Pergamon

Cement and Concrete Research, Vol. 25, No. 8, pp. 1713-1725, 1995
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0008-8846/95 $9.50+.00

0008-8846(95)00167-0

CHARACTERISTICS OF A THERMALLY ACTIVATED ALUMINO-SILICATE POZZOLANIC MATERIAL AND ITS USE IN CONCRETE

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(Refereed)
(Received February 15; in final form July 21, 1995)

ABSTRACT

This paper presents the results of the physical and chemical properties of a thermally activated alumino-silicate material (MK), and deals with the properties of fresh and hardened concrete incorporating this material. The properties of fresh concrete investigated included workability, bleeding, setting time, and autogenous temperature rise. The properties of the hardened concrete investigated included compressive, splitting-tensile and flexural strengths, Young's modulus of elasticity, drying shrinkage, resistance to chloride-ion penetration, freezing and thawing, and salt-scaling resistance. The properties of the MK concrete were also compared with those of the control portland cement concrete and the silica fume concrete.

The test results indicate that the MK material is highly pozzolanic and can be used as a supplementary cementing material to produce high-performance concrete. Although it requires a higher dosage of the superplasticizer and air-entraining admixture compared with that of the control concrete, the MK concrete can be produced with satisfactory slump, air content, and setting time. The concrete incorporating 10% MK had higher strength at all ages up to 180 days compared with the control concrete; in comparison with the silica fume concrete the MK concrete showed a faster strength development at early ages, but had lower strength after 28 days. At 28 days, the MK concrete had somewhat higher splitting-tensile and flexural strengths, Young's modulus of elasticity, and lower drying shrinkage compared with that of the control and the silica fume concretes. The resistance of the MK concrete to the chloride-ion penetration was significantly higher than that of the control concrete, but similar to that of the silica fume concrete. The MK concrete showed excellent performance in the freezing and thawing test. The performance of the MK concrete subjected to the de-icing salt scaling test was similar to that of the silica fume concrete, but marginally inferior to the control concrete.

Introduction

Canada Centre for Mineral and Energy Technology (CANMET) has an ongoing program dealing with the development of concrete having long-term durability. One of the means of achieving this objective is to incorporate supplementary cementing materials such as silica fume, fly ash, slag, and rice husk ash in concrete. The incorporation of these supplementary cementing materials in concrete leads to reduction in its porosity; this, in turn, leads to reduced permeability and increased durability of concrete.

This paper presents the results of an experimental study on a thermally activated alumino-silicate material* available in North America, and discusses its effect on the properties of fresh and hardened concrete. The objective of this study is to explore the potential of this material as an alternative for silica fume to produce high-performance concrete.

Research Significance

The long-term durability of concrete is of paramount concern to the concrete industry, and one of the methods to achieve this is to produce concrete having very low permeability. The thermally activated alumino-silicate material is one such pozzolanic product which may reduce the permeability of concrete significantly when incorporated in concrete, and thus may result in its increased durability. This research is undertaken to develop data to support the above.

Concrete Mixtures

The concrete mixtures were made at CANMET in 1994 using the following materials and mixture proportions.

Cement

ASTM Type I normal portland cement was used, and its physical properties and chemical analysis are given in Table 1.

Thermally Activated Alumino-Silicate Material (MK)

The physical properties and chemical analysis of the thermally activated alumino-silicate, hereafter designated as "MK" are also given in Table 1. MK is a whitish powder consisting mainly of silicon dioxide (51.34%) and aluminum oxide (41.95%). The material contains only a small quantity of calcium oxide (0.34%) and alkalies, and therefore it does not have a capacity to hydrate itself. The material has a specific surface of 16.8 m^2/g and a median particle size of approximately 1.3 μm, which is smaller than that of ordinary cement (approximately 10 μm) but larger than that of silica fume (approximately 0.1 μm). For the MK tested, 99.8% of the particles passed a 45 μm sieve. The specific gravity of the material is 2.5. Scanning electron micrograph (Fig. 1) shows that the particles of the material are angular like that of cement. The X-ray diffraction analysis indicates that the MK is mainly amorphous material with only a small quantity of crystallized phases (Fig. 2). The main crystallized phases identified include anatase (TiO_2), cristobalite (SiO_2), and quartz (SiO_2).

