

PROGRESS REPORT
INVESTIGATION OF PAPERMILL SLUDGE
AS A TACONITE BINDER

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Introduction

Bentonite has been used by the taconite industry since commercialization of the process in the early 1950's. Although many alternate binders have been tested, including organic materials, bentonite still remains the preferred binder. Bentonite has the unique qualities that promote stable green balling conditions and provides acceptable strengths during drying, preheating, and contributes to the fired pellet strength at an acceptable cost. The incentive to consider other binders is based on such factors as improving the metallurgical properties of the pellets, decreasing their silica content, and lower overall costs.

The USBM conducted a number of small scale tests using papermill sludge and other additives in place of bentonite as a binder for taconite pellets. Their balling tests indicated that acceptable green-ball strength could be achieved with papermill sludge. The fired pellet evaluations, however, were based on muffle furnace tests which do not accurately predict the fired pellet quality. Although the fired pellet data were also encouraging it was recognized that pot-grate tests were needed to obtain more definitive test data that could be directly related to commercial practice. Subsequently, the USBM recommended that the IRRRB fund an extension of the program to include pot-grate tests to verify the fired pellet quality obtained in the muffle furnace work. A test program was prepared by the Coleraine Research Laboratory of the Natural Resources Research Institute (NRRI) in cooperation with Dr. J. Nigro and Dr. L. Haas, USBM, and Mr. S. Dickinson, IRRRB. The contract proposal was submitted to the IRRRB January 27, 1988. The contract included evaluation of concentrate and sludge filtering and blending aspects, green-ball quality, and fired pellet quality based on pot-grate tests. This report presents data on all three aspects as stated in the contract and conclusions regarding future efforts in this area.

Raw Material

The concentrate for the pelletizing tests was obtained from National Steel Pellet Company in Keewatin, MN and the papermill sludge from the Grand Rapids Waste Treatment Filtration Plant. The sludge filter cake from the filtration plant is reportedly over 90 percent papermill sludge. Analyses of the papermill sludge and bentonite binders are given in Table I. The sludge filter cake contains 73 percent water. The dried sludge has 78 percent loss-on-ignition (LOI). Most of the LOI is from the organic material (primarily wood fibers) that would burn out during pelletizing. Analysis of the residual ash from the LOI, representing only 5.9 percent of the as received sludge filter cake, shows high amounts of Al_2O_3 and SiO_2 reflecting the kaolin clay used in papermaking. Size analysis of the sludge was not conducted due to the fibrous nature of this material. The sludge contains a high proportion of coarse wood fibers that interfere with the balling step and are difficult to assimilate into the green balls. The concentrate filter cake obtained from National Steel was 83.4 percent minus 270 mesh, contained 8.9 percent moisture, and had a Blaine surface area of 1719 cm^2/g (Table II).

Test Program and Procedures

The primary objective of the test program was to establish the quality of fired taconite pellets that could be produced with papermill sludge as the binder. In addition, to help the practicality of using papermill sludge, it was necessary to determine if it would be practical to add the sludge to the concentrate as a slurry prior to filtering, or whether partial drying and pulverizing combined with intensive mixing would be required. Green-ball quality was also a critical parameter to be tested. Evaluation of the sludge as a binder was based on a comparison to baseline data generated with bentonite as the binder. Detailed procedures for the filtering, balling, and pot-grate testing are given in the following discussion.

Bench-Scale Filter Tests. These tests were carried out using a 0.1 ft^2 test-leaf apparatus. Feed material was slurried to the desired density and temperature in 4000-gram batches. The slurry was continuously agitated with a variable speed mixer that was slowed down during the form time. The form and dry times for the disc speeds of 0.77, 1.0, and 1.43 rpm were based on a standard filter cycle. A vacuum of 25 to

26 inches of Hg was normally used as this level is required in the laboratory to produce cake moistures similar to the plant due to the large edge effect on the 0.1 ft² leaf. The filter cloth used for this test work was the basic nylon bag designated as 801RF. The papermill sludge was first dispersed in a 4 percent slurry with a Waring blender before it was added to the concentrate slurry. This was the highest percent solids that could be mixed without stalling the blender which represents a real problem in plant application. An addition rate of 1.4 percent papermill sludge on a dry basis was used in all the tests.

