ABSTRACT

Recent studies indicate that the optimum separation performances achieved by multiple stage cleaning using various column flotation technologies and single stage cleaning using a Packed-Flotation Column are superior to the performance achieved by the traditional release procedure, especially in terms of pyritic sulfur rejection. This superior performance is believed to be the result of the advanced flotation mechanisms provided by column flotation technologies. Thus, the objective of this study was to develop a suitable process utilizing the advanced froth flotation mechanisms to characterize the true flotation response of a coal sample.

This investigation resulted in the development of a modified coal flotation characterization procedure, termed as the Advanced Flotation Washability (AFW) technique. The apparatus used for this procedure is a batch operated Packed-Column device which provides enhanced selectivity due to a plug-flow environment and a deep froth zone. The separation performance achieved by the AFW procedure was found to be superior to those produced by the conventional tree and release procedures for three nominally -100 mesh coal samples and two micronized samples. The largest difference in separation performance was obtained on the basis of product pyritic sulfur content. A comparison conducted between the AFW and the release procedures at an 80% recovery value showed that the AFW technique provided a 19% improvement in the reduction of pyritic sulfur. For an Illinois No. 5 coal sample, this improvement corresponded to a reduction in pyritic sulfur content from 1.38% to 0.70% or a total rejection of 66%. Micronization of the sample improved the pyritic sulfur rejection to 85% while rejecting 92% of the ash-bearing material. In addition, the separation performance provided by the AFW procedure was superior to that obtained from multiple cleaning stages using a continuous Packed-Column under both kinetic and carrying-capacity limiting conditions.
EXECUTIVE SUMMARY

The goals of this project were to evaluate the current status of the coal flotation characterization procedures, such as release and tree analyses with respect to the advanced froth flotation technologies presently being introduced and to modify the traditional procedures so that a true theoretical optimum recovery-grade curve for any froth flotation process can be obtained from the analysis.

The traditional release and tree analysis procedures are recognized internationally as the analyses which provide the ultimate recovery-grade relationship that can be achieved by any flotation process for the treatment of a given coal. An analogous to release analysis is the washability analysis for gravity-based separations. Dell introduced the concepts of release analyses in 1953 and refined the procedure in 1964 and 1972. To date, release analysis, which is conducted using a Denver flotation device, has been successfully used as a tool by preparation plant operators and researchers for evaluating the efficiency of new flotation technologies and for optimizing current flotation systems. However, Dell et al. (1972) recognized the fact that potentially better performances could be achieved by other flotation devices. “The (release) approach towards this absolute measurement is, however, a function of cell design, and it is yet impossible to say whether results even better than those with the Denver unit are possible.”

In agreement with Dell’s statement, the introduction of advanced flotation technologies has resulted in separation performances superior to that predicted by the traditional release analysis procedure. This fact was found to be especially true on the basis of pyritic sulfur rejection with single stage cleaning using the Packed-Column and multiple stage cleaning using other flotation column technologies. It is believed that the superior performance is due to an improvement in the hydrodynamic conditions in the flotation cell and to the utilization of selectivity mechanisms in the froth zone. Due to the inherent constraints associated with the Denver cell which prevent the use of deep froth depths, the phenomena, such as reflux, selective detachment and froth washing of entrained materials, are not easily achieved in the traditional release analysis process. It is believed that these deficiencies have resulted in several steady-state column flotation results being superior to the corresponding release data.

A theoretical simulation of the release analysis procedure conducted by the principal investigators supports the above statements that release analysis should be conducted with a flotation device providing a plug-flow hydrodynamic environment and a deep froth depth. Plug-flow conditions were found to provide a higher recovery of particles to the froth zone when compared to perfectly-mixed conditions, which is characteristic of the Denver conventional cell used in the release analysis procedure. It was also found that the selectivity between particles of varying hydrophobicity is best achieved in the froth phase where the selective detachment mechanism can be utilized. Deep froth zones provide more reflux and a separation performance approaching the optimum separation performance. Unfortunately, conventional cells do not support deep froth zones and, therefore, have limited ability to provide sufficient reflux. Since release analysis is based
on selectivity, a flotation column apparatus was the desired separation device to replace the Denver cell for obtaining the optimum separation performance. Thus, the project objectives were: 1) to develop a new release analysis apparatus which would provide plug-flow conditions while allowing a significant froth reflux action; 2) to compare the optimum separation performance predicted by the modified release analysis procedure for the treatment of fine coal sample with that obtained by the traditional release and tree analyses, and washability analysis; 3) to compare the separation performances obtained for several coal samples using the proposed release procedure with the optimum recovery-grade curves obtained from the single-stage treatment provided by, Packed-Column flotation technology, which was found to produce the best separation performance among the six different flotation column technologies studied in a recently completed ICCI project. Achieving these objectives would result in a modified release analysis procedure which utilizes the advanced flotation mechanisms common to the modern flotation technologies and strategies being used today by coal preparation plant operators and researchers. Thus, a true optimum theoretical separation performance for any froth flotation process could be obtained for fine coal characterization.

During this investigation, a modified coal flotation characterization procedure, termed as Advanced Flotation Washability (AFW) technique was developed. The apparatus used for this procedure was essentially a batch operated 2-inch diameter, 5 ft tall Packed-Column, in which the feed slurry was continuously recirculated to avoid deposition of solid particles in the cell and to provide a feed flow counter-current to that of the air bubbles to effect superior bubble-particle collision. The cell was equipped with a PID controlled wash water system, which was mainly used to mobilize the deep froth in the cell and to adjust the pulp level to operate the cell at a desired froth depth to facilitate column reflux action.

