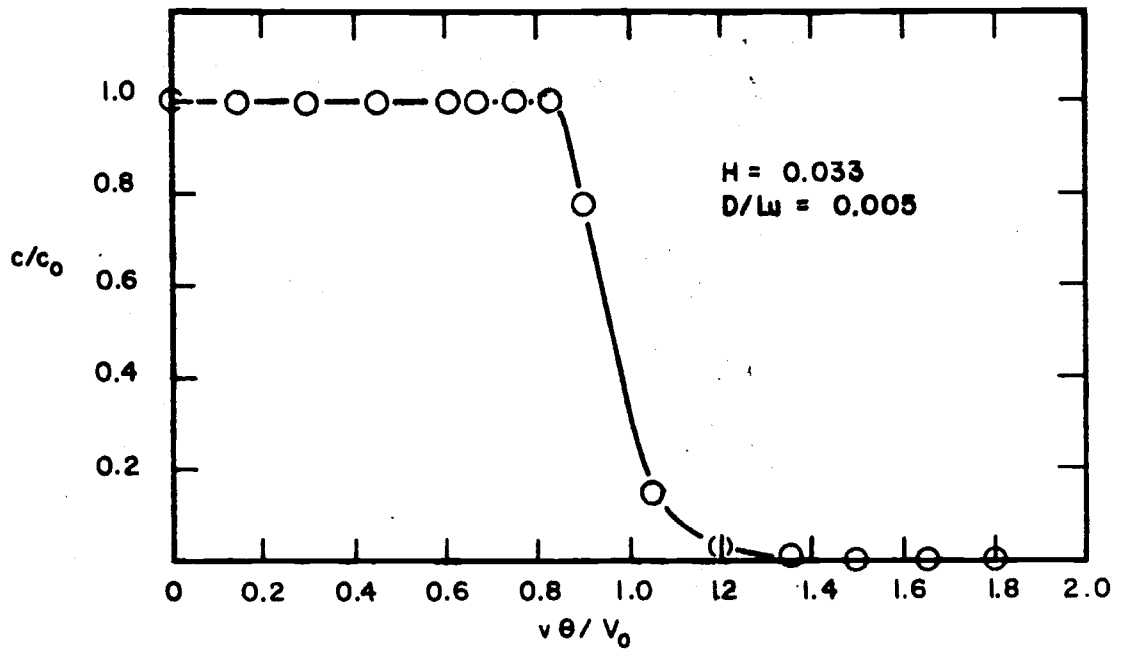
Fig. 8 : RUN B-8 POLYETHYLENE BEADS
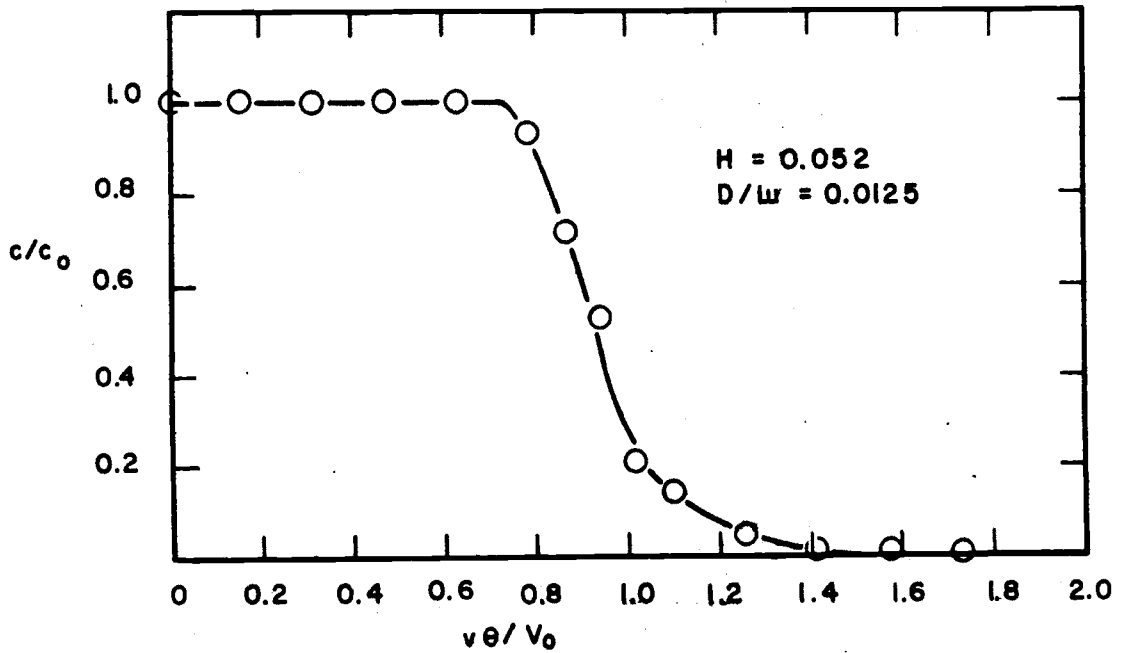
Fig. 9 : RUN B-11 POLYETHYLENE BEADS
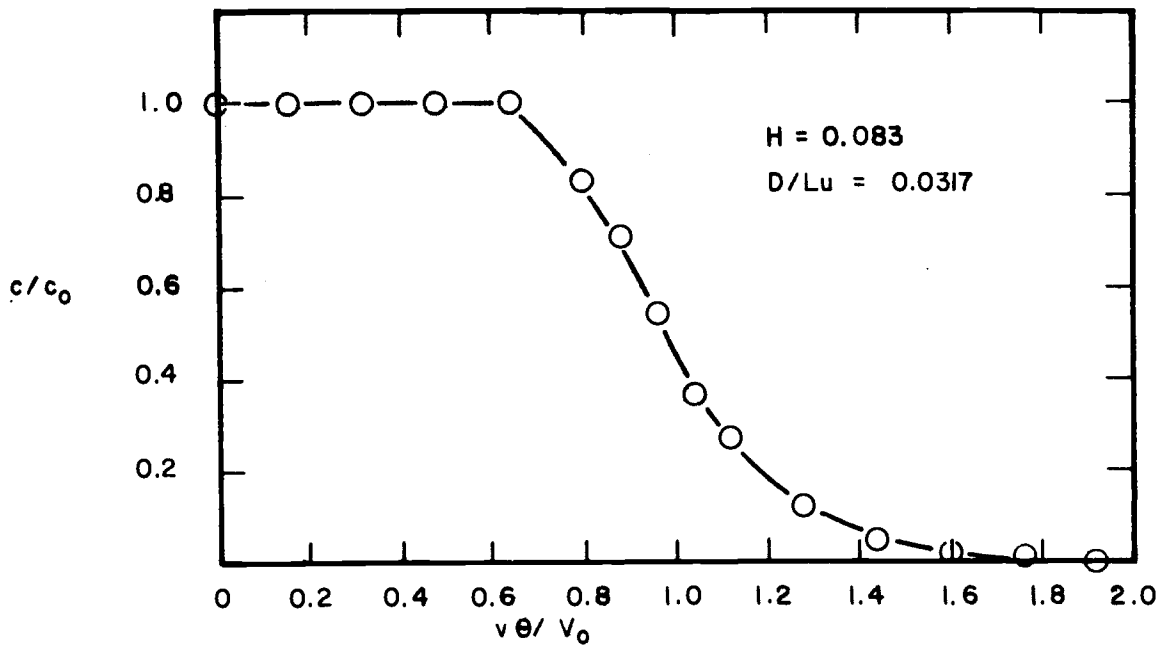
Fig. 10 : RUN B⁻13 - POLYETHYLENE BEADS
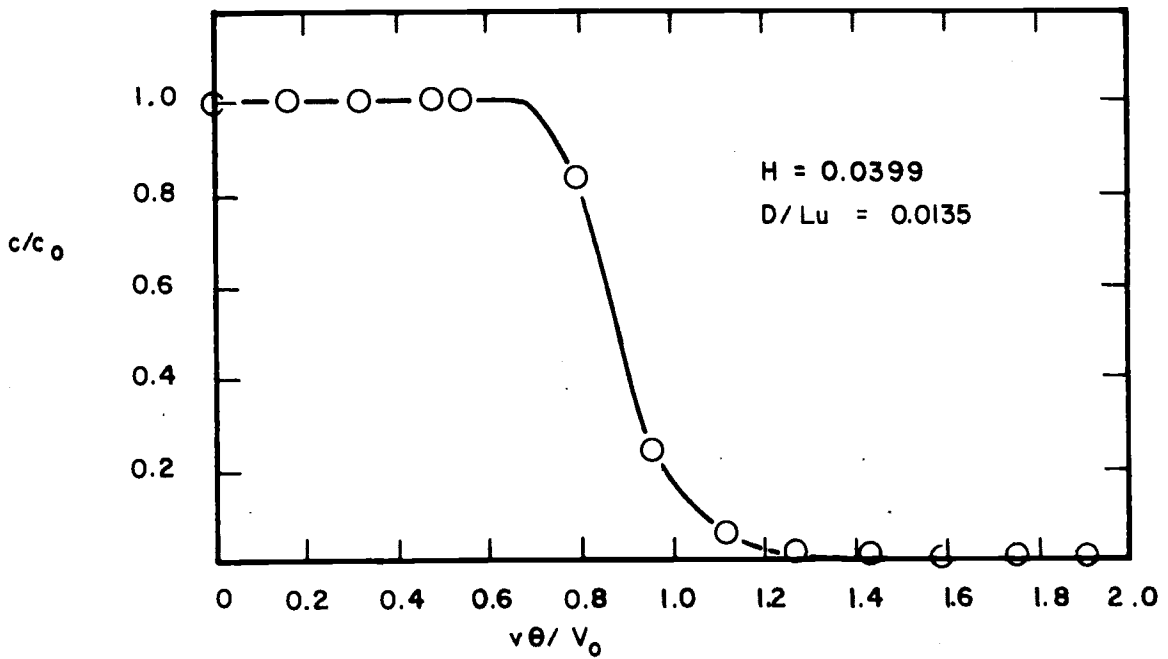
Fig. 11 : RUN B-14 POLYETHYLENE BEADS
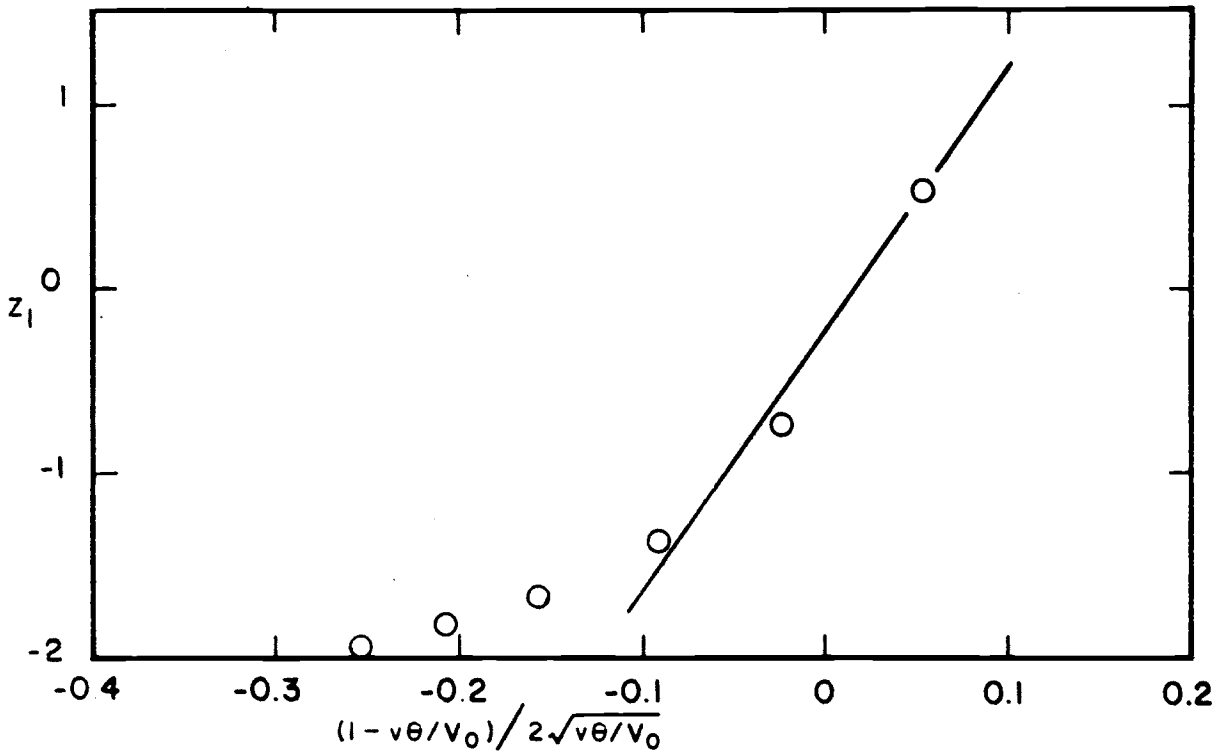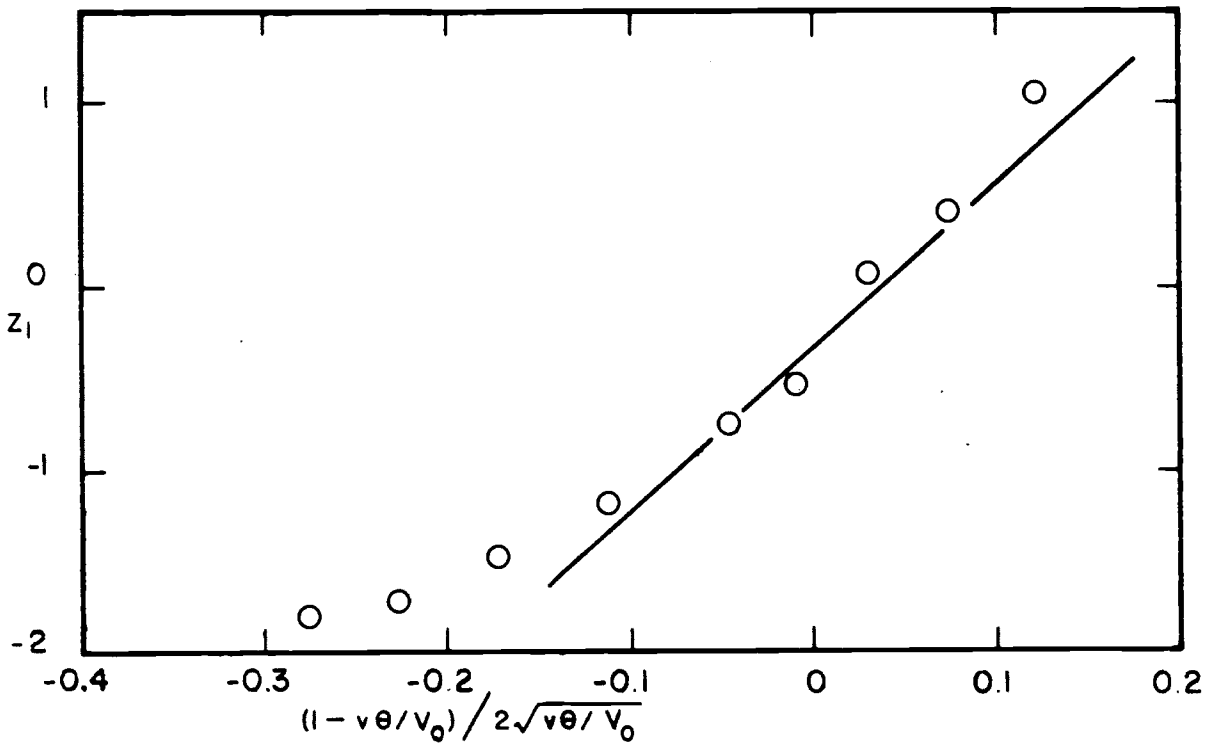
Fig. 12 : RUN B-8 POLYETHYLENE BEADS

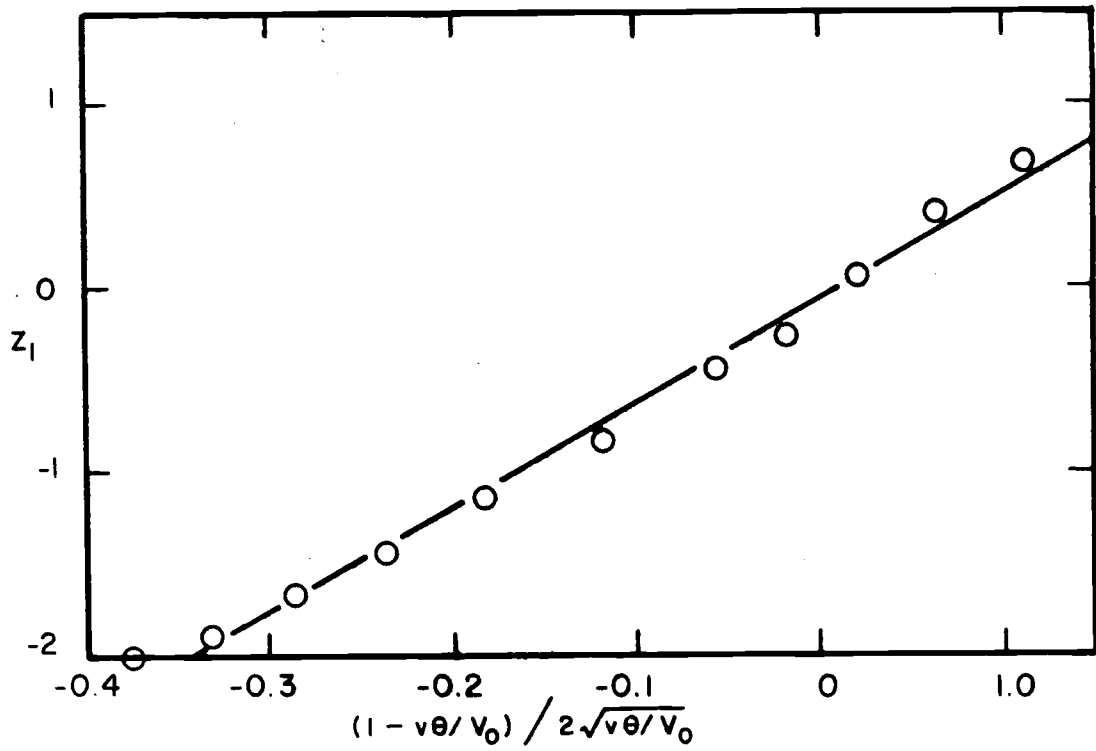Fig. 13 : RUN B-11 POLYETHYLENE BEADS
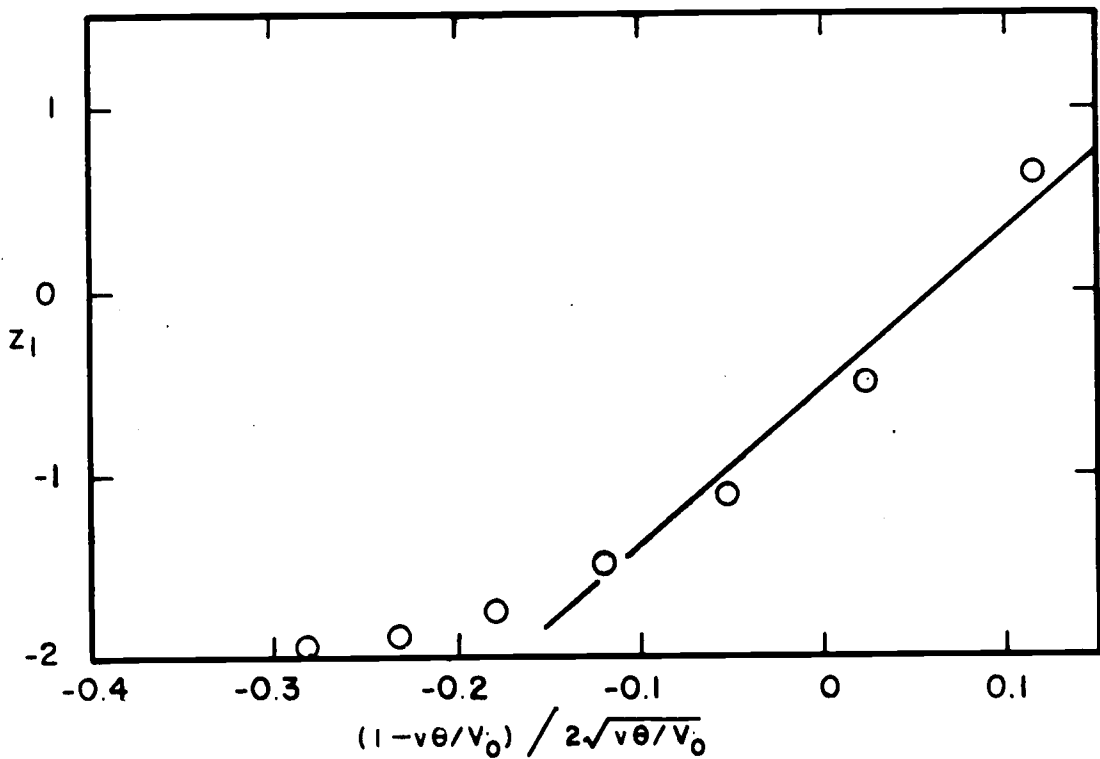
Fig. 14 : RUN B-13 POLYETHYLENE BEADS
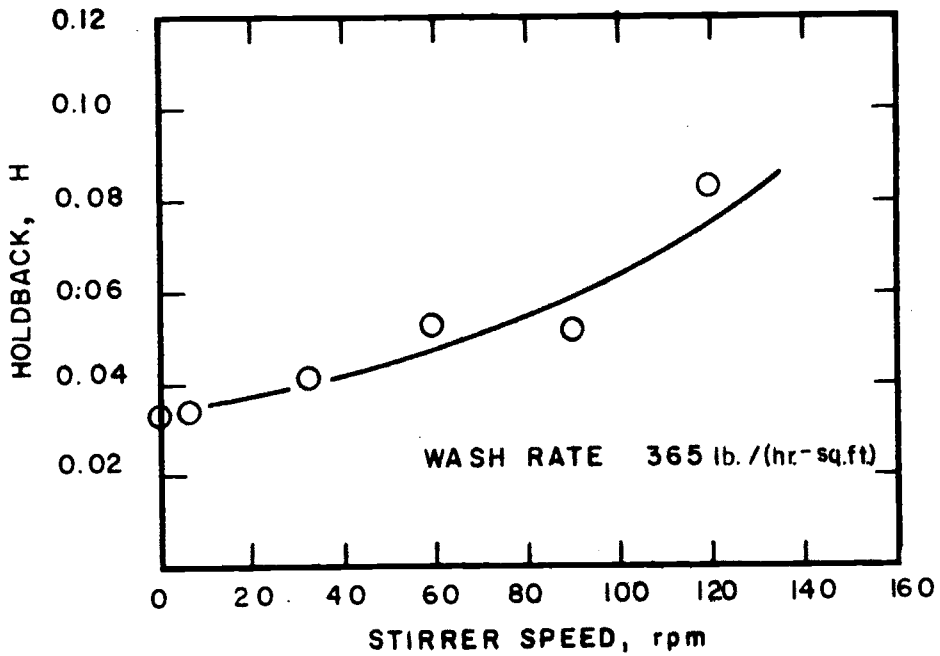

