POC-SCALE TESTING OF OIL AGGLOMERATION TECHNIQUES
AND EQUIPMENT FOR FINE COAL PROCESSING

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Principal Investigators

B. Ignasiak

W. Pawlak

K. Szymocha

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Alberta Research Council

Environmental Technologies

250 Karl Clark Road

Edmonton, Alberta

Canada T6N 1E4

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ABSTRACT

This report covers the technical progress achieved from July 01, 1997 to September 30, 1997 on the POC-Scale Testing Agglomeration Techniques and Equipment for Fine Coal Processing project.

Experimental procedures and test data for recovery of fine coal from coal fines streams generated at a commercial coal preparation plant are described. Two coal fines streams, namely Sieve Bend Effluent and Cyclone Overflow were investigated. The test results showed that ash was reduced by more than 50% at combustible matter recovery levels exceeding 95%.

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EXECUTIVE SUMMARY

This report presents the results of work completed during July 1 to September 30, 1997 on the POC-Scale Testing Agglomeration Techniques and Equipment for Fine Coal Processing project, under FETC/DOE Contract No. DE-AC22-95PC95152.

The objective of the project is to demonstrate a Proof-of-Concept (POC) 3 t/h scale Aglofloat technology capable of increasing the recovery and improving the quality of fine coal streams.

Two distinctly different coal conditioning devices, namely a conventional high-shear mixer and a jet processor were selected to:

- demonstrate the potential of Aglofloat technology for fine coal recovery, and
- develop a simple, economic, and reliable piece of equipment for coal conditioning (microagglomeration).

During the reporting period two samples of coal fines generated by Drummond's Shoal Creek Mine preparation plant were tested. Both samples represent current froth flotation feed and are identified as:

Sample 1	Sieve Bend Effluent, with nominal size range 600µm x 250µm (28M x 60M) and ash content of 43.7%
Sample 2	Cyclone Overflow, with nominal size range 250µm x 0 (60M x 0) and ash content of 30.6%

Preliminary Aglofloat tests were carried out for both samples using light oil (Diesel) and heavy oil (mixture of Maya Crude and Diesel oils at 1:1 ratio). The oil concentration was 0.5% and 1.0% on dry coal basis. The solids concentration was 20% and 30% respectively.

The test results are presented in Appendix B - 1 of this report, and were presented and discussed during the September 25, 1997 meeting at Drummond Company Ltd.

The results of preliminary agglomeration tests show that:

- significant reduction in ash content was achieved:
 - from 43.7% to about 18.5% for the Sieve Bend Effluent sample
 - from 30.6% to about 14.5% for the Cyclone Overflow sample
- high combustible matter recovery (about 95%) was obtained for both samples.

Centrifuge float-sink washability analyses were performed for both coals for evaluation of the Aglofloat process performance

Figure 1. Comparison of Float-Sink Tests with Aglofloat Process for Sieve Bend Effluent.

Comparison of the results for float-sink separation and Aglofloat process (see Figure 1 and Figure 2) shows that Aglofloat process performance is as good or better than float-sink separation. The better performance by the Aglofloat process most likely results from the additional liberation of mineral matter that occurs during the intense

mixing/conditioning of the coal slurry. In the region of impeller a high turbulence is generated that results in coal particle collisions and their further desintegration.

Figure 2. Comparison of Float-Sink Tests with Aglofloat Process for Cyclone Overflow

During the September 25, 1997 meeting at Drummond Company, it was found (based on results of coal characterization) that the coal samples ARC obtained for testing were not representative of typical coal fines streams generated by the Shoal Creek Mine preparation plant. As a result further tests with the first set of coal samples were suspended and ARC and Drummond Company representatives agreed that:

- ARC will get a new representative sample of coal fines (60M x 0) from Drummond's Shoal Creek Mine preparation plant
- ARC will get an additional sample taken from the ponds
- ARC will repeat the characterization tests with these new coal samples

New coal samples will be characterized and tested in the next phase of the project.