Batch Balling Tests. These tests were carried out with an open airplane tire and a standard procedure followed for all tests. Basically, the filter cake (as received moisture) and binder were premixed in an intensive mixer-muller for 2 minutes with enough water to give close to the target moisture. Binder addition was calculated on a dry concentrate basis. This material was then used in the 6-inch by 12-inch airplane tire to produce a batch of seed pellets which were sized at minus 3 plus 4 mesh. A set weight (125 grams) of seeds was returned to the tire and the remainder of the feed was added at an increasing rate to achieve the desired ball size by the end of 3 minutes. An additional 1 minute reroll period was given to complete the procedure. Green balls were then hand screened at minus 1/2 inch plus 7/16 inch to produce the balls for physical testing and moisture determinations. A portable water spray containing distilled water was used during the balling to control moisture level and ball growth. On each batch of sized pellets the moisture, 18-inch drop strength, wet compression, and dry compressions were measured using 20 pellets for each test. The dry strength was measured on balls from the moisture samples while still warm. Wet and dry compression strengths were measured on a mechanically operated spring scale, which indicates the force in pounds at the moment of ball breakage.

When possible a series of three tests were carried out at different moisture levels. However, it was impossible to achieve a low moisture level with the wet or partially dried sludge. The drop-test values from these tests is then used to determine a linear regression and the value for any given moisture can be calculated. Usually the drop strength at 9.2 and 9.5 percent moisture are chosen for comparison. Wet and dry compression are primarily dependent on the level of binder and therefore, an average value for the moisture series was calculated.

Pot-Grate Tests. Production of green balls for the pot-grate tests was carried out by a standard procedure. Binder additions were calculated on a dry basis as a percentage of the concentrate weight. Approximately 200 pounds of concentrate and the appropriate amount of additives were added to a Simpson mixer-muller and mixed for several minutes. The wet mix was balled in a laboratory 36-inch-diameter disc with alternating additions of spray water and feed. After a sufficient number of pellets had formed, the load was removed from the disc and screened. The minus 1/2-inch plus 7/16-inch balls were stored in a closed container until the necessary 70 pounds for a pot-grate test was produced.

A sample of the green balls from each test was reserved for moisture analysis, compression, and drop tests. Twenty balls were used for each of these quality tests and were carried out by the same methods as in the batch balling tests.

The green balls were transferred to a McKee-type pot-grate apparatus for induration. This apparatus consists of a 1-sq-ft cross sectional area, 16-inch deep, refractory-lined pot with grate bars to support the charge. The pot has a windbox and hood with attendant fan and combustion chamber to supply the processing gases. Oxygen is added to the combustion gases to give an oxidizing atmosphere during preheating and firing (about 15% O₂). The apparatus has thermocouples for controlling the temperature and to indicate the bed-temperature profile. Pressure taps are provided to maintain the desired constant pressure drop during processing. A 6-inch bed of green balls was charged to the pot atop a 2-inch hearth layer of fired pellets. The sequence of firing was downdraft drying, preheating, and firing at the desired temperatures. After updraft cooling, the pellets were removed from the core of the bed to avoid wall effect and samples were split out for product-quality tests, such as compression, tumble, reducibility, low-temperature breakdown, and chemical analyses. The tumble tests were by the standard ASTM procedure on a 25-pound sample while the fired pellet compression was an average of 60 pellets crushed with an automated machine according to the ASTM procedure.

Test Results

The results of the tests are presented for the three phases of work carried out. This included filtering, balling, and pot-grate pelletizing tests. Base case tests for each phase without sludge were also conducted for comparison purposes.

