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Report No. CBU-2006-02
REP-601
October 2006

For presentation and publication at the Terry Holland Symposium, Warsaw, Poland, June 2007.

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Use of Wood Micro-Fibers to Increase Freezing and Thawing Durability of Concrete

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Synopsis: Fibrous residuals generated from several pulp and paper mills were included in concrete. The average length of wood micro-fibers from these residuals was approximately 0.9 to 1.7 mm. By using the proper amounts of the fibrous residuals, water, and high-range water-reducing admixture (HRWRA), concrete mixtures containing wood micro-fibers were produced nearly equivalent to a reference concrete (no fibers) in slump and compressive strength. In general, the amount of HRWRA increased in proportion to the amount of wood micro-fibers incorporated into the concrete. At a somewhat lower level of compressive strength, these non-air entrained concrete mixtures containing wood fibers showed equivalent length change (drying shrinkage), and equivalent or lower resistance to chloride-ion penetration and abrasion when compared with the reference concrete without fibers. On the other hand, five out of the seven available sources of wood micro-fibers greatly improved the resistance of these non-air entrained concrete mixtures to freezing-and-thawing (FT) and salt-scaling. An outdoor demonstration slab was also constructed in 2002, and test specimens were cast for evaluation of resistance to FT and resistance to salt-scaling. Test results again showed that non-air entrained concrete could be made highly resistant to FT with the use of the wood micro-fibers from pulp and paper mills. It was concluded that concrete without entrained air could be made resistant to FT with the proper use of micro-fibers from pulp and paper mills.

Keywords: abrasion resistance; chloride-ion penetration; compressive strength; concrete pavements; deicing salt scaling; drying shrinkage; durability; freezing and thawing; micro-fibers; wood cellulose fibers.

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INTRODUCTION

Pulp and paper mill wastewater treatment plant residuals (also called sludge) are the solid residue removed from mill wastewater before the water is discharged into the environment or reused in the mills. Residuals are removed via a two-step process of treating the wastewater [1-5]. A primary residual is the solid removed from the primary clarifier. Primary clarification is usually carried out by sedimentation and sometimes by dissolved air flotation. In the sedimentation process, chemical additives are used to make the non-settleable solids settleable through flocculation. A primary residual consists mainly of wood cellulose fibers, papermaking fillers (kaolinitic clay, calcium carbonate, and/or titanium dioxide), and water. In some cases, ash generated by the mill and inert solids rejected during chemical recovery processes become part of the primary residual. The water clarified by the primary treatment is passed on to the secondary treatment. The secondary treatment is usually a biological process in which micro-organisms convert soluble organic matter to carbon dioxide and water while consuming oxygen. A secondary residual is mainly microbial biomass (also called biosolids) grown during this process and removed through clarification. Many times primary and secondary residuals are combined to facilitate handling. In most cases, the residuals are dewatered before disposal or beneficial use.

Because of the cellulose fibers present in the residuals, the use of the residuals as a source of wood microfibers in concrete could become an economical and beneficial alternative to other use options or landfilling [8-11].

OBJECTIVES

This research was conducted to establish mixture proportions for concrete containing the fibrous residuals from pulp and paper mills and to determine the benefits of using the residuals in concrete.

MATERIALS

Cement, sand, coarse aggregate, and chemical admixture

ASTM Type I portland cement was used in this research. The sand used in this research had bulk density of 1800 kg/m^3, specific gravity of 2.73, absorption of 1.3%, and fineness modulus of 2.88. Crushed stone with a 19-mm maximum size was used in the laboratory. The crushed stone had bulk density of 1570 kg/m^3, specific gravity of 2.67, and absorption of 0.4%. Gravel with a 19-mm maximum size was used in the field demonstration concrete. The gravel had bulk density of 1600 kg/m^3, specific gravity of 2.72, and absorption of 1.0%. The sand and the coarse aggregates met the requirements of ASTM C 33. The HRWRA used was a carboxylated polyether liquid admixture meeting the requirements of ASTM C 494.

