Experimental Investigations on Dissolution of Benzoic Acid in Agitated Vessels

Koppolu Karthik[1], Dr. Pallam Sreedhar[2]

[1]M. Tech, Indian Institute of Technology Madras, Department of Chemical Engineering, Chennai (India)
Email: *kaarthikkoppolu[at]gmail.com*

[2]Faculty, SVU College of Engineering, Department of Chemical Engineering, Tirupati, Andhra Pradesh (India)

Abstract: ***The mass transfer rates occurring in the rotating circular cylinders situated in the middle of the fixed container have been studied in the context of the dissolution rate of the Benzoic acid in water and the Potassium Hydroxide. The increasing stirrer speed causes the enhancement in the rate of dissolution. The larger mould size causes the reduction in the dissolution time. The mass transfer coefficient is decreased with the enhancement in the mould size. The experiment covered the range of the Reynolds numbers from 4500 to 37000. The results are given in the form of Sherwood numbers (Sh) and the Reynolds numbers (Re).*** $N_{Sh} = 0.55(N_{Re})^{0.49}(d/D_t)^{-1.78}$***. For mass transfer with the chemical reaction,*** F_R ***is proposed to be unaffected by the value of the Reynolds numbers.***

Keywords: Benzoic acid, Mass transfer rates, Sherwood number, Reynolds number

1. Introduction

1.1 Molecular Diffusion

Molecular diffusion refers to the movement of individual molecules in a substance due to their thermal energy. The kinetic theory of gases interprets the process of molecular diffusion and shows what happens in that process, and the success makes it a useful quantitative tool to describe diffusive phenomena; for this reason, the kinetic theory has been widely accepted. As a result of molecular diffusion, substances in a solution that may have an initially uniform concentration end up having a perfectly uniform concentration. However, molecular diffusion differs from the faster process of mixing. Molecular diffusion is relatively slower compared to rapid mixing, which is achieved by mechanical stirring and convective fluid motion. The latter mechanical stirring usually results in rapid motions of large fluid eddies; this characterizes turbulent flow.

Rates are defined in terms of molar flux, expressed as mol/ (area) (time) where the area is typically measured in a direction perpendicular to the diffusive process. Diffusivity, the diffusion coefficient, DAB, of the diffused substance A in the solution B, can now be defined as the ratio of the diffusive flux, JA, to the diffusive concentration gradient[1].

$J_A = -D_{AB}\ (\partial C/\partial Z) = -C^*D_{AB}\ (\partial X/\partial Z)$, which is a representation of Fick's first law in the Z-direction. The negative sign indicates that diffusion occurs toward regions of lower concentration. The diffusivity is influenced by the characteristics of the substance as well as its surroundings, including temperature, pressure, concentration, the state of matter (liquid, gas, or solid solution), and the nature of other components involved [1].

1.2 Agitation

Agitation is described as movement of particles when subject to the force in a circular direction within a container. Liquids are agitated for many purposes, Suspending solid particles, Blending miscible liquids, Dispensing a gas through the liquid in form of small baffles, Dispersing a second liquid with first to form an emulsion or suspension of fine drops, Promoting heat exchange between the liquid and cooling jacket [1].

1.3 Agitation Equipment

Liquids are normally mixed in a container, which of is often a vertical cylinder. The top may be open to the atmosphere or covered, and the depth of liquid is on the order of magnitude of the diameter of the tank. An impeller is mounted on an impeller shaft, which is usually hung from and driven by a motor. The action of the impeller creates a pattern of flow in the system, causing the liquid to circulate through the tank and back to the impeller. The flow characteristics in the mixing vessel are determined by the impeller design, the fluid properties, the dimensions and proportions of the tank, whether or not baffles are present, and the agitator [5].

