

## 2.1 Introduction:

Present investigation was undertaken with a view to study in detail certain aspects of drop dynamics and mass transfer from falling drops in a liquid-liquid spray extraction column. This involved the measurement of drop size and frequency of formation, drop velocity during free fall and mass transfer during formation and free fall periods.

Different concentrations of acetone and methylethylketone in aqueous solutions were used as dispersed phase and kerosene as continuous phase. The systems were so chosen that besides the dynamics, both dispersed and continuous phase resistances could also be studied for mass transfer mechanism. In addition, liquids with high interfacial tension ( $>21$  dynes/cm) had been selected to study the terminal velocity for oscillating drops.

## 2.2 Experimental set-up:

The investigation was carried out in a glass test column provided with a distributor at the top to disperse a single stream of liquid drops and an exit valve at the bottom to take out the collected drops from the column. The schematic diagram of the experimental set-up is shown in Fig.2.1.

The dispersed phase liquid was stored in an overhead feed tank (1) from which it flowed to a glass cylinder (2) provided with a constant level arrangement. The liquid from the constant head cylinder entered the extraction column (6) through a fine control stainless steel needle valve (4), with accurately calibrated precision flowmeter (3) and a distributor (9). The liquid

drops after travelling through the continuous phase were withdrawn from the column through an exit valve (7).

The extraction column consisted of a 8 mm thick pyrex glass column of 15.5 cm internal diameter and 150 cm height. The diameter of the column was large enough to eliminate wall effect<sup>1</sup>. The column was erected vertically on a slotted angle-iron frame provided with proper supports (Plate 2.1). The column details are shown in Fig.2.2.

The flanged bottom end of the glass column was fitted to a conical section (5) made of glass. A stainless steel needle valve (6) was attached to the end of this conical section for withdrawing the dispersed phase. The purpose of this part was to maintain an interfacial area, as small as possible, for the dispersed phase after reaching the column end<sup>2</sup>.

A short cylindrical section (8) of mild steel of the same internal diameter as that of the column was attached to the top of the column with flanges and is shown in Fig.2.2. The cylindrical section had two inlets, one for the introduction of the continuous phase without disturbing the distributor and the other for the introduction of the dispersed phase through the distributor.

The stainless steel distributor (7) was made in three parts: (1) the distributor cone, (2) the orifice plate, and (3) the nozzle. The distributor details are shown in Fig.2.3. The cone end was joined with the stainless steel feed pipe. Nozzles and hypodermic needles of different inside diameters were screwed to the orifice plate. Stainless steel and brass nozzles of different inside

diameters were carefully fabricated and finished with standard reamers and their diameters were measured accurately with a microscope. Dimensions of the nozzles used in the present work are summarised in Table 2.1.

## 2.3 Experimental techniques and procedures

### 2.3.1 Drop frequency and drop size measurement.

The test column was filled with the continuous phase liquid till the nozzle tip touched the liquid level. The feed from the constant head entered the column through the distributor. The exit valve at the column outlet was then adjusted accordingly such that a constant coalescing interfacial layer was maintained at the narrowest section of the conical section. At steady state condition, the time taken for formation and detachment of ten drops were noted. Ten such readings were taken for each flow rate using different dispersed phases, from which the drop frequency and drop-size were determined.

### 2.3.2 Measurement of free fall velocity of the drops.

The free fall velocity of the drops was measured for the entire column height for very short intervals of length. The technique adopted is as follows:

Two extremely narrow beams of strong light were allowed to pass through the liquid column at two accurately measured heights. Two photocells were placed in the paths of the light beams emerging from the column. Phillips OC71, phototransistors were modified to photodiodes and used as photosensitive energisers for this purpose. The photocells were then connected to the input terminals of two

identical auxiliary circuits whose outputs were fed to an electronic Time Interval Meter, (Airmec Ltd. Type 771). The intensity of light focussed on the photodiodes was varied by passage of the drops across the light beams, due to large a difference in refractive index of the dispersed and continuous phases. These variations in the photocell inputs were sufficient to energise the auxiliary circuits which, in turn could activate the Time Interval Meter.

The schematic diagram of the arrangement for measuring the drop velocity is shown in Fig. 2.4. The circuit diagram of the auxiliary circuits is given in Fig. 2.5. It was designed and assembled for use with the photocells and the Time Interval Meter. The travelling time of the droplets was measured accurately upto 0.001 se with this arrangement. Two suitable structures held the light source and photodiodes at appropriate positions. Each of these structures held one light source and the corresponding photocell and could be set at different heights of the column without disturbing the focussing of the light beams on the photodiodes.

