CHAPTER 1
INTRODUCTION

1.1 Background

In the field of mineral processing, ideal separation techniques are commonly employed to assess the cleanability of a sample or to determine the efficiency of a separation device. In the case of gravity separation, for example, float-sink analyses are used to generate washability curves which provide an estimate of the ultimate separability of a given sample. If float-sink analyses are applied to the feed and product streams from a gravity separation device, a partition curve is produced which is used to assess the efficiency of the separation device.

In order for an ideal separation to be useful as a measurement standard, two criteria must be met:

1. The ideal separation must be based on the same separation property as the actual separation process.

2. The separation property must be some intrinsic property of the material that can be readily adjusted to any specific value.

In the case of a gravity separation device, for example, the separation property in the actual process and in the float-sink analysis is specific gravity. Clearly it would not be appropriate to use float-sink analysis to characterize froth flotation, just as it would not be appropriate to use sieve analysis to characterize gravity separation. Furthermore, specific gravity can be adjusted to any required value through the use of a variety of heavy liquids or aqueous suspensions. Thus, a particle having a specific gravity of 1.6 will always sink in a solution with a specific gravity of 1.5 while a particle with a specific gravity of 1.4 will always float. There is never any case, in an ideal separation, where particles of the same specific gravity both float and sink.

Unfortunately, the same is not true for froth flotation. Froth flotation is a probabilistic process. At any point in time, a middling particle containing a small inclusion of hydrophobic material could encounter an air bubble and report to the froth product, while a particle composed entirely of hydrophobic material could remain in the pulp. In other words, with the exception of those particles that have no hydrophobicity whatsoever, there is always a finite probability that any particle can float at any time. Furthermore, the so-called property of “floatability” is a derived property made up of several factors including the hydrophobicity of the various phases, the particle morphology, the particle size, etc. Thus, floatability is not an intrinsic property which can be set to a predetermined value in an ideal separator. As a result, there is no truly ideal separation technique which can act as a counterpart in froth flotation to float-sink analysis in gravity separation.

In the case of froth flotation, the best separation that anyone can hope to achieve is one that approaches ideal behavior in the limit as the number of separation stages are increased. This is the approach that has been taken over the past 40 years with the development of the various forms of release analysis and tree analysis.

Release analysis was developed by C.C. Dell as the "counterpart in froth flotation to float and sink analysis in the gravity concentration of coal"(Dell, 1953). It was originally carried out using a standard laboratory batch flotation cell. The material to be separated was placed in the cell and froth products were collected for various increments of time. Each of these froth products was, in turn, floated and refloated, ultimately generating a series of products ordered in terms of grade. By appropriately combining the analyses from these products, a yield-ash curve could be constructed which purportedly represented the ultimate flotation separation possible for the specific sample tested, i.e., the release curve. The release curve was considered to be independent of cell type, reagent dosage, pulp density and operator interaction, and was believed to be a characteristic of the material and its degree of liberation or "state of release". Because Dell's original release analysis technique was based on time fractionation, it is often referred to as "timed release analysis". A slightly modified version of Dell's timed release analysis technique was later used by Cavallaro and Deurbrouck to characterize the flotation behavior of eastern U.S. coals (Cavallaro and Deurbrouck, 1965). For the most part, the procedure was identical to that developed by Dell; however, the time intervals were reduced due to the highly floatable nature of the coals tested.

In 1964, Dell outlined a simplified version of his release analysis procedure which was experimentally less difficult than the original version (Dell, 1964). In this case, the sample was fractionated by changes in operating conditions (i.e., aeration rate and impeller speed) as opposed to changes in flotation time. A comparison of the simplified release analysis technique with the timed release analysis technique showed both approaches to yield identical results; however, a later study indicated that the simplified technique was more sensitive to operator expertise. Furthermore, the release curve obtained by the simplified procedure seemed to be sensitive to cell design; although the reason for this observation was unclear. Tests conducted as a function of pulp density and collector type showed the simplified procedure to be relatively independent of these parameters (Dell et al., 1972). Similar results were reported by Forrest, Adel and Yoon who found that the release curves generated by the simplified procedure were relatively independent of frother and collector concentration as well as frother type for several highly floatable eastern U.S. coals (Forrest, Adel and Yoon, 1994).