Fig. 15 : RUN B-14 POLYETHYLENE BEADS

Fig. 16 : POLYETHYLENE BEADS
CONSTANT FLOW RATE
VARIABLE STIRRER SPEED
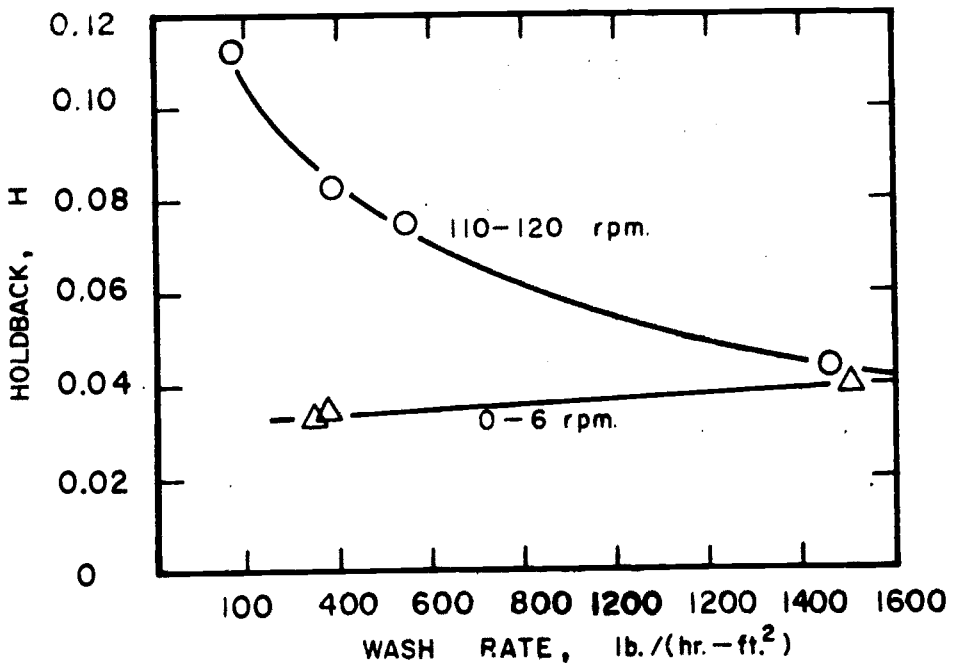

Fig. 17 : POLYETHYLENE BEADS
CONSTANT STIRRER SPEED
VARIABLE FLOW RATE

Criterion for Degree of Longitudinal Mixing -- In order
to determine the dispersion coefficient D, Z_1 (calculated by
means of Equation 2) was plotted versus $\dfrac{1 - v\theta/V_o}{2\sqrt{v\theta/V_o}}$, as
shown in Figures 12-15 for the four typical runs. In drawing
the best straight lines through the points, little weight was
given to points with values of c/c_o close to unity, since, in
this range, small errors in measuring the concentration lead
to large errors in the quantity Z_1. Points near $c/c_o = 0$
were given little weight because of inaccuracies in measuring
very small concentrations. As can be seen, only in the case
of Run B-13 was it possible to draw a good straight line through
the intermediate points. Thus the dispersion model did not
appear to fit most of the data on agitated polyethylene beads.

Although the value of D/Lu was difficult to determine by
the use of Equation 3, the hold-back H was comparatively easy
to determine from Equation 9. The value of H is recorded in
Table 1, and values of both H and D/Lu (as calculated from
the slopes of the lines shown on Figures 12-15) are shown in
Figures 8-11. It can be seen that a high value of H corre-
sponds to a high value of D/Lu and a highly skewed curve of
c/c_o vs. $v\theta/V_o$. Because this dispersion model did not well
represent most of the data on floating beads, the hold-back
H was used as a criterion of longitudinal mixing in such beds.

Effect of Changes in Agitation and Flow Rate -- Six
different runs were made at the same wash rate (~ 365 lb/
(hr sq ft)) and with different degrees of agitation

(Runs B-8,-9,-10,-11,-12, and -13). As can be seen from
Figure 16, agitation tends to increase longitudinal mixing,
as indicated by progressive increases in the hold-back as the
agitator speed was increased.

A number of runs were made at constant agitation and
varying flow rate. The effect of flow rate evidently depended
on the degree of agitation, as can be seen from Figure 17.
Thus in the four runs made at 110-120 rpm (B-3,-4,-6, and
-13), the hold-back decreased as the flow rate increased.
The opposite effect was noted in the three runs made at 0-6 rpm
(B-8,-12, and-14), as the hold-back increased slightly with
flow rate.

Results with "Scotchlite" Beads

As will be recalled, the "Scotchlite" beads were smaller
than the polyethylene beads, and had a specific gravity
greater than unity, thus they formed a bed at the bottom of
the column, and did not float. As with the floating poly-
ethylene beads, a series of runs was made at the same flow
rate and with different degrees of agitation. A number of
runs were also made at constant agitation and varying flow
rate. Two runs were made without agitation.

Figures 18-21 show typical results obtained in four runs
with the "Scotchlite" beads. Pertinent data for all runs are
given in Table 1. The runs with "Scotchlite" beads were made
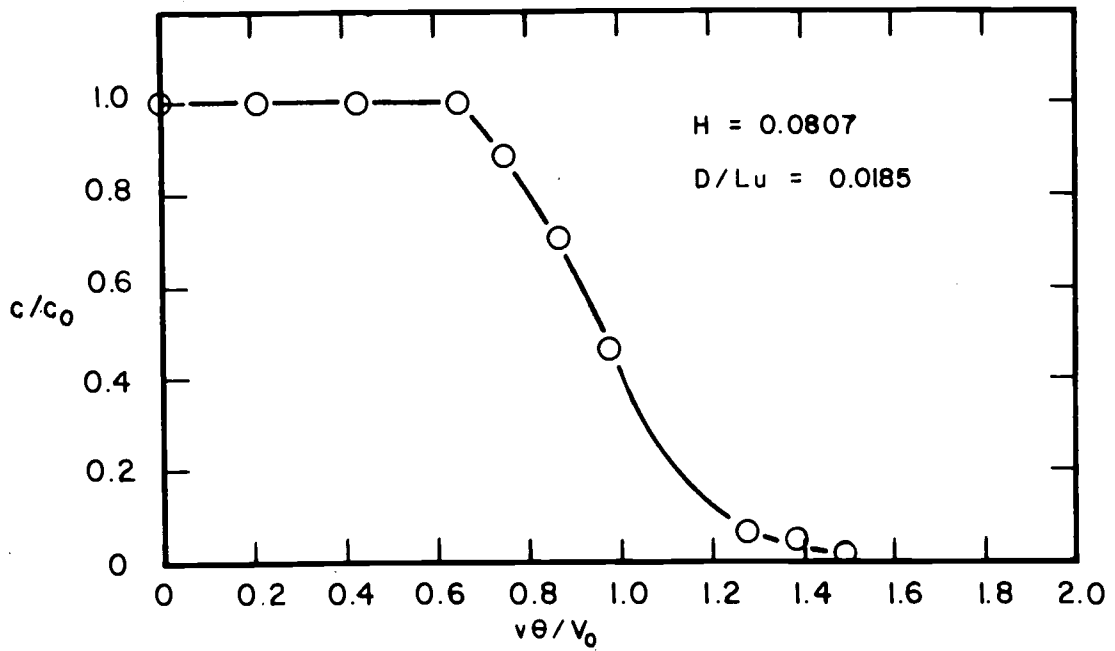after the completion of the runs with the floating beads and

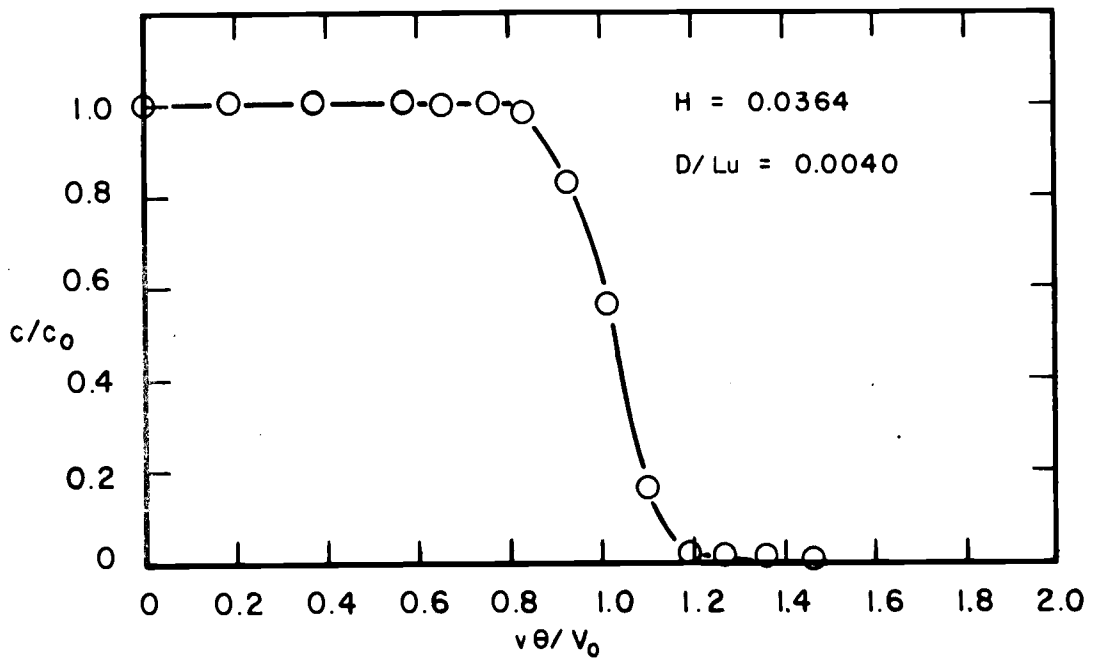Fig. 18 :   RUN   B-21   "SCOTCHLITE"   BEADS

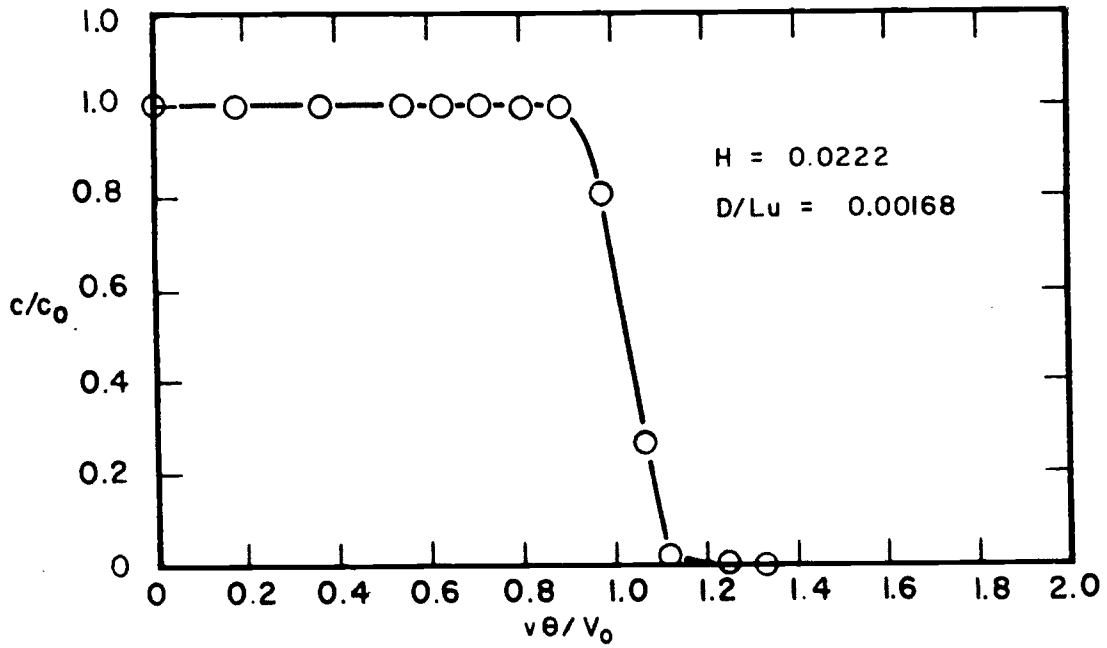Fig. 19:   RUN   B—28   "SCOTCHLITE"   BEADS

Fig. 20 : RUN B-30 "SCOTCHLITE" BEADS
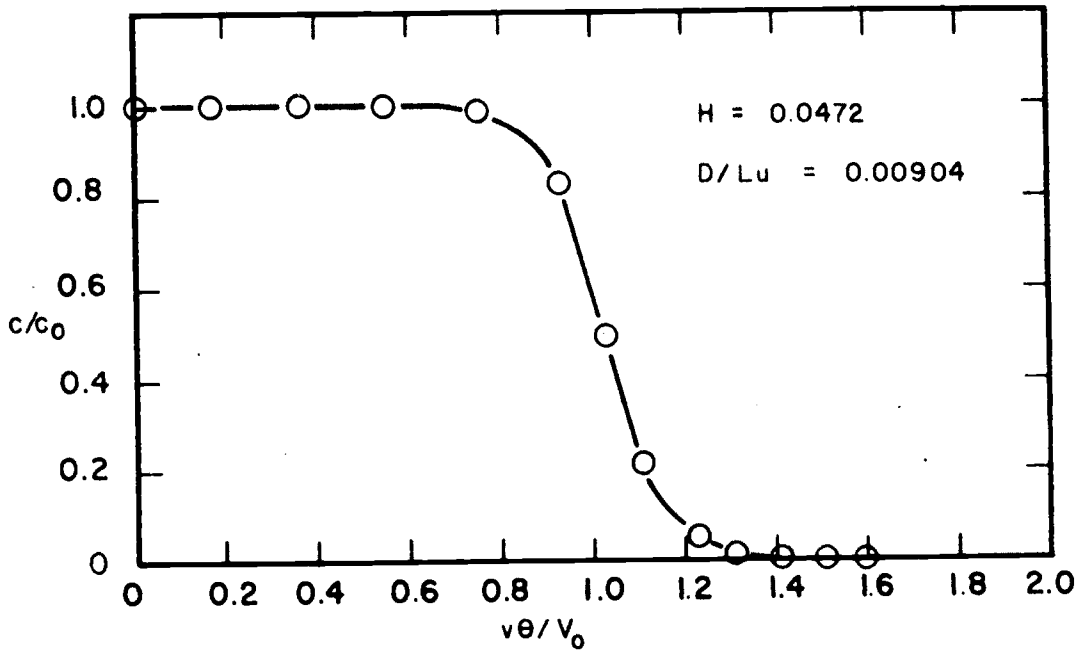

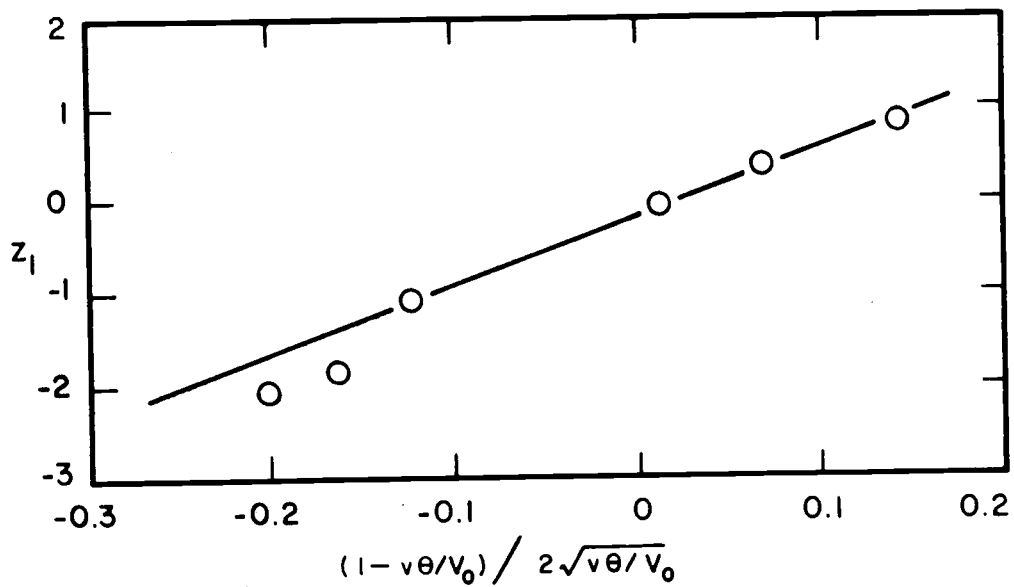Fig. 21 : RUN B-31 "SCOTCHLITE" BEADS

Fig. 22 : RUN B-21 " SCOTCHLITE " BEADS
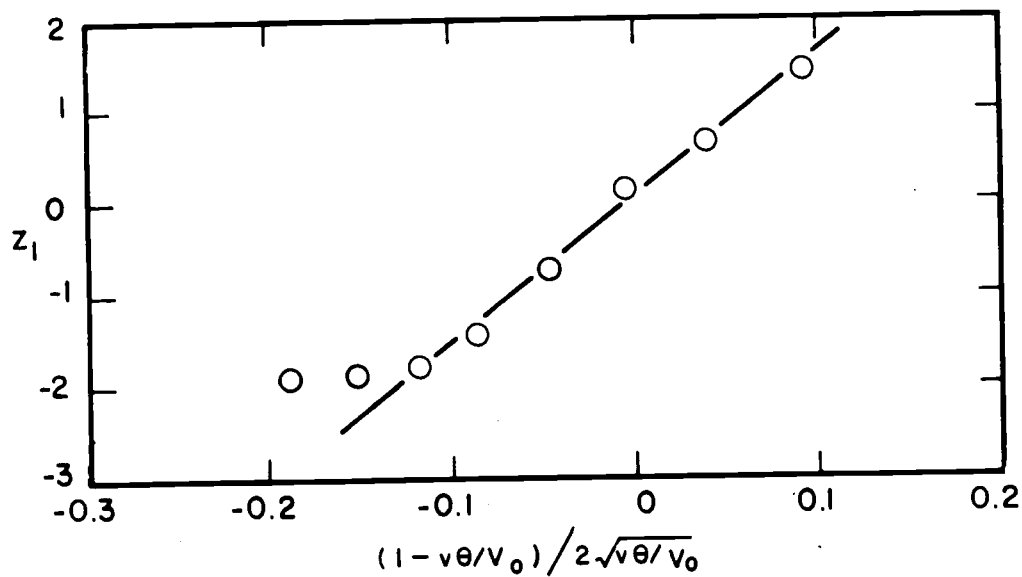

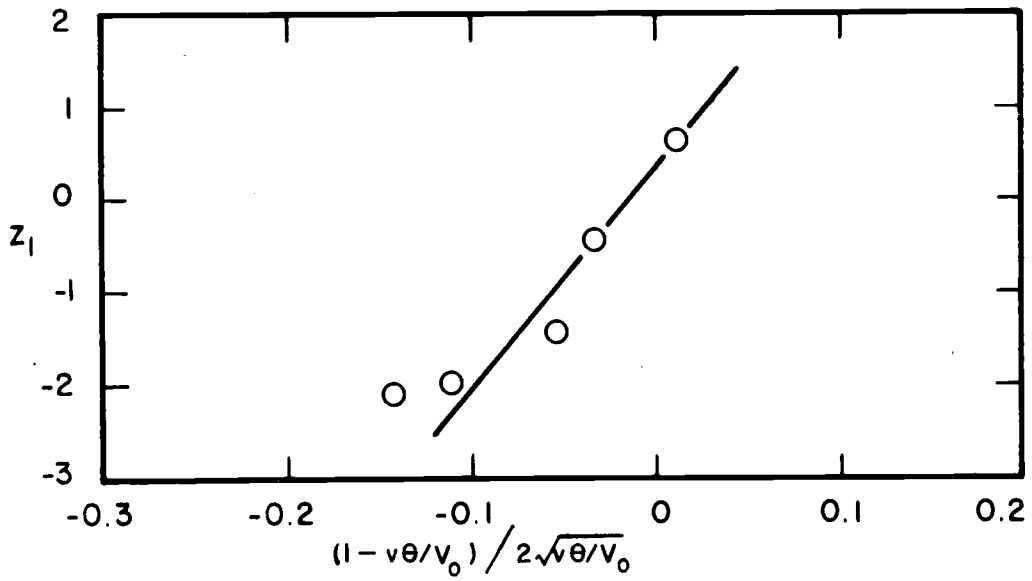Fig. 23 : RUN B—28 " SCOTCHLITE " BEADS

Fig. 24:   RUN   B—30   "SCOTCHLITE"   BEADS
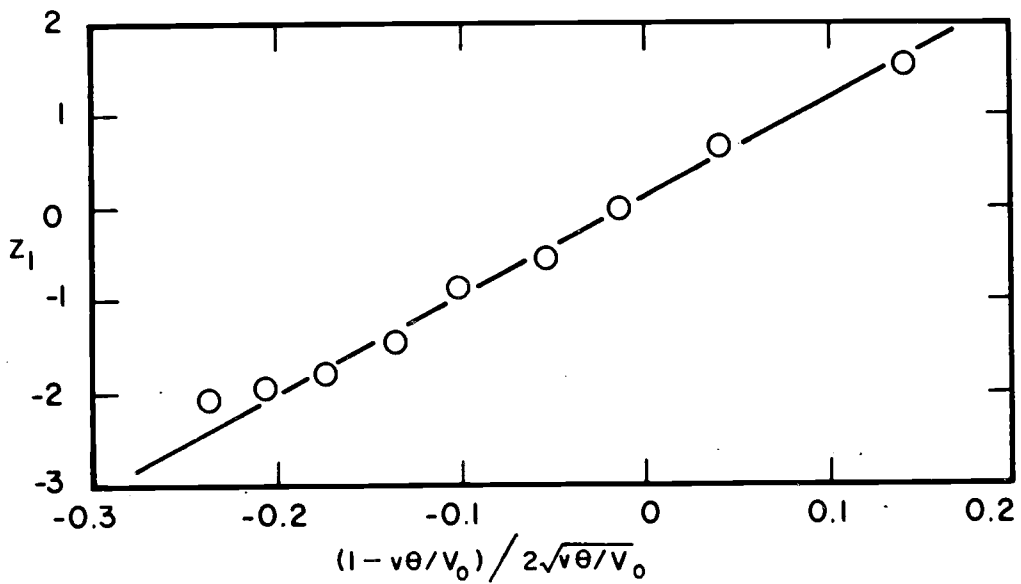

