ABSTRACT

We completed two related tasks relevant to the design of more cost effective coal flotation processes. Simulation of packed and staged flotation columns and the elementary flotation collection process were completed. A two phased experimental program was completed to the point where a pilot packed column guided by simulations in a process we call NUCOL appears capable of superior cleaning when compared to another interfacial cleaning processes ISGS aggregate flotation (ISGSAF) treating the same coal.

We completed a paper "Rate of Collection of Particles by Flotation." The most detailed analysis to date for the flotation rate constant expressed in terms of measurable parameters our analysis includes the effects of mixing, and colloidal forces. Available experimental data for induction times can be correlated using our result. In the future, it would be very desirable to incorporate the results of the rate analysis in our packed column design algorithm (also completed this year).

A 10 lb/hr packed flotation column pilot plant was redesigned, fabricated, shaken down and successfully operated to complete 12 flotation runs this year. We explored effects of pulp level, reflux, froth washing and two reagent systems. We found product grade and Btu recovery for an Indiana No. 5 coal (IBC 106) that were comparable or superior to ISGSAF continuous flotation unit (CFU) performance (Btu recovery (77%), ash rejection (72%) and pyrite rejection (71%)). More importantly, product grade at >60% Btu recovery was superior (2.8-3.0% ash and 0.45-0.6% pyrite). A 2% ash product (not obtainable by AF) was also achieved. Chemical doses, while higher than ISGSAF are not yet optimized. These results on product pyrite and ash suggest that our packed column process technology developed to date may be able to approach cleaning limits dictated by liberation.

There is need to further demonstrate our packed column flotation technology, NUCOL. The experimental program should be extended to determine limits and sensitivity of system response as indicated by simulation. Recent results suggest considerably more economic reagent doses may be possible and should be examined along with other measures for cost effective and robust operation. Simulations should be extended to simplify design and aid in operation and control before further scale up.

We believe our approach could be made sufficiently generic to describe most column designs recently introduced.
EXECUTIVE SUMMARY

We proposed two tasks relevant to the design of flotation processes for improved separation of pyrite and ash from Illinois coals.

TASK 1: A NEW APPROACH TO THE DESIGN OF FLOTATION COLUMNS

Our approach here was to develop both the macroscopic design of flotation columns as well as a new microscopic approach for estimating the flotation rate constants required by these flotation column designs.

Staged flotation columns

We have completed a paper describing the design of staged flotation columns (Idlas et al. 1989a submitted for publication). Our analysis provides a means to estimate quantitatively the effects on grade and recovery and sensitivity of the several dominant control variables. A primary conclusion is that product grade can be improved by using reflux, returning a portion of the clean coal product, to the top of the column. This provides additional opportunities for filling the liquid-gas interface with desired product and displacing undesired species. This has not been previously suggested in the context of column flotation, although it is common practice in the context of distillation.

However, there is a penalty. Product recovery decreases as the reflux ratio and product grade are increased. Product recovery can be increased by increasing the height of the column below the feed. As might be expected, both operating costs and capital costs must be increased, in order to improve the product grade while maintaining constant product recovery.

Packed flotation columns

We have also completed a paper describing the design of packed flotation columns (Idlas et al. 1989b submitted for publication). Our primary conclusion here is again that product grade can be improved by using reflux. The reason for this improvement is the same as that described above.

As with the staged column above, there is a penalty to achieving the highest product grade. If all other operating variables are held fixed, product recovery decreases as the reflux ratio and product grade are increased. In order to maintain the product recovery constant as the product grade increases, one must solve an optimization problem in which both the excess interfacial area available for flotation and the packing height below the feed may be increased. As one would expect, both operating costs and capital costs must be increased, in order to improve the product grade while maintaining the product recovery constant.

Rate of collection of particles by flotation

We have completed a paper which gives the most detailed analysis to date for the flotation rate constant expressed in terms of measurable parameters (Li et al. 1989 submitted for publication). It includes the effects of macroscopic fluid mixing but, most importantly that of both London-van der Waals and electrostatic double layer forces on the coalescence rate process. Several conclusions result from this analysis.

1) The flotation rate constant increases as bubble size decreases. This is supported by experimental observations in the literature.

2) There may be a maximum in the flotation rate constant as a function of the particle size. This appears to be consistent with experimental observations in the literature.

3) There may be a maximum in the flotation rate constant as a function of the intensity of turbulence. This also appears to be consistent with experimental observations in the literature.

4) When the electrostatic forces are repulsive, the flotation rate constant decreases as the electrostatic surface potentials increase. This has been observed experimentally.
5) Available experimental data for induction times can be correlated using our result - Appendix A-Slattery and FitzPatrick (1989).

We would like to incorporate the results of this analysis in our packed column design algorithm (Idlas et al. 1989b). We then would hope to use this information as a basis for interpreting our own studies of induction time designed to optimize our choice of reagent systems.

**TASK 2: EXPERIMENTAL STUDY OF PACKED FLOTATION COLUMNS**

Our second objective was to construct and operate a small scale packed flotation column. This is necessary for two reasons. First, the design algorithm for a packed column must be based upon experimental data from an operating column. Secondly, even with a sound design basis outlined in task I, aspects of column operation are not captured by the simulation at this time and need to be addressed experimentally.

*Phase I experimental program: design and construction*

Experiments were planned to provide data to test major features of the packed column design algorithm and provide necessary insight into other important details of design, operation and scale-up. The column consists of a reagent dosing and preconditioning circuit and a four inch diameter column with washing and reflux appurtenances. Ancillary processes include rod and attrition mills, tailings treatment, and a clarifier for waste streams. It should be pointed out that this is not a bench cell, but a full continuous pilot plant operation. For reference, the NUCOL pilot column system is one-third to one-half of the ISGS aggregate flotation pilot plant and was completed between summer 1988 and spring 1989.