The general approach used in the traditional release analysis for removing the hydrophilic mineral particles in the first stage and collecting the concentrate samples having varying degrees of hydrophobicity in the second stage was pursued in the AFW procedure to obtain an ultimate combustible recovery-grade curve representing the true flotation response of a coal sample. Several experiments were conducted using the AFW device on a nominally -100 mesh Illinois No. 5 coal to establish the appropriate operating parameter values and the step-by-step procedure for the AFW technique. Upon solidifying the experimental procedure, two additional -100 mesh and two micronized coal samples were treated by the AFW device and the separation performances were compared to those obtained from the conventional release and tree procedures. In addition, each nominally -100 mesh coal was used to conduct continuous Packed-Column tests and compare the performance with that obtained from the AFW and release procedures.

The performance curve generated using the AFW technique was found to be superior to those obtained using the traditional tree and release procedures on all the coal samples tested in this study. While treating an Illinois No. 5 coal having an ash, total sulfur and pyritic sulfur contents of about 20%, 2.60% and 1.38%, respectively, the AFW procedure
produced a product of 4.80% ash, 1.55% total sulfur and 0.70% pyritic sulfur at a combustible recovery value of 80%. In comparison, the optimum performance results obtained from the traditional procedures at the same combustible recovery are 5.6% ash, 1.86% total sulfur and 0.90% pyritic sulfur. After micronizing the coal, the pyritic sulfur content was further reduced to 0.32% at the same combustible recovery value which corresponds to a pyritic sulfur rejection of 85%. The ash content for the micronized sample was significantly reduced to 2.5% at 80% recovery which equates to an ash rejection of 92%. These separation performances achieved from the AFW procedure were found to be much superior to those obtained from the conventional tree and release procedures. Similar separation performance differences were also observed while treating an Illinois No. 6 coal, a West Kentucky No.9 coal and a micronized Illinois No. 6 coal.

The maximum selectivity achieved from a continuously operated Packed-Column was obtained under carrying-capacity limiting conditions, which was achieved by running the column at a high feed solids content. Under these conditions, the single stage cleaning of a -100 mesh Illinois No. 5 coal sample was found to provide nearly equal performance on the basis of product ash content as compared to that of the AFW procedure. On the other hand, test results from the Packed-Column when operated under kinetic limiting conditions indicate that approximately three cleaning stages are needed to achieve a separation performance nearly equal to the AFW performance. The continuous Packed-Column tests conducted on the Illinois No. 6 coal were inferior to both release and AFW procedure results which may be due to insufficient reflux caused by a low solids loading in the continuous Packed-Column tests. On the other hand, the Packed-Column tests conducted on the West Kentucky No. 9 sample produced separation performances that were superior to the results obtained from the traditional release procedure but inferior to the AFW performance curve.

The results obtained from the traditional flotation characterization procedures conducted on the Illinois No. 5 coal indicated that the tree procedure consistently produced inferior performance in the high recovery region of the combustible recovery-grade curve. Since a substantial portion of the combustibles in the tree procedure is recovered in the scavenger flotation steps, the heavy middling type particles tend to report to the concentrate, which considerably increases the ash content of the concentrate. Coal pyrite particles, being weakly hydrophobic also report to the concentrate during the scavenger flotation steps which enhances the sulfur content of the concentrates. Thus, the superior performance obtained from the successive cleaning stages conducted on the individual flotation concentrate samples produced during the tree procedure is heavily offset by the flotation of heavy middling and coal pyrite particles in the scavenger flotation steps.

The gravity-based washability results were found to be superior to the separation performance obtained from each of the flotation characterization techniques conducted on a -100 mesh Illinois No. 5 coal sample. However, the difference in separation performance achieved by the gravity-based washability and the AFW procedure was reduced as the coal sample was ground to a finer particle size.
OBJECTIVES

The goals of this project were to evaluate the current status of the release analysis procedure with respect to the advanced froth flotation technologies presently being introduced and to modify the traditional release analysis procedure so that a true theoretical optimum recovery-grade curve for any froth flotation process can be obtained from the analysis. In light of these goals, the project objectives were:

1. To develop a new release analysis apparatus which will provide plug-flow conditions while allowing a significant reflux action. The reflux action is critical for obtaining maximum separation performance.

2. To compare the optimum separation performance predicted by the proposed release analysis procedure for the treatment of a fine coal sample (-100 mesh) with that obtained by the traditional release and tree analyses, and washability analysis.

3. To compare the separation performances obtained for several coal samples using the proposed release procedure with the optimum recovery-grade curves obtained from the single-stage treatment provided by an advanced flotation system, i.e., Packed-Column.

These objectives were achieved in the investigation which resulted in the successful development of a novel test apparatus for evaluating the optimum flotation response for fine coal samples. It is believed that the separation performance obtained from the novel apparatus more truly represents the theoretically “best” performance and, thus, represents an important new tool for coal researchers and coal preparation plant operators when evaluating modern coal flotation technologies and processing strategies.

INTRODUCTION AND BACKGROUND

The release analyses procedure has long been used to predict the theoretically best separation performance that can be achieved by a flotation process for the treatment of fine coal. The release analysis for flotation is analogous to the washability analysis for gravity separation. The release procedure was originally introduced by Dell in 1953 as a new method for characterizing coal. The procedure, which utilizes laboratory conventional flotation cells, was revised by Dell in 1964 and by Dell et al. in 1972. The separation performance results obtained by release analysis is commonly used by preparation plant operators and researchers to evaluate new technologies and to optimize current flotation processes. However, the introduction of column flotation technology has created some controversy with the release analysis process which can be attributed to better hydrodynamic conditions and the advanced flotation mechanisms utilized by flotation columns.
The release analysis procedure is a two phase process. In the first phase, hydrophobic particles are separated away from hydrophilic particles by repetitive flotation of the concentrate to remove the entrained material. The key to the success of the first phase is to ensure complete flotation of the hydrophobic material while removing all of the entrained material. This must be accomplished while minimizing the amount of frother and collector additions. Excessive chemical additions result in a reduction in the selectivity of the second phase, which was confirmed in a study reported by Pratten et al. (1989). In this study, the release procedure was compared to another procedure known as the tree analysis. This procedure is similar to release analysis in that it uses conventional flotation cells to treat and retreat tailings. The apparent advantage of the tree procedure is its insensitivity to collector dosages at the low product ash region.