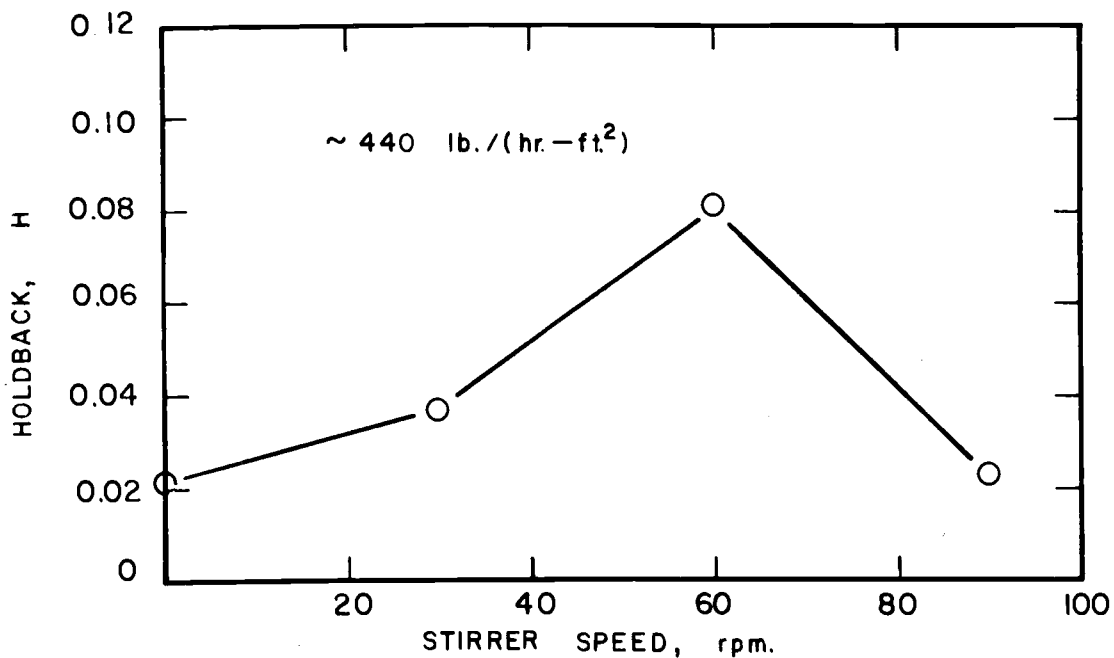Fig. 25:   RUN   B—31   "SCOTCHLITE"   BEADS

Fig. 26 : "SCOTCHLITE" BEADS, CONSTANT FLOW RATE,
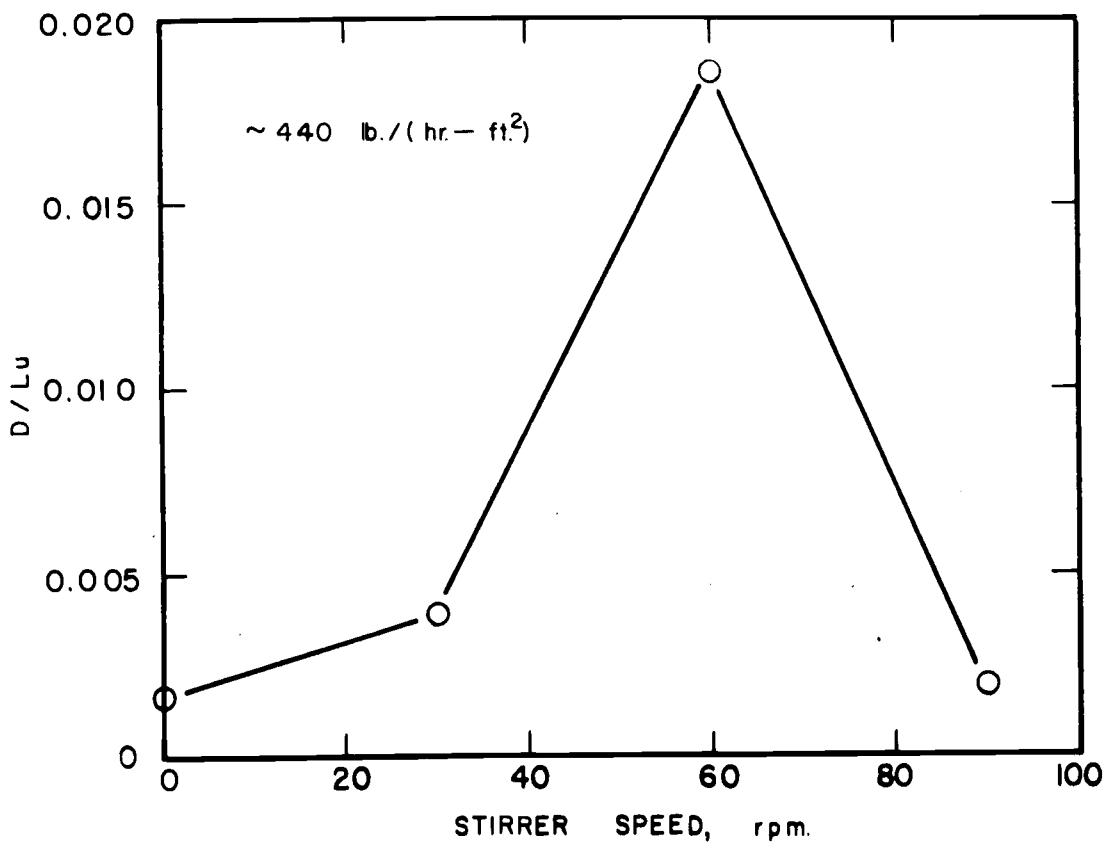VARIABLE SPEED

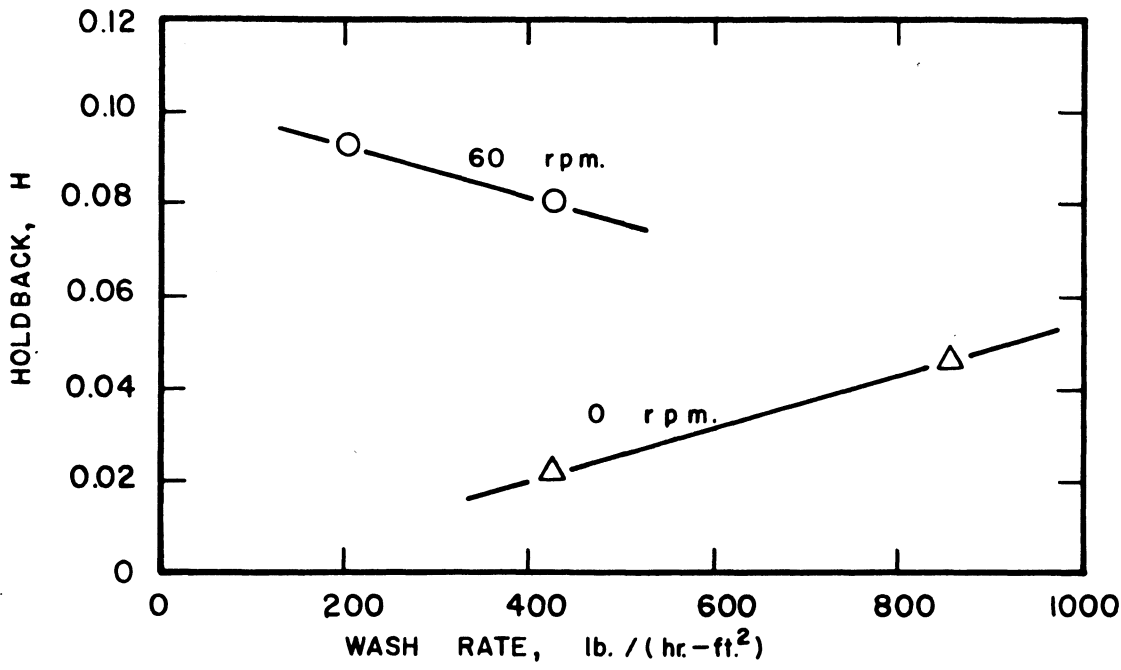Fig. 27: "SCOTCHLITE" BEADS, CONSTANT FLOW RATE,
VARIABLE SPEED

Fig. 28 :   "SCOTCHLITE"   BEADS
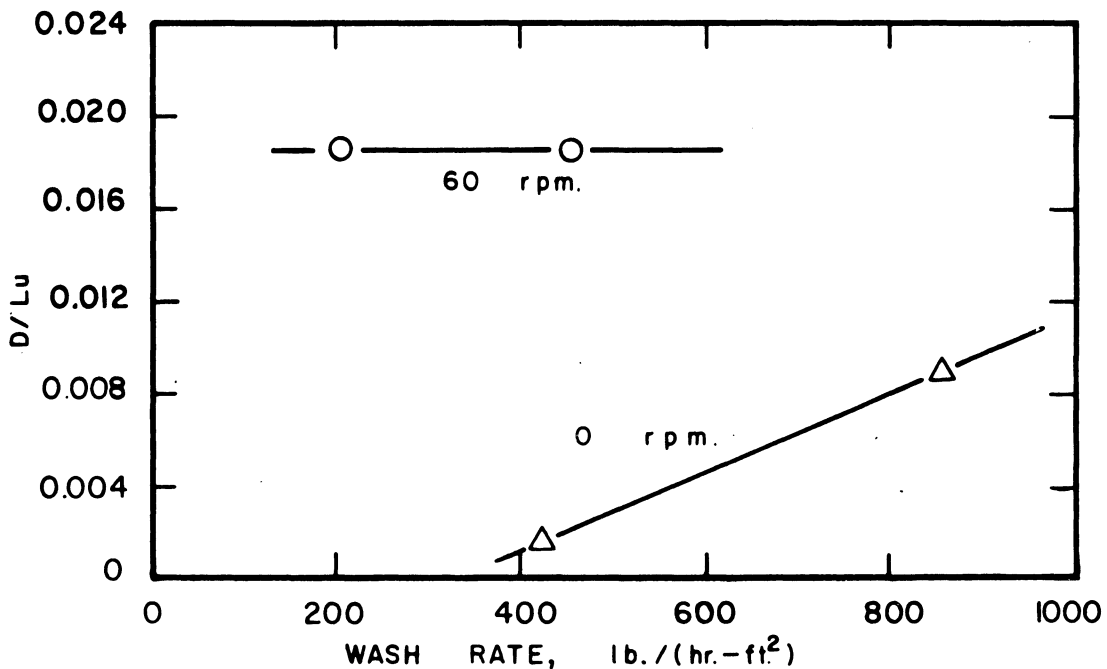CONSTANT   STIRRER   SPEED
VARIABLE   FLOW   RATE

Fig. 29 :   "SCOTCHLITE"   BEADS
CONSTANT   STIRRER   SPEED
VARIABLE   FLOW   RATE

experimental techniques had improved somewhat, as evidenced
by the better material balances. The value of the integral
(see Equation 13) varied from 0.9-1.04, with an average
deviation of two percent.

Criterion for Degree of Longitudinal Mixing -- An attempt
was made to determine the dispersion coefficient D from the
slopes of graphs of Z_1 vs. $\dfrac{1 - v\theta/V_o}{2\sqrt{v\theta/V_o}}$, as shown in Figures 22-25.
Although there is still some question as to whether the dis-
persion model is entirely applicable, it was possible to draw
straight lines through the data points for most of the runs.
Because the dispersion model did fit most of the data
moderately well, the effect of varying operating conditions
was shown in terms of the effect on D/Lu as well as on the
hold-back, H.

Effect of Changes in Agitation and Flow Rate -- With
"Scotchlite" beads, the effects of changing operating conditions
were not clear-cut, as was the case with polyethylene beads.
Figures 26 and 27 show results of varying the degree of
agitation while maintaining constant wash rate. (Runs B-21,
-28,-29, and -30). Both the hold-back and D/Lu increased as
stirrer speed was increased, reached a maximum at 90 rpm, and
decreased to a value almost the same as with no agitation as
the agitator speed was increased to 120 rpm. Figures 28 and 29
show the results of varying the flow rate at constant agitator

speed. Although insufficient data are available to characterize
the trends, it appears that in the absence of agitation, both
hold-back and D/Lu increase with increasing flow rate. As
the flow rate was increased at 60 rpm, there was a slight
decrease in the hold-back and no discernable change in D/Lu.

DISCUSSION OF RESULTS - INERT BEADS

The experimentally-determined facts can be summarized as
follows. With floating beads, agitation definitely had a
deleterious effect. With "Scotchlite" beads, the effect of
agitation was unclear -- the hold-back increased with agitator
speed, then decreased. With either type of bead, the effect
of flow rate on hold-back depended on the degree of agitation
during the experiment. At high agitation, the hold-back
decreased slightly as the flow rate was increased; at low
agitation, the hold-back increased slightly. Material balances
with the early experiments on floating polyethylene beads were
only fair, whereas those with the "Scotchlite" beads were quite
good.

Discussion of Results - Floating Polyethylene Beads

The simple model involving a combination of piston flow
with dispersion at the interface is evidently not a realistic
model for experiments with the large floating polyethylene
beads, as is evidenced by Figures 12-15. All curves seemed to
be concave upwards.

The intensity function, $I(\theta)$, was useful in determining
why the dispersion model did not fit the data. The intensity

function was determined for a typical run (Run B-13) by first plotting ln c/c_0 vs. $v\theta/V_0$ (Figure 30), and then plotting the slope of this curve vs. $v\theta/V_0$ (Figure 31). As can be seen, the intensity function increased, reached a maximum, and then fell off.

The fact that Figure 31 exhibits a maximum is strong evidence that stagnancy existed in the system (see Section III). It seems likely that the stagnancy was caused by differences in the degree of longitudinal mixing at any given cross section. Mixing near the tips of the stirrer rods should be more intense than mixing near the axis. Such a flow pattern would give rise to deviations from Equation 1, and in simple cases it can be shown that a maximum in the intensity function would result.

Although some longitudinal mixing occurred throughout the vessel at the surface of the floating polyethylene pellets, intense mixing occurred at each of the agitator blades, thus the behavior should perhaps resemble that of a number of stirred pots in series. However, such behavior would not lead to a maximum in the intensity function, and the true behavior can probably best be explained by a combination of this effect and that described in the preceding paragraph.

It would be expected that the simple dispersion model should fit the data for no agitation, even though it was not valid in runs with agitation. Only one run was made without agitation, and the data were not sufficient to prove whether or not the model was valid.

The insight into the mechanism of mixing provided by the intensity function allows the anomalous behavior shown in Figure 17 to be better understood. The increase in hold-back, H, with increase in stirrer speed (see Figure 16) is to be expected. The rather large increase in the hold-back when the stirrer speed is increased to 120 rpm suggests that at high speeds, longitudinal mixing by the agitator completely over-shadows longitudinal mixing by random motion about the particles themselves.

The explanation of the different behavior at high and low agitator speeds (see Figure 17) is now evident. With no agitation, all mixing is caused by random fluctuations at the surface of the floating particles. In such a case, most investigators have found that the Peclet number is only a weak function of Reynolds number (see Figure 4). Assuming the Peclet number to be essentially independent of Reynolds number, the coefficient of longitudinal dispersion, D, should be nearly proportional to u, and the term D/Lu should be independent of flow rate. If (13) the Peclet number varies inversely as $N_{Re}^{0.2}$, it can be shown that D/Lu should vary as $u^{0.2}$ (see Appendix p. 78). In any event, D/Lu is but a weak function of flow rate: this conclusion is supported by the data represented by the lower line on Figure 17.

The opposite effect occurs at high agitator speeds, however. Increasing the velocity through the bed significantly decreased the hold-back. Increasing the linear velocity through the bed would have no effect on mixing by the agitator, but would

affect mixing at the surface of the particles. Longitudinal mixing by the agitator is probably far greater than longitudinal mixing at the surface of the particles themselves; thus, increasing the linear velocity through the floating beads did not significantly increase the longitudinal dispersion, as was the case without agitation. Physically, at low flow rates the agitator has a long time to achieve good mixing of the contents of the vessel; thus the hold-back should be greatest at low flow rates. As the flow rate is increased, the dispersion at the surface of the particles makes a more important contribution to the total dispersion, and the curves at both high and low agitation should approach each other as seen in Figure 17.

Although the simple dispersion model did not correlate the data, an apparent value of D/Lu was determined from the slopes of the best straight lines in Figures 12-15, and the corresponding value of the Peclet number entered in Table 1. In the one run made with no agitation, the Peclet number was 1.32. This value may be compared with the value 1.1 that would be obtained by the use of Figure 4. The Peclet number in runs with agitation was only a fraction of that obtained by use of Figure 4.

In summary, any agitation had a deleterious effect upon the washing of floating beads. The simple dispersion model superimposed on plug flow (Equation 1) does not correlate the data. The deviation from the simple model was probably caused

by the stirrer. Data obtained in the one run without agitation were not sufficient to confirm or disprove the simple dispersion model.

Unpublished work recently done at M.I.T. indicates that the simple dispersion model is correct when floating beads are washed without agitation. In this work, a plot of Z_1 vs. $\dfrac{1 - v\theta/V_o}{2\sqrt{v\theta/V_o}}$ yielded a straight line. In addition, the shape of the curve of intensity function versus $v\theta/V_o$ was identical to that which would be predicted for this model. (See Figure 3b.) Values of the Peclet number varied from 0.75-1.0 over a range of Reynolds numbers of from 5-20, values close to those that would be predicted from Figure 4 for packed beds under identical conditions.

Discussion of Results - Scotchlite Beads

While a satisfactory explanation for the behavior of the floating polyethylene beads can be given, the results with the "Scotchlite" beads were inconclusive and difficult to interpret. Summarizing the experimental results:

1. In plots of Z_1 vs. $\dfrac{1 - v\theta/V_o}{2\sqrt{v\theta/V_o}}$, it was possible to draw straight lines through most of the data. Thus the fit of the data to the dispersion model was fair.

2. Both the hold-back and D/Lu increased as the stirrer speed was increased, reached a maximum at 90 rpm, and

decreased to a value almost the same as with no agitation as the agitator speed was increased to 120 rpm.

3. In the absence of agitation, both hold-back and D/Lu increased moderately with increasing flow rate. At 60 rpm, as the flow rate was increased there was a slight decrease in the hold-back and no discernable change in D/Lu.

The intensity function was again useful in determining whether or not segregation did occur. Figures 32 and 33 represent a test of the data from Run 31, one of two runs without agitation. The normalized intensity function curve was derived by taking the slope of the solid line in Figure 32. (The last two points were disregarded because of doubt regarding the reliability of the data at low concentrations.) As can be seen from Figure 33, the normalized intensity function increases but its slope tends to decrease; however, there is no maximum, and the curve does not appear to be approaching an asymptote. The curve shows no indication of stagnation, but does not show the form expected for a simple combination of piston flow and dispersion at the water-brine front.

