CHAPTER 2
PROCEDURE

2.1 Introduction

A comparison of the three major release analysis techniques, timed release analysis, simplified release analysis, and tree analysis, can be conducted in two ways. From a theoretical perspective, it is possible to model each technique and compare the procedures through simulation. From an experimental perspective, it is possible to experimentally compare the procedures by conducting tests on a common sample or set of samples. In this investigation, both methods were used in order to compare the techniques and determine which technique gives the best approximation of the ideal separation. This chapter serves to describe both the mathematical and experimental procedures used in this comparison.

2.2 Modeling and Simulation

All three common release analysis procedures are very easily studied on a theoretical basis because they can be modeled using simple mathematics. Three models are needed to completely represent any of these procedures. These include a process model, a material model, and a procedural model. The process model represents the flotation process itself, the material model represents the mineral being studied, and the procedural model represents the actual testing procedure. The following sections describe each of these three models as they were used in this investigation.

2.2.1 Process Model

All of the various release analysis procedures being studied in this investigation use a laboratory-scale, semi-batch flotation cell. Assuming the cell is perfectly mixed and flotation can be represented as a first-order rate process, a mass balance on floatable component i yields:

\[
\frac{dC_i}{dt} = -k_i C_i
\]  \hspace{1cm} (1)

where \( C_i \) represent the concentration of floatable component i in the cell at any time, and \( k_i \) represents the flotation rate constant for floatable component i. If make-up water is added to the cell to keep the pulp volume constant, then concentration can be replaced with mass and the expression becomes:

\[
\frac{dM_i}{dt} = -k_i M_i
\]  \hspace{1cm} (2)
where \( M_i \) represents the mass of floatable component \( i \) in the cell. Assuming an initial mass of component \( i \), \( M_{i,0} \), is placed in the cell at time zero, and integrating from time zero to some time, \( t \), Equation (2) becomes:

\[
\frac{M_i}{M_{i,0}} = \exp(-k_i t)
\]  

(3)

By definition, recovery of component \( i \), \( R_i \), at any time, \( t \), is given as:

\[
R_i = 1 - \frac{M_i}{M_{i,0}}
\]  

(4)

Substituting Equation (4) into Equation (3), the model equation representing the recovery of component \( i \) as a function of time in a semi-batch flotation cell is given by the standard plug-flow model:

\[
R_i = 1 - \exp(-k_i t)
\]  

(5)

When Equation (5) is applied to all of the various components in the feed material, the overall recovery and grade for a single cell can be determined. Thus, the application of Equation (5) requires a knowledge of flotation time, \( t \), and the flotation rate constant, \( k_i \), for each component present in the feed.

2.2.2 Material Model

The material model consists of three parts: the component composition, the mass of material in each component class, and the flotation rate constant for each component class. In this investigation, the component composition classes were preset at 10% ash increments, while the mass of material in each class and the flotation rate constant for each class were determined by back-fitting these values to experimental data obtained from simplified release analysis tests performed on coal samples from the Stockton and Beckley seams. The set of flotation rate constants and component masses that produced the minimum residual between the experimental and simulated ash and yield values for all points on each release curve were determined to be the best-fit solution. The Solver feature on Microsoft Excel was used to perform this fit.

The material models developed for each of the two coal samples are shown in Tables 1 and 2, and the results of the data fit are shown in Figures 5 and 6. As shown, the fit to the experimental data is excellent, and the models developed for each of the two coal samples are also in excellent agreement with the commonly known properties of these two samples. For example, the Stockton seam is known to have a much higher middlings content than the Beckley seam. This is exemplified by the fact that the simulated Stockton seam contains over 25% of its weight in the 20-80% ash classes, while the simulated Beckley seam contains only 5% of its weight in this same range.
Table 1. Material model for the simulated Stockton seam coal sample.