In 1983, a new procedure, known as tree analysis, was suggested by Nicol et al. (1983). Under this approach, a laboratory batch flotation cell was used to separate a coal sample into a concentrate and tailings fraction. Each concentrate and tailings fraction was subsequently refloated, and the procedure was repeated so that the testing sequence branched out in the form of a tree. After four or five levels of branching, the resulting fractions were analyzed and the data arranged according to increasing ash content. The results were then combined to give the final yield-ash curve. A comparison of tree analysis and simplified release analysis (Pratten, Bensley and Nicol, 1989) showed that the tree procedure was better at establishing the low-ash, low-yield portion of the ultimate separation curve, while the simplified release procedure was slightly superior in the high-ash, high-yield range. However, the tree procedure was found to be totally independent of collector dosage, while the data suggested that this was not the case for the simplified release procedure. As a result, the tree procedure was considered to be the superior technique.

In spite of the work that has been conducted to define and develop the various release analysis procedures, these techniques are by no means considered standards for characterizing coal flotation. Over the years, a number of techniques have been developed for characterizing floatability and flotation performance. In addition to release analysis, these include film flotation, locked-cycle tests, test tube floatability tests, "frothless" flotation tests, etc. The following is a review of the procedures commonly employed to characterize flotation performance.

1.2 Literature Review

There have been many different methods of testing floatability and characterizing flotation performance reported in the literature over the past forty years. These include but are not limited to release analysis, test-tube floatability, film flotation, frothless flotation, locked-cycle tests, contact angle measurements, Hallimond tube flotation, and the use of novel cell designs such as the dissolved air Hallimond tube. This literature review discusses the advantages and disadvantages of the above floatability measurement techniques and shows that there is much work to be done within this area as none of them are accepted as standards in determining a mineral's floatability.

The Hallimond tube is a fairly well accepted method of testing floatability despite some serious shortcomings. In the original version, the test sample is placed as a loose bed of particles on top of a porous septum. Air is pushed through the bed and into contact with the particles. Since the particles are restricted in their movement, it is generally easier for the bubbles to attach themselves to the mineral particles than is the case in an industrial flotation cell. As a result, particles which would not float under industrial conditions may float in a Hallimond tube (Klassen and Makrousov, 1963). The introduction of a stirrer in a later version of the tube appeared to minimize this problem; however, some shortcomings still remain. These include (i) incomplete flotation chemistry (i.e., no frother is added), (ii) inability to generate real grade vs. recovery data, and (iii) unrealistic flow conditions (Chudacek, 1990). Therefore the Hallimond tube test is not considered representative of industrial flotation.

Contact angle measurements have been used for many years as an assessment of a mineral's hydrophobicity. Unfortunately, the method is very tedious and does not simulate the conditions of a conventional or column flotation cell. As a result, it is not considered to be a good representation of an industrial flotation circuit. It is, however, considered to be a good measurement of the degree of hydrophobicity of a coal or mineral surface (Macdonald and Brison, 1962).

The test-tube floatability test is based on the stoppered cylinder shaking test. In this test a graduated cylinder is filled to 30-50% of its volume with a solution containing approximately 1 gram of sample. It is then shaken vigorously either manually or mechanically. The solution in the test-tube contains both frother and collector so the flotation chemistry for the test is complete. The froth is then removed using a pipette and analyzed. This procedure is repeated three times to produce a grade-vs.-recovery curve. Accelerometry tests performed on the shaken test-tube and on a conventional flotation cell have shown that both produce bubbles in the 50-1000 micron range. This indicates that the shaken test-tube is hydrodynamically similar to the conventional cell (Chudacek, 1990). Initially, the major problem associated with the test-tube floatability procedure was its heavy dependence on the operator when the test-tube was manually shaken. This problem seems to have dissipated with the advent of mechanical shaking devices. The major advantages of this technique are that it is quick and simple to conduct, it is reproducible, and it can yield floatability, concentrate grade, and mineral recovery data from a very small sample. Unfortunately, this test is limited in that only a small amount of sample may be used which may or may not be representative of the bulk material. Another drawback is that only a fairly fine particle size range can be tested (i.e., around 38 microns). Therefore, this test is probably not representative of conditions in an industrial flotation cell. However, the test-tube floatability test can be used to get a quick "snapshot" of the floatability of a coal or mineral since it is easy to perform and requires only a small amount of sample.

