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WASHING OF BRINE FROM
ICE CRYSTALS

BY
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
CAMBRIDGE, MASSACHUSETTS

OFFICE OF SALINE WATER
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WASHING OF BRINE FROM ICE CRYSTALS

by

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for

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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 principal 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 supercooled 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 interpretation and correlation of data on washing. The washing studies herein reported were carried out by "batch" operation, that is, by washing brine from a fixed bed of ice-brine mixture. This is experimentally simpler than countercurrent 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 $V_0$. The voids are filled with brine having a salt concentration $c_0$. 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

$$\frac{c}{c_0} = \frac{1}{2} + \frac{1}{2} \text{erf} \left[ \frac{1 - \frac{v \theta}{V_0}}{\sqrt{4D/v \theta \cdot \frac{u}{L}}} \right]$$
Fig. 1: Concentration Profiles
In previous residence-time distribution studies, the data were successfully correlated by describing the Peclet number, $N_{\text{Pe}}$, as a function of the Reynolds number, $N_{\text{Re}}$. The Peclet number is defined as

$$N_{\text{Pe}} = \frac{d_p u}{D}$$

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

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

where $d_p$ is the particle diameter, $u$ is the linear velocity of the fluid past the particle, $\rho$ is the fluid density, and $\mu$ 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 be characterized by a model involving a simple combination 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 correlations 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 excellent 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. INTRODUCTION

The Freezing Process

A general discussion of the freezing process for desalination 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 empiricism 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 understanding 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, $V_0$. The voids are filled with brine having a salt concentration $c_0$. 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 $c_0$ to 0 at the instant the water "front" reaches the outlet, that is at $\theta = V_0/v$ (or $\theta V_0/V_0 = 1$), as shown in Figure la. This "piston flow" is the ideal case, since all of the wash water can be recovered with the melted ice.

Ideal piston flow (Figure la) never occurs, since there is always some longitudinal mixing due to viscous effects and to molecular or eddy diffusion. Figure lb illustrates the departure from ideal piston flow caused by longitudinal mixing. Figure lc 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 ld illustrates still another possibility -- non-uniform flow with channeling in which stagnation occurs.

Danckwerts (I) 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

$$\frac{c}{c_0} = \frac{1}{2} + \frac{1}{2} \operatorname{erf} \left( \frac{1 - v \theta / V_0}{2 \sqrt{\frac{v \theta D}{V_0 Lu}}} \right)$$

(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 $u_0/\varepsilon$, where $u_0$ is the mean or superficial velocity, and $\varepsilon$ 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

$$\frac{c}{c_0} = \frac{1}{2} + \frac{1}{2} \operatorname{erf} Z_1$$

(2)

where

$$Z_1 = \frac{1 - v \theta / V_0}{2 \sqrt{\frac{v \theta D}{V_0 Lu}}}$$

(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 (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/co versus vθ/Vo 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 throughput as indicated by Figure 1b.

Rifai, Kaufman, and Todd (2) 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

\[ \frac{c}{c_0} = \frac{1}{2} + \frac{1}{2} \operatorname{erf} Z_1 - \frac{1}{2} e^{\frac{Lu}{D}} (1 - \operatorname{erf} Z_2) \]  \hspace{1cm} (4) \]

where \( Z_1 \) is given by Equation 3, and \( Z_2 \) is given by

\[ Z_2 = \frac{1 + \frac{\nu \theta}{V_0}}{2\sqrt{\frac{\nu \theta D}{V_0} \frac{L}{D}}} \]  \hspace{1cm} (5) \]

As pointed out by these investigators, the term \( e^{\frac{Lu}{D}} \) is extremely large when the term \( (1 - \operatorname{erf} Z_2) \) 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:

\[ \operatorname{erf} Z_2 = 1 - \frac{e^{-Z_2^2}}{Z_2 \sqrt{\pi}} \left( 1 - \frac{1}{2Z_2^2} + \frac{1.3}{(2Z_2^2)^2} - \frac{1.3.5}{(2Z_2^3)^3} + \ldots \right) \]  \hspace{1cm} (6) \]

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

\[ \frac{c}{c_0} = \frac{1}{2} + \operatorname{erf} Z_1 - \frac{e^{-Z_2^2}}{2Z_2 \sqrt{\pi}} \]  \hspace{1cm} (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 $v\theta/V_0$ 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 $v\theta/V_0$ 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 $V_0$. Danckwerts (7) suggests that the value of $D$ is more easily determined from the slope of the $c/c_0$ curve at $v\theta/V_0 = 1$, by using the relation

$$
\left[ \frac{d\left(\frac{c}{c_0}\right)}{d\left(\frac{v\theta}{V_0}\right)} \right]_{v\theta/V_0=1} = -\frac{1}{2} \frac{Lu}{\sqrt{\pi D}} \tag{8}
$$

