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ABSTRACT

Automated image analysis (AIA) of mineral association with the coal matrix was shown to be helpful in predicting froth flotation behavior of an Indiana No. 3 and an Upper Freeport coal when the association was expressed in terms of the relative amount of particle surface covered by coal and mineral phases. AIA measurements of the amount and quality of material with more than 80% of the particle surface covered by coal were found to relate to the pyrite and ash reduction observed in froth collected after 1.5 minutes of flotation. Results accurately predicted more ash reduction by froth flotation for the Indiana No. 3 coal than for the Upper Freeport coal. The association distribution derived from AIA also showed the changes in ash reduction as a function of cleaning severity.

In another task, analysis of the repeatability of coal-mineral matter association results for a single pellet of coal indicated that characterization of 3,000 particles should be adequate to provide $\pm 16\%$ relative repeatability for phases measured to be present at the 5% level. Comparison of results between two pellets indicated that the reproducibility of AIA results is about twice as large as the repeatability due to additional variation from instrumental and sampling effects. However, the distribution histograms for the coal-mineral matter association showed the same relative shape for both pellets analyzed, which indicates that analysis of 3,000 particles is adequate for describing the association of mineral phases with coal.

MICROCHARACTERIZATION OF COAL COMPONENTS FOR BENEFICIATION

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INTRODUCTION

Whereas physical coal cleaning processes utilize differences in the characteristics of coal and minerals to separate coal from mineral matter, and whereas coal and mineral matter are not completely separated from each other except at prohibitively small particle sizes, scanning electron microscopy-based automated image analysis (SEM-AIA) has been undergoing development at the Ames Laboratory for the past several years for predicting and evaluating the cleanability of coals. SEM-AIA has been used to measure the association of individual mineral grains with coal in both raw and processed coals. Results are presented in the form of the mass fraction of coal and mineral phases present at different levels of association. Coal-mineral matter association is measured and expressed in two ways: as the weight fraction of minerals present in coal particles, and as the fractional coverage of particle surface by mineral matter. The first format is suited to predicting cleanability for density-based processes, since particle mineral content can be easily related to particle density. The second format is suited to predicting cleanability for surface-based processes, such as froth flotation or oil agglomeration, where the amount of coal or mineral matter on the surface of a particle is related to the likelihood that a particle will appear in the clean coal stream.

Current efforts are directed at further defining the relationship between the fraction of mineral coverage of a particle's surface and the probability that it will be recovered in the clean coal stream. Another task is directed at establishing the repeatability of AIA results.

PROGRESS

Correlation of AIA and cleaning test results

Last quarter, results were reported (1) which indicated that AIA results for the distribution of the relative amounts of coal and mineral matter present on the surface of particles provided a general indication of

the behavior to be expected during froth flotation. AIA predictions of froth flotation behavior did not appear to be as good as AIA predictions of float-sink behavior. However, further examination and comparison of the AIA association results and froth flotation results has shown that AIA results may be able to predict ash and pyrite reduction for froth flotation as closely as it can predict float-sink behavior (2).

Samples of Indiana No. 3 and Upper Freeport coal were tested for float-sink and froth flotation behavior by AMAX Research and Development at different levels of cleaning severity (3). Test results for the float sample at 1.6 sp. gr. were selected for comparison with AIA predictions based on recovery of particles with less than 60% mineral content, as determined by AIA, and results for the 1.5-minute froth collection sample were compared with AIA predictions based on recovery of particles with less than 20% mineral matter on the surface. These samples were chosen from the cleaning tests which showed some ash and pyrite reduction by their respective processes. The corresponding points in the AIA distributions were chosen so that AIA-predicted mineral matter reduction aligned with the ash reduction from the cleaning tests. AIA-predicted reductions in pyrite, and thus in pyritic sulfur, were compared to pyritic sulfur reductions from the cleaning tests.

Tables 1 and 2 show the comparison of predicted reductions based on AIA results with actual reductions measured from cleaning tests along with the ash and pyrite contents measured for the raw coals. Results show good correlation between AIA predictions and cleaning test results. For the float-sink results in Table 1, both AIA and cleaning tests showed that in both coals pyrite was more liberated than mineral matter in general. For example, the relative reduction of pyrite, as predicted by AIA, was 23% higher than the reduction in mineral matter for Upper Freeport coal. Actual cleaning results indicated that Upper Freeport and Indiana No. 3 coals were similar in their cleanability, whereas AIA results predicted that the Upper Freeport coal might be slightly more cleanable.

