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THE FEASIBILITY OF PRODUCING A DRY–PRESSED BUILDING BRICK WITH TAILINGS FROM A QUEBEC IRON MINE

by

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MINERAL PROCESSING DIVISION

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POSSIBILITE DE PRODUCTION D'UNE BRIQUE DE CONSTRUCTION PRESSEE A SEC A PARTIR DE RESIDUS D'UNE MINE DE FER DU QUEBEC

par

R. K. Collings*et G. A. Brown**

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RESUME

La Direction des Mines a reçu, au début de 1973, une requête de Hilton Iron Mines de Shawville, Québec, afin d'examiner la possibilité d'utilisation de ses résidus de minerai de fer dans la fabrication de briques. Une investigation fut donc entreprise sur l'utilisation de ces résidus dans cette application en se servant de lignosulfonate de calcium comme agent liant, ce dernier étant un déchet de l'industrie de la pulpe et du papier.

Un lien satisfaisant fut obtenu dans les tests préliminaires, lesquels comportaient l'utilisation de 1 pour cent de lignosulfonate et de 6 pour cent d'eau. Les spécimens d'essai avaient été pressés à 5000 lb/po^2 et cuits dans un petit four électrique à des températures variant entre 1000 et 1200° C pendant une période de 5 à 6 heures. Il fut cependant constaté que les spécimens se fracturaient à la cuisson, probablement dû au changement de volume du quartz présent, un phénomène qui se produit à 573° C. Ce problème fut toutefois résolu en donnant au matériel original un broyage plus fin, c'est-à-dire une proportion d'environ 50 pour cent passant le tamis No. 200. D'autres essais de cuisson furent effectués dans un plus grand fourneau. Des spécimens rencontrant les normes de la CSA en regard avec la résistance, l'absorption et les caractéristiques de gel et dégel furent obtenus après 6 heures de cuisson, cependant, dans une zone de cuisson relativement restreinte (1150 à 1175° C). De la recherche additionnelle, comportant l'utilisation d'additifs afin d'élargir la zone de cuisson est recommandée.

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INTRODUCTION

The Mines Branch was consulted early in 1973 by Hilton Iron Mines, Shawville, Quebec, regarding the feasibility of using its iron ore tailings for brick manufacture. Company officials indicated that the projected life of the mine, based on current reserves, was about five years. To ensure continuing employment for mine personnel, officials would like to see subsidiary or alternative industries established in the Shawville area, e.g., a brick manufacturing facility using mine tailings as a basic raw material.

Bureau of Mines researchers in the United States have demonstrated the feasibility of producing good-quality dry-pressed brick from copper mill tailings(1) and, as Hilton tailings are mineralogically similar, the possibility of producing a like product with the latter appeared worthy of study. An investigation similar in nature to that undertaken by the USBM was therefore initiated using Hilton tailings with small quantities of calcium lignosulfonate, a waste product from the pulp and paper industry, as binder.

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DESCRIPTION OF SAMPLES

Two samples of the Hilton tailings were received - one, in February, weighing 25 lb (S-1), the other in March, weighing 100 lb (S-2). The bulk of the work outlined in this report was carried out with the latter, a composite sample of current tailings obtained over an extended period. Both samples principally consisted of quartz, feldspar and amphibole, with minor mica, magnetite, and pyrite, and were mostly minus 20 mesh, as noted in Table 1.

TABLE 1

Wet Sieve Analyses-Hilton Tailings
(Wt %)

Mesh Size	Sample S-1	Sample S-2
+10		1.8
-10+20		6.7
-20+28		6.8
-28+35		7.3
-35+48	7.2	10.6
-48+65	32.0	12.6
-65+100	27.0	18.8
-100+150	17.3	13.5
-150+200	6.2	9.9
-200+325	5.1	5.2
-325	5.2	6.8
	100.0	100.0

INVESTIGATIVE WORK

Preliminary Tests

A number of preliminary tests were made to determine the amount of calcium lignosulfonate and water required for bonding, the degree of blending or mixing required, and the optimum forming pressure, firing time and temperature for test specimen preparation.

Samples of minus 20-mesh Hilton tailings were thoroughly dry mixed in a laboratory blender with various quantities of calcium lignosulfonate in both solid and liquid form, water added, and the whole mixed for an additional period. One hundred gram samples of each mixture were pressed on a Carver, hand-operated press to form test cylinders measuring 1.74-in. in diameter by 1.2- to 1.4-in. in height.