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* commercially known as Metakaolin.

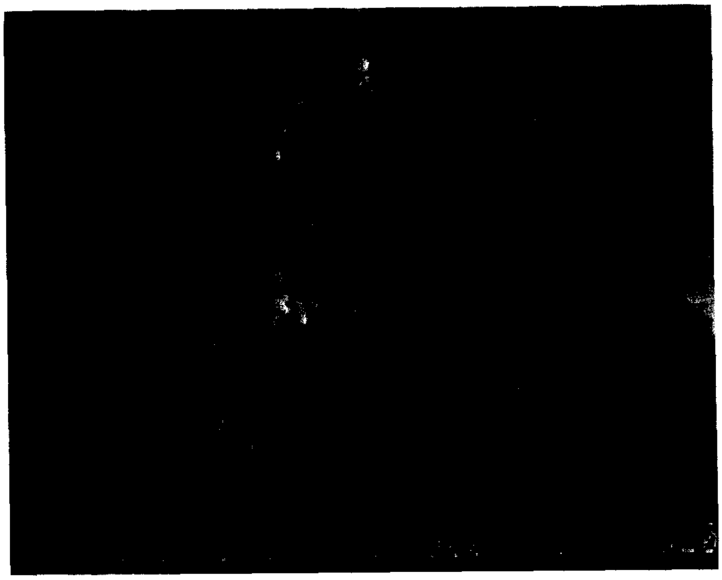

FIG. 1
Scanning electron micrograph of the MK particles.

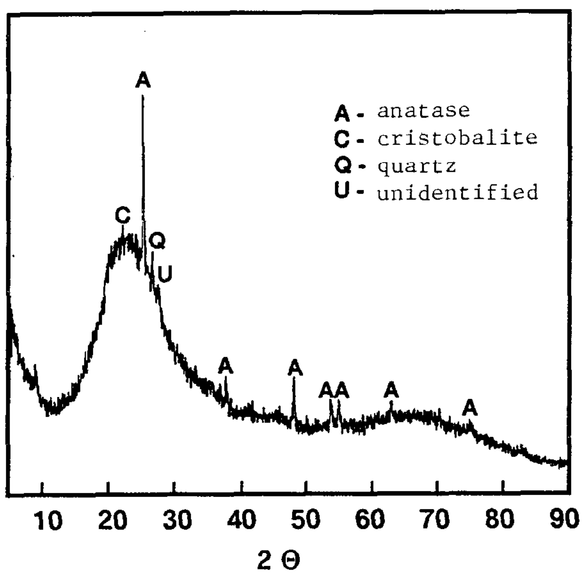

FIG. 2
X-ray spectrum of the MK.

Silica Fume

Silica fume (SF) used was a dry uncompacted powder. The chemical composition and physical properties of the silica fume are given in Table 1.

TABLE 1
Physical Properties and Chemical Analysis of the Portland Cement, Thermally Activated Alumino-silicate (MK), and Silica Fume

	ASTM Type I Cement	MK	Silica Fume
Physical Tests			
Specific gravity	3.09	2.50	2.16
Fineness			
passing 45μm, %	93.0	99.8	98.9
specific surface, Blaine, m^2/kg	373	-	-
nitrogen adsorption, m^2/g	-	16.8	26.1
median grain size, μm	-	1.3	-
Setting time, min,			
initial	135	-	-
final	-	-	-
Compressive strength of 51-mm cubes, MPa			
7-day	27.8	-	-
28-day	39.1	-	-
Chemical Analysis, %			
Silicon dioxide (SiO_2)	20.1	51.34	93.71
Aluminum oxide (Al_2O_3)	4.51	41.95	0.21
Ferric oxide (Fe_2O_3)	2.50	0.52	0.31
Calcium oxide (CaO)	61.3	0.34	0.35
Magnesium oxide (MgO)	3.13	0.03	0.47
Sodium oxide (Na_2O)	0.24	0.34	0.19
Potassium oxide (K_2O)	0.39	0.11	1.19
Phosphorous oxide (P_2O_5)	<0.9	0.28	0.14
Titanium oxide (TiO_2)	0.24	1.74	0.01
Sulphur trioxide (SO_3)	4.04	0.07	0.29
Loss on ignition	2.41	0.72	2.72
Bogue Potential Compound			
Tricalcium silicate C_3S	51.3	-	-
Dicalcium silicate C_2S	19.0	-	-
Tricalcium aluminate C_3A	7.7	-	-
Tetracalcium aluminoferrite C_4AF	7.6	-	-