No reasonable explanation can be offered for the maximum in curves of hold-back and D/Lu as agitation is increased. Behavior up to 60 rpm is as expected, but the sharp drop-off at 90 rpm is surprising.
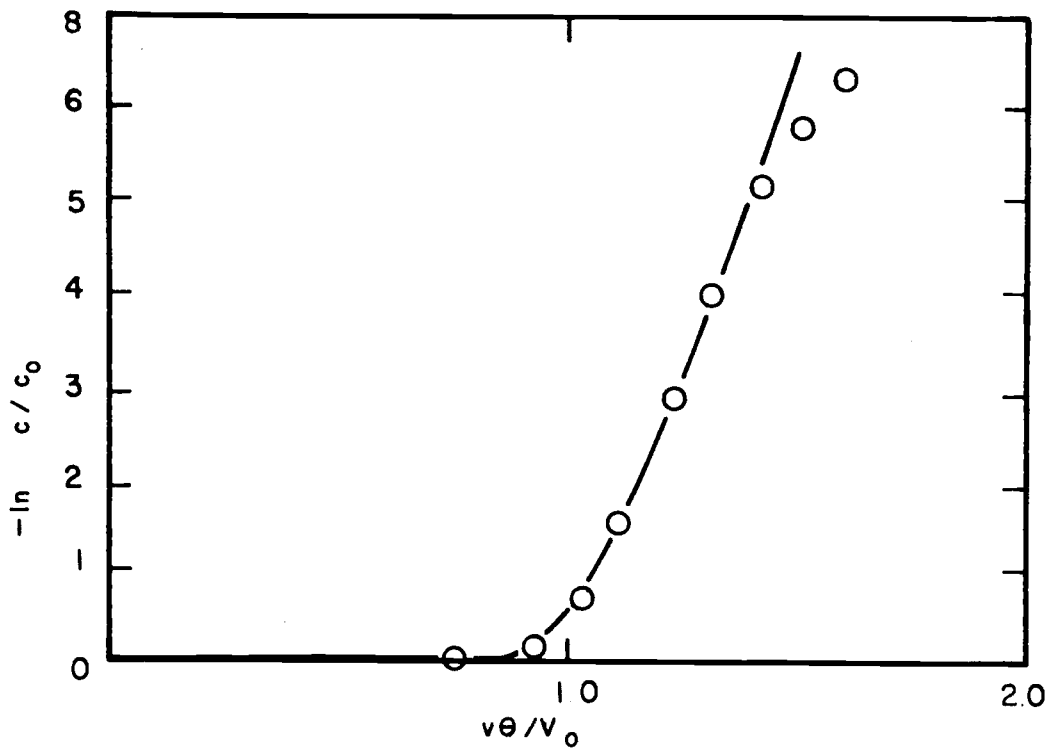
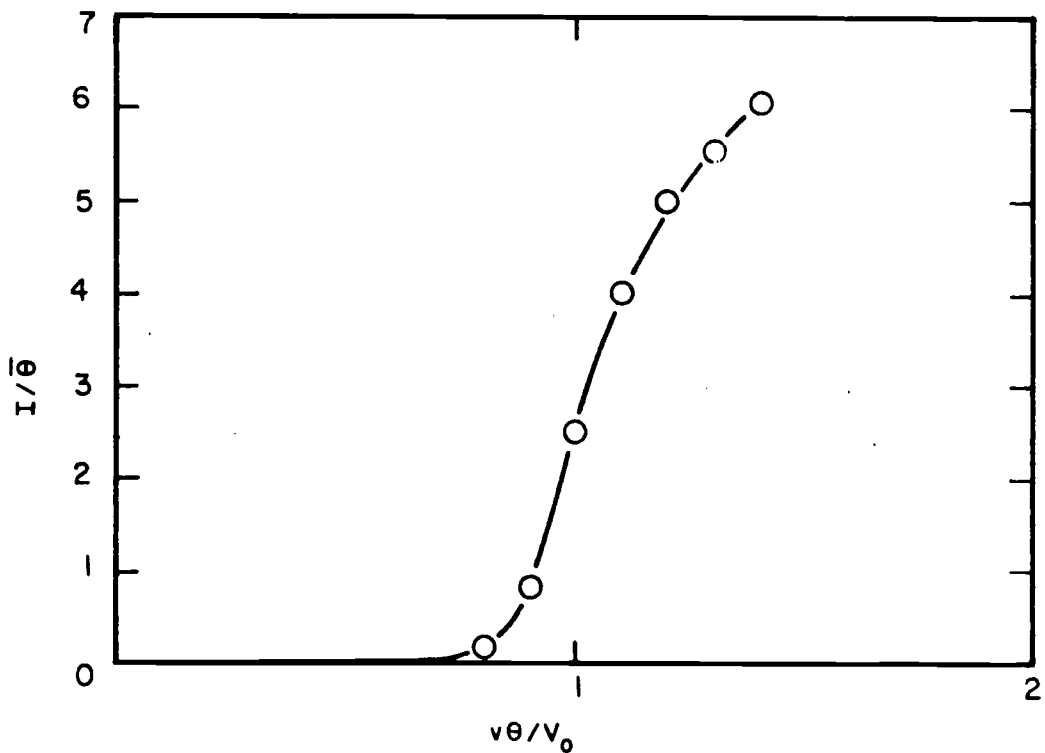Fig. 32: RUN B-31 "SCOTCHLITE" BEADS

Fig. 33: RUN B-31 INTENSITY FUNCTION

The results at variable flow rate and constant stirrer speed are about as expected. The results are similar to those already discussed in experiments with floating polyethylene beads.

An apparent value of D/Lu was determined from the slopes of the best straight lines in Figures 22-25, and the corresponding value of the Peclet number entered in Table 1. As expected, low values for the apparent Peclet number were obtained at high stirring speeds and low flow rates. Even in the two runs without agitation, the values of the Peclet number were considerably below those determined by other investigators. The reason for this discrepancy is unknown.

Comparison of Results

From the data obtained, it is not possible to make many generalizations. Without agitation, the simple dispersion theory would predict that it should be easier to wash the smaller particles and that deviations from piston flow should be insensitive to wash rate.

Where the dispersion model is valid, the dispersion coefficient D can be determined equally well by the slope method (see Equation 3 et seq.), or by determining the hold-back (see p. 23), since H should be a unique function of D/Lu. Thus if the dispersion model is valid, it should be possible to plot H versus D/Lu. Figure 34 is such a graph, with the theoretical relation obtained by combining Equations 1 and 9. With one exception, points representing runs with "Scotchlite" beads fell close to the calculated line, while points for polyethylene beads deviated considerably. Figure 34 shows that D/Lu and H are related. It
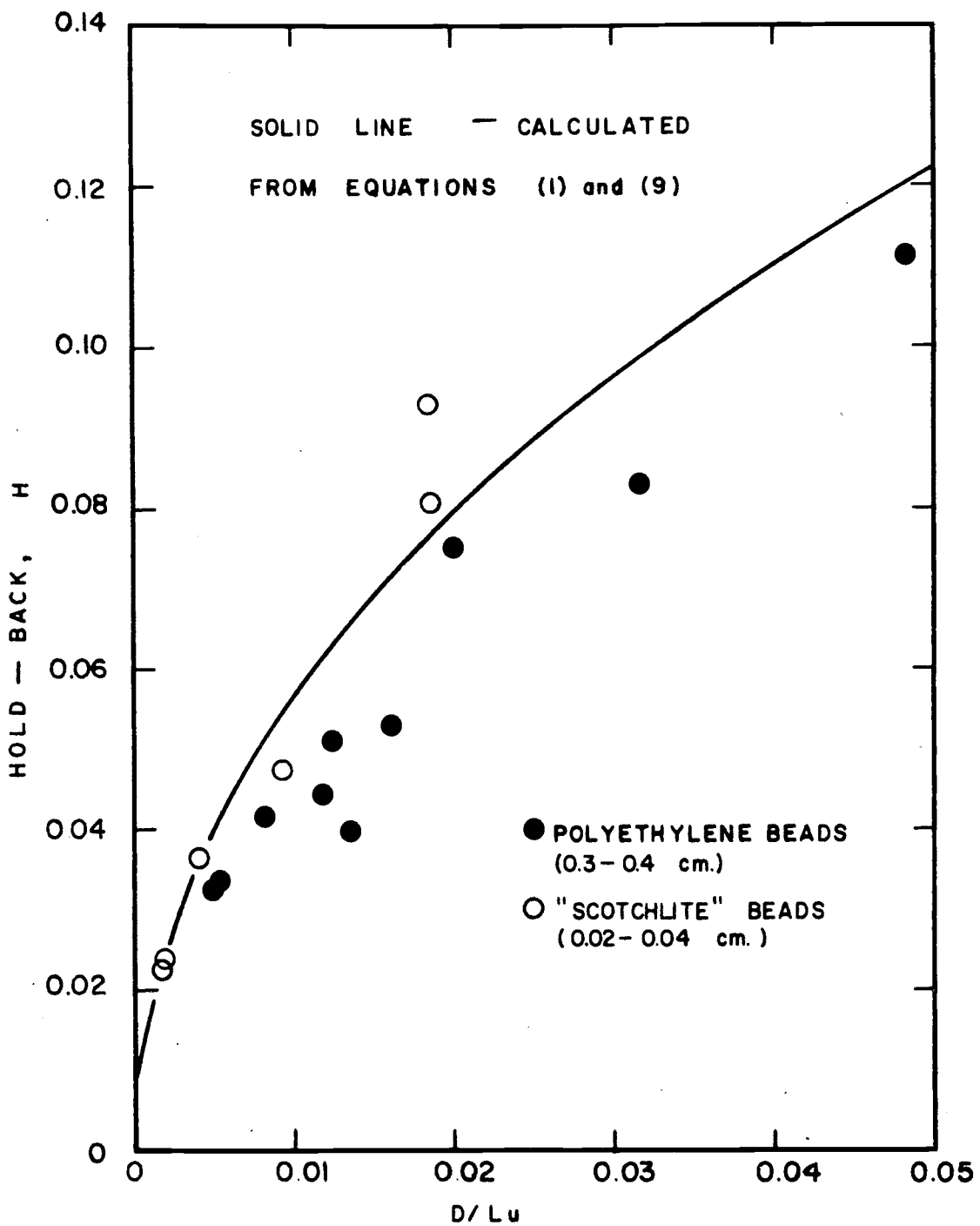
Fig. 34 : H vs D/Lu FOR BEADS

suggests the simple dispersion model to be consistent with the data on "Scotchlite" beads, but that this simple model does not adequately represent the data on the larger floating beads.

EXPERIMENTAL STUDIES - WASHING ICE CRYSTALS

Experimental Results

A total of 14 runs was made to study the effect of various operating variables in washing brine from ice crystals. Four runs were made without agitation to study the effect of wash rate. Five runs were made with varying heights of slush in the column to study the effect of bed height. Two runs were made with the agitator running, and two runs were made with a pulsed column. A single run was made with a drained bed. In the treatment of the data, it was assumed that no melting or freezing took place during the washing operation. Data for all runs are summarized in Table 2.

The results demonstrated that a close approach to "piston flow" of the wash water was accomplished in displacing brine from ice crystals, just as had been the case in washing inert beads. Except for a short period at the very start of the run, the salt concentration in the exit stream remained essentially constant till 85-95 percent of the original volume had been displaced. The concentration at the exit then dropped rapidly and closely approached zero by the time 1.15-1.40 times the void volume had been displaced. In general, the results in washing ice crystals were better than those in washing beads, in that there was less mixing at the water-brine front, and a closer approach to piston flow was obtained.

Table 2

Washing Brine From Ice Crystals

Run	I-5	I-6	I-7	I-8	I-12	I-13	I-14	I-16	I-17	I-10	I-15	I-18	I-19	I-9
Remarks	VARIABLE FLOW RATE				VARIABLE COLUMN HEIGHT					VARIABLE STIRRER SPEED		PULSING		DRAINED BED
Wash Rate, lb/(hr ft^2)	138	63.8	198	262	137	137	136	141	146	136	136	128	131	134
Column Height, in.	28	28	28	28	10	19	16	14	10	28	28	28	28	28
Stirrer Speed, rpm	0	0	0	0	0	0	0	0	0	8	4	0	0	0
Pulses Per Min.	0	0	0	0	0	0	0	0	0	0	0	4	12	0
Ice, Grams	2129	2086	1805	1784	688	1208	1266	1078	741	1736	2212	2217	1742	1882
Liquid ml(initial)	4245	4185	4320	4490	1556	3100	2330	2080	1642	4480	4050	4160	4480	1070
Liquid, ml(final)	4032	3962	4136	4333	1572	2982	2432	2069	1704	3864	3253	3928	4533	1064
Hold-back, H	0.0241	0.0195	0.0265	0.0258	0.0413	0.0410	0.0340	0.0411	0.0402	0.0865	0.105	0.0346	0.0339	0.58
D/Lu	0.00129	0.00083	0.00141	0.00145	0.0041	0.00391	0.00298	0.0049	0.00427	0.0414		0.00475	0.0065	
N_{Pe}	0.545	0.848	0.499	0.485	0.48	0.265	0.413	0.287	0.461	0.017		0.148	0.108	
N_{Re}	0.147	.068	0.197	0.256	0.138	0.133	0.146	0.150	0.147	0.135	0.148	0.139	0.126	

Ice Crystal Size -- The general shape of the ice crystals
remained unchanged from run to run, but the nominal size
varied somewhat. The great majority of the crystals were of
the oyster shape, but a number resembling horseshoes and
perforated platters were observed (see Figure 35). The crystals
occurred in very narrow size distributions, nearly all grouped
very closely about the nominal size. The nominal large dimen-
sion ranged between 0.40 and 0.85 millimeters for all the runs.
When the nominal large dimension was 0.40 millimeters, the
maximum size was 0.70 millimeters, with nearly all the crystals
above 0.20 millimeters, and a very few small crystals perhaps
0.04 millimeters long. The horseshoe shape was frequently
found among the very small crystals, but it was rarely found
among larger crystals. The perforated platters occurred only
as large crystals 1 millimeter in diameter or larger, and
were only seen in batches with a nominal large diameter of 0.85
millimeters. A number of measurements of ice crystal diameter
were made, but unfortunately these measurements were not made
in all of the runs. Insufficient measurements were made to
allow complete characterization of the shape.

One of the major difficulties in interpreting the results
was that the size and shape of crystals varied from batch to
batch. Although data within any one run appeared consistent,
changes in the size of ice crystals from batch to batch made
it difficult to compare results. If N_{Pe} varies as $N_{Re}^{-0.2}$, it
can be shown that D/Lu should vary as $d_p^{1.2}$. (See Appendix II).

TOP

SIDE

"OYSTER"   SHAPES

TOP

SIDE
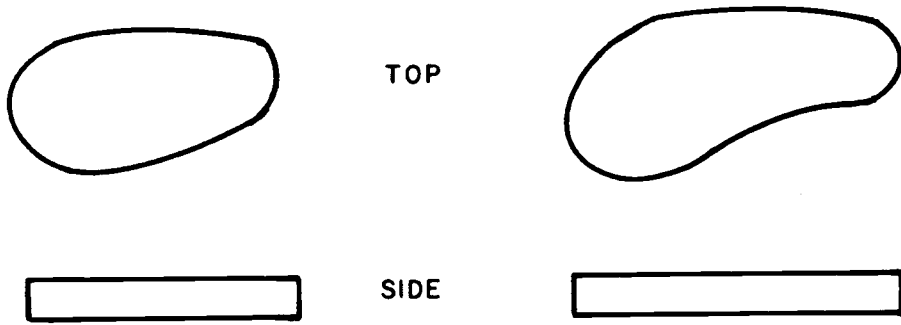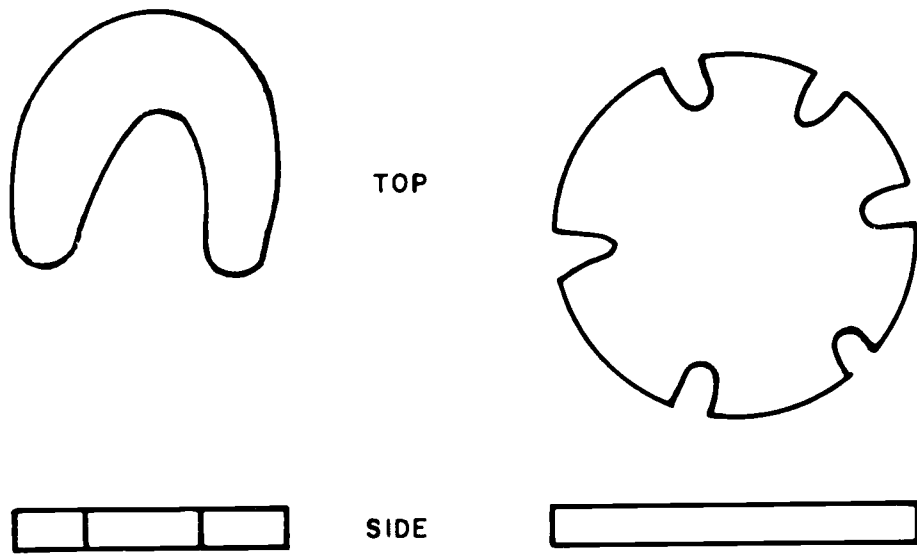
HORSESHOE   " PERFORATED   PLATTER"

Fig. 35 :   SCHEMATIC   DIAGRAMS   OF   THE   SHAPES
           OF   TYPICAL   ICE   PARTICLES

Thus, if the simple dispersion model is correct, variations in particle size between runs should have an important effect on washing efficiency. There was always uncertainty as to whether the different results in two runs were due to an actual change in operating conditions or merely to a change in the size of the ice crystals.

Typical Results -- Representative data for washing ice crystals in flooded beds are presented in Figures 36-39. Figure 36 is typical of some of the best results obtained at low wash rate, maximum column height, and no agitation. Figure 39 is representative of some of the worst results obtained with high agitation. In all runs there was a sharp rise in the exit concentration at the very start of a run. Probably the reason for this behavior was that a small amount of water remained in the exit pipe and diluted the first part of the liquid that was drained from the bed.

Material Balances -- Material balances closed about as well as the average for the experiments with washing inert beads. The material balances were checked by plotting the concentration of salt versus the volume of drainings in a run. Graphical integration of this curve permitted calculation of the initial salt concentration. The average deviation of the calculated value from the actual measured value was 3.6 percent.

As noted earlier, stoichiometry dictates that the area above the curve to the left of the line $v\theta/V_o = 1$ must be
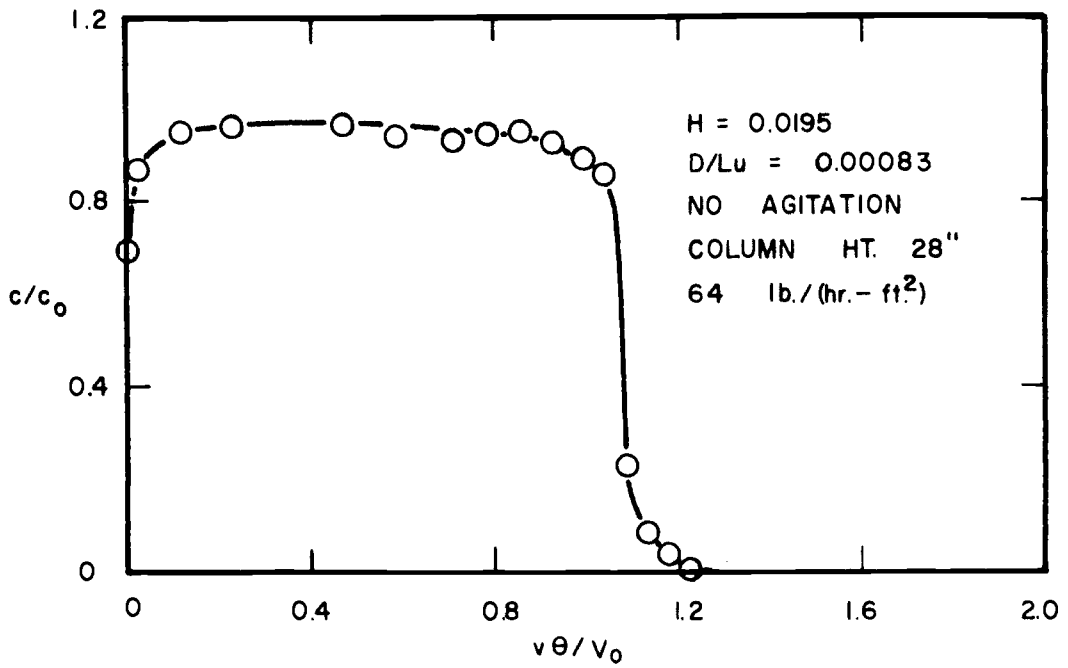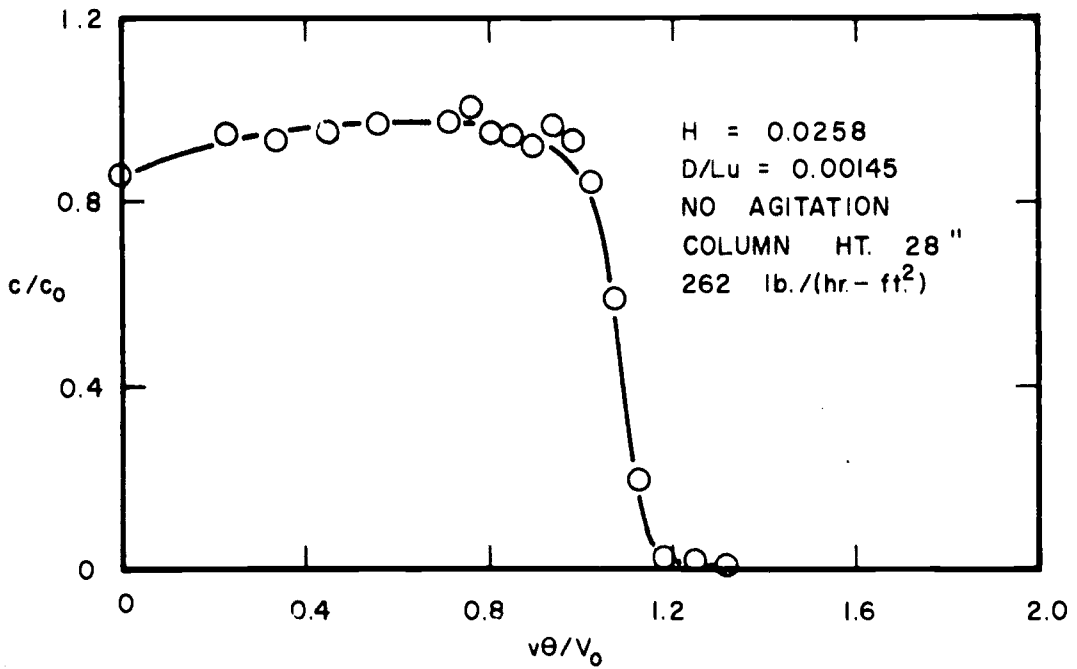
Fig. 36: RUN I-6, ICE