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 $\frac{1 - v\theta/V_0}{2 \sqrt{v\theta/V_0}}$. As can be seen from Equation 3, this plot should yield a straight line
passing through the origin with a slope equal to \( \frac{1}{\sqrt{D/Lu}} \). An important advantage of this method is that small errors in the determination of the void volume \( V_0 \) do not lead to correspondingly large errors in the dispersion coefficient \( D \). An unexpected advantage of this method of determining the dispersion 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 conveniently defined as the area above the curve of \( c/c_0 \) between \( v\theta/V_0 = 0 \), and \( v\theta/V_0 = 1 \)

\[
H = 1 - \int c/c_0 \, d \left( \frac{v\theta}{V_0} \right)
\]  

(9)
This area is denoted by $A_1$ in Figure 2 below.

![Diagram](image)

**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(θ) 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:

\[ I(\theta) = -\frac{d}{d\theta} \ln \frac{c(\theta)}{c_0} \]  

(10)

Thus I(θ) dθ is the probability that a particle in the system for a time θ will leave during the period θ to θ + dθ. Usually the intensity function and time are plotted in normalized form by plotting I/\bar{\theta} vs \( \theta/\bar{\theta}/V_0 \), where \( \bar{\theta} \) is the average residence time \( V_0/v \). The quantity of I/\bar{\theta} is readily obtained by taking the slope of the curve of \( \ln c/c_0 \) vs. \( \theta/\bar{\theta}/V_0 \) at any point (See Appendix I). This normalized intensity function often gives information difficult to see on the usual plot of \( c/c_0 \) vs \( \theta/\bar{\theta}/V_0 \), 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(θ) dθ 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 $d\theta$, 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 $I(\theta)$ 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 $(I(\theta)/\bar{\theta} \rightarrow n)$ which is equal to the number of vessels.

Another typical case is that of piston flow with superimposed 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 $\theta$ is increased. Instead, the intensity function grows without bounds.

The intensity function is particularly useful in determining 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. \( \nu \theta/V_0 \). 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 \( \frac{c}{c_0} \) vs. \( \nu \theta/V_0 \) 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
Thus

\[ \int_0^{\infty} \frac{c}{c_0} d\left(\frac{v\theta}{V_0}\right) = 1 \]  

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 \( v\theta/V_0 = 1.0 \). 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 \( c/c_0 \) vs. \( v\theta/V_0 \) will cross the line \( v\theta/V_0 = 1.0 \) at a value of \( c/c_0 = 0.50 \), since \( \text{erf}(0) = 0 \). Actually the curve always intersects at a value of \( c/c_0 \) 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 \( c/c_0 \).

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

\[
N_{Pe} = \frac{d_p u}{D}
\]

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

\[
N_{Re} = \frac{d_p u \rho}{\mu}
\]

where \( d_p \) is the particle diameter, \( u \) is the linear velocity (superficial velocity divided by void fraction) of the fluid past the particle, \( \rho \) is the density, and \( \mu \) 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 dispersion 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 longitudinal 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
Fig. 4: Dispersion of salt brine in beds of spheres (13)

$N_{Re} = d_p u / \nu$

$N_{Re} = d_p u / \nu$

$N_{Re} = d_p u / \nu$

$N_{Re} = d_p u / \nu$

0.001
0.01
0.05
0.1

0.5
1.0
5.0
10.0

$N_{Pe} = d_p u / D$

$N_{Pe} = d_p u / D$

$N_{Pe} = d_p u / D$

$N_{Pe} = d_p u / D$

$N_{Pe} = d_p u / D$

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$N_{Pe} = d_p u / D$
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

WASH-WATER DISTRIBUTOR

WASH COLUMN

RESERVOIRS

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 \( \frac{3}{4} \) 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 \(-10^\circ C\) and a 3-percent salt solution freezes at about \(-2^\circ C\). 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 \(8^\circ C\) to \(3^\circ C\). After this change, all the ice was formed as discrete particles and ball formation ceased.
Ice-Washing -- 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 allow 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
ICE - REMOVING SPOUT

