

**Final Report on the Integration of the GranuFlow Process  
With a Packed Flotation Column, CRADA 98-F020**

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## **BACKGROUND**

The Department of Energy's National Energy Technology Laboratory (NETL) entered into a Cooperative Research and Development Agreement (CRADA) with Mineral Technologies International, Incorporated (MTI) in 1998 for the purpose of experimenting with the integration of the GranuFlow Process with a packed flotation column for fine coal processing. NETL scientists had previously developed and patented the GranuFlow Process which utilizes bitumen emulsions to dewater fine coal while reducing dustiness and the loss of fines. Further development and experimentation of GranuFlow with outside partners is an ongoing activity.

MTI was awarded a Phase II SBIR grant for a project entitled "An Integrated Process for the Recovery of Coal Fines from Waste Streams." The objective of this project was to demonstrate at the pilot scale a novel process system which integrated the high performance of the MTI packed column flotation cell with the dewatering ability of the GranuFlow Process. With the development of CRADA 98-F020, NETL worked with MTI in support of the project.

NETL's role was to provide technical information and input to the project with regard to the GranuFlow Process and its application to the proposed project, and to conduct GranuFlow Process testing. Information about the chemistry and interactions of coal, emulsions, and frothers were analyzed to determine optimum experimental conditions. Standard procedures for testing the dustiness and moisture content of the treated coal product were also outlined by NETL.

NETL also advised MTI about the types and amounts of bitumen emulsions that might have performed well in the proposed coal dewatering tests. Based on extensive laboratory experiments using NETL standard procedures and equipment, five emulsions were recommended for testing by MTI. Small-scale coal dewatering tests using these emulsions produced favorable results. Larger dewatering tests using the solid- and screen-bowl centrifuges were then conducted at the NETL facility during April 2000.

## **EXPERIMENTAL APPROACH**

MTI supplied NETL with the coal samples for the centrifuge tests from the Sentinel Plant, near Morgantown WV, where the packed column was tested. Both a flotation concentrate sample and a mixture of flotation and spiral concentrates were tested, in order to look at the effectiveness of emulsion addition on two different plant streams. Two bitumen emulsions were used in the centrifuge tests and are described below.

### **Test materials**

As described above, two coal slurries were used in the test series. The flotation product sample was 16% solids, and had a particle size of about 100 mesh by 0. The mixture of flotation and spiral concentrates consisted of approximately 19% solids, and had a particle size of about 16 mesh by 0. The coal slurries used in each test will be identified as flotation or mixed

concentrate. The two emulsions used in testing were made by Bitor (70% bitumen by weight) from Venezuela and an asphalt emulsion by Monsey (60% bitumen by weight) from Pennsylvania, USA. Both of these emulsions had been used and tested before in previous GranuFlow tests at NETL.

**Centrifuge and feed system configurations**

*Screen bowl centrifuge* -- The screen bowl centrifuge used was a 6” x 12” Bird centrifuge with a 5” x 3.75” screen section, 2” long beach zone, 0.5” pool depth, 2500 rpm bowl rotation, 100:1 conveyor differential, feed ports 2.75” from beach zone, and 35 mesh screen. A 50-gallon feed tank was used with a Turbon mixer and centrifugal recirculation pump. The coal slurry was fed to the centrifuge using a 6 gpm Moyno pump and a Micromotion mass flow meter. The pump used to inject the emulsions was a 0.1 gpm pump which fed into a 0.5” x 20’ feed line. The injection point was at the beginning of the feed line, right after the Micromotion, in order to maximize the mixing of the emulsion with the coal slurry. This is an important factor in successfully implementing the GranuFlow Process.

*Solid bowl centrifuge* -- The solid bowl centrifuge used was a 14” Sharples hi-g centrifuge. A 100 gallon feed tank was used with mixer and inversion recirculation for mixing. The coal slurry was fed at a constant rate using a calibrated pump. The pump used for emulsion injection was a 0.1 gpm pump, which fed into the beginning of a 0.5” x 45’ feed line to maximize mixing.

**Test series**

Four series of tests were run utilizing the flotation concentrate and the mixed concentrate. Both the screen-bowl and solid-bowl centrifuges were used to remove water from the coal samples. Each test series consisted of treating the coal slurries with 0, 4, and 8% of the emulsions. The details of the testing conditions are shown in the table below. All tests were run at approximately 20 degrees C.

**Table 1. Test series conditions for MTI tests**

Test series	1	2	3	4
Centrifuge used	Screen-bowl	Screen-bowl	Solid-bowl	Solid-bowl
Slurry flowrate (gpm)	3	3	4	4
Solids feedrate (gram/min)	1895	2269	2527	3025
Coal sample used	Flotation	Mixed	Flotation	Mixed
Feed solids, %	15.2	20.4	16.7	18.8
Dry ash, %	5.0	8.2	5.1	9.1