H = 0.0195
D/Lu = 0.00083
NO AGITATION
COLUMN HT. 28"
64 lb./(hr.-ft.^{2})

Fig. 37: RUN I-8, ICE

H = 0.0258
D/Lu = 0.00145
NO AGITATION
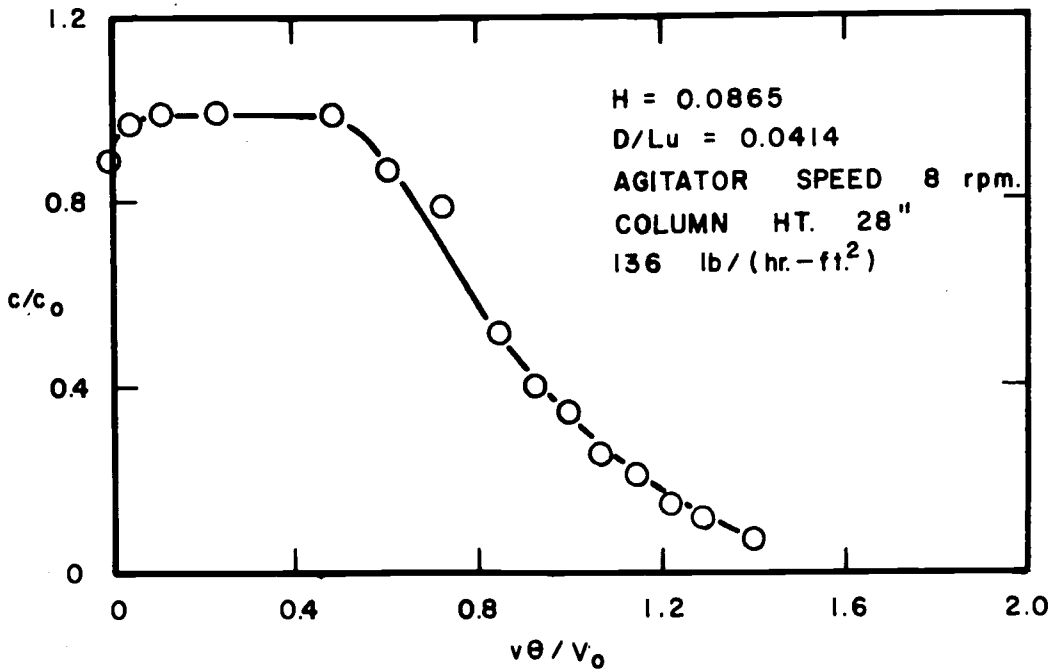COLUMN HT. 28"
262 lb./(hr.-ft.^{2})
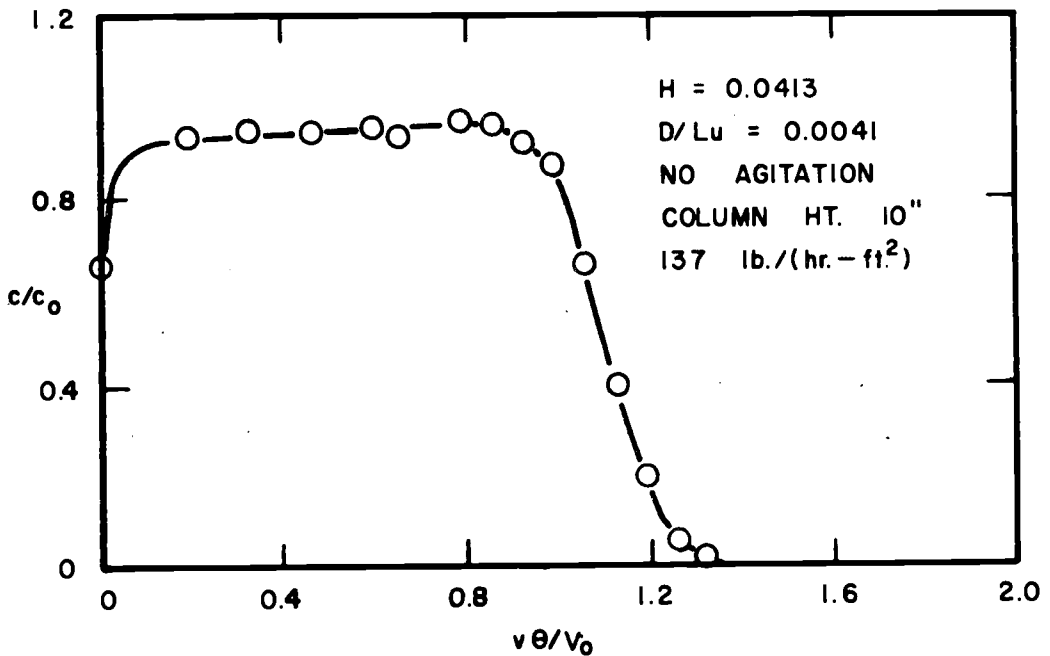
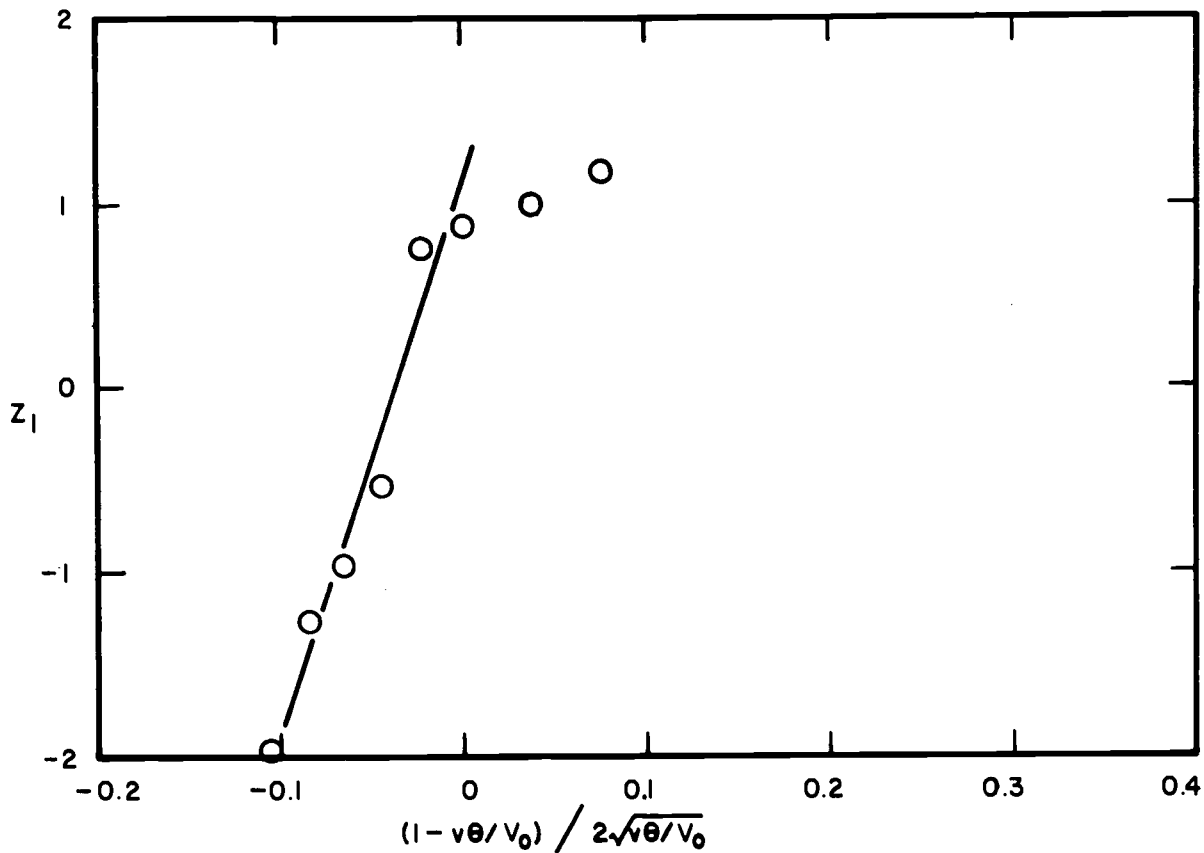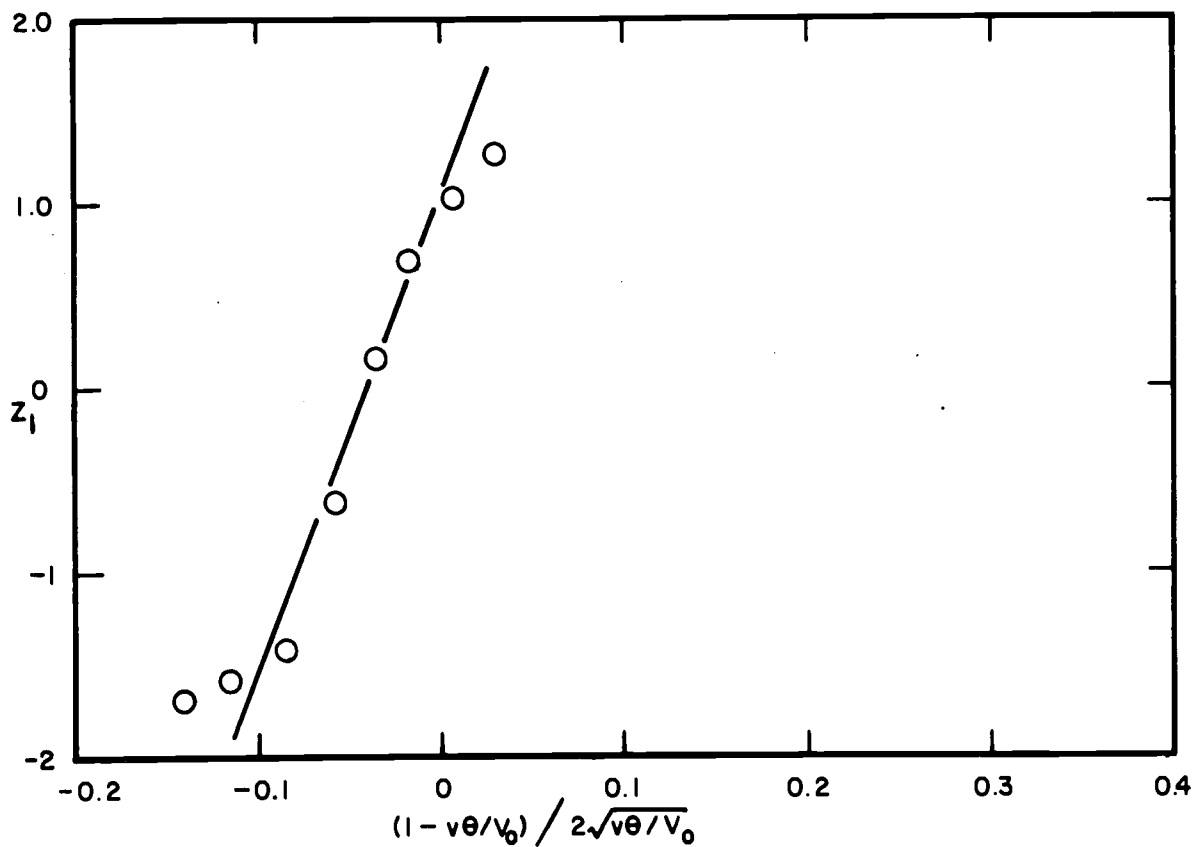Fig. 38: RUN I-10, ICE

Fig. 39: RUN I-12, ICE
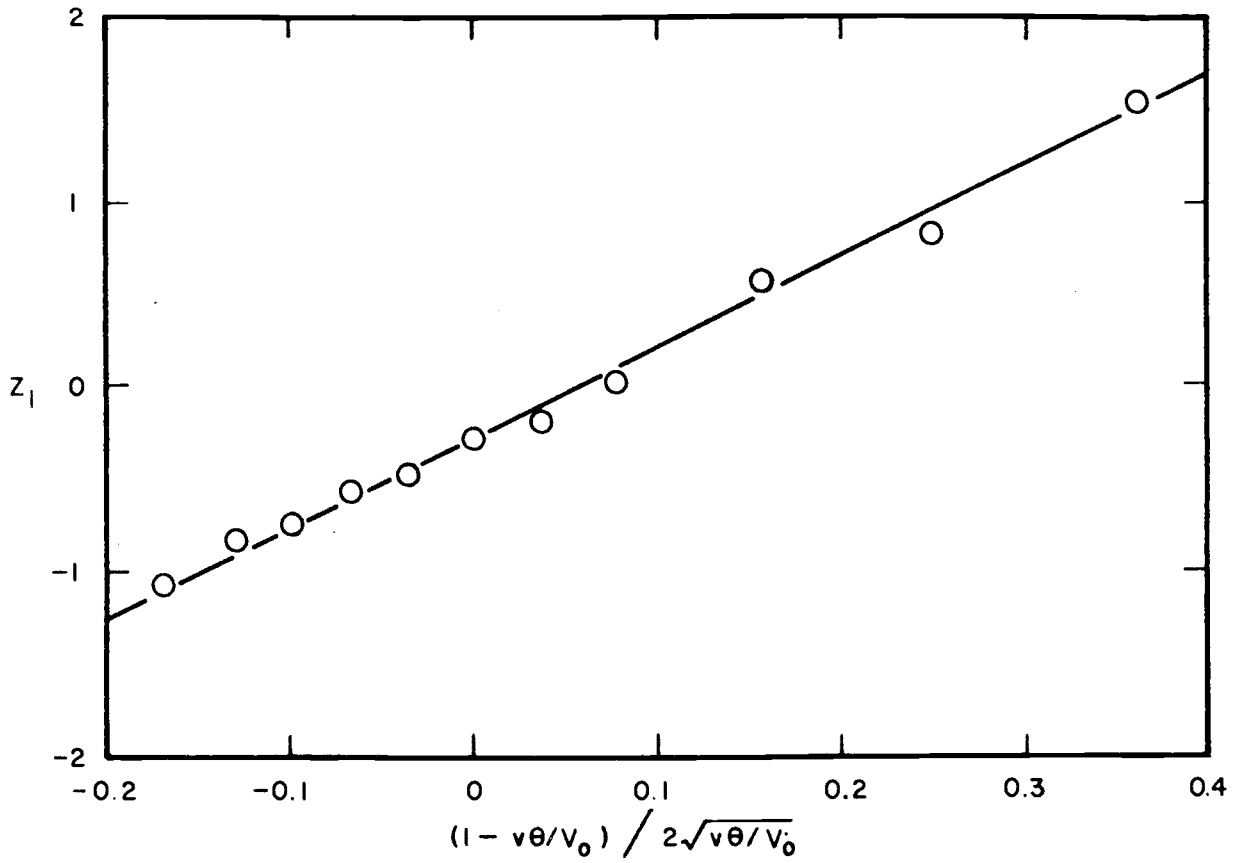
Fig. 40: RUN I-6, ICE

Fig. 41: RUN I-8, ICE
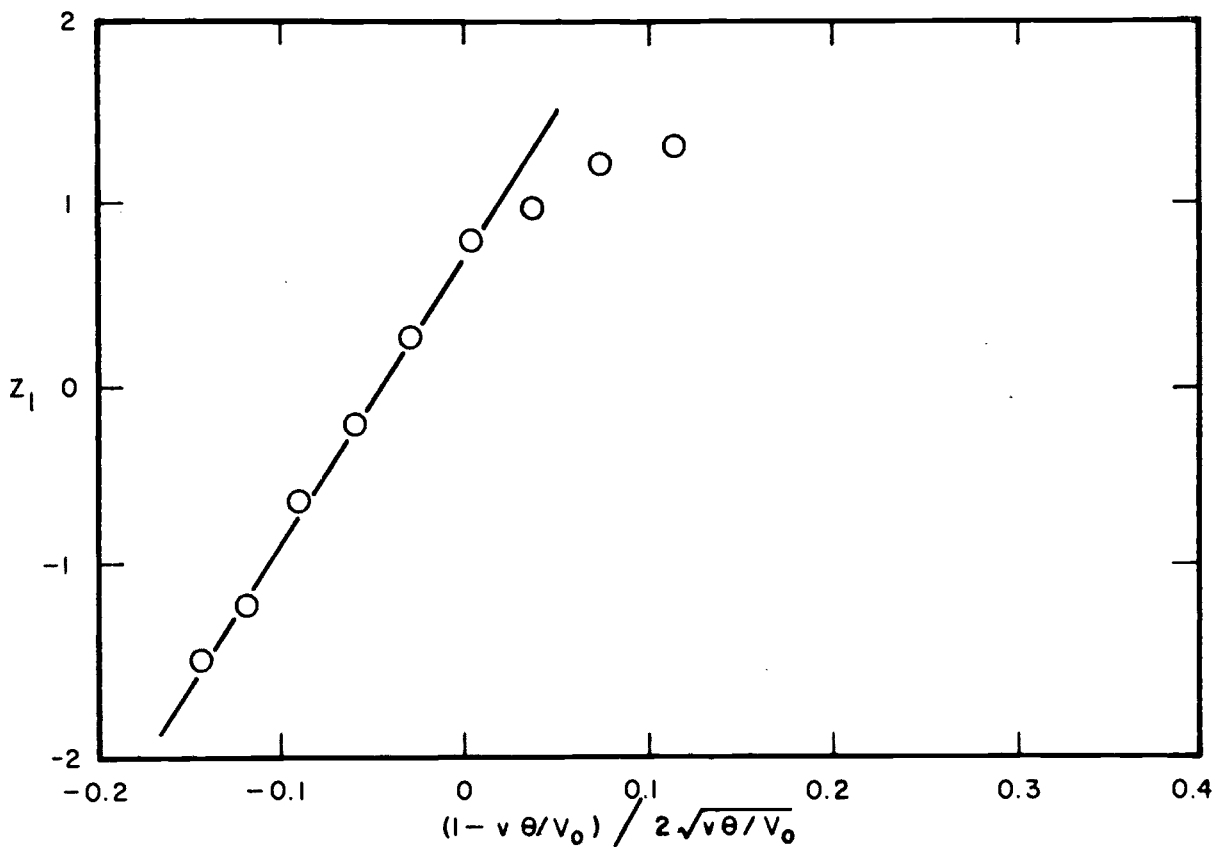
Fig. 42: RUN I-10, ICE

Fig. 43: RUN I-12, ICE

COLUMN HEIGHT 28"

FLOW RATE ∼ 135 lb./(hr.−ft.^{2})

Fig. 44: ICE

COLUMN HEIGHT 28"

FLOW RATE ∼ 130 lb/(hr.−ft.^{2})

Fig. 45: ICE

HOLDBACK, H

WASH RATE, lb./(hr.−ft.²)

NO AGITATION

28" COLUMN HT.

Fig. 46 : ICE

D/Lu

WASH RATE, lb./(hr.−ft.²)

NO AGITATION

28" COLUMN HT.