The second phase of the release procedure involves the separation of the hydrophobic particles comprising the first phase concentrate into fractions varying in their degrees of hydrophobicity. The ash content in coal particles typically increases with a decrease in surface hydrophobicity. Thus, a recovery-grade curve can be developed based on floatability. The first cleaning stage of Phase II in the release procedure is performed under starvation conditions (i.e., low aeration rate, low impeller speed and low chemical additions) to float the most hydrophobic particles. The conditions are then improved by increasing the amount of air which ideally floats the particles having the next highest degree of floatability. This process is continued until all of the coal has been floated and a total of 5 clean coal products and 1 tailings sample has been produced. A flowsheet of the release analysis process and the results obtained from a typical analysis are shown in Figure 1.

This procedure has been found to be very successful for comparisons with in-plant conventional cell plant. However, there are numerous reports of column flotation data out performing the release analysis, which is considered as being theoretically impossible. This phenomena may be due to the high mixing conditions of the conventional cell which is used to conduct the release analysis. This is compared to the near-plug flow conditions that are achieved in laboratory flotation columns. The Packed-Column, which is completely filled with corrugated plates spaced at 1/4-inch apart, is one column that consistently out performs the release analysis in the low ash content-low recovery region. Figure 2 shows a comparison of the results obtained by the principal investigators from the treatment of two different coals by the release analysis procedure and the Packed-Column. As shown, the Packed-Column produced cumulative product ash contents that were 1% to 2% lower than those achieved by release analysis in the low recovery region. Results showing a superior performance by the Packed-Column were also reported in a
Figure 1. Step-by-step analysis of results obtained from a traditional release analysis conducted on a -65 mesh Illinois No. 6 coal sample.

Figure 2. Flotation results showing the superior performance of the Packed-Column over that achieved by the traditional release analysis in the low recovery-low ash content region. Fine coal samples (-100 mesh) treated were from the (a) Paradise Preparation Plant and (b) Illinois No. 5 coal seam.
EXPERIMENTAL PROCEDURES

Sample

Three fifty-five gallon drums of dry Illinois No. 5 run-of-mine coal sample were collected from a local preparation plant for this investigation. The samples were crushed using a laboratory Jaw cruscher and a hammermill to obtain a -100 mesh product which was split into representative lots of about 5 lbs each and placed into storage bags. The sample bags were stored at -20° C to minimize surface oxidation of the coal particles. The coal sample was found to have ash, total sulfur and pyritic sulfur contents of 19.9%, 2.75% and 1.53%, respectively. Size-by-size sample characterization data are provided in Table 1.

Table 1. Size-by-size analysis data of the -100 mesh run-of mine Illinois No. 5 coal seam sample used in the present investigation.

<table>
<thead>
<tr>
<th>Size Fraction (mesh)</th>
<th>Weight (%)</th>
<th>Ash (%)</th>
<th>Total Sulfur (%)</th>
<th>Pyritic Sulfur (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>+200</td>
<td>35.8</td>
<td>14.8</td>
<td>2.61</td>
<td>1.17</td>
</tr>
<tr>
<td>200 x 325</td>
<td>24.8</td>
<td>15.6</td>
<td>2.68</td>
<td>1.25</td>
</tr>
<tr>
<td>325 x 400</td>
<td>3.0</td>
<td>16.1</td>
<td>3.03</td>
<td>1.60</td>
</tr>
<tr>
<td>-400</td>
<td>36.3</td>
<td>28.3</td>
<td>2.90</td>
<td>1.68</td>
</tr>
<tr>
<td>Total</td>
<td>100.0</td>
<td>19.9</td>
<td>2.75</td>
<td>1.38</td>
</tr>
</tbody>
</table>

A portion of the Illinois No. 5 coal sample was micronized to investigate the liberation effect on the proposed flotation procedure. The hammer mill product having a size distribution as described in Table 1 was ground for 20 minutes in a Turbo-Mill containing 1/8-inch diameter zircon balls as the grinding media. The size characteristic data of the micronized sample are shown in Table 2.

Table 2. Size distribution data for the micronized coal samples in this study.

<table>
<thead>
<tr>
<th>Cumulative % Finer</th>
<th>Particle Size (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>75</td>
<td>42</td>
</tr>
<tr>
<td>50</td>
<td>20</td>
</tr>
<tr>
<td>25</td>
<td>6</td>
</tr>
</tbody>
</table>

In addition to the Illinois No. 5 seam coal sample, two additional coal samples were used in this study. A sample lot of approximately 200 pounds of IBC-105 (Illinois No. 6) coal, which is known to be difficult to clean, was obtained from the Illinois Coal Sample Bank. A West Kentucky No. 9 coal slurry sample was collected from the fine refuse stream of an operating coal preparation plant near Henderson, Kentucky. The size-by-size data for both samples are provided in Table 3.
Table 3. Size-by-size analysis data of the IBC-105 (Illinois No. 6) and West Kentucky No. 9 coal samples used in the present investigation.