Phase 0 experiments in 1988 with an alpha version prototype packed flotation column demonstrated operational feasibility but also pointed out some significant shortcomings of the initial design. Phase I consisted in the redesign and reconfiguration of the 1988 (alpha) version of the pilot plant. Feeding, sampling and control equipment were significantly changed in constructing the Beta version in fall/winter 1988/89.

Experimental progress was initially slow this year for two reasons: 1) a key research engineer (Stan Zagula) left the University in the first project quarter and 2) long-term availability of laboratory space to set up the intended pilot operation was uncertain. The major and total renovation of The Technological Institute was scheduled to displace Northwestern University's Coal Research Lab as it had twice before in the last three years.

*Phase II experimental program: shakedown and testing*

Our second (Beta version) prototype (10 lb/hr) packed flotation column was made operational over the second and third project quarters this year. Shake down was accomplished in the second quarter and resulted in change in the reflux and washing equipment and transfer tank sizes and mixing and tail flow slurry flow control. By third quarter we began successful operation and completed 12 runs in the fourth quarter. At the end of the project we have planned a Gamma version of the prototype.

Experimentally, we explored effects of two reagent systems and operating conditions including reflux and froth washing. In our first three runs with IBC 106 coal, our primary concern was to identify and solve operational problems. Initially, we used an anionic surfactant frother and kerosene collector reagent system. Subsequently, we also analyzed alcohol and kerosene reagent combinations. Further, significant reagent optimization should be done.

We have produced superior grade recovery data for IBC 106 (77% BTU recovery at rejection of pyrite and ash of 72% and 71%, respectively) This is better than other continuous flow unit published results for this coal (Lytle, et. al. 1989, Final Technical Report to CRSC). Results were obtained with an unoptimized operating and reagent system and could be improved further. Our simulations show that product recovery decreases as the reflux ratio and desired product grade are increased. Product recovery could be increased beyond levels reported here by increasing the scavenging section of the column; e.g. Ken-Flote utilized about three times the length of columns utilized here. We regret that without more time and resources, use of longer columns and other optimization steps could not be undertaken in the grant period. Nevertheless, pyrite and ash rejection are particularly encouraging, suggesting NUCOL may be able to produce very clean coals that approach closer to the limit of liberation than current mechanical / pneumatic cell technologies.
Our simulations and their guide of experimental accomplishments suggest the attractiveness of our packed column approach and the need to further optimize the system. A more cost effective reagent package, along with other improved operating conditions and column characteristics should be pursued with IBC 106 and other coals to evaluate / demonstrate / develop the technology before larger scale - up. Furthermore, operation stability and freedom from long term operational problems such as fouling / plugging should also be shown.

Overall, it is little short of remarkable that with our limited resources and significant personnel changes we still redesigned, constructed, shook down and successfully operated a rather flexible pilot plant system in one years time. Our progress might be compared to development of Aggregate Flotation, where although conventional subaeration cell technology was used; four years were required from time of conception of pilot plant (summer 1984) through design, construction, shake down and successful consistent operation (summer 1988). We achieved a major fraction of the experimental development path with NUCOL in 18 months with the major leg accomplished in 12 months.

The remainder of this report contains proprietary information and as such is only available to the project sponsor.
OBJECTIVES

The objective of this project is to improve our fundamental understanding of flotation processes, in order that they may be designed and operated to achieve maximum cleaning of fine and ultrafine Illinois coals. We have sought both to enhance our understanding and modeling of governing mechanisms and to take advantage of proven concepts for the design of open, packed, and staged columns.

This project consists of two related tasks that are relevant to the design of flotation machines for the separation of coal from undesirable minerals.

TASK 1: A NEW APPROACH TO THE DESIGN OF FLOTATION COLUMNS

Our first objective was to extend the established methods for the design of foam separation columns and of distillation columns to the design, scale-up, and control of open, packed, and staged flotation columns.

TASK 2: EXPERIMENTAL STUDY OF PACKED FLOTATION COLUMNS

Our second objective has been to experimentally study a small scale packed flotation column. Our design algorithm for a packed column requires experimental data from an operating column. We could find no data for packed flotation columns in the open literature that are sufficiently complete for this purpose.

INTRODUCTION AND BACKGROUND

TASK 1: A NEW APPROACH TO THE DESIGN OF FLOTATION COLUMNS

Rate of collection of particles by flotation

Froth flotation is a process whereby particles are captured selectively from a suspension by gas bubbles. The capture of a particle by a bubble can be thought of as occurring in a series of stages: bubble-particle approach, thinning and rupture of the liquid film between them, and formation of a stable particle-bubble aggregate.

Promotion of the attachment of desired mineral particles to gas bubbles and prevention of attachment of undesired mineral particles are the most fundamental requirements for successful flotation. When as the result of mixing a solid particle is brought into near contact with an air bubble for a sufficiently long period of time, a thin liquid film forms between them and begins to drain (Schulze, 1984). The thin film is not bounded by parallel planes. As a drop or bubble approaches an interface, it develops a dimple: the film is thicker at its center than at its rim (Derjaguin and Kussakov, 1939; Allan et al., 1961; Platikanov, 1964; Hartland, 1967, 1969; Hodgson and Woods, 1969; Hartland and Woods, 1973; Burrill and Woods, 1973). As the thickness of the draining film becomes sufficiently small (about 1000 angstroms), the effects of the disjoining pressure attributable to the London-van der Waals forces and to any electrostatic double layer become significant. The liquid film drains until coalescence occurs, and the particle is attached to the bubble.