Fig. 47: ICE

WASH RATE $\cong$ 140 lb./(hr. – ft.^{2})
NO AGITATION

Fig. 48: ICE

FLOW RATE $\cong$ 140 lb./(hr. – ft.^{2})
NO AGITATION
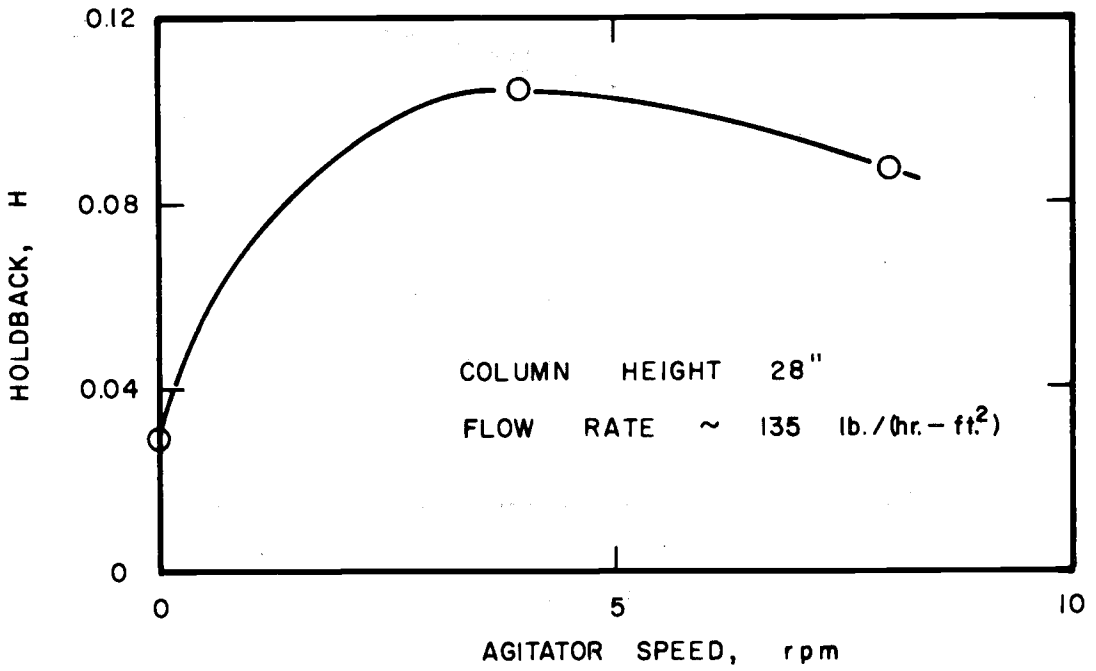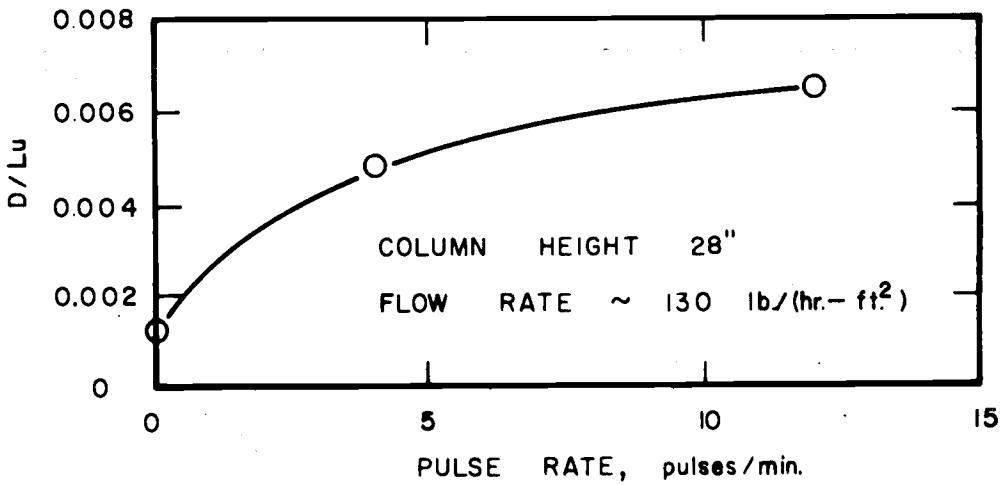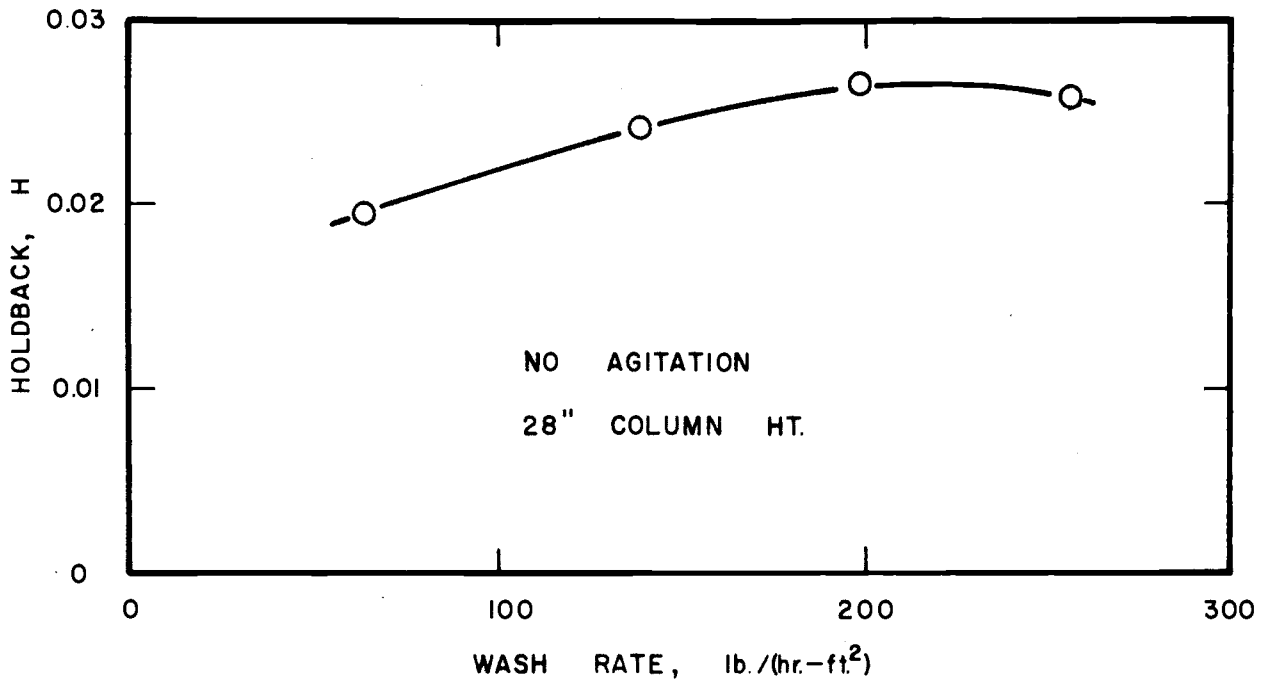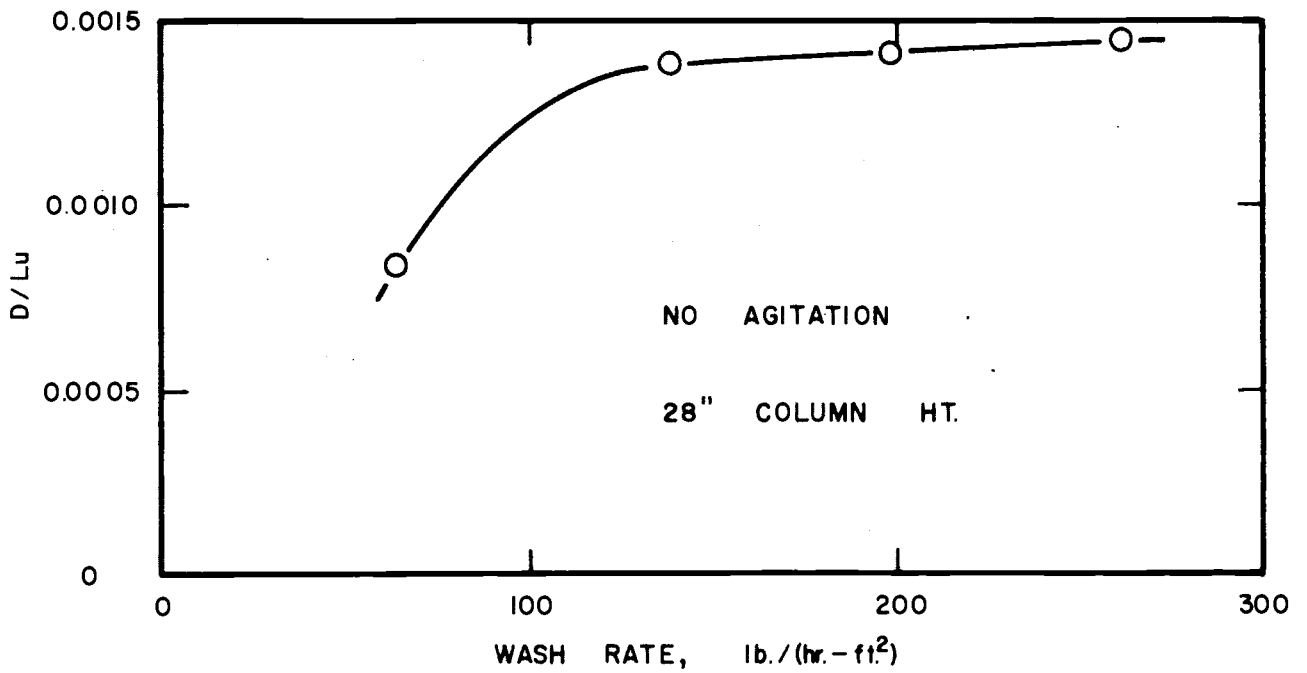
Fig. 49: ICE

Fig. 50: RUN I-9, ICE, DRAINED BED

exactly equal to the area below the curve on the right of this same line. Another criterion is that c/c_o should have a value of very nearly 0.50 when $v\theta/V_o = 1$. Fairly consistently the curves of c/c_o vs. $v\theta/V_o$ intersected the line $v\theta/V_o = 1$ at a value considerably higher than 0.50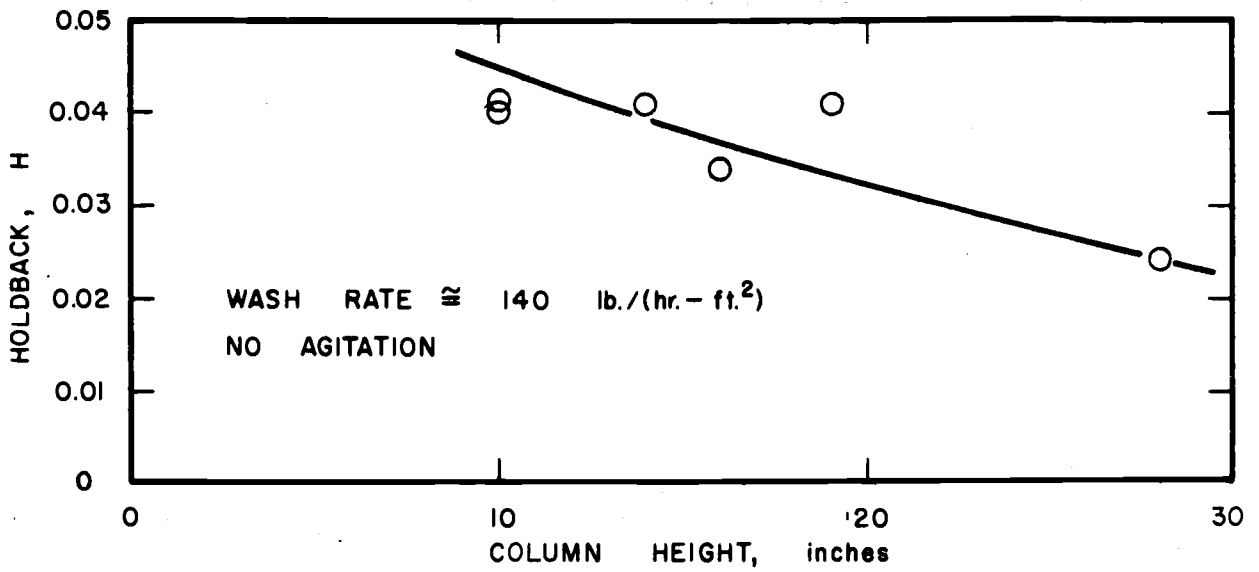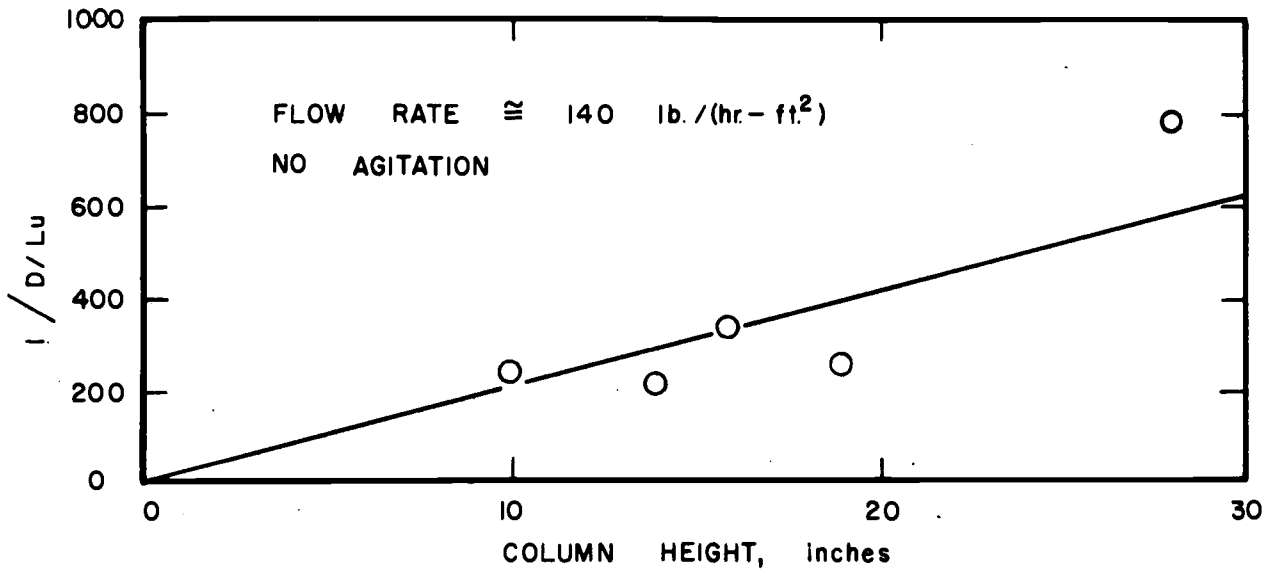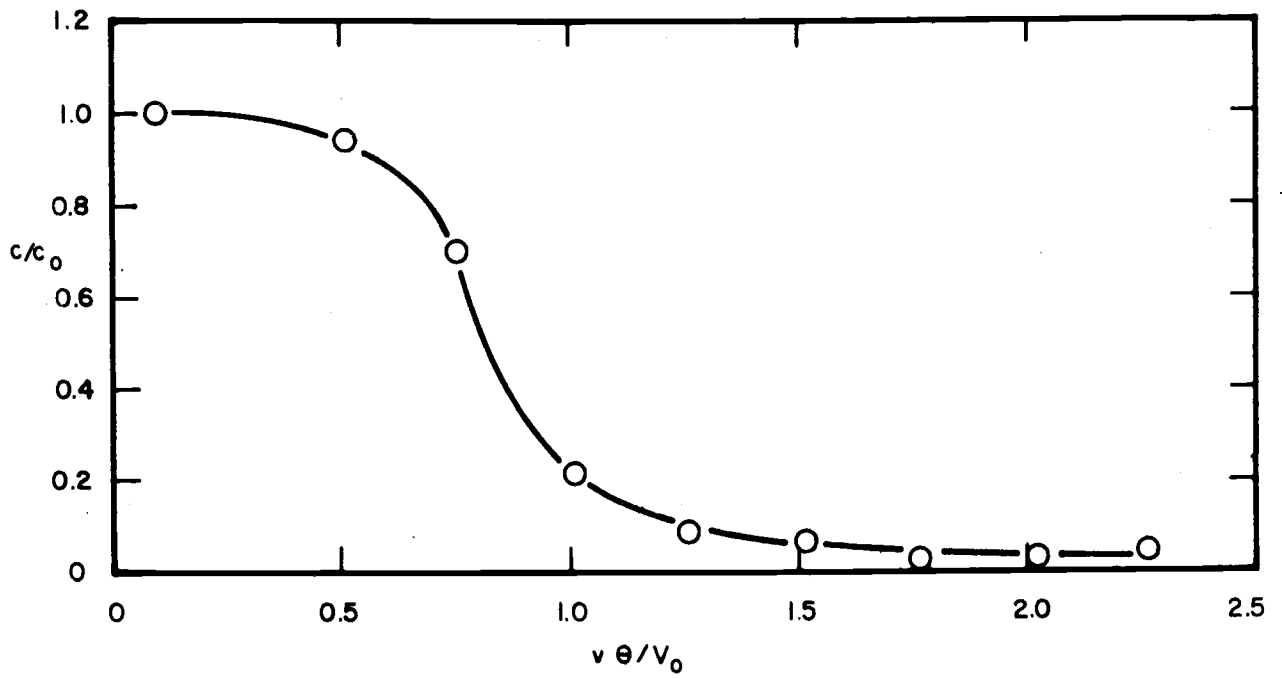, that is, the entire curve was displaced farther to the right than it should have been. Reasons for this behavior are suggested on page 59.

Determination of the Dispersion Coefficient, D -- The dispersion coefficient D was again determined by plotting Z_1

$$vs \left[\frac{1 - v\theta/V_o}{2 \sqrt{v\theta/V_o}} \right]$$ as shown in Figures 40-43 for four typical runs.

Figure 42 is an example of one of the best correlations; Figure 41 represents one of the worst fits of the data to Equation 1. Little weight was given to points near $c/c_o = 1$, or near $c/c_o = 0$. Outside of this range, it was generally possible to draw a good straight line through most of the data points, indicating the applicability of the simple dispersion model. The effect of varying operating conditions can be expressed in terms of either D/Lu or the hold-back, H.

Effect of Agitation -- Three runs were made under identical conditions except that the agitator speed was set at 0, 4, and 8 rpm. The results are shown in Figure 44. Agitation obviously has a deleterious effect. Any beneficial effect in promoting displacement of the brine by radial mixing was counterbalanced by the additional longitudinal mixing. The reason for the maximum in the curve is not understood. It is

conceivable that differences in ice crystal size between runs
may have lead to the small decrease in hold-back as the agitator
speed was increased from 4 to 8 rpm; however, the dramatic
increase in hold-back from 0 to 4 rpm is a real effect that
could not be caused by such differences.

Two runs were made in which the entire wash column was
pulsed by the alternate expansion and contraction of a balloon
at the bottom of the column. Two pulsing rates were used --
4 per minute, and 12 per minute. Results are shown in Figure 45.
It is clear that pulsing had a deleterious effect. Apparently
pulsing contributed to longitudinal mixing, and this effect
counterbalanced any possible beneficial effect of improved mass
transfer.

Effect of Varying Flow Rate -- Four runs were made under
identical conditions, except that the flow rate was varied.
The results of this study are shown in Figure 46 and 47.
Increasing the wash rate from 50 to 130 lb/(hr ft^2) caused a
small increase in the holdback. This is probably within the
precision of the measurements, and it cannot be concluded that
there is a definite trend of H and D to increase with flow rate;
the change may possibly be due to variation in ice particle
size, which was difficult to control.

Effect of Varying Column Height -- Five runs were made
with varying amounts of slush in the column to study the effect
of variations in column height. The results are shown in

Figures 48 and 49. There appears to be a trend to higher hold-back as the column height is decreased, but the scatter of the data is such as to make it hard to draw any quantitative conclusions.

Drained Bed -- One run was made with a drained bed. In this run, as much as possible of the brine was allowed to drain by gravity before the start of the run. The initial slush contained 64 percent ice, as against an average of 29 percent ice in the other runs. The results are shown in Figure 50. It can be seen that the concentration of salt in the outlet began to decrease when a quantity far less than one void volume was used for washing. The washing process was character-ized by serious "tailing", that is, excessive amounts of wash water were required to reduce the salt concentration to an acceptable level. When an amount of wash water equivalent to 2.27 times the void volume was used, the salinity of the ma-terial in the column after melting was 1670 ppm, still above the acceptable concentration of 500 ppm. In this run, the quantity of wash water wasted was equivalent to nearly 80 percent of the ice washed, and still potable water was not obtained after melting

Comparison of Figure 50 with either Figure 36 or 37 illustrates the superiority of washing in a flooded bed, even though direct comparison tends to exaggerate the advantage of a flooded bed. (The amount of liquid originally on the

crystals, V_o, was only about one-quarter of that in the run with flooded beds.)

DISCUSSION OF RESULTS - ICE CRYSTALS

Summarizing the results: (1) In plots of Z_1 vs $\dfrac{1 - v\theta/V_o}{2\sqrt{v\theta/V_o}}$,

it was possible to draw straight lines through most of the data; thus the fit of the data to the dispersion model was quite good. (2) Any agitation had a deleterious effect, whether it was caused by rotating the stirrer, or by pulsing. (3) The hold-back increased somewhat as the flow rate was increased. (4) The hold-back increased somewhat as the column height was decreased. (5) Although the data within any one run were consistent, there was a noticeable scatter when comparing data of different runs. (6) Results in a drained bed were far inferior to those in a flooded bed.

Material Balances

As pointed out earlier, the curves of c/c_o vs. $v\theta/V_o$ are consistently displaced to the right of where they should be. It is believed that this displacement is due to inaccurate measurement of the void volume, in turn due to inaccurate measurement of the concentration of salt in the brine at the start of the run. The salt was analyzed by measuring the con-ductivity and comparing this measured value with that of standard solutions. At low concentrations, the conductivity changes rapidly with concentration and high precision analysis is possible except at very low concentrations. The salt content

of moderately concentrated solutions (above 1.0 percent salt)
becomes progressively more difficult to measure because con-
ductivity changes relatively slowly with concentration in this
range. A simple solution to the problem is to dilute the con-
centrated samples with a known volume of water. This precaution
was taken during analytical work with the inert beads, but it
was not taken in the ice-washing experiments.

Freezing or Melting During a Run

Freezing or melting of ice during a run could occur
because of heat transfer from the cold room to the wash column;
however, even under adiabatic conditions some additional ice
would be formed during washing. The temperature of the ice-
brine mixture was about 27.5°F at the start of a run. During
washing, contact of fresh water with 27.5°F ice results in
freezing an additional amount of ice equivalent to about three
percent of that present at the start of the run.

Freezing this additional amount of ice can be conveniently
considered to have two separate effects as shown in Figure 51.