<table>
<thead>
<tr>
<th>Size Fraction (mesh)</th>
<th>Weight (%)</th>
<th>Ash (%)</th>
<th>Total Sulfur (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>IBC-105 Coal</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>+100</td>
<td>17.7</td>
<td>10.9</td>
<td>3.17</td>
</tr>
<tr>
<td>100 x 200</td>
<td>29.4</td>
<td>11.0</td>
<td>3.53</td>
</tr>
<tr>
<td>200 x 325</td>
<td>16.4</td>
<td>11.8</td>
<td>3.92</td>
</tr>
<tr>
<td>325 x 400</td>
<td>4.3</td>
<td>12.0</td>
<td>3.87</td>
</tr>
<tr>
<td>-400</td>
<td>32.2</td>
<td>26.2</td>
<td>4.07</td>
</tr>
<tr>
<td>Total</td>
<td>100.0</td>
<td>16.0</td>
<td>3.72</td>
</tr>
<tr>
<td>W. Kentucky No. 9</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>+65</td>
<td>1.9</td>
<td>6.5</td>
<td>2.13</td>
</tr>
<tr>
<td>65 x 100</td>
<td>1.9</td>
<td>5.8</td>
<td>2.23</td>
</tr>
<tr>
<td>100 x 200</td>
<td>13.5</td>
<td>6.4</td>
<td>2.30</td>
</tr>
<tr>
<td>200 x 400</td>
<td>14.6</td>
<td>11.3</td>
<td>2.85</td>
</tr>
<tr>
<td>-400</td>
<td>68.1</td>
<td>45.9</td>
<td>1.98</td>
</tr>
<tr>
<td>Total</td>
<td>100.0</td>
<td>34.0</td>
<td>2.16</td>
</tr>
</tbody>
</table>

AFW Apparatus

A modified coal flotation characterization procedure, termed as Advanced Flotation Washability (AFW) technique, was developed which replaces the conventional flotation cell with a flotation column when measuring the true flotation response of a coal sample. The new apparatus, as shown in Figure 3, consists essentially of a batch-operated, 2-inch diameter, 5 ft tall Packed-column.

Prior to the tests, coal slurry containing approximately 20% solids by weight was added directly to the column. The pulp level was adjusted to 2 ft from the cell volume which provided a froth depth of about 3 ft. The slurry was conditioned with the desired amount of collector (i.e., kerosene) for a two minute period prior to adding the slurry to the flotation column. The frother was added as needed directly into the cell.

As shown in Figure 3, the feed slurry was continuously recirculated during the experiments to avoid deposition of solid particles in the cell and to provide a feed flow counter-current to that of the air bubbles to effect superior bubble-particle collision. The air was injected through a flow meter directly into the cell. The cell was equipped with a proportional, integral, derivative (PID) controlled wash water system which was mainly used to mobilize (or lubricate) the deep froth in the cell and to conveniently adjust the pulp level to operate the cell at a desired froth depth. For example, during the flotation test, froth concentrate continuously reports to the overflow launder which causes a reduction in the pulp level. This reduction in the cell pulp level is received by a pressure transducer placed in the lower section of the column, which activates a PID controller to
send a constant analog signal to a peristaltic pump. The wash water flow rate entering the top of the froth zone is adjusted by the PID controller to maintain the pulp level at the desired preset value. This system has several improvements over the traditional conventional cell:

1. The batch Packed-column provides support for deep froth depths and a plug-flow environment which, theoretically, improves separation performance.

2. The froth concentrate is removed without the assistance of the operator. The traditional conventional cell required assistance by the operator which increased the possibility of experimental error and hindered test repeatability.

3. The use of a controller allows the system to be operator independent while providing wash water to the froth zone for mobilization and cleaning purposes.

Figure 3. A schematic of the experimental apparatus used to conduct the AFW procedure.
AFW Procedure

The traditional release analysis approach of attempting to remove all the hydrophilic mineral particles in the first stage and collect concentrate samples having varying degrees of hydrophobicity in the second stage was also pursued in the AFW procedure. The first stage of the procedure consisted of two to three flotation steps as shown in Figure 4, which was conducted with the addition of the desired amount chemical reagents to separate the hydrophilic mineral particles from the hydrophobic coal particles. The tailings obtained from each step of the first stage were combined to form the final tailings. The froth concentrate collected from the first stage, which is considered to be comprised of only hydrophobic particles, is segregated into about six different fractions according to a decreasing order of hydrophobicity by varying the aeration rate from 1.5 liters/min to nearly 5 liters/min and by maintaining a deep froth of about 3 ft to facilitate a superior column reflux action.

Initially, several tests were conducted to determine the optimum amount of air required in each flotation step of the first stage to ensure proper flow of the froth concentrate and to simultaneously minimize the pulp water recovery to the product launder. The chemical reagent dosages were also varied to arrive at an optimum level to ensure a complete flotation of all the hydrophobic particles in the first stage. The step-by-step configuration

![Diagram](image)

Figure 4. A schematic illustration of the step-by-step AFW procedure.
and the operating parameter values used in the first step of the important tests are listed in Table 4. As shown, the first four experiments were conducted using the configuration in Figure 4. For experiment 5 and 7, the flotation steps in the first stage was conducted using the Denver cell, whereas experiment 6 started by segregating the feed into three initial fractions using the AFW device and then separately floating the three fractions using variable amount of air to obtain a series of concentrate samples of varying degrees of hydrophobicity (i.e., similar to Tree Analysis).

Table 4. A summarized list operating parameter values used in the first stage of several important experiments conducted using the AFW procedure.

