COAL FLOTATION WITH IBS/OIL FROTH

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INTRODUCTION

International coal markets are expected to grow in the near future [1]. U.S. has sufficient coal reserves that will last for centuries, but a major problem in coal utilization is sulfur. Fifty five to 80% of the sulfur in most coals is in the form of pyrite, and if this pyritic sulfur can be removed by advanced physical coal cleaning techniques, sulfur dioxide (SO_2) emissions in the U.S. could be cut by as much as 50% [2]. Presently, the alternative for the prevention of SO_2 emissions is post-combustion scrubbing, but is too costly and consumes as much as 5% of the power station's output (adding to CO_2 emissions). Therefore, incentives exists to explore pre-combustion cleaning. Coal has to be grounded to -325 mesh in order to liberate pyrite and other minerals from the organic components. At this fine particle size attractive forces between particles are much greater than the differences in the gravity forces exerted on the particles, and thus the gravity separation based conventional coal cleaning processes become inefficient [3].

Of the existing fine coal cleaning techniques froth flotation is most effective, but is inefficient in making good separation with ultrafine coal [4]. There is a wide range of chemicals that are required for coal preparation in these process, for improved process efficiencies and to meet stricter environmental standards [5]. This makes process control very complex. At ultrafine sizes, the small mass and momentum of clay particles, is the primary cause of their physical entrainment and transportation into the froth. This lowers the efficiency of the froth flotation process when excessive amounts of clay are present. The entrained ash can be washed from the froth by counter-current washing as in column flotation, but at the same time it may rupture the bubble and reduce the recovery.

Coal flotation by Intrinsic Bubble Separation has shown to circumvent surface phenomena which reduce the efficiencies of the conventional cleaning processes [3]. This process takes the advantage of the natural porosity of the organic fraction of the coal. In this approach bubbles are formed directly on the organic coal particles, eliminating the bubble-particle collision and attachment probabilities. The nonporous minerals do not form bubbles, assuring 100% bubble-particle contact. Thus, flotation can be highly selective. Since the bubbles form from the pore openings, this minimizes free bubble formation and enhances the cleaning of high ash coals.

Washability curves are the graphs showing the ash-density distribution. These curves are indicative of the maximum cleaning potential of any physical coal cleaning process for a particular coal sample. Washability curves were obtained for four coals from the Illinois Basin Coal Sample Program (IBC 101, 102, 104, and 106). Southern Illinois University at

Carbondale has developed a method for rapidly and accurately evaluating fine and ultrafine coal liberation using Density Gradient Centrifugation (DGC), Micro Sulfur and Thermogravimetric Analysis (TGA) methods to establish washability curves [3]. For the IBC 102 coal there was small difference in the liberation of minerals between -100 and -400 mesh material. The ash yields went through a minimum with increasing density, indicating that the inertinite and the liptinite macerals have higher inherent ash yields than the vitrinites. Washability curves for the IBC 101 and 106 coals were like the 102 Coal but had much higher ash yields which indicated that even at ultra-fine size there is substantial amount of unliberated minerals in these coals [6].Previous results on intrinsic bubble formation process showed that pressure, in general, increased the hydrophobicity of the organic portion of the coal. Except for Octanol, additives did not have any positive effect on the process. Lower pHs seem to favor the process. The process works effectively on weathered coals and pulp densities as high as 20% [2].

All the IBC coals at -32 mesh have been cleaned to their respective -100 mesh washability lines. Results from the high clay IBC 104 coal are the best thus far. For this coal substantial ash (> 90%) and sulfur rejection (> 80%)have been obtained. These results compare quite favorably with those of the Illinois State Geological Survey Aggregate Flotation process (Fig. 1) [6].

Recoveries and ash rejections with this process compared very favorably with other processes. Flotation kinetics are much faster. There were some problems with the wetting of coal at high pulp densities (> 20 wt.%) which were overcome by using a bigger mixing chamber and two 10,000 rpm mixing motors. This high mixing rate should help in breaking some of the mixed phase mineral/coal particles which caused problems in the early phases of this project.