We usually refer to the time required for the thinning and rupture of the liquid film between particle and bubble as the induction time. The induction time depends not only upon the disjoining pressure, but also upon many other variables including particle size, the bubble size, the surface tension, and the viscosity of the continuous phase. Particles having a sufficiently short induction time will be captured, and these are said to have a higher selectivity. Experimentally, there have been attempts to characterize the induction time by measuring the particles of different species captured by an air bubble on the tip of a capillary tube that is forced against a bed of particles for a short period of time (Laskowski and Iskra 1970, Schulze 1984, Yordan and Yoon 1985, Yoon and Luttrell 1985, Ye and Miller 1988). From the viewpoint of theory, Ralston (1983) and Schulze (1984) have pointed out that an analysis of induction time which assumes a plane-parallel film misses the strong influence of film dimpling upon film drainage.
Staged and packed flotation columns

The recent round-robin test conducted by the Department of Energy (Kilimeyer and Hucko, 1989; Hucko et al., 1988) suggests that columns have an inherent advantage over standard mechanical/pneumatic flotation machines in cleaning coal. The overall best performer at this point appears to be Static Tube Flotation (a packed flotation column), with columns in general always outperforming cell systems.

Three types of columns have been suggested for use in flotation: open, packed, and staged (Kawatara and Eisele, 1987).

Open flotation columns with counter-current washing of the foam generated are currently being used for refining a variety of minerals (Nicol et al., 1988; Subramanian et al., 1988; Egan et al., 1988; Moon and Sirois, 1988; Ynchausti et al., 1988; Wheeler, 1988). They are reported to be more efficient than the traditional agitated vessel (Luttrell et al., 1988; Kawatara and Eisele, 1988; Misra and Harris, 1988; Nicol et al., 1988). Backmixing (axial dispersion) can be a problem with these columns. This has been specifically addressed in the analyses of Dobbs and Finch (1986), of Luttrell et al. (1988), and of Sastry and Lofftus (1988).

Until recently, coal and other minerals were typically ground too coarsely to be used in packed or staged columns without serious plugging problems. Particularly in the case of coal, where there is an increased emphasis on the physical removal of as much of the pyrites as possible, this limitation appears to have been removed by grinding to 90% < 200 or 400 mesh, e.g.

A staged flotation column has been proposed (Dell and Jenkins, 1976; Degner and Sabey, 1988). In commercial-scale tests, the WEMCO-Leeds column was reported to be more efficient than standard cells (Degner and Sabey, 1988). Scale-up of the number of stages to increase product purity and product recovery has been discussed as part of this project (Idias et al., 1989a). Scale-up of the column diameter to increase throughput has not been addressed.

The addition of packing reduces the effects of backmixing seen in open columns. Packed flotation columns have been reported by Yang (1984, 1987, 1988) to achieve the same separation for which eight traditional stages were required. Scale-up of the column height to increase product purity and product recovery has been previously addressed as part of this project (Idias et al., 1989b). Scale-up of the column diameter to increase throughput was considered to be direct by Yang (1988), because residence times could be estimated to be nearly those expected in a plug flow.

An overall summary of some relevant aspects of column fabrication and analysis are summarized on Table 1.

PROCEDURES

Our approach to the two tasks employed theoretical and experimental procedures outlined below.

TASK 1: A NEW APPROACH TO THE DESIGN OF FLOTATION COLUMNS

Our approach to the design of an open, packed, or staged column has been to develop simulations for their operation that can be used to scale up the process or to optimize the operating conditions. These simulations (Idias et al., 1989a,b; see also Appendices B and C-Slattery and FitzPatrick (1989) involve parameters that can be determined only by studying an operating column. Unfortunately, there are no data currently available in the literature for packed columns that are sufficiently complete for our purposes.

TASK 2: EXPERIMENTAL STUDY OF PACKED FLOTATION COLUMNS

In order to test our design algorithm, we require data from an operating column. A continuous column with 10 lb/hr coal capacity was set up and shaken down and fully put into operation this year.

Pilot column development

Initial experiments in 1988 with a prototype packed flotation column were successful within their initial scope, but pointed up significant shortcomings of the first design; principally, systems for slurry feed and reflux and gas introduction needed greater flexibility and dynamic range and we appeared to need a larger pilot plant area. The latter problem was overcome in the New pilot plant constructed in 1988 - 89 by using a Uni Strut™ support system
giving a vertical structure much like an actual gravity fed preparation plant facility. The unit schematized in Figure 1 operates in a limited laboratory or pilot plant floor space and could be modularized for transport to a field site in the future.

**Pilot plant elements**

The pilot plant layout shown in Figure 2 consists of a reagent dosing and preconditioning circuit with capability of adding frother, collector, suppressants and modifying agents. Multiple reagent addition and sampling points are provided. The main column is four inch inside diameter with tailing discharge eductor, pulp level controller and froth washing and reflux appurtenances. Foam breaker and reflux and wash water tanks are also included. Ancillary feed slurry preparation processes include a batch rod ball mill and semi-continuous stirred ball mill and a multistage slurry prep tank. Tailings treatment includes a dual polymer feed to the discharge from an intermittently operated sump which then passes to final drain through an upflow sludge blanket clarifier. Thickened solids are intermittently bled off to pressure filter dewatering for other project use or disposal. Concentrate and tails are analyzed from timed composites and feed from grabs. Pumping rates are monitored within 5% and have proven to be usually within 2%.

The physical design of the packed flotation column is somewhat different from that described in (Slattery and FitzPatrick, 1988). A total of six pumps and up to four dynamic and four static tanks and necessary mixers are used to prepare (condition), transport, and control slurry and washwater through the packed column system. Calibrated Masterflex pumps with digital flow meters regulate and monitor flows in the conditioning and feed loops, and tails, the reflux, the rinse water, and wash water stream. Stripping and enriching sections of fixed length were used initially. Proprietary cleanable packing material and proprietary feed and reflux distribution devices are used.

