MASS TRANSFER STUDIES IN A VIBRATION MIXED REACTOR

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Fulfillment of the Requirements
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by
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CERTIFICATION OF THESIS WORK

We, the undersigned, certify that Tariq Mahmud candidate for the degree of Master of Science in Engineering (Chemical) has presented his thesis on the subject Mass Transfer studies in a Vibration Mixed Reactor, that the thesis is acceptable in form and content, and that the student demonstrated a satisfactory knowledge of the field covered by this thesis in an oral examination held on 2nd April, 1982.

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Mass transfer characteristics of a vibration mixed reactor has been studied by measuring the rate of evaporation of naphthalene particle. The effects of flow rate, particle arrangements and frequency of vibration on mass transfer rate have been studied.

The results of the present study are compared with the previous literature on mass transfer in fixed bed. It is found that the rate of mass transfer in this work is lower than the other investigations.

The results for three types of particles are different and the data cannot be correlated by using a simple equation. Therefore, separate correlations, relating $N_{Sh}$ and $N_{Re}$ have been developed for different particle sizes and arrangements.

Generally, stack arrangement of particles reduces mass transfer rate because of the "area blocking effect". In the present study data for different arrangements are brought together by using an effective area for mass transfer.
ACKNOWLEDGEMENTS

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Abstract

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Rational design of industrial reactor depends on reliable informations regarding the chemical reaction rate. The overall rate depends on both the rate of mass transfer and the intrinsic rate of chemical reaction. The rate data are generally procured by means of experiments with suitable laboratory scale reactor. In a reactor, transfer of mass and heat also occur during reaction and these physical rate processes may significantly affect the overall rate of chemical conversion. The flow pattern and mass and heat transfer characteristics of an industrial reactor are generally different from a laboratory reactor. The overall rate data from laboratory reactor cannot, therefore, be used directly to scale up an industrial reactor. The accepted practice for rational design of industrial reactor is to procure intrinsic chemical reaction rate data in the laboratory in absence of heat and mass transfer and then combine the intrinsic rate data with other physical rate processes, such as mass transfer and heat transfer rates to obtain the overall rate of chemical conversion. This overall rate may then be used as a basis for the design of an industrial reactor.

A basic prerequisite for the study of the intrinsic chemical reaction rate in a laboratory reactor is a satisfactory characterization of heat and mass transfer behaviour in the reactor. The present work is aimed at studying the
mass transfer characteristic in the reactor for a non-reacting system under isothermal conditions. The objective of this research was to develop simple correlations to relate mass transfer rate with operating conditions of a given reactor. The resulting correlations would then be used in conjunction with other data to obtain the intrinsic chemical reaction rate.

Two types of laboratory reactors have acquired eminence in the study of kinetics of reaction: recycle reactor and continuous stirred tank reactor (CSTR). Both the reactor systems have some advantages and disadvantages. However, a large number of designs of continuous stirred tank reactors have been advocated in the literature. The present study involves a vibration mixed reactor which has the novel feature of retaining the advantages of both the recycle reactor and the CSTR.
CHAPTER-2
LITERATURE REVIEW

2.0.0 PARTICLE-FLUID MASS TRANSFER

Adequate knowledge of mass transfer rate between a fixed bed of solid particles and flowing gas or liquid is needed in the design and development of the many important industrial equipments including fixed bed reactors. Mechanisms and correlations for mass transfer for single and multi-particle systems have been widely investigated and the volume of the literature on the subject is extensive. This section contains a review on some of the theoretical works and experimental works carried out by various investigators on mass transfer in fixed beds.

2.1.0. Mass Transfer in Particulate Beds:

Mass transfer problem in a particulate bed can be analyzed theoretically by setting up appropriate differential equations. The steady state mass balance equation without any chemical reaction is written as

\[ \nabla \cdot \mathbf{V} C_A = D_{AB} \nabla C_A \]  \hspace{1cm} (2.1)

Although the equation may be readily written, no general method of solution exists. Before solving equation (2.1), the equation of motion must be solved to obtain the velocity distribution. But it has not yet been possible to solve the full equation of motion analytically even for a single sphere placed in an infinite flowing medium. Only in certain ranges of Reynolds number, it is possible to obtain analytical solution of equation of motion. As a result, the mass balance equation can at best be solved analytically
for certain limiting ranges of Reynolds number.

When there is no convection, left hand side of equation is zero and we have the well known result for the stagnant medium:

\[ N_{Sh} = \frac{2}{1 + \frac{R}{b}} \]  

(2.2)

When the thickness at the fluid medium, \( b \to \infty \), \( N_{Sh} \to 2 \) which is the result in stagnant infinite medium.

For convective mass transfer from a single sphere, the left hand side of equation (2.1) remains and we have to know the velocity distribution. It has been mentioned that analytical solution of the equation of motion is available only for certain special cases. We can solve the equation of motion for two extreme situations. One for very slow motion and other when the velocity is very high. For the latter case, only analytical solution is possible. In the intermediate range where the motion is neither very small nor very high, and which is usually the case in most chemical engineering applications one has to solve the equation of motion numerically.

For creeping motion, when \( N_{Re} \to 0 \), there exists two solutions for the mass balance equation, equation (2.1) depending on the dimensionless Peclet number, one for \( N_{Pe} \to \infty \) and the other for \( N_{Pe} \to 0 \).

When the Peclet number is large and the Reynolds number is low, we have
This expression is known as Levich (1) solution. It is found from experimental studies that equation (2.3) is valid for \( N_{Pe} > 70 \).

For liquids, the Schmidt number is in the range \( 10^2 \) to \( 10^3 \) so that the solution of the mass balance equation for high Peclet number is generally valid for solid liquid systems at low Reynolds number.

When the Peclet number is very low the Sherwood number is given by

\[
N_{Sh} = 2 + \frac{1}{6} N_{Pe} + \frac{1}{2} N_{Pe} \ln N_{Pe}
\]  

Since equation (2.4) is valid for low Peclet number i.e. low Schmidt number, therefore this expression is in general applicable for gas solid systems.

The problem of mass transfer in a multiparticle system is much more complicated because of the complex flow patterns that exist in such system. Only using some simplified assumptions one can get some estimates of mass transfer in a particulate bed.

First we consider a simplified situation in a particulate bed where there is no flow and each particle is surrounded by a fluid envelope of radius \( b \), where \( b \) is related to the voidage by the following equation

\[
\frac{R}{b} = (1 - \varepsilon)^{1/3}
\]  

(2.5)
Replacing the value of $b$ from the above equation in equation (2.2) we have

$$N_{Sh} = \frac{2}{1-(1-E)^{1/3}}$$ (2.6)

When there is convection, different authors have proposed various models for solving the mass transfer problems in a multiparticle bed. Discussion on these models are given below:

**Cell Model:** The cell model developed by Happle (2), where it was assumed that a three dimensional assembly of spheres can be considered to be a number of identical unit cells consisting of a particle surrounded by a spherical fluid envelope. Each cell contains the same amount of fluid as the relative volume of fluid to particle volume in the entire assemblage. With this model the equation of motion can be solved for the unit cells and the solution of the mass balance equation can be obtained. Pfeffer (3) solved analytically the mass balance equation in a multiparticle system for high Peclet number and low Reynolds number case and obtained the following relation.

$$J_D = 1.26 \left[ \frac{1-(1-E)}{W} \right]^{5/3} N_{Re}^{1/3}$$ (2.7a)

or alternatively

$$N_{Sh} = 1.26 \left[ \frac{1-(1-E)}{W} \right]^{5/3} N_{Re}^{1/3}$$ (2.7b)

Where $W$ is complicated function of voidage. Equation (2.7) has been compared with the experimental data taken in packed beds and at different values of the void volume (between
0.33 to 0.55) and agreement is reasonable for $N_{Re} < 100$.