1.0 INTRODUCTION

This report covers the technical progress achieved from July 01, 1997 to September 30, 1997, on the POC-Scale Testing Agglomeration Techniques and Equipment for Fine Coal Processing project, under FETC/DOE Contract No. DE-AC22-95PC95152.

The objective of this project is to develop and demonstrate a Proof-of-Concept (POC) scale technology capable of increasing the recovery and improving the quality of fine coal streams. The terms and conditions of the project include the following:

- the technology should have a near term implementation status
- the POC-scale equipment should be demonstrated at a commercial coal preparation plant in the United States
- there should be a large reserve of coal available for processing using the new techniques/equipment at a selected commercial coal preparation plant.

The oil agglomeration and especially the Aglofloat technology was selected for demonstration of POC-scale equipment for fine coal processing. This process can be applied successfully particularly for coal fines below 150 μm (100M). Low oil addition levels combined with exceptional selectivity place the Aglofloat process in the forefront of technologies for fine coal clean-up and recovery. Two distinct agglomeration devices, namely a conventional high-shear mixer and a jet processor were selected in order to:

- demonstrate the potential of Aglofloat process for fine coal recovery, and
- develop a better, more economic and more reliable piece of equipment for coal slurry conditioning (microagglomeration).

To meet the overall objectives, an 11 task work plan was designed. The work ranges from batch and continuous bench-scale testing through the design, commissioning and field testing of POC-scale agglomeration equipment.

The project commenced in June 1995, and was suspended by DOE in June 1996, when two coal producers (first Cooney Bros. Coal Co. and subsequently, PBS Coals Inc.) made decisions not to proceed with the project because they had obtained long term contracts that did not require cleaning of coal fines prior to their blending with clean coarse coal.

On February 13, 1997 ARC signed a Host Agreement with Drummond Company Inc. and the project was formally reactivated by DOE on May 01, 1997.

During the current reporting period there were activities relative to the following tasks:

- Task 1 - Project planning and management
- Task 2 - Host site selection and plan formulation
- Task 4 - Coal characterization and laboratory (batch) and bench-scale testing

The work on the remaining tasks is scheduled for completion in the next months.

2.0 SUMMARY OF WORK COMPLETED AND IN PROGRESS

2.1 Host Site - Shoal Creek Mine Preparation Plant

The Shoal Creek Mine preparation plant owned and operated by Drummond Company Inc. serves as a host site for POC-scale equipment demonstration. The plant is located in Oak Grove, Jefferson County, Alabama. Drummond provided ARC with the following information about the Shoal Creek Mine preparation plant:

- Site Grading Plan (mine)
- Site Plan
- Material, Handling, Coal Preparation and Barge Load Facilities flow chart

The preparation plant consists of two trains each with a capacity of 1815 t/h (2000 TPH). The plant processes three streams of coal (coarse, intermediate and fine) and contains the following major pieces of equipment:

- Rotary breakers that crush the feed coal below 75 mm (3")
- Dry and wet screens for separation of coarse 75 mm x 6.35 mm (3"x 1/4") and intermediate 6.35 mm x 0 (1/4" x 0) streams
- Coarse coal jig for the 75 mm x 6.35 mm material and fine coal jig for the 6.35 mm x 0 material
- Froth flotation circuits that process fines 250 µm x 0 (60M x 0)
- Classifying cyclone for 250 µm x 0 (60M x 0) material

Figure 2.1 illustrates a simplified flowsheet of the host site facility.

On September 25, 1997 ARC's Project Manager visited Drummond's office in Jasper, Alabama and toured the Shoal Creek preparation plant. During the plant tour a site for future location of POC-scale equipment was selected.

2.2 Project Coal Fines

2.2.1 Coal feed for Shoal Creek Mine preparation plant

The Shoal Creek preparation plant processes coal mined from the Mary Lee Coal Group located at Warrior Coalfield, Alabama. This group consists of five beds and some of

these beds attain thickness throughout much of the basin. The majority of the coal is ranked as high volatile A-bituminous coal and generally contains a low percentage of sulfur. Adjacent to Drummond Company Inc., Jim Walter Resources also mines the Mary Lee Coal Group.