1.4 Suspension of solid particles

Particles of solids are dispersed in liquids for many purposes, mainly to achieve a homogeneous mixture. Most mixing produces flow in the horizontal and vertical directions, besides up and down circulation. To keep solid particles suspended in a tank, much higher velocities are required. If solids are kept suspended in an agitated tank, there are several ways to define the condition of suspension. 1)Nearly complete suspension, 2) Complete particle motion 3) Complete suspension or complete off bottom suspension [6].

1.5 Impellers

Impellers are classified into two types 1. Axial flow impellers and 2. Radial flow impellers.

Axial flow impellers male an angle of less than 90^0 with the shaft. They generate flow currents parallel to the axis of shaft. Radial flow impellers have blades parallel to the axis of the shaft. They generate the flow currents in the tangential to the circular path or radial direction which is perpendicular to shaft. Impellers are further classified into three types 1. Propellers 2. Paddles 3. Turbines.

Propellers and pitched blade turbines are axial flow impellers, whereas paddles, flat blade, curved blade, disc flat bade turbines are radial flow impellers. Axial impellers are used at high speeds to promote rapid dispersion and used at low speeds to keep solids in suspension. Radial flow impellers used for large scale mixing of solid/liquid suspension. A propeller is an axial flow; high speed impeller commonly used for low viscous liquids. It mounted at center, off center or at an angle to the vessel. The diameter of propeller is usually between 15 to 30% of the diameter of the vessel. Propeller drives the liquid straight down to the bottom of vessel, at the bottom the stream spreads radially in all directions towards the wall, then the liquid floes upward along the wall and finally returns to the suction of impeller from the shaft. Turbine impellers drive the liquid radially against the wall, where the stream divides into two portions. One of the portions flows downward to the bottom and then returns to the center of impeller from bellow, while the other flows from upward towards the surface and finally returns to the impeller from the above. Paddle agitators with two or four blades are very common. The blade of these agitators are usually vertical and extended close to the wall of the vessel. They are simply pushers and cause the mass to rotate in laminar swirling motion with practically no radial flow. The circulation is poor and the mixing action is insufficient. Rotate at speed of 20 to 150 RPM. The length of the impeller lies between 50 to 80% of the inside diameter of the vessel [1].

1.6 Benzoic Acid:

Benzoic acid is a colorless crystalline solid that features a carboxylic acid functional group. Its IUPAC designation is benzene carboxylic acid, and it has a CAS number of 65-85-0. The molecular formula is C_6H_5COOH, and its molar mass is 122.12 g/mol. The melting point is 122.50 °C, while the boiling point is 249 °C, although it decomposes at that temperature. Its density is 1.32 g/cm³ at 20 °C. Solubility in water is measured as 3.4 g/L at 25 °C, equivalent to 0.028 mol/L; benzoic acid also dissolves in ethanol, acetone, ether, and benzene, but it does not dissolve in alkanes like hexane. It possesses an acidity with a pKa of 4.2 and undergoes electrophilic aromatic substitution, forming salts and esters with bases and alcohols. When burned, it emits irritating smoke. The heat capacity (Cp) is 1.42 J/g·K, with a heat of vaporization around 58 kJ/mol and a heat of fusion of approximately 18.2 kJ/mol. Benzoic acid is biodegradable and has low toxicity to aquatic organisms at environmental levels, with a lethal dose estimated at about 1700 mg/kg. It can cause irritation to the skin, eyes, and respiratory system, but it is classified as non-carcinogenic [10].

2. Objectives

To study the mass transfer with and without chemical reaction and finding

- Effect of shaft speed.
- Effect of size of mould.
- Estimation of mass transfer coefficient.
- Mass transfer data correlation in terms of dimensionless groups.