### Table 1. Construction, Analysis and Performance Attributes of Various Flotation Equipment

<table>
<thead>
<tr>
<th>TYPE</th>
<th>CONSTRUCTION</th>
<th>ANALYSIS &amp; PERFORMANCE</th>
</tr>
</thead>
</table>
| OPEN | • SIMPLE TO BUILD  
   • VARIOUS AIR INJECTION DEVICES APPEAR REQUIRED TO CUT DISPERSION | • ANALYSIS IS MOST DIFFICULT  
   • CONTROL MORE DIFFICULT THAN OTHERS  
   • POOREST GRADE/RECOVERY OF COLUMN  
   • SEVERAL EXAMPLES ARE COMMERCIAL |
| STAGED | • DIFFICULT STAGE CONSTRUCTION  
   • ONLY ONE DESIGN EVEN EXISTS | • ANALYSIS IS EASY COMPARED TO OTHERS  
   • CONTROL EASIER THAN OPEN COLUMN  
   • HIGH GRADE/RECOVERY ARE POSSIBLE  
   • FINE IS GRIND NECESSARY  
   • ONLY LIMITED DATA AVAILABLE  
   • NO TRUE EXAMPLE IS COMMERCIAL |
| PACKED | • SIMPLE PACKING DESIGNS AVAILABLE  
   • AIR INJECTION DESIGN LESS CRITICAL | • ANALYSIS IS SIMPLER THAN OPEN COLUMN  
   • CONTROL IS SIMPLER THAN OPEN COLUMN  
   • HIGHEST GRADE/RECOVERY OF COLUMNS  
   • RELATIVELY FINE GRIND IS NECESSARY  
   • BEST PERFORMER IN DOE ROUND ROBIN  
   • ONLY ONE DESIGN IS COMMERCIAL |
| CELLS | • SIMPLE  
   • COMMERCIAL CELLS  
   • ADVANCED FLOWSHEETS AVAILABLE | • ANALYSIS IS RELATIVELY EASY  
   • CONTROL DIFFICULTY IS MODEST  
   • CELL PERFORMANCE IS OFTEN NON-IDEAL  
   • OPTIMAL FLOWSHEETS MAY BE COMPLEX  
   • SHOWS LOWEST GRADE/RECOVERY  
   • THE STANDARD TECHNOLOGY AVAILABLE |
**Pilot column operating conditions and ranges**

Experiments were designed and carried out using IBC-106 coal to provide data to test the design algorithms outlined in Appendices B and C (Slattery and FitzPatrick, 1989). We further wished to evaluate other important details of design, operation and scale-up. Some ranges of design and operating variables and conditions for prototype laboratory column are given on Table 2.

<table>
<thead>
<tr>
<th>Table 2: Column design, operating and control variables and intended ranges</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed characteristics</td>
</tr>
<tr>
<td>size</td>
</tr>
<tr>
<td>grade (liberation of pyrite and ash)</td>
</tr>
<tr>
<td>Conditioning steps</td>
</tr>
<tr>
<td>dispersant addition</td>
</tr>
<tr>
<td>collector dose</td>
</tr>
<tr>
<td>conditioning time</td>
</tr>
<tr>
<td>Column operation</td>
</tr>
<tr>
<td>gas flow rate</td>
</tr>
<tr>
<td>feed slurry solids concentration</td>
</tr>
<tr>
<td>feed slurry flow rate</td>
</tr>
<tr>
<td>tailings flow rate</td>
</tr>
<tr>
<td>reflux flow rate</td>
</tr>
<tr>
<td>wash water flow rate</td>
</tr>
<tr>
<td>pulp level</td>
</tr>
<tr>
<td>foam height above packing</td>
</tr>
<tr>
<td>Packing design</td>
</tr>
<tr>
<td>column height (overall)</td>
</tr>
<tr>
<td>packing</td>
</tr>
<tr>
<td>non clogging elements</td>
</tr>
</tbody>
</table>

**Feed characteristics and reagents**

An IBC 106 sample of 700 lbs was riffled, roll crushed to 14 mesh and stored in 3.5 kg quantities under Argon at NUCRL. Samples of minus 100 M rod milled and minus 270 M attrition ground coal were sent to ISGS for petrographic analysis; results from selected runs are also since sent for analysis, since their levels of ash and pyrite (mentioned later) may approach limits of liberation.

We adapted our attrition mill to do semi continuous grinds in parallel with rod mill operation, and achieved our goal of cutting pilot plant manpower needs to levels quite below ISGS AFCFU.

Since our simulation algorithm suggests how to operate a single column of fixed configuration to achieve best results, this was tested first. Feed characteristics, were not optimized with regrind circuits, etc. With added resources we could examine liberation effects in combined two stage circuits (columns in series) with regrind, but did not feel this was warranted initially. Our first goal was not to produce a superclean product as Yang (1989) attempted.

Read et. al. (1988) and Lytle, et. al. (1989) spent considerable time examining reagent packages for Aggregate Flotation. This provided valuable guidance in selecting initial reagent packages to independently control ash, pyrite and coal flotability. Initially, frother only and frother - collector combinations are employed. An APT 100 induction time measuring device was used to further screen frother doses. Alcohol frotthers give less stable froth compared to charged surfactants. Both emulsified and unemulsified collector were used.
Data reduction and analyses

A series of continuous flow tests were done to extract measured coal (BTU) recovery and grade (pyritic sulfur and ash) under independent variation of solids flow rates, reagent doses and column liquid flow rate and level control. Feeds, concentrates and tails were analyzed. For moisture ash and sulfur forms and BTU. Component mass balances are obtained by hand calculation using METABAL code.