EXPERIMENTAL PROCEDURES

The coals selected for use in this research were IBC 101, 102, 104 and 106 from the Illinois Basin Coal Sample Program (IBCSP) and a Herrin # 6 coal from the Monterey #2 mine located near St. Louis. The choice of these coals was made on the basis of their differing rank (reflectance), ash yields and pyrite/organic sulfur ratios (3). The particle sizes used for the IBC coal samples were -32, -100,-400 mesh and for the Monterey coal -400 mesh sample was used. The particle size distribution for -32 and -100 mesh were determined by wet sieving, and that for -400 mesh was performed using the Microtrack analyzer. Washability curves were plotted for -100 and -400 mesh samples. A schematic of the experimental setup used is shown in figure 2. Dried coal is fed into the lock hopper where it is pressurized using air. Water and the flotation media used are fed into the mixing chamber and are pressurized to the same pressure as the coal. Coal is then dumped in the mixing chamber, mixing starts simultaneously (two 10,000 rpm motors are used for mixing). A plug valve is used for slurry output at the bottom of the mixer and for depressurization control. The outlet pipe is immersed in a separation column with a water cushion. Coal slurry depressurizes through a nozzle in the separation column; float froth rises to the top and is collected in a trough attached to the separation column. The liquid left in the column is termed as the suspension and the solids settled at the bottom are called the sink. Recovery and ash values are determined on each of the collected fractions. Runs with good recovery and separation are subjected to additional analyses.

Initial runs, to study the effects of various conditions, were performed with IBC coals. Pressure variations were made ranging from 15 to 300 psig on the three mesh sizes of these coals. pH variations were made using NaOH and HCl. After the optimum pHs were determined, the effect of various gases (N_2, CO_2, and Air) on the process were studied. Runs were performed with various additives to assess their effect on recovery and separation. Also, the effect of mixing rate, mixing time, pulp density, and weathering (oxidation) on cleanability and recovery were studied [6].

Effects of additives (Octanol, Corn oil, Pentane, and various dispersants), pH (2.3 to 11.0) and pulp density on the Monterey coal were studied. These runs were performed at 60 psig and 5% pulp density (-400 mesh samples), in the new apparatus shown in Fig. : 2. Various loadings of additives were used to study the effect of the additive loadings.

RESULTS AND DISCUSSION

Ultrafine (8μm mean size) Monterey coal was subjected to sink float analysis by centrifuging at 34,000 rpm and specific gravity of 1.6. The float from this run had a recovery of 59 wt.% (5.4 wt.% ash) and the sink had a recovery of 41 wt.% (45.6 wt.% ash). This result showed that even at ultrafine size minerals are unliberated. Also, from the optical microscopy it is seen that there are a lot of mixed phase mineral/coal particles present at this fine particle size.

Increase in mixing rates and time have a positive effect on the process, though very high mixing time (> 30 min.) tend to wet the organic matter and caused a decrease in the recovery (Fig. : 3). With increased mixing rate the recoveries go up but the ash rejections tends to level off, this could be due to unliberated minerals or mixed phase mineral/coal particles. The problem of mixed phase is more evident at -400 mesh where the electrostatic charges are much stronger.

Additive loadings had mixed effect on the cleaning of ultrafine Monterey coal. With increased octanol loading there was a very small increase in the recovery but the ash yield in the float almost doubled (6.4 to 11.2 wt.%). Thus lower octanol loadings are favorable for the process. This in contradiction to results reported previously for -32 and -100 mesh fractions of IBC 104 coal and may be due to change in wetting angle, resulting in more particle bubble detachment in the case of ultrafine coal. Increased bubble detachment would be expected to entrain liberated ultrafine clay particles. In the case of larger particle size fractions the detached bubbles are not sufficient to float the larger sized clay particles.

Oil froth agglomerates generated with corn oil gave higher recovery and high ash yields, when the oil loading was increased there was a slight drop in recovery but the ash yields went down almost to half of that at lower loadings (7.4 from 12.5 wt.%). Since Ocatnol and corn oil had different effects on the process a run was performed with both octanol (at lower loading) and corn oil (at higher loading), this combination of additive gave a very high carbon recovery (> 90 wt.%) and an ash yield of 9.3 wt.%. Other additives had no positive effect on the process. In fact dispersants tend to give very low recovery. This is due to wetting of organic particles that results in less gas being trapped in the coal pores.

Effects of pH on the process were also studied. pH seems to depend on the additive. When corn oil was used higher pH (11.0) was optimum for the process and both the recovery and ash rejection increased with the increasing pH. Whereas, with pentane neutral pH favored the process and any change in pH resulted either in the lower recovery (with lower pH) or lower ash rejection (with an increase in pH). At higher pHs minerals were dispersed in the separation column(high clay content in the suspension), lower pH on the other hand tends to sink not only the minerals (clear suspension) but also the organic coal particles.