The specimens were fired in a laboratory, Thermolyne electric furnace over 5-to 6-hr periods, the temperature being increased 200 Celsius degrees per hour to a maximum that varied between 1000° and 1200°C for individual tests. The maximum temperature was maintained for one hour, followed by cooling at the natural rate of the furnace. The fired specimens were examined and tested for compressive strength. These preliminary tests indicated that the dry lignosulfonate powder was as effective as the liquid for bonding. Satisfactory bonding was achieved by blending the tailings with 1 per cent lignosulfonate, 6 per cent water, and mixing the whole for a 10-min period in the laboratory blender. A forming pressure of 5000 psi produced specimens with adequate dry strengths. The 5- to 6-hr firing cycle appeared satisfactory, however, specimens fired to 1200°C in the Thermolyne furnace were glazed and showed some tendency to fuse to the bottom furnace lining.

A point of particular concern was the fact that all specimens made with the as-received tailings, fired to 1000° to 1200°C, exhibited various degrees of fracture. Although other factors may have contributed, the prime reason undoubtedly was the quartz in the tailings because fracturing was initiated at about 600°C. Quartz undergoes a reversible inversion between the α and β forms at 573°C, which is accompanied by a substantial volume change. Specimens made with finer, artificial mixtures of tailings, or with samples ground to 40 per cent, or more, minus 200 mesh (Table 2) did not fracture on firing and specimens showed high compressive strengths. Several preliminary tests were also made with material treated by dry and wet magnetic separation and/or froth flotation. These latter tests were not pursued beyond the preliminary stage owing to relatively large losses through discarding the magnetic fraction which, in some instances, represented over 50 per cent of the feed (Table 3). Also, any additional processing, especially if wet, would tend to render commercial production uneconomic.

These preliminary tests are summarized and further commented upon in Table 4.

TABLE 2

Wet Sieve Analysis - Sized or Ground Tailings

(Wt %)

Mesh Size	Head Sample (S-1)	Blended Sample	Ground Samples	
			40%-200 mesh	50%-200 mesh
+10	1.8			
-10+20	6.7		0.7	0.2
-20+28	6.8		0.4	0.3
-28+35	7.3		0.5	0.4
-35+48	10.6	10	1.1	0.8
-48+65	12.6	10	3.5	2.5
-65+100	18.8	10	15.4	11.1
-100+150	13.5	10	16.1	21.6
-150+200	9.9	10	19.9	8.5
-200+325	5.2	50(-200)	38.1	25.0
-325	6.8	--	4.3	29.6
	100.0	100.0	100.0	100.0

TABLE 3

Magnetic Separation Tests

Unit and Feed	Product Recovery-Wt %	
	Mag.	Non-Mag.
Permanent Magnet, Head Sample	43.2	56.8
Carpco, Head Sample	55.0	45.0
Carpco, +150 mesh	63.4	36.6
Sala, wet, Head Sample	6.8	93.2

TABLE 4

Typical Data - Preliminary Tests, Thermolyne Furnace

Test	Specimen No.	Specimen Forming Material [1]	Forming Pressure psi [2]	Heating Rate 200°C/hr	Compressive Strength psi	Remarks
T-1	46	-35 mesh, head	5000	5hr to 1000°C	1495	fired specimens light pink-brown or salmon, all specimens fractured, especially No. 57, no evidence of bloating or blistering
	44	-35 mesh, non-mag.	"	1 hr at 1000°C	1265	
	57	-20 mesh, non-mag., flotation tails	"		-	
T-2	47	-35 mesh, head	"	6hr to 1200°C,	5482	fired specimens very dark grey or brown, all fractured especially No. 61, some distortion, no evidence of bloating or blistering
	45	-35 mesh, non-mag.	"	1 hr at 1200°C	6722	
	61	-20 mesh, non-mag., flotation tails			-	
T-3	28	tailings, special [3] blend	"	5½hr to 1100°C, 1 hr at 1100°C	8907	fired specimens brown, no evidence of fracturing, bloating or blistering
	39	tailings, non-mag., special blend	"		8403	
T-4	56	non-mag.,flotation tails, ground to 40%-200 mesh	"	5hr to 1000°C, 1 hr at 1000°C	1620	fired specimen light pink-brown or salmon, no evidence of fracturing, bloating or blistering
T-5	60	" " "	"	6hr to 1200°C 1 hr at 1200°C	6750	fired specimens dark brown, no evidence of fracturing, bloating or blistering but some distortion
T-6	83	head, ground to 40% minus 200 mesh	3000	5½hr to 1100°C, 1 hr at 1100°C	3865	fired specimens light red-brown, no evidence of fracturing, bloating or blistering, final strengths proportional to forming pressure
	84	" " "	4000		6134	
	85	" " "	5000		6470	
T-7	75	head, ground to 40% minus 200 mesh	3000	6hr to 1200°C, 1 hr at 1200°C	3361	fired specimens dark brown, no evidence of fracturing, bloating or blistering, some distortion, final strengths proportional to forming pressure
	77	" " "	4000		6890	
	79	" " "	5000		8697	
T-8	62	head, ground to 44% minus 200 mesh	5000 [4]	6 hr to 1200°C, 1 hr at 1200°C	-	fired specimens dark brown, specimens showed considerable degree of bloating and blistering (these specimens pressed in Hydrolair unit)
	64	" " "			2100	