Fibrous residuals

In total, seven sources of pulp and paper mill residuals were used representing a wide variation in the type of wood fibers and processes. Table 1 presents the type, physical properties, loss on ignition (LOI), and wood fiber contents of the residuals. Table 2 presents the mineralogical composition of the residuals. Fig. 1 shows scanning electron micrographs (SEM) of oven-dried samples of the residuals.

Deflocculation (or "repulping") of residuals

As-received residuals contained fibrous clumps that consisted of wood fibers (Residual BR), clay (in Residuals C1, C2, I, S, & WV), and/or other particulates (in Residuals C2, I, S, WG, & WV). Had these clumps been included in concrete, they would have remained clumps in the hardened concrete. For fibers to function as strengthening filaments in concrete, they must be separated into well-dispersed individual fibers.

Therefore, all seven sources of residuals were deflocculated, or "repulped", into separated wood fibers and particulates before they were introduced into concrete mixtures [9]. The "pulper" used for this purpose in the concrete laboratory at the University of Wisconsin-Milwaukee (UWM) consisted of a 19-liter (5-gal.) plastic bucket and a high-speed mixer with a two-blade propeller positioned above the bottom of the bucket. The propeller blades subjected mixtures of water and fibrous residuals to high-speed rotation for at least 20 minutes. It was easy to repulp Residuals BR and C1. It was relatively easy to repulp Residuals C2 and WG. On the other hand, it was hard to repulp Residuals S, I, and WV. The main reason for the difficulty was that Residuals S, I, and WV were relatively thoroughly dewatered at the mill wastewater-treatment plants.

SPECIMEN PREPARATION

Concrete mixing in the laboratory was done in accordance with ASTM C 192 using a revolving-drum tilting mixer. The properties of freshly mixed concrete were determined, and test specimens were cast for the evaluation of mechanical and long-term properties of concrete.

In general, compressive strength of each mixture was determined by testing three 100 × 200 mm cylinders at each test age. Flexural strength was determined by testing three 100 × 100 × 400 mm beams at each test age. The length change specimens used were 75 × 75 × 285 mm prisms, which were stored in lime-saturated water until the age of 28 days and subsequently stored in a drying room maintained at 23 ± 2°C and a relative humidity of 50 ± 4%.

PRELIMINARY INVESTIGATION

A series of preliminary concrete mixtures were made to establish mixture proportions for concrete containing the fibrous residuals [8, 10]. Major findings from the preliminary investigation were as follows:

(1) The fibrous residuals did not affect the compressive strength development of concrete.

(2) By using a proper combination of residual and HRWRA contents, the slump and compressive strength of concrete can be adjusted.

(3) By achieving an equivalent density, concrete mixtures containing the residuals can be produced equal in slump and strength to a reference concrete (no residuals).

MAIN LABORATORY MIXTURES

Mixture proportions

Based on the mixture proportions established during the preliminary investigation, concrete mixtures were produced in the laboratory in two groups: (1) Reference 1 (no residuals), C1, WG, C2, and WV; and (2) Reference 2 (no residuals), BR, I, and S. The as-received residual content used in concrete mixtures was 0.65% for C1, C2, WG, WV, I, and S; and, 0.35% for BR based on the mass of concrete. An air-entraining admixture was not used. Table 3 presents the mixture proportions and fresh properties of the main laboratory concrete mixtures.

Depending on the source of residuals, the amount of wood fibers in concrete ranged from 2.4 to 4.9 kg/m^3 (4.0 to 8.3 lb/yd^3) on oven-dry basis. In order to achieve a target slump of 75 to 150 mm (3 to

6 in.), the amount of HRWRA was increased in an approximate proportion to the amount of wood fibers in concrete. The density of concrete mixtures was almost uniform. Overall, the air content of concrete mixtures containing the residuals was comparable to that of the reference concrete mixtures (1.9 vs. 1.8% on average).