Aggregate

The coarse aggregate was a crushed limestone with a maximum nominal size of 19 mm, and the fine aggregate was local natural sand from the Ottawa area. Both the coarse and fine aggregates were separated into different size fractions and recombined to a specified grading shown in Table 2. The coarse and fine aggregates had specific gravities of 2.69 and 2.70, and water absorptions of 0.82 and 1.10%, respectively.

TABLE 2
Grading of the Aggregates

Coarse Aggregate		Fine Aggregate	
Sieve size (mm)	Cumulative percentage retained	Sieve size (mm)	Cumulative percentage retained
19.0	0.0	4.75	0.0
12.7	40.0	2.36	10.0
9.5	65.0	1.18	32.5
4.75	100.0	0.60	57.5
-	-	0.30	80.0
-	-	0.15	94.0
-	-	pan	100.0

Superplasticizer

A superplasticizer of sulphonated, naphthalene formaldehyde condensate was used for all of the concrete mixtures. This superplasticizer was a dark brown solution containing 42% solids.

Air-Entraining Admixture

A synthetic resin type of air-entraining admixture was used in all of the concrete mixtures.

Mixture Proportions

A concrete mixture with 10% MK as cement replacement (by weight) and a water-to-cementitious materials ratio of 0.40 was selected for the investigation. A control portland cement concrete mixture, and a mixture incorporating silica fume were also included for comparison. The proportions of the concrete mixtures are summarized in Table 3. All concrete mixtures were air-entrained.

Preparation and Casting of Test Specimens

The concrete was mixed in a laboratory counter-current mixer for a total of 5 minutes. The properties of fresh concrete including slump, air content, density, bleeding, setting time, and temperature increase were determined, and are given in Table 4. The measurement of the temperature rise was carried out using a 152x305-mm cylinder with a temperature-measuring

TABLE 3
Proportions of Concrete Mixtures

Mixture no.	MK content (%)	Silica fume content (%)	W/C or W/C+MK or W/C+SF	Quantities (kg/m³)							
				Water*	Cement	MK	Silica fume	Fine agg.	Coarse agg.	SP**	A.E.A.*** (mL/m³)
C0	0	0	0.40	154	385	0	0	674	1102	2.5	158
MK10	10	-	0.40	155	349	39	0	675	1103	4.3	248
SF10	-	10	0.40	155	348	0	39	673	1098	3.7	248
C0-D	0	0	0.40	155	386	0	0	676	1105	2.5	129
MK10-D	10	-	0.40	155	349	39	0	676	1104	3.5	249
SF10-D	-	10	0.40	155	348	0	39	671	1096	3.7	248

* includes water in the superplasticizer, ** superplasticizer, naphthalene based, *** air-entraining admixture

TABLE 4
Properties of Fresh Concrete

Mixture No.	MK content (%)	Silica fume content (%)	W/C or W/C+MK or W/C+SF	Temp. (°C)	Slump (mm)	Density (kg/m³)	Air content (%)	Bleeding	Setting time h:min	
									Initial	Final
C0	0	0	0.40	21.0	80	2345	6.1	negligible	n/a	5:12
MK10	10	-	0.40	20.0	170	2345	5.6	negligible	3:06	4:24
SF10	-	10	0.40	20.5	150	2330	5.7	negligible	3:36	4:48
C0-D	0	0	0.40	19.0	140	2345	5.8	-	-	-
MK10-D	10	-	0.40	23.5	130	2350	5.5	-	-	-
SF10-D	-	10	0.40	23.0	130	2330	5.8	-	-	-

unit embedded in the middle of the cylinder. The mixtures C0, MK10, and SF10 were used for determining the properties of fresh and hardened concrete; the mixtures C0-D, MK10-D, and SF10-D were used for assessing the durability and drying shrinkage of concrete.