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Configuration in Terms of the 1st Stage</th>
<th>Collector (lb/ton)</th>
<th>Frother (lb/ton)</th>
<th>Air (lpm)</th>
<th>Froth Height (ft)</th>
<th>Wash Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3 cleaners</td>
<td>1.0</td>
<td>0.88</td>
<td>4</td>
<td>1.5</td>
<td>No</td>
</tr>
<tr>
<td>2</td>
<td>3 cleaners</td>
<td>1.39</td>
<td>0.88</td>
<td>2</td>
<td>2.0</td>
<td>Yes</td>
</tr>
<tr>
<td>3</td>
<td>4 cleaners</td>
<td>1.70</td>
<td>0.66</td>
<td>2</td>
<td>2.5</td>
<td>Yes</td>
</tr>
<tr>
<td>4</td>
<td>4 cleaners</td>
<td>1.70</td>
<td>0.66</td>
<td>2</td>
<td>2.5</td>
<td>No</td>
</tr>
<tr>
<td>5</td>
<td>Denver cell</td>
<td>2.0</td>
<td>0.88</td>
<td>-</td>
<td>-</td>
<td>No</td>
</tr>
<tr>
<td>6</td>
<td>Fractionation</td>
<td>0.55</td>
<td>0.66</td>
<td>2</td>
<td>2.5</td>
<td>Yes</td>
</tr>
<tr>
<td>7</td>
<td>Denver cell</td>
<td>2.58</td>
<td>0.88</td>
<td>-</td>
<td>-</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Conventional Methods

Similar coal characterization experiments were also conducted using the traditional release and tree analysis procedures. The procedures for both methods were conducted using a 4-liter laboratory Denver flotation cell. The modified release procedures described by Dell (1972) and Forrest et al. (1994) were followed in detail while the tree analysis procedure was performed according to Nicols (1983). A detailed study based on a statistical design was conducted on the Release and Tree procedures to identify the conditions providing the optimum separation performance. The optimum curves generated from both methods were used for comparison purposes.

In addition, fine coal washability data was also obtained for comparison purposes using the ASTM centrifugal procedure. Commercial Testing and Engineering Company performed the washability analyses. Washability analyses were only conducted on the +500 mesh (25 μm) size fraction since the technique is ineffective on the -500 mesh material.

The separation performance obtained from a continuously operated Packed-Column was also obtained for validating the new characterization procedure. Each of the coals samples were treated in a 4-inch (10-cm) diameter, 16 ft (4.9 m) tall Packed-Column. Typically, a froth depth of 10 ft (3 m) and an aeration rate of 4 cm/sec were used to ensure the rejection of entrainable material. The corresponding bias factors were greater
than 0.5. Wash water was added at the top of the froth zone at a flow rate of 2 liters/min. The air was injected directly into the bottom of the cell without the assistance of an external bubble generator. A PID controller was used to maintain the froth depth at the desired level through adjustment of the tailings pump.

RESULTS AND DISCUSSION

Conventional Techniques

Several tests were conducted to obtain the optimum separation performance for both the release and tree procedures. Figure 5 shows the “best” results obtained on the basis of ash rejection from the two procedures for the nominally -100 mesh Illinois No. 5 coal sample. As shown, the release procedure appears to provide a superior performance compared to the tree method. This finding is likely due to the inefficient recovery of the middling particles by the Tree analysis as indicated by the suppressed ‘elbow’ of the recovery-product ash curve. Therefore, the release procedure appears to provide a more efficient treatment of coals containing a significant amount of middling. As shown, the release analysis procedure provided ash reductions from 19.9% to nearly 5.5% while recovering 90% of the combustible material.

![Graph showing separation performance results]

Figure 5. Separation performance results obtained from the traditional release and tree procedures for the -100 mesh Illinois No. 5 coal sample; feed ash = 19.9%.
A review of the literature found little indication of the importance of feed solids content on the separation performance achieved by the tree and release analysis procedures. However, during this investigation, the feed solids content was found to be a critical parameter in determining the separation performance. The trend indicates that increasing the feed solids content increases the separation performance as shown for the release analysis procedure in Figure 6. This finding may be explained by the fact that a high solids content increases the amount of reflux occurring between the froth and pulp zones, which reportedly enhances selectivity. Therefore, conducting the release analyses at the low solids contents that are typical of flotation feed streams in operating coal preparation plants may not provide the “best” performance.

![Graph showing combustible recovery vs. product ash percentage](image)

**Figure 6.** Effect of feed solids concentration on the separation performance achieved by the traditional release analysis procedure on the -100 mesh Illinois No. 5 coal sample; feed ash = 19.9%.

**AFW Technique**

The optimum separation performances achieved on the nominally -100 mesh Illinois No. 5 coal sample using the Advanced Flotation Washability (AFW) technique and the conventional analyses are compared in Figures 7 (a) and (b) with that obtain from the centrifugal gravity-based washability analysis. For each of the characterization techniques, the near horizontal portion of the curve represents the rejection of liberated gangue material while recovering nearly all of the combustible material. The vertical portion of the curve, which represents a sharp decrease in recovery with decreasing product ash, is formed according to the manner in which the middling particles report to
Figure 7. A comparison of the separation performances obtained on the basis of ash rejection from the proposed Advanced Flotation Washability (AFW) method, the conventional techniques and the gravity washability analysis on the (a) -100 mesh and (b) micronized Illinois No. 5 coal sample; feed ash content = 19.9%.
the clean coal concentrate. A comparison of the AFW and release analysis procedures indicates that the separation performances on the basis of ash content are nearly equal along the horizontal portion of the curve. However, the AFW method provides a superior performance along the vertical portion, which indicates that the AFW procedure provides a more efficient treatment of middling particles.

As shown in Figure 7(a), the ash reduction obtained from the AFW procedure at 80% combustible recovery was from 19.9% to 4.5% for the -100 mesh Illinois No. 5 coal sample which corresponds to an ash rejection of nearly 85%. However, this performance is substantially inferior to that of the gravity-based washability which confirms the fact that gravity-based separations treat middling particles more efficiently than surface-based separations. However, the reduction in the amount of middling particles through grinding allows the surface-based separations to approach the efficiency of the gravity-based separations, which is shown by a comparison between Figures 7(a) and (b). After micronization of the Illinois No. 5 coal sample, the product ash content obtained at 80% recovery was approximately 2.5%, which is nearly equal to the gravity-based separation performance. This separation represents an ash rejection of 92% and a high separation efficiency of 72%. It is also worth noting that the difference in the separation performances between the AFW procedure and the release analysis increased upon micronization of the coal sample, which may be due to an improved bubble-particle collision environment offered by the Packed-Column. Also, the separation performance provided by tree analysis was found to be superior to the release analysis, which is likely caused by: 1) reduction in the amount of middlings and 2) better recovery mechanisms by the tree analysis when compared the release procedure.