Compressive strength

Fig. 2 presents the compressive strength of concrete. On the whole, the 28-day compressive strength was about 43 MPa (6300 psi) on average. In these particular groups of mixtures, the 28-day strength of the concrete mixtures containing the residuals was about 15% lower compared with the reference mixtures. The reference and residual-containing concrete mixtures showed similar patterns of strength development.

Length change (drying shrinkage)

Fig. 3 shows the length change (drying shrinkage subsequent to 28 days of immersion in water) of concrete mixtures. C1, C2, WG, and WV concrete mixtures showed somewhat higher drying shrinkage than their reference concrete (Ref. 1). BR, I, and S concrete mixtures showed slightly lower drying shrinkage than their reference concrete (Ref. 2). Thus, overall, the drying shrinkage of residual-containing concrete mixtures was similar to that of the reference mixtures.

Resistance to chloride-ion penetration

The test results for chloride-ion penetrability into concrete (ASTM C 1202) are presented in Table 4. A lower electrical charge passed implies a higher resistance of concrete to chloride-ion penetration. The chloride-ion penetration resistance of the residual-containing concrete mixtures was lower than the Reference Concrete; however, qualitatively, all concrete mixtures had equivalent chloride-ion penetration resistance.

Abrasion resistance

Fig. 4 presents the test results for mass loss of concrete due to abrasion (ASTM C 944 using double load (197 N)). A lower mass loss implies a higher abrasion resistance of concrete. In general, the abrasion resistance of the residual-containing concrete was somewhat lower than the Reference Concrete. The abrasion mass loss of the concrete WV was about three times as much as that of Reference 1 concrete.

Resistance to freezing and thawing (FT)

The test results for freezing and thawing durability factor (ASTM C 666, Procedure A) of concrete are presented in Table 4. The test results for the changes in relative dynamic modulus of elasticity of concrete due to cycles of freezing and thawing are shown in Fig. 5. Concrete containing residuals generally showed a much higher resistance to freezing and thawing than the Reference Concrete. All of the concrete mixtures did not contain any air-entraining admixture. Four out of seven residual concrete mixtures showed a durability factor of 85 or higher. Overall, the durability factor of residual-containing concrete was twice as high (64 vs. 32 on average) as the Reference Concrete. This could be attributed to the reduced rate of crack propagation due to micro-fiber reinforcement of concrete [6,7] by cellulose fibers. Also, the cellulose fibers are like thin-walled tubes with closed ends. Possibly, these central canals of fibers might have provided the space into which the freezing water expanded into without cracking/damaging the concrete.

Resistance to salt scaling

The test results for scaling resistance of concrete surfaces exposed to a deicing salt (ASTM C 672) are presented in Table 4, and in Figs. 6 and 7. The concrete specimens were subjected to cycles of freezing and thawing in the presence of calcium chloride. All concrete mixtures did not contain any air-entraining admixture. Concrete containing residuals generally showed much higher salt-scaling resistance than the Reference Concrete. Overall, concrete containing fibrous residuals withstood 2.8 times as many salt-scaling cycles as the Reference Concrete (85 vs. 30 cycles on average) before reaching severe surface scaling. This again could be attributed to the reinforcement of the concrete by wood fibers.

CONSTRUCTION DEMONSTRATION AND TESTING

In order to demonstrate the use of paper mill fibrous residuals in concrete construction, a demonstration slab was constructed outdoors using Residual C1 [11]. The demonstration site was near the Residual C1 source mill. Nearly five cubic meters of concrete was produced at a ready-mixed concrete plant, test specimens were cast, and a concrete slab-on-ground was constructed. The mixture proportions and fresh properties of the field demonstration concrete (Mixture C1 – Field) are included in Table 3. An air-entraining admixture was not used. The 3-day, 7-day, and 28-day compressive strength of the concrete were 32.5, 41.4, and 51.8 MPa, respectively. The 3-day, 7-day, and 28-day flexural strength of the concrete were 4.4, 4.9, and 6.2 MPa, respectively.