## Procedures for analysis of samples

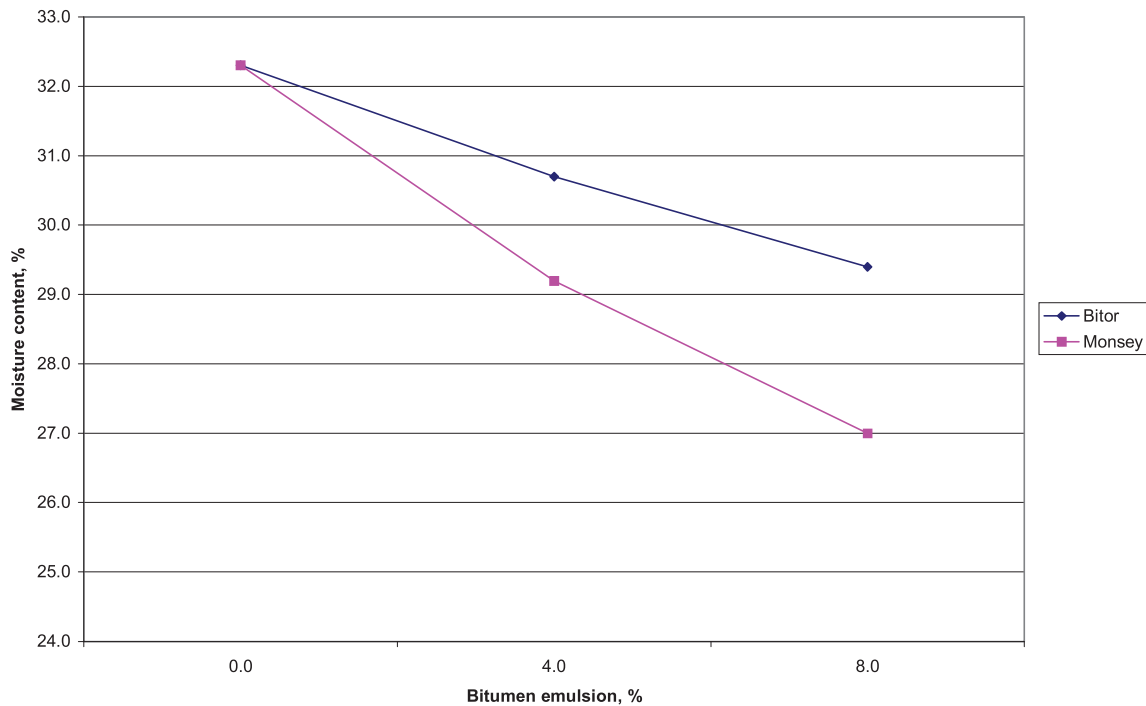
*Moisture content* -- The moisture contents of the product coal samples from each test were determined by placing the coal in a pan which was placed in a drying oven at 105 degrees C. The pans were removed and weighed several times until no change in the weight was noted.

*Dust index* -- The dust index is defined as the percent mass of a coal sample which passes through a 100 mesh screen. The coal sample is placed in a series of mesh screens on a RoTap for 5 minutes. The fractions in each screen are removed and weighed.