The cell model has also been used to solve mass transfer problem for high Peclet number and the intermediate range of flow. Leclaire and Hamiclec (4) solved the heat or mass transfer problem for large Peclet number and intermediate range of Reynolds number. From their result it can be concluded that for a Reynolds number upto 100 $J_D$ factors are almost same as obtained by Pfeffer (3). By least square analysis they obtained the following theoretical relationship:

$$J_D \varepsilon^{0.854} = 1.485 N_{Re}^{-0.632} \quad (2.8)$$

**Boundary-Layer Model**: Carberry (5) proposed the boundary layer model based on the concept of diffusion within a developing boundary layer which is repeatedly developed and destroyed as the fluid flows through an assembly of spherical particles. Carberry (5) obtained a theoretical solution for mass transfer in a packed bed under the assumption that the boundary layer develop and collapses over a distance approximately equal to one particle diameter and following an analysis similar to the laminar boundary layer flow on a flat plate and obtained the following result

$$N_{Sh} = 1.15 \left( \frac{N_{Re}}{E^2} \right)^{\frac{1}{2}} N_{Sc}^{\frac{1}{2}} \quad (2.9)$$

The boundary layer model is supported by literature data for particle fluid mass transfer data in packed beds over the range $0.5 < N_{Re} < 10^3$.

Various other theoretical models are also available in the literature, but these are less successful than the cell and the boundary layer models.
2.2.0 Experimental Studies of Mass Transfer in Fixed Beds

It has been reported in the previous section that theoretical studies have been developed on the basis of some simplifying assumptions. As a result, the theoretical solutions have not proved to be particularly successful in describing mass transfer processes in fixed beds.

Therefore, more effort has been made in carrying out experimental studies. Most of the experimental studies on mass transfer in fixed beds were carried out using the following systems.

1. Sublimation of solids into gases.
2. Evaporation of liquids from porous particles into gases.
3. Dissolution of solid into liquid.
4. Solution of liquids contained in porous particles into other liquids.
5. Mass transfer in a solid-gas reacting system.

In this section a review is given on the previous experimental studies on mass transfer in fixed beds.

2.2.1. Mass Transfer in gas-solid System:

Mass transfer study in gas-solid system has been carried out by the following methods:

a. Evaporation of liquids from the porous particles into gases.

b. Sublimation of solids into gases.
a. Evaporation of liquids from the porous particles into gases:

Mass transfer studies, by evaporation of water from the surface of spherical and cylindrical pellets into air stream, were carried out by a number of investigators, Gamson, Thodos and Hougen(6). The following correlations were obtained:

\[
J_D = 16.8 \, N_{Re}^{-1.0} \quad N_{Re} \ll 40 \tag{2.10a}
\]

\[
J_D = 0.989 \, N_{Re}^{-0.41} \quad N_{Re} \gg 350 \tag{2.10b}
\]

It should be noted that since the experiments were carried out using shallow bed their results were doubtful as there was possibility of large entrance and exit effects.

Wilke and Hougen(7) carried out a study similar to that of Gamson et al. (6) in a shallow bed with Reynolds number in the range 50 - 4000. Their results are correlated by the following equations:

\[
J_D = 1.82 \, N_{Re}^{-0.51} \quad N_{Re} \ll 350 \tag{2.11a}
\]

\[
J_D = 0.989 \, N_{Re}^{-0.41} \quad N_{Re} \gg 350 \tag{2.11b}
\]

Thacker and Hougen(8) extended the work of Gamson et al. using Rasching rings, Partition and Berl Saddles. They determined heat-transfer factor \( J_H \) and \( J_D \) was obtained by using the ratio \( J_H / J_D = 1.076 \). The Reynolds number was within the range of 70 to 20,000 but most of the measurements made between 250 to 3000. The correlations obtained were:
De Acetis and Thodos (9) studied mass and heat transfer by evaporation of water from the surface of porous spherical particles. The spheres were arranged into two types of packing: packed fixed bed consisting of layers of sphere in a very compact arrangement and expanded fixed beds consisting of porous spheres randomly mixed with an equal number of inert spheres of the same size. The experimental data obtained for the packed and expanded fixed beds were analyzed to establish the mass- and heat-transfer factors. Mass-transfer factors, \( J_D \), were plotted against the modified Reynolds number for both packed and expanded fixed beds. The Reynolds number ranged from 13 to 2136. The \( J_D \) factor relationship was expressed as

\[
J_D = 0.725 \div (N_{Re}^{0.44} - 0.15)
\]  

(2.13)

The \( J_D \) factors resulting from this study were compared with those of several other investigations for systems involving both gas and liquid films. The \( J_D \) factors previously reported by Gamson et al. (6) were consistently higher than the values obtained in this study. This was because a higher surface temperature would produce a larger driving force and consequently both lower of mass-transfer coefficient and \( J_D \) factor.

Petrovic and Thodos (10) carried out an investigation of gas-phase mass transfer at low Reynolds number region associated
Several studies have been carried out to extend the mass transfer investigations of Gamsor et. al. in the low reynolds number region. Extrapolation of their relationships between $N_{Re} - 170$ has been the subject of considerable speculation and criticism(11). Reliable results for $N_{Re}$ above and below $N_{Re} = 170$ is difficult to obtain because of the relevance of saturated conditions at the end of the fixed bed.

In the studies of Gamson, Thodos and Haugeen(5), the surface temperature of the wet porous pellets was assumed to be the same as the wet-bulb temperature of the inlet air. Although this assumption is undoubtedly valid at high air
velocities, considerable speculation exists as to its validity at low velocities. De Mecis and Thodoe (9) measured directly the surface temperature of the particles. It was found that the temperature of the evaporating surface was the same as the wet bulb temperature of inlet air only at high velocity.

(h) Sublimation of solid into gases:

Several investigators have studied mass transfer rates by evaporation of naphthalene into a stream of air or inter gases.

Hurt (12), Resnick and White (13) studied the rate of mass transfer by vaporization of naphthalene into air streams. In the experiments of Resnick and White (13), the gas leaving the bed was nearly saturated with naphthalene. Hurt (13) did not present sufficient information on the particle and bed properties to permit an accurate calculation of the mass transfer factor. Some doubt exists as to the numerical accuracy of the results of Hurt, Resnick and White.

Chu, Kalil and Wetteroth (14) measured the rate of mass-transfer between regular shaped solids and a turbulent air stream for both fixed and fluidized granular beds. The study was carried out by use of celite cylinder lead shot, glass beads, and rape seed coated with naphthalene. Particle diameters ranged from 0.18 to 1.4 cm. The Reynolds number was varied from 1 to 5000. The smaller particles required the use of very small bed heights to prevent the exit air from becoming over 90% saturated. As a result, very small bed heights were necessary. The mass-transfer factor was
correlated with the modified Reynolds number for both fixed and fluidized beds. The following correlations were proposed

\[
J_D = 5.7 \left( \frac{N_{Re}}{P} \right)^{-0.78} \quad 1 < \frac{N_{Re}}{P} < 30 \quad (2.15a)
\]

\[
J_D = 1.77 \left( \frac{N_{Re}}{P} \right)^{-0.44} \quad 30 < \frac{N_{Re}}{P} < 5000. \quad (2.15b)
\]

Where \( \frac{N_{Re}}{P} = \frac{D_p \cdot G}{(1 - \varepsilon)} \)

Bar-Ilan and Mesnick(15) determined the mass transfer characteristics of a fixed bed by measuring the rate at which naphthalene particles in a fixed bed vaporized into air passed through the bed. It was reported that the particle diameter appeared as a parameter for the spherical granules but not for cylindrical pellets. For pelleted material of two different diameters, a single line correlated the \( J_D \) factor with the modified Reynolds number. A departure from a straight line correlation was obtained at the extremely low Reynolds number. The maximum in the \( J_D \) factor appeared at a Reynolds number of about one; for values of the Reynolds number greater than one, the \( J_D \) factor decreased with increase in Reynolds number.