Fig. 8 shows the resistance of the concrete test specimens to FT (ASTM C 666, Procedure A). Although the concrete was not air entrained, it exhibited a high resistance to FT. The relative dynamic modulus of elasticity of concrete specimens remained above 95% when subjected to 300 cycles of FT. Fig. 9 shows the deicing salt scaling of the concrete test specimens (ASTM C 672). When subjected to 50 cycles of FT in the presence of calcium chloride, the surface showed moderate to severe scaling (visual rating of 4).

CONCLUSIONS

Based on the data presented, the following conclusions can be drawn:

(1) By using proper amounts of fibrous residuals, water, and HRWRA, the concrete mixtures containing the fibrous residuals were produced nearly equivalent to reference concrete mixtures (no residuals) in slump and compressive strength. In general, HRWRA was used in proportion to the amount of wood fibers in concrete.

(2) In general, the length change (drying shrinkage) of the concrete mixtures containing the residuals was equivalent to that of the reference concrete mixtures. The residual-

containing concrete showed equivalent or lower resistance to chloride-ion penetration and abrasion when compared with the reference concrete without fibers.

(3) The wood cellulose fibers in a fibrous residual significantly enhanced the resistance of non-air-entrained concrete to FT, bringing the resistance up to the level of air-entrained concrete.

(4) Use of pulp and paper mill fibrous residuals in concrete can save the pulp and paper industry disposal costs and produce a "greener" concrete for construction, and at the same time produce durable concrete.

ACKNOWLEDGEMENTS

The writers express their deep gratitude to the U.S. Department of Energy - Agenda 2020 Program; National Council for Air and Stream Improvement (NCASI), Research Triangle Park, NC; Weyerhaeuser Company, Federal Way, WA; and Stora Enso North America Company, Wisconsin Rapids, WI, for providing major funding for this project.

The UWM Center for By-Products Utilization was established in 1988 with a generous grant from the Dairyland Power Cooperative, La Crosse, WI; Madison Gas and Electric Company, Madison, WI; National Minerals Corporation, St. Paul, MN; Northern States Power Company, Eau Claire, WI; We Energies, Milwaukee, WI; Wisconsin Power and Light Company, Madison, WI; and, Wisconsin Public Service Corporation, Green Bay, WI. Their financial support and additional grant and support from Manitowoc Public Utilities, Manitowoc, WI, are gratefully acknowledged.

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Table 1 – Type, physical properties, loss on ignition (LOI), and wood fiber contents of the residuals.

Residual[a,b]	Moisture Content (%)	Specific Gravity	Bulk Density (kg/m^3)	Fiber Length[c] (mm)	LOI (%)	Wood Fiber (%)
C1	185	1.77	1080	1.22	54.9	43
C2	220	1.69	1000	1.20	73.1	64
I	95	2.04	830	0.85	49.7	40
S	84	2.00	660	1.11	57.9	49
WG	116	2.17	750	1.51	43.6	35
WV	142	1.62	570	1.68	82.3	77
BR	230	1.56	450	1.34	99.6	94
Avg.	153	1.83	760	1.27	65.9	57

[a] Type: Primary, except for BR which is fiber reclaim.
[b] Fiber source: Virgin, except for I (recycled) and S (80% recycled, 20% virgin).
[c] Length-weighted average.

Table 2 – Mineralogical composition of the residuals by powder diffraction analysis (% by mass).

Mineral	C1	C2	I	S	WG	WV
Calcite, $CaCO_3$	0	15	51	36	63	21
Kaolinite, $Al_2Si_2O_5(OH)_4$	52	14	16	7	0	2
Magnesite, $MgCO_3$	0	0	0	7	0	0
Quartz, SiO_2	0	5	0	1	2	3
Talc, $Mg_3Si_4O_{10}(OH)_2$	0	< 1	0	3	0	0

Table 3 – Mixture proportions of concrete.