3. Experimental Setup

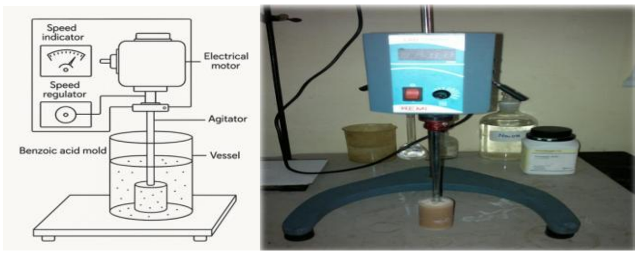

Figure 1: Experimental Setup

4. Experimental Work

These processes employed in the study are presented in the following four categories: (1) Preparation of Benzoic Acid Moulds, (2) mass transfer without a chemical reaction, (3) mass transfer with a chemical reaction, and (4) extent of the variables considered.

Preparation of Benzoic acid

"Pure Benzoic acid" is the solute used while "distilled water" is the solvent. Benzoic acid is normally available in a powdered form. Benzoic acid is packed inside a steel kettle and heated to 130°C, thereby turning into a liquid at that temperature. The molten Benzoic acid is then poured into a Brass mold that consists of a Brass rod at its center. The rod helps form a cylinder that is 3.9 cm and 2.8 cm long with a smooth and sturdy surface. All the resulting cylinders are removed from dust, washed with water, and then dried inside "desiccators" before being ready to be used in the actual experiment. Safety measures are also taken to prevent the inhalation of Benzoic acid vapors that may have adverse effects on one's health. Precautions are also taken to ensure that the Benzoic acid molds are always moisture-free.

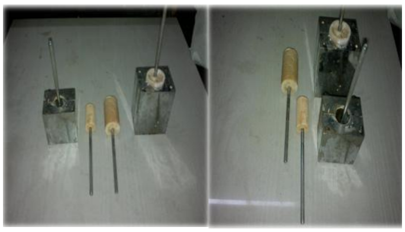

Figure 2: Moulds of Benzoic acid

4.1 Mass Transfer without reaction

Benzoic Acid Molds are placed perfectly in the middle of a vessel containing a fixed volume of distilled water. The rotation speed of the cylinder was set, and samples of 5ml were taken every 10 minutes for titration using standardized Potassium Hydroxide solutions until a constant titration value was obtained. Initial and final measurements for length and diameter were taken before and after the experiment. Measurements for the diameter of each cylinder were taken in four spots, and the average value was used for calculations. The experiment was repeated for different rotation speeds, namely 200, 300, and 400 rpm, using molds of two different sizes.

4.2 Mass Transfer with reaction

A certain amount of Potassium hydroxide solution was filled into a cylindrical container. To this solution, 3–4 drops of the "phenolphthalein" indicator were added, which colored the solution pink; the solution was well mixed before the start of the experiment. The Benzoic acid-laden stirrer continued to rotate till there was a change in color of the solution from pink to colorless, and the time taken for this process was recorded. The solvent used was standardized solution of Potassium hydroxide of concentration 0.01. The initial and final diameter and length of the cylinder are measured before and after the experiment. The rotational speeds at which the experiment is conducted are 200, 300, and 400 rpm, respectively. Mold is supported at the center of the vessel, should not touch the bottom of the vessel, and be properly submerged in the solution [5].

5. Variables

The variables examined includes the following.
With and Without Chemical Reaction
(1) Three distinct shaft speeds
(i) 200 rpm, (ii) 300 rpm , (iii) 400 rpm
Concentration of KOH at 0.01 N

(2) Two varying lengths and diameters
(i) 4.2 and 2.8 cm
(ii) 2.5 and 3.8 cm

6. Result and Discussion

The experimental study performed in the current investigation includes both mass transfer without a chemical reaction and mass transfer with a chemical reaction. More specifically, the investigation includes the mass transfer of Benzoic acid of two different mold sizes at three different speeds in water, as well as the mass transfer of Benzoic acid at the same speed in two different concentrations of the water solution with Potassium hydroxide.

6.1 Mass Transfer without reaction

Study of mass transfer without chemical reactions was conducted using different moulds of solid Benzoic acid in water, and the findings are discussed below:

6.2 Effect of shaft speed

Concentration of Benzoic Acid vs Time curve for small mould is given in Fig 3 below while that of big mould is given in Fig 4 below at three different stirring speeds.

