Pergamon

Cement and Concrete Research, Vol. 25, No. 3, pp. 511-521, 1995

0008-8846/95 $9.50+.00

0008-8846(95)00040-2

STRENGTH AND DURABILITY OF POLYPROPYLENE FIBRE REINFORCED GROUTS

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(Refereed)
(Received January 13, 1994; in final form February 17, 1995)

REFEREED PAPERS

ABSTRACT

Fibrillated polypropylene fibres were added to cementitious grouts to determine whether improved mechanical properties and durability could be achieved. The grouts were studied for suitability as subsurface containment barriers around stabilized hazardous waste landfills. Strength, wet-dry and freeze-thaw durability and shrinkage crack control were investigated. Fibres added at volume fractions of 0.1 and 0.2% were found to reduce crack widths of restrained shrinkage specimens by bridging action. Compressive and flexural strengths were not consistently affected by incorporation of fibres. Fibres did not significantly change the residual compressive strength of air entrained grouts subjected to freeze-thaw cycles.

Introduction

Cementitious grouts are being evaluated for subsurface containment barriers around a chemical waste landfill at Sandia National Laboratories in New Mexico as part of the Mixed Waste Landfill Integrated Demonstration sponsored. The waste will be stabilized to reduce mobility of hazardous species prior to installation of impermeable, durable subsurface barriers and caps to isolate landfill contents. The landfill is located in the vadose zone of an arid environment. Further details of this project are available elsewhere (1,2). The results of this study on fibre reinforced grouts are also relevant to other applications where improved performance is sought.

Integrity of grout barriers is compromised if an unacceptable degree of cracking occurs. Therefore, fibre reinforcement was investigated as a means of decreasing the risk and extent of cracking and improving durability. Fibrillated polypropylene fibres were added to grouts to study potential improvement in durability that may arise if the fibres can toughen grouts and reduce the extent and width of cracks. Polypropylene fibres were chosen in preference to steel fibres to minimize segregation in the fluid grouts. Previous work has shown that pumpable grouts with permeabilities of the order of 10^{-11} to 10^{-10} cm/s can be formulated and that addition of fibrillated polypropylene fibres does not alter permeability significantly (2). The influence of mix proportions and fibre reinforcement on strength, wet-dry cycling durability, freeze-thaw durability and restrained shrinkage cracking resistance are reported in this paper. Since the proposed subsurface barriers will surround waste that has been chemically and physically stabilized, there is no major threat of leachate interaction with grout.

Experimental Procedure

Materials

Superplasticized grouts based on Type I (ordinary Portland) cement were studied. Wyoming bentonite was added to the grouts to prevent excessive bleeding and settling. The superplasticizer used was a sodium salt of sulphonated naphthalene formaldehyde condensate with a solids content of 42% by mass. For the freeze-thaw tests a vinsol resin air entraining agent was added at a rate of 1 ml/kg cement.

Three grout mixes containing silica sand were evaluated and compared with an unsanded cement grout. Unsanded grout is not recommended for barriers or other relatively thick sections due to lack of shrinkage resistance. One grade of sand had 98% of particles between sieve sizes 200 and 40 (74 to 420 μm) and a coarser sand with 98% of particles between sieve sizes 60 and 30 (250 to 595 μm). One mix contained a blend of the two sand grades so that an increased proportion of sand could be used without causing an excessive increase in water demand. The mix proportions of the unreinforced grouts are given in Table 1. The letters "f" and "c" refer to fine and coarse sand respectively.

TABLE 1. Mix Proportions of Grouts

Mix	Cement (kg/m^3)	Water (kg/m^3)	Sand (kg/m^3)	Bentonite (kg/m^3)	Superplasticizer (l/m^3)
1	1435	545	0	10	21.5
2	795	437	795 (f)	22	15.9
3	832	399	832 (c)	20	16.6
4	769	384	538 (c) 384 (f)	19	15.4

The fibres used were 19 mm long collated, fibrillated polypropylene. Relatively low volume fractions of 0.1 and 0.2% were used to retain pumpability of the grouts and as an attempt to prevent clumping, or "balling", of fibres. The fibre reinforced grouts are identified by the % volume fraction and the letter "F" following the Mix Number. For example, 1 0.1F refers to Mix 1 reinforced with 0.1% volume fraction fibres.

Mixing and Curing

Grouts were mixed in a planetary type mixer. Fibres were added incrementally to the grout to prevent clumping and mixed until a uniform distribution was achieved. The hardened grouts were demoulded 24 hours after casting and cured for 28 days. Two different types of curing were employed. Wet curing by submersion in water at 25°C was conducted to determine the properties when adequate cement hydration occurs. Simulation of the in-situ curing conditions for subsurface barriers was achieved by burying 24 hour old specimens in 200 litre drums of site soil that contained 8% moisture by mass.