RESULTS AND DISCUSSION

TASK 1: A NEW APPROACH TO THE DESIGN OF FLOTATION COLUMNS

Our approach here has been to develop both the macroscopic design of flotation columns as well as a new microscopically based approach for estimating the flotation rate constants required by these flotation column designs.

Staged flotation columns

We have completed a paper describing the design of staged flotation columns (Idlas et al. 1989a).

The primary conclusion is that product grade can be improved by using reflux. This provides additional opportunities for filling the liquid-gas interface with the desired product and for displacing any undesired species by competition. This does not appear to have been previously suggested in the context of flotation, although it is common practice in the context of distillation, liquid-liquid extraction, and gas adsorption (Coulson et al., 1978; Geankoplis, 1978; Sherwood et al., 1975; Treybal, 1980).

However, there is a penalty. If all other operating variables are held fixed, product recovery decreases as the reflux ratio and product grade are increased. Increasing the number of stages while maintaining the height of the column constant can help, only if there is a mechanism for scavenging gangue particles from the froth as it passes from one stage to the next (Degner and Sabey, 1988). More generally, one must solve an optimization problem in which both the excess interfacial area available for flotation and the height of the column below the feed may be increased. As might be expected, both operating costs and capital costs must be increased, in order to improve the product grade while maintaining the product recovery constant.

Packed flotation columns

We have also completed a paper describing the design of packed flotation columns (Idlas et al. 1989b).

Our primary conclusion here again is that product grade can be improved by using reflux. The reason for this improvement is the same as that described above.

However, there is a penalty. If all other operating variables are held fixed, product recovery decreases as the reflux ratio and product grade are increased. In order to maintain the product recovery constant as the product grade increases, one must solve an optimization problem in which both the excess interfacial area available for flotation and the packing height below the feed may be increased. As one would expect, both operating costs and capital costs must be increased, in order to improve the product grade while maintaining the product recovery constant.

Rate of collection of particles by flotation

We have completed a paper which gives the most detailed analysis to date for the flotation rate constant expressed in terms of measurable parameters (Li et al. 1989). It includes the effects of both London- van der Waals forces and of electrostatic double layer forces. Several conclusions have resulted from this analysis.

1) The flotation rate constant increases as bubble size decreases. This is supported by experimental observations in the literature.

2) There may be a maximum in the flotation rate constant as a function of the particle size. This appears to be consistent with experimental observations in the literature.

3) There may be a maximum in the flotation rate constant as a function of the intensity of turbulence. This appears to be consistent with experimental observations in the literature.
4) When the electrostatic forces are repulsive, the flotation rate constant decreases as the electrostatic surface potentials increase. This has been observed experimentally.

5) Available experimental data for induction times can be correlated using our result (see Appendix A, Slattery and FitzPatrick, 1989).

With added resources, we would hope to incorporate the results of this analysis in our packed column design algorithm (Idlas et al. 1989b). We also would hope to be able to use this analysis as a basis for interpreting induction times, designed to optimize choice of reagent systems.

**TASK 2: EXPERIMENTAL STUDY OF PACKED FLOTATION COLUMNS**

Experimental studies were planned and executed achieving several results.

a) Construction of all principal elements of the packed flotation column pilot plant was completed by beginning of fourth quarter.

b) Twelve runs using coal and two frother/collector reagent systems were conducted. Analysis of these runs are sufficiently complete to describe superior operation to other cell flotation with Illinois coals and performance similarities to Yang's Static Tube Flotation. There is still room to optimize our reagent system and column operation but this could not be completed within the grant period.

c) We are at a point to accelerate the development of the combined process we now call NUCOL. We have added several additional elements to the column design and operation that we consider innovative. These include algorithms and techniques for selection and use of coal specific reagents and doses required. More development work is required on this latter aspect.

d) We were able to operate our column in reflux mode with sufficient stability in several runs to determine that our simulations are qualitatively correct in several essential features. Operation of the column required extra manpower to makeup for an incomplete control system. Some minimal added control elements to achieve stabilizing control were requested for a grant renewal period.

e) We think added improvement in grade and recovery is still possible by manipulating air/pulp ratio, froth level and froth quality. A proprietary device to include this in a control system has been conceived but not constructed. More work is required on this as well.

*Pilot flotation runs*

Table 3 shows the principle independent variables and Table 4 the principle performance related dependent variables for the 12 column runs. Missing values in table 4 were not yet available from laboratory analysis at report time. Figures 3 and 4 summarize the results of NUCOL and ISGS AF using conventional grade, recovery plots. Principal dependent variables measured are concentrate and tail solid and liquid flow rates and solids compositions. These are converted to grade and recovery data using metallurgical balances. In table 4, the recovery, rejection values give a range of uncertainty of the results based on our technique at the time. Mass balances were checked to close within 10 - 15%. Runs 105 and 107 could only be closed to 25%. The second number weights the more accurately determined concentrate flow.

**Actual column conditions**

For actual runs, flow rate of air and liquid were kept constant along with the grind, still to be verified. Pulp densities were kept within the 2.5 - 4.5 % range and pulp height between 1.5 and 1.7 m while overall column height remained constant at 2.4 m. Conditions were not employed that would exceed the column carrying capacity. The reflux flow rates were varied such that reflux ratio between 0.6 and 0.9 resulted. Rinse water was used to move the slurry to the concentrate tank at dilution ratios of rinse water to concentrate of 0.3 - 0.8, and in some cases was zero. In early runs, this diluted the concentration of the reflux stream. To better understand the effect of reflux, equipment was modified to push the reflux pulp concentration to the highest value possible (run 112). This points up the value of guiding experiments by simulation results such that the number of tests are minimized and those
needed are designed around the dominant control (independent) variables. Collector (kerosene) doses not counting the first three runs were in the range of 0.3 to 2.2 kg/ton, with most 0.7 to 1. In most runs, an anionic surfactant frother system #68 was used at 1.8 - 3.5 kg/ton while a more common alcohol #62 was used in two runs at much lower doses 0.4 - 0.8 kg/ton.