Heat and mass transfer studies were carried out in a vibration mixed reactor by Ahmed and Sunderland (16). Mass transfer experiments were carried out by evaporation of naphthalene in a stream of air at 30°C. The rate of evaporation of spherical naphthalene pellets of different sizes (¼ in., ⅛ in., and ⅛ in.) was measured as a function of frequency and amplitude of vibration and velocity of gas. The effect of flow rates on mass transfer without vibration in the reactor is represented by the following correlation:
\[ N_{sh} = 2.25 \left( N_{Re} \right)^{0.83} \]  \hspace{1cm} (2.16)

The flow rate range studied was 50-700 C.C./min. It was reported that the rate of mass transfer increased with increase in frequency of vibration against flow rate by as much as 450%. To study the effect of amplitude, mass transfer rates were determined at three amplitudes (1/8 in., 3/32 in., and 1/16 in.). It was observed that the value of the mass transfer coefficient increased considerably, especially at higher frequencies when the amplitude is increased. The data for \( \frac{3}{4} \) in. pellets were represented by the following correlations for different amplitudes:

\[ N_{sh} = 1.98 \left( N_{Re} \right)^{0.47} \] \hspace{1cm} (2.17) low frequency regime

\[ N_{sh} = 1.28 \times 10^{-2} N_{Re}^{1.3} \] \hspace{1cm} (Amplitude 1/8 in high frequency regime

\[ N_{sh} = 2.89 N_{Re}^{0.41} \] \hspace{1cm} (2.18) low frequency regime

\[ N_{sh} = 5 \times 10^{-2} \left( N_{Re} \right)^{1.11} \] \hspace{1cm} (Amplitude 3/32 in high frequency regime

\[ N_{sh} = 4.29 N_{Re}^{0.36} \] \hspace{1cm} (2.19) low frequency regime

\[ N_{sh} = 1.37 \times 10^{-1} \left( N_{Re} \right)^{1.02} \] \hspace{1cm} (Amplitude 1/16 in high frequency regime

The effect of particle size was also studied and the result for the three particle sizes (\( \frac{3}{4} \) in., 3/16 in. and 1/8 in.) do not fall into the general pattern and hence separate correlation was written down for each particle size, as follows:

\[ N_{sh} = 1.49 N_{Re}^{0.49} \] \hspace{1cm} (2.20) low frequency regime

\[ N_{sh} = 2.95 \times 10^{-3} \left( N_{Re} \right)^{1.59} \] \hspace{1cm} high frequency regime.
Size 3/16 in. and amplitude 1/16 in.
\[ N_{Sh} = 2.93 \times 10^{0.38} \text{ -- low frequency regime} \]
\[ N_{Sh} = 0.029 \times 10^{1.31} \text{ -- high frequency regime} \]

Size 1/8 in. and amplitude 1/8 in.
\[ N_{Sh} = 1.09 \times 10^{0.49} \text{ -- low frequency regime} \]
\[ N_{Sh} = 1.24 \times 10^{-3} \times 10^{1.82} \text{ -- high frequency regime} \]

Size 1/8 in. and amplitude 1/16 in.
\[ N_{Sh} = 1.7 \times 10^{0.42} \text{ -- low frequency regime} \]
\[ N_{Sh} = 2.98 \times 10^{-2} \times 10^{1.35} \text{ -- high frequency regime} \]

### 2.2.2 Mass Transfer in Liquid-Solid System

One of the earlier works on mass transfer to liquids were carried out in fixed and fluidized beds by Wilhelm(17). They measured the rate of mass transfer of \( \beta \) (or 2) - naphthol ball-shaped pellets and flakes into water. Two separate mass transfer correlations were obtained as given below,

\[ J_D = 1.625 \times 10^{-0.507} \times N_{Re} \quad \text{for} \quad N_{Re} < 120 \]  
(2.24a)

\[ J_D = 0.687 \times 10^{-0.327} \times N_{Re} \quad \text{for} \quad N_{Re} > 120 \]  
(2.24b)

A study similar to McCune and Wilhelm was carried out by Gaffney and Drew(18) with benzene-sulicylic acid, n-butyl
alcohol-succinic acid systems using pelleted particles. The following correlations were obtained from the data of Gaffney and Drew:

\[
J_D = 1.97 \left( \frac{N_{Re}}{E} \right) - 0.613 \quad (2.25a)
\]

\[
\begin{align*}
N_{Re}/E < 200 & \\
J_D = 0.29 \left( \frac{N_{Re}}{E} \right)^{-0.254} N_{Re}/E > 200 & 
\end{align*}
\]

(2.25b)

Hobson and Thodos studied the removal of organic liquids (saturated solution of water-isobutyl alcohol or water-methyl ethyl Ketone) on impregnated porous celite spheres arranged in a fixed bed, and reported the following correlation:

\[
\log J_D = 0.7683 - 0.9175 \log N_{Re} + 0.0817 \log N_{Re}^2 \quad (2.26)
\]

It may be seen that Hobson and Thodos correlation predicts higher mass transfer rate compared to the previous two works.

Evans and Gerald measured rate of mass transfer for fixed and fluidized beds of benzoic acid granules dissolving in water stream at low Reynolds number. The study was carried in the range of Reynolds number from 1 to 100. The results for fixed bed are correlated by the following equation:

\[
J_D = 1.48 N_{Re}^{-0.52} \quad (2.27)
\]

This correlation for fixed-bed data is nearly identical with the relation for liquid-phase fixed-bed mass-transfer developed by McCune and Wilhelm for this range of Reynolds number.
Theoenes and Kramers (21) investigated mass transfer rate between a single sphere of benzoic acid and water where the sphere was a part of a regular arrangement of inert spheres. Eight different kinds of packing were used. The voidage was varied between 0.25 and 0.50. The authors correlated mass transfer data by the following equation:

\[ N_{Sh} = 1.0 \cdot N_{Re}^{1/3} \cdot N_{Sc}^{1/3} \text{ where } N_{Re} = \frac{DV}{1 - \varepsilon} \]  

(2.28)

The Reynolds number ranged from 40 to 4000 and the Schmidt number from 1 to 1000.

Mass transfer rates have been measured by Mullin and Treleaven (22) for spheres of benzoic acid placed alone and among groups of inert spheres in a flowing stream of water. The voidage was varied from approximately 1.0 (single particles) to 0.5 (fixed and fluidised beds) and Reynolds number 50 to 700. The data for fixed and moving isolated spheres were correlated by

\[ N_{Sh} = 0.94 \cdot N_{Re}^{1/2} \cdot N_{Sc}^{1/3} \]  

(2.29)

It was reported that the presence of a single neighbouring sphere had little effect on the mass transfer rates from a given sphere. For multiparticle systems the mass transfer rates were found to increase with decreasing voidage. This increase in mass transfer rate was due to increase in velocity gradient and also possibly due to turbulence produced by the interactions between the fluid and the particles.
2.3.0 Summary of Literature on Particle - Fluid Mass Transfer.

Mass transfer rates for single particles can be predicted from the theory for the limiting case of creeping flow. However, the theoretical predictions are not exact, because of the difficulty in describing the flow. The situation in multiparticle systems is complicated. Various models have been suggested to predict theoretically the mass transfer rates in a multiparticle assembly. The most widely used models are the cell model of Pfetter (3) and the boundary layer model of Carberry(5).