The process has been operated at pulp densities as high as 20 wt.%, after which wetting of coal and mineral entrainment in the froth becomes a problem. We think that this problem can be overcome when the process is scaled up to the pilot plant.

CONCLUSIONS

Fine and ultrafine coal cleaning by the IBS/Oil froths process is feasible. Coals have been cleaned to their washability limits by this process. Additive loadings have no systematic effect on the process and higher additive loadings work just as well as the lower additive loadings. Octanol gave very high ash rejections (> 90 wt.%), the carbon recoveries were depressed (50 wt.%). Corn oil/Octanol (neutral pH) and pentane at lower pH all cleaned Monterey coal to the washability limits. Organic particle flotation is almost instantaneous. Results thus far have shown that the process has much better recovery and ash rejection than the conventional flotation processes. Increased mixing rates have a positive effect on the efficiency. The process is limited by the unliberated minerals and especially by the interaction of coal and mineral particles. There is a need to find a dispersing agent that could break the phase of mineral/coal interactions without adversely affecting the wetting angle or a better grinding/liberation process is required. This would improve the effectiveness of the process for ultrafine coal cleaning.

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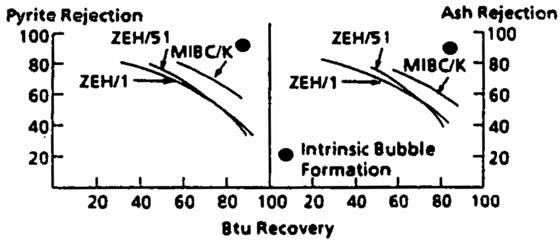

FIGURE : 1 Comparison of intrinsic bubble formation with aggregate flotation.

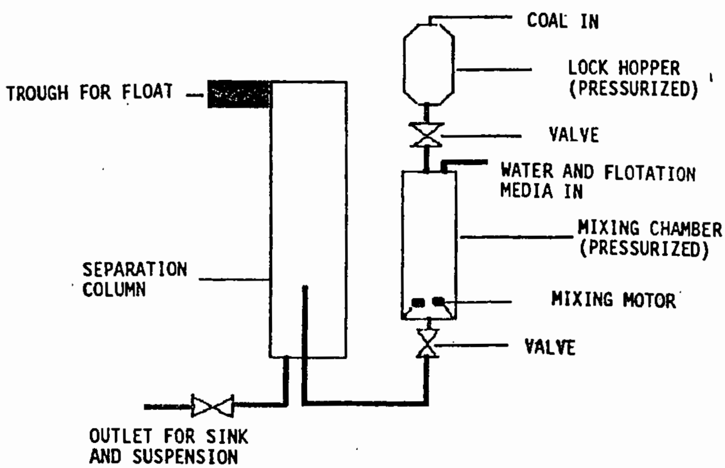

FIGURE : 2 SCHEMATIC OF FLOTATION APPARATUS

WASHABILITY CURVE FOR HERRIN #6 HIGH ASH COAL

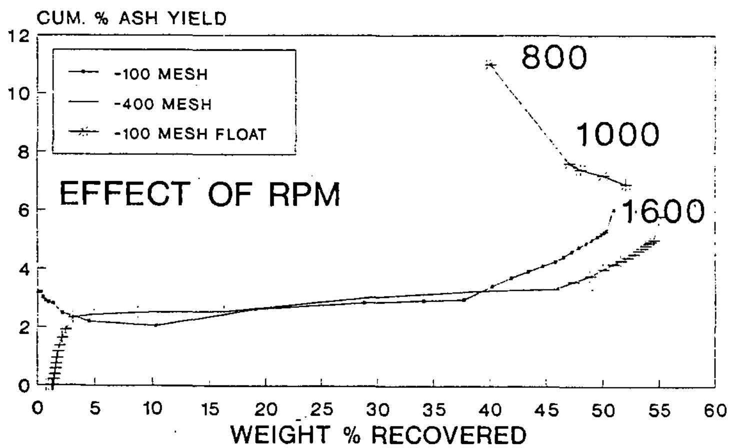

FIGURE : 3 EFFECT OF RPM ON THE RECOVERY AND ASH REJECTION OF IBC 104 (HERRIN # 6) COAL.