<table>
<thead>
<tr>
<th>Class</th>
<th>Composition (% Ash)</th>
<th>Weight Percent</th>
<th>Ash Units</th>
<th>Rate Constant (min⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>26.55</td>
<td>0.00</td>
<td>1.00</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td>23.80</td>
<td>2.38</td>
<td>0.49</td>
</tr>
<tr>
<td>3</td>
<td>20</td>
<td>10.19</td>
<td>2.04</td>
<td>0.18</td>
</tr>
<tr>
<td>4</td>
<td>30</td>
<td>10.24</td>
<td>3.07</td>
<td>0.18</td>
</tr>
<tr>
<td>5</td>
<td>40</td>
<td>1.96</td>
<td>0.79</td>
<td>0.16</td>
</tr>
<tr>
<td>6</td>
<td>50</td>
<td>0.98</td>
<td>0.49</td>
<td>0.16</td>
</tr>
<tr>
<td>7</td>
<td>60</td>
<td>0.98</td>
<td>0.59</td>
<td>0.15</td>
</tr>
<tr>
<td>8</td>
<td>70</td>
<td>0.97</td>
<td>0.68</td>
<td>0.14</td>
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<td>9</td>
<td>80</td>
<td>0.95</td>
<td>0.76</td>
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</tr>
<tr>
<td>10</td>
<td>90</td>
<td>2.05</td>
<td>1.84</td>
<td>0.12</td>
</tr>
<tr>
<td>11</td>
<td>100</td>
<td>21.31</td>
<td>21.31</td>
<td>0.04</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>100.00</td>
<td>33.95</td>
<td></td>
</tr>
</tbody>
</table>

Table 2. Material model for the simulated Beckley seam coal sample.

<table>
<thead>
<tr>
<th>Class</th>
<th>Composition (% Ash)</th>
<th>Weight Percent</th>
<th>Ash Units</th>
<th>Rate Constant (min⁻¹)</th>
</tr>
</thead>
<tbody>
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<td>30.64</td>
<td>0.00</td>
<td>1.00</td>
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<td>2</td>
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<td>34.16</td>
<td>3.42</td>
<td>0.49</td>
</tr>
<tr>
<td>3</td>
<td>20</td>
<td>0.00</td>
<td>0.00</td>
<td>0.18</td>
</tr>
<tr>
<td>4</td>
<td>30</td>
<td>0.00</td>
<td>0.00</td>
<td>0.18</td>
</tr>
<tr>
<td>5</td>
<td>40</td>
<td>0.94</td>
<td>0.38</td>
<td>0.16</td>
</tr>
<tr>
<td>6</td>
<td>50</td>
<td>2.48</td>
<td>1.24</td>
<td>0.16</td>
</tr>
<tr>
<td>7</td>
<td>60</td>
<td>0.00</td>
<td>0.00</td>
<td>0.15</td>
</tr>
<tr>
<td>8</td>
<td>70</td>
<td>0.00</td>
<td>0.00</td>
<td>0.14</td>
</tr>
<tr>
<td>9</td>
<td>80</td>
<td>0.00</td>
<td>0.00</td>
<td>0.13</td>
</tr>
<tr>
<td>10</td>
<td>90</td>
<td>14.93</td>
<td>13.44</td>
<td>0.12</td>
</tr>
<tr>
<td>11</td>
<td>100</td>
<td>16.85</td>
<td>16.85</td>
<td>0.04</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>100.00</td>
<td>35.32</td>
<td></td>
</tr>
</tbody>
</table>
Figure 5. Comparison of simplified release analysis curves obtained for Stockton seam coal using the experimental procedure and a simulated procedure with back-fitted flotation rate constants.
2.2.3 Procedural Model