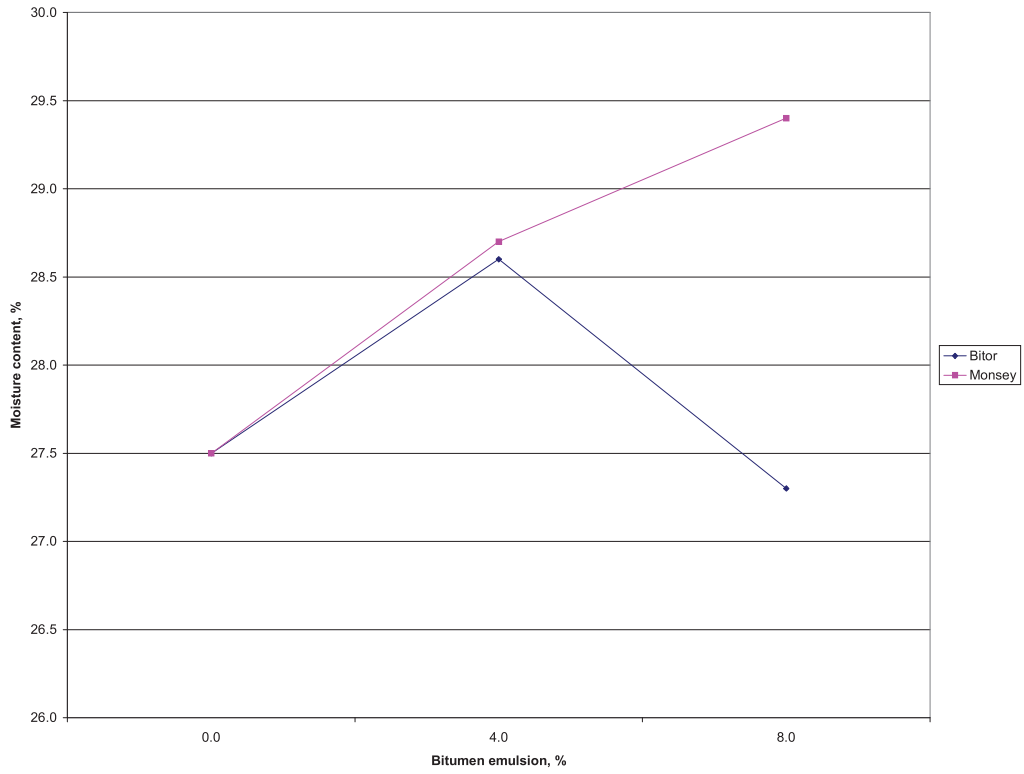
## RESULTS AND DISCUSSION

### Moisture reduction

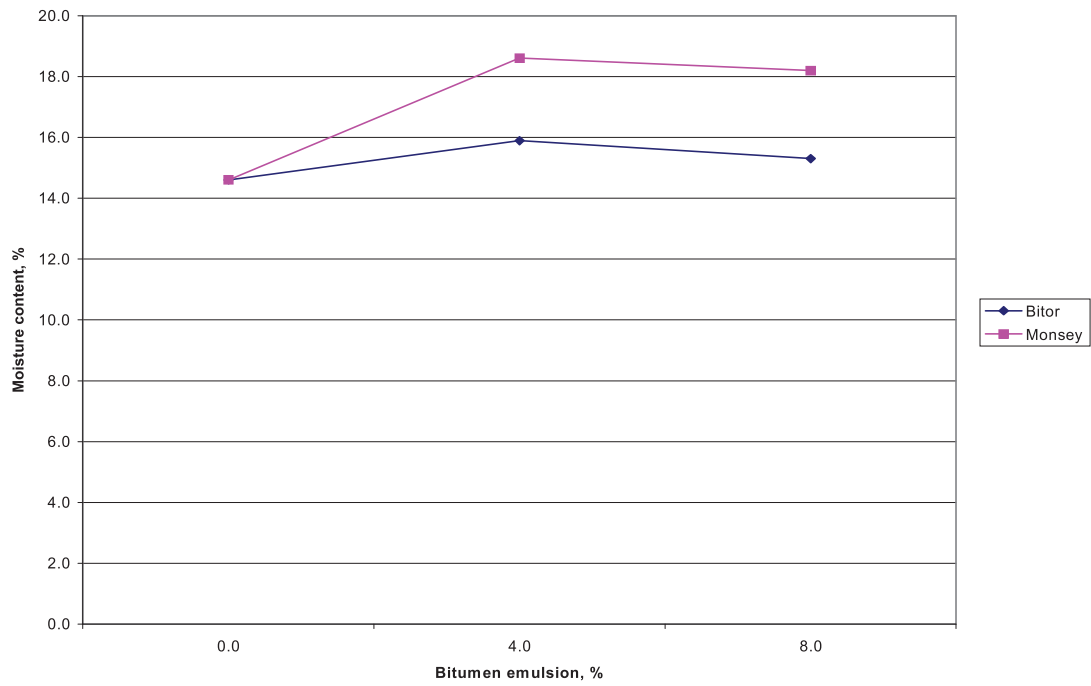
The moisture contents of the untreated and bitumen-treated coals for each of the four tests are shown in Figures 1-4.



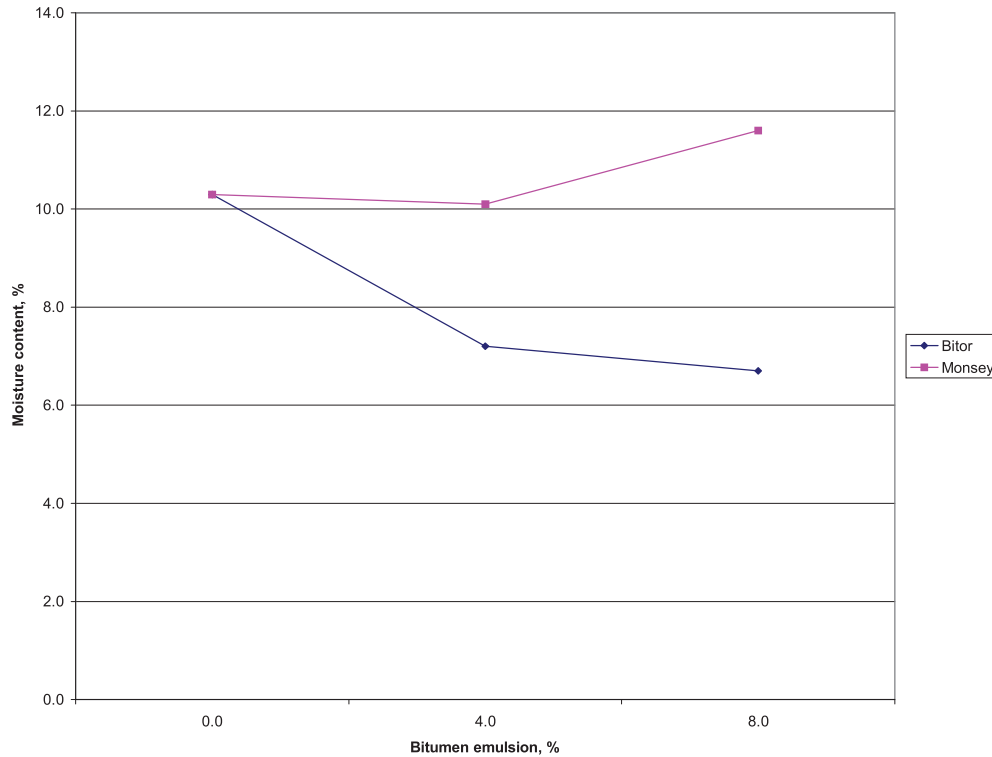
**Figure 1. Moisture content of flotation product dewatered with screen bowl centrifuge**



**Figure 2. Moisture content of flotation product dewatered with solid bowl centrifuge**



**Figure 3. Moisture content of mixed concentrate dewatered with screen bowl centrifuge**



**Figure 4. Moisture content for mixed concentrate dewatered with solid bowl centrifuge**

The moisture content results of the treated samples varied greatly. The flotation product dewatered with the screen-bowl centrifuge had lowered moisture content with both emulsions at both dosage rates, as shown in Figure 1. The Monsey 8% emulsion dosage produced the lowest moisture content of 27% versus 32.3% in the untreated flotation concentrate. That is a 16% reduction in moisture for the treated coal. The mixed concentrate treated with the emulsions and dewatered with the screen-bowl centrifuge did not have a reduction in moisture content, as shown in Figure 3. Instead, the moisture actually increased slightly with the 4% and 8% additions of both emulsions.