Bench-Scale Filtering Tests. The first series of filter leaf tests was conducted without sludge. A concentrate slurry was produced at 60 percent solids to provide baseline data. Tests 1 to 4 were run to establish a relationship between filter rate and filter cake moisture that would be in the range produced at the plant. At 60 percent solids in the feed the filter cake moisture ranged from 8.7 to 9.2 percent when the filter rate was varied from 320 to 494 lb/hr/ft² as shown in Table III. These tests are consistent with plant practice because the filter cake received from the plant contained 8.9 percent moisture. Since the papermill sludge contains about 73 percent water it was suggested that the most convenient method of mixing with the concentrate would be in the slurry form. Therefore, the sludge was repulped with water in a blender at 4 percent solids. This slurry was extremely viscous and appeared to be the highest density that would be practical. In Tests 5 to 10 the sludge was added as a slurry to the concentrate slurry. The combined filter feed slurry was held at 60 percent solids and contained 1.4 percent sludge on a equivalent dry basis. The results of these tests show that at filter rates of 236 to 354 lb/hr/ft² the filter cake moisture was very high at 11.6 to 13.1 percent. The sludge addition produced a thick and viscous slurry feed and the cake thickness was high even at simulated faster disc speeds. Air holes or bubbles also seem to form in the cake. With less form time and a thinner cake, the moisture was even higher than with normal form time. Thus, at similar capacities, the sludge added over 3 percent moisture to the filter cake which would be unacceptable in a plant because of balling problems and collapse on the grate during drying.

In the next series of tests, the 4 percent sludge slurry was added to a 65 percent solids concentrate slurry. The resulting filter feed, at 54 percent solids, was used for Tests 11 to 15 (Table III). Varying the rate from 196 to 282 lb/hr/ft² resulted in filter cake moistures from 11.2 to 11.8 percent. Thus, a 50 to 100 percent reduction in rate still produced a moisture content about 2 percent higher than normal filter cake. This material at 11 percent moisture would, however, be acceptable to produce green balls and

acceptable in a plant if excess drying capacity was available on the pelletizing machine.

Batch Balling Tests. The batch balling tests using papermill sludge were carried out using several methods of adding the binder to look at green-ball quality. A sludge addition of 1.4 percent (31.4 lb/LT) on a dry basis was used in all tests except Tests 1 to 3 which used bentonite. The bentonite tests at the 15 lb/LT addition level produced excellent quality green balls. At 9.2 percent moisture the 18-inch drop number was 10, and the dry strength was 17.3 pounds. In comparison, when the dried (pulverized) sludge was used the green-ball quality was considerably lower. At 9.2 percent moisture the 18-inch drop number was 4 and the dry strength 2.3 pounds. The 18-inch drop number is below the 5 drops minimum commonly cited by industry. A summary of the data is given in Table IV. Also, low moisture balls were difficult to produce as the tendency was to form at high levels.

In Tests 7 and 8 the effect of adding the sludge as a slurry was investigated and the data are presented in Table V. Using feed that was filtered with the binder showed good green-ball quality was obtained except the moisture was high (10 to 11%).

In Test 9 the "as received" sludge (73% moisture) was shredded and then intensively mixed with the concentrate, but the green-ball quality was not satisfactory. This is probably because the sludge is very difficult to break up and mix with the concentrate as it self-agglomerates and forms small seeds. It should be noted, that adding 1.4 percent (dry basis) of the 73 percent moisture sludge to an 8.9 percent filter cake would add about 3 percent moisture to the balling feed, i.e. to 11.9 percent moisture which may be too high for good balling and probably too high to dry effectively on the grate.

To avoid adding high levels of water to the balling feed a partially dried sludge was used in Tests 10 to 12. The sludge was dried to 31 percent and shredded in a Littleford intensive mixer prior to mixing with the concentrate. Again, the green-ball quality at 9.2 percent moisture appeared unacceptable, the 18-inch drop number was 3.4 and the dry strength 3.3 pounds. Even at the higher moisture there

was little improvement. The data are given in Table VI.