Samples of two coal fines streams that were generated at Drummond's coal preparation plant were submitted for testing. Both samples represented current froth flotation feed and were marked and described as follows:

Figure 2.1. Simplified flowsheet of the Shoal Creek Mine preparation plant.

Sample 1 - Sieve Bend Effluent – 600 μm x 250 μm (28M x 60M)
Flotation Feed – Shoal Creek Preparation Plant

The particle size of the Sieve Bend Effluent varies depending on cleaning regimes utilized at the plant, but is nominally 600 μm x 250 μm (28M x 60M). The contribution of this fraction to the flotation feed varies from a minimum of 1% to approximately 6 % of the plant feed (9 t/h to 54 t/h (10 TPH to 60 TPH) for each train).

Sample 2 - Cyclone Overflow – 250 μm x 0 (60M x 0)
Flotation Feed – Shoal Creek Preparation Plant

The Cyclone Overflow sample is representative of the majority of the feed to the existing flotation system at Shoal Creek plant. It represents approximately 7% to 12% of the plant feed (approximately 63 t/h to 109 t/h (70 to 120 TPH) for each train).

The coal samples were collected directly from plant streams and were shipped as taken in 19 L (5 gallon) containers (2 containers of each sample).

Sample 1 – Sieve Bend Effluent did not contain free water; it was moist and after homogenization was riffled into 9.5 L (2.5 gallon) pails. In order to keep the moisture level constant, the pails were tightly sealed and stored in a cold room.

Sample 2 – Cyclone Overflow contained free water above a bed of coal fines. In order to homogenize the sample and prepare the feed for testing, the free water was drained and the moist coal fines were spread on a plastic sheet, thoroughly mixed, and then riffled into 9.5 L (2.5-gallon) pails.

2.2.2 Coal Fines Characterization

Project coal fines characterization included:

- routine analyses including proximate and ultimate composition, calorific value and particle size distribution, and
- float-sink separation for cleanability determinations

ASTM standards were used for coal fines properties determinations, while an in-house developed centrifugal float-sink procedure was utilized for washability (cleanability determinations). A list of analytical methods used for coal characterization and a description of the float-sink procedure are included in Appendix A.

Table 2.1 provides the proximate and ultimate composition for both project coal samples.

Both samples were characterized by high-ash contents of 43.7 % and 30.6 % for the Sieve Bend Effluent and Cyclone Overflow, respectively. Both of them can be considered as low volatile bituminous coal.

Table 2.1 Properties of Project Coal Fines

Property	Coal Fines Samples	
	Sieve Bend Effluent 600 μm x 250 μm (28M x 60M)	Cyclone Overflow 250 μm x 0 (60 M x 0)
Moisture, wt % (ar)	23.4	34.4
Ash, wt % (db)	43.7	30.6
Volatile Matter, wt % (db)	19.0	21.0
Fixed Carbon, wt % (db)	37.3	48.4
Carbon, wt % (daf)	86.5	86.9
Hydrogen, wt % (daf)	5.0	4.9
Nitrogen, wt % (daf)	1.8	1.8
Sulfur, wt % (daf)	1.7	0.8
Oxygen, wt % (daf by diff.)	5.0	5.6
Calorific Value (db) MJ/kg	18.35	23.79
Btu/lb	7,890	10,230

Note: ar - as recieved
db - dry basis
daf - dry, ash free

Table 2.2 and Figure 2.2 present particle size distribution data for the Sieve Bend Effluent sample. A Drummond representative indicated that this sample should contain particles in the 600 μm x 250 μm size range.

Table 2.2. Particle Size Distribution for Sieve Bend Effluent
(600 μm x 250 μm)

	Size Fraction, μm							
	-45	45-63	63-90	90-150	150-180	180-250	250-355	355-1000

Percent	14.2	8.6	13.6	28.3	9.3	19.2	4.2	2.6
Cum. %	14.2	22.8	36.4	64.7	74.0	93.2	97.4	100

However, the coal sample sent to ARC was out of the Shoal Creek plant specification for this stream. About 93 % of the coal particles were smaller than 250 μm which is nominally the bottom limit given for this sample and mass median diameter was about 115 μm .