The flotation concentrate dewatering tests with the solid-bowl centrifuge show mixed results. Figure 2 indicates that with both 4% emulsion dosage rates, the moisture content increased slightly from 27.5% to around 28.5%. The 8% Monsey addition showed a further increase to almost 29.5% while the 8% Bitor addition resulted in a slight decrease to 27.3% moisture. Differences of  $\pm 1$  percentage point or so are generally within experimental error. The final moisture contents for the mixed concentrate dewatered with the solid-bowl centrifuge in Figure 4 show the Bitor additions decreasing the moisture from 10% to 7.2% and 6.7% respectively for the 4% and 8% dosages. The Monsey emulsion added some moisture to the coal as the final treated product contained more water than the initial concentrate. In some cases, slight increases in product moisture with the addition of emulsion may be due to the fact that the recovery of fine coal to the product is increasing.

## Dust index results

The results of the dust index analysis are shown in Tables 2-5 . Results for test series 1 to 4 include the untreated coal as well as the treated products of both emulsions at both dosage rates.

**Table 2. Test 1 dust index results of flotation concentrate coal**

Emulsion added, %	Dust index (% -100 mesh)
0%	49.7
Bitor, 4%	23.2
Bitor, 8%	8.9
Monsey, 4%	29.5
Monsey, 8%	11.2

**Table 3. Test 2 dust index results of mixed concentrate coal**

Emulsion added, %	Dust index (% -100 mesh)
0%	15.5
Bitor, 4%	9.2
Bitor, 8%	6.2
Monsey, 4%	9.1
Monsey, 8%	7.3

**Table 4. Test 3 dust index results of flotation concentrate coal**

Emulsion added, %	Dust index (% -100 mesh)
0%	52.3
Bitor, 4%	25.6
Bitor, 8%	9.2
Monsey, 4%	27.7
Monsey, 8%	12.0

**Table 5. Test 4 dust index of mixed concentrate coal**

Emulsion added, %	Dust index (% -100 mesh)
0%	17.1
Bitor, 4%	9.9
Bitor, 8%	6.5
Monsey, 4%	13.8
Monsey, 8%	8.1

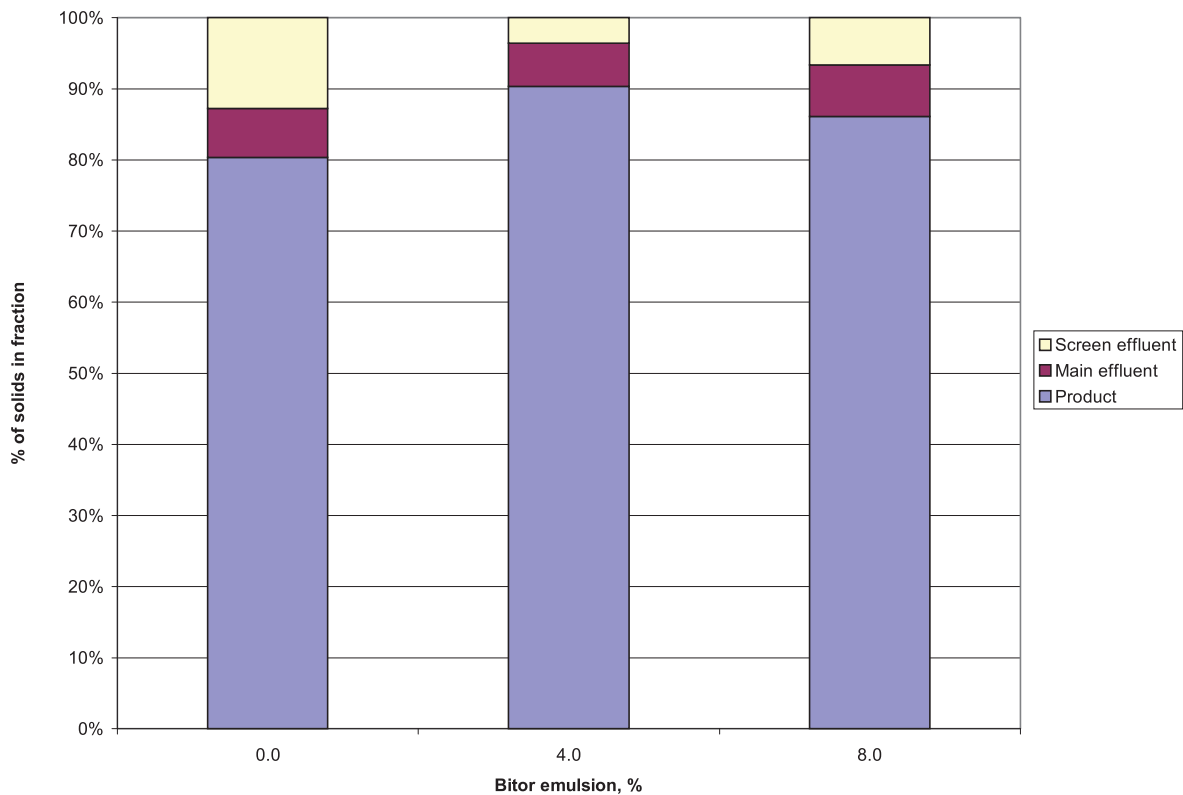
The dust index results were quite similar for the coal samples dewatered with both centrifuges. The flotation concentrate treated with the 4% emulsion addition had a reduction in dust of approximately 50% while the 8% additions reduced dust by about 75%. The Bitor emulsion reduced dust slightly more than the Monsey emulsion.