Pot-Grate Tests. The objective of the pot-grate tests was to determine what effect the sludge would have on fired pellet quality even though it would have to be partially dried to use as a binder. A base case test with bentonite was also conducted (Test 1) for comparison. Pellet quality in this test was very good with a 96.9 percent plus 1/4-inch tumble index and 649 pound compression strength. The metallurgical quality showed a 0.73 percent/minute reducibility and a 97.0 percent plus 6.3 mm LTB. In Tests 2 to 4 an addition of 1.4 percent (dry basis) sludge containing 31.2 percent moisture was used as the binder and no bentonite was added. Green-ball quality using the 3-ft disc was slightly better than in the batch balling tire; although, it was still marginal compared with the bentonite. In Tests 2 to 4 the firing temperature was varied from 2250 to 2350°F. In Test 2 with 2250°F firing the tumble index was 91.1 percent plus 1/4 inch and the compression strength was 412 pounds. An increase of 50°F in Test 3 improved quality slightly to a 91.8 percent tumble index and 419 pounds compression which compared with the bentonite test at the same temperature was much lower quality. A higher temperature of 2350°F in Test 4 again improved the physical quality to a 93.7 percent tumble index and 461 pounds compression. However, the reducibility (R₄₀) decreased from 1.09 to 0.86 percent/minute. Pellet temperatures at the bottom were now approaching 2400°F and thus, higher firing was not considered. It also appears that the reducibility benefit would be small at equivalent quality.

A final test was carried out using a combination of bentonite and sludge with each at half the addition used in the previous tests. Pellet quality improved slightly, but the tumble index was still unacceptable. Observation of the pellet surfaces indicates obtaining a good tumble would be difficult to achieve because of the large crater-like holes left from burning the sludge particles.

Chemistry of the fired pellets shows a lower SiO₂ of about 0.3 percent would be obtained with the sludge compared with bentonite which would be an advantage.

Conclusions

The test data show that direct substitution of papermill sludge for bentonite as a binding agent is not attractive for the following reasons:

- 1) The addition and mixing of the papermill sludge with the concentrate presents practical problems.
 - A) Addition of the papermill sludge as a slurry before filtration, which allows for ideal mixing with the concentrate, is not practical because filter capacity is greatly reduced and the moisture level in the filter cake is significantly increased. Both of these items represent significant costs to the producer that would require capital investment for added filter capacity and added operating costs related to the excess moisture.
 - B) Addition of the papermill sludge as a damp filter cake is impractical because it does not blend with the concentrate even with intensive and prolonged mixing times. This would also add about 3 percent moisture to the balling feed.
 - C) Addition of the papermill sludge as a "dry" material requires installation of both a dryer facility and a grinding system to produce a 200-mesh product suitable for mixing with the concentrate. The tests also show that intensive mixing would be required to obtain acceptable green-ball quality. These again represent significant capital investment and operating costs.
- 2) The quality of the green balls is marginal compared with that obtained with bentonite. Allowing higher moisture would probably improve green-ball quality to the point where it would be acceptable to the operator, but at an added energy cost.

3) Pellets produced in pot-grate tests with the papermill sludge were markedly inferior to those produced with bentonite. The physical quality, compression, and tumble index are unacceptable by current industry standards.

4) The ISO reducibility of the pellets was higher than that obtained with bentonite. However, when the firing cycle was modified to produce better physical quality the reducibility decreased to almost the same level as with bentonite.

5) The papermill sludge filter cake also readily grows a mold and has a noxious odor which would probably be unacceptable in a taconite plant environment.

Use of the papermill sludge in its "as is" condition, either slurry, or as filter cake does not appear practical or economically attractive. Pretreatment of the sludge by drying, grinding, or separation of the wood fibers may make it more useable, at a cost, but was beyond the scope of this study. Additional work under the current contract does not appear warranted.