Each of the release or tree analysis procedures is modeled by combining the data from the material model with the process model and repeatedly applying these models to represent the flowsheets shown in Figures 2-4. This is most easily accomplished by use of a spreadsheet program. For example, in the case of timed release analysis, Equation (5) is used in conjunction with the data in Tables 1 and 2 and the flotation times specified by Dell (1953), or those used by Cavallaro and Deurbrouck (1965). The mass of material recovered in each composition class is calculated, and these concentrates subsequently become the feed to the next level of the procedure. In the case of the
simplified release analysis procedure, the first stage, which requires rigorous flotation conditions, is represented by applying Equation (5) several times in series for long flotation times (e.g., 15 minutes). In the second stage, where operating conditions are used to fractionate the sample, the rate constants are multiplied by a coefficient prior to each float to represent the progressive increases in aeration rate, impeller speed, and flotation time. In this investigation, coefficient values of 0.72, 2.38, and 7.37 were used to produce each of the three fractions for the simulated Stockton seam sample, and values of 0.43, 1.36, and 5.51 were used for the simulated Beckley seam sample. These values were determined from the same back-fitting procedure described previously. Finally, tree analysis is represented by using Equation (5) with a fixed flotation time (e.g., 2 minutes) to repeatedly process the concentrate and tailings produced until the desired number of stages has been achieved.

2.3 Experimental

Experimental tests were conducted to compare the three common release analysis techniques: timed release analysis, simplified release analysis, and tree analysis. The tests were carried out using two different coal samples, one a high-middlings coal and the other a “black-and-white” coal, as a means of comparing the effect of material type on each release procedure. In addition, two coals were selected that were sufficiently hydrophobic so that no collector would be needed in the test work. In this way, the procedures could be compared without chemistry effects masking process differences. The following describes the sample, sample preparation procedures, and experimental procedures used in this investigation.

2.3.1 Sample

Run-of-mine coal samples were collected from two Cyprus-Amax preparation plants located in West Virginia. The first sample was collected from the storage pile of the Lady Dunn plant and placed in a lined 55 gallon drum. The sample was collected from just beneath the surface of the storage pile to avoid obtaining oxidized coal. This sample originated from the Stockton seam and had a high middlings content. It was classified as a high volatile B bituminous coal with a calorific value between 13,000 and 14,000 Btu/lb. The second sample was collected from the feed belt to the Maple Meadow preparation plant and placed in a lined 55 gallon drum. This sample originated from the Beckley seam and had a lower middlings content than that of the Stockton sample. It too was classified as a high volatile B bituminous coal with a calorific value between 13,000 and 14,000 Btu/lb. A head sample was taken from each drum after the samples were prepared for flotation. These head samples were sized and analyzed for ash content. The results of these analyses are shown in Table 3.
Table 3. Sample characterization.

<table>
<thead>
<tr>
<th>Size</th>
<th>Beckley Seam</th>
<th>Stockton Seam</th>
</tr>
</thead>
<tbody>
<tr>
<td>+28</td>
<td>0.00</td>
<td>0.00%</td>
</tr>
<tr>
<td>28x35</td>
<td>0.00</td>
<td>0.00%</td>
</tr>
<tr>
<td>35x65</td>
<td>42.86</td>
<td>21.43%</td>
</tr>
<tr>
<td>65x100</td>
<td>27.47</td>
<td>13.74%</td>
</tr>
<tr>
<td>100x150</td>
<td>26.37</td>
<td>13.19%</td>
</tr>
<tr>
<td>150x0</td>
<td>103.30</td>
<td>51.65%</td>
</tr>
<tr>
<td>Totals:</td>
<td>200</td>
<td>100.00%</td>
</tr>
</tbody>
</table>

After the samples were transported back to the lab, each sample was crushed in two stages. The first stage involved crushing the sample with a jaw crusher. In the second stage, the jaw crusher product was further crushed with a roll crusher to -6 mm. Each crushed sample was split into eleven five-gallon lots with the cone and quartering technique. The lots were then split into 1000 gram charges with a Jones Riffler. These charges were stored in the laboratory freezers to minimize oxidation of the coal. When a test was conducted, a charge was removed from the freezer and pulverized to -28 mesh using a disc pulverizer. The pulverized product was then split into 200 gram test samples using a Jones Riffler. This sample preparation procedure was conducted to ensure that each flotation test sample was representative of the bulk run-of-mine sample.