"Frothless" flotation tests are conducted by manipulating the surface tension of the flotation medium. This is done by creating a series of methanol solutions with decreasing surface tensions which yield sequentially graded products. The analyses of these products results in the generation of grade-vs.-recovery curves. The technique is based on Zisman's concept of critical surface tension which states that a particle will float if the surface tension of the medium is above some critical value and sink if the surface tension is below this value (Hornsby and Leja, 1984). The test is carried out in the "frothless" flotation cell designed by Partridge and Smith with about 2 grams of sample placed on the fritted glass disc at the bottom of the cell. Nitrogen is introduced into the cell and the various products are collected from each solution with a specific surface tension. The main advantages of this procedure are that it is quick and easy to complete and the procedure produces a number of products of varying floatability so that grade-vs.-recovery curves can be produced (Hornsby and Leja, 1984). Unfortunately, the procedure is not really comparable to industrial practice since flotation is rarely carried out in methanol solutions.

Film flotation is often considered similar to frothless flotation in that it too uses a methanol solution. In this instance a thin monolayer of particles (0.06 - 0.3 g) is placed on the surface of a liquid of a certain surface tension in a shallow vessel. The floatable particles remain afloat while the non-floatable particles sink. If the procedure is conducted with a range of solution surface tensions, a grade-vs.-recovery curve can be generated. This test is good for measuring a sample's overall floatability and for finding its critical surface tension. Another advantage is that it is quick and fairly easy to perform (Fuerstenau and Williams, 1987). Unfortunately, this test has a number of shortcomings. First, it does not accurately simulate the chemical conditions found in an industrial cell since methanol is rarely used as a medium in industrial scale flotation. Second, the flotation mechanism is completely different from that of an industrial cell. In this test flotation is achieved by manipulating the surface tension of the medium, while in most industrial cells flotation is caused by bubbles and particles attaching themselves to one another. Therefore, this test is not considered an ideal candidate for characterizing the flotation response of a material under industrial flotation conditions.

Up to this point the various floatability tests which have been discussed use properties such as critical surface tension and contact angle to test the floatability of a mineral. These tests are good procedures for finding a mineral's general flotation behavior but they do not necessarily predict how a mineral will behave in an industrial flotation circuit. Tests such as release analysis, tree analysis, locked-cycle tests, and the dissolved-air flotation microcell test are generally considered better for characterizing a mineral's flotation behavior in an industrial environment.

The dissolved-air flotation microcell test is carried out in a modified Hallimond tube adapted to use the dissolved-air technique. The dissolved-air procedure involves introducing pressurized water into a cell through a needle valve. When the pressure is released as the water enters the cell, fine bubbles (-120 micron) "precipitate" out (Matis, Mavros and Kydros, 1991). These fine bubbles increase the recovery rate by improving bubble-particle collision efficiency (Yoon, Luttrell and Adel, 1989). In a typical test a 200 ml suspension is produced with a pulp density of 0.5% and a particle size range of 25 x 10 microns (Matis, Mavros and Kydros, 1991). The suspension is conditioned with a xanthate collector and appropriate pH modifiers. Pressurized water is then introduced into the cell and the release of pressure produces the very fine bubbles. A concentrate is then collected and analyzed. The main advantage of this test is that the experiments are performed under conditions similar to those in large-scale flotation cells. For example, laminar flow conditions are prevalent in the collection and flotation areas of these cells, due mainly to the relatively slow bubble rise velocity (Matis, Mavros and Kydros, 1991). This improves the rate of particle adhesion which in turn improves the flotation efficiency. Other advantages of this test are that it provides the capability of conducting floatability tests with small-size bubbles, a release analysis procedure can easily be adapted to it, and it allows for fast experiments with low mineral consumption. Unfortunately, the test still possesses some of the disadvantages of the original Hallimond tube including incomplete flotation chemistry, low operating solids range, and unrealistic flow conditions as compared to conventional flotation. Therefore, this test is generally not used for characterizing flotation behavior on an industrial basis.