A large amount of experimental work is available in the literature on the mass transfer problem in a particulate bed. Some of the important works in gas-solid system are summarized in Table (2.1).
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<td>$J_D = 16.8 N_{Re}^{-0.10}$</td>
<td>2.10a</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$N_{Re} &gt; 350$</td>
<td>$J_D = 0.989 N_{Re}^{-0.41}$</td>
<td>2.106</td>
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<tr>
<td>Wilke and Hongen (7)</td>
<td>Water-air</td>
<td>$N_{Re} &lt; 350$</td>
<td>$J_D = 1.82 N_{Re}^{-0.51}$</td>
<td>2.11a</td>
</tr>
<tr>
<td></td>
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<td>$N_{Re} &gt; 350$</td>
<td>$J_D = 0.989 N_{Re}^{-0.41}$</td>
<td>2.11b</td>
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<tr>
<td>Thacker and Hongen (8)</td>
<td>Water-air</td>
<td>$70 &lt; G/\sqrt{\delta_p}/\mu$</td>
<td>$J_D = 0.855 (G/\sqrt{\delta_p}/\mu)^{0.34}$</td>
<td>2.12</td>
</tr>
<tr>
<td>De Acetis and Thodos (9)</td>
<td>Water-air</td>
<td>$30000 &lt; G/\sqrt{\delta_p}/\mu$</td>
<td>$J_D = 1.07 (G/\sqrt{\delta_p}/\mu)^{-0.41}$</td>
<td>2.12b</td>
</tr>
<tr>
<td>Petrovic and Thodos (10)</td>
<td>Water-air</td>
<td>$13 &lt; N_{Re} &lt; 2136$</td>
<td>$J_D = 0.725 (N_{Re})^{0.15}$</td>
<td>2.13</td>
</tr>
<tr>
<td>Chu, Kalil and Wettersoth (14)</td>
<td>Naphthalene-air</td>
<td>$1 &lt; N_{Re} &lt; 30$</td>
<td>$J_D = 5.7 (N_{Re})^{-0.78}$</td>
<td>2.15a</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$30 &lt; N_{Re} &lt; 5000$</td>
<td>$J_D = 1.77 (N_{Re})^{-0.44}$</td>
<td>2.156</td>
</tr>
</tbody>
</table>
3.0.0. EXPERIMENTAL

3.1.0 Basis of Experimental Design.

The objective of this work was geared to the study of heterogenous catalytic reactions. For the design and analysis of catalytic reactors one uses the overall rate of chemical conversion which depends on both the rate at which mass is supplied to the catalyst surface i.e., mass transfer rate and the intrinsic rate of chemical reaction i.e., kinetic rate. As in the case of mass and heat transfer rates, the kinetic parameter in general cannot be predicted from available correlations as the latter data are often specific for a given system. Therefore, the kinetic parameter is determined experimentally. To develop a meaningful expression for the kinetic rate from laboratory data it is required that mass and heat transfer effects are accurately known. The main aim of this work was to provide gas-solid mass transfer data using a laboratory reactor that would be well suited for subsequent studies on heterogenous catalytic reactions.

Various types of laboratory reactors have been designed and developed for catalytic reactions.

A brief discussion of the advantages and disadvantages of the different reactors will aid us in understanding the reasons for selection of the particular reactor that was used in the present study.

Differential Reactors: A differential reactor contains a shallow bed of catalyst so that relatively small changes in
composition are obtained by passing a reactant through the catalyst bed. Generally when heat and mass transfer effects are prominent, evaluation of concentration and temperature gradients are highly uncertain for differential reactors.

To overcome this difficulty, high flow rates are employed in differential reactor to minimize the effects of mass and heat transfer on overall rate of chemical conversion. For complex exothermic reactions differential reactors have proved useful. The chief difficulty with this type of reactor is that extreme precision of chemical analysis is required.

Integral Reactors: Integral reactors utilize a much larger amount of catalyst bed than differential reactors. Since the conversion in an integral reactors would be higher, extreme precision in measurement is not required. Some of the principal disadvantages of integral reactor is the difficulty of obtaining isothermal condition and proper flow distribution and higher transport gradients within the bed and is clearly inferior to differential reactors.

Recycle Reactor: Recycle reactors have been designed and developed to combine the desirable features of the differential and integral reactors. A recycle reactor consists of a recycle loop around a small reactor between the feed and product streams. The analytical difficulty of a differential reactor is thus avoided due to a large difference in the concentrations of fresh feed and product streams. Also the
disadvantages of the integral reactors are overcome due to small interphase gradients and large flow rates resulting from low conversion and high recycle ratio respectively.

Not every reaction can be studied by using a recycle reactor because in some cases various side reactions may be promoted, owing to the admixture of products to the fresh feed. Another possible difficulty is the length of time required for the recirculating portion of the system to reach a steady state operation. Pressure drops across the reactor may be higher than those usually observed in ordinary differential beds. Owing to the transient period after the change of any experimental variable, one must have a continuous method of analysis when using this type of reactor. One of the major difficulties with these reactors is the design of a suitable recycle pump capable of operating at the high temperature and pressure usually encountered.

Continuous stirred tank reactor (CSTR):

Although stirred tank reactors have been successfully employed to determine the intrinsic chemical reaction rate, but it has some shortcomings. The ratio of free volume to catalyst volume is often too large. For this reason, there is a possibility of homogeneous reaction to take place simultaneously with the catalytic reaction. The extent of gas solid contact is not always well defined. There are some mechanical
problems for example; protection of the shaft bearing from over heating when high temperature is involved. It is also difficult to stop leakage of reactants through the glands between reactor and stirrer shaft.

Vibration Mixed Reactor: To achieve the ideal stirred tank reactor behaviour, Sunderland and El Kanzi have designed a laboratory reactor in which a piston reciprocating at high speed provided the mixing needed to minimize the heat and mass transfer effects that frequently obscure the intrinsic chemical kinetics of gas-solid catalytic reaction. The vibration mixed reactor is similar in concept to previous stirred tank reactors, but it contains several new important features. The reactor is shown in Fig. 3.1 and 3.2.

The design of the reactor is unusual in that mixing within the reactor is done by a piston reciprocating at high speed rather than the more normal rotary devices. The frequency and amplitude of reciprocation can be varied giving the flexibility of operation. The direction of flow is altered for each stroke and this provides better mixing than a system in which the flow is always in the same direction. The turbulent motion also ensures that the mixing of the gases within the bed is good, reducing interfacial resistances and allowing the reaction to be carried out under chemical control condition.

The piston is driven through a piston rod by means of a coil oscillating in the annulus of a powerful pot magnet.
Degree of mixing can be varied by varying the applied voltage which alters the amplitude of vibration. Frequencies can be varied by using a frequency oscillator and power amplifier.

It can be concluded that the Vibration Mixed Reactor has considerable potential for studying gas-solid catalytic reactions and hence, the present mass transfer studies were carried out using a Vibration Mixed Reactor.
3.2.0 Design Characteristics of Vibration-Mixed Reactor

The vibration mixed reactor was originally designed by El Kanzi and Sunderland (23). Complete design details and a scale drawing are given in El Kanzi's thesis (24). Figure (3.1) shows the reactor designed by El Kanzi and Sunderland. The characteristics of this reactor and its performance in measuring intrinsic rate of a chemical reaction has been discussed in previous section.

Some modifications to the original design of El Kanzi and Sunderland's reactor (23) was made by Sunderland and Ahmed (16) and the modified design has been used in the present work. Figure (3.2) shows the reactor used by Sunderland and Ahmed (16) and in the present investigation.

The reactor base Figure (3.2) consists of a stainless steel cylindrical vessel (18) 7 5/8 in. in diameter. The cylindrical vessel is fixed to a 12 in. bottom plate (19) by a flange (20). Within the base, a flat spring, 5/8 in. in diameter and 0.006 in. thick, is clamped and screwed to the bottom of the connecting rod. There is a hollow aluminium cone (8), screwed to the bottom of the connecting rod. The moving coil of the drive unit is fixed to the cone. The moving coil 2 in. in diameter and 1 in. high, is mounted in the annulus of a magnet (7).