For each of the mixtures C0, MK10, and SF10, eighteen 102x203-mm cylinders and three 102x76x406-mm prisms were cast for the determination of the compressive and flexural strengths, respectively. Also, four 152x305-mm cylinders were cast from each mixture for determining the splitting-tensile strength and Young's modulus of elasticity.

For mixtures C0-D, MK10-D, and SF10-D, three 102x203-mm cylinders each were cast for the determination of compressive strength. Another four cylinders of the same size were cast from each mixture for determining the resistance to chloride-ion penetration. Also, from each mixture ten 102x76x390-mm prisms were cast for determining the freezing and thawing resistance and drying shrinkage and two 280x305x76-mm slabs were cast for the de-icing salt scaling test.

Most specimens were cast in two layers, and compacted on a vibrating table, except for 152x305-mm cylinders which were cast using an internal vibrator. After casting, all the specimens were left covered in the casting room at 20±3°C for 24 h. The specimens were then demoulded and transferred to a moist curing room at 20±3°C and 100% relative humidity until the time of testing.

Testing of Specimens

For the mixtures C0, MK10, and SF10, the compressive strength was determined at 1, 3, 7, 28, 90, and 180 days. The splitting-tensile and the flexural strengths were determined at 28 days using two large cylinders and three prisms, respectively and the Young's modulus of elasticity was determined at 28 days on two cylinders. The drying shrinkage of the two prisms was measured at 7, 14, 28, 56, and 112 days after an initial curing of 1 day in the mould and 6 days in lime-saturated water, and the measurements will be continued for 224 days. Another two prisms were stored in the lime-saturated water 24 h after casting, and their length changes were measured for reference purposes. All the above tests were carried out following the relevant ASTM standards.

The resistance of the concrete to the chloride-ion penetration was measured according to ASTM C 1202 at the ages of 28 and 90 days using the top portion of the two small cylinders at each age.

The resistance of the concrete to freezing and thawing cycling was determined following ASTM C 666 Procedure A, freezing and thawing in water. The changes in length, weight, pulse velocity, and resonant frequency of the two prisms were determined after every 50 freezing and thawing cycles. The flexural strength of the concrete prisms were also determined after the completion of the test, and compared with that of the reference concrete specimens cured in the moist room. The air content and spacing factor of the hardened concrete were determined following ASTM C 457.

The de-icing salt scaling test, based on ASTM C 672, was started after an initial moist curing of the two test slabs for 28 days, followed by 14 days drying in laboratory air. The test slabs were exposed to 50 cycles of freezing and thawing in a 3% solution of NaCl solution. The performance of the concrete was evaluated visually, and by determining the weight loss.

Results and Discussion

Properties of Fresh Concrete

The properties of fresh concrete are given in Table 4. The concretes had slumps ranging from 80 to 170 mm, and unit weights from 2331 to 2352 kg/m^3. The air content of the concrete was in the range of 5.5 to 6.1%. The concrete incorporating the MK showed good finishing qualities. Because of the fine particle size of MK, the concrete incorporating 10% MK required more superplasticizer than the control concrete. It required almost as much superplasticizer as the silica fume concrete in order to achieve similar slump; this is contradictory to the findings by other researchers [1,6]. At the replacement level of 10%, the MK concrete required a similar amount of air-entraining admixture as the silica fume concrete.

The initial and final setting times of the control, MK, and silica fume concrete were comparable (Table 4). The bleeding of all three concretes were negligible.

Figure 3 shows the autogenous temperature rise in the concretes with time. The placing temperature of the concrete mixtures was about 20°C. The maximum temperature reached for the MK concrete was 52.8°C, which was somewhat higher than that of the silica fume concrete (46.1°C) and the control concrete (45.6°C). The maximum temperature of the MK concrete

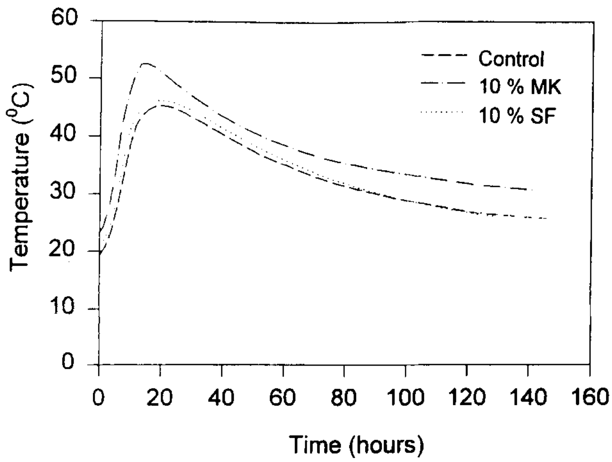

FIG. 3
Autogenous temperature rise in 152x305-mm concrete cylinders.