The locked-cycle flotation test is one of the better procedures available since it is capable of providing a lot of information about a continuous flotation circuit (Nishimura, et. al, 1989). The test consists of a series of batch tests conducted with a small scale laboratory apparatus, where the products generated in one test (nth cycle) are added to the next test ((n+1)th cycle). A flow sheet of the test is shown in Figure 1. Note that the test consists of five batch tests or stages which include a rougher, 2 scavenger, and 2 cleaner stages. The test can be arranged in any circuit configuration, but Figure 1 shows the ideal 2 stage Rougher-Cleaner-Scavenger circuit as presented by Meloy (1983). In order to obtain acceptable information from a locked-cycle test, steady-state must be reached. Unfortunately, this leads to the major disadvantage of this procedure which is the fact that it is very time consuming. It has been shown that the test normally requires at least five cycles to reach steady-state, even in a simple circuit, and the time to reach steady-state increases as the amount of recycle material increases (Nishimura et al., 1989). The procedure's main advantage is that the results provide good agreement with tests conducted on continuous flotation circuits. In other words the locked-cycle test is a good simulation of a plant circuit because it accurately reproduces the conditions of an industrial flotation circuit (Agar, 1985). Therefore, the locked-cycle test is as good a characterizing procedure as any found within the literature.

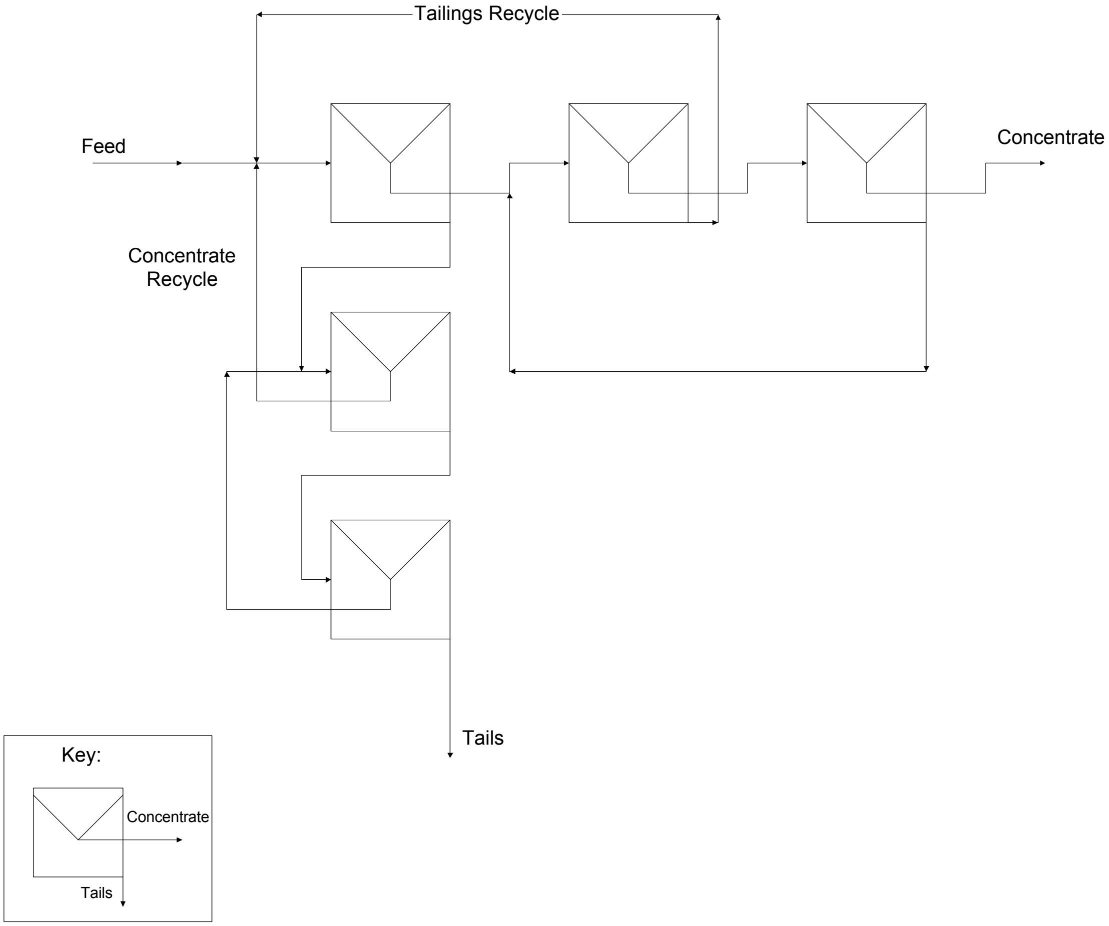

Figure 1. Schematic diagram of the locked-cycle flotation test procedure.