TABLE 4 (Continued)

Typical Data - Preliminary Tests, Thermolyne Furnace

Test	Specimen No.	Specimen Forming[1] Material	Forming[2] Pressure psi	Heating Rate $200^{\circ}C/hr$	Compressive Strength psi	Remarks
T-9	68	head, ground to 42% minus 200 mesh	5000[4]	6hr to $1200^{\circ}C$, 1 hr at $1200^{\circ}C$	10,714	fired specimens dark brown, evidence of minor fracturing with some spalling and distortion (these specimens also pressed in Hydrolair unit)
	69	" " "	"		13,235	

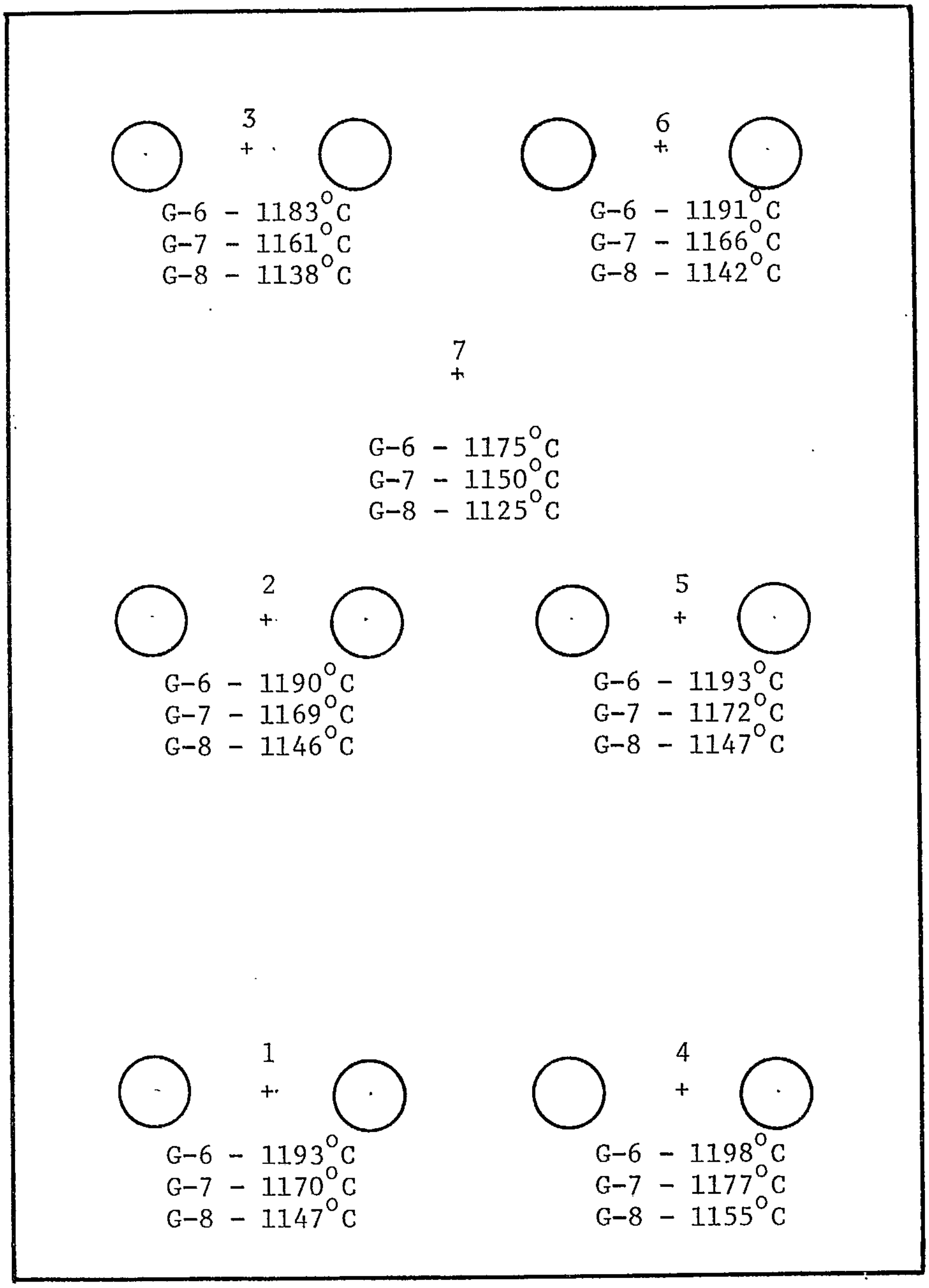
(1) Tailings-sized, beneficiated and ground to size noted, with 1% calcium lignosulphonate and 6% water.
(2) Maximum pressure used in forming cylindrical test specimens.
(3) Tailings, as received, blended to provide sample containing 50% of minus 200-mesh material. See Table 2.
(4) Specimens used in Tests 8 and 9 were pressed in a Hydrolair, air-operated, hydraulic press, all others were pressed in a Carver, hand-operated, hydraulic press.