The separation performance differences were greater on the basis of pyritic sulfur content reductions as shown in Figures 8(a) and (b) for the -100 mesh and micronized Illinois No. 5 coal samples, respectively. The AFW procedure provided product pyritic sulfur contents that were about 0.2% weight units lower than that of the release method. This is likely due to the more efficient ability of the Packed-Column to reject the floatable pyrite particles, both liberated and non-liberated. The large reflux action occurring as a result of the deep froth, which also reduces the possibility of entrainment, and a heavy solids loading causes the superior rejection of the pyrite particles. For the -100 mesh coal sample, the pyritic sulfur content was reduced during the AFW procedure from 1.38% to about 0.70% while recovering 80% of the combustibles, which equates to a pyritic sulfur rejection of 66%. This separation performance is substantially inferior to that of the gravity-based washability which obtained an 81% pyritic sulfur rejection at 80% recovery.

After micronization, the AFW procedure provided substantially lower product pyritic sulfur contents and a superior performance compared to the conventional characterization methods as shown in Figure 8(b). While recovering 80% of the combustible material, the AFW procedure produced a coal concentrate containing 0.32% pyritic sulfur which equates to a relatively high pyritic sulfur rejection of 85%. It should be noted that this separation performance represents an improvement over that provided by the gravity-
Figure 8. A comparison of the separation performances obtained on the basis of pyritic sulfur rejection from the proposed Advanced Flotation Washability (AFW) method, the conventional techniques and the gravity washability analysis on the (a) -100 mesh and (b) micronized Illinois No. 5 coal sample; feed pyritic sulfur = 1.38%.
based separation shown in Figure 8(a). Another difference that is observed between Figures 8(a) and (b) is that the release analysis procedure provided a superior pyritic sulfur rejection in the high recovery region when compared to the performance of the tree analysis. As explained previously, the tree analysis procedure provides a larger number of opportunities to float hydrophobic particles when compared to the release procedure, which enhances recovery but reduces the selectivity among the hydrophobic particles such as coal pyrite. This may explain the inferior performance provided by the tree analyses for the rejection of liberated pyrite.

Figure 9 provides a comparison of the sulfur dioxide emission rating obtained from the treatment of the -100 mesh Illinois No. 5 coal sample using the AFW and release analysis procedures. Due to improvements obtained in ash and pyritic sulfur content reductions, the AFW procedure provided a product containing substantially lower amounts of SO$_2$/MBTU. At a combustible recovery of 80%, the difference was almost unity with the AFW procedure achieving a 2.45 lbs SO$_2$/MBTU, which represents a Phase I compliance coal.

Figure 9. A comparison of the reductions obtained in the potential sulfur dioxide emissions predicted for the -100 mesh Illinois No. 5 coal sample by the Advanced Flotation Washability (AFW) and release analysis techniques.
An important feature of any characterization process is repeatability. Previous reporting of data (i.e., literature, reports and professional meetings) obtained from the traditional release analysis procedure indicated significant variations between tests involving different operators. The automatic control system in the AFW procedure removes the operator from the product collection task, thereby, enhancing repeatability. Five AFW tests conducted under the same conditions were conducted and the separation performance data are plotted in Figure 10.

Figure 10. Separation performance results obtained from 5 separate tests showing the excellent repeatability of the AFW analysis; -100 mesh Illinois No. 5 coal sample; feed ash = 19.9%.

Comparison with Continuous Column Results

As mentioned previously in this report, test results achieved from single stage cleaning by a continuously operated Packed-Column were found to be superior in a previous ICCI project report to those obtained from the traditional release analysis. Flotation columns can be operated to provide two different limiting conditions which provide a variation in separation performance. When the feed to the flotation column contains a large amount of solids (e.g., > 10% by weight) and/or the particle size is very fine (e.g., 100% -200 mesh), the column operates under carrying-capacity limiting conditions in which insufficient bubble surface area exists to float all of the hydrophobic particles reporting to the froth zone. Thus, upon bubble coalescence, particles detach from the bubbles and report back to the pulp zone where the particles can re-attach to a bubble and report to the
froth zone. This recycling event is referred to as reflux and contributes to an increase in selectivity, which may be further enhanced by the possibility that the bubble-particle detachment process is selective. On the other hand, if sufficient bubble surface area exists, the column is considered to be kinetic rate limited. Multiple stage cleaning under kinetic rate limiting conditions (i.e., low feed solids content and/or relatively large particle sizes) has been found to improve the overall separation performance, whereas this finding was not realized for columns that were carrying-capacity limited.

Figures 11 (a) and (b) compare the results obtained from a continuous Packed-Column operated under kinetic and carrying-capacity limiting conditions with those obtained from the AFW and release analysis procedures. Under carrying capacity conditions, a rougher cleaning stage was the only treatment cleaning step evaluated whereas rougher, rougher-cleaner, and rougher-cleaner-cleaner circuit arrangements were tested using the Packed-Column under kinetic limiting conditions. As shown, the separation performance provided by the extensive three stage cleaning (R-C-C) under kinetic limiting conditions remained inferior to that of the AFW procedure on the basis of both product ash (Figure 11a) and pyritic sulfur (Figure 11b) contents, although the performance approached the AFW curve with each subsequent cleaning stage. On the other hand, operating the Packed-Column under carrying-capacity conditions provided a nearly equal separation performance on the basis of product ash content when compared to the AFW curve and a superior performance when compared to the traditional release results as shown in Figure 11(a). This finding is consistent with the explanation of improved selectivity with carrying-capacity conditions. However, in terms of the product pyritic sulfur content, the performance from the AFW procedure remained superior.

