D. BUBBLE SIZE DISTRIBUTION MEASUREMENTS

The bubble size distribution, together with the average gas hold-up determines the gas-liquid interfacial area available for mass transfer. Bubble properties such as bubble size and rise velocity, and the axial and radial profiles of bubble size distributions are very important in analyzing the hydrodynamics and gas-liquid mass transfer in the slurry bubble column reactor. Studies were undertaken to determine the bubble size distributions for FT-300 wax and reactor waxes (Sasol's Arge reactor wax and Mobil reactor wax). The effect of operating and design parameters were also investigated during the course of these studies.

The Sauter mean bubble diameter (d_s) is commonly used to represent the bubble size distributions for mass transfer studies in two-phase systems. It is a volume to surface ratio and together with the average hold-up value (ϵ_g) determines the specific gas-liquid interfacial area (a).

The specific gas-liquid interfacial area is the total surface area of all bubbles in the dispersion divided by the volume (V_T) of the dispersion. However, V_T can be obtained by dividing the total volume of the bubbles by the void fraction (or gas hold-up). Assuming spherical bubbles, the following equations can then be used to obtain a definition for the Sauter mean bubble diameter:

$$a = \frac{\Sigma \pi d_i^2}{V_T} = \frac{6\epsilon_g \Sigma d_i^2}{\Sigma d_i^3} = \frac{6\epsilon_g}{d_s} \qquad \text{(V-2)}$$

where

$$d_s = \frac{\Sigma d_i^3}{\Sigma d_i^2} \qquad \text{(V-3)}$$

The diameters of bubbles in a representative sample of the bubble population can now be used to determine the Sauter mean bubble diameter and therefore the specific interfacial area.

D.1. Selection of Techniques

Several techniques are available for the measurement of bubble size distributions. Detailed reviews of these techniques along with their limitations are available in literature (e.g. Buchholz and Schugerl, 1979). Two such techniques were selected for our work; the photographic technique and the dynamic gas disengagement (DGD) technique. These techniques were the most suitable for the hostile environment in the hot flow columns. The photographic technique provides point estimates of the bubble size distributions at various locations in the bubble column, whereas the DGD technique gives an average bubble size distribution for the entire dispersion.

D.2. Bubble Size Distribution Using The Photographic Technique

The photographic method is a direct procedure for measuring bubble size distributions. Despite the tedious process of identifying individual bubbles, this technique is by far the most widely used method for obtaining bubble sizes. The photographic technique was used to obtain estimates of bubble size distributions in FT-300 wax. Selected photographs of the bubbles were enlarged and bubble sizes measured using image analysis. Experiments were conducted in the 0.051 m ID and 0.229 m ID glass columns, and the 0.241 m ID stainless steel column in order to study the effect of various parameters on the bubble size distribution. Majority of the experiments were conducted at 265°C, with a few at 200°C in order to study the effect of temperature. The 1.85 mm orifice plate and the 40 μm sintered metal plate (SMP) distributors were employed in the 0.051 m ID column, while the 19 X 1.95 mm perforated plate distributor was used in the 0.229 and 0.241 m ID columns.

The major highlights of these investigations are:

- The Sauter mean bubble diameter (d_s) decreases with an increase in height above the distributor. The extent of this decrease depends on the flow regime, with more significant decreases in cases where foam is present.
- The presence of foam results in lower Sauter mean bubble diameters compared to values when foam is absent. Sauter mean diameter for pure foam was found to be around 0.5 mm.
- The value of d_s appears to be fairly constant with changes in superficial gas velocity in the 0.051 m ID column, however in the large columns (0.229 m ID glass and 0.241 m ID SS), it decreases marginally with an increase in superficial gas velocity. In general, d_s approaches a fairly constant value for higher values of u_g (> 0.07 m/s).
- The 40μm SMF distributor produced significantly smaller bubbles (d_s = 0.5-0.7 mm at 265°C) compared to the 1.85 mm orifice plate distributor (d_s - 1.2-2.2 mm under similar conditions). However, d_s in the 0.229 m and 0.241 m columns, with improvements in the technique (significantly larger bubble count), were around 0.5 mm at the wall and around 0.8 mm at the center (for velocities greater than 0.05 m/s). The latter values are comparable with results reported in literature for studies conducted using molten wax as the liquid medium.
- Sauter mean diameter is greatly affected by radial position with larger values in the center of the column than at the column wall

This is compatible with observations reported in literature for the air-water system.