It has been observed that the amount of the acid gradually increases with time at a fixed stirrer speed. The rate of mass transfer is also seen to gradually decline as time passes by, which may be ascribed to the reduction in the driving force due to the increase in the concentration of the acid, which in turn reduces the flux rate. In both cases, the rate of dissolution is increased by the rise in the stirrer speed.

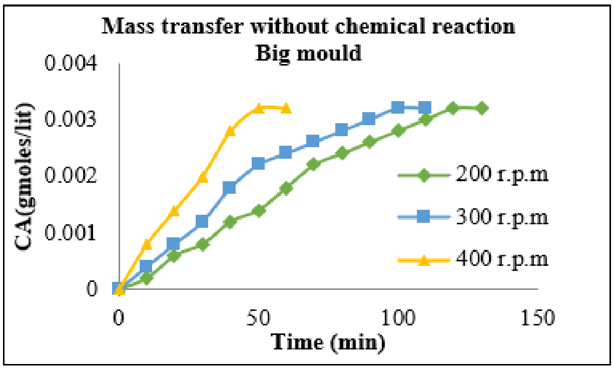

Figure 3: Concentration vs Time for big mould at shaft speed of 200, 300, 400 R.P.M

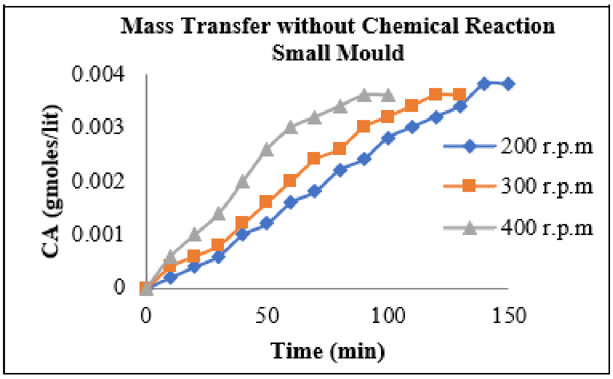

Figure 4: Concentration vs Time for small mould at shaft speed of 200, 300, 400 R.P.M

6.3 Effect of size of mould

Figures 5,6 and 7 shows the acid concentration with time for the two types of moulds used at a stirring speed of 200,300 and 400 respectively. It is observed that the increase in mould size decreases the mass transfer flux. It has been found that the time required for the dissolution of the larger mould is less than that in the case of the small mould. Also, it has been found that for any given time, the concentration for the larger mould is always higher than the smaller mould. The main reason behind this will be due to the fact that the larger mould has a greater surface area than that for the smaller mould.

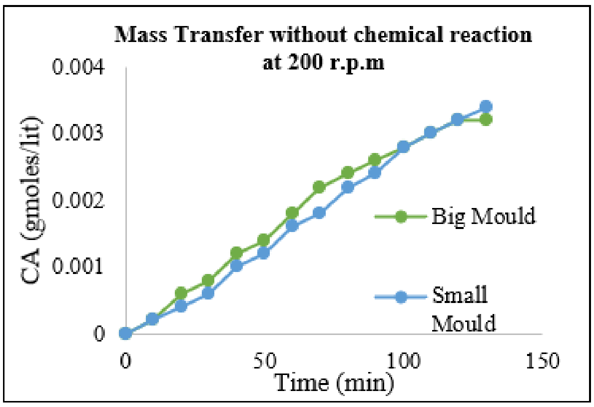

Figure 5: Concentration vs Time for small and big mould at shaft speed of 200 R.P.M

