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

**QUARTERLY TECHNICAL PROGRESS REPORT
(April 1, 1996 - June 30, 1996)**

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1.0 INTRODUCTION

The overall objective of the 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 last quarter, we investigated the fine coal beneficiation behaviors in the multistage column and the conventional column^[1]. In this quarter, we have initiated the **wastewater treatment tests program** to verify the multifunction features of the multistage column. We also performed data analysis of the bubble sizes using a model based on the unified proportionality equation^[2-3]. Table 1.1, the project schedule, shows work accomplished to date.

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. Separation 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

This task was completed in September 1994^[4].

2.2 Task 2: Equipment Design and Construction

2.2.1 Objective

Currently, the objective is to modify an existing multistage column system in our lab for general use in wastewater treatment. This unit includes a multistage flotation column, a gas supply system and associated ancillaries.

2.2.2 Current Status of Task 2

During this reporting period, the design and construction have been completed for the wastewater treatment tests to demonstrate the multifunction features of the multistage column. Shake-down tests in a modified multistage column have been carried out. Both liquid and gas flowmeters, as well as an agitator speed controller have been re-calibrated. A Zeta-potential meter (ZM-80) and a particle size analyzer (Microtrac 7995) have been set up and tested. The other property measurement devices including a pH meter and a surface tension measuring device have also been tested. All these instruments have been calibrated with the standard samples provided by the manufactures.

2.3 Task 3: Hydrodynamic Tests

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 multistage separation column. This task was completed in December 1995^[5-7].

2.4 Task 4: Separation Tests

To demonstrate the multifunction features of the multistage column, the separation tests in Task 4 have focused on the investigation of wastewater treatment since April 1996. The goal of this phase of the study is to examine the potential application of the column to the general areas of fluid/particle separation and evaluate the effectiveness of the multistage column in these processes. The detailed description of the technical progress for this quarter is presented below.

2.4.1 Objective and Scope

The objective of this phase of our study is to explore the potential application of the multistage column for wastewater treatment. The experimental work focused on identifying the effect of operating parameters on the column performance used as wastewater treatment device and demonstrating its multifunctional features in the removal of contaminants (such as dispersed oil) from wastewater.

2.4.2 Experimental Setup and Procedure

The experiments is to be performed in a three-stage flotation column. The column configuration is depicted in Figure 2.4.1. It consists of a plexiglass column which is 0.1 m in diameter and 1.35 m in height. In the column, three contactors were installed to create multiple loop flow patterns. The diameter ratio of draft tube to column is 0.75. For comparison purposes, experiments will also be carried out in the conventional column (without contactor) in the next quarter.

1- Flowmeter;	2- Baffle;	3- Vortex-Inducing Contactor;
4- Impeller;	5- Shaft;	6- Gas Sparger;
7- Gas Tank.		

Figure 2.4.1 Multistage Flotation Column for Wastewater Treatment

A simulated wastewater sample is prepared by adding a pre-determined amount of a light mineral oil with a density of 874 kg/m^3 at 25°C . The oil/water mixture (with <1 ppm surfactant by volume) is emulsified in the feed tank by recirculating and mixing for about 30 minutes. Based on our previous experience, the oil-water emulsion prepared in this manner is very stable and no coalescence of oil droplets is expected. The initial oil concentration in water is 430 mg/L . Methyl isobutyl carbinol, (MIBC) and 2-ethyl-1-hexanol are used as frothers. The superficial gas velocity varies over a range up to 3.0 cm/s . Table 2.4.1 lists the operating conditions used in the study.

Table 2.4.1 Operating Conditions

Parameters	Conditions
Superficial gas velocity (V), 10^{-2} m/s	0.5 - 3.0
Agitation Speed (N), Hz	0.0 - 45.0
Reagent dosage (C), mg/L	0.0 - 20.0
Temperature, $^\circ\text{C}$	25

The oily water is fed to the top of column and a constant water level is maintained. Gas bubbles are generated by a vertical perforated metal sparger (mean pore size of $5 \mu\text{m}$), which is located at the bottom of the column. Gas bubbles pass through each contactor upward in contact with the down-flowing wastewater stream. The gas-lift action causes a circulation (or loop flow) of fluids around each contactor. The treated water leaves the bottom of the column via the clean water discharge line, while the oil-laden foam overflows into the foam discharge tank. Clean water samples are collected and analyzed for oil content using a chromatographic technique.

