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**A STUDY OF
MULTISTAGE/MULTIFUNCTION COLUMN
FOR FINE PARTICLE SEPARATION**

Quarterly
~~ANNUAL~~ TECHNICAL PROGRESS REPORT
(~~July 8, 1994 - July 7, 1995~~)
*considered 2nd quart. 1995
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1.0 INTRODUCTION

The overall objective of the proposed research program is to explore the potential application of a new invention involving a multistage column equipped with vortex-inducing loop-flow contactors (hereafter referred to as **the multistage column**) for fine coal cleaning process. The research work will identify the design parameters and their effects on the performance of the separation process. The results of this study will provide an engineering basis for further development of this technology in coal cleaning and in the general areas of fluid/particle separation.

In the first year of the project, we completed **equipment design, construction of the new column for hydrodynamic tests and gas holdup measurements**. Also, we have initiated the **determination of bubble size** as part of the hydrodynamic measurements. The program schedule is shown in Table 1.1. A detailed description of technical progress is presented below.

Table 1.1 Project Schedule

Tasks	1994				1995				1996				1997			
	7	9	12	3	6	9	12	3	6	9	12	3	6			
1. Project Planning	■															
2. Equipment		■	■	■												
3. Hydrodynamic Tests				■	■	■	■									
4. Beneficiation Tests							■	■	■	■	■	■	■			
5. Conventional Column Tests									■	■	■	■	■			
6. Data Analysis				■	■	■	■	■	■	■	■	■	■			
7. Reports	□	□	□	■	■	■	■	■	■	■	■	■	■			

Notes: □ = Quarterly Technical Progress Report;
 ■ = Annual Report; ● = Final Report.

2.0 TECHNICAL PROGRESS

2.1 Task 1: Project Planning

During the first year of the project (July 8, 1994 - July 7, 1995), significant progress has been made in the preparation of this experimental study of a multistage column for fine coal cleaning. A project schedule (see Table 1.1) has been developed, and technical issues and approaches associated with the project (see Table 2.1) have been addressed. A research team has been assembled (see Figure 2.1.1).

2.2 Task 2: Equipment Design and Construction

The equipment design and construction have been completed. Shake-down tests in the new column have also been carried out. Both liquid and gas flowmeters, as well as an agitator speed controller have been calibrated. A U-tube manometer system, a high-speed videocorder, an optical fiber viewing device and a conductivity meter have been tested.

2.3 Task 3: Hydrodynamic Tests

2.3.1 Objective and Accomplishments

The objective of this task is to pursue a basic understanding of the hydrodynamic behavior and to characterize the flow and mixing conditions in the new column. In the first year, we have completed the testing of the *local and overall gas holdups* in the three-stage column and conventional column (without contactors). In June, 1995, we initiated the *measurement of the bubble size* in the three-stage column and conventional column. Much of the current work has

focused on developing an image analysis method to determine the bubble sizes from the photographs.

Table 2.1
Technical Issues and Approaches for the Project

<u>Technical Issues</u>	<u>Technical Approaches</u>	<u>Expected Results</u>
1. Project Planning	Formulate a work plan Identify the two coal samples to be tested	Establish a work plan for the project and for each task
2. Equipment Design and Construction	Use expertise in coal cleaning and fluid flow to design and construct the proposed column	Provide the required multistage separation column for hydrodynamic and fine coal beneficiation tests
3. Hydrodynamic Testing a. Gas holdup b. Mixing time c. Bubble size d. Circulation velocity e. Interfacial area	Conduct experiments with and without coal particles in the system; varying flow rate, contactor geometry, and agitation intensity. A manometer, a high-speed videocorder, an optical fiber viewing device, and a conductivity meter are used.	Establish correlations of operating parameters in terms of dimensionless groups, such as Peclet, Reynolds, Froude, power numbers and geometric ratios or semi-empirical relations
4. Coal Beneficiation Tests: a. Heating Value Recovery b. Pyritic sulfur rejection c. Ash rejection	Conduct coal beneficiation tests in the proposed multistage column	Provide experimental verification of the potential application of the proposed multistage column for fine coal beneficiation
5. Conventional Column Tests	Perform flotation tests using a conventional column	Provide baseline data for comparison with results obtained in the proposed multistage column
6. Data Analysis	Use statistical methods for analyzing the data to demonstrate application of the proposed multistage column as a fine coal beneficiation device	Identify the design and operating parameters for optimal performance of the proposed multistage column

RESEARCH TEAM

Figure 2.1.1 Research Team

For engineering design and scale-up, an understanding of the hydrodynamic behavior of the multistage column is required. Gas holdup and specific interfacial area are two key parameters for evaluating the performance of the multistage column. For these purposes, gas holdup and bubble size data are to be determined. The following is a summary of the experimental results for these two parameters.