The first release analysis procedure was developed by C. C. Dell in 1953 and is known as timed release analysis (Dell, 1953). In this procedure time is used to separate a sample into fractions of varying floatability. The flotation procedure is carried out in a laboratory batch cell and an excess of reagent is used throughout. A serious drawback of this method is that water has to be decanted off the collection pan before a concentrate can be reintroduced to the cell in subsequent flotation stages to avoid pulp overflow and entrainment of gangue material. In the case of coal flotation, decantation is not very practical since low-density coal tends to remain in suspension and is often removed when water is decanted.

In 1965, the decantation problem was partially solved by adjusting the flotation times associated with the procedure (Cavallaro and Deurbrouck, 1965). The procedure was additionally modified to allow for varying mechanical flotation conditions. Instead of only varying the flotation time for each concentrate, the impeller speed was also varied. This supposedly improved the separation because the fractionation was not just based on time but also on the physical flotation conditions.

The only major problem associated with timed release analysis is that it is time consuming and rather tedious. However, it has been found to be reproducible, independent of cell type, reagent dosage, pulp density, and operator interaction. A flowsheet of the procedure is shown in Figure 2.

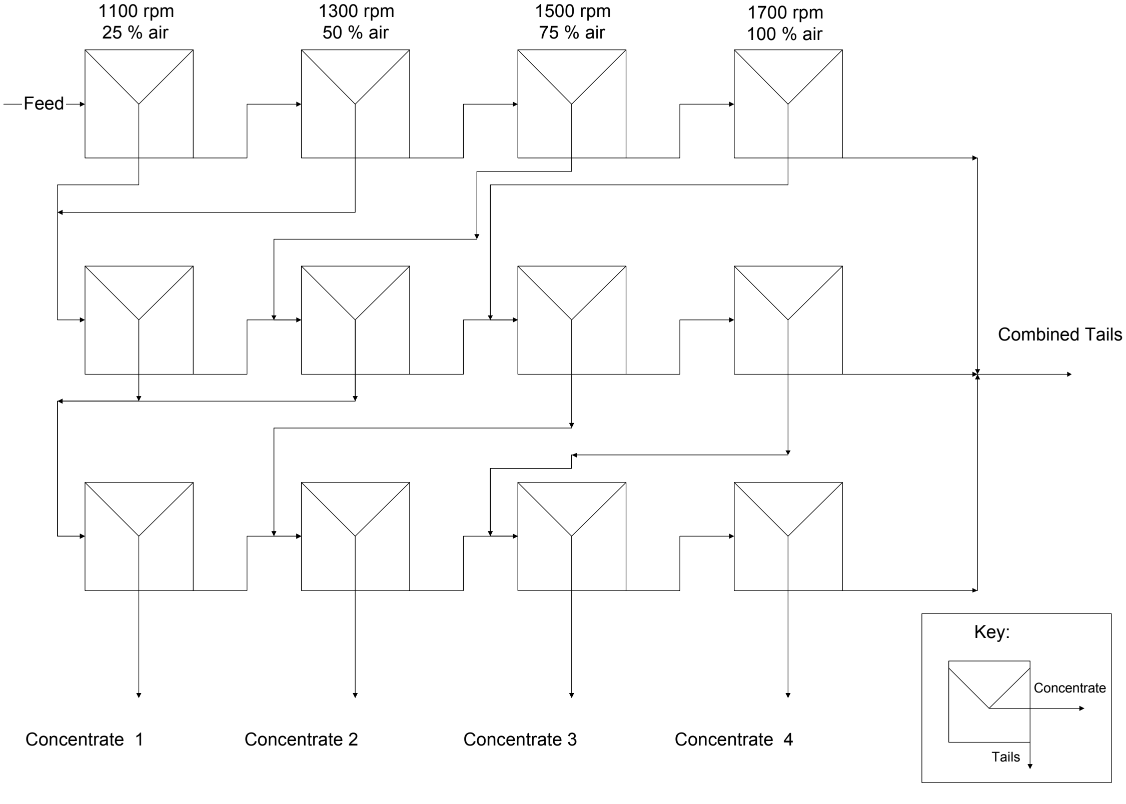

Figure 2. Schematic diagram of the timed release analysis procedure.