Both cleaning tests and AIA results indicated that these coals were more cleanable by density-based separations than by froth flotation, especially in the case of the Upper Freeport coal. Selective removal of pyrite was not indicated for the surface-based cleaning. Relative reduction of pyrite by froth flotation was only slightly higher than the reduction in mineral matter in general.

Table 1. Comparison of AIA predictions with cleaning results of float sink tests (% removal)

	Original Content	Predicted ¹ Removal, %	Actual ² Removal, %
Upper Freeport			
Pyr. S.	0.95	93	87
MM or Ash	9.88	70	61
Indiana No. 3			
Pyr. S.	2.23	65	85
MM or Ash	7.35	50	55

¹ Prediction based on recovery of particles containing less than 50% mineral matter, by weight

² Based on results of 1.6 sp. gr. separation

Table 2. Comparison of AIA predictions with cleaning results of froth flotation tests (% removal)

	Original Content	Predicted ¹ Removal, %	Actual ² Removal, %
Upper Freeport			
Pyr. S.	0.95	27	30
MM or Ash	9.88	25	21
Indiana No. 3			
Pyr. S.	2.23	53	59
MM or Ash	7.35	47	41

¹ Prediction based on recovery of particles with surfaces more than 80% covered by coal

² Based on results of 1.5 min flotation

These preliminary comparisons of AIA results with results of cleaning tests are very encouraging and suggest that AIA may be used to provide valuable data for predicting the cleanability of coal by either density or surface-based processes. Even though froth flotation recovery and ash rejection can vary widely depending on process conditions, AIA association distributions should provide an estimate of how dependent recovery and mineral reduction for a given coal may be on changes in processing, such as frother or collector concentration or flotation time. The details of the correlation between AIA results and actual cleaning performance for a specific set of process parameters remain to be determined.

Determination of AIA repeatability

Three sets of analyses on a different sample of Upper Freeport coal have been completed in order to determine the repeatability of AIA association results. One pellet of the coal was analyzed twice, and a second pellet of the same coal was analyzed once to assess the influence of limited particle counts and sampling effects on repeatability.

The three sets of association results are shown in Figures 1-3. Figures 1 and 2 show two portions of an analysis of a single pellet. The data from a single analysis of 6,000 particles were split into two data sets of 3,000 particles each, so that the differences in Figures 1 and 2 are entirely due to the effect of limited particle counts. Figure 3 shows the results from the analysis of a second pellet. Differences between Figure 3 and Figures 1 and 2 include sample preparation effects and potential differences in instrumental setup, along with effects due to limited particle statistics.

About 3,000 particles are represented by each of the distribution histograms shown in Figures 1 and 2. For such distributions obtained by counting particles, the variation in the results is related to the square root of the number of particles counted within any given class. Counting more particles leads to a smaller relative error in the results. Without explicit values for the number of particles counted in each category, the count per category for these analyses may be approximated as 30 times the measured percentage (i.e., 1% of the sample corresponds to about 30 particles). The variation (standard deviation) may then be calculated as shown in the following equations.

$$\text{Std.Dev.} = \text{fraction} / \sqrt{(\text{number of particles})},$$

but $\text{number of particles} \approx 30 \times \text{fraction},$

$$\begin{aligned} \text{so } \text{Std.Dev.} &= \text{fraction} / \sqrt{(30 \times \text{fraction})} \\ &= \sqrt{(\text{fraction}/30)} \end{aligned}$$

Therefore, an estimated standard deviation of 0.41% would be calculated for a weight fraction of 5%, and 95% of the reported values should fall within two standard deviations, i.e., 0.82%, of their true value.

Figure 1. Association of coal and mineral matter in the first analysis of the first pellet of Upper Freeport coal

Figure 2. Association of coal and mineral matter in the second analysis of the first pellet of Upper Freeport coal

Figure 3. Association of coal and mineral matter in a second pellet of Upper Freeport coal

The graphical presentations in Figures 1 and 2 are supplemented by Table 3 which shows the fractions of coal, pyrite, and a combination of other minerals for six classes of association, and by Table 4 which shows the variation to be expected in the results due to particle count. Table 4 shows repeatability in the results calculated for selected weight fractions. Using standard statistical principles, the variation in the difference between values from these two sets has been calculated as $\sqrt{2}$ times the variation in the results for either set taken by itself. For example, values near 5% by weight should be within 1.2% of each other 95% of the time. Most of the values in Table 3 are well within these bounds determined by particle count alone. The one notable exception is in the pairs showing the amount of coal in particles with 0% mineral matter and 1-20% mineral matter. Individually, these pairs are outside the range of repeatability; however, when the values are combined to indicate the fraction of sample with 0-20% mineral matter, the agreement is very good. Therefore, the variation appears to be quite adequately explained by variation in particle count.

