CHAPTER-3

EXPERIMENTAL SETUP AND PROCEDURES

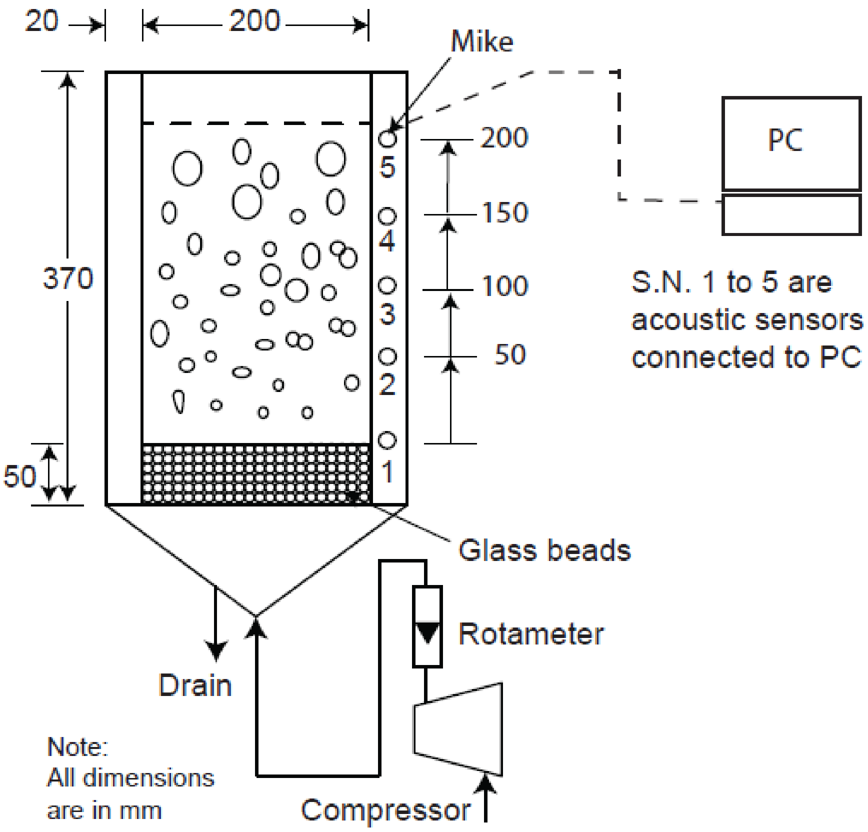
Experimental setup and procedure for acoustic measurements of bubble behaviour have been presented in this chapter. The algorithm for estimation of bubbler size distribution, Sauter-mean bubble diameter and specific interfacial area of bubbles from acoustic signals are also presented.

3.1 Experimental Set-Up:

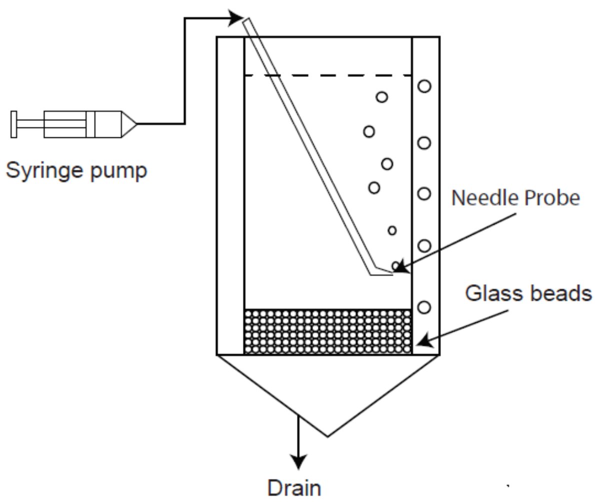
Schematic diagram of present experimental setup is given in Figure 3.1 (a) and 3.1(b). It consisted of a rectangular column with 0.37m height, 0.2m width and 0.02m depth. The set-up was made of Perspex sheet. Two glass walls were used to avoid erosion of the wall and facilitate easy cleaning. It also made visual observation possible.

During experiments with bubble column, air was sparged through a distributor to provide uniform bubbling. The sparger consisted of a perforated plate having 200 holes of 0.0015 m diameter. The holes were placed in a triangular pitch with an opening area of 8.34%. Over the plate, 0.005 m glass beads were filled up to a height of 0.05 m. A 200 mesh of SS (0.00074m Nominal Sieve Opening) was placed over the beads. The glass beads between perforated plate and wire mesh acted as calming section. The bubble column consisted of a conical bottom below the perforated plate. A drain was provided to remove the liquid collected during startup and shutdown of the operation.

The gas was supplied using a compressor. A rotameter was provided for flow rate measurement. On one side of the setup five condenser mikes were provided at 0.0 m, 0.05m, 0.1m, 0.15m and 0.2 m measured from SS mesh.