VARIABLE SPEED STIRRER, RADIAL SPINES 1" APART, OFFSET 90°

100-MESH SCREEN

BYPASS

2-GALLON WASHWATER RESERVOIR

3/8" COPPER TUBING

POSITIVE DISPLACEMENT PUMP, VARIABLE SPEED

SAMPLING POINT

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>
<thead>
<tr>
<th>Run</th>
<th>Type</th>
<th>Stirrer Speed</th>
<th>Wash Rate</th>
<th>$\frac{c}{c_0}d(v\theta/V_0)$</th>
<th>Holdback, H.</th>
<th>D/Lu</th>
<th>$N_{Re}$</th>
<th>$N_{Pe}$</th>
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</thead>
<tbody>
<tr>
<td>B-3</td>
<td>P.E.</td>
<td>110</td>
<td>174</td>
<td>1.13</td>
<td>0.112</td>
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<td>2.68</td>
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<td>B-4</td>
<td>P.E.</td>
<td>110</td>
<td>543</td>
<td>1.01</td>
<td>0.075</td>
<td>0.0200</td>
<td>8.37</td>
<td>0.326</td>
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<td>P.E.</td>
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<td>1460</td>
<td>0.90</td>
<td>0.044</td>
<td>0.0118</td>
<td>22.50</td>
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</tr>
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<td>354</td>
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<td>0.0050</td>
<td>5.46</td>
<td>1.32</td>
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<td>32</td>
<td>355</td>
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<td>0.0082</td>
<td>5.47</td>
<td>0.796</td>
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<td>B-10</td>
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<td>59</td>
<td>374</td>
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<td>0.053</td>
<td>0.0162</td>
<td>5.77</td>
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<td>B-11</td>
<td>P.E.</td>
<td>90</td>
<td>375</td>
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<td>0.052</td>
<td>0.0125</td>
<td>5.78</td>
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<td>B-12</td>
<td>P.E.</td>
<td>6</td>
<td>380</td>
<td>0.99</td>
<td>0.034</td>
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<td>5.86</td>
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<tr>
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<td>P.E.</td>
<td>120</td>
<td>382</td>
<td>1.04</td>
<td>0.083</td>
<td>0.0317</td>
<td>5.89</td>
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<tr>
<td>B-14</td>
<td>P.E.</td>
<td>6</td>
<td>1510</td>
<td>0.90</td>
<td>0.040</td>
<td>0.0135</td>
<td>23.50</td>
<td>0.484</td>
</tr>
<tr>
<td>B-21</td>
<td>Scotch.</td>
<td>60</td>
<td>454</td>
<td>1.00</td>
<td>0.081</td>
<td>0.0185</td>
<td>0.64</td>
<td>0.0311</td>
</tr>
<tr>
<td>B-22</td>
<td>Scotch.</td>
<td>60</td>
<td>204</td>
<td>0.99</td>
<td>0.093</td>
<td>0.0185</td>
<td>0.29</td>
<td>0.0311</td>
</tr>
<tr>
<td>B-28</td>
<td>Scotch.</td>
<td>30</td>
<td>439</td>
<td>1.02</td>
<td>0.036</td>
<td>0.0040</td>
<td>0.62</td>
<td>0.144</td>
</tr>
<tr>
<td>B-29</td>
<td>Scotch.</td>
<td>90</td>
<td>430</td>
<td>1.04</td>
<td>0.024</td>
<td>0.0020</td>
<td>0.61</td>
<td>0.288</td>
</tr>
<tr>
<td>B-30</td>
<td>Scotch.</td>
<td>0</td>
<td>423</td>
<td>1.03</td>
<td>0.022</td>
<td>0.0017</td>
<td>0.60</td>
<td>0.339</td>
</tr>
<tr>
<td>B-31</td>
<td>Scotch.</td>
<td>0</td>
<td>856</td>
<td>1.02</td>
<td>0.047</td>
<td>0.0090</td>
<td>1.21</td>
<td>0.064</td>
</tr>
</tbody>
</table>
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 \( \int \frac{c}{c_0} d\left(\frac{v\theta}{V_0}\right) \) must be unity, thus the area above the curve to the left of the line \( \frac{v\theta}{V_0} = 1 \) 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 \( \frac{v\theta}{V_0} = 1 \) at a value of \( c/c_0 \) 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-II 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 $\frac{1 - v\theta/V_0}{2\sqrt{v\theta/V_0}}$, 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 $c/c_0$ close to unity, since, in this range, small errors in measuring the concentration lead to large errors in the quantity $Z_1$. Points near $c/c_0 = 0$ 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 $H$ corresponds to a high value of $D/Lu$ and a highly skewed curve of $c/c_0$ vs. $v\theta/V_0$. 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 ($\sim 365$ 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 polyethylene 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