Figure 2.2 Particle Size Distribution for Sieve Bend Effluent (600µm x 250µm)

Table 2.3 and Figure 2.3 present particle size distribution data for the Cyclone Overflow sample with particles in the size range of 250 µm x 0 (60M x 0). The results show that this sample represents a typical plant stream produced by cyclones.

Table 2.3. Particle Size Distribution For Cyclone Overflow Coal Sample (250 µm x 0)

	Size Fraction, µm						
	-45	45-63	63-90	90-150	150-180	180-250	250-355
Percent	66.5	8.3	11.8	6.0	2.0	2.9	2.5
Cum. %	66.5	74.8	86.6	92.6	94.6	97.5	100.0

About 97.5 % of particles were smaller than 250 μm (60M) and 66.5 % of the particles were smaller than 45 μm (325M). Due to the wet screening limitations, exact mass median diameter was not determined, nevertheless it would be smaller than 45 μm .

Figure 2.3 Particle Size Distribution for Cyclone Overflow (250 μm x 0).

The project sample characterization program also includes determination of coal cleanability. For determination of fine-coal cleanability, the centrifugal float-sink washability method was used. Detailed information about the centrifugation float-sink procedure is given in Appendix A -1.

Centrifugal float-sink washability analyses were performed for both coal samples and the results obtained were used for evaluation of the performance of the Aglofloat process.

Table 2.4. Float-Sink Separation Results for Sieve Bend Effluent (600 μm x 250 μm)

Specific Gravity	Float				Sink	
	Yield wt %	CM Recovery wt %	Ash wt %	Total S wt %	Yield wt %	Ash wt %
1.3	23.5	38.5	7.8	0.49	76.5	54.1
1.4	36.9	58.7	10.4	0.57	63.1	64.5
1.6	58.8	91.9	11.7	0.69	41.2	83.7
1.8	61.9	90.6	17.6	0.75	38.1	84.9
2.0	73.3	97.4	25.2	0.77	26.7	88.8

Note: All results on dry basis
CM – Combustible Matter

Table 2.4 and Figure 2.4 present results obtained for centrifugation float-sink tests obtained for the Sieve Bend Effluent coal sample. Similar data are presented in Table 2.5 and Figure 2.5 for the Cyclone Overflow coal sample.

Another presentation of the washability results, which are very useful for the evaluation of other separation processes, is shown in Figure 2.6 and in Figure 2.7.

Figure 2.4 Washability Curve for Sieve Bend Effluent (600 µm x 250 µm).

Table 2.5. Float-Sink Separation Results for Cyclone Overflow (250 µm x 0)

Specific Gravity	Float				Sink	
	Yield wt %	CM Recovery wt %	Ash wt %	Total S wt %	Yield wt %	Ash wt %
1.3	46.0	60.2	9.2	0.68	54.0	44.4
1.4	55.0	68.3	13.8	0.67	45.0	55.1
1.6	78.4	95.0	15.9	0.65	21.6	67.7
1.8	81.1	96.1	17.8	0.71	18.9	82.7

2.0	83.4	97.0	18.3	0.72	16.6	89.3
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Note: All results on dry basis
CM – Combustible Matter

Figure 2.5 Washability Curve for Cyclone Overflow (250 μ m x0).

2.3 Laboratory Batch Tests

Preliminary Aglofloat tests were carried out using light Diesel oil and heavy oil which was a mixture of Maya Crude oil and Diesel oil at 1:1 ratio. The oil concentration was 0.5% and 1.0% on a dry coal basis.

Table 2.6 shows the test matrix for Aglofloat batch tests as used for processing both coal samples. Information about the Aglofloat procedure and the equipment used is given in Appendix A-2.