(a)



(b)

Figure 3.1: (a) Experimental Set-up for bubble column (b) Single bubble study.

Microphones used were electret omnidirectional condenser microphone cartridge of size 9.0×5.0 mm. These were rated to record sound having frequency in the range of 20 Hz-16,000Hz, enough to record all the sounds related to bubble size. The microphones were purchased from the local market. These mikes were fixed on the wall using '**M Seal'** without affecting the recording surface. This side was properly sealed with cotton to avoid the mixing of the produced acoustic signal with outer noise. The cotton was used, for its sound absorbing property due to its porous and fibrous nature. Acoustic signals were measured by one mike at a time.

During experiments with single bubble, a probe 0.5m long and of 0.0015m internal diameter capillary was prepared. It was bent at about 0.003m at the lower end at an angle of 90^0. The bottom tip of the capillary was fitted with a 0.024m long 0.00056m internal diameter needle (Dispo Van injection syringe needle). The probe was connected to a syringe pump (Miclins SYNINGE PUMP, SP-01) using a silicone tube (internal diameter = 0.003m). To produce bubble in the upward direction, the capillary probe was placed into the tank at an angle of 105^0 with the support of a 0.03m×0.03 m ×0.03m movable cubical wooden block placed on the top of the tank (Figure 3.1b). The needle was placed at a height of 0.085m from the bottom of the tank.

A U-Tube manometer filled with carbon tetra-chloride (Density= 1584.39 kg/m^3) was used for measurement of the pressure drop in the bubble column. For this purpose one side of the tank opposite to the side on which mikes were placed, 10 tapping points were provided. All the connections for air supply were provided by 5 mm silica tubes and the entire column was supported on an aluminium stand.

All acoustic signals were measured at 44100 Hz in 'wma' format using microsoft's 'voice recorder' and were converted to 'wav' file before storing.

For validation of the results a few experiments were also conducted to capture images of bubble column. The photographic study was made using a high speed Nikon Camera (J4 Model) was used to capture the images. The column was illuminated from the back of the column using two 500W halogen bulbs.

A photograph of the entire experimental setup is presented in the Figure 3.2.

3.2 Physical Properties of the System:

Experiments were conducted with distilled water, aqueous solutions of Ethylene Glycol (EG), Carboxy Methyl Cellulose (CMC) and sodium hydroxide (NaOH). While EG was used to vary viscosity of the solution, CMC was used to study the applicability of acoustic technique in non-Newtonian solution. NaOH was taken to study the effect of addition of electrolytes. Physical properties of these solutions at 30 ^{o}C are presented in in Table 3.1 and 3.2.

Aqueous solutions of CMC follow power-law given below.

$$\tau_w = K\dot{\gamma}^n \tag{3.1}$$

However, the values of K and n depend upon the molecular weight of CMC. Therefore, these constants have to be determined experimentally. In the present studies, the values of K and n were determined by using capillary flow method. Flow in a capillary with ID=0.001 m and length=1.00 m was measured by collecting the solution for 30 s. The pressure drop, ΔP, is estimated from the applied head as $\Delta P = \rho_L gH$. Apparent shear rate and shear stress at the wall are given as

$$\tau_w = \Delta PR / 2L \tag{3.2}$$

and $$\dot{\gamma} = 4Q \big/ \left(\pi R^3\right) \tag{3.3}$$

Values of K and n were determined by applying non-linear regression to Equation (2.1).