**Comparisons Using Other Coal Sources**

Test results on the basis of product ash content obtained from the treatment of a nominal -100 mesh and micronized IBC-105 (Illinois No. 6) coal sample are shown in Figures 12 (a) and (b), respectively. The ash rejections achieved by both the continuous Packed-Column and the release analysis procedure were inferior to those provided by the AFW method. The AFW results indicate a reduction of ash content from 16.0% to 7.2% while recovering about 96% of the combustibles. This equates to an ash rejection of 61% and a separation efficiency of 57%. However, liberation of the gangue material by micronization resulted in a further reduction in ash content to approximately 4.0% at 90% recovery as shown in Figure 12(b). Thus, micronizing the IBC-105 coal sample resulted in an improved in separation efficiency to about 70%.

Substantial differences in the separation performances were realized on the basis of the pyritic sulfur content as shown in Figures 13 (a) and (b). Because of the relatively coarse particle size distribution of the IBC-105 coal sample, it is likely that the Packed-Column was operated under kinetic-limiting conditions which may explain the significantly inferior results shown in Figure 13(a) when compared to those from the AFW procedure. On the -100 mesh sample, the AFW procedure achieved a reduction in the pyritic sulfur
Figure 11. A comparison of the separation performances achieved on the basis of a) product ash and b) pyritic sulfur contents by the AFW and conventional flotation analysis methods, the continuous Packed-Column under carrying-capacity limiting conditions and rougher R, rougher-cleaner (R-C), and rougher-cleaner-cleaner (R-C-C) arrangements of the Packed-Column under kinetic limiting conditions; -100 mesh Illinois No. 5.; feed ash and pyritic sulfur contents = 19.9% and 1.38%, respectively.
Figure 12. Separation performances on the basis of product ash content achieved on the a) standard and b) micronized IBC-105 coal samples by the Advanced Flotation Washability (AFW) and release analyses and the continuously operated Packed-Column; feed ash = 16.0%.
Figure 13. Separation performances achieved on the a) standard and b) micronized IBC-105 coal samples by the Advanced Flotation Washability (AFW) and release analyses and the continuously operated Packed-Column; total and pyritic sulfur contents = 3.72% and 1.62%, respectively.
content from 1.62% to 0.62% at 96% recovery which corresponds to a pyritic sulfur rejection of 66.7% and a product total sulfur content of 2.94%.

Figures 14 and 15 show the separation performance achieved on the basis of product ash and pyritic sulfur contents, respectively, for a Kentucky No. 9 coal sample. For this sample, the performance achieved on the basis of product ash content by the continuous Packed-Column was found to be inferior to the AFW results but superior to the traditional release results. The difference in separation performances between the AFW curve and the Packed-Column curve increased on the basis of pyritic sulfur, indicating that the pyrite in the West Kentucky No. 9 coal seam may be moderately hydrophobic and relatively easy to float. The AFW performance curve indicates an ash content reduction of 34.0% to about 5.0% for the West Kentucky No. 9 sample while recovering 90% of the combustibles. At the same recovery, the pyritic sulfur content can be reduced from 1.28% to 0.46% which equates to a pyrite rejection of nearly 78%.

![Figure 14](image)

**Figure 14.** Separation performances on the basis of product ash content achieved on a West Kentucky No. 9 coal sample by the Advanced Flotation Washability (AFW) and release analyses and the continuously operated Packed-Column; feed ash and pyritic sulfur contents = 34.0% and 1.20%, respectively.
Figure 15. Separation performances on the basis of product pyritic sulfur content achieved on a West Kentucky No. 9 coal sample by the Advanced Flotation Washability (AFW) and release analyses and the continuously operated Packed-Column; feed ash and pyritic sulfur contents = 34.0% and 1.20%, respectively.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

Based on the analysis of the test results obtained during this investigation, the following conclusions were derived:

1. The Advanced Flotation Washability (AFW) procedure developed in this investigation provides a superior separation performance when compared to the conventional and modified release analysis procedures. This finding was obtained from the treatment of three nominally -100 mesh coal samples and two micronized coal samples. The superior performance is due to the use of a batch-operated Packed-Column which provides: 1) a plug-flow environment which provides a better bubble-particle collision environment than the perfectly-mixed conditions provided by the conventional cell and 2) a deep froth to eliminate entrainment and to provide a competitive particle recovery environment through a reflux action.
2. The largest difference in separation performance was obtained on the basis of product pyritic sulfur content which is most likely due to the apparent natural floatability of the coal pyrite particles. At 80% combustible recovery, a 19% improvement in the reduction of pyritic sulfur was obtained from the AFW technique when compared to the optimum result achieved using the release analysis procedure when treating a -100 mesh Illinois No. 5 coal sample. From the AFW results, this improvement corresponds to a reduction in pyritic sulfur content from 1.38% to 0.70% which equates to a pyritic sulfur rejection of 66%. After micronizing the coal, the pyritic sulfur content was further reduced to 0.32% at the same combustible recovery value which corresponds to a pyritic sulfur rejection of 85%. The ash content for the micronized sample was reduced from 19.9% to 2.5% at 80% recovery which is an ash rejection of 92%.

3. The maximum selectivity achieved from a continuously operated Packed-Column was obtained under carrying-capacity limiting conditions. Under these conditions, the single stage cleaning of a -100 mesh Illinois No. 5 coal sample was found to provide nearly equal performance on the basis of product ash content as compared to that of the AFW procedure. However, in terms of the product pyritic sulfur content, the AFW curve was significantly inferior. On the other hand, test results from the Packed-Column when operated under kinetic limiting conditions indicate that approximately three cleaning stages are needed to achieve a separation performance nearly equal to the AFW performance.