Compressive and Flexural Strength

Cylindrical compressive strength measurements were performed in accordance with ASTM C 39-86. The cylinders were 75 mm diameter and 150 mm long. At the completion of the 28 day curing period the compressive strength of six specimens per batch was measured. Both wet and soil curing conditions were used for compressive strength tests.

Prismatic beams 300 mm x 50 mm x 50 mm of selected grouts were cast and cured by burial in soil and measured for flexural strength. Third-point loading following ASTM C 78-84 was conducted on 6 specimens per batch for Mixes 1 to 3 and 12 specimens per batch for Mix 4.

Restrained Shrinkage

Shrinkage cracking tendency of the unreinforced and fibre reinforced grouts was studied using restrained shrinkage (shrinkage ring) tests. The objective of the tests was to compare the shrinkage cracking resistance of the grouts under simulated environmental conditions. In practice, the barriers will be subjected to internal restraint arising from moisture variations from the surface to the interior. The restrained shrinkage test has been used to study shrinkage cracking of cementitious materials and the effect of fibres and admixtures (3-9).

Grouts were cast in an annular shape around a rigid steel ring. The grout annulus, or ring, had an inner radius of 82.5 mm, an outer radius of 125 mm and a height of 65 mm. Twenty four hours after casting, the outer mould was removed and the grout ring bonded to the steel ring was buried in oven dried soil (0% moisture) to simulate dry subsurface exposure conditions. Shrinkage was restrained by the inner steel ring and radial cracks eventuated. Specimens were examined daily and the time to cracking and the crack width at failure were noted. After failure, the specimens were removed from the soil and the top and circumferential surfaces were exposed to air at 25°C and 40-50% relative humidity. Crack width was monitored with time as the specimens continued to dry and shrink. Exposure to air after initial cracking allowed study of ongoing crack opening and the role of fibres in crack width control.

Wet-Dry Cycling

The durability of the grouts to wet-dry cycles was investigated. Grout cylinders of 75 mm diameter and 150 mm length were cured in water for 28 days and then cycled by submersion in water at 25°C for 5 hours followed by drying in air at 25°C and relative humidity of 40-50% for 43 hours. The 48 hour cycle was repeated 12 times. The drying temperature of 25°C was used in preference to higher values to simulate subsurface temperatures to which the grouts will be exposed in service. Drying at 25°C was sufficient to cause loss of surface moisture. More aggressive wet-dry cycling at higher temperatures would be expected to increase cracking associated with stresses induced by repeated shrinkage and swelling of the surface layer. At the completion of 12 cycles the ultrasonic pulse velocity and residual compressive strength were measured. Prior to strength testing, the specimens were soaked to increase the moisture content to a level similar to that of the wet cured specimens since moisture content can effect results (10). Ultrasonic pulse velocity was measured following ASTM C 597-83. The values reported refer to those measured in the wet condition so that the effect of moisture content was minimized. The residual compressive strength and ultrasonic pulse velocity were compared to those measured after 28 days wet curing.

Freeze-Thaw Durability

The resistance of unreinforced and fibre reinforced versions of Mix 2 to freeze-thaw cycles was assessed using the method described in ASTM C 666-90, Procedure A. Although the subsurface barriers are unlikely to be subjected to extensive freeze-thaw cycles in the environment under consideration, the test gives an indication of the effect of fibres on durability. Beams 406 mm x 102 mm x 76 mm were cured in water at 25°C for 14 days. One beam of each material underwent freeze-thaw cycles while control beams were maintained in water at room temperature. The temperature range used in the freeze-thaw cabinet was -18 to 10°C. The beams were exposed to 112 cycles (14 days). Instead of measuring the dynamic modulus, as given in ASTM C 666, the compressive strength of 76 mm cubes cut from the cycled beams was measured and compared with that for cubes cut from the control beams. This gave an indication of relative compressive strength loss.

Results

Figures 1 and 2 show the mean 28 day compressive strength of the plain and fibre reinforced grouts for different curing conditions. The effect of fibres on mean 28 day flexural strength for the grouts cured in soil is shown in Figure 3. The error bars on these figures represent one standard deviation. Figure 4 shows the increase in crack width with time for plain and fibre reinforced versions of Mix 4 after the shrinkage ring was removed from soil and left to dry in air.