H = 0.0807
D/\text{Lu} = 0.0185

c/c_0

\nu \theta / \nu_0

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

H = 0.0364
D/\text{Lu} = 0.0040

c/c_0

\nu \theta / \nu_0
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 \( Z_i \) vs. \( \frac{1 - \frac{v}{V}}{2 \sqrt{\frac{v}{V} + \frac{v_0}{V_0}}} \), as shown in Figures 22-25. Although there is still some question as to whether the dispersion 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 \( \ln \frac{c}{c_0} \) vs. \( \frac{v\theta}{V_o} \) (Figure 30), and then plotting the slope of this curve vs. \( \frac{v\theta}{V_o} \) (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 overshadows 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 \( Z \) vs. 
\[
\frac{1 - v\theta/V_0}{2\sqrt{v\theta/V_0}}
\]
yielded a straight line. In addition, the shape of the curve of intensity function versus \( v\theta/V_0 \) 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 \( Z \) vs. 
\[
\frac{1 - v\theta/V_0}{2\sqrt{v\theta/V_0}}
\]
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
SOLID LINE — CALCULATED FROM EQUATIONS (1) and (9)

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>
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<th>I-5</th>
<th>I-6</th>
<th>I-7</th>
<th>I-8</th>
<th>I-12</th>
<th>I-13</th>
<th>I-14</th>
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<th>I-17</th>
<th>I-10</th>
<th>I-15</th>
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<td>Remarks</td>
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<td>VARIABLE COLUMN HEIGHT</td>
<td>VARIABLE STIRLER SPEED</td>
<td>PULSING</td>
<td>DRAINED BED</td>
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<td>137</td>
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<td>10</td>
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<td>0.197</td>
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<td>0.135</td>
<td>0.148</td>
<td>0.139</td>
<td>0.126</td>
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</tr>
</tbody>
</table>
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 dimension 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).
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 \( \frac{v\theta}{V_0} = 1 \) must be
**Fig. 36: RUN I-6, ICE**

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

**Fig. 37: RUN I-8, ICE**

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

Fig. 39: RUN I-12, ICE

H = 0.0413
D/Lu = 0.0041
NO AGITATION
COLUMN HT. 10''
137 lb./(hr.-ft.²)

H = 0.0865
D/Lu = 0.0414
AGITATOR SPEED 8 rpm.
COLUMN HT. 28''
136 lb./(hr.-ft.²)
Fig. 40: RUN I-6, ICE

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

Fig. 43: RUN I-12, ICE
Fig. 44: ICE

**Graph 1:**
- **Y-axis:** Holdback, H
- **X-axis:** Agitator Speed, rpm
- **Conditions:** Column Height 28"; Flow Rate ~ 135 lb./hr-ft²

Fig. 45: ICE

**Graph 2:**
- **Y-axis:** D/Lu
- **X-axis:** Pulse Rate, pulses/min.
- **Conditions:** Column Height 28"; Flow Rate ~ 130 lb/(hr-ft²)
Fig. 46: ICE

Fig. 47: ICE
**Fig. 48: ICE**

WASH RATE = 140 lb./(hr. ft.²)
NO AGITATION

**Fig. 49: ICE**

FLOW RATE = 140 lb./(hr. ft.²)
NO AGITATION
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 \( \frac{c}{c_0} \) should have a value of very nearly 0.50 when \( \frac{v\theta}{V_0} = 1 \). Fairly consistently the curves of \( \frac{c}{c_0} \) vs. \( \frac{v\theta}{V_0} \) intersected the line \( \frac{v\theta}{V_0} = 1 \) 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. \( \frac{1 - \frac{v\theta}{V_0}}{2 \sqrt{\frac{v\theta}{V_0}}} \) 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 \( \frac{c}{c_0} = 1 \), or near \( \frac{c}{c_0} = 0 \). 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 ft²) 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 characterized 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 material 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, \( v_0 \), 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 \( \frac{1 - \frac{v\theta}{V_0}}{2\sqrt{\frac{v\theta}{V_0}}} \)

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 \( c/c_0 \) vs. \( v\theta/V_0 \) 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 conductivity 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 conductivity changes relatively slowly with concentration in this range. A simple solution to the problem is to dilute the concentrated 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.
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 assumption 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 $Z_l$ vs. $1 - v\theta/V_o$. 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 relatively 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

\[ Z \sim \frac{1 - \frac{v\Theta}{V_0}}{2\sqrt{\frac{v\Theta}{V_0}}} \]