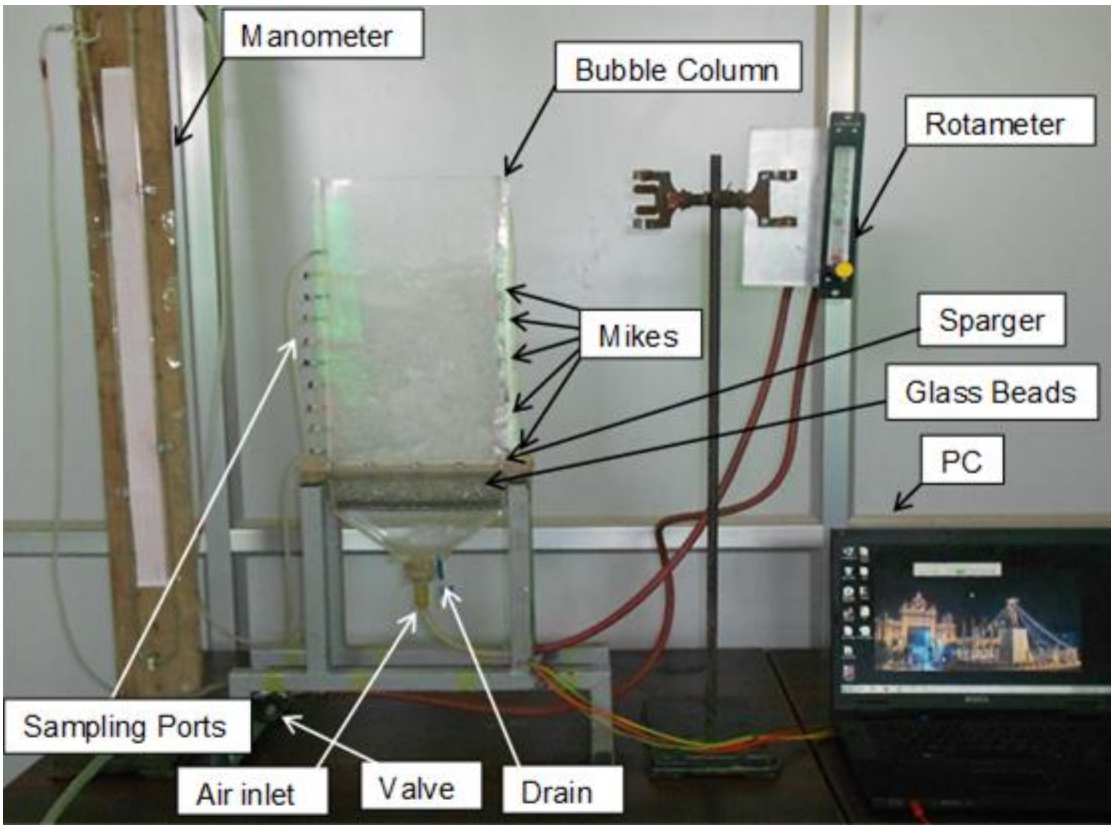


Figure 3.2: Photograph of experimental setup:

Table 3.1: Physical Properties of the Distilled water, aq. Solutions of EG and NaOH.

Liquid	Density $kg\ m^{-3}$	Surface Tension $N\ m^{-1}$	Viscosity $kg\ m^{-1}\ s^{-1}$
Distilled water	996	72.0	0.000894
Ethylene Glycol (0.1 wt%)	996.3	71.9*	0.000897#
Ethylene Glycol (0.5 wt%)	997.5	71.9*	0.000906#
Ethylene Glycol (1.0 wt%)	1002.0	71.2*	0.001029#
NaOH (0.1 wt%)	1001.0**	72.0	0.000903
NaOH (0.5 wt%)	1006.0**	72.1	0.000942
NaOH (1.0 wt%)	1011.0**	72.2	0.000952

* Estimated from data of Jerome et al. (1968)
\# Estimated from data of Nakanishi et al. (1971)
** Estimated from IS 4016:2002.

Table 3.2: Physical Properties of the CMC solution.

Conc. (wt% CMC in water) at 30°C	Density, kg m^{-3}	Surface Tension N m^{-1}	K	n
0.1	998	71.2	0.546	0.22
0.5	998	70.0	0.456	0.23
1.0	999	69.1	0.268	0.26

3.3 Experimental Procedure:

3.3.1 Single Bubble Experiments: Experiments with single bubbles were carried out to study the behaviour single bubble and the distance upto which the condenser mikes can record the acoustic signal. To carry out these experiments tap water in atmospheric condition was filled up to 0.2m height of the column. The capillary tube and needle arrangement was submerged into water filled bubble column. Air was supplied at a particular rate by the syringe pump through the needle probe. The flow rate was kept so low that discrete and distinguishable bubbles were generated. At the same time images and videos were captured using the high speed camera.

Air was pumped through a calibrated syringe pump. The calibration chart is presented in Figure 3.3.

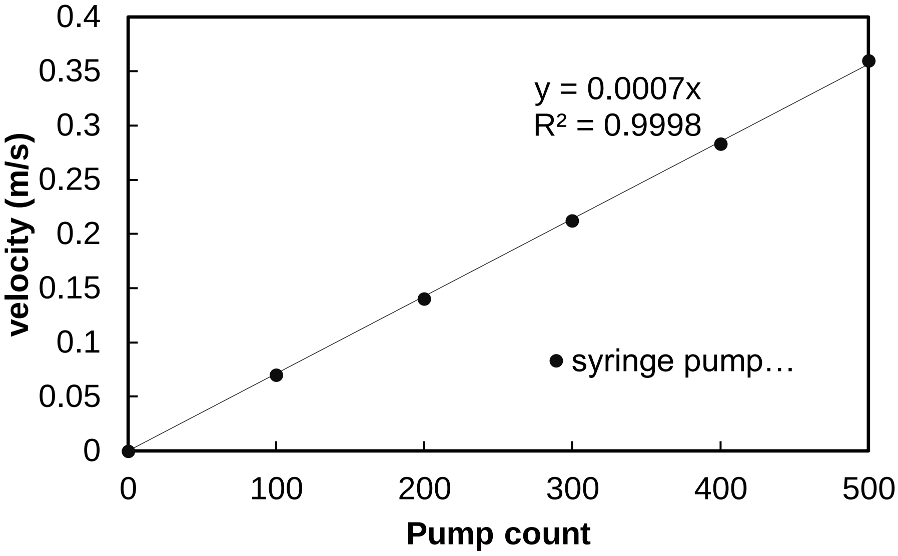


Figure 3.3: Calibration of Syringe Pump.