In 1964, Dell outlined an experimentally simpler version of the original timed release analysis procedure referred to here as simplified release analysis (Dell, 1964). In the simplified release analysis procedure, the sample is fractionated by operating conditions as opposed to time used in the original procedure. Again the experiment is carried out in a standard laboratory flotation cell which allows for independent control of impeller speed and aeration rate. As in the original procedure an excess of reagent is used.

The simplified release analysis procedure consists of two stages. The first stage objective is to completely separate the floatable material from the non-floatable material by repeated flotation of the concentrate under intense flotation conditions (i.e., high aeration rate and impeller speed). The second stage objective is to separate the floatable material from the first stage into fractions beginning with the low-recovery, high-grade material and proceeding to the high-recovery, low-grade fraction. The concentrate from the first stage is floated at several impeller speeds and aeration rates of steadily increasing intensity. Under these flotation conditions, concentrates of decreasing quality are collected and a release curve is generated. Therefore, the fractionation is dependent on the flotation intensity and not the timing of the concentrate collection. A flowsheet of this procedure is shown in Figure 3.

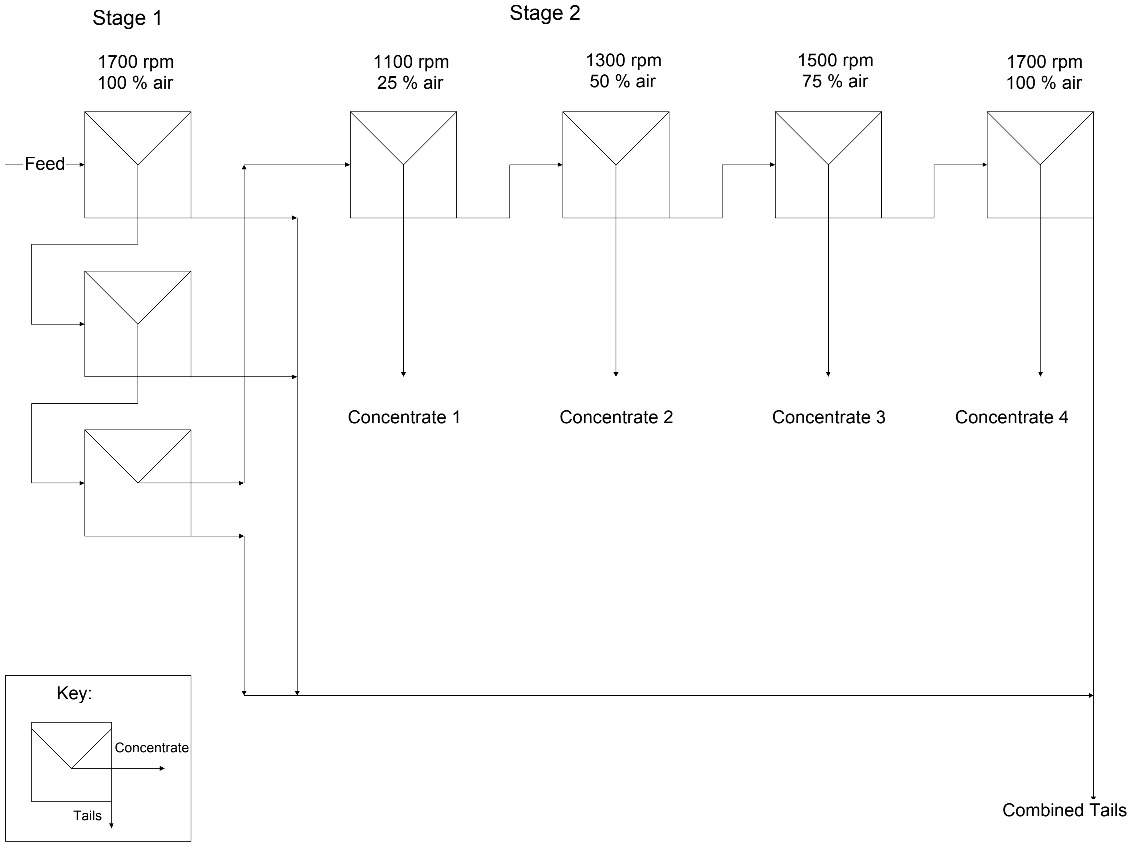

Figure 3. Schematic diagram of the simplified release analysis procedure.