Table 2.6. Test Matrix Utilized for Project Coal Fines Batch Testing

Coal Fines	Sieve Bend Effluent (600 μm x 250 μm) Cyclone Overflow (250 μm x 0)
Bridging Oil <ul style="list-style-type: none"> Type Addition 	Diesel Oil Maya Crude Oil & Diesel Oil (1:1 ratio) 0.5 % (dry coal fines basis) 1.0 % (dry coal fines basis)
High-Shear Mixer <ul style="list-style-type: none"> Solids Concentration Mixing Intensity Residence Time 	20 % (dry coal fines basis) 30 % (dry coal fines basis) 2 000 rpm 1 min
Froth Flotation <ul style="list-style-type: none"> Solid Concentration Mixing Intensity Frother Residence Time 	6.5 % (dry coal fines basis) 1 100 rpm MIBC at 45 g/t (0.1lb/t) No addition 6 min

Aglofloat test results for the Sieve Bend Effluent coal sample are presented in Table 2.7 and in graphical form in Figure 2.6.

Comparison of the test results obtained for the Aglofloat process with float-sink tests results shows that the Aglofloat process performance is as good as float-sink separation or sometimes better. Similar trends were observed for Cyclone Overflow tests and are presented in Table 2.8 and in Figure 2.8.

The better performance by the Aglofloat process most likely results from additional liberation of mineral matter that occurs during intense mixing/conditioning of the slurry in the High Shear Mixer.

Table 2.7. Aglofloat Process Test Results for Sieve Bend Effluent (600 μm x 250 μm)

Test I. D.	High-Shear Solids Concentration wt %	Oil Add. wt %	Clean Coal Ash wt %	CM Recovery, wt %	Ash Reduction, wt %
DR-1	20	0.5 Diesel	19.5	92.6	53.4
DR-2	20	1.0 Diesel	18.5	94.4	57.8
DR-5	20	0.5 MC +D	18.6	95.6	57.3
DR-6	20	1.0 MC +D	18.6	94.6	57.4
DR-9	30	0.5 Diesel	19.4	96.1	55.6
DR-10	30	1.0 Diesel	19.9	97.7	54.4
DR-13	30	0.5 MC +D	19.3	93.8	55.8
DR-14	30	1.0MC +D	19.2	95.0	56.1
DR-17*	30	0.5 Diesel	17.3	95.1	60.3
DR-18*	30	1.0 Diesel	18.7	95.8	57.3
DR-21*	30	0.5 MC +D	17.6	93.1	59.7
DR-22*	30	1.0 MC +D	19.7	94.8	54.8

Note: MC + D – Blend of Maya Crude Oil and Diesel Oil (1:1 ratio)

CM – Combustible Matter

* – no Frother

For more detail results see Appendix B Table B-1

Figure 2.6 Evaluation of the Aglofloat Process by Comparison with Float-Sink Tests
Results for Sieve Bend Effluent.

Table 2.8. Aglofloat Process Test Results for Cyclone Overflow (coal fines 250 µm x 0)

Test I. D.	High-Shear Solids Concentration wt %	Oil Add. wt %	Clean Coal. Ash wt %	CM Recovery, wt %	Ash Reduction, wt %
DR-3	20	0.5 Diesel	14.8	94.2	51.6
DR-4	20	1.0 Diesel	14.4	93.5	52.8
DR-7	20	0.5 MC +D	14.5	94.7	52.5
DR-8	20	1.0 MC +D	12.9	95.7	57.8
DR-11	30	0.5 Diesel	15.7	96.9	48.9
DR-12	30	1.0 Diesel	16.3	97.3	46.6
DR-15	30	0.5 MC +D	17.3	97.7	43.4
DR-16	30	1.0MC +D	18.2	97.2	40.5
DR-19*	30	0.5 Diesel	15.5	97.4	49.3
DR-20*	30	1.0 Diesel	16.0	95.1	47.7
DR-23*	30	0.5 MC +D	15.8	96.1	48.5
DR-24*	30	1.0 MC +D	15.8	99.2	48.5

Note: MC + D – Blend of Maya Crude Oil and Diesel Oil (1:1 ratio)

CM – Combustible Matter

* – no Frother

For more results see Appendix B Table B-1

Figure 2.7 Evaluation of the Agfloat Process by Comparison with Float-Sink Tests
Results for Cyclone Effluent.