The distance of the needle probe tip from the wall was placed at the wall and gradually it was moved away from the wall. The acoustic signal was measured after bubbling. It was observed that no acoustic signal was captured beyond a distance of 0.01 m from the wall. It clearly indicates that the condenser mike used measures only acoustic signals produced by bubbles present only ion the close proximity of the wall.

3.3.2 Bubble Column Study: Air from the compressor was supplied through the empty bubble column, and the required flow rate was maintained using the Rotameter for 15 to 20 minutes to achieve steady state conditions. This Rotameter reading was used to estimate superficial gas velocity, U_g. The amount of water was estimated to achieve a particular static bed height. After achieving steady state conditions, required amount of water was poured into the column from top. A 40 ml of water was required for increase bed height by 10.01 m.

Acoustic signal were measured by one condenser mike at a time. The signal was stored as 'wav' file. The room temperature and temperature of liquid in the column were recorded. The Acoustic data were collected for all five different mikes mounted on the column wall for the same experimental conditions. The acoustic data and image/video data were obtained by varying superficial gas velocity and for different static bed height. Same procedure was followed for other liquid solutions as given in Table 3.1 and Table 3.2.

3.4 Acoustic Signal Analysis:

Acoustic signal was analysed in the following manner.

(i) The acoustic signal measured at 44100 Hz as 'wma' files were converted to 'wav' file and stored as 'wav' file. The signal was cropped as non-overlapping sequential sets of 2048 data points. It corresponds to 0.0464 s, during which not more than 4 to 5 bubbles were present.

(ii) Matlab's 'fft' function was use for each such acoustic segment. The peaks in signal were determined using Matlab's 'fndpeak' function with peaks separated by 100 data points.

(iii) The signal has low (<200 Hz) and high frequency components which were not considered due to unrealistic bubble size not confirmed visually. High frequencies may be due to turbulence etc. Bubbles of 0.5 mm were discarded. Low frequency signal may be due to hydrodynamics process such as fluctuation of the liquid height and hence discarded.

(iv) The bubble diameter, d_b, corresponding to each frequency was determined. Since, while obtaining value of d_b, low frequencies were discarded, bubble size smaller than 0.0005 m were not considered. Bubble-size distributions were obtained using MATLAB's 'histogram' command with 25 equally spaced bins. The bubble size distribution (BSD) is plotted between numbers of bubble, N_b, as a function of bubble diameter, d_b, thus obtained. This procedure gave the values of minimum and maximum values of d_b.

(v) Sauter-mean bubble diameter, d_{32}, was estimated using individual values of bubble diameter, d_b in the following formula.

$$d_{32} = \sum_i d_i^3 \Big/ \sum_i d_i^2 \tag{3.4}$$

Any data, for which a mike was not submerged throughout the duration of acoustic signal measurement, was discarded.

3.5 Mass Transfer Studies:

Gas liquid mass transfer rate was measured by dissolved oxygen method. The method was first validated by Titration Method.

3.5.1 Titration method: As soon as the column starts operating, 15 ml of sample was collected from the column after every 3 minutes. Each sample was divided into three parts of 5 ml each and transferred to 20 ml narrow-mouth conical flasks. A few drops of Phenolphthalein indicator were added to the solution. After addition of indicator, the color of the solution turned to light pink. The solution was titrated with freshly prepared ammonia solution (conc. = 0.2 mole/l). ISO 9001: 2008 certified LABRONICS LT-28 model Digital DO Meter was calibrated with the titration method.

3.5.2 DO Meter Method: Initial dissolved oxygen was also measured using the calibrated DO meter. A 15 ml of sample was collected from the bubble column for 30 minutes at an interval of 3 minutes.

These data were used to estimate gas-liquid mass-transfer coefficient.

Analysis of the acoustic signals and data collected during mass transfer studies and the results are presented in the following chapters.

Acoustic signals were measured at 44100 Hz using 'voice recorder' of Microsoft Windows. Such recordings were made at several superficial gas velocities, U_g, and static bed height, H_s = 0.10, 0.15 and 0.20 m and height above the sparger, Z = 0.00, 0.05 and 0.15 m for air-water system and air-aq. soln. of ethylene glycol. The value of U_g was varied in the range of 0.00417 – 0.167 m s^{-1} for air-water system and in the range of 0.0556 – 0.222 m s^{-1} air-aq. soln. of ethylene glycol.