A new powerful magnet has been used. The whole assembly is bolted on the bottom plate (19). The driving unit housing case (13) is then fixed to the base plate by the flange. The power to the coil is supplied from the mains through a frequency oscillator and amplifier.
FIG. 3.1 REACTOR USED BY SUNDERLAND AND EL KANZI (23).

NUMBERED PARTS.

1. Reciprocating piston
2. Catalyst pockets
3. The moving coil
4. Connecting rod
5. Top spider
6. Bottom spider
7. The magnet
8. The Al cone
9. The reactor
   a) main body of the reactor
   b) piston sleeve
   c) reactor top
10. Inlet to reactor
11. Thermocouple well
12. Grooves to take piston
13. Slots in piston sleeve
14. Effluent outlet
15. Lower entry port
16. Upper inlet port
17. Sleeve for piston rod
NUMBERED PARTS
1. Reciprocating Piston
2. Catalyst pockets
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   a) Main body of the reactor
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   c) Reactor top
10. Inlet to reactor
11. The thermocouple well
12. Grooves to take piston sleeve
13. Slots in piston sleeve
14. Effluent outlet
15. Lower entry port
16. Upper inlet port
17. Sleeve for piston rod
18. Reactor Base
19. Bottom plate
20. Flange

FIG. 3.2 CROSS SECTIONAL VIEW OF THE REACTOR.
3.3.0 Selection of the Experimental System:

As discussed earlier, the objective of this work was to obtain reliable mass transfer data in vibration mixed reactor, using a gas-solid system. Among the different methods for mass transfer studies the two most suitable methods are:

(i) Evaporation of liquids from porous pellets
(ii) Sublimation of suitable solids in a stream of inert gases.

Evaporation of liquid from a porous pellet in a hot air stream possesses some difficulties because of the following reasons:

In addition to mass transfer, significant heat transfer may also occur due to evaporation of liquid, and simultaneous heat and mass transfer complicate the necessary calculations. The main difficulty is in the estimation of surface temperature of the particle. The liquid film on the solid surface is not uniform because of the complication of diffusion of liquid through the capillaries of the pellets to the surface.

Considering the above difficulties of the first method, the sublimation method was chosen. In this method the heat effect due to sublimation is generally negligible and surface concentration remains uniform. The system used was evaporation of naphthalene in air. Naphthalene is easily available
FIG. 3. FLOWDIAGRAM OF THE EXPERIMENTAL SETUP.

TC THERMOCouple
PG PRESSURE GAUGE
PR PRESSURE REGULATOR
V VALVE
and can be made into suitable shapes and size without much difficulty. Also different physico-chemical mass transfer studies using naphthalene-air system have been used by many workers.

3.4.0 Experimental Setup.

A flow diagram of the experimental setup is shown in Figure (3.3). Compressed air from the main supply line was passed through a pressure regulator for pressure reduction. Air was passed through a filter to remove any dust particle and was then dried by passing through a silica-gel dryer. The flow rate of dry compressed air was measured by a calibrated rotameter (FM) and pressure at the rotameter inlet was measured by a pressure gauge (PG). The flow rate of air was controlled manually by using valve (V-2). Before entering the reactor, air was split into two streams. The main stream of air was preheated in an electrically heated heater that flowed to the reactor beds through the piston rod sheath. The other stream was passed to the reactor beds through the bottom part of the reactor, where it served as cooling medium for the coil of the driving unit. This flow was controlled by valve (V-4). The temperature inside the reactor bed was measured by a calibrated Chromel-Alumel thermocouple (TC) and the temperature at the reactor outlet was measured by a glass thermometer (TM). The air flowing through the reactor beds was exhausted from the outlet at the top of the reactor.
3.5.0 Experimental Procedures:

For conducting the experiment the following steps were followed.

1. The air flow was started through the reactor beds and the flow rate was controlled by the valve (v-2).

2. The electrical heater was switched on and heating rate was controlled by regulating the input voltage with a voltage regulator.

3. Sufficient time was allowed for the system to attain thermal balance under flow condition.

4. The temperature reading of the calibrated thermocouple (TC) was observed. It took about an hour for the reading of the thermocouple and thermometer to attain steady state.

5. When the reactor assembly was heated to a steady state, the air flow was stopped and the reactor top plate was removed and weighed. Naphthalene pellets were placed into the catalyst pockets. After packing the pellets, the top plate was bolted and air flow was resumed at a preset frequency and amplitude of oscillation of the piston.

The time required for removal of reactor top plate and reweighing of the naphthalene pellets was 3% of the total experimental time.
The amplitude of oscillation was measured with a travelling microscope. In order to do this, the vibrating piston was illuminated by a stroboscope turned to the same frequency. The frequency oscillator was calibrated against a stroboscope for frequency up to 32 Hz.

The bulk gas temperature reading of the thermocouple set into the reactor was assumed to be the surface temperature of the naphthalene pellets. Since the rate of naphthalene evaporation was too low to produce any noticeable thermal effect, therefore, the above assumption was not invalid. The temperature was used to calculate vapour pressure of naphthalene (Appendix A.4).

6. At the end of two hours, which was the duration of all experimental runs, the air flow was stopped and the naphthalene pellets were removed from the reactor and reweighed immediately.

7. From the vapour pressure data and the amount of naphthalene evaporated, it was checked if the air was saturated with naphthalene.

3.6.0 Experimental Precautions:

The following precautions were found necessary in order to obtain reproducible results.

1. The loss in weight of naphthalene pellets was kept around 4.5%. This resulted in a negligible change in the surface
area of the pellets during time of the run.

The change in weight was measured by a balance with an accuracy of ± 0.0001 gm.

2. Naphthalene pellets were stored in a stoppered bottle to avoid any loss in weight.
3. Saturation of air must be avoided to obtain meaningful data.

3.7.0 Experimental Conditions:

The following range of experimental conditions were used in the present work.

1. Flow rate of air was within the range of 120-900 c.c./min.
2. Frequency of vibration was varied from 15 to 32 Hz.
3. Amplitude of vibration was 1 mm.
4. Reactor pressure was within the range of 765 to 775 mm Hg.
5. Temperature was within the range of 27°C to 29°C.
6. Pellet of three different shapes and sizes were used.
7. Duration of each experiment was two hours.

3.8.0 Preparation of Naphthalene Particles:

Many mass transfer investigation are carried out using spherical particles. However, since the objective of the work
was likely to work: on heterogenous catalytic reactions, it was felt that particles chosen for this work should have similar size and shape as those that will be used for kinetics studies. The catalysts available in the present laboratory for the later work are cylindrical (size: 1/8 in.) and thus it was decided that mass transfer work was to be carried out using cylindrical particles. In the present study, cylindrical and pellet shaped particles of naphthalene were used (Fig. 3.3).

Following methods were tried for preparation of naphthalene pellet for mass transfer studies:

1. Compression of powdered naphthalene in a die.
2. Casting of molten naphthalene in a mould.
3. Coating of naphthalene on a desired shaped inert particle.

Due to nonavailability of spherical die and mould, it was not possible to prepare spherical pellets of naphthalene. Third method was also tried but the pellets produced were not uniform. Finally three different shapes of naphthalene pellets were prepared by compressing naphthalene powder in a pharmaceutical tablet machine. Steel plungers and dies of different shapes and sizes were used with the tablet machine. The pellets produced by this methods were hard, smooth and uniform. The shape and dimension of the pellets are shown in Figure.
FIG. 3.3  MASS TRANSFER PELLETS

<table>
<thead>
<tr>
<th>Particles</th>
<th>$D_p$ (cm)</th>
<th>Surface Area (sq. cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TYPE I</td>
<td>0.448</td>
<td>1.129</td>
</tr>
<tr>
<td>TYPE II</td>
<td>0.456</td>
<td>1.137</td>
</tr>
<tr>
<td>TYPE III</td>
<td>0.5686</td>
<td>1.259</td>
</tr>
</tbody>
</table>
CHAPTER 4

4.0.0 RESULTS AND DISCUSSIONS:

4.1.0 Treatment of Experimental Data:

In this experimental work, overall mass transfer coefficients were determined in a vibration mixed reactor by naphthalene evaporation in a stream of air. Naphthalene pellets of three different shapes and sizes were used for the studies. The rate of evaporation of naphthalene was measured as a function of velocity of air (without vibration) and frequency of vibration. Rate of evaporation was also measured for different orientation of pellets of same shape and size (Fig. 4.1).