4. The Advanced Flotation Washability (AFW) procedure reduces the role of the operator during the performance of the procedure which ultimately improves the statistical repeatability of the test data using different operators. This represents an important improvement over the tree and release procedures. In fact, the operator's role is limited to the setting and adjustment of the operating parameters. The concentrate is collected in an overflow launder while an automatic control system maintains a constant pulp level. Tests conducted using the AFW procedure under constant test conditions indicate excellent repeatability of the test results.

5. The results obtained from the traditional flotation characterization procedures conducted on the Illinois No. 5 coal indicated that the tree procedure consistently produced inferior performance in the high recovery region of the combustible recovery-grade curve. Since a substantial portion of the combustibles in the tree procedure is recovered in the scavenger flotation steps, the heavy middling type particles tend to report to the concentrate, which considerably increases the ash content of the concentrate. Coal pyrite particles, being weakly hydrophobic also report to the concentrate during the scavenger flotation steps which enhances the sulfur content of the concentrates. Thus, the superior performance obtained from the successive cleaning stages conducted on the individual flotation concentrate samples produced during the tree procedure is heavily offset by the flotation of heavy middling and coal pyrite particles in the scavenger flotation steps.
6. The feed solids content used in the AFW and conventional procedures was found to significantly affect the separation performance curve. The separation performances based on both ash and pyritic sulfur contents were found to improve with an increase in the feed solids content up to 20% by weight. This is believed to be due to an enhancement in the competitive environment in the flotation process which improves the separation results. Thus, the "best" possible flotation response for a given coal must be obtained at high solid contents of approximately 20% by weight. However, plant operators may desire to obtain a "real" picture for their plant conditions and, thus, will conduct the analysis at the solids content of the stream in the plant. As a result, the optimum flotation performance achieved from the AFW or conventional techniques will be optimum only for the feed solids content at which the test was conducted.

7. The gravity-based washability results were found to be superior to the separation performance obtained from each of the flotation characterization techniques conducted on a -100 mesh Illinois No. 5 coal sample. However, the difference in separation performance achieved by the gravity-based washability and the AFW procedure was reduced as the coal sample was ground to a finer particle size.

**Recommendations**

The followings represent recommendations or future work plans which are based on the findings presented in this final technical report:

1. A fundamental study of the selectivity of the bubble-particle detachment process needs to be conducted to fully understand and improve the performance of advanced flotation system.

2. Petrographic and scanning electron microscope analyses need to be conducted on the flotation concentrates from the AFW procedure to identify the particles being floated and quantify the true efficiency of the proposed procedure.

**DISCLAIMER STATEMENTS**

This report was prepared by Dr. R. Q. Honaker of Southern Illinois University at Carbondale with support, in part by grants made possible by the U. S. Department of Energy Cooperative Agreement Number DE-FC22-92PC92521 (Year 4) and the Illinois Department of Commerce and Community Affairs through the Illinois Coal Development Board and the Illinois Clean Coal Institute. Neither Dr. R. Q. Honaker of Southern Illinois University at Carbondale nor any of its subcontractors nor the U. S. Department of Energy, the Illinois Department of Commerce and Community Affairs, Illinois Coal Development Board, Illinois Clean Coal Institute, nor any person acting on behalf of either:
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REFERENCES


PROJECT MANAGEMENT REPORT
June 1, 1996, through August 31, 1996

Project Title:  A MODIFIED RELEASE ANALYSIS PROCEDURE USING
ADVANCED FROTH FLOTATION MECHANISMS

DOE Cooperative Agreement Number:  DE-FC22-92PC92521 (Year 4)
ICCI Project Number:  95-1/1.2B-1P
Principal Investigator:  R. Q. Honaker
                     Department of Mining Engineering
                     Southern Illinois University at Carbondale
Other Investigator:  M. K. Mohanty
                     Department of Mining Engineering
                     Southern Illinois University at Carbondale
Project Manager:  K. Ho, ICCI

COMMENTS

None.
## PROJECTED AND ESTIMATED EXPENDITURES BY QUARTER

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*Cumulative by Quarter
CUMULATIVE COSTS BY QUARTER

A Modified Release Analysis Procedure Using Advanced Froth Flotation Mechanisms

![Graph showing cumulative costs by quarter]

- ● = Projected Expenditures
- ▲ = Actual Expenditures

Total Illinois Clean Coal Institute Award $39,280
SCHEDULE OF PROJECT MILESTONES

A
B
C
D
E
F
G

Begin
Sept. 1
1995

Hypothetical Milestones:

A: Equipment ordered and received
B: Sample Acquisition and Characterization (Task 1)
C: Construction of Release Analysis Apparatus (Task 2)
D: Procedure Development for Modified Release Analysis (Task 3)
E: Comparison with Traditional Methods (Task 4)
F: Comparison with Advanced Floatation Technologies (Task 5)
G: Reporting

Comments:

None.
HAZARDOUS WASTE REPORT
June 1, 1996, through August 31, 1996

Project Title: A MODIFIED RELEASE ANALYSIS PROCEDURE USING ADVANCED FROTHER FLOTATION MECHANISMS

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 4)
ICCI Project Number: 95-1/1.2B-1P
Principal Investigator: R. Q. Honaker
Department of Mining Engineering
Southern Illinois University at Carbondale

Other Investigator: M. K. Mohanty
Department of Mining Engineering
Southern Illinois University at Carbondale

Project Manager: K. Ho, ICCI

HAZARDOUS WASTE REPORT

All hazardous wastes were turned over to SIUC Center for Environmental Health and Safety to be disposed of in accordance with EPA/SIUC approved disposal plan.