2.4 Supplementary Tests - Reconstitution

During the September 25, 1997 meeting at Drummond Company, it was concluded that the coal samples ARC obtained for testing were not representative of typical coal fines streams generated by the Shoal Creek Mine preparation plant. To verify the ARC results, additional analyses were performed to determine the ash content in the tailings in order to

calculate reconstituted ash values. Reconstitution of the sample was done to confirm feed ash content values that were obtained during the coal fines characterization.

The ash content of the process tailings was very consistent for both samples and on the average was about 88%. Table 2.9 presents the ash contents in products (clean coal and tailings) and the ash levels in feed coals obtained by direct determination and by reconstitution based on ash content and yields of process products.

Table 2.9 Ash Content in Clean Coal, Tailings and Reconstituted Feed Coal

Test I.D.	Ash Content in Products, wt %		Ash Content in Feed, wt %	
	Clean Coal	Tailings	Direct Determination	Reconstituted
Sieve Bend Effluent				
DR- 1	19.5	88.2	43.7	43.3
DR- 2	18.5	88.1	43.7	42.4
DR- 5	18.6	87.8	43.7	42.0
DR-6	18.6	88.2	43.7	42.3
DR-13	19.3	88.5	43.7	42.9
DR-14	19.2	87.5	43.7	41.6
DR-17	17.3	88.1	43.7	42.3
DR-18	18.7	88.1	43.7	41.5
DR-21	17.6	88.9	43.7	44.2
DR-22	19.7	88.1	43.7	42.0
Cyclone Overflow				
DR- 3	14.8	88.0	30.6	29.5
DR- 4	14.4	88.5	30.6	29.2
DR- 7	14.5	88.2	30.6	29.9
DR- 8	12.9	88.6	30.6	29.1
DR-11	15.7	88.6	30.6	29.3
DR-12	16.3	88.5	30.6	29.1

Reconstitution of the ash content in the feed coal (based on the ash contents in clean coal and tailings) confirmed that the ash results obtained by direct determination were correct.

3.0 CONCLUSIONS

The coal samples subjected to testing showed a very good response to the Aglofloat process treatment.

The results obtained for coal samples shows significant reduction of ash content, in the range from 48% to 60%, at coal matter recovery exceeding 95%.

A good selectivity of the Aglofloat process, expressed by a very high tailings ash (about 90%), was observed.

A comparison of the Aglofloat test results with float-sink test shows that the Aglofloat process performance might be better if optimized process conditions are applied.

Based on the results obtained it was concluded that the samples ARC received for testing, did not represent typical, average coal fines streams produced by the Shoal Creek Mine preparation plant.

As a result of the September 25, 1997 meeting at Drummond Company, a discussion of different options to resolve the problem of coal fines representativeness, resulted in the following recommendations:

- Further tests with the samples ARC had received will be suspended
- ARC will get a new, representative sample of the coal fines stream with a size range of $250\ \mu\text{m} \times 0$ (60M $\times 0$) from Drummond's Shoal Creek Mine preparation plant
- ARC will get an additional coal sample taken from the existing settling ponds
- ARC will repeat the characterization tests with these new coal samples.