Although the simplified procedure is generally considered simpler than the timed release analysis technique, it may be more sensitive to operator bias. An example of this is found in the first stage of the experiment when the floatable material is separated from the non-floatable material. The number of floats used and the way in which the froth is removed can greatly affect this separation. If any floatable material is lost during this part of the experiment, the release curve can be impacted significantly. During the second stage, it is often difficult to control the rate of flotation by simply adjusting aeration rate and impeller speed. In the case of a highly floatable coal, for example, the valuable material may tend to float within the first two fractions leaving very little material for the latter fractions. This results in the data being grouped at either the high-yield end or low-yield end of the release curve, depending on the floatability of the material. The problem may be avoided by using flotation time to fractionate the sample in the second half of the test (Pratten et al, 1989); however, this procedure deviates from Dell's original approach, and there is some question as to how the release curve is impacted by time fractionation in the second stage.

As a result of the possible experimental biases associated with release analysis, an alternative technique, known as tree analysis, was developed in 1983 (Nicol et al., 1983). The procedure was based on the concept of fractionating a sample by progressively refloating the concentrate and tailings such that the test branches out in the form of a tree.

According to the tree analysis procedure, a sample is initially floated at some arbitrary set of flotation conditions (i.e. impeller speed, aeration rate, and flotation time) and is split into a concentrate and a tailings fraction. These fractions are then refloated under the same flotation conditions, and their subsequent concentrate and tailings fractions are refloated. This procedure is continued until the final products contain less than 2% of the initial feed mass or until the desired flotation level is reached. The final products are then analyzed and sorted in order of decreasing grade. These results are then used to construct a release curve. In order to produce data points in the high-grade region of the curve, collector dosages are generally kept low. A flowsheet of this procedure is shown in figure 4.

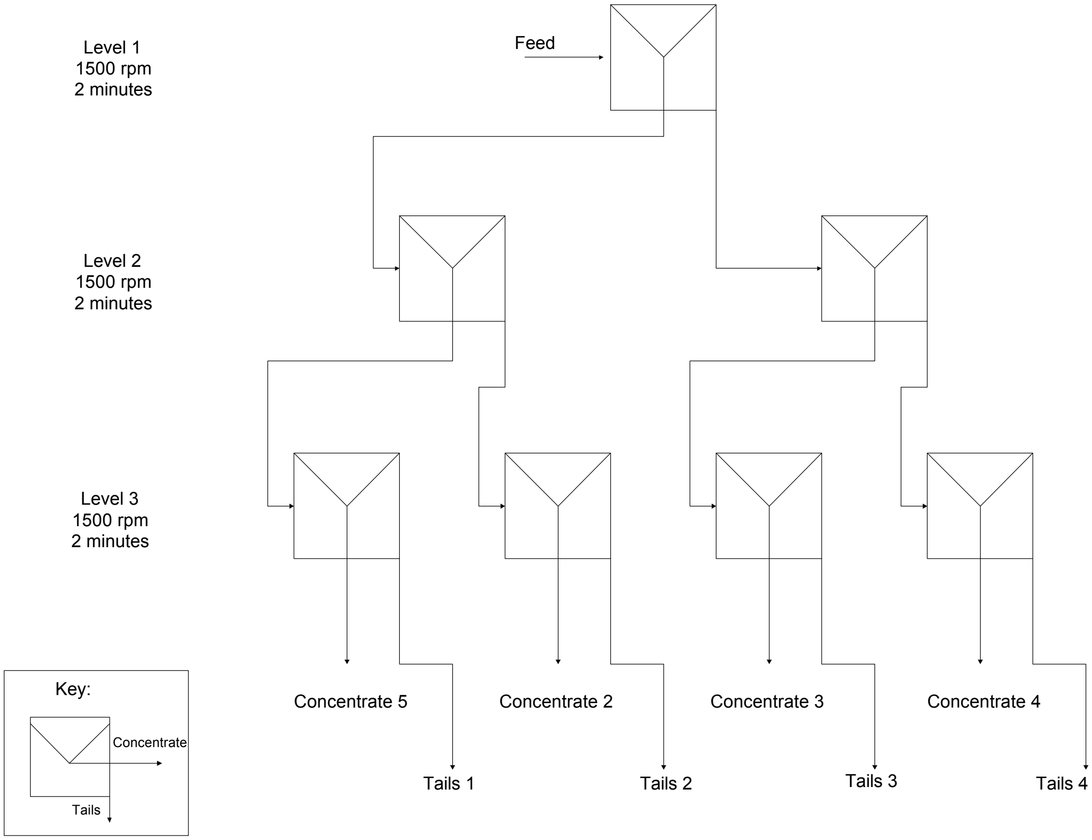

Figure 4. Schematic diagram of the tree analysis procedure.