Table 3. Reproducibility of AIA results for two subsets of an association analysis on a single pellet of Upper Freeport coal

Phase	Wt. % mineral matter (MM) in particle							Total
		0	1-20	21-40	41-60	61-80	81-100	
Coal	A	58.37	20.84	5.01	1.78	0.90	0.58	87.48
	B	52.10	26.47	5.11	2.34	0.70	0.43	87.15
Pyrite	A	0.00	0.23	0.34	0.08	0.40	1.54	2.59
	B	0.00	0.27	0.30	0.20	0.50	1.26	2.53
Other MM	A	0.00	1.43	1.64	1.58	1.71	3.57	9.93
	B	0.00	1.78	1.76	2.11	1.20	3.46	10.31
Total	A	58.37	22.50	6.99	3.44	3.01	5.69	100.00
	B	52.10	28.52	7.17	4.65	2.40	5.15	100.00

Table 4. Estimated variation in AIA results at selected weight fractions showing two standard deviations for a single analysis (i.e., % wt. fraction $\pm 2\sigma$) and the minimum significant difference between values from two different analyses (i.e., Result₁-Result₂)

% weight fraction	variation in a single analysis	minimum significant difference
1	0.37	0.52
2	0.52	0.73
5	0.82	1.15
10	1.15	1.63
20	1.63	2.31
50	2.58	3.65

Comparison of Figure 3 with Figures 1 and 2 shows additional variation in the results due to minor changes in instrument setup and due to sampling effects. The variation appears to be about twice that due to limited particle counts alone. However, the same patterns of association appear in all three figures, such as the preferential liberation of pyrite and calcite.

Characterization of additional coals

Analyses are in progress on three 100-mesh samples of Upper Freeport, Illinois No. 6, and Pittsburgh No. 8 coals from the Argonne Premium Coal Sample Program. Data collection has been completed, and results will be available for the next report. Results will be presented to show association based on both particle composition (i.e., density) and particle surface for these samples of three coals widely used in coal preparation experiments.

CONCLUSIONS

The AIA technique has been improved for determining the association of mineral matter with coal in terms of the relative amount of coal and mineral matter present on the particle surface. This format appears to be suitable for predicting the behavior of samples undergoing surface-based cleaning processes. Detailed results available through AIA helped to explain the results of lab-scale froth flotation performance of an Indiana No. 3 and an Upper Freeport coal. Both lab tests and AIA predictions indicated that surface-based processes would be much less effective than density-based processes for removing mineral matter from these coals. They also showed that surface-based cleaning would be somewhat more effective in reducing mineral matter in the Indiana coal than in the Upper Freeport coal, even apart from explicit consideration of the hydrophobic properties of the coals. AIA predictions of pyrite removal also agreed well with the results of the cleaning tests. Based on these results, it should be possible to develop AIA for predicting the cleanability of a coal with regard to mineral matter or specific minerals, and for helping to explain difficulties with the application of cleaning processes to certain coals.

In the future, characterization of the particle surfaces will be developed further. Additional samples of raw and cleaned coals will be examined in an effort to better relate AIA-measured distributions to the behavior under various process conditions. Also, a complete set of samples (raw coal, clean product and tailings) from a surface-based cleaning process will be analyzed so that a mass balance may be performed in terms of particle association, and to check for misplacement of material during cleaning.

Repeatability of AIA association results was confirmed by multiple analyses of a single sample of coal. Variation based on the finite number of particles analyzed was in line with expectations. Relative repeatability was on the order of $\pm 16\%$ for weight fractions around 5%. Repeatability was somewhat better for larger fractions including more counts and respectively worse for smaller fractions with less counts. Inter-pellet sampling effects and changes in instrument setup lead to additional variation so that relative reproducibility was on the order of 32% for fractions of 5% by weight. Analysis of 3,000 particles appears to be adequate to describe the character of a coal. For special cases, more particles may need to be analyzed to determine the character of minor mineral components. Examination of the data will be completed during the next quarter and final conclusions will then be tendered.

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