Table 3a. Conditions for Pilot Column Runs 101 - 104

<table>
<thead>
<tr>
<th>RUN NUMBER</th>
<th>COAL TYPE</th>
<th>FEED SIZE (90% PASS MESH SIZE)</th>
<th>FEED % SOLIDS</th>
<th>FLOW RATE (LITERS/MIN.)</th>
<th>PULP DEPTH (CM)</th>
<th>AIR FLOW RATE (L/MIN)</th>
<th>WASH WATER FLOW (ML/MIN)</th>
<th>CONCENTRATE DILUTION RATIO</th>
<th>REFLUX RATIO</th>
</tr>
</thead>
<tbody>
<tr>
<td>101</td>
<td>IBC 106</td>
<td>270</td>
<td>2.6</td>
<td>1</td>
<td>80</td>
<td>2</td>
<td>150</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>102</td>
<td>IBC 106</td>
<td>270</td>
<td>2.4</td>
<td>1</td>
<td>80</td>
<td>2</td>
<td>350</td>
<td>0</td>
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<tr>
<td>103</td>
<td>IBC 106</td>
<td>270</td>
<td>2.4</td>
<td>1</td>
<td>80</td>
<td>2</td>
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Table 3b. CONTD. Runs 105 - 108

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<th>PULP DEPTH (CM)</th>
<th>AIR FLOW RATE (L/MIN)</th>
<th>WASH WATER FLOW (ML/MIN)</th>
<th>CONCENTRATE DILUTION RATIO</th>
<th>REFLUX RATIO</th>
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### Table 3c. CONTD. Runs 109 - 112

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### Table 4. Recovery, Grade Results for Pilot Column Runs 101 - 112

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<th>% Product Ash Ratio</th>
<th>% BTU Recovery Pyr S</th>
<th>% Rejection Pyrite Ash</th>
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<td>(68) 83 (67) 81</td>
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Figure 3a BTU Recovery vs. concentrate ash for ISGS CFU and NUCOL

Figure 3b BTU Recovery vs. Ash Rejection for ISGS CFU and NUCOL
**PRODUCT PYRITE GRADE %**

Figure 4a BTU Recovery vs. concentrate pyrite for ISGS CFU and NUCOL.

**PYRITE REJECTION %**

Figure 4b BTU Recovery vs. Concentrate Pyrite for ISGS CFU and NUCOL.
Discussion of column runs and performance

Highest recovery and best grade were obtained for a condition without reflux and relatively high surfactant dose; 77 % Btu was recovered and 72 and 71 % ash and pyrite were rejected, respectively and product grades of 3 % ash and 0.6 % pyrite were reached. Our simulations indicate that this should be the case, i.e. best recovery, but the results do not mean that this is an optimum, or best achievable grade and recovery with this coal and with our packed column. In fact product grades of 2 % ash and 0.45 % pyrite were obtained at Btu recovery of 46 and 58 %, conditions with reflux.

shakedown runs

The first three runs were part of our shakedown and showed serious overdosing with kerosene collector reagent which was not properly applied in the conditioning step. Recoveries were generally very low and product grades poor because of a rapidly draining froth and tailings insufficiently stable flow control.

overview of major runs

Starting with run 104, conditions of operation were stabilized and improved with a drop in collector from the early runs to the more reasonable range of around 2.5 lb/ton and resulted in product ash around 3% and pyrite 0.6%. Runs 105, 106 were designed to evaluate replication at higher pulp density than run 104 and with different coal batch age. Each run had 3.8% pulp density, similar reagent doses, pulp levels and other operating characteristics. Doses were dropped further in runs 105 and 106 such that a threshold was reached around the conditions shown of 3.2 kg/ton of frother. In run 106 where the reflux ratio was increased, the pulp level increased, reagent doses dropped and recovery dropped with a slight improvement in ash grade. Ash and pyrite rejection improved in this run over 105 because the feed pyrite assayed higher (discussed later).

Runs 107 and 108 operated with higher pulp level than 106. Runs 106 and 107 differed only in absence of collector in the latter and produced the expected result of lower Btu recovery but unexpected poorer ash and pyrite grade. Perhaps, the pulp level, was too high and froth level too shallow for good gangue rejection in run 107.

Run 108, 109 and 110 were operated with still lower frother doses, than 107 which may be about the lowest effective with surfactant #68. Run 108 has the highest kerosene dose and a relatively high pulp level. Ash and pyrite grades worsened here as might be expected with an overdose of collector.

We wished to test the hypothesis that frother #68 needed to be above 3.0 kg/ton to obtain good grade. Runs 109 and 110 were designed to look at more modest ranges in doses without reflux. Pulp levels were relatively high and collector doses were on the marginally low side. Run 109 and 110 show that doubling collector for a relatively low frother rate gives similar product ash but dramatic (almost twofold) increase in recovery. Run 109 showed one of the best ash rejections but not the best ash grades. Run 109 interestingly had an almost 80% ash rejection but a product grade of 4.9 whereas 112, with the 62 alcohol frother had an 88% ash rejection and product grade of 2.1 % ash.

In runs 111 and 112 major variables investigated were a different alcohol surfactant system and the effect of reflux with that system. By using an alcohol surfactant, system, the effect of charge and relative frother adsorption may be apparent such that a threshold concentration for the surfactant system could be identified. Run 111 and 112 showed the expected result that by adding reflux, recovery drops (from 54 to 46 % and grade improves (from 3.0 % to 2.1 % ash).

