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DEVELOPMENT OF THE CHEMICAL AND ELECTROCHEMICAL COAL CLEANING PROCESS

by

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ABSTRACT

Studies concerning the use of flotation as an alternative method of separating liberated mineral matter from CECC treated coal were continued using Elkhorn No. 3 coal. The results obtained using different methods of recovering the clean product after CECC treatment showed trends similar to those observed previously with Pittsburgh No. 8 coal. The use of flotation after CECC treatment of the Elkhorn No. 3 coal was found to be more efficient than wet-screening.

Construction of the continuous bench-scale unit was completed in this quarter. The unit was modified to process 2 lbs/hr instead of the original design of 1 lb/hr so as to ensure efficient operation of the vacuum filter unit. Start-up of the continuous unit was successfully conducted and shakedown testings were started. Initial results show that the continuous unit was able to reduce the ash content of a recently acquired Middle Wyodak coal sample to as low as 1.16%.

PROJECT OBJECTIVES

The objectives of this work are to: (a) determine the mechanisms by which the CECC process removes ash and pyritic sulfur from coal, (b) learn more about the operating parameters of the process, (c) collect engineering information for the scale-up of the process, and (d) test the CECC process as a bench-scale continuous operation.

PROJECT TASKS

Task 4 - Batch Testing

Tests studying the effect of using flotation as an alternative method of separating liberated mineral matter from coal after CECC treatment were continued in this quarter.

Subtask 4.3 - Mechanistic Studies

Alternative Method of Separating Liberated Mineral Matter from Coal

The use of conventional flotation to separate liberated mineral matter from the treated coal was previously found to be more effective than screening for Pittsburgh No. 8 coal. Tests were continued here studying the use of different methods of recovering the clean coal after CECC treatment. The 65 x 325 mesh size Elkhorn No. 3 coal samples were treated using the following conditions: 6% solids, 3 M H_2SO_4 , 10^{-5} M Fe^{3+} ions and a temperature of 40°C . For Test No. 1, conventional flotation of the sample in a 1-liter Denver flotation cell was conducted without any CECC pretreatment, while Test No. 2 involved CECC treatment with the clean coal recovered by wet-screening at 325 mesh. In Test No. 3, the +325 mesh CECC-treated fraction from Test No. 2 was floated to get the final CECC product. The coal sample was floated directly after CECC treatment without wet-screening in Test No. 4. A flotation time of 1 minute was used for all flotation tests with 1 lb/ton MIBC added as frother.

Table I shows the results obtained using different methods for removing the liberated mineral matter from CECC-treated Elkhorn No. 3 coal samples. Also shown for comparison is the result for the test using flotation without CECC treatment (Test No. 1). The ash content was reduced in the conventional

TABLE I

A. Ash Analysis Results for Elkhorn No. 3 Coal

Test No	Process	Ash Content (% wt)			Ash Rejection (% wt)			Coal Recovery (% wt)
		Feed	Product	Reject	Total	Liberated	Dissolved*	
1	Flotation	18.46	15.40	87.57	20.79	-	-	94.96
2	CECC	18.46	14.00	63.44	29.33	92.68	7.32	93.16
3	CECC and .. Flotation	18.46	10.88	53.29	49.14	95.63	4.63	86.30
4	CECC and Flotation	18.46	9.05	45.57	59.53	79.32	20.68	82.55

* and/or lost
 .. wet-screened (325 mesh) prior to flotation
 no wet-screening

TABLE I

B. Sulfur Analysis Results for Elkhorn No. 3 Coal

Test No	Process	Sulfur Content (% wt)			Sulfur Rejection (% wt)		
		Feed	Product	Reject	Total	Liberated	Dissolved*
1	Flotation	1.91	1.79	-	12.75	-	-
2	CECC	1.91	1.65	3.10	21.93	56.80	43.20
3	CECC and .. Flotation	1.91	1.70	1.05	25.48	63.23	36.77
4	CECC and ... Flotation	1.91	1.72	1.62	28.32	55.32	44.77

* and/or lost

.. wet-screened (325 mesh) prior to flotation

... no wet-screening

flotation test from 18.46% to about 15.4%, which corresponds to a % ash rejection of 20.8%. For Test No. 2, which used wet-screening after CECC treatment, the % ash rejection was improved to about 29.3% without a significant decrease in coal recovery. From the mass balance of the ash contents, liberation accounted for about 92.7% of the total mineral matter removed.

Test No. 3, which used flotation instead of wet-screening after CECC treatment, showed a reduction of the ash content to around 10.9% with a coal recovery of about 86.3%. The % ash rejection was calculated to be 49.1%, which is significantly better than the values obtained in Test Nos. 1 and 2. The amount of mineral matter removed that may be attributed to liberation also increased. These results would indicate that a significant amount of coarse liberated mineral matter is being produced in the CECC processing of this sample.

The last test in this series was conducted using flotation but without screening to remove the -325 mesh particles produced during the CECC treatment (Test No. 4). The results for this test show that the amount of mineral matter rejected also increased significantly. The ash content was reduced to about 9% with a slight decrease in coal recovery. This last test resulted in a % ash rejection of almost 60%, which is higher than the other three values. However, the amount of mineral matter removed that may be attributed to liberation decreased to about 79.3%. The increase in mineral matter removal may be due to an increase in the dissolution of the mineral matter, as indicated by the mass balance. Additionally, it was found that the product size distribution of Test No. 4 was coarser than that of Test No. 3, and this may also account for the difference in their product ash contents.