APPENDIX A

A-1 Float-Sink Separation

A-1.1 Float-Sink Apparatus

The centrifugal float-sink tests are carried out using the apparatus shown in Figure A-1. This apparatus has been made based on the method developed and described by J.P. Franzidis and M.C. Harris*. This equipment has been devised for speeding up float-sink analysis of coal fines. The float-sink device consists of two chambers:

- outside chamber – a standard stainless 250mL centrifugation tube, and
- inside chamber – a stainless steel custom made to fit centrifugation tube

The inside chamber is a tapered tube with the bottom sealed by a conical plug. This plug is connected via a rod to a spring at the top of the chamber. When sufficient downward forces act on the plug, the spring is compressed just opening the bottom of the chamber. At the beginning of the float-sink test the coal-solvent slurry remain in the inside chamber of the apparatus. The outside chamber is filled with the same solvent and the fluid level is the same as in the inside chamber and enables sink material to enter the outside chamber. During spinning, at certain value rpm, the centrifugal force acting on the conical plug pulls it down just compressing the spring. This links the fluid in the inside chamber with that in the outside chamber. When the separation is completed and rpm is reduced, the downward force decrease and the conical plug seals the inside chamber isolating the float material from the sink material.

*J.P. Franzidis and M.C. Harris, "A Method for the Rapid Float-Sink Analysis of Coal Fines", J.S. Afr. Inst. Min. Metallo., Vol. 86 No. 10, Oct 1986, pp. 409-414.

A-1.2 Centrifugal Float-Sink Experimental Procedure

1. Wet screen coal fines samples into three size fractions: +500 μm , 500 μm x 150 μm and 150 μm x 0.
2. Dry the coal size fractions in a convention oven, which is set at 105⁰C for 3 hours to achieve a moisture content of essentially 0%. Place the samples in a desicator.
3. Weigh 2 g of coal sample, using analytical balance into 100 mL Erlenmeyer flask with $\overline{\text{\$}}$ stopper, add 50 mL of solvent with required density and shake for 20 min at 216 rpm on a shaker.
4. Ultrasonically condition the coal-solvent slurry for 10 min.
5. Transfer the content of the flask into inside chamber of the float-sink apparatus using small funnel; wash out the content of the flask with additional 44 mL of the solvent.
6. Add 121 mL of the solvent into outside chamber of the float/sink apparatus.
7. Do everything in duplicate.
8. Equalize the weight of both apparatus before centrifugation using the same solvent.
9. Place both apparatus into centrifuge and centrifugate 15 min at 1000 rpm and 10 min at 1300 rpm.
10. Filtrate the content of inside and outside chamber separately using Whatman paper No. 40 or 42 and wash out all the content carefully.
11. Dry filter papers in vacuum oven at 60⁰C to a constant weight.

12. Subject dry float and sink fraction for analysis.

A) 1000 RPM
the Inside chamber
remains closed

B) 1300 RPM
the spring is compressed
and the bottom of the inside
chamber is open

Figure A-1. Float-Sink Apparatus

1 - Outside chamber, 2 - Inside chamber,
3 - Conical plug and rod, 4 - Spring

Figure A-1. Float-Sink Apparatus

1 - Outside chamber, 2 - Inside chamber,
3 - Conical plug and rod, 4 - Spring

A-2. Batch-Scale Test Procedure

Experiments are performed in a 1L mixer equipped with variable speed stirrer. The tank contain four removable baffles. Radial four-blade, turbine type impeller on centrally located shaft provides agitation. Detailed data on high-shear mixer geometry is shown in Figure A-2.

Recovery of clean coal flocs is performed in a standard Denver flotation machine model D-12 equipped with a 2.5L flotation cell.

In a typical experiment, a weighed coal fines sample is placed in the stirrer tank and mixed with water to make up the required solids concentration. The resulting suspension is mixed at 1750 rpm and a predicted amount of bridging liquid (0.5% to 3%) is added. Bridging liquid (usually oil) is introduced to the coal slurry without emulsification. The slurry is agitated for a predetermined period of time, then transferred to the flotation cell and diluted. The volume of slurry is adjusted by adding water to achieve 6% to 10% solids concentration. The frother (when used) is added to the slurry and the slurry is conditioned for 60 seconds. The agitation rate is set at 1100 rpm and then air applied to the cell. Coal microagglomerates (flocs) are manually removed for six minutes.

Subsequently, the coal concentrate is dried at 40°C, weighed and analyzed for proximate composition and total sulfur (optional). For selected samples, if necessary, forms of sulfur and calorific values are also determined.

The tailings are filtered, weighed and analyzed for ash.