was reached after about 15 h, which was earlier than those of the control concrete (20 h) and the silica fume concrete (19 h). The temperature of the MK concrete was higher than those of the control and silica fume concretes at all ages up to 6 days, indicating a high reactivity of the MK material.

Mechanical Properties of the Hardened Concrete

The compressive, flexural, and splitting-tensile strengths, and the Young's modulus of elasticity of the concretes investigated are summarized in Table 5, and the compressive strength development of the concrete with age is illustrated in Fig. 4.

The concrete incorporating the MK had higher strength at all ages up to 180 days compared with the control concrete; and when compared with the silica fume concrete, the MK concrete showed a faster strength development at early ages, but had lower strength after 28 days. The faster strength development of the MK concrete at early ages as compared with the silica fume concrete is probably due to the faster rate of hydration and reaction as indicated by the higher temperature rise discussed earlier.

TABLE 5
Mechanical Properties of Hardened Concrete

Mix no.	MK content (%)	Silica fume content (%)	W/C or W/C+MK or W/C+SF	Unit weight (kg/m^3)	Strength (MPa)								"E" Modulus^{++} (GPa)
					Compressive*						Splitting-tensile**	Flexural^{+}	
					1d	3d	7d	28d	90d	180d	28d	28d	28d
C0	0	0	0.40	2350	20.9	25.5	28.9	36.4	42.5	44.2	2.7	6.3	29.6
MK10	10	-	0.40	2330	25.0	32.9	37.9	39.9	43.0	46.2	3.1	7.4	32.0
SF10	-	10	0.40	2320	23.2	28.6	34.7	44.4	48.0	50.2	2.8	7.0	31.1

* average of three 102x203-mm cylinders, ** average of two 152x305-mm cylinders
$^{+}$ average of two 102x76x406-mm prisms, $^{++}$ average of two 152x305-mm cylinders.

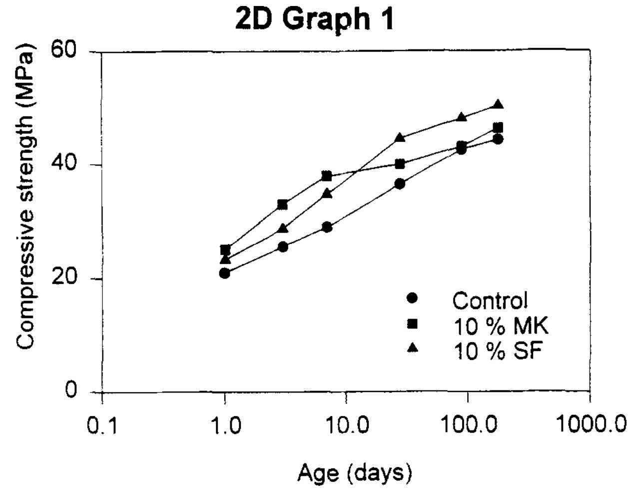

FIG. 4
Development of the compressive strength of concrete (W/C, W/C+MK, or W/C+SF = 0.40).

At 28 days, the MK concrete had somewhat higher splitting-tensile and flexural strengths and modulus of elasticity compared with that of the control and silica fume concretes. For example, splitting-tensile and flexural strengths of the control, silica fume, and MK concrete were 2.7, 2.8, and 3.1 MPa and 6.3, 7.0, and 7.4 MPa, respectively; the corresponding Young's modulus of elasticity values were 29.6, 31.1, and 32.0 GPa, respectively.