Globar Furnace Tests

The results obtained in the preliminary tests were carefully reviewed and a second series of tests initiated using a Globar electric furnace, a much larger unit than the Thermolyne. As in the preliminary tests, test specimens (cylinders) were prepared using 1 per cent calcium lignosulfonate and 6 per cent water to bind the tailings which previously had been ground to about 50 per cent minus 200 mesh. Specimen forming pressure was 5000 psi. All specimens were fired in the Globar over a 5- to 6-hr period to maximum temperatures that ranged from 1000^{o} to $1200^{o}C$, and held at that temperature for one hour before the furnace was allowed to cool at its natural rate.

The final specimens were examined upon removal from the furnace and colour, quality, and shrinkage noted. The compressive strength was then determined and typical specimens checked for absorption and freeze-thaw as per CSA Specification A 82.2-1967 [(2)]. The results are noted in Table 5.

Globar furnace Tests G-6, G-7, and G-8, (maximum temperature 1175^{o}, 1150^{o} and 1125^{o}, respectively), were made to investigate temperature variation inside the furnace and the effect of this variation on the various physical properties of the fired specimens. Twelve specimens were placed in the furnace in each of these three tests in the positions shown in Figure 1. When the furnace reached the desired maximum temperature, as indicated by the control thermocouple, position 7, an automatic controller came into play to control the temperature within $\pm 5^{o}C$. The temperatures at six furnace locations, 1 to 6, Figure 1, were then recorded by a second thermocouple. These are also noted in Figure 1. Temperature-check positions 1 to 6 were about 1 in. above the test specimens whereas position 7 was 2 to 3 in. higher in the furnace.



Front End of Furnace

Figure 1 - Specimen Placement and Temperature Check, Globar Furnace, Test Nos. 7, 8 and ~~8~~ 6.

Note: Control thermocouple at position 7, temperature checks made at positions 1-6 inclusive.

TABLE 5

Typical Data - Globar Furnace Tests

Test	Specimen No.	Heating Rate 200°C/hr	Compressive Strength psi	% Absorption 24 hr imm.	% Absorption 5 hr boil	Sat. Coeff	Freeze Thaw-cycles	%Shrinkage Diam.	%Shrinkage Height	Weight Loss %	Remarks
G-1	102-111	5hr to 1000°C, 1 hr at 1000°C	3100 - 3600 av. of 10=3300								fired specimens light pink-brown or salmon, no evidence of fracturing, bloating or blistering variation in compressive strength probably due to variation in internal temperature of furnace-
G-2	112-120	5½hr to 1100°C 1 hr at 1100°C	3100-4400 av. of 19=3800								as above
	121-126	"		12.9 av. of 6	15.7 av. of 6	0.83					little variation in % absorption in each of the two absorption tests
	127-132	"					see remarks				all except two of the six specimens tested failed before 30 cycles and only one of these went the full 50 cycles without failure (this latter specimen had been fired in the right-front, hotter portion of the Globar)
	133-141	"									
G-3	146-160	6hr to 1200°C, 1 hr at 1200°C	7500-20,000 av. of 9=12,000				50+				fired specimens were dark brown, no evidence of fracturing, bloating or blistering, wide variation in compressive strength probably due to specimen location in furnace, four specimens tested for freeze-thaw all went full 50 cycles without failing
G-4	161-166	5½hr to 1150°C 1 hr at 1150°C	16,600-18,200 av. of 2=17,400	6.6 av. of 2	12.5 av. of 2	0.52	see remarks				fired specimens medium brown, no evidence of fracturing, bloating or blistering, high compressive strengths but low absorption indicating dense product, the two specimens tested for freeze-thaw failed after a few cycles
G-5	167-172	5 hr to 1100°C, 1 hr at 1100°C	3600-3700 av. of 2=3650	13.7 av. of 2	16.4 av. of 2	0.84	see remarks				fired specimens light pink-brown or salmon, low compressive strengths, higher absorption than G-4, the two specimens tested for freeze-thaw failed after a few cycles