- In the presence of oxygenates lower Sauter mean bubble diameters were obtained.
- Temperature did not have a significant effect on d_s for the 0.051 m ID column, whereas d_s decreased (between 25-50% relative) in the 0.241 m ID column as temperature was decreased from 265°C to 200°C.

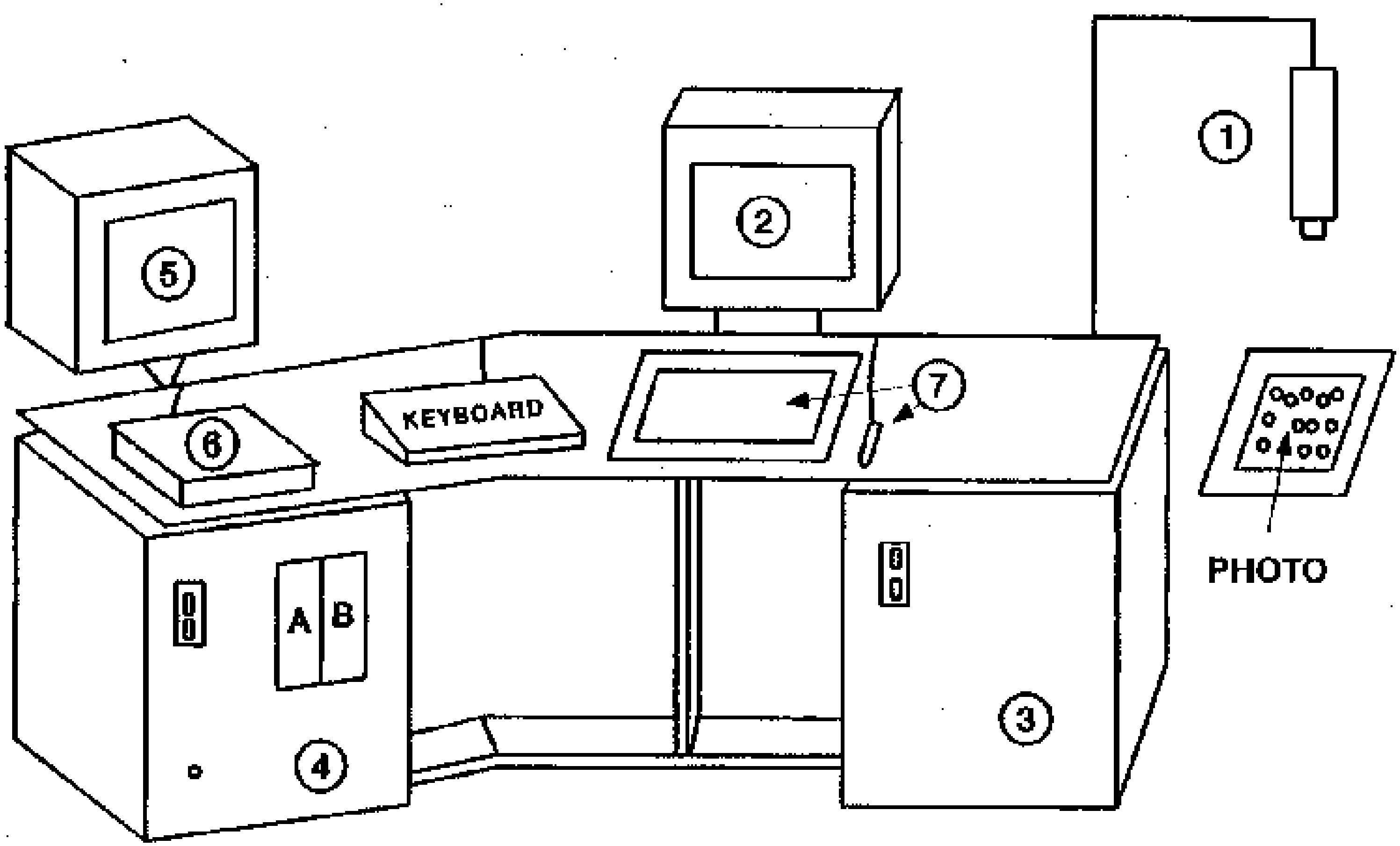
D.2a. Image Analysis

Photographs selected for analysis were enlarged to 8" by 10" and analyzed on a Zeiss image analyzer located in the Biology Department at the University of Texas at Austin. An illustration of the image analysis system is shown in Figure V-48. A description of the hardware and software associated with image analysis is given below.

D.2a.1. Hardware

The TV camera (1) converts an image of the photograph falling on its light sensitive pickup tube into an analog electrical signal. This signal is sent to an array processor, IBAS II (3), where it is converted to numerical form by an analog to digital converter so that it can be manipulated by the computer. Processed images are sent through a digital to analog converter and then to a display monitor (2). The image analyzer is only able to distinguish between shades of gray, therefore, when bubbles overlap, the image analyzer treats such bubbles as a single bubble. However, IBAS II allows the user to edit the original image in order to circumvent this problem. This is done using the digitizer tablet with mouse or light pen (7). With the light pen the user can trace over the bubbles that are to be analyzed. Once this is complete, a variety of object speci-

FIGURE V-48
IMAGE PROCESSING SYSTEM



1 - TV CAMERA 2 - DISPLAY MONITOR 3 - IBAS II 4 - IBAS I
5 - DATA MONITOR 6 - PRINTER 7 - DIGITIZER TABLET & LIGHT PEN

fic parameters can be calculated and displayed on the data monitor (5) and/or printer (6). A host computer, IBAS I (4), performs the tasks of a manager. It communicates with the user, manages the image processing programs (or software) for IBAS II, and is also capable of some statistical analysis. IBAS I is equipped with floppy disk drives and hard disk drives that allow the storage of parameters for further analysis.