Tree analysis is probably the simplest of the release analysis procedures in concept but it is also the most tedious since it may require anywhere from 16-32 fractions (i.e. 4-5 levels) to span the appropriate grade-recovery range. In theory, the tree procedure should provide the best separation since both the tailings and concentrate are refloated numerous times to ensure that all of the material is properly fractionated according to grade.

One of the most recent forms of release analysis is a column-based procedure described by McClintock, Walsh and Rao (1995) which uses a flotation column in conjunction with a slightly modified version of Dell's simplified release analysis procedure. The procedure uses a laboratory column with feed rates, wash water flow rates, and aeration rates appropriate for the size of the column being used. In the specific application presented, a glass column with an inside diameter of 25 mm and a height of 2.4 m was used. The aeration rate ranged from 50-250 ml/min, wash water rate from 15-20 ml/min, and feed rate from 40-50 g/min. The column was operated in a continuous mode and feed was passed through the column to produce a concentrate and tailings fraction. After the initial concentrate was set aside, the tailings were pumped back through the column to produce a second concentrate. The procedure was repeated until the froth was barren. After this first stage of separation in which the floatable material was separated from the non-floatable material, the final tailings were set aside for analysis and the concentrates were combined for separation in the second stage of the experiment. To begin the second stage, the combined concentrates were pumped through the column and a low aeration rate was used to produce a high-grade fraction. Again the concentrate was saved for analysis and the tailings were pumped back through the column at a slightly higher aeration rate. The procedure was repeated at higher and higher aeration rates until the froth was barren. All of the resulting concentrates and the final tailings were then analyzed and the results used to construct the release curve.

The main difference between column-based release analysis and Dell's simplified release analysis technique is that the column-based procedure uses repeated flotation of the tailings in the first stage rather than the concentrate. This is possible because the wash water used in a flotation column prevents entrainment, so additional cleaning of the froth product is not necessary. Repeated flotation of the tailings also ensures that all floatable material reports to the second stage for fractionation.

In spite of the work that has been conducted to define and develop the various release analysis procedures, these techniques are by no means considered standards for characterizing flotation. In fact, it is still unclear as to which technique comes closest to representing the ideal flotation separation. It is also unclear as to whether the separation obtained by release analysis can be surpassed using a novel cell design. In a recent comparison of column flotation technologies, Honaker, Mohanty and Ho (1995) reported that on several occasions, the separation performance of the flotation columns exceeded the separation results obtained using Dell's simplified release analysis procedure. Furthermore, the Packed Column was found to give consistently better results under high-grade, low-recovery conditions than would be indicated by simplified release analysis. Clearly, additional work is needed in the effort to define a method of characterizing flotation performance that is comparable to float-sink analysis in gravity separation.

1.3 Objective

There are currently a number of techniques that have been proposed for characterizing the flotation behavior of a given material. All of these techniques have their own advantages and disadvantages, all are claimed to be comparable to float-sink analysis in gravity separation, and yet none have been proven to produce an ideal grade-vs.- recovery curve.

The objective of this work is to use mathematical modeling and experimental testing to compare various techniques used to determine optimum flotation response. The techniques to be compared in this study are based on the common forms of release analysis including: (i) timed release analysis, (ii) simplified release analysis, and (iii) tree analysis. The best procedure will be identified and alternative procedures will be proposed and discussed. It is ultimately hoped that this work will determine whether any of the current procedures is capable of producing an ideal grade-vs-recovery curve for a flotation separation.

1.4 Scope

The information in this thesis has been organized into six chapters. Chapter 1 deals with the literature review, background, and objectives of this investigation. Chapter 2 describes the procedures, both theoretical and experimental, used in conducting the simulated and experimental comparisons of the release analysis techniques. Chapter 3 presents the results from the simulated and experimental comparisons of the release analysis techniques, while chapter 4 describes alternative techniques that resulted from the experimental and theoretical comparisons. Finally, chapter 5 presents the major conclusions of this work and chapter 6 proposes possible future work which seems to logically follow from the findings reported here.

Link to Chapt. 2