2.3.2 Release Analysis Procedures

Timed Release Analysis: The timed release analysis procedure was begun by placing a 200-gram sample of coal into a four-liter batch flotation cell along with 3800 grams of water. This produced a pulp of 5% solids by weight. Next, five drops of frother (MIBC) were added and the slurry conditioned for five minutes at 1700 rpm. No collector was used since the material in question was naturally hydrophobic. A standard Denver Model D-12 laboratory flotation machine was used for all tests. It was selected because both the aeration rate and impeller speed could be controlled independently throughout the experiment. A group of four concentrates was then collected in succession. The first was collected for 30 seconds at 1100 rpm, the second for 60 seconds at 1300 rpm, the third for 90 seconds at 1500 rpm and the fourth for 120 seconds at 1700 rpm. The tailings from the cell were then emptied into a bucket and saved. To begin the second phase of the test the first concentrate was reconditioned for 5 minutes at 1700 rpm and five more drops of frother were added. Concentrate 1 was then floated alone for 15 seconds at 1100 rpm. Concentrate 2 was then added and collected for an additional 15 seconds at 1100 rpm. The collection pan was changed and concentrate 2 was collected for 30 more seconds at 1300 rpm. Concentrate 3 was then added and a product was collected for 30 seconds at 1300 rpm. Again the collection pan was changed and concentrate 3 was floated for 45 additional seconds at 1500 rpm. The fourth concentrate was then added and a product was collected for 45 seconds at 1500 rpm. A new collection pan was added and a concentrate was collected for 2 minutes at 1700 rpm. The tailings fraction for this phase was saved and combined with those from the first
phase. During the third phase of the test, the same procedure was used except that the
new concentrate was not added until two-thirds of the way through the collection time of
the previous concentrate. Again the tailings from this stage were saved and combined
with those from the previous two phases (Dell, 1972). The concentrates and tailings
were then filtered, dried, and assayed. The entire procedure is shown schematically in
Figure 2.

Simplified Release Analysis: The simplified release analysis test was started by
adding a 200-gram test sample to a four-liter batch cell along with 3800 grams of water.
This produced a slurry of 5% solids by weight. The slurry was then conditioned with 5
drops of MIBC frother for five minutes. A concentrate was then collected at an aeration
rate of 100% and an impeller speed of 1700 rpm until the froth was barren. The tailings
were then placed in a bucket and saved. This procedure was repeated two additional
times to complete the separation of floatable material from non-floatable material. More
floats may have been necessary if the coals or minerals in question were less hydrophobic
than the ones used in this investigation.

The second phase of the test involved the floatable material being separated into
fractions of varying floatability. This was done by floating the material over a range of
aeration rates and impeller speeds. The second phase was started by reconditioning the
floatable material for five minutes with 5 drops of MIBC frother. The impeller speed
was then set to 1100 rpm and the aeration rate to 25% of maximum, and the first
concentrate was collected until the froth was barren. The collection pan was then
changed and the impeller speed increased to 1300 rpm while the aeration rate was
changed to 50% of maximum. Again the froth was collected until it was barren. This
procedure was repeated twice more at 1500 rpm and 1700 rpm to produce a total of four
concentrates. The final tailings fraction was combined with those of the first stage and
all five fractions were then filtered, dried, weighed, and assayed. The entire simplified
release analysis procedure can be seen schematically in Figure 3.

Tree Analysis: A four-level tree was selected to characterize the coals being
studied in this investigation. The impeller rate was set at 1500 rpm and the aeration rate
at 50% of maximum, while the collection time was selected to be 2 minutes per float.
These mechanical flotation conditions were held constant throughout the test. A two-
minute collection time was selected based on the operator’s previous experience working
with the coals in question. No collector was used since the coals in question were
naturally hydrophobic and about 5 drops of MIBC frother were added to each float.
Each float was conditioned for five minutes at an impeller speed of 1700 rpm. The
initial feed was then floated and its product and tailings fractions were then refloated
separately. This procedure was repeated as needed to produce a four-level tree. A total
of 16 products were obtained, and they were subsequently filtered, dried, weighed, and
assayed. The products were then arranged in order of increasing ash content to produce a
release curve. A diagram of this procedure is included in Figure 4.