D.2a.2. Software

The image analysis software consists of two separate programs, the first program controls the operations of the array processor (IBAS II) and the second program is used to perform statistical analysis on data generated by IBAS II. Both programs are stored on a floppy disk and are loaded into the computer's memory using the disk drives in IBAS I.

IBAS II Controller Program: This program consists of predefined function groups and functions that allow the image analyzer to perform the various tasks necessary for bubble size measurements. Following is a list of tasks handled by this program:

a. Input of image into the computer.

b. Scaling of the image or calibration: A wire piece of known length (usually 12 mm long) is glued to the wall of the bubble column in the field of the camera used to take photographs. Thus, every photograph of the bubbles also contains an image of this reference piece. During the calibration process the user locates the two ends of the reference piece on the TV image and marks them using the light pen and digitizer tablet. A corresponding value of the actual length is also entered into the computer. The computer then evaluates the scaling factor.

c. Enhancement of the image in order to correct for uneven lighting or poor contrast in the photographs.

d. Interactive editing, which allows the user to trace the individual bubbles using the light pen and digitizer. Bubbles that overlap or touch one another are separated by redrawing them in open spaces on the image. At the end of this step only those bubbles have been identified which are clear and distinct. This step involves some subjective judgement on the part of the user, thus introducing human error. There is a definite bias toward the inclusion of a greater proportion of larger bubbles at the expense of tiny bubbles. The implication of this prejudice towards larger bubbles is discussed later in the Section V-D.2e.

e. The actual sizing and classification of bubbles based on the parameters selected (see Section V D 2b.).