2.4.3 Oily Water Treatment Test Program

Tests will be performed in the multistage column to study the effects of *contactor number*, *frother concentration*, and *two different flotation gases* on the oil removal. The oil removal efficiency, η , is defined as

$$\eta = \frac{C_o - C_f}{C_o} \times 100\% \quad (2.4.1)$$

where C_o and C_f are the initial and final oil concentrations, respectively.

The experimental test program will be initiated next quarter.

2.5 Task 5: Conventional Column Tests

In the last quarter, we completed the investigation of the fine coal beneficiation behavior in the conventional column. The results were reported in the last quarterly report^[5]. In this quarter, no additional experiment was carried out in the conventional column.

2.6 Task 6: Data Analysis

In the past several quarters, data for the gas holdup, bubble size and specific interfacial area^[4] were analyzed in terms of three key operating parameters: V , N and C . Empirical correlations were established in terms of three second-order polynomial equations^[7] representing the experimental data.

In this quarter, an attempt was made to analyze the bubble size distribution in the multistage column using the unified proportionality equation^[2-3].

2.6.1 Basic Principle of the Model Equation

The unified proportionality equation is a multiscale, multidimensional equation. The equation not only embraces both linear and logarithmic scales, but also includes a multilogarithmic dimension. The equation has shown to be useful for modeling flotation kinetics data in the multistage column^[7]. In this study, we performed the modeling of bubble size distribution data using the same form of equation derived from the general unified proportionality equation. Model equation parameters were obtained using Microsoft Excel (version 5.0). Statistical parameters were used for identification of model applicability. Graphs for expressing the proportionality of the model are discussed below.

2.6.1.1 General Equation

The unified proportionality equation is given as:

$$d(q^m Y) = K d(q^n X) \quad (2.6.1)$$

where q is a notation of logarithm, K is a proportionality constant, and m and n are logarithmic dimensions of Y and X . In this study, Y represents the cumulative frequency density (%) and X the bubble size (mm). The model equation needs to incorporate the cumulative frequency density asymptote (Y_i) as reference point to account for the effective cumulative frequency density (Y), as will be discussed in the following section.

2.6.1.2 Model for Cumulative Frequency Density of Bubble Sizes- phenomena with ($Y_i > Y$)

In flotation, one is interested in finding what the bubble size distribution profile looks like in the multistage column. Because the profile of cumulative frequency density (cfd) of bubble sizes tends to

approach an asymptote, it is imperative that the asymptote be identified first and be used as a key reference point in analyzing the effective frequency density of bubble sizes. Similar to the flotation kinetics, the bubble size distribution is a $2q$ by $1q$ phenomenon, which can be described by the equation:

$$\frac{d(qY_i - qY)}{qY_i - qY} = K \frac{dX}{X} \quad (2.6.2)$$

The equation state that the fractional change of effective Y in logarithmic dimension is proportional to the fractional change of X.

The integrated form of the bubble size distribution equation can then be expressed as:

$$\log \frac{1}{(qY_i - qY)} = K \log X + \log A \quad (2.6.3)$$

In the above equation, the term $1/(qY_i - qY)$ is known as the *proportionality factor*. A is an integration constant. According to Equation (2.6.3), $1/(qY_i - qY)$ versus X can be plotted in a log-log graph to obtain a straight line if the proportionality exists. Microsoft Excel software was used to determine the asymptote Y_i and parameters A and K in the above model equation.