2.3.2 Gas Holdup

2.3.2.1 Principles

Gas hold-up is one of the key parameters for evaluating the performance of the multistage column. The overall gas hold-up (Φ) is defined as:

$$\Phi = \frac{V_G}{V_G + V_L} = \frac{V_{GL} - V_L}{V_{GL}} = \frac{H_{GL} - H_L}{H_{GL}} \quad (2.3.1)$$

where V_G is the volume of gas; V_{GL} is the volume of liquid-gas layer; V_L is the volume of liquid without gas bubble; H_{GL} is the height of liquid-gas layer under bubbling conditions; and H_L is the height of liquid level without gas bubbles. Thus, the overall gas holdup can be directly determined by volume expansion (or change in height) of the liquid phase.

The overall gas holdup can also be determined by measuring the hydrostatic pressure difference at two positions along the column. A U-tube manometer was used for this purpose^[1]. If the gas density is neglected, the relation between gas holdup and manometer reading can be expressed as:

$$\Phi = \frac{H_b - \rho_m H_m - H_w}{H_b} \quad (2.3.2)$$

where H_b denotes the liquid height in the column under bubbling condition; H_w is the liquid height in the connecting tube; H_m is the manometer reading; ρ_m is the density of the manometer liquid^[1-2].

2.3.2.2 Experimental Setup and Procedure

The experiments were performed in the three-stage agitated column and the conventional column. Both columns were operated under the same conditions. The flow diagram of the

multistage column is shown in Figure 2.3.1. The column consists of a plexiglass column of 0.1-m diameter and 1.83-m height. In the column, three vortex-inducing contactors are installed to create multiple loop flows. Within each contactor there are two impellers. The diameter ratio of contactor to column is 0.75. The multistage column can be divided into three working sections: foam discharge section, contacting section and gas stream injection section.

Water was used as the liquid phase and air as the gas phase. In each run, a predetermined amount of water was added to the column and its initial volume (or height) was recorded. Air was then introduced into the bottom of the column through three porous ceramic spargers (with mean pore size of 25 μm). The gas flow rate was controlled by a flowmeter. The superficial gas velocity (SGV) ranged from 0.5 to 8 cm/s. The agitation speed (N) was varied over a range up to 45 Hz. The frother used in this study was 4-methyl-2-pentanol (or methyl isobutyl carbinol, MIBC). MIBC was added to the tap water at various dosages up to 25 ppm. The frother was premixed with water in the column prior to start-up. All tests were carried out under ambient temperature and pressure. Table 2.2 lists the range of the key operating conditions employed in this study.

Table 2.2 Operating Conditions

Parameters	Conditions
Superficial gas velocity, 10^{-2} m/s	0.5 - 8.0
Agitation Speed, Hz	0.0 - 45.0
MIBC dosage, mg/L	0.0 - 25.0

Figure 2.3.1 Flow Diagram of the Multistage Column

2.3.2.3 Results and Discussion

A series of experiments on overall gas holdup of the multistage agitated column were conducted using both hydrostatic pressure difference method and volume expansion method. The comparative test results confirmed that the gas holdup data determined by the direct measurement of liquid levels (see Eq. 2.3.1) are in good agreement with those obtained from measuring the hydrostatic pressures (see Eq. 2.3.2). The relative deviation of the data obtained from the two methods are less than 10% (see Table 2.3). Chisti has reported similar results for evaluating these two methods^[3]. All subsequent gas holdup data presented in this report are obtained using the hydrostatic pressure difference method.

Table 2.3 Comparison of the Gas Holdup Data Obtained from Two Different Methods

Method	Overall gas holdup									
Volume expansion	0.08	0.085	0.093	0.110	0.130	0.285	0.296	0.318	0.364	
Pressure difference	0.08	0.085	0.092	0.109	0.126	0.261	0.271	0.293	0.340	

Note: SGV : 1.76 - 7.63 cm/s; Agitation speed : 0 - 45 Hz; MIBC dosage: 7 mg/L

Gas holdup measurements were first carried out for the whole column. The data thus obtained from the column show that a gas holdup as high as 45% can be achieved by adding a small amount of frother. The experimental results revealed that there are two distinct flow regimes in the multistage column as shown in Figures 2.3.2 and 2.3.3. A sharp break point occurs near the superficial gas velocity of about 0.06 m/s and at the agitation speed of 14.5 Hz. This break point corresponds to a visually observed change in the flow pattern from bubble flow to emulsion flow.