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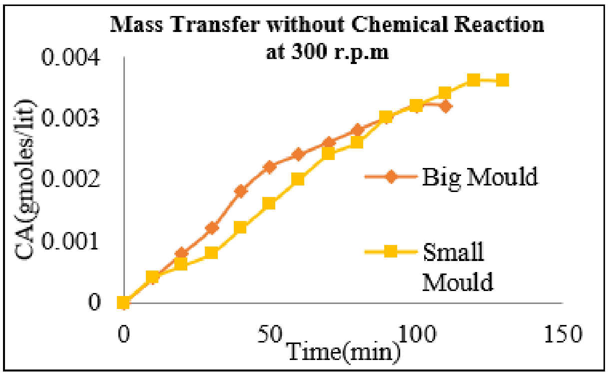

Figure 6: Concentration vs Time for small and big mould at shaft speed of 300 R.P.M

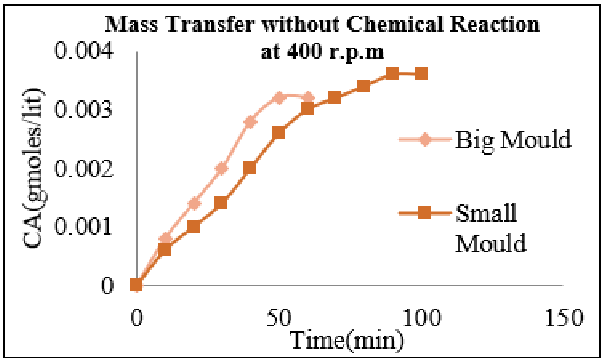

Figure 7: Concentration vs Time for small and big mould at shaft speed of 400 R.P.M

6.4 Estimation of mass transfer coefficients

In the process of solid dissolution, the flux is proportional to the driving force and the proportionality constant, which is called the mass transfer coefficient. Molar flux can be written as:

$$N_A = \frac{1}{a}\frac{dC_A}{dt} = K_s \Delta C_A = K_s(C^* - C_A) \text{ ---- (1)}$$

where C* represents the solubility of the Benzoic acid in water. By integrating Equation 1 we obtain:

$$\ln\left(\frac{C^*}{C^* - C_A}\right) = K_s a t \text{ ------ (2)}$$

Equation 2 illustrates that the plot of $\ln\left(\frac{C^*}{C^*-C_A}\right)$ against time results in a straight line, with a slope equal to $K_s a$. Figures 8 9 10 11 12 and 13 display the relationship between $\ln\left(\frac{C^*}{C^*-C_A}\right)$ and time for two different types of molds at stirring speeds of 200, 300 and 400 rpm, respectively. The data aligns well with a straight line, from which the mass transfer coefficient, "K_s" can be determined using the slope of these lines.Additionally, it has been noted that the mass transfer coefficient reduces as the diameter of the cylindrical mold increases.

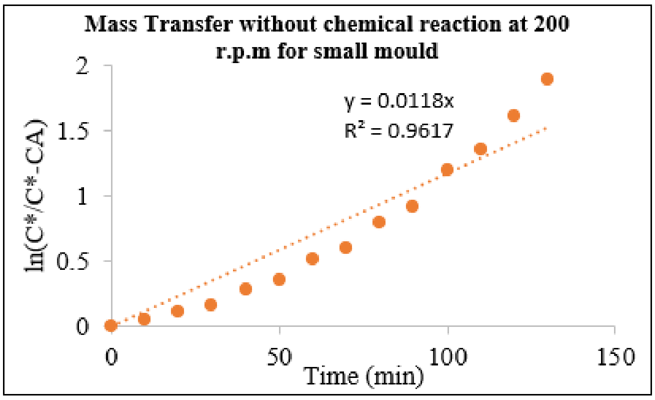

Figure 8: $\ln\left(\frac{C^*}{C^*-C_A}\right)$ vs Time for small mould at 200 R.P.M