Table I

Analyses of Blandin Papermill Sludge and Bentonite

	<u>Total Sludge*</u>	<u>Ash**</u>	<u>Bentonite</u>
SiO ₂ , %	9.95	45.57	64.2
Al ₂ O ₃ , %	8.38	38.39	17.3
Fe ₂ O ₃ , %	0.86	3.96	4.0
CaO, %	0.58	2.65	1.1
MgO, %	0.24	1.08	1.9
TiO ₂ , %	0.98	4.48	-
K ₂ O, %	0.13	0.60	-
LOI, %	78.16	-	6.0
Moisture, %	73.0	-	5.0

* As received sludge taken from waste treatment filter on 12/9/88, analyses on a dry basis.

** The high Al₂O₃ and SiO₂ reflect the Kaolin clay used in papermaking which provides some of the binding qualities of the sludge.

Table II

Size Analysis of National Steel Concentrate Used
For Sludge Binder Test Program

<u>Size, Mesh</u>	<u>Wt, %</u>	<u>Cumulative % Passing</u>
+100	0.5	
100/150	1.4	99.5
150/200	3.6	98.1
200/270	11.1	94.5
270/325	2.8	83.4
325/400	9.2	80.6
400/500	19.5	71.4
-500	51.9	51.9
Total	100.0	-
Moisture	8.9%	

Table III

Summary of Filter Tests With National Concentrate and Blandin Sludge

Test No.	Feed			Filter Cloth	Vacuum in. Hg			rpm	Cake		Capacity, lb/hr/ft ²
	% Solids	°F	Sludge, %		Form	Dry	Inches		Moisture, %		
1	65	68	-	801 RF	26	26	1.43	5/8	9.6	610	
2	60	68	-	801 RF	25	26	1.43	3/8	9.2	494	
3	60	68	-	801 RF	25	26	1.00	7/16	9.0	375	
4	60	68	-	801 RF	25	26	0.77	5/8	8.7	320	
5	60	68	1.4	801 RF	25	26	1.43	3/8	12.7	352	
6	60	68	1.4	801 RF	25	26	1.43	3/8	12.6	354	
7	60	68	1.4	801 RF	25	26	1.00	7/16	12.3	279	
8	60	68	1.4	801 RF	26	26	1.00	7/16	12.3	280	
9	60	68	1.4	801 RF	25	26	0.77	9/16	11.6	236	
10	60	68	1.4	801 RF	26	26	1.43*	5/16	13.1	336	
11	54**	68	1.4	801 RF	25	26	1.43	5/16	11.8	282	
12	54	68	1.4	801 RF	25	25	1.43	4/16	11.6	257	
13	54	68	1.4	801 RF	25	26	1.00	3/8	11.5	228	
14	54	68	1.4	801 RF	25	26	1.00	3/8	11.6	218	
15	54	68	1.4	801 RF	25	26	0.77	3/8	11.2	196	

* Form time cut by 25 percent to get thinner cake for same drying time as No. 5.

** Percent solids resulting from adding 4 percent slurry sludge to 65 percent solids concentrate.

Table IV

Summary of Batch Balling Tests With Bentonite Binder
and Dried Papermill Sludge Binder

Test No.	Concentrate	Binder		Green-Ball Quality			
		Type	lb/LT	Moisture, %	Drops, No.	Compression, lb	
						Wet	Dry
1	National	Bentonite	15.0	8.76	7.2	3.1	15.9
2	National	Bentonite	15.0	9.11	7.9	3.1	17.9
3	National	Bentonite	15.0	9.74	15.1	3.1	18.1
Calc	National	Bentonite	15.0	9.2	10.0	3.1	17.3
Calc	National	Bentonite	15.0	9.5	12.6	3.1	17.3
4	National	Sludge*	31.4	9.12	4.1	2.3	2.8
5	National	Sludge*	31.4	10.10	4.4	1.6	2.2
6	National	Sludge*	31.4	10.39	4.5	1.5	1.9
Calc	National	Sludge	31.4	9.2	4.1	1.8	2.3
Calc	National	Sludge	31.4	9.5	4.2	1.8	2.3

* Oven dried sludge at 105°C and pulverized. Added 1.4 percent on dry basis (31.4 lb/LT).