Mixture Designation	Ref. 1	C1	C2	WG	WV	Ref. 2	BR	I	S	C1 – Field
Wood Fibers from Residuals* (kg/m^3)	0	2.4	3.1	2.5	4.9	0	2.4	3.3	4.2	1.8
Cement (kg/m^3)	368	360	363	359	361	367	365	368	363	329
Water (kg/m^3)	156	149	156	162	158	158	151	156	159	119
Water/Cement, *w/c*	0.43	0.41	0.43	0.45	0.44	0.43	0.41	0.42	0.44	0.36
Sand, SSD (kg/m^3)	855	835	850	835	840	850	845	855	840	860
Stone, 19-mm maximum, SSD (kg/m^3)	1050	1030	1030	1020	1030	1050	1040	1050	1030	1050
Residuals, as-received moist (kg/m^3)	0	15.6	15.7	15.6	15.7	0	8.5	16.0	15.7	11.6
HRWRA (L/m^3)	0.81	1.79	2.97	2.25	3.36	0.82	1.47	3.50	5.51	1.77
Slump (mm)	115	90	150	180	125	75	125	90	75	210
Air Content (%)	1.6	2.8	1.6	1.8	1.7	1.9	2.3	1.3	1.8	3.0
Density (kg/m^3)	2430	2390	2420	2400	2410	2420	2410	2440	2410	2370

* As-received moist fibrous residuals were used. The quantities of the fibers shown are on oven-dry basis.

Table 4 – Chloride-ion penetrability into concrete, freezing and thawing (FT) durability factor of concrete, and salt-scaling resistance of concrete.

Mixture Name	Ref. 1	C1	C2	WG	WV	Ref. 2	BR	I	S
Charge Passed (Coulombs)	3680	3740	4340	4780	5030	3600	3730	3650	4020
FT Durability Factor	21	94	44	14	23	42	94	85	92
Cycles to Reach Severe Scaling	30	85	65	65	30	30	140	85	125

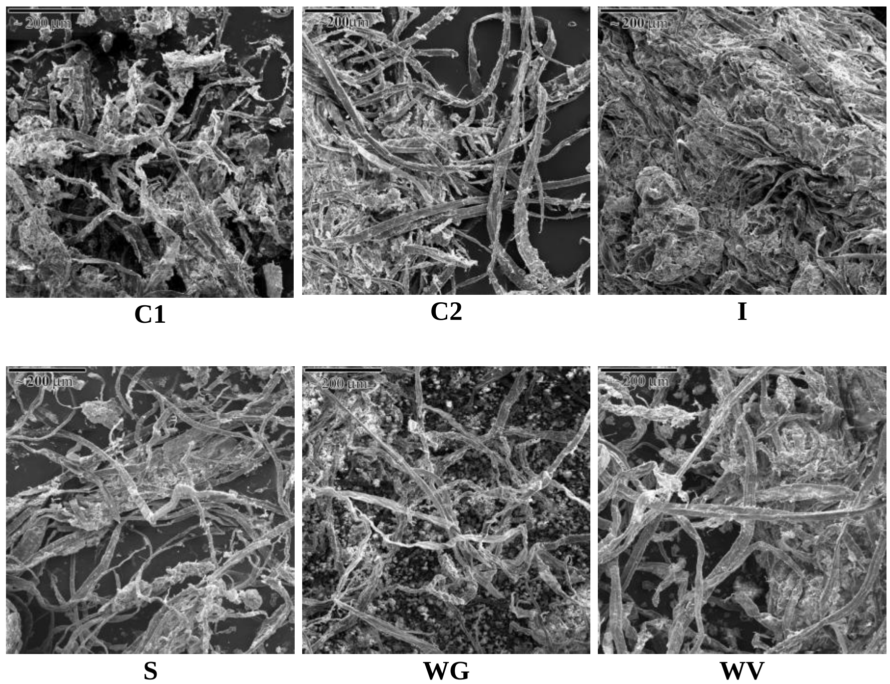
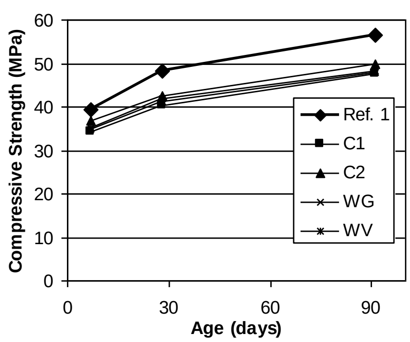
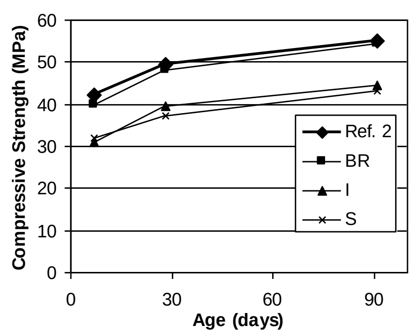