The mixed concentrate treated with 4% Bitor emulsion displayed a dust reduction of 41 to 45%, while the 8% Bitor treatment showed a dust reduction of 60%. The Monsey emulsion treatment produced slightly less reduction in dust, with 4% and 8% dosages achieving reductions of 18 to 41% and 53%, respectively.

### Solids distributions

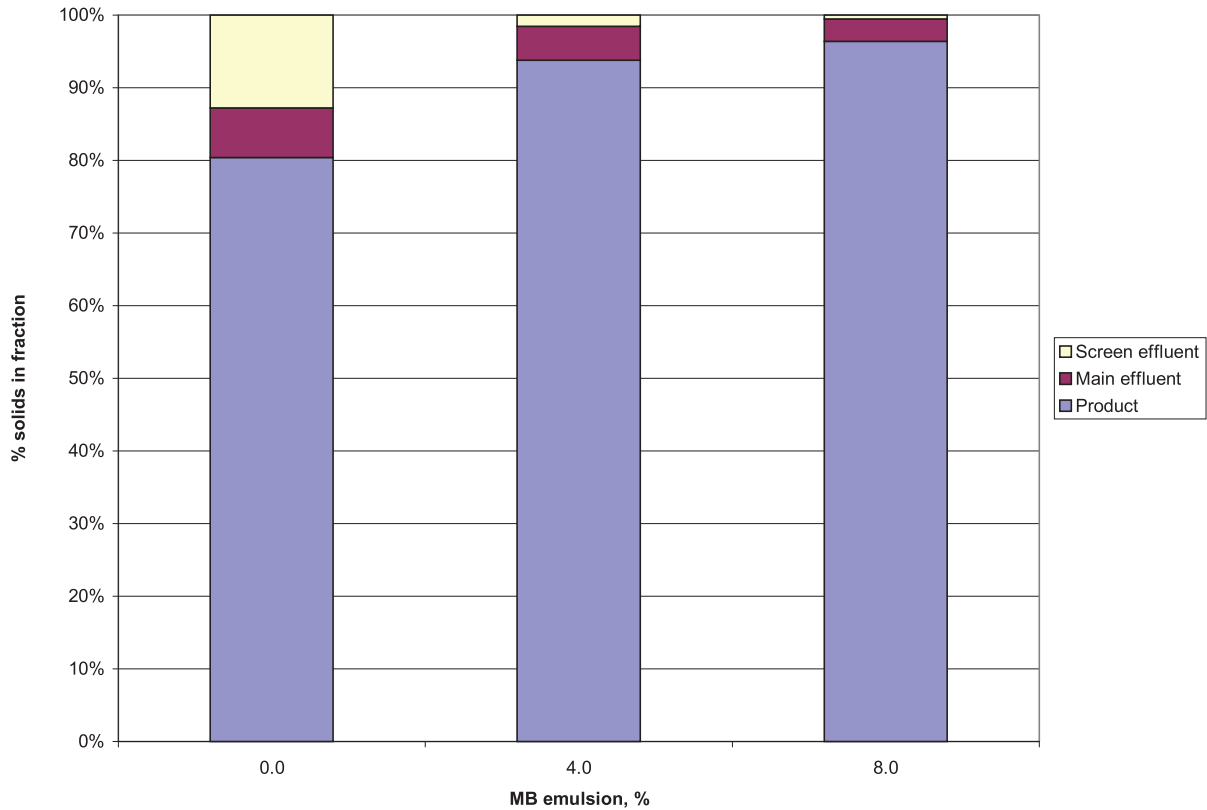
The effect of the GranuFlow Process on distributions of solids in the untreated and treated coals for the screen-bowl centrifuge tests are shown in Figures 5-8 .

In general, treating the flotation concentrate with either emulsion resulted in a substantial increase in the amount of solids in the product, with a marked reduction of solids in one of the effluents. For the Bitor emulsion, the solids were removed primarily from the screen effluent, as seen in Figure 5.