Figure 2.3.2 Effect of Superficial Gas Velocity on Gas Holdup

Figure 2.3.3 Effect of Agitation Speed on Gas Holdup

The effects of SGV, N and MIBC dosage on overall gas holdup were investigated. The experimental data for the roles of these operating parameters were plotted in a three dimension axes' system as shown in Figures 2.3.4 to 2.3.7.

Figures 2.3.4 to 2.3.7 show the effect of SGV on the gas holdup. The data indicate that when the SGV is less than 0.06 m/s, the gas holdup increases significantly with an increase in SGV. Once the SGV becomes greater than 0.06 m/s, the increase in gas holdup apparently slows down. Based on our observation, this corresponds to a visual change in the flow regime. In the first regime, gas stream with uniform large-size bubbles enters into the contacting region and the bubbles break up into small bubbles under agitation. As the SGV increases, loop-flow around the contactors was intensified. In this case, the bubbles become smaller and lead to increasing gas holdup. When the SGV increases beyond 0.06 m/s, there is a more rapid increase in the turbulence. Consequently, an emulsion flow pattern appears around each contactor. This corresponds to the second flow regime. In this regime, the gas holdup behavior is quite different from the first flow regime. On the one hand, the vortex turbulent motion caused by the loop flow in the multistage contactors does enhance the bubble break up and inhibit, to some extent, the effect of bubble coalescence; thus, relatively uniform fine bubbles fills the entire column volume which in turn leads to a high gas holdup. On the other hand, after gas bubble size is reduced to a limiting size range (i.e., the bubble breakup rate is equal to the bubble coalescence rate) the rate of increase in gas holdup declines.

The agitation speed is another important operating parameter affecting the gas holdup. As shown in Figures 2.3.4 through 2.3.7, a similar sharp break point is noted on the behavior of the

MIBC: 7 mg/L

Figure 2.3.4 Effects of SGV and Agitation Speed on Gas Holdup

Figure 2.3.5 Effect of SGV and Agitation Speed on Gas Holdup (MIBC: 15 mg/L)

Figure 2.3.6 Effects of SGV and Agitation Speed on Gas Holdup (MIBC: 20 mg/L)

Figure 2.3.7 Effects of SGV and Agitation Speed on Gas Holdup (MIBC: 25 mg/L)

gas holdup with increasing agitation speed at various SGV levels. This phenomenon also corresponds to the visually observed change in the flow regime discussed in the previous paragraphs. The initial gradual increase in gas holdup with increasing agitation speed quickly gives way to a more rapid rise of gas holdup when the agitation speed is greater than 14.5 Hz.

Since frother are expected to alter bubble coalescence and breakup behaviors, the effect of such agents on gas holdup was also studied. The effect of MIBC dosage on the gas holdup under varying SGV and agitation speed is seen in analyzing the three-dimension plots from Figures 2.3.4 through 2.3.7. The results indicate that the MIBC concentration does have a significant effect on the gas holdup at higher SGVs. These phenomena may be attributed to the fact that at higher SGVs, a greater surface activity would result in the presence of a frother. This in turn would reduce coalescence and lead to higher gas holdup. It is also noted from the figures that as the MIBC dosage increases beyond 15 mg/L, the rate of increase in gas holdup tends to decline. This is also consistent with the visually observed change in flow regime discussed above.

The local gas holdup in the contacting section (i.e., the column section occupied by contactors) and bubble injection section was also measured. The data indicate that a relatively high gas holdup (more than 40%) was observed in the contacting section while a gas holdup of less than 10 % is found in bubble injection section. This demonstrates that the contacting section in the multistage column plays a predominant role in the separation operation.

In addition, gas holdup measurements were conducted in a conventional agitated column for comparison with results obtained from the multistage column (Figure 2.3.8). The data show that under the same operating conditions the gas holdup in the multistage column (up to 45%) is significantly greater than those in the conventional agitated column (less than 30%). This finding

indicates that the multistage column equipped with vortex inducing contactors should give a superior performance as a fine coal cleaning device.