Statistical Analysis Program: This program requires very little user interaction. It reads the data output by the previous program and performs the necessary statistical calculations. After the initial classification of bubble sizes into 40 classes, it was found that in majority of the cases 95% of the bubbles were classified in the first 15 to 20 classes with the other 5% distributed in the remaining 20 to 25 classes. In order to improve upon this distribution, bubbles in the first 15 to 20 classes were reclassified into 40 classes instead. As a result, for each photograph, bubbles were distributed in 60 to 65 classes or groups based on the diameter of the area equivalent circle (d_B). Three user-defined parameters were estimated for each bubble; $(d_{MAX}+d_{MIN})/2$, d_B^2, and d_B^3. The following statistical quantities were also calculated for each of these parameters for all bubbles in a given photograph: arithmetic and geometric means, variance, and the second and third moments. The program also produced a plot of the number frequency of bubbles as a function of bubble diameter (histogram) and a cumulative frequency plot (or a plot of the total number of bubbles smaller than a given diameter versus bubble diameter) of bubbles in a given photograph, based on the d_B values for the groups.

D.2a.3. Data Reduction - Sauter Mean Diameters

Data obtained from image analysis were used to estimate the Sauter mean diameter d_s for bubbles in individual photographs. Two values of Sauter mean diameter were calculated. The first was estimated from diameters of individual bubbles using Equation (V-4).

$$d_s = \frac{\Sigma d_{Bi}^3}{\Sigma d_{Bi}^2} \qquad \text{(V-4)}$$

where d_{Bi} is the diameter of the area equivalent circle. The second value of d_s was estimated for the bubble groups (classified during the image analysis step). The groups were formed according to the size of bubbles, and typically between 60 to 65 bubble groups were formed for a given photograph. The upper and lower size limits for each group were averaged and it was assumed that this averaged value represented the diameter of all bubbles in that group. Equation (V-5) was then used to estimate a d_s value for these groups.

$$d_s = \frac{\Sigma n_i d_{Bi}^3}{\Sigma n_i d_{Bi}^2} \qquad \text{(V-5)}$$

where d_{Bi} is the representative diameter for the i'th group and n_i is the number of bubbles in that group. The difference between the two values of

d_s was less than 0.1% for most cases. Therefore, parameters based on groups were used in subsequent analysis. A cumulative density function was generated for each photograph analyzed.

D.2b. Parameter Selection for Bubble Size Measurements

The photographic technique to measure bubble size distributions approximates the true particle size distribution (particles having three dimensions) by a profile size distribution (measurements in two dimensions). Errors involved in this approximation depend partly on the parameter chosen to represent the diameter of the bubble. The size of a particle or bubble in profile can be characterized by four different measures (Weibel, 1980).

a. Maximum and minimum diameters: These are also defined as the largest and smallest caliper diameters. A caliper diameter is defined as the distance between two parallel lines that are tangent to the particle profile. These dimensions in effect describe the shape of the particle. An average of these two dimensions could be used as a representative diameter of the particle.

b. Feret diameter: This is a random caliper diameter. It is measured by the distance between two parallel lines that are tangent to a particle which is in any random orientation. However, when a profile is used to measure the diameter of a solid particle, the mean Feret diameter would be a better representation of the particle's dimension.

c. Chord or intercept length: This is the intercept of a random test line by the particle's boundaries. A mean value for this length could be used as the particle diameter.

d. Area equivalent diameter: This is the diameter of the circle which has the same area as the particle.

The image analysis system can measure all four diameters, however the area equivalent diameter requires the least amount of computation. It has been shown (Weibel, 1980) that for a circle, the mean Feret diameter and the area equivalent diameter are the same, however the mean chord length is slightly lower (mean chord length = 0.79 times area equivalent diameter). However, for ellipses of axial ratio greater than one, the following relation holds true:

mean Feret diameter > area equivalent diameter > mean chord length

The four parameters discussed above were estimated from image analysis results for photographs of the FT-300 wax - nitrogen system. The results are summarized in Table V-1. These values are in agreement with the relationship given above for ellipses.

Akita and Yoshida (1974) used the average of the minimum and maximum diameters to represent the size of bubbles obtained from photographic measurements in their study. In the present study it was decided that the area equivalent diameter would be used as an estimate of the bubble diameter. The choice of the area equivalent diameter is justified by two factors; (1) it requires the least amount of computation, and (2) the primary purpose of measuring diameter is to use it to estimate the Sauter mean diameter, which assumes a spherical particle. As shown in Table V-1, the area equivalent diameter is between 4 to 10% lower than the average of the maximum and minimum diameters. This difference is not too significant considering other sources of error.