In a gas-solid system, it is convenient to define an average mass transfer coefficient \( K_g \), based on partial pressure driving force as

\[
K_g = \frac{N}{a_p (P_s - P_b)} R^N T^T
\]  

The mass transfer coefficient under steady state conditions was calculated by using equation (4.1). For a well mixed system at steady state, the bulk gas partial pressure of naphthalene \( P_b \), is equal to its exit concentration. \( P_b \) is determined from the air flow rate and loss in weight of naphthalene pellets (Appendix A.5). The surface partial pressure \( P_s \), is the vapour pressure of naphthalene at the temperature of the surface. Since the naphthalene evaporation
rate is not fast enough to establish a temperature difference between the solid surface and the bulk gas, therefore $T_s$ is determined at the bulk gas temperature. This was also noted by (16). The vapour pressure of solid naphthalene is given by the following equation (25).

$$\log P_s = 6.84577 - 1606.529/(187.227 + T)$$  \hspace{1cm} (4.2)

Where $P_s =$ vapour pressure, mmHg

$T =$ absolute temperature, °C

In mass transfer studies the data are often correlated using dimensionless groups as

$$N_{Sh} = f(N_{Re}, N_{Sc})$$  \hspace{1cm} (4.3)

In the present study the diffusion coefficient remained almost constant and hence there was very little variation in the Schmidt number. The value of Schmidt number varied between 2.46 and 2.48. Hence in the present study data was correlated using the Sherwood number and Reynolds number only.

The Reynolds number in a particulate bed is usually defined as

$$N_{Re} = \frac{V_R D_p \rho}{\mu}$$  \hspace{1cm} (4.4)

Where $V_R$ and $D_p$ are characteristic velocity and length of the particle respectively. In most mass transfer studies $V_R$ is taken as the superficial velocity. However, in the present case,
the characteristic velocity must also reflect the effect of vibration. The velocity term $V_R$ was thus calculated considering the following factors.

(i) velocity based on the air flow rate.
(ii) velocity based on the vibration of piston.

The resultant average velocity of air flowing through the catalyst beds is given by the equation

$$V_R = \frac{L}{Q_o} \left[ (\sqrt{2\pi tf} D_f^2 + (F)^2) \right]^{\frac{1}{2}} \quad (4.5)$$

The detail derivation of equation (4.5) is given in the Appendix A. For without reciprocation, the velocity is calculated by neglecting the term in equation (4.5) caused by the vibration of the piston. The equivalent diameter of the pellets $D_p$, in the Reynolds number is defined by

$$D_p = \frac{6}{S_v} \quad (4.6)$$

4.2.0 Results:

To determine the effect of vibration on mass transfer in a vibration mixed reactor, the mass transfer rate was measured with and without vibration. The effects of particle shape and arrangement on mass transfer were also studied by using pellets of three different shapes and sizes. The arrangement of the particles in the catalyst beds are shown in Fig. 4.1.
The particle Type-II was randomly packed in the catalyst beds. The experimental results were correlated in terms of Sherwood number and Reynolds number.

4.2.1 Mass Transfer (Without Vibration)

Mass transfer rate was measured without vibration for three types of particles. For particle Type-I, two different arrangements in the catalyst beds of the reactor were used. The air flow rate range was from 120 c.c./min to 900 c.c./min. The mass transfer results are tabulated in Tables (A.6-1 to A.6-4).

The $N_{Sh}$ Vs. $N_{Re}$ plots for different particles are shown in Figures (4.2, 4.3 and 4.4). From these figures and tables it is evident that the results for the three types of particle are different and the data cannot be correlated using a simple equation. Even for particle of same shape and size (Type-I), different results were obtained for different arrangements. Therefore, separate correlations were developed for different particle sizes and arrangements. The experimental results are correlated by the following equations:

<table>
<thead>
<tr>
<th>Particle Type</th>
<th>Arrangement</th>
<th>Correlation</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>I</td>
<td>$N_{Sh} = 0.73 N_{Re}^{0.41}$ $5 &lt; N_{Re} &lt; 33$ (4.7a)</td>
</tr>
<tr>
<td>I</td>
<td>II</td>
<td>$N_{Sh} = 0.8 N_{Re}^{0.43}$ $5 &lt; N_{Re} &lt; 41$ (4.7b)</td>
</tr>
<tr>
<td>II</td>
<td>Random</td>
<td>$N_{Sh} = 1.64 N_{Re}^{0.24}$ $4 &lt; N_{Re} &lt; 26$ (4.7c)</td>
</tr>
<tr>
<td>III</td>
<td>III</td>
<td>$N_{Sh} = 1.1 N_{Re}^{0.321}$ $5 &lt; N_{Re} &lt; 34$ (4.7d)</td>
</tr>
</tbody>
</table>
4.2.2 Mass Transfer (with vibration)

The effect of vibration on mass transfer from the same particle was studied. The frequency of vibration was varied from 15 Hz to 32 Hz. It was not possible to vary the amplitude of vibration. The amplitude of vibration was 0.01 cm. The experimental results are tabulated in Table A.6-5 to A.6-7. The results are graphically represented by Figs. (4.5, 4.6). A separate correlation was developed for each particle. The correlations are:

<table>
<thead>
<tr>
<th>Particle Type</th>
<th>Arrangement</th>
<th>Correlation</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>I</td>
<td>( N_{Sh} = 7.86 \times 10^{-3} N_{Re}^{0.01} ) ( 200 &lt; N_{Re} &lt; 433 ) (4.8a)</td>
</tr>
<tr>
<td>I</td>
<td>II</td>
<td>( N_{Sh} = 0.015 N_{Re}^{0.95} ) ( 193 &lt; N_{Re} &lt; 420 ) (4.8b)</td>
</tr>
<tr>
<td>III</td>
<td>III</td>
<td>( N_{Sh} = 0.054 N_{Re}^{0.84} ) ( 158 &lt; N_{Re} &lt; 342 ) (4.8c)</td>
</tr>
</tbody>
</table>
4.3.0 Discussion

In the present work the effect of flow rate, frequency of vibration, particle shape and their arrangements on mass transfer have been studied. The results are discussed below:

Inspection of data show that the rate of mass transfer increases with increase in flow rate as expected and found by other investigators. However, the effect of flow on mass transfer rate is found to be less pronounced than other data available in the literature. Sunderland and Ahmed (16) carried out experimental works using similar system and they reported much higher values of mass transfer coefficient at comparable flow rates.

The main reason for the difference between the results of Sunderland and Ahmed (16) and the present work is the difference in the values of vapour pressure. The original source which Sunderland and Ahmed quoted for the vapour pressure data could not be checked. Use of the vapour pressure values reported by Sunderland and Ahmed sometime gave negative result in the present case and thus literature was checked. The vapour pressure values of the present study at a given temperature are higher than those used by the above two authors. Thus for equal amount of evaporation the mass transfer coefficient in the present case will be lower because of higher value of driving force than that predicted.
by the values used by Sunderland and Ahmed.

Comparison with other data on fixed beds also indicate the present mass transfer results are much lower. However it is doubtful whether a direct comparison between present value with fixed bed results would be meaningful. In fixed bed experiments the particle to bed diameter is usually much greater than 20, whereas in the present case these dimensions are comparable ($D_p / D_t \approx 2$). The Sherwood number values predicted in this work was near to the values for isolated single particles.