Fig. 1 − Scanning electron micrographs of the residuals.

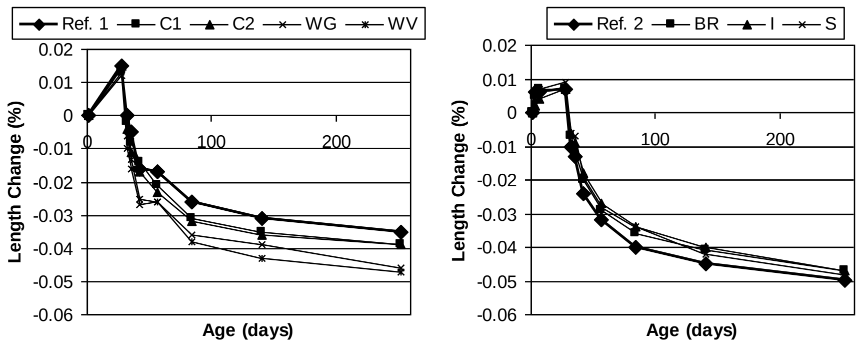


(a) C1, C2, WG, & WV.



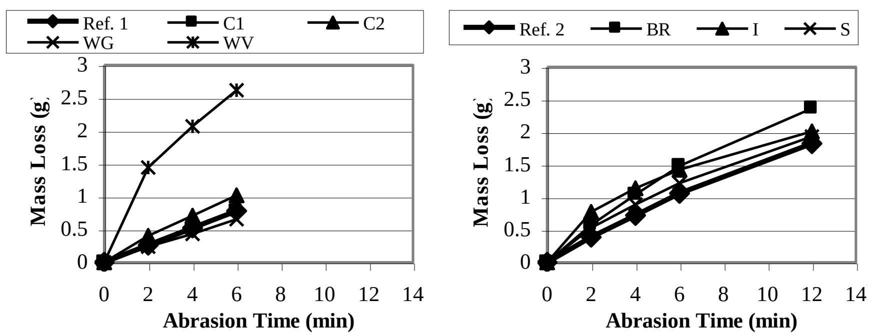
(b) BR, I, & S.

Fig. 2 – Compressive strength of concrete.



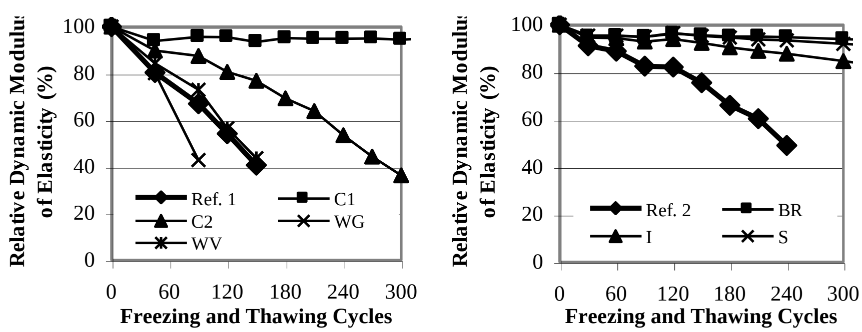
(a) Ref. 1, C1, C2, WG, & WV. (b) Ref. 2, BR, I, & S.