2.3.3 Bubble Size

In the design and scaleup of the multistage column, the interfacial area of gas bubbles is one of the critical parameters. The interfacial area of gas bubbles is related to the magnitude of the gas holdup and the mean gas bubble size, d_{av} . Mathematically, the interfacial area of gas bubbles, a , can be expressed as:

$$a = \frac{\Phi}{d_{av}} \quad (2.3.3)$$

In order to calculate the interfacial area, a series of bubble size measurements were performed.

Figure 2.3.8 Comparison of Gas Holdup Between Conventional Column and Multistage Column

2.3.3.1 Experimental Setup for Bubble Size Measurements

The bubble sizes in the multistage column were investigated using photographic method. The experimental setup consisted of a camera, a strobe light, and a video camera recorder. The key specifications for the camera and strobe light are listed in Table 2.3.

Table 2.3 Specifications for Camera^[4]

Item	Model
Camera	Solid State Camera, CID 2710
Lens	Olympus 100 x 52 1:2.8 macro lens, No. 22706714
Strobe	EG&G MVS - 2600

2.3.3.2 Experimental Method

In order to take photographs through the wall of the multistage column, elimination of any optical distortion due to the curvature of column wall should first be considered. In our case, because the micro lens of camera was focused on a field of view of only one (1) centimeter wide, there was no need to concern about the distortion caused by diffraction (see Figure 2.3.9.).

In the image analysis, the acquisition of clear image of bubble shape is crucial. For this purpose, a strobe light was applied as a light source. When the frequency of the flash synchronizes with the exposure frequency of the camera, the moving bubble recorded in the photograph will appear to be a “frozen” bubble. Thus, it results in a greatly enhanced quality of the bubble image.

Figure 2.3.9 Scale of the Field of View

To accomplish this, a proper selection of exposure time is a critical step. Table 2.4 lists the order of magnitude of exposure time for different cameras and light sources.

Table 2.4 Comparison of Camera Performance

	Regular Camera	High-Speed Camera	Regular Camera with Strobe
Frames per second	30	2000	30
Exposure time (μ s)	33333	500	1 - 5
Quality of Image	poor	fair	good

2.3.3.3 Experimental Design

A central composite design method was applied to investigating the effects of operating parameters on the bubble size and its distribution. Theoretically, the central composite design is a 2^k fractional design augmented by $2k$ axial points and a number of replicates at the central point to estimate the effects of operating variables on the object variable^[5]. Based on the previous experimental results, four variables, i.e., MIBC dosage, agitation speed (N), superficial gas velocity (SGV), and axial position (H) in the column, were selected as operating variables. Five levels of each variable were chosen and denoted as -2, -1, 0, 1, 2 in the design matrix, as shown in Table 2.5. Table 2.6 presents the experimental design matrix obtained from using the commercial statistical software NCSS.

Table 2.5 Experimental Design Matrix

Variables	Levels				
	-2	-1	0	1	2
MIBC, mg/L	0	7	15	20	25
N, Hz	0	14.7	21.7	30.0	45.0
SGV, 10 ⁻³ m/s	0.6	1.8	2.9	4.1	5.3
H	a*	b*	c*	d*	e*

Note: a* - gas bubble injection section; b* - inlet of the 3rd contactor;
c* - midway of the 3rd contactor; d* - outlet of the 3rd contactor;
e* - foam zone.

2.3.3.4 Image Analysis of Bubble Size

In order to save time in counting and sizing of a large number of gas bubbles, information concerning the size and shape of gas bubbles was obtained from the photographs with the aid of a computerized image analyzer. A premier software package^[6] for image analysis (**HARMONY**) was used to examine the bubble images. The procedure for the image analysis is consisted of the following steps:

1. Acquire and freeze a single frame by means of frame grabber;
2. Enhance and clean up the image by functions offered by **HARMONY** software;
3. Identify the features of the object of interest, i.e., the area of the bubbles;
4. Export the analysis results into Microsoft Excel spreadsheets in the computer;
5. Plot the bubble size distribution using the FREQUENCY function of Excel.