It is found from Fig. (4.2 to 4.4) that the particle arrangement has significant effect on mass transfer rates. This explains why equation (4.3) do not successfully correlate all data. The above correlation does not show the effect of geometrical arrangement, although it may take into account the effect of average voidage by incorporating this in the definition of Reynolds number. Thus correlations obtained by using equation (4.3) do not make any distinction between two different arrangements having the same voidage. Clearly this is not satisfactory for different geometrical arrangements. Same voidage but different arrangement lead to different flow distribution and hence different rates of mass or heat transfer.
The fluid pattern also greatly depends on particle to tube diameter ratio. When this ratio is large, the effect of the wall of the container cannot be neglected. In most works, the effect of wall may be neglected but this cannot be true in the present case where the particle to tube diameter is small. Hence data for the present case cannot be compared directly with most mass transfer studies which are generally carried out with large particle to tube diameter ratio.

The effect of bed structure may clearly be seen in Figure 4.2, which shows that the data for same particle size but different arrangement cannot be correlated by a single equation, though the voidages for both cases are the same.

Within the range of experimental observation the mass transfer results are lower when particles are arranged in a one stack (arrangement I) than in a two stack (arrangement II) arrangement. In the case where the particles are placed at random in the bed and in which case no definite order can be maintained in the bed, the mass transfer results are higher than the above two cases in Figure (4.2). The decrease in the mass transfer results due to ordered arrangement may be attributed to an "area blockage" effect. It may be seen from a consideration of the geometrical arrangements that with an ordered structure, the effective surface available to flow is low and hence
mass transfer is lower than when the particles are placed randomly in the bed. For a random geometry the flow can "see" the particles from all sides whereas for an ordered arrangement there are preferential zones through which flow occurs. It was felt that if the value of the effective surface could be calculated, it would be possible to correlate data for various geometry by a single correlation. Obviously, calculation of the effective surface area is a difficult task. However, based on the geometry of arrangement, it may be said that if the particles stack one above another, the surfaces where two particles are in contact are not available to flow, and hence, may be neglected from the total surface to calculate the effective surface area. Based on this simple consideration, effective surface areas were calculated and used to correlate data for different geometrical arrangements and shown in Figure (4.7). The various arrangements and the corresponding effective areas are shown in Table A.6-10. It may be seen from Figure (4.7) that the data for various arrangements may be brought closer and reasonably well correlated by a single correlation, and is given by equation (4.7c).
The effect of frequency of vibration is shown in Table (A.6 to 6-7) and Figure (4.5 and 4.6), where it is seen that the rate of mass transfer increases with the increase in the frequency of vibration. The frequency ranged from 15 Hz to 32 Hz. For example, at a flow rate of 270 c.c/min mass transfer coefficient with vibration is 1014.44 cm/hr and without vibration is 555.54 cm/hr. This increase in mass transfer rate is due to the fact that vibration of the piston at high speed reduces interfacial resistance markedly to give high rates of mass transfer. The results of mass transfer of the present study is compared with the results of Sunderland and Ahmed. The results differ both in absolute value and trend. Mass transfer coefficient is again not as high as that of Sunderland and Ahmed. Sunderland and Ahmed found that with increasing frequency, a transition occurs from a relatively low to a relatively high transfer regime. But in the present study there is no such distinct regime for mass transfer. Sunderland and Ahmed studied the effect of amplitude on mass transfer rate at three different amplitudes (3.2 mm, 2.4 mm and 1.6 mm). The value of mass transfer coefficient increased when the amplitude was increased.

In the present work, the amplitude of vibration could not be varied and hence its effect could not be observed. The amplitude of vibration in the present study was 1 mm, and which is lower than the smallest value used by Sunderland
and Ahmed. It is possible that there is a minimum value of amplitude of vibration before which transition does not occur for a given range of frequency. The amplitude of vibration was lower than that of Sunderland and Ahmed and hence again, direct comparison could not be made. The absolute values of mass transfer was lower because of the different values of vapour pressure used in the two works as has been mentioned before.

The effect of particle arrangement is also evident in the case of mass transfer with vibration and is shown in Figure(4.8). Again correlation based on effective surface area for mass transfer brings data for different geometrical arrangements together. However, this method is less satisfactory for arrangement-I.
FIG. 4.1: ARRANGEMENT OF PARTICLES IN THE REACTOR.
FIG. 4.2 $N_{Sh}$ AGAINST $N_{Re}$

- ○ Particle Type-I
- □ Arrangement-I
- ○ Particle Type-I
- □ Arrangement-II
FIG. 4.3 \( N_{Sh} \) AGAINST \( N_{Re} \)

Particle Type-II
Arrangement - Random

FIG. 4.4 \( N_{Sh} \) AGAINST \( N_{Re} \)

Particle Type-III
Arrangement - III
FIG. 4.5 $N_{Sh}$ AGAINST $N_{Re}$

- $N_{Re}$
- Particle Type-I
- Arrangement-I
- Particle Type-I
- Arrangement-II
FIG. 4.6  $N_{Sh}$ AGAINST $N_{Re}$

Particle Type-III  
Arrangement-III
FIG. 4.7 $N_{Sh}$ AGAINST $N_{Re}$

- $\square$ Particle Type-I
  - Arrangement-I
- $\triangle$ Particle Type-I
  - Arrangement-II
- $\bigcirc$ Particle Type-II
  - Arrangement-Random
- $\times$ Particle Type-III
  - Arrangement-III
FIG. 4.8 $N_{Sh}$ AGAINST $N_{Re}$

- △ Particle Type-I
- Arrangement-I
- □ Particle Type-I
- Arrangement-II
- ○ Particle Type-III
- Arrangement-III
NOMENCLATURE

A = Cross sectional area of piston, ft^2
a = Amplitude of piston
ap = Surface area of pellets, cm^2
CA = Concentration of component A,
DAB = Diffusion coefficient, ft^2/hr.
DP = Equivalent diameter of the particle, cm.
F = Volumeetric flow rate, cm^3/min.
f = Frequency, Hz.
G = Superficial mass velocity, lb./ft.^2 hr.
KG = Mass transfer coefficient, cm/hr.
L = Length of bed, ft.
N = Rate of evaporation of naphthalene,
Ps = Surface partial pressure, atm. gmoles/hr.
Pb = bulk gas partial pressure, atm.
Qo = Void volume of bed.
R = radius.
RN = Gas constant, cm^3.atm/gm moles °K.
SV = Specific surface of solid, surface per unit volume of solid, cm^-1.
T = Absolute temperature, °K.
V = Velocity
VR = Resultant velocity, Equation
W = function of voidage, W=2-3(R/b) + 3(R/b)5-2(R/b)6.
Dimensionless Groups

\[ N_{Pe} = \text{Peclet number, } N_{Re} \times N_{Sc} \]
\[ N_{Re} = \text{Reynolds number, } \frac{VD_p \rho}{\mu} \]
\[ N_S = \text{Schmidt number, } \frac{D \rho}{\mu} \]
\[ N_{Sh} = \text{Sherwood number, } \frac{K D}{\rho D_p} \]
\[ J_H = \text{Chilton and Colburn heat transfer factor.} \]
\[ J_D = \text{Chilton and Colburn mass transfer factor.} \]

Greek Symbols

\( \varepsilon \) = Void fraction.
\( \rho \) = Density of gas, lb/ft\(^3\).
\( \mu \) = Viscosity of gas, lb/ft-hr.
BIBLIOGRAPHY


APPENDIX A.1

EXPRESSION FOR VELOCITY OF OSCILLATORY FLOW

The velocity of an oscillatory flow is calculated by taking into account the combination of

(i) Velocity based on the gas flow rate

(ii) Velocity based on vibration of piston superimposed on the flow of gas.