(a) DISPLACEMENT OF CURVE DUE TO DENSITY DIFFERENCE
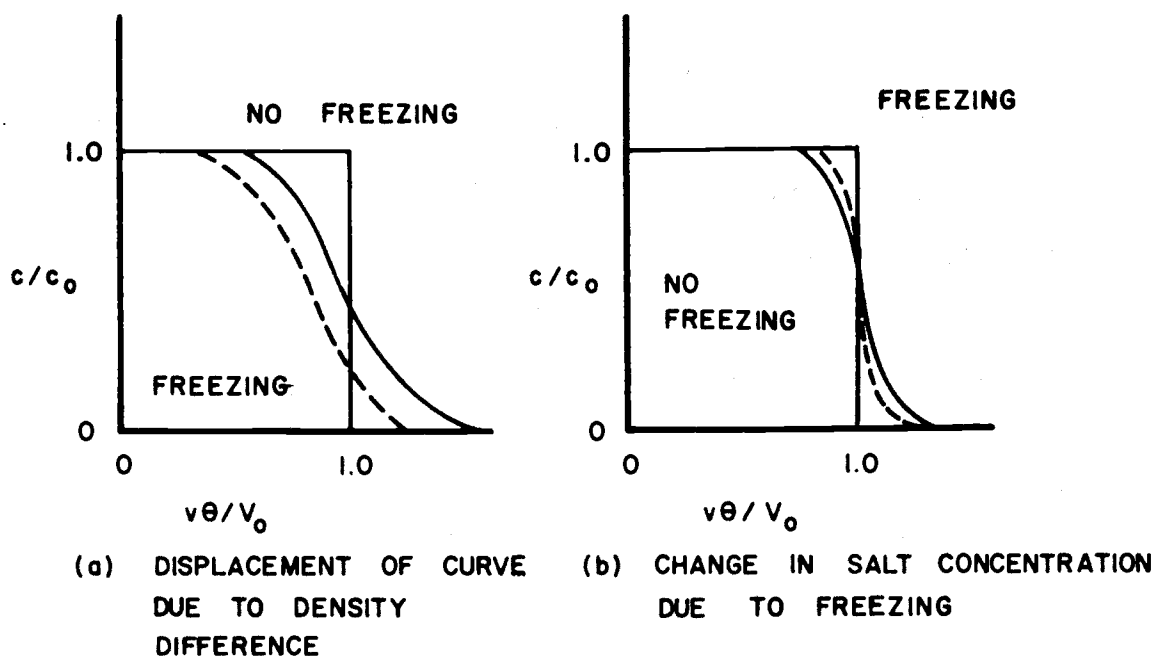
(b) CHANGE IN SALT CONCENTRATION DUE TO FREEZING

Fig. 51: EFFECT OF PARTIAL FREEZING

If the level in the wash column is held constant, freezing of additional ice will tend to increase the quantity of washings because of the difference in density of ice and water, thus displacing the entire curve to the left, as shown in (a). Freezing the additional amount of ice raises the concentration of brine at any point in the moving front, but the front tends to move through faster, giving rise to the curve shown in (b). Melting of ice caused by heat transfer would have the opposite effect. Calculations indicate heat transfer would cause melting or freezing of not more than a few percent.

In a typical run with constant liquid level in the wash column, three percent additional ice formation would result in

a displacement of the line at $v\theta/V_o = 1.0$ of less than 0.2
percent. The change in concentration at any point is somewhat
more serious, but still relatively unimportant. This same
amount of additional ice formation would cause a change in the
concentration profile of about one percent. This change is
again within the limits of experimental error. Thus the as-
sumption of no melting or freezing of ice appears to be well
justified.

Reliability of Results

Runs under optimum washing conditions were difficult to
analyze, whereas runs that gave inefficient washing were easy
to analyze. The reason for this is that, under optimum washing
conditions, there was a sharp break in the curve of c/c_o vs.
$v\theta/V_o$ in the neighborhood of $v\theta/V_o = 1$. Values of c/c_o near
unity or zero are of doubtful accuracy, thus in runs made
under optimum conditions, only three or four points could be
used to prepare plots of Z_1 vs. $\dfrac{1 - v\theta/V_o}{2\sqrt{v\theta/V_o}}$. For this reason,
runs under optimum conditions (low flow rate, no agitation,
maximum column height) usually provided only a few points in
the range of interest. The best runs for testing the validity
of the dispersion model were therefore those made under rela-
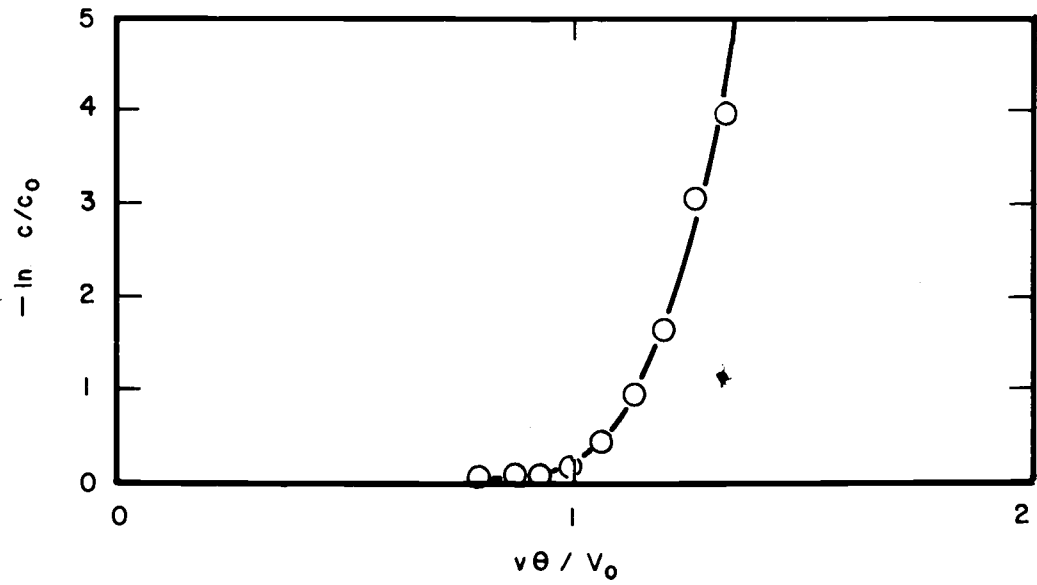tively poor washing conditions (high flow rate and minimum
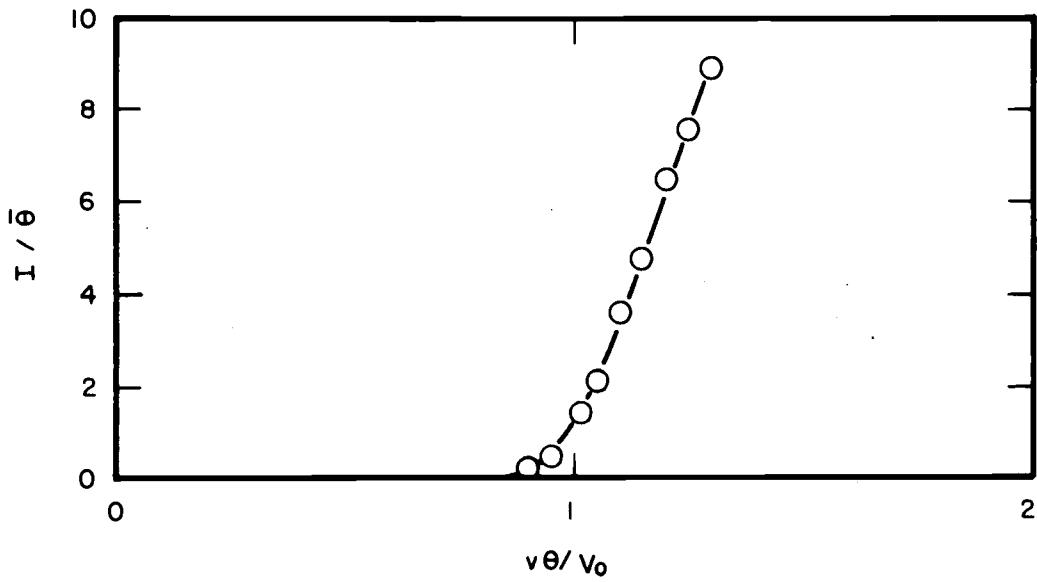column height).

Fig. 52: RUN I—12, ICE

Fig. 53: RUN I—12, ICE

Validity of the Simple Dispersion Model

Most of the data fell on a straight line (excluding points at high and very low concentration) when plotted in the form Z_1 vs. $\dfrac{1 - v\theta/V_o}{2\sqrt{v\theta/V_o}}$. This in itself lends strong support to the simple dispersion model.

The intensity function, $I(\theta)$, was again very useful in determining whether stagnancy existed, as it is frequently possible to glean much pertinent information by this sensitive test of the data. Figure 52 is a plot of the logarithm of c/c_o vs. $v\theta/V_o$ for run I-12, a run made under wash conditions far removed from the optimum. Figure 53 is the intensity function curve derived from Figure 52. There appears to be no tendency for the intensity function to reach a maximum; thus, there is no evidence of short circuiting. Instead, the behavior is precisely that would be expected for a combination of piston flow and dispersion at the water-brine front (see Figure 3b); hence Figure 53 is additional confirmation of the validity of the dispersion model.

If the simple dispersion model is valid, the dispersion coefficient D could again be determined equally well by the slope method or by determining the hold-back, since H should be a unique function of D/Lu. A plot of hold-back, H, versus D/Lu is given in Figure 54.
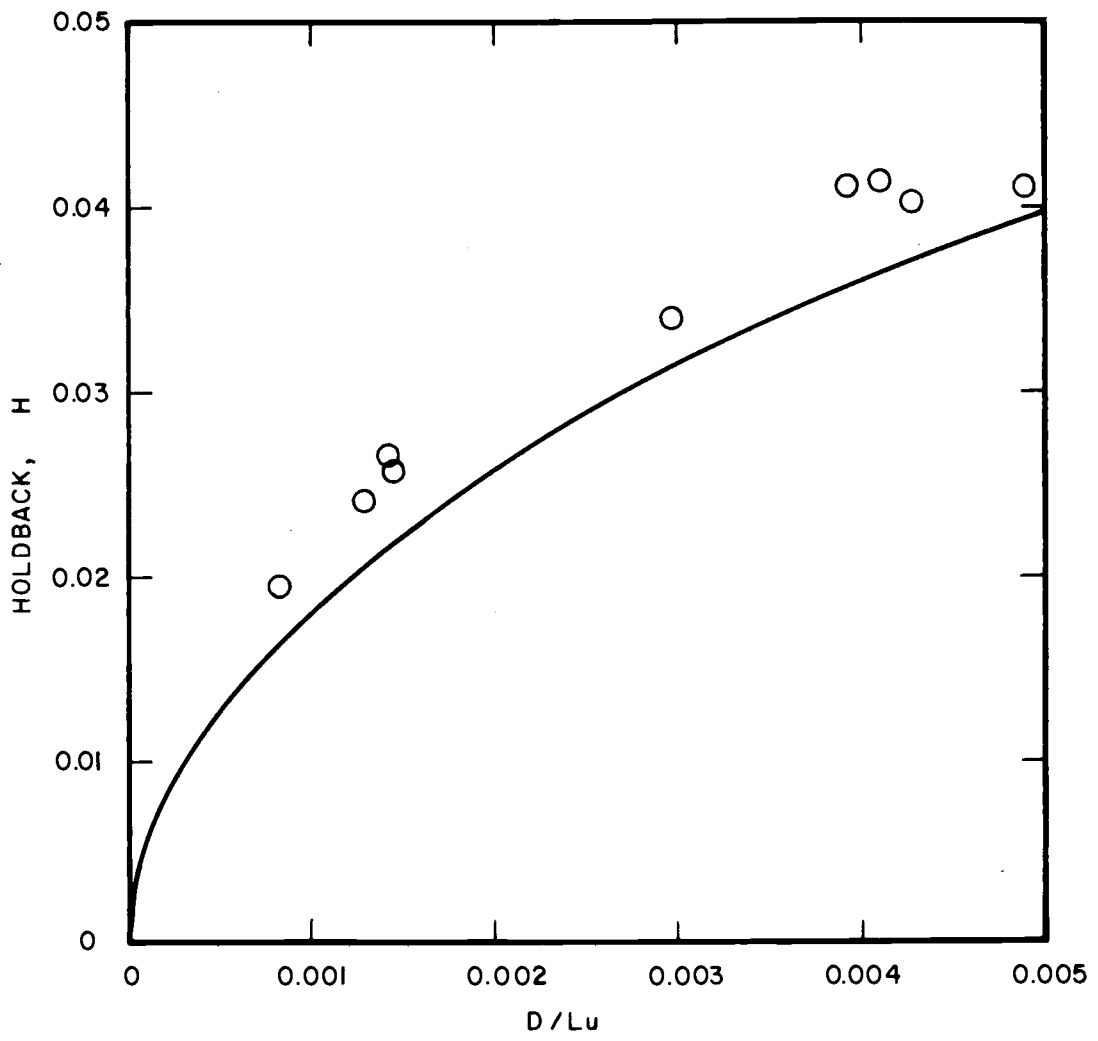
Fig. 54: H vs D/Lu FOR ICE

Effect of Varying Flow Rate -- The performance of the
column with varying wash rate is as expected. Hold-back and
D/Lu increased somewhat with flow rate, but the increase was
not linear. The reason for this behavior has been discussed.

Specifically, increasing the wash rate from 50 to 130
lb/(hr-ft²) caused nearly a 20 percent increase in the hold-
back, and a 67 percent increase in D/Lu. This four-fold change
in flow rate should have caused only a 32 percent increase in
D/Lu. The reason for this deviation from the predicted behavior
is again probably due to variations in the size of ice crystals
between runs.

Effect of Varying Column Height -- The performance of the
column as the slush height was varied shows the trends predicted
by theory although the serious scatter of the data plotted on
Figures 48 and 49 makes quantitative interpretation difficult.
At constant flow rate, D/Lu should vary inversely with L;
alternatively 1/(D/Lu) should vary directly with column height.
Figure 49 indicates that the proper trend is followed. Again
it is probable that variations in the size of crystals between
runs contributed to the serious scatter.

Flooded Bed vs. Drained Bed -- There seems to be no question
that washing in a flooded bed is far superior to washing in a
drained bed. In a flooded bed, water tends to displace salt in
a uniform manner with little channeling. The difference in the
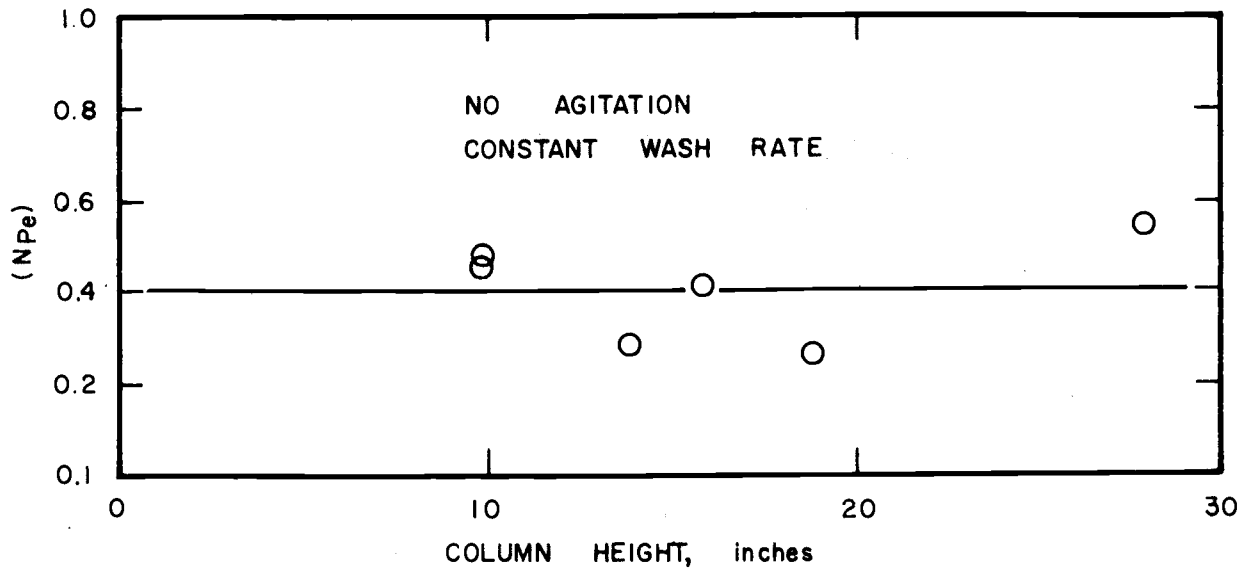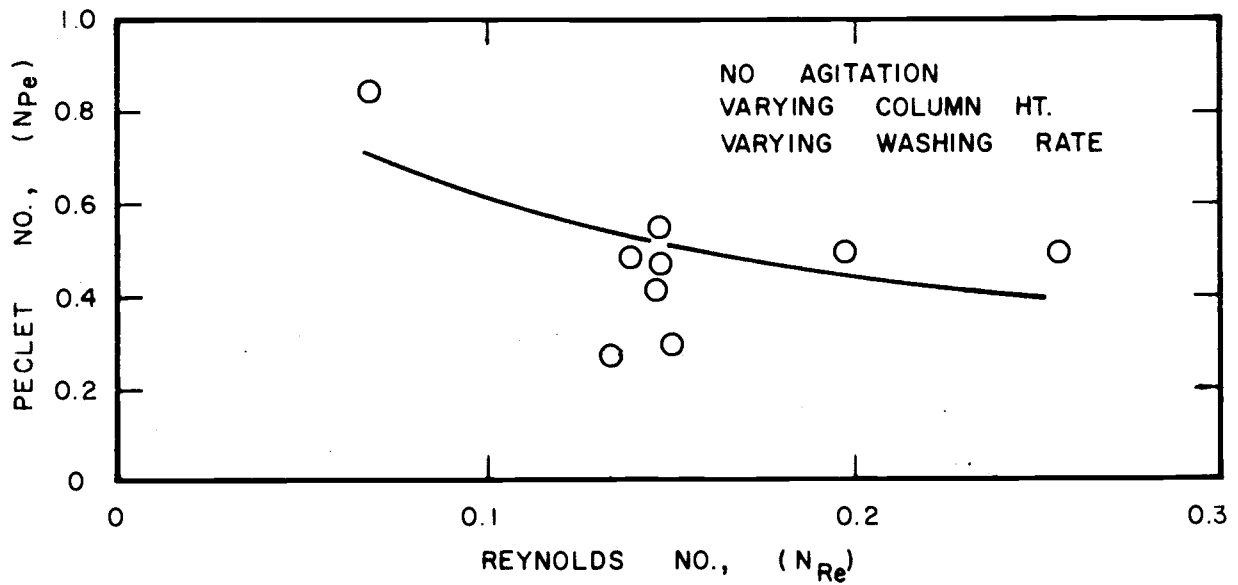specific gravity of brine and water stabilizes the advancing

Fig. 55: ICE

Fig. 56: ICE

front. In a drained bed, channeling with resultant tailing
is very serious.

Peclet Number -- If the ice crystal size had remained
constant between runs, it should have been possible to test the
theoretical prediction that N_{Pe} should vary as $N_{Re}^{-0.2}$, and
that N_{Pe} should be independent of column height. Unfortunately
the ice crystal size did vary between runs.

To compare the general range of Peclet numbers obtained,
the Peclet number was calculated for all runs based on an
effective particle size. Since the "size" of the crystals was
between 0.4 and 0.8 millimeters in most runs, an effective
"diameter" of 0.5 millimeters was used to calculate a Peclet
number. The results of this calculation were entered in Table 2,
and are presented in Figures 55 and 56. As can be seen, Peclet
numbers in the range of 0.25-0.85 were obtained. Figure 55
shows that the Peclet number does not appear to be a function of
column height. Figure 56 shows the general trend of decreasing
Peclet number with increasing Reynolds number. Once more the
scatter of the data is probably due to differences in the size
of ice crystals between runs.

Conclusions -- Probably the most significant finding in the
ice-washing tests was that the process can be characterized by
a model involving a simple conbination of piston flow and
dispersion at the water-brine interface. The fact that most

of the data fell on a straight line when plotted in the form

$$Z_1 \text{ vs. } \frac{1 - v\theta/V_o}{2\sqrt{v\theta/V_o}}$$ lends strong support to the simple dispersion

model. The form of the intensity function confirmed this

behavior.

The work on ice crystals proves that the simple dispersion

model is correct, and strongly suggests that previous studies of

fluid flow in packed beds can be directly applied to the

washing problem. Because of the difficulty of characterizing

the size of ice crystals, and because of differences in crystal

size between runs, it was not possible to show conclusively that

the Peclet numbers established as correct in packed beds would

also apply to flooded beds of ice crystals. However, all the

relations predicted for packed beds seem to apply. It was

shown that both hold-back and D/Lu should be only a weak

function of the wash rate, as predicted by the simple theory.

It was shown that agitation promoted axial mixing, and was not

necessary to remove brine trapped around and between the

crystals. It was likewise demonstrated that a tall column

should work better than a short column, and that $1/(D/Lu)$ should

vary directly with column height, again as predicted by theory.

Finally it was shown that washing in a flooded bed is far

superior to washing in a drained bed.

VI. CALCULATED PERFORMANCE OF WASH COLUMNS

The purpose of this section is to predict the washing
efficiency under ideal conditions in which only one variable at
a time is changed. It is hoped that the calculated performance
under various operating conditions will permit a better
understanding of column performance.

Wash Water Wasted

The calculated performance under ideal conditions is
particularly useful in predicting the amount of wash water
wasted. Because errors in measuring the void volume were of the
order of 3-5 percent, this quantity was difficult to measure
experimentally. (A 3-5 percent error in measuring the void
volume would produce a very large error in the magnitude of the
wash water wasted.) For this reason, the quantity of wash
water wasted to produce potable water (500 ppm of salt) was
not reported. Instead, the theoretical behavior under various
operating conditions was calculated, and is presented in this
section.

Prediction of D/Lu

If the ice particles were spherical and of uniform size,
the work of previous investigators in packed beds could be
used to predict the dispersion coefficient. Because of the
non-uniform size distribution and non-spherical shape of ice
particles, it is as yet not possible to predict the dispersion
coefficient D. However, it should be possible to calculate the

effect of changed operating conditions if the value of D/Lu
is known under one set of operating conditions.

Basis for Calculations

As a basis for the calculations, a set of standard opera-
ting conditions was assumed. As set forth in Table 3 below,
these operating conditions would be expected to result in a value
of D/Lu of 0.0015. If the value of D/Lu is known under this
particular set of conditions, the value under any other
condition can be readily calculated.