Figure A-2. Laboratory High-Shear Mixer
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Laboratory High-Shear Mixer

A-3. Analytical Procedures and Standards

Proximate Analysis:	Fisher Coal Analyzer (ASTM draft stage) ASTM D3174-93 and D3175-89a (Reapproved 1993)
Total Sulfur Analysis:	Leco SC-32 ASTM D 4239-85 High temperature combustion method with IR absorption procedure
Forms of Sulfur :	ASTM D 2492 (SO ₄ ——— S ICP) (Pyrite ——— Fe ICP)
Calorific Value:	ASTM D 2015
Particle Size Distribution:	Granulometer 723 (Laser Light Diffraction) or wet screening

The following laboratories provide analytical services for this project:

- Analytical Laboratory of National Centre for Upgrading Technology (NCUT),
Devon Research Centre, 1 Oil Patch Drive, Devon, Alberta

- Norwest Labs, 14203- 129 Avenue, Edmonton, Alberta

APPENDIX B

Batch Test Results

Table B-1. Test Conditions and Results for Sieve Bend Effluent (600 μm x 250 μm)

Moisture 23.4%

Ash Content 43.7%

High Shear Mixer - 1 min, 2000 rpm

Flotation - 3 min, 1100 rpm

Test	Cs	Oil Add.	Yield	C M Recovery	Product Ash	Ash Reduction	Tailings Ash
	%	%	%	%	%	%	%
DR- 1	20	0.5 Diesel	65.1	92.6	19.47	53.4	88.2
DR- 2	20	1.0 Diesel	65.8	94.4	18.46	57.8	88.1
DR- 5	20	0.5 MC+D	66.5	95.6	18.64	57.3	87.8
DR- 6	20	1.0 MC+D	66.0	94.6	18.62	57.4	88.2
DR- 9	30	0.5 Diesel	67.5	96.1	19.41	55.6	-
DR-10	30	1.0 Diesel	69.2	97.7	19.94	54.4	-
DR-13	30	0.5 MC+D	65.8	93.8	19.32	55.8	88.5
DR-14	30	1.0 MC+D	66.7	95.0	19.19	56.1	87.5
DR-17*	30	0.5 Diesel	65.1	95.1	17.33	60.3	88.1
DR-18*	30	1.0 Diesel	67.0	95.8	18.67	57.3	88.1
DR-21*	30	0.5 MC+D	64.0	93.1	17.6	59.7	88.9
DR-22*	30	1.0 MC+D	67.2	94.8	19.74	54.8	88.1

* - no frother added

Table B-2. Test Conditions and Results for Cyclone Overflow (250 µm x 0)

Moisture 34.4%
Ash Content 30.6%
High Shear Mixer - 1 min, 2000 rpm
Flotation - 3 min, 1100 rpm

Test	Cs %	Oil Add. %	Yield %	C M Recovery %	Product Ash %	Ash Reduction %	Tailings Ash
DR-3	20	0.5 Diesel	76.9	94.2	14.81	51.6	88.0
DR-4	20	1.0 Diesel	76.5	93.5	14.43	52.8	88.5
DR-7	20	0.5 MC+D	77.0	94.7	14.52	52.5	88.2
DR-8	20	1.0 MC+D	76.5	95.7	12.90	57.8	88.6
DR-11	30	0.5 Diesel	80.0	96.9	15.65	48.9	88.6
DR-12	30	1.0 Diesel	81.3	97.3	16.33	46.6	88.5
DR-15	30	0.5 MC+D	82.3	97.7	17.31	43.4	-
DR-16	30	1.0 MC+D	83.0	97.2	18.21	40.5	89.5
DR-19*	30	0.5 Diesel	80.2	97.4	15.52	49.3	-
DR-20*	30	1.0 Diesel	79.1	95.1	15.99	47.7	-
DR-23*	30	0.5 MC+D	79.4	96.1	15.75	48.5	-
DR-24*	30	1.0 MC+D	82.2	99.2	15.76	48.5	-

* - no frother added