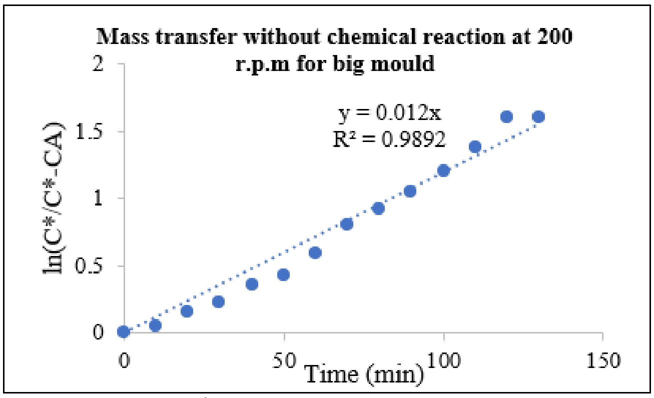

Figure 9: $\ln\left(\frac{C^*}{C^*-C_A}\right)$ vs Time for big mould at 200 R.P.M

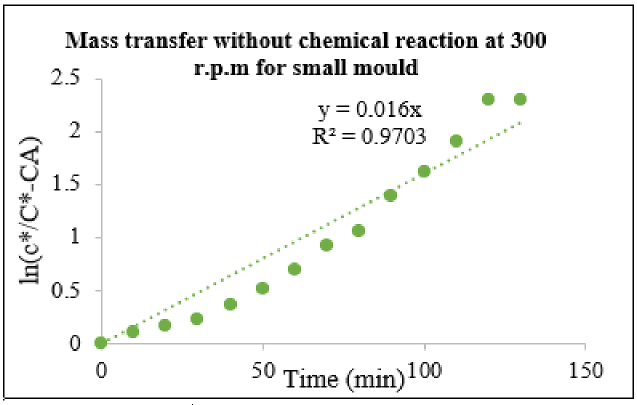

Figure 10: $\ln\left(\frac{C^*}{C^*-C_A}\right)$ vs Time for small mould at 300 R.P.M

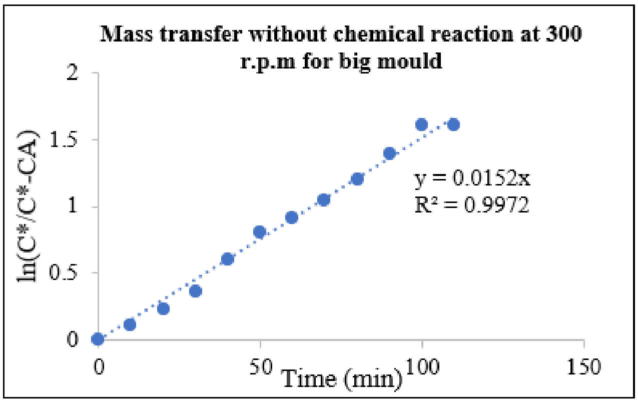

Figure 11: $\ln\left(\frac{C^*}{C^*-C_A}\right)$ vs Time for big mould at 300 R.P.M

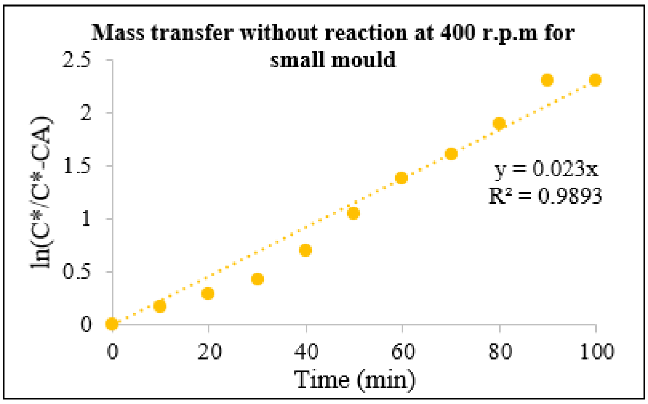

Figure 12: ln $\left(\frac{C^*}{C^*-C_A}\right)$ vs Time for small mould at 400 R.P.M