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 \( c/c_0 \) vs. \( \frac{v\Theta}{V_0} \) 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 combination 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
\[ Z \propto \frac{1 - v\theta/V_0}{2 \sqrt{v\theta/V_0}} \]
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 operating 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**

<table>
<thead>
<tr>
<th>STANDARD WASHING CONDITIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Column Height</td>
</tr>
<tr>
<td>Nominal Crystal Size</td>
</tr>
<tr>
<td>Column Diameter</td>
</tr>
<tr>
<td>Ice in Charge</td>
</tr>
<tr>
<td>Initial Salt Concentration</td>
</tr>
<tr>
<td>Wash Rate, up</td>
</tr>
<tr>
<td>D/Lu</td>
</tr>
<tr>
<td>Salt Concentration in Wash Water</td>
</tr>
<tr>
<td>Salt Concentration in Potable Water</td>
</tr>
</tbody>
</table>

The data of Harleman, Mehlhorn, and Rumer (13) are particularly 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).

\[
\frac{D}{Lu} = 0.0015 \left( \frac{d_p}{0.5} \right)^{1.2} \left( \frac{u}{200} \right)^{0.2} \left( \frac{28}{L} \right)
\]  \hspace{1cm} (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

$$F_k = 3\mu d_p u$$  \hspace{1cm} (17)$$

where $\mu$ is the viscosity, $d_p$ is the particle diameter and $u$ is the linear velocity. The buoyant force $F_S$ is

$$F_S = \frac{1}{6\pi d_p^3} \Delta \rho$$  \hspace{1cm} (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 $F_k$:

$$u_{\max} = \frac{1}{18} \frac{d_p^2 \rho g}{\mu}$$  \hspace{1cm} (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 $t$, lb/ft$^3$
c$_0$  initial salt concentration lb/ft$^3$
D  dispersion coefficient
d  particle diameter, ft
erf $Z$  \[ \frac{2}{\sqrt{\pi}} \int_0^Z e^{-y^2} dy \]
H  hold-back, Equation 9
HTU  height of a theoretical unit
I($t$)  intensity function; Equation 10
L  length of bed, ft
ln  logarithm to the base e
n  number of vessels
N$_{Pe}$  Peclet number, $\frac{d п}{D}$
N$_{Re}$  Reynolds number, $\frac{d п u_p}{\mu}$
u  linear (interstitial) velocity, $\frac{u_0}{\epsilon}$, ft/hr
u$_0$  superficial velocity, ft/hr
v  volumetric flow rate, ft$^3$/hr
V$_0$  void volume, ft$^3$
Z$_1$  1st order term in eqn. for fluid displacement (see eqn.3)
Z$_2$  2nd order term in eqn. for fluid displacement (see eqn.5)
\(\epsilon\)  void fraction
\(\theta\)  time, hr
\(\bar{\theta}\)  mean residence time, hr
\(\mu\)  viscosity, lb/(hr)(ft)
\(\rho\)  density, lb/ft$^3$
Bibliography


Appendix I

Normalized Intensity Function

The intensity function $I(\theta)$ is

$$I(\theta) = -\frac{d}{d\theta} \ln \frac{c(\theta)}{c_0}$$  \hspace{1cm} (1)

$$d\left(\frac{v\theta}{V_o}\right) = \frac{v}{V_o} \ d\theta$$  \hspace{1cm} (2)

Thus

$$d\theta = \frac{V_o}{v} \ d\left(\frac{v\theta}{V_o}\right) = \frac{1}{\theta} \ d\left(\frac{v\theta}{V_o}\right)$$  \hspace{1cm} (3)

substituting (3) in (1)

$$\frac{I(\theta)}{\bar{\theta}} = -\frac{d}{d\left(\frac{v\theta}{V_o}\right)} \ \ln \frac{c(\theta)}{c_0}$$  \hspace{1cm} (4)

Thus the normalized intensity function is simply the negative of the slope of the curve of $\ln \frac{c}{c_0}$ 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

\[
\frac{d_p u}{D} = \frac{K}{(d_p u)^{0.2}} = \frac{K'}{(d_p u)^{0.2}} \tag{2}
\]

Rearranging (2) gives

\[
D = \frac{1}{K'} (d_p u)^{1.2} \tag{3}
\]

Dividing (3) by Lu

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

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

\[
\frac{D}{Lu} = 0.0015 \left( \frac{d_p}{0.5} \right)^{1.2} \left( \frac{u_p}{200} \right)^{0.2} \left( \frac{28}{L} \right)
\]