2.6.2 Data Analysis Procedure

Microsoft Excel was used in data analysis and graphing. Using an object oriented interactive approach, the asymptote Y_i was first sought. For each model, the K, A, and unique Y_i were determined simultaneously for Equation (2.6.3) using the trendline (or regression) analysis. The objective was to identify the unique asymptote Y_i for the best fit of the trendline or the maximum of the *coefficient of determination* (R^2). The value of R^2 can be defined as follows:

$$R^2 = 1 - \frac{SSE}{SSTO} = 1 - \frac{\sum (Y_i - \hat{Y}_i)^2}{\sum (Y_i - \bar{Y}_i)^2} \quad (2.6.4)$$

where, SSE is the error sum of squares, which is used to measure the variation in the observations Y_i when a regression model utilizing the independent variable X is employed; $SSTO$ is a measure of the uncertainty in predicting Y when the independent variable X is not considered.

The bubble size data obtained from the multistage column and the calculated proportionality factors are listed in Tables 2.6.1 through 2.6.4. Based on a regression analysis, the model equation parameters were determined and presented in Table 2.6.5. Using the model equation parameters in Table 2.6.5, the values of the theoretical Y were calculated using the model equation as listed in Tables 2.6.1 through 2.6.4.

Table 2.6.1 Bubble Size Distribution in the Multistage Column (Run 12)

				Y_i
X	Fd	$Y(\text{experimental})$	$Y(\text{calculated})$	99.999
$d_p, \text{ mm}$	%	<i>Cum. Fd, %</i>	<i>Cum. Fd, %</i>	$1/(qY_i - qY)$
0.25	0	0		
0.50	11.4	11.4	8.233	1.060
0.75	45.6	57.0	52.88	4.096
1.00	20.1	77.1	78.53	8.854
1.25	10.5	87.6	89.23	17.393
1.50	4.39	91.99	94.02	27.582
1.75	2.63	94.62	96.396	41.645
2.00	3.51	98.13	97.68	122.04
2.25	0.877	99.01	98.44	230.96

Note: Fd = Frequency density, %; $Cum. Fd$ = Cumulative frequency density, %.

Table 2.6.2 Bubble Size Distribution in the Multistage Column (Run 28)

				Y_i
X	Fd	Y(experimental)	Y(calculated)	99.999
d_p , mm	%	Cum. Fd, %	Cum. Fd, %	$1/(qY_i - qY)$
0.25	2.86	2.86	0.143	
0.50	42.86	45.71	43.41	1.060
0.75	35.24	80.95	77.88	4.096
1.00	8.57	89.52	89.92	8.854
1.25	2.86	92.38	94.67	17.393
1.50	3.81	96.19	96.87	27.582
1.75	1.91	98.09	98.01	41.645
2.00	0.95	99.04	98.65	122.04
2.25	0	99.04	99.05	230.96

Note: Fd = Frequency density, %; Cum. Fd = Cumulative frequency density, %.

Table 2.6.3 Bubble Size Distribution in the Multistage Column (Run 30)

				Y_i
X	Fd	Y(experimental)	Y(calculated)	99.999
d_p , mm	%	Cum. Fd, %	Cum. Fd, %	$1/(qY_i - qY)$
0.00	0.00	0.00		
0.50	48.54	48.54	51.46	3.429
0.75	27.18	75.73	72.15	10.155
1.00	5.83	81.55	81.15	15.085
1.25	4.85	86.41	85.81	24.284
1.50	0.97	87.38	88.48	27.527
1.75	2.91	90.29	90.13	45.280
2.00	0.97	91.26	91.23	57.339
2.25	0.97	92.23	92.00	77.852
2.50	0.00	92.23	92.55	77.852

Note: Fd = Frequency density, %; Cum. Fd = Cumulative frequency density, %.

Table 2.6.4 Bubble Size Distribution in the Multistage Column (Run 33)

				Y_i
X	Fd	Y(experimental)	Y(calculated)	99.999
d_p , mm	%	Cum. Fd, %	Cum. Fd, %	$1/(qY_i - qY)$
0.00	0.00	0.00		
0.50	53.33	53.33	52.06	3.753
0.75	26.67	80.00	76.43	11.069
1.00	6.667	86.67	86.33	17.991
1.25	4.444	91.11	90.98	29.529
1.50	1.481	92.59	93.47	37.226
1.75	2.963	95.56	94.93	75.856
2.00	0.00	95.56	95.85	75.856
2.25	0.741	96.30	96.47	101.75
2.50	0.00	96.30	96.90	101.75

Note: Fd = Frequency density, %; Cum. Fd = Cumulative frequency density, %.