Discussion of major effects in column runs

liberation and limits of cleanability

Runs 105 and 106 shed insight on both liberation and cleanability. The good recovery, and excellent product grade (mean values were all identical) for runs 105 and 106 suggest that replication with the pilot plant is quite good, even with different feeds at two different points in time. Since feed pyrite grade differ (0.83 in 105 and 1.5 in 106) but product grades do not, the difference in rejected pyrite percent corresponds to the coarse pyrite missing in run 105. The lower feed pyrite grade in 105 most likely reflects a transfer tank problem whereby settling loss of coarser pyrite occurred. The removed pyrite likely has the identical size (liberation) consist in both cases. We have been unable to verify this hypotheses, since particle size and petrography are not done yet. Feed pyrite grades are essentially
constant for all other runs including 106 - 108 at 1.5 %, slightly lower than the 1.85% reported Kruse, et. al. (1989) for IBC 106. Feed ash grade is also essentially constant at about 8.4 %, slightly lower than the reported 9.0% (Kruse, et. al.,1989).

Product grade for runs 105, 106 and 107 deserve further discussion. Both concentrate ash and pyrite in run 106 are essentially identical suggesting a limiting concentrate grade for both ash and pyrite for the chosen degree of liberation and reagent system. Ash and pyrite rejection are about 82 % for run 106, but only 77 % and 67 % respectively for run 105. By eliminating the kerosene in run 107 while maintaining all other variables recovery dropped dramatically to 33 % as expected; however, both ash and pyrite grades worsened and rejections remained constant at about 83 %.

Grades are puzzling but high rejection is not since with low recovery, by definition everything else appears as rejected. Lytle et. al. (1989) claims that at the finer liberation of IBC 106, ash somehow reabsorbs to coal surfaces and ends up enriched in the product. While run 107 results are not expected in error they have not been duplicated.

Dose effects

Runs 104, 108 and 109 - 111 had no reflux and thus can be directly compared. Run 108 had twice the collector and only one third the frother of run 104. Prior to selecting final dose for 108, a dose only 20 % higher than for run 104 resulted in essentially zero recovery. The froth suggested collector failure. Cutting back to 1.7 kg/ton was necessary to get a proper froth.

Run 108 was designed to show effects of high frother and collector dose. Initial ionic surfactant dose was so high that the coal was stabilized and not collected. By successively cutting the dose, a stable rich froth was produced. However, the collector dose was not sufficiently and proportionately reduced, resulting in a probable overdose. While kerosene was excessive, relatively modest frother at 1.7 kg/ton was almost adequate; the combination still did not bring the high recovery and grade seen in run 104. Recovery decreased to 67 % and ash and pyrite rejection dropped dramatically to about 54%. The froth was rich but selectivity was lost as the extra collector floated most of the ash and pyrite. This kerosene dose, the highest used to date, is too high and not the correct strategy.

Run 109 had a rather low collector dose by comparison to 108, but about the same frother. Recovery dropped suggesting that either the collector was required or insufficiently stable froth was produced to transmit high product recovery. Note that run 107 had similar grade and recovery, but at about half the feed solids and twice the frother. From these results, it appears that the collector dose necessary to obtain selectivity and a stable froth is at least above 0.5 kg/ton. Since reagent addition affects froth quality / stability as well as the k ratio, our system needs to be guided with added measures to separate out these effects.

In run 110, more stable operation was achieved at 2.5 % solids. Now, with the same frother dose, increasing collector brought recovery as high as run 104 but only with a 62 % ash rejection. The conclusion for this run without reflux is that collector at 0.9 kg/ton is high enough to achieve almost 80% Btu recovery but frother / collector balance is needed to obtain better ash rejection. Comparing runs 104, 108 and 109 suggest that the charged surfactant 68 may require a relatively high dose of over 3 kg/ton to get high (>66%) ash rejection.

Runs 111 and 112 define a preliminary way how an alcohol surfactant behaves with and without reflux. Basically we used rather low doses of both frother and collector, ~ 0.5 lb/ton collector and 0.75 - 1.5 lb/ton frother. Our simulations predict grade increase with reflux with an attendant drop in Btu recovery. For no reflux, 54 % Btu is recovered at 3 % ash grade and with reflux, 46 % recovery at 2.1 % ash grade is realized. This latter is the lowest ash measured for this coal at this high a recovery.

Reflex effects

In each case with reflux, the simulation predicted trends are observed. Adding reflux in runs 105 or 106 compared to 104 drops recovery by 20 % and improves product ash and pyrite grade. The latter, perhaps limited by liberation, only improves 3 - 6 % but the former by 25 %. For run 111 and 112, reflux cuts Btu recovery by 15 % and improves product ash grade by one third to 2.1 % ash, the lowest ash measured for this coal at this high (46%) Btu recovery. In run 106 compared to 105 where the reflux ratio was increased slightly, recovery dropped with a slight improvement in ash grade. Even in the shakedown runs 102 and 103 where doses are identical, reflux of 0.6 cuts the recovery by 33 % and improves product ash grade by 10 %. What we would like to and need to show next is how to move on the optimum grade recovery curve with reflux.
Comparison of NUCOL and ISGS AF

Figure 3a shows product Btu recovery vs. product ash and a clear demarcation for best values for NUCOL and the cluster for ISGSAF CFU (Lytle, et al., 1989). For these best values, the recovery increases with reagent dose and best grade is obtained with reagent system 62, K. The two data with poorer performance than the CFU were runs 108 and 109. Both cases may be underdosed with frother. In run 108 kerosene dose was too high and in 109 too low for effective system operation from any standpoint. The intermediate values with 4.1 and 4.4 % ash fall within normal CFU range. The lowest recovery of the best data on Figure 3a were obtained with a noticeably lower doses of the 62, K reagent system.