Two 500 watt projection lamps were used as light sources. Light from these sources were allowed to pass through suitably located apertures to avoid total internal reflection and focussed on the photodiodes through a set of lenses. The lamps were connected to mains through a voltage stabiliser to maintain constant intensity and were cooled by airblast for continuous working. The experiments were carried out in a dark room and at 30°C.

The height of the column between the two light beams was calibrated first. The continuous phase liquid was filled up in the column till the nozzle tip just dipped into the liquid. The

first light beam was set at the position where the drop would just release from the nozzle tip and the photodiode was properly aligned to receive the light beam. The second light beam and photodiode were set at a distance of 10 cm from the first one. The output voltage of the circuit was checked with an avometer for proper functioning of the Time Interval Meter. In order to eliminate any refraction error of the light beams on passing through the column, horizontality of the beams on both sides of the column was checked by a circular gauge.

The flow rate of the dispersed phase and the exit valve were now adjusted. As the drop crossed the first light beam, the Time Interval Meter started on and when the same drop crossed the second light beam, the meter closed. The time taken for the drop to travel 10 cm was recorded correct to one millisecond. The meter and the auxiliary circuits were reset for the next set of readings.

The experiment was conducted for different solutions at various flow rates and different frequencies of drop formation throughout the entire column height at an interval of 10 cm.

### 2.3.3 Estimation of mass transfer from drops.

During experimental runs, the height of the continuous phase was varied by filling up the liquid in the column upto the required height and lowering the distributor head accordingly till the nozzle tip just dipped into the liquid. The inlet and exit valves were carefully adjusted so as to maintain the interfacial layer constant at the narrowest section of the conical bottom for any required flow rate of the dispersed phase. The drops after passing through the continuous phase were collected in sampling

bottles. Ten such samples were collected for analysis for each run.

The refractive index of the collected samples was measured at constant temperature with an Abbe Refractometer and solute content determined from previously calibrated plots of refractive index vs. concentration (Figs.2.7 and 2.8). The quantitative estimation of acetone in acetone-water solutions was also made by Messinger's iodoform method.

The continuous phase liquid was usually taken out from the extraction column at an interval of one hour to avoid appreciable buildup of the solute. The kerosene was washed with water to remove the solute and dried in a packed column of activated silica gel. Regenerated kerosene was recycled for the experiment.

The time of contact of the drops with the continuous phase was measured with the electronic set-up described earlier.

#### 2.3.4 Schlieren technique for studying interphase mass transfer.

Experiments on interphase mass transfer during drop formation were carried out using Schlieren photographic technique<sup>3,4</sup>. The Schematic diagram of the Schlieren set-up is shown in Fig.2.6. The light source, S, was placed at the focus of a concave mirror  $M_1$ , so that suitably positioned working section specially the nozzle and its immediate vicinity were illuminated by the parallel beam of light of the Schlieren system. A second concave mirror  $M_2$  placed beyond the working section, produced an image of the source in its focal plane K, beyond which a focussing lens L was used

to give an image of the model on a screen. The light source used was a high pressure mercury vapour lamp (Phillips SP 900 W). Light from the source was allowed to pass through a square aperture. The lamp was cooled by an airblast for continuous running.

The working section W consisted of a rectangular chamber made of perspex. Two parallel sides of this chamber focussing in the path of the light beam were provided with optically homogeneous glass plate to avoid any deflection of the light beam. The volume of this chamber was large enough compared to the drop volume to eliminate wall effect and appreciable build up of solute in the continuous phase.

The continuous phase liquid was filled up in the working chamber till the nozzle tip dipped into the liquid. Drops were allowed to form at the nozzle tip at the required frequency of formation and the working section illuminated by switching on the light source. Shadow Schlieren photographs of the drops were taken at suitable intervals with a high speed camera Leica 111f and panchromatic film Ilford HP3, 27DIN. An exposure time of  $1/1000$ th second was used during the experiment and it was done in a dark room. Visual observations of the Schlieren images were made by projecting the images of the droplet on a screen which was suitably positioned for this experiment.

Mass transfer studies during drop formation were carried out in this chamber containing the continuous phase. As soon as the drops were formed at the nozzle tip, they could be removed immediately and the concentrations were checked by a polarograph.