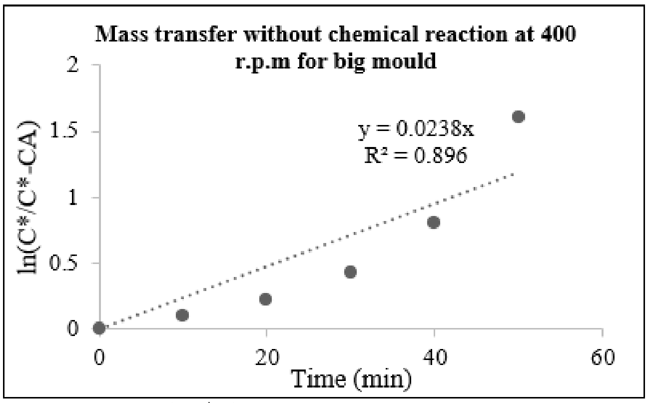

Figure 13: ln $\left(\frac{C^*}{C^*-C_A}\right)$ vs Time for big mould at 400 R.P.M

Table 1: K_s values for both moulds at 200, 300, 400 R.P.M

Speed (R.P.M)	Mould	K_s
200	Small	0.00236
	Big	0.001539
300	Small	0.00336
	Big	0.002147
400	Small	0.00481
	Big	0.003127

6.5 Correlations

The mass transfer data are represented through dimensionless groups, with various researchers utilizing different characteristic length parameters for the Reynolds number, Sherwood number, and other relevant dimensionless parameters linked to the transfer process in agitated systems. In this study, the diameter of the cylinder has been selected as the characteristic length parameter. The size of the mould affects the Sherwood number, and an increase in mould diameter leads to a decrease in the Sherwood number, resulting in a straight line represented by the following equation.

$$Sh \ \alpha \ Re^n \text{ --------------------------------------- } (3)$$

$$Sh = a \ Re^n \text{ ------------------------------------- } (4)$$

The constant "a" differs for the two distinct sizes of solid mould. To account for the size of the solid mould, another dimensionless group is introduced as the ratio of the mould diameter to the tank diameter, leading to the equation for the Sherwood number given by $Sh = B\,(Re)^{n1}\,(d/Dt)^{n2}$.

It is observed that n1 = 0.49 and n2 = 1.78. A least square analysis of the data yielded the following equation $Sh = 0.55\ Re^{.049}(d/Dt)^{-1.78}$.

The values of Reynolds number and Sherwood number for big mould and small mould at different speeds of 200, 300, 400 R.P.M. are tabulated in table 2. The plot of $Sh(d/D_t)^{-1.78}$ vs Re was shown in figure 14.
Diameter of cylindrical vessel D_t = 12.5 cm
Diameter of big mould d = 3.8 cm
Diameter of small moulds d = 2.5 cm

Table 2: Reynolds and Sherwood number for both moulds at 200, 300, 400 R.P.M

	R.P.M	Reynolds Number N_{Re}	Sherwood number Sh
Big mould	200	16362	520
	300	22619	696
	400	27698	972
Small mould	200	6982	522
	300	9637	711
	400	11780	975