Table 2.6 NCSS Design Matrix

Run	MIBC	N	SGV	H
1	-2	0	0	0
2	2	0	0	0
3	-1	-1	-1	-1
4	0	2	0	0
5	1	1	-1	1
6	1	-1	1	1
7	-1	1	-1	1
8	-1	-1	1	1
9	0	0	0	0
10	0	0	0	2
11	1	1	1	1
12	0	0	-2	-1
13	-1	1	1	-1
14	-1	-1	-1	1
15	1	1	1	-1
16	0	0	0	-2
17	1	-1	-1	-1
18	0	0	0	0
19	-1	-1	1	-1
20	0	0	0	0
21	0	0	0	0
22	0	-2	0	0
23	1	1	-1	-1
24	0	0	0	0
25	0	0	0	0
27	-1	1	-1	-1
28	1	-1	1	-1
29	0	0	0	0
30	0	0	0	0
31	1	-1	-1	1
32	0	0	2	0
33	-1	1	1	1

Figures 2.3.10 through 2.3.14 show the original image acquired by frame grabber and the binary image after manipulation. In order to calculate the statistical average bubble sizes and plot their distribution, more than 100 bubbles were counted for each run using the image analyzer. Work for this task is in progress. Preliminary results indicate that considerably smaller bubble size can be obtained in the multistage column as opposed to the conventional column. Additional data on the calculated bubble sizes and interfacial areas will be submitted in the next technical progress report.

2.3.4 Summary

Experimental measurements of the overall gas holdup and gas bubble size were conducted to evaluate the roles of various operating parameters in the hydrodynamic behaviour of the multistage column. The results show that

1. The relationship of the gas holdup with the SGV or N can be used to define the two flow regimes in a multistage agitated column. The experimental results revealed that the break point on the curves (i.e., near the SGV of about 0.06 ms^{-1} and N beyond 14.5 Hz) corresponds to a visually observed change in the flow regime from bubble flow to emulsion flow.
2. In bubble flow regime, the SGV plays a more important role than the agitation speed in increasing the gas holdup. In contrast, the effect of the agitation speed on gas holdup is more significant than that of SGV in the emulsion flow regime.
3. The effect of frother dosage on gas holdup was profound. However, when frother dosage exceeds 15 mg/L , the increase in gas holdup levels off.

Frother : MIBC 0.0 ppm; $N = 21.7$ Hz;
 $SGV = 2.9 \times 10^{-2}$ m/s; H: midway of the 3rd contactor.

Figure 2.3.10 Photograph of Gas Bubbles
(a) Full Greyscale; (b) Binary Images.

(a)

(b)

Frother : MIBC 7 ppm $N = 14.7 \text{ Hz}$
 $SGV = 1.8 \times 10^{-2} \text{ m/s}$ H: inlet of the 3rd contactor.

Figure 2.3.11 Photograph of Gas Bubbles

(a) Full Greyscale; (b) Binary Images.

(a)

(b)

Frother : MIBC 7 ppm $N = 30.0 \text{ Hz}$
 $SGV = 4.1 \times 10^{-2} \text{ m/s}$ H: inlet of the 3rd contactor

Figure 2.3.12 Photograph of Gas Bubbles

(a) Full Greyscale; (b) Binary Images.

Frother : MIBC 7 ppm $N = 30.0 \text{ Hz}$
 $SGV = 1.8 \times 10^{-2} \text{ m/s}$ H: inlet of the 3rd contactor

Figure 2.3.13 Photograph of Gas Bubbles
(a) Full Greyscale; (b) Binary Images.

Frother : MIBC 7 ppm $N = 14.7 \text{ Hz}$
 $SGV = 4.1 \times 10^{-2} \text{ m/s}$ H: inlet of the 3rd contactor

Figure 2.3.14 Photograph of Gas Bubbles
 (a) Full Greyscale; (b) Binary Images.

4. The gas holdups in the multistage column are significantly greater than those in the conventional column.
5. The gas bubble sizes in the multistage column are much smaller than those in the conventional column.

3.0 WORK FORECAST

In the next year, the following tasks will be performed:

1. Continue to conduct the image analysis of the gas bubbles:
 - a. Compare the average bubble sizes between the multistage column operation and conventional column operation;
 - b. Calculate the specific interfacial areas using the gas holdup data and the average bubble size data.
2. Initiate investigation of mixing and liquid circulation behaviors :
 - a. Set up and calibrate a conductivity device;
 - b. Measure the mixing time;
 - c. Investigate the liquid circulation behavior around the contactors.
3. Perform fine coal beneficiation tests in the multistage column and conventional column.

The geometric dimension of and, the range of operating parameters to be used in the conventional column will be the same as those employed in the multistage column. Thus, a direct comparison between the results obtained from the two columns can be made.

4.0 REFERENCES

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