Table V-1. Comparison between parameters used for bubble diameter measurements (FT-300 wax, 265°C, 0.051 m ID column, 1.85 mm orifice plate distributor)

u_g (m/s)	Height (m)	Parameter value (mm)			
		d_{ave}	d_{Feret}	d_{chord}	d_{circ}
0.01	0.3	0.69	0.74	0.45	0.65
0.01	1.2	0.81	0.87	0.54	0.77
0.01	1.8	0.52	0.56	0.35	0.50
0.02	1.2	0.61	0.66	0.39	0.58
0.03	0.3	0.59	0.63	0.38	0.56
0.03	1.2	0.59	0.63	0.38	0.55
0.03	1.8	0.45	0.52	0.26	0.40
0.04	0.3	0.63	0.72	0.35	0.56
0.04	1.2	0.67	0.76	0.37	0.60
0.04	1.8	0.46	0.52	0.26	0.41
0.07	1.2	0.51	0.58	0.29	0.46
0.09	0.3	0.53	0.62	0.30	0.49
0.09	1.2	0.58	0.62	0.30	0.52

D.2c. Experimental Procedure

Various lighting arrangements, cameras, and lenses were tried in the 0.051 m ID column in order to improve the quality of photographs for bubble size measurements. The best arrangement for this column consisted of two 1000 watt lights (Colortran) placed at angles of 90° with respect to the front of the column in a staggered position (i.e. one 0.15 m above the field of view and the other 0.15 m below the field of view). A shield (flat black metal plate) was placed between the lower light and the field of view. Milar paper was placed between the field of view and the light at the top in order to reduce the glare. A Canon, AE1/P (35 mm SLR) camera was used with Canon auto bellows and a 135 mm Canon lens with a polarized filter. Photographs were taken for all velocities (0.01 to 0.12 m/s) at heights of 0.45, 1.20 and 1.95 m, except when foam filled the entire column (photographs were taken only at 1.20 m for such cases).

Two different lighting arrangements were used while photographing bubbles in the 0.229 m ID column, one for low gas velocities (< 0.05 m/s) and one for gas velocities greater than 0.05 m/s. A Vivitar Model 283 flash was attached to the Canon camera for all photographs. The flash was mounted approximately 0.25 m from the column at an angle of 45° with respect to the front face of the column. At low gas velocities, the Canon 135 mm lens mounted on the Canon auto bellows with an extension of 110 mm was employed. At higher velocities, a 50 mm Canon lens was mounted on the Canon auto bellows with an extension of 70 mm. An f/stop of 16 was found to produce the best results. Photographs were taken for all velocities at heights of 0.41, 1.12 and 1.96 m. The arrangement used in the 0.241 m ID stainless steel column (viewing port) was the same as that used with the 0.229 m ID column at higher gas velocities.

The photographs were taken after a run time of one hour at a given velocity, for velocities of 0.01 to 0.05 m/s, and after 45 minutes for gas velocities greater than 0.05 m/s. A minimum of four photographs were taken at each velocity using Kodak 400 ASA Tri-X Pan film. In the 0.229 m ID column, photographs were also taken just after the gas input to the column was shut off, after each velocity. This was done in order to obtain bubble size distributions after the large bubbles had disengaged from the dispersion.

A major problem with the use of the photographic technique with cylindrical columns is the 'curvature effect' The curvature of the column wall distorts the image of the bubbles in the column since the wall behaves like a convex lens. In order to alleviate this problem, the area of the column that was photographed was kept at a minimum (approximately 15 X 20 mm). The distance between the lens and the column wall was typically between 30 to 50 mm This also helped in the image analysis procedure since the individual bubbles were more distinct when photographed at a close range.

D.2d Experimental Results

The photographic technique could be used only with FT-300 wax because of its clarity and water-like appearance. Measurements were made mostly in the 0.051 m ID and 0.229 m ID glass columns, with some photographs of the flow field near the center of the column taken through the specially constructed port (see Figure V-79) in the 0.241 m ID stainless steel column. It was not possible to use this technique with the Mobil and Sasol reactor waxes due to their dark color.