**Figure 5. Solids distribution for flotation product treated with Bitor emulsion and dewatered with the screen-bowl centrifuge**

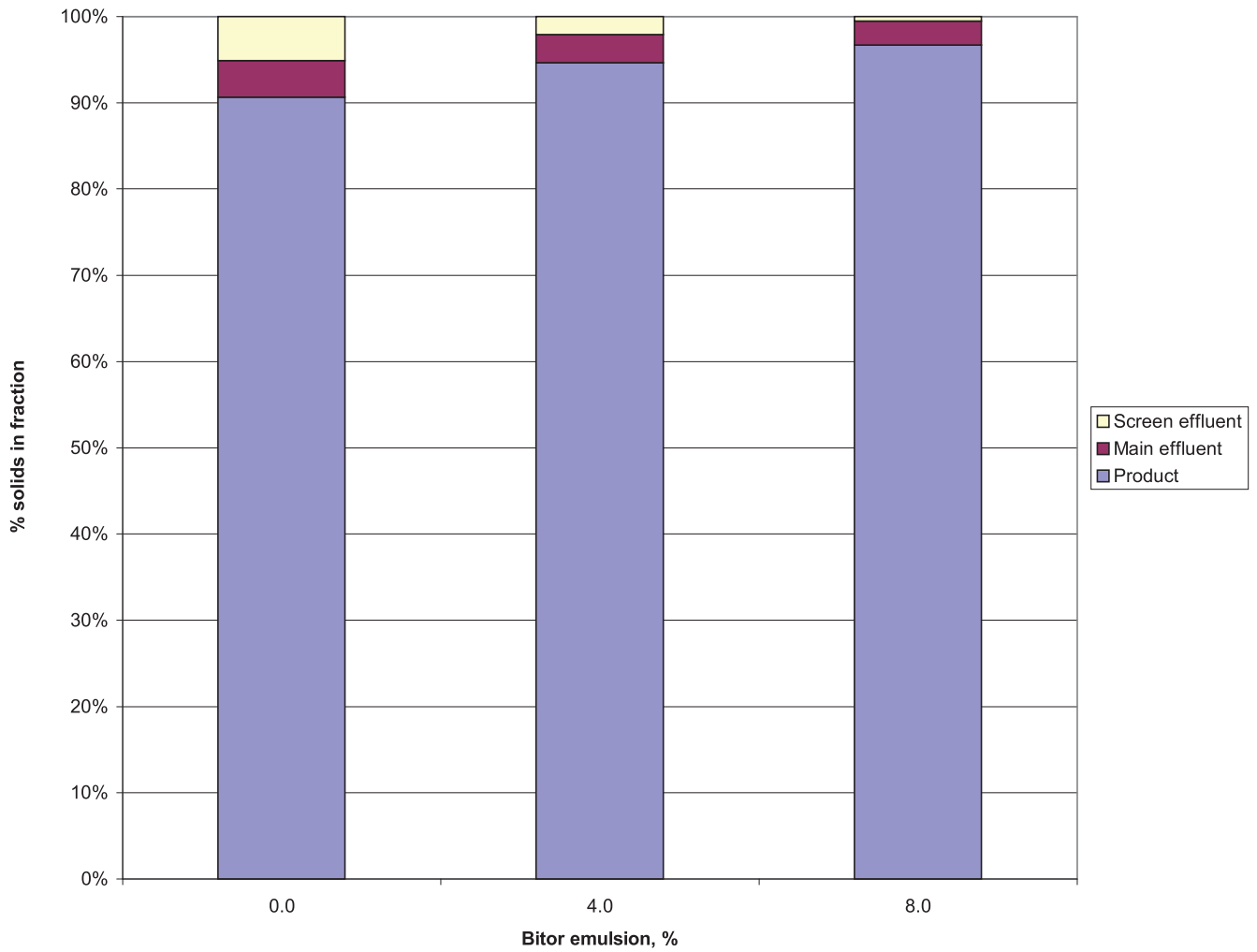
The Monsey emulsion treatments reduced the amounts of solids in both the main and screen effluents, as seen in Figure 6.



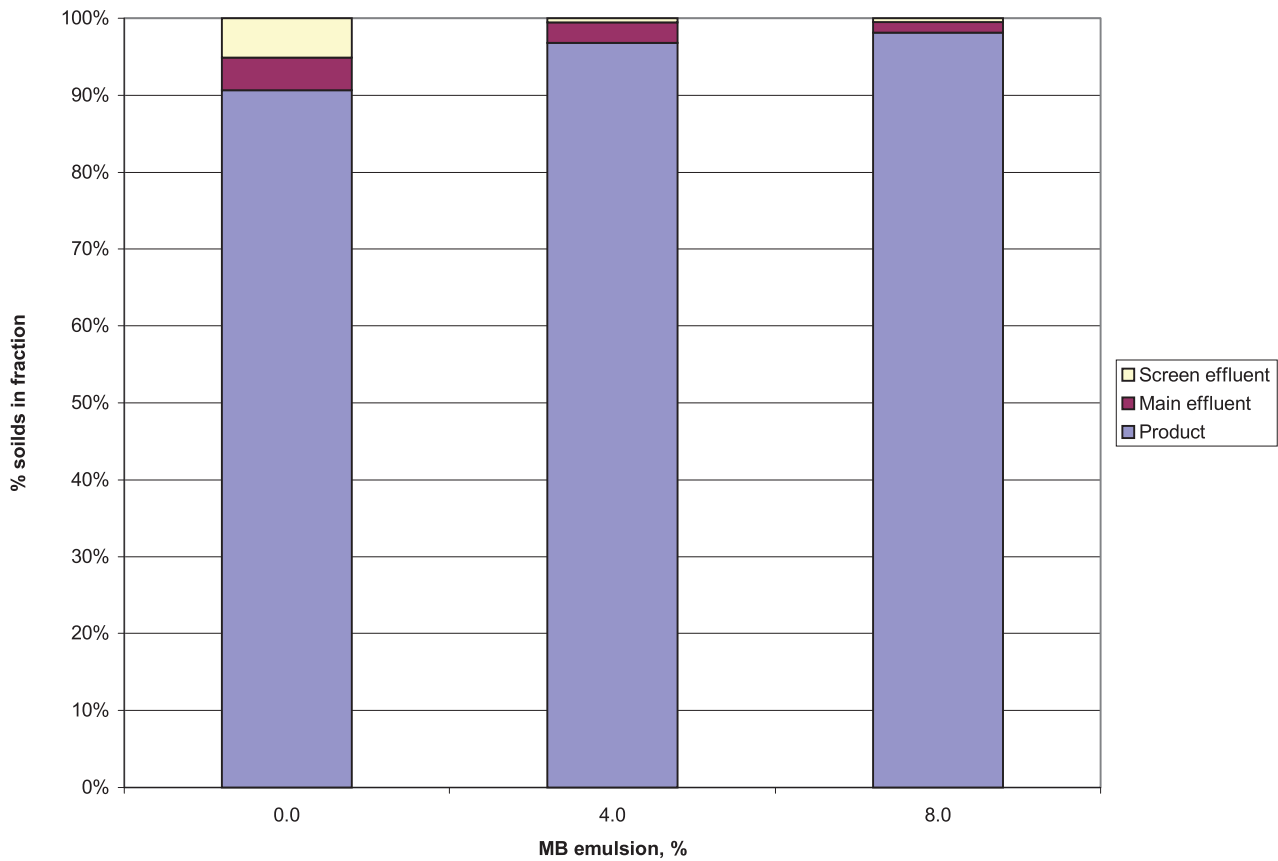
**Figure 6. Solids distribution for flotation product treated with Monsey emulsion and dewatered with the screen-bowl centrifuge**