Table 2.6.5 Parameter Values

Run Parameters	12	28	30	33
Y_i	99.999	99.999	95.00	98.5
R^2	0.9734	0.9803	0.9943	0.9934
K	3.369	2.973	1.961	2.274
A	9.526	21.667	14.618	17.461

2.6.3 Discussion and Remark

Figure 2.6.1.a through 2.6.1.d are the plots of cumulative cfd vs. X for Run 12, 28, 30 and 33. These plots show a series of sigmoid curves. When the scale of cumulative cfd is changed from linear to logarithmic, a series of hyperbolic curves with continuous changes of slope are obtained. This is a prerequisite curve for interpretation of Equation (2.6.2).

Figure 2.6.1.a Plot of Cumulative cfd vs. Bubble Size (Run 12)

Figure 2.6.1.b Plot of Cumulative cfd vs. Bubble Size (Run 28)

Figure 2.6.1.c Plot of Cumulative cfd vs. Bubble Size (Run 30)

Figure 2.6.1.d Plot of Cumulative cfd vs. Bubble Size (Run 33)

Figures 2.6.2 through 2.6.5 depict the proportionality plots for the data obtained from Run 12, 28, 30 and 33, where the proportionality factor $1/(qY_i - qY)$ is plotted against bubble sizes in a log-log graph. In these figures, all trendlines are drawn to give unique Y_i using the Excel software. Figures 2.6.6 through 2.6.9 show the comparison of experimental data with calculated values using Equation 2.6.3. The results reveal that the data points are matched by Equation 2.6.3.

Figure 2.6.2 Proportionality Plot of Model 3 (Run 12)

Figure 2.6.3 Proportionality Plot of Model 3 (Run 28)

Figure 2.6.4 Proportionality Plot of Model 3 (Run 30)

Figure 2.6.5 Proportionality Plot of Model 3 (Run 33)

In Table 2.6.5, the R^2 values range from 0.97 to 0.99. These R^2 values are high, indicating that the Equation (2.6.3) can be considered as a feasible candidate equation for describing the bubble size distribution.

From the above discussion, it can be concluded that the relationship between the cumulative frequency density and the bubble sizes for the multistage column is not linear. Instead, their relationship is changing in a logarithmic scale and the relative change is varying in a fractional change rather than simple linear change. For this reason, the bubble size distribution can best be described by a high dimensional subset of the unified proportionality equation, which may be written as:

$$\frac{1}{\log Y_i - \log Y} = A \bullet X^\kappa \quad (2.6.5)$$

where, all the parameters in this equation have been defined in the previous section. The applicability of this equation for describing the relation between cumulative frequency density and bubble size can also be confirmed from Figures 2.6.6 through 2.6.9.

2.7 Summary

The accomplishments for this quarter may be summarized as follows:

1. The experimental investigation on the multifunctional features of the multistage column has been initialed. The modified equipment design and construction have been completed. The preparation work for the wastewater treatment tests are underway.
2. The unified proportionality Equation (2.6.5) has been shown to be applicable for describing the bubble size distribution profile in the multistage column.

Figure 2.6.6 Comparison of calculated values with experimental data (Run 12)

Figure 2.6.7 Comparison of calculated values with experimental data (Run 28)

Figure 2.6.8 Comparison of calculated values with experimental data (Run 30)

Figure 2.6.9 Comparison of calculated values with experimental data (Run 33)

3.0 WORK FORECAST

In the next quarter, separation tests will be continued as the part of work under Task 4 to study the application of the multistage column to the other separation processes and to demonstrate its multifunction capability. Work will include the following:

- (1) Conduct wastewater treatment
- (2) Continue to prepare a data basis for engineering design and economic analysis of the multistage flotation system.

4.0 REFERENCES

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