There is a net flow of gas \( F \) through the bed as well as flow caused by the piston. This complex situation is treated by assuming that the two effects are independent of each other. Therefore the total flow rate in the reactor bed at any instant is the sum of the two

\[
F_T = F + 2\pi f a A \cos 2\pi ft
\]

where \( F \) = flow rate of gas, c.c/min

and \( 2\pi f a A \cos 2\pi ft \) = flow due to simple harmonic motion of piston.

The instantaneous velocity is given by

\[
V = F_T \times \frac{L}{Q_o}
\]

where

\( L \) = Length of bed

\( Q_o \) = Void volume of bed

By substituting the value of \( V \) in Equation (A.1.1)

\[
V = \frac{L}{Q_o} \left( F + 2\pi f a A \cos 2\pi ft \right)
\]
By averaging over the time for 1 cycle we may write:

\[ V_R = \frac{L}{Q_0} \left[ \int_0^{1/f} V^2 \, dt \right]^{1/2} \quad (A.1.3) \]

By substituting the value of \( V \) from equation (A.1.2), in equation (A.1.4) we get:

\[ V_R = \frac{L}{Q_0} \left[ \int_0^{1/f} \left( F + 2 \pi f L \alpha A \cos \pi f t \right)^2 \, dt \right]^{1/2} \quad (A.1.4) \]

\[ V_R = \frac{L}{Q_0} \left[ (\sqrt{2} \pi f L \alpha A)^2 + (F)^2 \right]^{1/2} \quad (A.1.5) \]

This resultant mean velocity given by equation (3.7) has been used to calculate the Reynolds number.
APPENDIX A.2

CALIBRATION OF FLOWMETER

The flowrates of air through the reactor were controlled by a rotameter (Manufactured by Fischer and Porter Ltd., England). The rotameter was calibrated by using a soap bubble flowmeter. Supply pressure of air at the rotameter inlet was maintained constant by a pressure regulator (PR). Time was measured by a stopwatch. The flow measurements for each position of the float of the rotameter were taken. Average of three consecutive readings were taken. The flowrate was varied from 80 to 1220 c.c./min.

The flowrates were plotted against the reading of the rotameter. The calibration curve is shown in Figure A.1.
FLOW RATE (cm³/min.)

FIG. A.1 FLOAT PETER CALIBRATION CURVE.
APPENDIX A.3

CALIBRATION OF THERMOCouple

Reactor bed temperature measurements were made by a Chromel - Alumel thermocouple. The output potentials were recorded by a millivolt recorder (PHILIPS PM 8251 Single-pen recorder). The thermocouple was calibrated by using a constant temperature bath. The millivolt reading corresponding to the temperature was plotted. The calibration curve is shown in Figure A.2.
FIG. A.2  THERMOCOUPLE CALIBRATION CURVE.
Appendix A.4

Vapour Pressure of Naphthalene

The vapour pressure of naphthalene used to calculate the mass transfer coefficient is given by the following expression:

\[ \log P_s = 6.84577 - \frac{1606.529}{(187.227 + T)} \]

where \( P_s \) = vapour pressure in mmHg.
\( T \) = temperature in °C.

Figure A.3 shows the plot of Naphthalene vapour pressure against the temperature.

Heat of sublimation of Naphthalene: 916.5 Cal/g-mol
APPENDIX A.5

SAMPLE CALCULATION

Experimental Data:

Flow rate of air - 660 cm³/min.
Surface area per particle - 1.129 cm²
Number of particles - 32
Loss in weight of naphthalene particles - 0.0696 gm.
Temperature - 29.4°C.
Pressure - 772 mm Hg.
Time - 2 hr.

Calculated Results:

Molecular weight of naphthalene - 128.16
Value of Gas Constant, \( R_N \) - 82.057 cm² atm. gmoles/K.

Equation:

\[ K_g = \frac{N}{a_p (P_s - P_b)} R_N T \]

Rate of evaporation of naphthalene:

\[ N = \frac{0.0696}{2 \times 128.16} = 2.7154 \times 10^{-4} \text{ gmoles/hr.} \]

Total mass transfer area:

\[ a_p = 1.129 \times 32 \]
\[ = 36.128 \text{ cm}^2 \]
Bulk gas partial pressure:

Molar flow rate of air = 1.7668 g/mole/hr.

\[ P_b = \frac{2.7154 \times 10^{-4}}{1.7668 + 2.7154 \times 10^{-4}} \times 1.016 \]

\[ = 1.5613 \times 10^{-4} \text{ atm.} \]

Vapour pressure of naphthalene:

From Appendix (A.4) at 29.4°C \( P_g = 3.5526 \times 10^{-4} \text{ atm.} \)

Mass transfer coefficient:

\[ K_g = \frac{2.7154 \times 10^{-4}}{36.128(3.5526 \times 10^{-4} - 1.5613 \times 10^{-4})} \times 82.057 \times 302.4 \]

\[ = 936.87 \text{ cm/hr.} \]
## APPENDIX A.6

### MASS TRANSFER RESULTS

**Table A.6-1**

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**Particle Type** - I

**Arrangement** - I
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**Particle Type - I**

**Arrangement - II**
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Particle Type - II

Arrangement - Random
### Table A.6-4

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**Particle Type - III** ☐

**Arrangement - III** ☐
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**Arrangement - I**
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Particle Type - III

Arrangement - III
## Table A.6-8

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<table>
<thead>
<tr>
<th>Particle</th>
<th>Arrangement</th>
<th>Effective Area (Fraction of the total area)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TYPE - I</td>
<td>I</td>
<td>0.496</td>
</tr>
<tr>
<td>TYPE - I</td>
<td>II</td>
<td>0.50</td>
</tr>
<tr>
<td>TYPE - II</td>
<td>Random</td>
<td>1</td>
</tr>
<tr>
<td>TYPE - III</td>
<td>III</td>
<td>1</td>
</tr>
</tbody>
</table>

**Calculation:**

1. **Particle Type - I, Arrangement - I**

   \[
   \text{Area available for mass transfer} = \pi \times DL
   \]

   \[
   = \pi \times 0.602 \times (4 \times 0.296)
   \]

   \[
   = 2.239 \text{ cm}^2
   \]

   Total area = 4 \times 1.129

   \[
   = 4.516 \text{ cm}^2
   \]

   Fraction of the total area available = \[
   \frac{2.239}{4.516} = 0.496
   \]

2. **Particle Type - I, Arrangement: II**

   \[
   \text{Area available for mass transfer} = \frac{4 \times (\pi \times D^2)}{4}
   \]

   \[
   = \pi \times (0.602)^2
   \]

   \[
   = 1.1385 \text{ cm}^2
   \]
Fraction of the total area available \( \frac{1.1385}{4.516} = 0.25 \)

ii) Area available for mass transfer (neglecting inner area and considering outer area and edges)

\[
\begin{align*}
&= 4X \frac{\pi}{4} B^2 + 4X (\pi DL) \\
&= \pi (0.602)^2 + 4X \pi X 0.602X X \frac{1}{2} \\
&= 1.1385 + 2.392 \\
&= 3.5775 \text{ cm}^2
\end{align*}
\]

Fraction of the total area available \( \frac{3.5775}{4.516} = 0.75 \)

It seems intuitively reasonable to assume that the ends of the particles are not fully exposed to the flow and hence the effective surface area available for mass transfer is taken as a mean of the areas (I) and (II) as shown above.

Thus fraction of the area for arrangement-II is

\[
\frac{0.25 + 0.75}{2} = 0.5
\]

3. Particle Type-III, Arrangement-III

From Figure (4.1) it may be considered that except for the contact line the entire area is available to flow and hence the fraction of the total area available for mass transfer is taken as unity.

4. Particle Type-II, Arrangement—Random

For the random arrangement the fraction is also taken as unity, since it may be considered that the entire surface area is available to flow and hence mass transfer.