Figure 5 shows the drying shrinkage strain of the concretes after 7 days of initial curing in lime-saturated water. The MK concrete had a lower drying shrinkage compared with that of the control and silica fume concretes. After 112 days of drying at the relative humidity of 50%, the

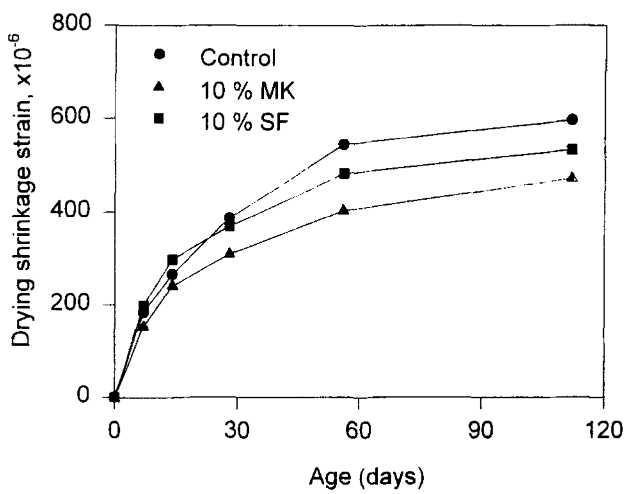

FIG. 5
Drying shrinkage strain of the concrete.

TABLE 6
Resistance of Concrete to Chloride-ion Penetration

Mix no.	Type of concrete	W/C or W/C+MK or W/C+SF	Unit weight (kg/m^3)	28-d Compressive strength (MPa)	Resistance to chloride-ion penetration (coulomb)	
					28d	90d
C0-D	Control	0.40	2320	36.5	3175	1875
MK10-D	10% MK	0.40	2330	37.9	390	300
SF10-D	10% SF	0.40	2310	42.8	410	360

MK concrete had a drying shrinkage strain of 472x10^{-6} compared with 596x10^{-6} and 532x10^{-6} for the control and silica fume concretes, respectively.

Durability Aspects of the Hardened Concrete

The 28- and 91-day test results for the resistance to chloride-ion penetration of the concretes, measured in terms of the electric charge in coulombs passed through the specimens (ASTM C 1202), are given in Table 6.

The resistance of the MK concrete to the chloride-ion penetration was significantly higher than that of the control concrete, but similar to that of the silica fume concrete. At 28 days, the charge passed through the MK concrete (390 coulombs) and through the silica fume concrete (410 coulombs) was very low in comparison with the control portland cement concrete (3175 coulombs). As expected, the charge passed through the concrete decreased with increased curing. At 90 days, the charge passed through the MK and the silica fume concretes was 300 and 360 coulombs, respectively. The corresponding value for the control concrete was 1875 coulombs. According to ASTM C 1202, when the charge passing through concrete is below 1000 coulombs, the concrete has very high resistance to the chloride-ion penetration.

Table 7 summarizes the results of the freezing and thawing testing according to ASTM C 666 Procedure A, freezing and thawing in water, together with the residual flexural strength which was based on the flexural strength of the concrete prisms after the completion of the freezing and thawing cycling and that of the reference concrete specimens cured in the moisture room.

TABLE 7
Summary of Test Results after 300 Cycles of Freezing and Thawing

Mix no.	Type of concrete	28-d compressive strength (MPa)	Air content of		Specific surface (mm^{-1})	Spacing factor $\bar{L}$ (mm)	Change at the end of 300 freezing/thawing cycles (%)				Durability factor (%)	Residual flexural strength (%)
			fresh concrete (%)	hardened concrete (%)			W*	L*	PV*	RF*		
C0-D	Control	36.5	5.8	6.6	21.2	0.15	0.08	0.006	-0.55	-0.84	98.3	85
MK10-D	10% MK	37.9	5.1	4.9	17.9	0.22	0.09	-0.003	0	0.16	100.3	89
SF10-D	10% SF	42.8	5.8	5.0	22.2	0.17	0.12	0.001	0.19	0.47	100.9	96

* W-Weight, L-Length, PV-Pulse Velocity, RF-Resonant Frequency

The control, MK, and silica fume concretes showed excellent performance after 300 cycles of freezing and thawing with durability factors of about 100. There were insignificant changes in length, weight, pulse velocity, and resonant frequency of the test prisms after the above cycling. The residual flexural strength of the MK concrete prisms subjected to freezing and thawing cycling was 89% of the reference specimens, which was slightly higher than that of the control concrete (85%), and lower than that of the silica fume concrete (96%).