Fig. 3 – Length change of concrete.



(a) C1, C2, WG, & WV. (b) Ref. 2, BR, I, & S.

Fig. 4 – Mass loss of concrete specimens due to abrasion.



(a) C1, C2, WG, & WV. (b) Ref. 2, BR, I, & S.

Fig. 5 – Change in relative dynamic modulus of elasticity of concrete due to cycles of freezing and thawing

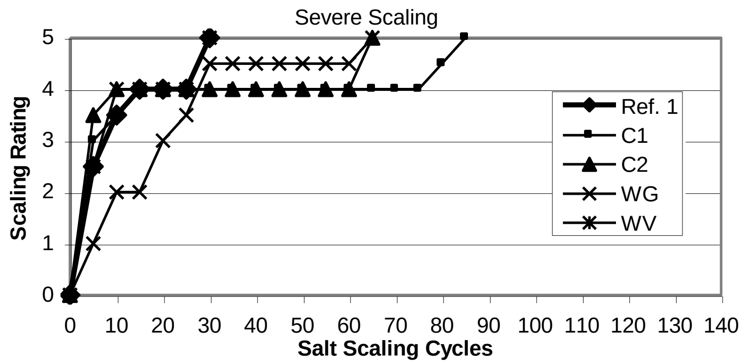


Fig. 6 – Salt-scaling of concrete (C1, C2, WG, WV)

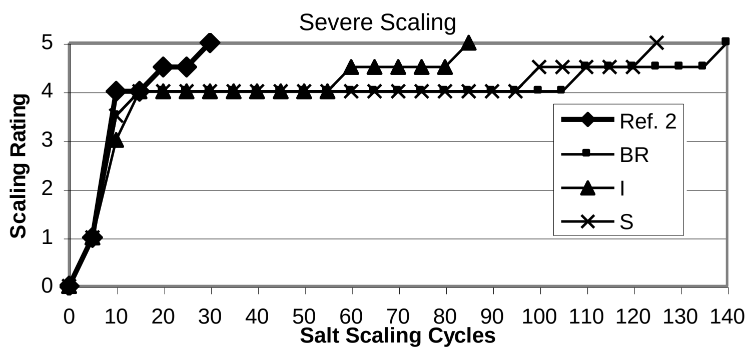


Fig. 7 – Salt-scaling of concrete (BR, I, S)

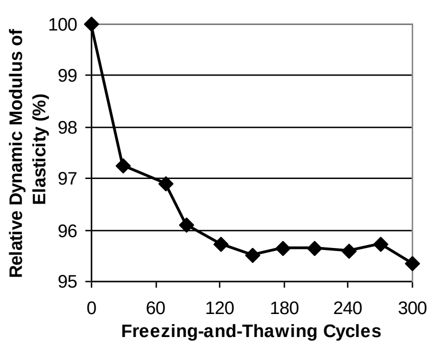


Fig. 8 – Freezing-and-thawing resistance of concrete used for field demonstration.

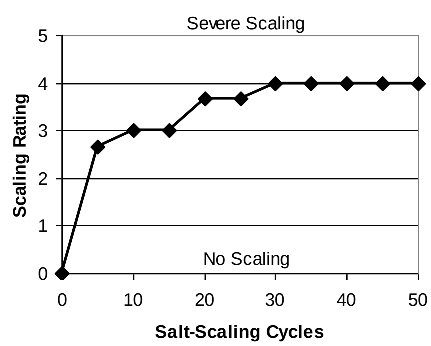


Fig. 9 – Salt scaling of concrete used for field demonstration.