Replot of these data in Figure 3b in terms of Btu recovery vs. ash rejection shows again a clustering of the data about the CFU results, Two data have higher grade recovery than CFU, but most are lower. The two better values correspond to run 112 (62,K reagent system, 46 % recovery at 2.1% grade) and run 104 (68, K reagent system, 77 % recovery at 3.0 % grade). However, to achieve the superior grade recovery of run 104 required 6 times the frother and 3.5 times the collector of run 112.

Product pyrite is shown next in Figure 4a. This shows a clear clustering of values for AF and NUCOL, with the latter having noticeably superior grade quality below 0.6% pyrite grade (at recovery of 58 - 78 %). The two cases where the pyrite grades are poorest (above 0.95 %) toward the upper limit of AF, correspond to runs 107 and 108, respectively, which (beyond run 103) also have the lowest and highest collector doses, respectively. Without collector, recovery is poor; with highest collector, Btu recovery is 67 % but everything is floating.

When plotted in Figure 4b, results show some different trends: the single NUCOL case superior to AF is run 104; the same was true for ash rejection. Run 106 performance meets the central trend of the AF CFU line. Reagent system 68, K frother and collector doses were respectively, 4.8 kg/ton + 1.1 kg/ton and 3.2 kg/ton + 0.7 kg/ton for runs 104 and 106. Relatively high doses were required for grade/recovery that has not yet been optimized.

We conclude that with 12 tests we have been able to demonstrate in comparison with AFCFU several operating conditions where NUCOL produces superior product ash and pyrite grade at comparable Btu recovery. Furthermore, two operating conditions have been identified showing better ash and pyrite rejection, Btu recovery characteristics than AF.

The recent round-robin test conducted by the Department of Energy (Killmeyer and Hucko, 1989; Hucko et al., 1988) indicated that several columns outperformed cell systems in cleaning Pittsburgh and Upper Kittanning seam coals. As we and others have already pointed out, columns have an inherent advantage over standard mechanical/pneumatic flotation machines in cleaning coal. The overall best performer in the round robin appeared to be a packed flotation column. Unfortunately, a design and operating algorithm has not been available until now to explain and further develop and optimize this promising cleaning technology.

Future development

As it turns out we have just enough archived sample of each of the round robin coals to do a test if we knew the optimal reagent dose. Our sample is insufficient to determine the optimum at this time. With an extension of this study, we could demonstrate a small scale test for optimal reagent dose selection and use it to demonstrate superior column performance for several coals. An experimental program should continue along lines of reducing reagent costs while trying to achieve 90 % Btu recovery and 80 to 90% pyrite rejection even with difficult to float coals like IBC 106. It should be appreciated that we have by no means optimized our column operation at this point in time. However, compared to other CRSC pilot plant developments, Northwestern University's record of getting sophisticated coal cleaning pilot plants up and running (FitzPatrick, et al, 1987), in a short period is remarkable. We have accomplished more than an adequate experimental program this year. The packed column technology conceived at NU should be given further developmental support.
CONCLUSIONS AND RECOMMENDATIONS

We have reached several conclusions and have attendant recommendations.

a) Construction of all principal elements of a beta version of the packed flotation column pilot plant was completed. Significant redesign of some elements was required. Revision of our beta to a gamma version was planned and needs to be done.

b) Twelve runs using coal and two frother/collector reagent systems were conducted. Analysis of these runs although still preliminary is sufficiently complete to describe superior operation to other cell flotation with Illinois coals and performance similarities to Static Tube Flotation. We were able to clean IBC 106 coal (9% feed ash and 1.5 % pyritic sulfur) to levels higher than reported by physical interfacial cleaning in unoptimized systems. We find very high product grade (2.9 % ash) at 77 % Btu recovery and 2.1 % ash at 46 % Btu recovery. Pyrite grades of 0.6 % at 77 % Btu recovery and 0.45 % at 58 % Btu recovery were obtained. There is still considerable room to optimize our reagent system and column operation but this could not be completed within the grant period.

c) We are at a point to accelerate the development of the combined process we now call NUCOL. We have an algorithm that could extend the physical cleaning concept of NUCOL and have added several additional elements to the column design and operation that we consider innovative, including techniques for selection and use of coal specific reagents and doses required. More development work is required on using the simulation directly for specific run conditions and the reagent selection algorithm.

d) We were able to operate our column with sufficient stability in several runs to determine that our simulations are qualitatively correct in several essential features. Trends in the effect of reflux and chemical variables (type and dose) appear to agree with simulation trends and other empirical measures, but with some surprises. Added data are still needed with reflux and other control variables to compare predictions more quantitatively.

e) We think added improvement in grade and recovery is still possible by manipulating air/pulp ratio, froth level and froth quality. An innovative device to include this in a control system has been conceived but not constructed. Operation of the column required extra manpower to makeup for an incomplete control system. Some minimal added control elements to achieve stabilizing control were requested for a grant renewal period. These elements should be added to the gamma version and demonstrated before further scale up.

f) Perhaps, failure of ISGS aggregate flotation to advance more quickly was due to not getting started early enough with a more inherently superior cell technology. This is confirmed by the Homer City tests with WEMCO and BF-AM Bechtel column cells. Without adequate provision for innovations made possible by column technologies, aggregate flotation was unnecessarily handicapped, and ended up one of the poorer performers in the DOE round robin tests.

REFERENCES


Li, D. M.; FitzPatrick, J. A.; Slattery, J. C. "Rate of Particle Collection in Flotation". Currently being reviewed 1989.