Table V

Summary of Batch Balling Tests With
Papermill Sludge at As Received Moisture

<u>Test No.</u>	<u>Concentrate</u>	<u>Binder</u>		<u>Green-Ball Quality</u>			
		<u>Type</u>	<u>lb/LT</u>	<u>Moisture, %</u>	<u>Drops, No.</u>	<u>Compression, lb</u>	
						<u>Wet</u>	<u>Dry</u>
7	National	Sludge*	31.4	10.1	6.4	2.3	7.0
8	National	Sludge*	31.4	11.3	15.3	2.8	7.7
9	National	Sludge**	31.4	10.4	3.9	1.9	3.8

* 1.4 percent sludge (dry basis) added to concentrate as 4 percent slurry and filtered together in pressure filter to 10 percent moisture.

** 1.4 percent sludge (dry basis) added to concentrate in mixer muller at 73 percent moisture after shredding to minus 1/4 inch and mixed for 5 minutes.

Table VI

Summary of Batch Balling Tests With Partially Dried
Papermill Sludge Binder

<u>Test No.</u>	<u>Concentrate</u>	<u>Binder</u>		<u>Green-Ball Quality</u>			
		<u>Type</u>	<u>lb/LT</u>	<u>Moisture, %</u>	<u>Drops, No.</u>	<u>Compression, lb</u>	
						<u>Wet</u>	<u>Dry</u>
10	National	Sludge*	31.4	8.86	3.1	2.1	3.3
11	National	Sludge*	31.4	9.62	3.8	1.9	3.5
12	National	Sludge*	31.4	10.21	4.2	1.6	3.1
Calc	National	Sludge*	31.4	9.2	3.4	1.9	3.3
Calc	National	Sludge*	31.4	9.5	3.7	1.9	3.3

* Sludge oven dried to 31.2 percent moisture and shredded in Littleford intensive mixer prior to mixing with concentrate at 1.4 percent addition rate (dry basis).

Table VII

Summary of Pot-Grate Tests With Blandin Sludge Binder
and National Concentrate

<u>Test No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
<u>Green Balls</u>					
<u>Additives</u>					
Bentonite, lb/LT	15.0	-	-	-	7.5
Blandin Sludge, % (Dry Basis)	-	1.4	1.4	1.4	0.7
Sludge Moisture, %	-	31.2	31.2	31.2	31.2
<u>Ball Quality</u>					
Moisture, %	9.1	9.1	9.0	9.0	9.1
18" Drops, No.	14.2	4.9	4.6	5.0	6.3
<u>Compression</u>					
Wet, lb	3.2	2.5	2.9	2.9	3.0
Dry, lb	20.7	4.7	5.5	5.4	10.3
<u>Indurating Temperatures</u>					
Drying, °F	700	700	700	700	700
Preheating, °F	1900	1900	1900	1900	1900
Firing, °F	2300	2250	2300	2350	2300
Peak Bottom, °F	2330	2320	2360	2390	2360
<u>Pellet Quality</u>					
<u>Before Tumble</u>					
+1/4", %	99.4	97.3	97.7	98.0	98.6
<u>Tumble Index</u>					
+1/4", %	96.9	91.1	91.8	93.7	94.4
-28 Mesh, %	3.1	8.7	8.0	6.1	5.4
<u>Compression</u>					
lb	649	412	419	461	482
-200 lb, %	0	0	1.7	1.7	0
<u>LTB</u>					
+6.3 mm, %	97.0	93.0	93.7	93.2	94.2
-0.5 mm, %	2.2	5.8	5.1	5.0	4.0
ISO R40, %/min	0.73	1.29	1.09	0.86	0.95
<u>Chemistry</u>					
Fe _T , %	65.8	66.0	66.1	66.0	65.6
Fe ⁺⁺ , %	0.05	0.15	0.10	0.25	0.06
SiO ₂ , %	5.12	4.78	4.83	4.78	4.90
Al ₂ O ₃ , %	0.17	-	0.14	0.13	-
CaO, %	0.28	-	0.32	0.25	-
MgO, %	0.35	-	0.35	0.33	-