TABLE 5 Continued

Typical Data - Globar Furnace Tests

Test	Specimen No.	Heating Rate 200°C/hr	Compressive Strength psi	% Absorption		Sat, Coeff	Freeze-Thaw cycles	% Shrinkage		Weight Loss %	Remarks
				24 hr imm.	5 hr boil			Diam.	Height		
G-6	173-178	5 3/4hr to 1175°C, 1 hr at 1175°C	9495-20,000 av. of 6=15,013								fired specimens medium brown, no evidence of fracturing, bloating or blistering, variation in compressive strength generally coincides with variation in internal temperature of furnace
	179-181	"		2.32 av. of 3	7.49 av. of 3	0.31					
	182-184	"					50+				all three specimens tested withstood the required 50 freeze-thaw cycles without fracture
	173-184	"						4.0	5.0	10.3	
G-7	185-190	5½hr to 1150°C 1 hr at 1150°C	10,588-22,142 av. of 6=15,536								fired specimens dark pink-brown, no evidence of fracturing, bloating or blistering, variation in compressive strength generally coincides with variation in internal temperature of furnace- only one of the three specimens tested for freeze-thaw withstood required 50 cycles without fracture - this specimen was located in the rear, right-hand section of the furnace which was about 8°C above the average temperature of the furnace, remaining two specimens fractured horizontally at a point midway between the top and bottom surfaces of specimen after 9 freeze-thaw cycles - this fracture, in part, could be due to a plane of weakness in the specimens that may have developed during pressing
	191-193	5½hr to 1150°C, 1 hr at 1150°C	10,588-22,142 av. of 6=15,536				see remarks				
	194-196	"		5.79 av. of 3	12.06 av. of 3	0.47					
	185-196	"						2.3	3.3	11.2	

TABLE 5 Continued

Typical Data - Globar Furnace Tests

Test	Specimen No.	Heating Rate $200^{\circ}C/hr$	Compressive Strength psi	% Absorption 24 hr imm.	% Absorption 5 hr boil	Sat, Coeff	Freeze-Thaw cycles	% Shrinkage Diam.	% Shrinkage Height	Weight Loss %	Remarks
G-8	197-202	5 1/4 to $1125^{\circ}C$ 1 hr at $1125^{\circ}C$	8,571-15,366 av. of 6=11,943								fired specimens medium pink brown, no evidence of fracturing, bloating or blistering, variation in compressive strength generally coincides with variation in internal temperature of furnace
	203-205	"					see remarks				only one of three specimens tested for freeze thaw withstood required 50 cycles without fracture-this specimen was located in the rear, right-hand section of the furnace which was about $9^{\circ}C$ above the average temperature of the furnace, remaining two specimens fractured horizontally at a point midway between the top and bottom surfaces of the specimen after 9 freeze-thaw cycles- this fracture, in part, could be due to a plane of weakness in the specimens that may have developed during pressing
	206-208	"		8.11 av. of 3	13.52 av. of 3	0.59					
	197-208	"						2.2	2.5	10.9	