TABLE 3

STANDARD WASHING CONDITIONS

Column Height	28 inches
Nominal Crystal Size	0.5 millimeters
Column Diameter	4 inches
Ice in Charge	29 percent/by weight
Initial Salt Concentration	4 percent
Wash Rate, uρ	200 lb/(hr ft^2)
D/Lu	0.0015
Salt Concentration in Wash Water	0
Salt Concentration in Potable Water	500 ppm

The data of Harleman, Mehlhorn, and Rumer (13) are par-
ticularly valuable in predicting the effect of wash rate and
particle size on the dispersion coefficient. These investigators

showed that N_{Pe} varies inversely as $(N_{Re})^{0.2}$. Assuming that this relation holds, and that the standard conditions are as in Table 3, the dispersion coefficient under any other conditions can be calculated by the following relation: (for derivation of this equation see Appendix II).

$$\frac{D}{Lu} = 0.0015 \left(\frac{d_p}{0.5}\right)^{1.2} \left(\frac{u}{200}\right)^{0.2} \left(\frac{28}{L}\right) \tag{16}$$

Calculation of Wash Water Wasted

Figure 57 is a plot of wash water wasted vs. D/Lu. (For convenience, the hold-back curve is also shown.) The calculation of the amount of wash water wasted is based upon batch washing a slush containing 29 percent ice and 4 percent salt. It was intended that, after washing, the ice in the column would be melted, and that the resultant mixture of melted ice and wash water contained in the voids and would contain 500 ppm of salt. It will be noted that, under the standard conditions assumed above, 0.024 pounds of water would be wasted per pound of ice washed.

This curve was used in conjunction with Equation 16 to predict the performance of the column under various conditions. It can be seen that relationship between wash water wasted and D/Lu is almost linear. It will be noted that the curve does not pass through the origin; thus a negative amount of wash water is predicted for perfect piston flow (D/Lu = 0). The reason for this apparently anomalous result is that the calculations are based on using pure water for washing; hence no

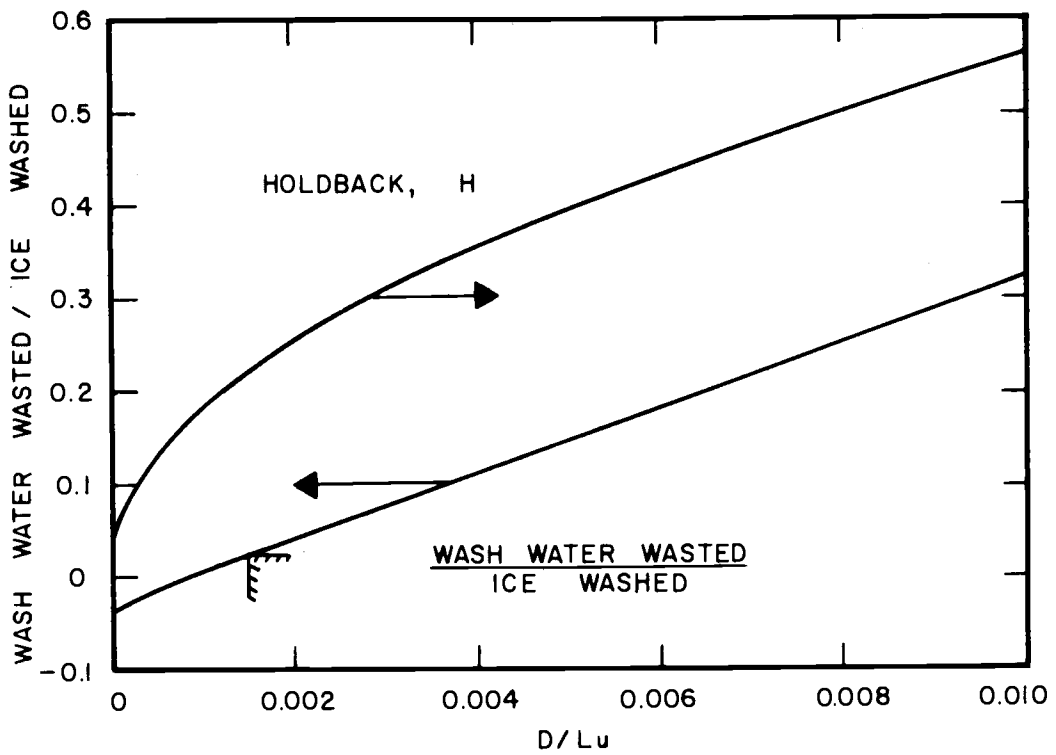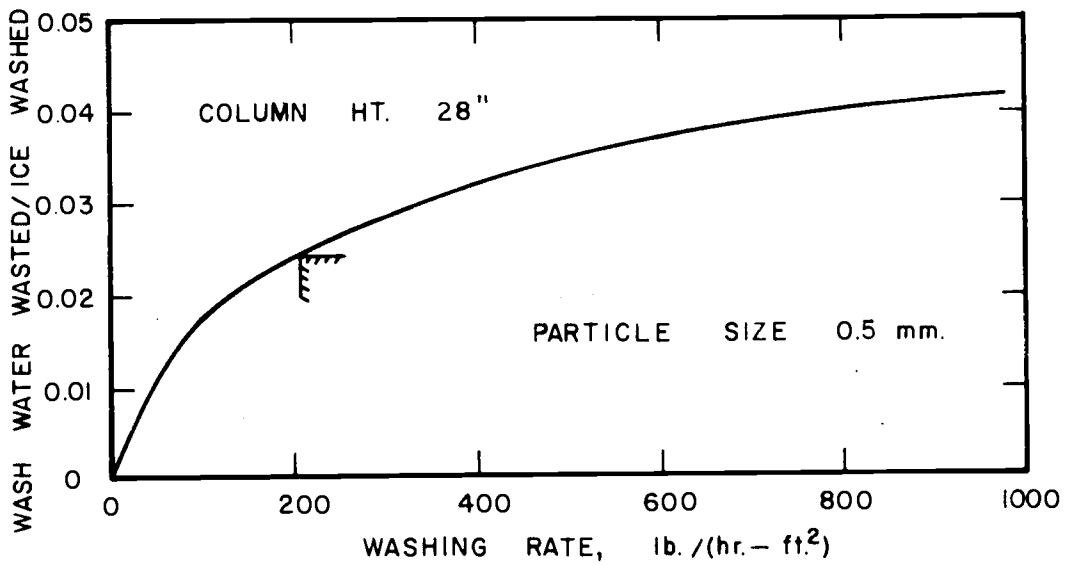Fig. 57: CALCULATED ICE—WASHING CURVES

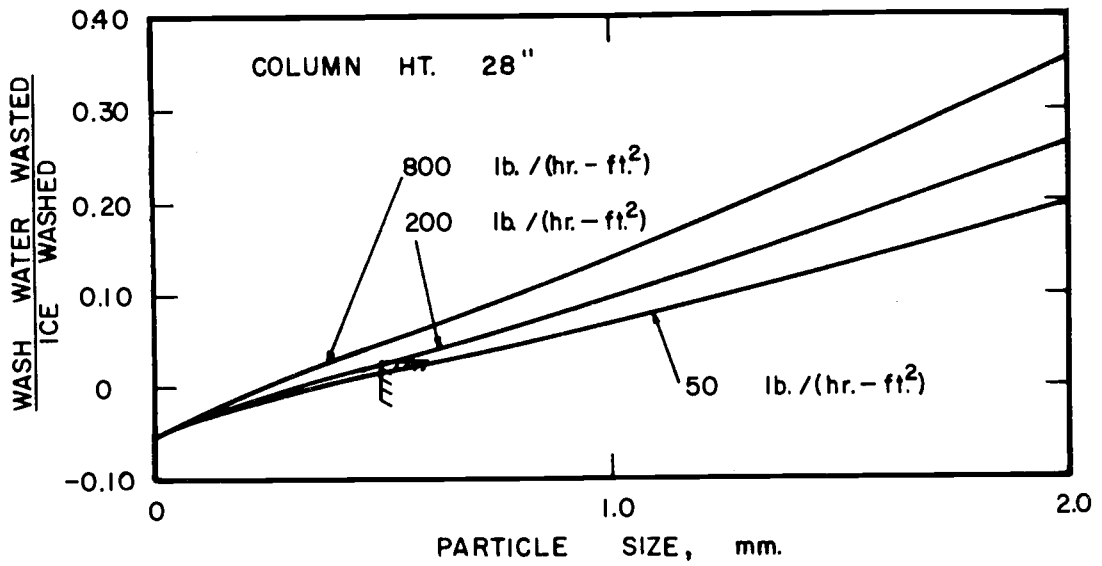Fig. 58: CALCULATED EFFECT OF WASH RATE
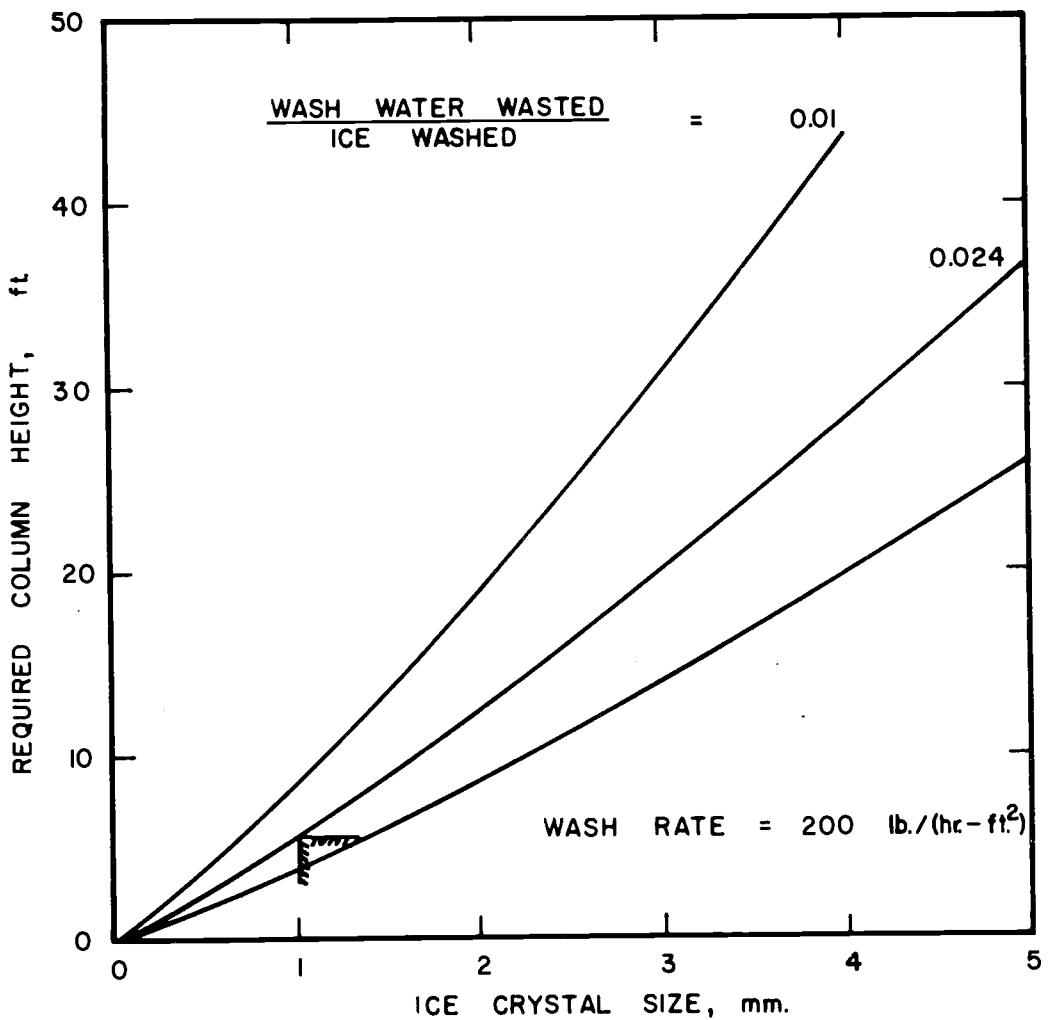
Fig. 59: CALCULATED EFFECT OF PARTICLE SIZE

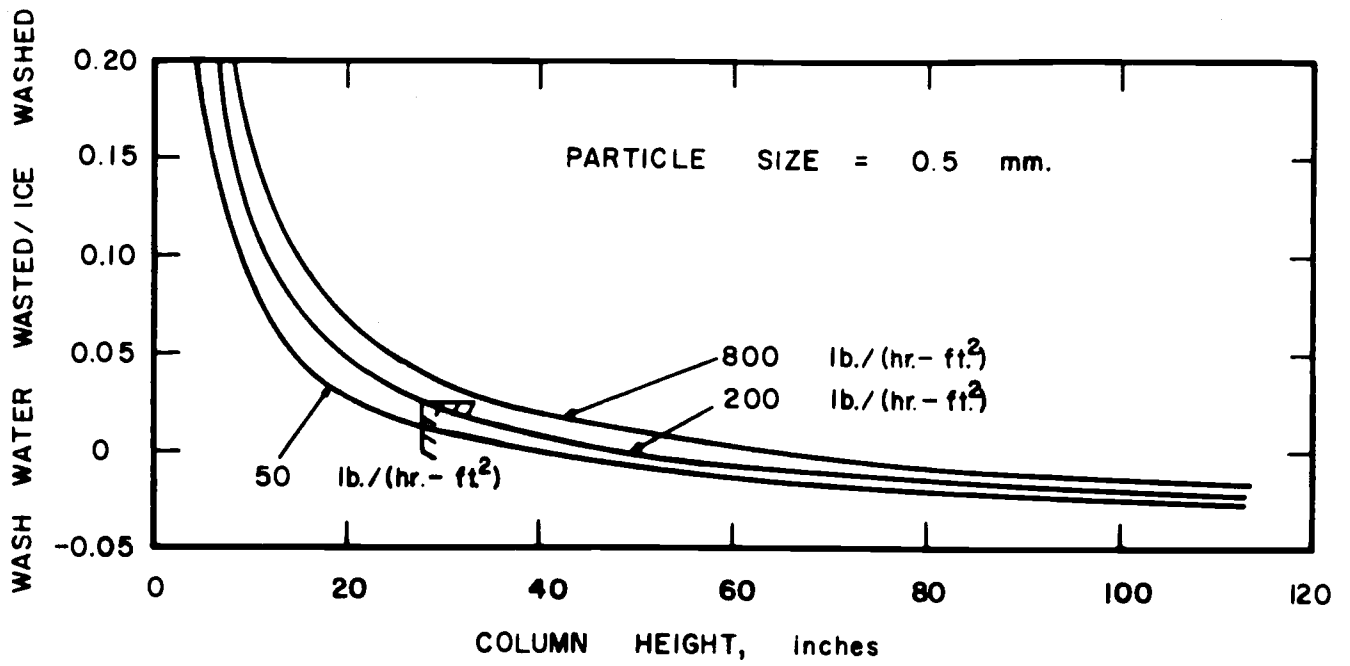Fig. 60: CALCULATED EFFECT OF CRYSTAL SIZE ON
COLUMN HEIGHT

Fig. 61: CALCULATED EFFECT OF COLUMN HEIGHT

net wash water is required at a finite value of D/Lu because
some pure water can be diluted with salt and still the purity
requirement of 500 ppm can be met.

Effect of Varying Wash Rate

The effect of varying the wash rate is seen in Figure 58.
Again, standard conditions are as listed in Table 3. It can
be seen that 0.024 pounds of wash water are wasted at a wash
rate of 200 lb/(hr-ft^2), which corresponds to a value of
D/Lu = 0.0015. In preparing this curve, it was assumed that
all variables except wash rate would be held as in Table 3.
As can be seen from Equation 16, D/Lu varies as the 0.2 power
of the wash rate. This relation was used together with Figure 57
to obtain Figure 58. This curve demonstrates that wash water
wasted is a weak function of wash rate, in conformance with
experimental results showing that washing rate does not have a
very strong effect on the value of D/Lu.

Effect of Varying Crystal Size

Figure 59 demonstrates the effect of particle size upon
wash water wasted, with wash rate as a parameter. Again the
base case is indicated on the graph. The curves indicate that
washing is more effective for small particles than for large
ones. Thus, for a given column height and wash rate the wash
water wasted increases approximately in proportion to the 1.2
power of the particle diameter, as shown in Figure 59. Looked
at another way, for a fixed amount of wash water wasted, the

required column height varies directly as the 1.2 power of the particle diameter as shown in Figure 60. Thus large ice crystals will require long columns for efficient washing. This would appear to be contrary to the view sometimes suggested that large crystals should be easier to wash. However, other factors in addition to the required column height will influence the economics of the washing process. For example, the capacity of the washing column would be expected to be greater for large ice crystals. Since washing efficiency is only a weak function of wash rate, wash columns will probably be designed to operate at capacities near the flooding capacity, and this will result in higher through-puts for larger ice crystals.

The drag F_k on a spherical particle is given by Stokes' Law as

$$F_k = 3\pi\mu d_p u \qquad (17)$$

where μ is the viscosity, d_p is the particle diameter and u is the linear velocity. The buoyant force F_s is

$$F_s = \frac{1}{6}\pi d_p^3 \Delta\rho \qquad (18)$$

where $\Delta\rho$ is the difference in density of the particle and the fluid. The "fluidization velocity" can be estimated by equating F_s and F_k:

$$u_{max} = \frac{1}{18} \frac{d_p^2 \rho g}{\mu} \qquad (19)$$

This indicates that the fluidization velocity varies as the square of the radius. If this velocity is exceeded, the particles will be dragged down in a down-flow column. This velocity is then the maximum allowable velocity in a flooded bed.

This analysis suggests that the required wash column cross-sectional area would be expected to decrease as the inverse square of the ice crystal size. Since the required column height varies directly as the 1.2 power of the particle size and directly as the 0.2 power of the wash water velocity, the volume of the washing column would vary as the inverse 0.4 power of the particle size. Thus large ice crystals will require longer columns but less column volume for a fixed amount of wash water wasted. It is not clear from these preliminary considerations how the cost of the washing column will vary with the ice crystal size.

There is another effect of ice crystal size which suggests an advantage for large crystals. A bed of large crystals can be drained of a greater percentage of the brine than a bed of small crystals can. It may be possible to capitalize on this by draining the ice crystals before their introduction into a continuous, countercurrent flooded wash column.

Effect of Varying Column Height

Figure 61 shows the effect of varying column height upon the performance of the wash column, with wash water rate as a

parameter. Again the base case is indicated on the graph. The curves clearly show that an increase in the column height is beneficial.

Whether the theoretical curves calculated for the 4-inch column would apply to larger commercial-size columns is not definitely known, but wall effects are expected to be minor, and the results should be applicable to large columns, unless unexpected serious channeling develops.

A   area, ft^2

c   effluent concentration at time θ, lb/ft^3

c_o initial salt concentration lb/ft^3

D   dispersion coefficient

d_p particle diameter, ft

erf Z $\dfrac{2}{\sqrt{\pi}} \displaystyle\int_o^z e^{-y^2}\, dy$

H   hold-back, Equation 9

HTU   height of a theoretical unit

$I(\theta)$ intensity function; Equation 10

L   length of bed, ft

ln   logarithm to the base e

n   number of vessels

N_{Pe} Peclet number, $\dfrac{d_p u}{D}$

N_{Re} Reynolds number, $\dfrac{d_p u \rho}{\mu}$

u   linear (interstitial) velocity, $\dfrac{u_o}{\epsilon}$, ft/hr

u_o superficial velocity, ft/hr

v   volumetric flow rate, ft^3/hr

V_o void volume, ft^3

Z_1 1st order term in egn. for fluid displacement (see egn.3)

Z_2 2nd order term in egn. for fluid displacement (see egn.5)

ϵ void fraction

θ time, hr

$\bar{\theta}$ mean residence time, hr

μ viscosity, lb/(hr)(ft)

ρ density, lb/ft^3

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Appendix I

Normalized Intensity Function

The intensity function $I(\theta)$ is

$$I(\theta) = -\frac{d}{d\theta} \ln \frac{c(\theta)}{c_o} \tag{1}$$

$$d\left(\frac{v\theta}{V_o}\right) = \frac{v}{V_o} \, d\theta \tag{2}$$

Thus

$$d\theta = \frac{V_o}{v} \, d\left(\frac{v\theta}{V_o}\right) = \frac{1}{\bar{\theta}} \, d\left(\frac{v\theta}{V_o}\right) \tag{3}$$

substituting (3) in (1)

$$\frac{I(\theta)}{\bar{\theta}} = -\frac{d}{d\left(\frac{v\theta}{V_o}\right)} \ln \frac{c(\theta)}{c_o} \tag{4}$$

Thus the normalized intensity function is simply the negative of the slope of the curve of $\ln\frac{c}{c_o}$ vs. $\frac{v\theta}{V_o}$.

Appendix II

Variation of D/Lu With Operating Conditions

The Peclet number, N_{Pe}, varies as $N_{Re}^{-0.2}$, therefore

$$N_{Pe} = \frac{K}{N_{Re}^{0.2}} \tag{1}$$

where K is a constant. Expanding

$$\frac{d_p u}{D} = \frac{K}{\left(\frac{d_p u \rho}{\mu}\right)^{0.2}} = \frac{K'}{\left(d_p u\right)^{0.2}} \tag{2}$$

Rearranging (2) gives

$$D = \frac{1}{K'} \left(d_p u\right)^{1.2} \tag{3}$$

Dividing (3) by Lu

$$\frac{D}{Lu} = \frac{1}{K'} \frac{\left(d_p u\right)^{1.2}}{Lu} = K'' \frac{\left(d_p\right)^{1.2} u^{0.2}}{L} \tag{4}$$

Thus from the standard conditions of Table 3, page 68,

$$\frac{D}{Lu} = 0.0015 \left(\frac{d_p}{0.5}\right)^{1.2} \left(\frac{u\rho}{200}\right)^{0.2} \left(\frac{28}{L}\right)$$