The air-void parameters of hardened concrete, that is, specific surface and spacing factor $\bar{L}$, are also shown in Table 7. The values of the specific surface were 21.2, 17.9, and 22.2 mm^{-1} for the control, MK, and silica fume concretes, respectively; the corresponding values of the $\bar{L}$ were 0.15, 0.22, and 0.17 mm, respectively.

It is generally agreed that air-entrained concrete should have a spacing factor not exceeding 0.2 mm for satisfactory resistance to freezing and thawing. In recent years, several publications [7-9] have reported that superplasticized concrete and concrete incorporating supplementary cementing materials show satisfactory resistance to freezing and thawing cycling even if the spacing factor exceeds 0.2 mm, and is between 0.2 and 0.25 mm [7-9]. The test results obtained in this investigation support these published data.

The results of the concretes investigated to determine the de-icing salt scaling according to ASTM C 672 are given in Table 8. The visual evaluation of the test slabs subjected to the scaling test for 50 cycles showed that the performance of both the MK and silica fume concretes was marginally inferior to that of the control concrete. For both the MK and the silica fume concretes, some coarse aggregate was visible at the surface of the slabs after 50 cycles of freezing and thawing, whereas for the control concrete, no coarse aggregate was visible. The total mass of scaling residue for the control, MK, and silica fume concretes was 0.3, 0.9, and 0.8 kg/m^2, respectively. According to the Ontario Provincial Standard Specification, the mass of scaling residue is limited to 0.8 kg/m^2 after 50 cycles of freezing and thawing. Further research is needed in this area.

TABLE 8
Test Results of De-icing Salt Scaling

Mix no.	Type of concrete	W/C or W/C+MK or W/C+SF	28-d compressive strength (MPa)	Visual rating* (ASTM C 672)	Total scaling residue (kg/m^2)
C0-D	Control	0.40	36.5	2	0.3
MK10-D	10% MK	0.40	37.9	3	0.9
SF10-D	10% SF	0.40	42.8	3	0.8

* Rating (ASTM C 672)
0 No scaling
1 Very slight scaling (3.2 mm, max, no coarse aggregate visible)
2 Slight to moderate scaling
3 Moderate scaling (some coarse aggregate visible)
4 Moderate to severe scaling
5 Severe scaling (coarse aggregate visible over entire surface)

Conclusions

The results of this limited investigation indicate that the thermally activated alumino-silicate material is highly pozzolanic and appears to have excellent potential as a supplementary cementing material for producing high-performance concrete.

The concrete incorporating 10% MK required more superplasticizer and air-entraining admixture than the control concrete. It required almost as much superplasticizer and air-entraining admixture as the silica fume concrete in order to achieve similar slump and air content. The initial and final setting times of the MK concrete were shorter than those of the control and silica fume concretes. The bleeding of the MK concrete was negligible. The maximum temperature reached for the MK concrete was higher, and was reached earlier than that of the control and the silica fume concretes.

The MK concrete had higher strength properties at all ages up to 180 days compared with the control concrete; when compared with the silica fume concrete, the MK concrete showed a faster strength development at early ages, but had lower strength after 28 days. The MK concrete had lower drying shrinkage compared with that of the control and silica fume concretes.

The resistance of the MK concrete to the penetration of chloride-ions was significantly higher than that of the control concrete, but similar to that of the silica fume concrete. The MK concrete showed excellent performance under the freezing and thawing cycling with a durability factor of about 100. The performance of the MK concrete subjected to the de-icing salt scaling test was similar to that of the silica fume concrete, but was marginally inferior to the control concrete.

Further comprehensive investigations should be performed to cover the effects of different percentages of MK as cement replacement and different W/C+MK on concrete properties, and the role of MK in controlling alkali-aggregate reactions in concrete. Data are also needed on the long-term strength characteristics and creep of the concrete incorporating MK type materials.

Acknowledgement

The MK material was provided by Englehard Corp., Iselin, New Jersey, USA. The authors acknowledge the assistance of Messrs. R. Chévrier and R. Canaj for the laboratory work on concrete and the assistance from J. Szymański, P. Carrière, L. Morin, and A.J. Hanson for determining the physical and chemical characteristics of the material.

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