DISCUSSION OF RESULTS

Preliminary test results, Table 4, indicate several significant points. The removal of select fractions, e.g., plus 20- or plus 35-mesh, magnetic (magnetite), flotation concentrate (pyrite), did not reduce specimen fracture on firing. However, fracture was nullified by using an artificial, more finely divided mixture of tailings and also by fine grinding to 40 per cent or more minus 200 mesh (Tests T-3 to T-9). Specimens fired to 1200°C were dark brown and showed some distortion whereas those fired to only 1000° or 1100°C were light reddish brown or salmon and showed no distortion. The final strengths achieved were generally proportional to the forming pressures, higher strengths being achieved with higher forming pressures (Tests T-5 and T-6). Strength also was proportional to firing temperature within limits, specimens fired at 1000°C being much weaker than those fired at 1100°C and above. Only one series of test specimens, those pressed in the Hydrolair press, showed evidence of bloating and blistering on firing (Tests T-8 and T-9). No apparent reason for this unusual behaviour could be found. Possibly, in error, the specimens may have been formed at a higher pressure than intended. This may, in turn, have prevented the escape of entrapped gases during firing which could account for the bloating and blistering. None of the several dozens of specimens formed with the hand-operated, Carver press, showed any bloating or blistering.

The Globar furnace test results, summarized in Table 5, generally confirm the results obtained in the Thermolyne unit. Specimens fired to 1000°C were pink-brown or salmon whereas those fired to 1200°C were dark brown. Intermediate colours were obtained at intermediate temperatures. None of the fired specimens showed any evidence of bloating or blistering. Compressive strengths were generally proportional to firing temperatures. Specimens fired to 1000°C exhibited strengths of about 3300 psi, those heated to 1100°C had strengths in excess of 3600 psi. A significant increase in compressive strength from 12,000 to 15,000 psi, was noted in the specimens fired at higher temperatures, 1125° to 1175°C. A wide variation in specimen strength was observed in some tests, (G-3, G-6, G-7, G-8), presumably due to specimen location in the furnace (Figure 1). Specimens placed in the right, hotter side of the furnace, generally, although not always, exhibited higher strengths. Specimen compressive strengths, in any event, were well above the 3500 psi minimum specified in CSA A82.1-1965[(3)]. The percentage absorption of the fired specimens was proportional to the firing temperature. Those fired to 1100°C showed absorptions of 13 and 16 percent, and those fired to 1175°C, 2 and 7 per cent respectively for the 24-hr immersion and 5-hr boil tests. Saturation coefficient varied from a low of 0.31 to 0.84. These values met CSA specifications for Type 1 (exposed) brick. All specimens fired to 1175°C or above withstood the required 50 freeze-thaw cycles (Test G-3 and G-6); however, only one specimen of the six tested in G-2, one of three in G-7, and one of three in G-8, withstood 50 cycles without fracture. The sound specimens in G-2 had been fired in the front-right or hottest position in the furnace (Figure 1) whereas those in Tests G-7 and G-8 were fired in the rear right of the furnace. While the temperature at this position was slightly below the mean for the furnace, this location was very close to the refractory lining of the furnace. Specimens fired in this position conceivably could have benefitted somewhat from the radiant heat given off by the lining during the long, 24-hr cooling period. The fact that all freeze-thaw specimens from Tests G-6, G-7 and G-8 that failed did so by virtue of a single, plane fracture that occurred midway between the top and bottom surfaces of the specimens may be significant. This failure could be associated with a suspected plane of weakness which formed during the initial pressing of the test specimens which was coincident with the fracture plane. Specimens fired at lower temperatures, i.e., below 1125°C, exhibited many and diverse fractures in freeze-thaw failure. Shrinkage on firing, as expected, was greater for specimens fired at higher temperatures. Shrinkage at 1175°C (Test G-6) was 4 to 5 per cent whereas it was only 2 per cent at 1125°C (Test G-8). Weight loss in all cases was in the order of 10 to 11 per cent and due to removal of water and the evolution of sulphur dioxide and other gases on firing. Shrinkage was well within CSA specifications.

A point of concern was the relatively narrow firing range within which specimens meeting CSA freeze-thaw specifications were produced, (1175° to 1200°C, as indicated by control thermocouple, position 7, Figure 1). As noted above, pressing techniques may have contributed to freeze-thaw failure of specimens fired below 1175°C, however, this would have to be verified by further experimentation. The firing range could possibly be extended by additives, e.g., a fluxing material such as ground glass to promote sintering at a lower temperature and/or a refractory material such as fire clay to extend the upper temperature.

RECOMMENDATIONS

Although the authors were unable to carry this investigation through to the actual fabrication and testing of full size brick due to pressure of other work commitments, this preliminary study of Hilton Mines tailings indicates the technical feasibility of producing a fired product which appears to have potential for use as building brick. Further research is recommended to investigate techniques for extending the firing range, should studies indicate an adequate and continuing market for this type of brick in the Shawville and surrounding area. This research should be augmented by pilot plant production of full size brick.

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