The results for the solids distribution are more consistent with the mixed concentrate dewatering tests. Figures 7 and 8 show that both the Monsey and Bitor emulsions treatments resulted in increases in the percent of solids in the product while removing solids from the other effluents.

For the solid-bowl centrifuge dewatering tests, all samples of both treated and untreated coals had greater than 99% of the solids in the product with less than 1% solids in the main effluent (see raw data in Appendix.)



**Figure 7. Solids distribution for mixed concentrate treated with Bitor emulsion and dewatered with the screen-bowl centrifuge**



**Figure 8. Solids distribution for mixed concentrate treated with Monsey emulsion and dewatered with the screen-bowl centrifuge**

## CONCLUSIONS

Based on the results from the few tests that were run, treating the coal concentrates with bitumen emulsion did not appear to produce consistent moisture results. Historically, the GranuFlow Process provides some improvement in product moisture. The moisture content of the treated coal product varied not only between emulsions but among the different dosages of the same emulsion. In some cases, emulsion treatment showed a slight increase, in others there was a negligible effect, and in others there was a marked decrease. Overall, a slight decrease in moisture content was noted with most emulsion treatments for both the flotation and mixed concentrates.

Both centrifuges produced coal products with significantly lower dust indices when treating with the same emulsion dosages. The type of centrifuge used did not appear to drastically affect the dust index results. The Bitor emulsion was more efficient at reducing the production of dust than the Monsey emulsion. The dust index was further reduced with an increase in the emulsion dosage rate. The flotation concentrate had a greater reduction in dust than the mixed concentrate, although the flotation samples initially contained more fine coal than the mixed samples.

The addition of a bitumen emulsion at 4% or 8% did significantly increase the percent solids recovered in the product streams of the screen-bowl centrifuge. This is beneficial as less material is lost in the effluent streams. The fine coal particles may have become agglomerated with the bitumen and other particles and less likely to be lost through the centrifuge screen section. The solid-bowl centrifuge results showed that an emulsion dosage may not be needed as greater than 99% of the solids are recovered with or without the expense of adding the emulsion.

In addition, in some cases, there appeared to be selective recovery of coal as opposed to mineral matter, as indicated by lower ash contents in the product solids, or higher ash contents in the effluent solids.

These cooperative tests have shown that implementing the GranuFlow Process with this particular coal had generally positive, if somewhat mixed, results summarized as follows: 1) moisture content results were inconsistent but showed some promise for improving product moisture, 2) the significant increase in recovery of solids makes the addition of a bitumen emulsion worthwhile if the costs of adding the emulsion can be offset by the increased product, 3) the marked decrease in dust index provides for a product that is easier to handle and transport.

## **Appendix**

### **Raw Data for NETL CRADA 98-F020**

Test 1

<b>CENTRIFUGE TYPE</b>	screen	screen	screen	screen	screen	screen
<b>BITUMEN EMULSION</b>						
type	(none)	Bitor	Bitor	(none)	Monsey	Monsey
dosage, %	0.0	4.0	8.0	0.0	4.0	8.0
<b>FEED SLURRY</b>						
concentrate type	flotation	flotation	flotation	flotation	flotation	flotation
flow rate, gpm	3.1	3.2	4.1	3.1	3.4	3.8
% solids	15.2	15.2	15.2	15.2	15.2	15.2
dry % ash	5.0	5.0	5.0	5.0	5.0	5.0
<b>PRODUCT</b>						
% water	32.3	30.7	29.4	32.3	29.2	27.0
dry % ash	4.0	4.0	3.9	4.0	4.1	4.0
<b>MAIN EFFLUENT</b>						
% solids	1.4	1.3	1.4	1.4	1.1	0.7
dry % ash	18.3	18.1	17.1	18.3	21.8	19.5
<b>SCREEN EFFLUENT</b>						
% solids	35.6	14.2	22.2	35.6	6.8	1.8
dry % ash	5.1	5.8	5.2	5.1	6.5	8.9
<b>SOLIDS DISTRIBUTION</b>						
product %	80.3	90.4	86.2	80.3	93.8	96.4
main effluent %	6.9	6.1	7.2	6.9	4.7	3.1
screen effluent %	12.8	3.6	6.7	12.8	1.5	0.5
<b>PRODUCT DUSTINESS</b>						
dust index, %	49.7	23.2	8.9	49.7	29.5	11.2