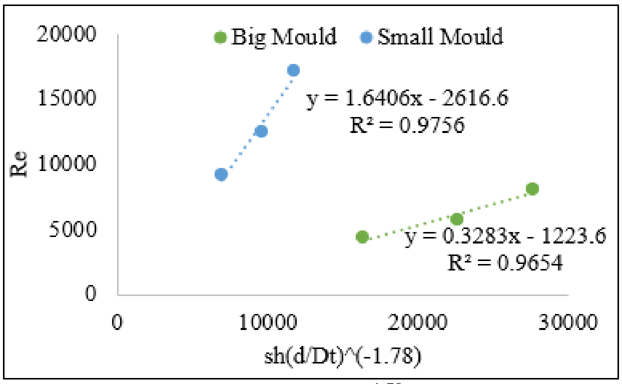

Figure 14: $Sh(d/D_t)^{-1.78}$ vs Re

6.6 Mass Transfer with Chemical Reaction:

A limited study on the effect of alkali in the water on the mass transfer coefficient in the presence of fast /Instantaneous chemical reaction is undertaken, Thus Potassium hydroxide, which reacts with Benzoic acid is included in the system. A cylinder of dimensions (length- 4.2 & 2.8 cm , Dia- 2.5 & 3.8 cm) is suspended in Potassium hydroxide solution of 0.01 N concentration and the stirrer is rotated at 200,300 and 400 rpm respectively, an indicator is also used and the colour change exhibited by the system when all the base in the liquid solution is neutralized by the Benzoic acid transferred due to solid dissolution .As the reaction is instantaneous the acid concentration in bulk liquid is considered to be zero ,throughout the experiment .Hence the driving force is constant throughout the experimental run. From the transferred data mass flux is calculated from which mass transfer coefficient is also estimated. It is observed that the mass transfer coefficient increases with increase in stirring speed and increase in concentration of Potassium hydroxide in aqueous solution. From the data Sherwood numbers are estimated in presence of mass transfer with chemical reaction by defining reaction factor F_R as the ratio of Sherwood numbers with and without chemical reaction , the values of F_R and Reynolds number are shown in table 3. It can be observed that F_R is independent of Reynolds number but increases with increase in Potassium hydroxide concentration.

Table 3: Values of F_R and N_{Re} for both moulds at different speeds at KOH concentration of 0.01

	R.P.M	F_R	N_{Re}
Big Mould	200	2.20	13057
	300	2.54	18434
	400	2.06	23089
Small Mould	200	1.84	5377
	300	1.68	7334
	400	1.73	8400

7. Comparison of benzoic acid and cinnamic acid at 400 R.P.M using Big mould.

We observed that the rate of dissolution of both Cinnamic acid and Benzoic acid is gradually increasing with time. Moreover, when we compare both, it is evident from Figure 15 that the rate of dissolution of benzoic acid is higher compared to cinnamic acid. Also, the mass transfer coefficient of benzoic acid is smaller compared to that of Cinnamic acid.

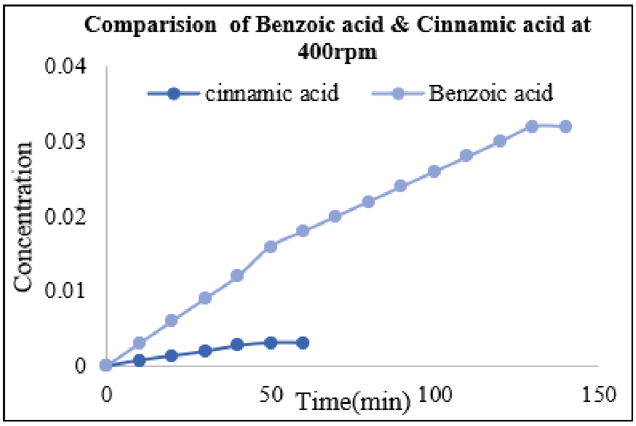

Figure 15: Time vs Concentration for the cinnamic acid and benzoic acid at the shaft speed of 400 R.P.M

8. Conclusions

The study covered a Reynolds number range of 4500 to 37000. The result is presented in the form of Sherwood number (Sh), Reynolds number (Re), and the ratio of mould diameter to the vessel diameter (d/Dt). As the speed of the shaft is increased, the rate of dissolution will also be higher. With the larger mould, the time needed for dissolution will be shorter. As the mould diameter gets larger, the mass transfer coefficient becomes smaller. The relation is given by

$N_{Sh} = 0.55(N_{Re})^{0.49}(d/Dt)^{-1.78}$ is the equation for mass transfer without chemical reaction

For the case of Mass Transfer with Chemical Reaction, the reaction factor (FR) is the ratio of the Sherwood Number in the presence of a chemical reaction to that in the absence of the reaction, and it has also been shown that it is independent of the Reynolds Number.

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