It should be noted, however, that the amount of mineral matter removed in these tests is significantly lower than those obtained previously. Using diffuse reflectance FTIR spectroscopy, the feed samples used here were found to be slightly oxidized already. As stated previously, the CECC process was found to be ineffective in treating oxidized feed samples. This would then explain why the results obtained in these tests are relatively poor. Another factor to consider is that although both samples were from the same batch, the ash and sulfur contents of the sample used in this quarter are higher than those used in the Eleventh Quarter. It is possible that an excessive amount of Fe^{3+} ions may have been present in the tests conducted in this quarter due to the higher pyrite content. As discussed previously, an increase in pyritic sulfur content may result in an increase in the amount of Fe^{3+} ion coming from pyrite dissolution. If this amount is in excess, then there will be insufficient counter ions present and/or jarosite formation would occur, which is detrimental to the CECC process.

The amount of sulfur rejected by the different tests is also shown in Table I. The use of flotation to recover the clean coal after CECC treatment resulted in slightly better sulfur removal. Also, the CECC treatment gave significantly better sulfur rejection than flotation. This may be attributed to the additional sulfur rejection brought about by pyrite dissolution in the CECC process.

Task 5 - Design and Construction of the CECC Continuous Unit

Subtasks 5.2 & 5.3 - CECC Unit Construction and Modification

The construction of the CECC continuous bench-scale unit was completed in this quarter. The vacuum filter was installed and connected according to

the design. Start-up of the unit was carried out using the optimum conditions determined for the Middle Wyodak coal, but in the absence of any acid. During the start-up, the flow rate feeding into the reactor was found to be slower than the filtration rate of the vacuum filter. Consequently, there was not enough slurry being fed into the filter for efficient filtration. This problem was solved by doubling the flow rate of all components of the system. Thus, the entire unit was modified to process coal at the rate of 2 lbs/hr, instead of the original design of 1 lb/hr. Aside from this, there were no other major problems found in the unit during the start-up procedures.

Task 6 - Continuous Unit Operation

Subtask 6.1 - Shakedown Testing

After the satisfactory start-up of the continuous unit, the shakedown testing was started using Middle Wyodak coal samples. The operating condition used, which was the optimum determined in Subtask 4.2, was: 6% solids, 2.28×10^{-4} M Fe^{3+} ion added, 2.32 M H_2SO_4 and a temperature of 80°C. The 65 x 325 mesh coal samples were fed at a rate of 2 lbs/hour with a total retention time of about 11 hours in the reactor. The coal used here, instead of the original sample, was a recently acquired Middle Wyodak coal from the Kerr McGee Coal Corporation. This change was made because the original sample (split-B) obtained from Task 2 was found to be already oxidized. As discussed previously, the removal of mineral matter by the CECC process is ineffective on oxidized samples.

Table II shows the results obtained for the Middle Wyodak coal at different time periods. For Day 1, the first batch of samples showed the ash content being reduced from 5.37% to about 2.02%. On Day 2, the ash content of

TABLE II

Results of CECC Shakedown Testing on Middle Wyodak Coal

<u>Sampling Period</u>	<u>Ash Content (% wt)</u>		<u>Sulfur Content (% wt)</u>	
	<u>Feed</u>	<u>Product</u>	<u>Feed</u>	<u>Product</u>
Day 1	5.37	2.20	0.375	0.650
Day 2	5.37	1.58	0.375	0.672
Day 5	5.37	1.16	0.375	0.616

the wet-screened product was reduced to about 1.58%, with the reject having an ash content of about 5.51%. After running the unit for 5 days, the ash content of the product obtained was 1.16%. The product ash contents obtained here were much lower than those obtained in the parametric tests. This may be attributed to this coal sample being relatively less oxidized than the original sample used in the parametric tests.

Regarding the removal of sulfur from this low sulfur coal, the % sulfur rejections obtained in all the tests were negligible. In fact, the sulfur content appeared to have increased due to the adsorption of the sulfate ion by the coal.

SUMMARY, CONCLUSIONS AND PLANNED FUTURE WORK

Studies were continued in this quarter to find alternative methods of separating the liberated mineral matter after CECC treatment. The results obtained for the Elkhorn No. 3 coal showed that the use of flotation after CECC treatment gave better mineral matter removal. This observation is similar to that observed previously for the Pittsburgh No. 8 coal. The use of flotation to separate the liberated mineral matter from the clean coal will be investigated in the continuous test work. Also, flotation will be used in the CECC processing of fine coals to produce superclean and ultraclean coals. A flotation column was incorporated into the continuous unit for these purposes.

Construction of the continuous bench-scale unit was completed in this quarter. Start-up of the unit was conducted successfully using optimum conditions determined in Subtask 4.2 but without any acid added to the system. From this start-up procedure, the feed flow rate was found to be inadequate

and was increased. Consequently, the continuous unit was modified to treat coal at a rate of 2 lbs/hr, instead of 1 lb/hr. This was necessitated by the filtration rate of the vacuum drum filter being higher than the original design, which caused it to operate inefficiently.

Shakedown testing of the continuous unit was started afterwards on a new batch of Middle Wyodak coal obtained from Kerr McGee Coal Corporation. The previous sample (split-B) was not used since it was already oxidized. Using the optimum conditions determined from Subtask 4.2, the CECC continuous unit was able to reduce the ash content of the sample from 5.37% to as low as 1.16%. This value is better than that obtained in the parametric (Subtask 4.1) and optimization tests (Subtask 4.2). Shakedown testing of the continuous bench-scale unit using the Middle Wyodak coal will be continued.

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