Test 2

<b>CENTRIFUGE TYPE</b>	screen	screen	screen	screen	screen	screen
<b>BITUMEN EMULSION</b>						
type	(none)	Bitor	Bitor	(none)	Monsey	Monsey
dosage, %	0.0	4.0	8.0	0.0	4.0	8.0
<b>FEED SLURRY</b>						
concentrate type	mixed	mixed	mixed	mixed	mixed	mixed
flow rate, gpm	3.1	3.1	2.8	3.1	3.2	3.2
% solids	20.4	20.4	20.4	20.4	20.4	20.4
dry % ash	8.2	8.2	8.2	8.2	8.2	8.2
<b>PRODUCT</b>						
% water	14.6	15.9	15.3	14.6	18.6	18.2
dry % ash	8.6	8.7	8.4	8.6	8.8	8.2
<b>MAIN EFFLUENT</b>						
% solids	1.1	0.9	0.8	1.1	0.6	0.3
dry % ash	22.9	27.4	27.8	22.9	23.3	25.1
<b>SCREEN EFFLUENT</b>						
% solids	31.6	11.9	2.9	31.6	2.1	1.8
dry % ash	13.0	14.7	23.2	13.0	14.3	9.8
<b>SOLIDS DISTRIBUTION</b>						
product %	90.6	94.7	96.8	90.6	96.9	98.3
main effluent %	4.2	3.3	2.8	4.2	2.7	1.4
screen effluent %	5.1	2.1	0.5	5.1	0.5	0.5
<b>PRODUCT DUSTINESS</b>						
dust index, %	15.5	9.2	6.2	15.5	9.1	7.3

### Test 3

<b>CENTRIFUGE TYPE</b>	solid	solid	solid	solid	solid	solid
<b>BITUMEN EMULSION</b>						
type	(none)	Monsey	Monsey	(none)	Bitor	Bitor
dosage, %	0.0	4.0	8.0	0.0	4.0	8.0
<b>FEED SLURRY</b>						
concentrate type	flotation	flotation	flotation	flotation	flotation	flotation
flow rate, gpm	4.0	4.0	4.0	4.0	4.0	4.0
% solids	16.7	16.7	16.7	16.7	16.7	16.7
dry % ash	5.1	5.1	5.1	5.1	5.1	5.1
<b>PRODUCT</b>						
% water	27.5	28.7	29.4	27.5	28.6	27.3
dry % ash	4.4	4.5	4.8	4.4	4.5	4.5
<b>MAIN EFFLUENT</b>						
% solids	0.2	0.2	0.2	0.2	0.2	0.2
dry % ash	73.1	74.7	74.2	73.1	77.1	78.2
<b>SCREEN EFFLUENT</b>						
% solids	n/a	n/a	n/a	n/a	n/a	n/a
dry % ash	n/a	n/a	n/a	n/a	n/a	n/a
<b>SOLIDS DISTRIBUTION</b>						
product %	>99	>99	>99	>99	>99	>99
main effluent %	<1	<1	<1	<1	<1	<1
screen effluent %	n/a	n/a	n/a	n/a	n/a	n/a
<b>PRODUCT DUSTINESS</b>						
dust index, %	52.3	27.7	12.0	52.3	25.6	9.2

## Test 4

<b>CENTRIFUGE TYPE</b>	solid	solid	solid	solid	solid	solid
<b>BITUMEN EMULSION</b>						
type	(none)	Bitor	Bitor	(none)	Monsey	Monsey
dosage, %	0.0	4.0	8.0	0.0	4.0	8.0
<b>FEED SLURRY</b>						
concentrate type	mixed	mixed	mixed	mixed	mixed	mixed
flow rate, gpm	4.0	4.0	4.0	4.0	4.0	4.0
% solids	18.8	18.8	18.8	18.8	18.8	18.8
dry % ash	9.1	9.1	9.1	9.1	9.1	9.1
<b>PRODUCT</b>						
% water	10.3	7.2	6.7	10.3	10.1	11.6
dry % ash	9.5	9.4	9.8	9.5	9.6	9.4
<b>MAIN EFFLUENT</b>						
% solids	0.1	0.1	0.1	0.1	0.1	0.1
dry % ash	65.9	69.4	71.7	65.9	72.0	83.0
<b>SCREEN EFFLUENT</b>						
% solids	n/a	n/a	n/a	n/a	n/a	n/a
dry % ash	n/a	n/a	n/a	n/a	n/a	n/a
<b>SOLIDS DISTRIBUTION</b>						
product %	>99	>99	>99	>99	>99	>99
main effluent %	<1	<1	<1	<1	<1	<1
screen effluent %	n/a	n/a	n/a	n/a	n/a	n/a
<b>PRODUCT DUSTINESS</b>						
dust index, %	17.1	9.9	6.5	17.1	13.8	8.1