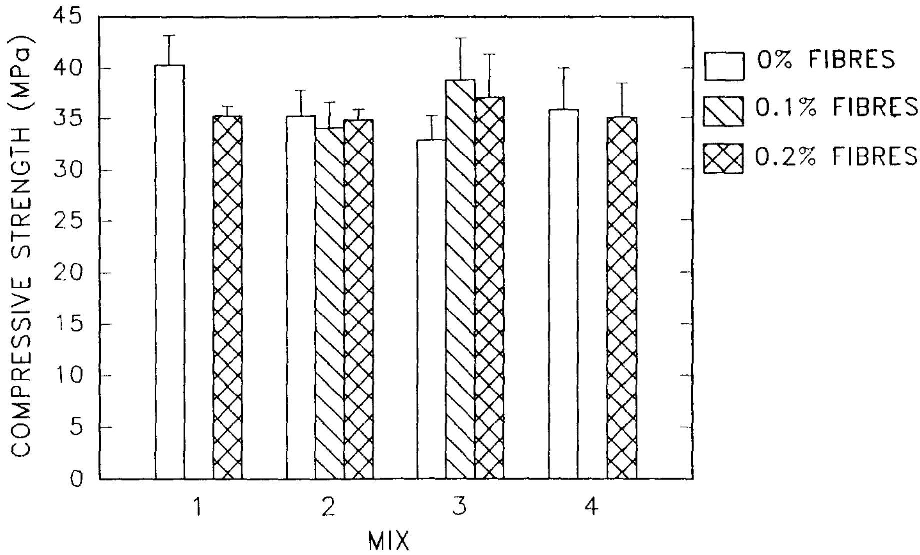

FIG. 1
Effect of Fibres on Compressive Strength of Wet Cured Grouts.

Table 2 indicates the residual strength and change in ultrasonic pulse velocity (UPV) measured on the specimens subjected to 12 wet-dry cycles at 25°C and re-soaked. The table reports the mean values ± the standard deviation

The cube compressive strengths of Mixes 2 and 2 0.2F after freeze-thaw cycling are compared with control specimens in Table 3. The mean and standard deviation are presented Both the

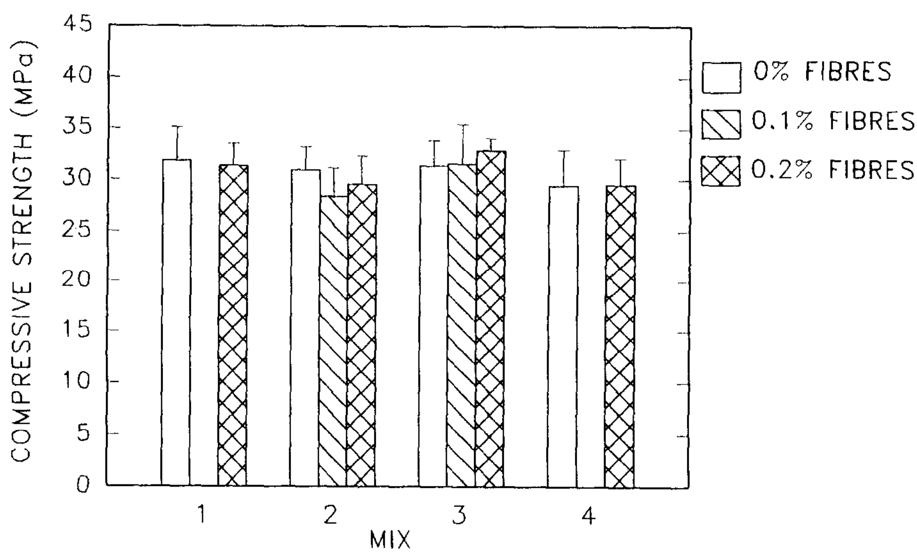

FIG. 2.
Effect of Fibres on Compressive Strength of Soil Cured Grouts.

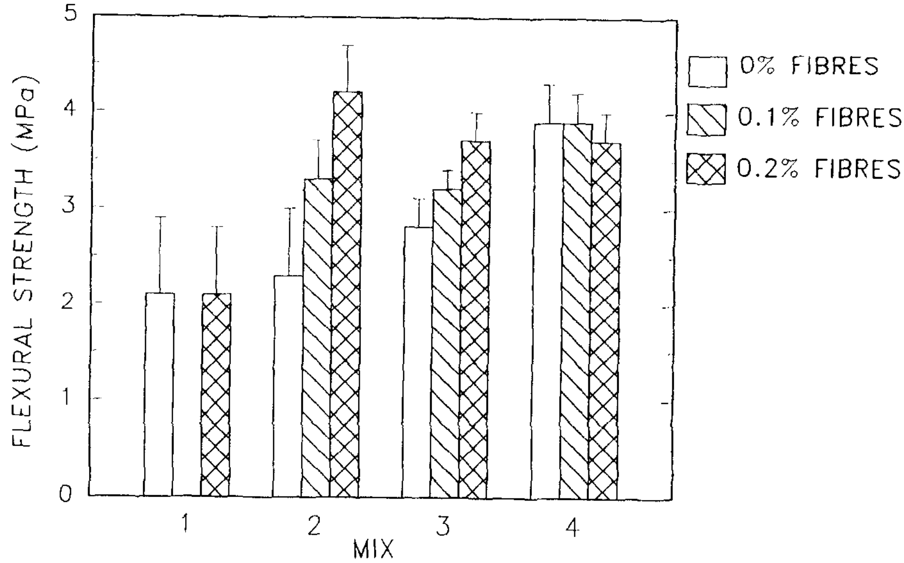

FIG. 3.
Effect of Fibres on Flexural Strength of Soil Cured Grouts.

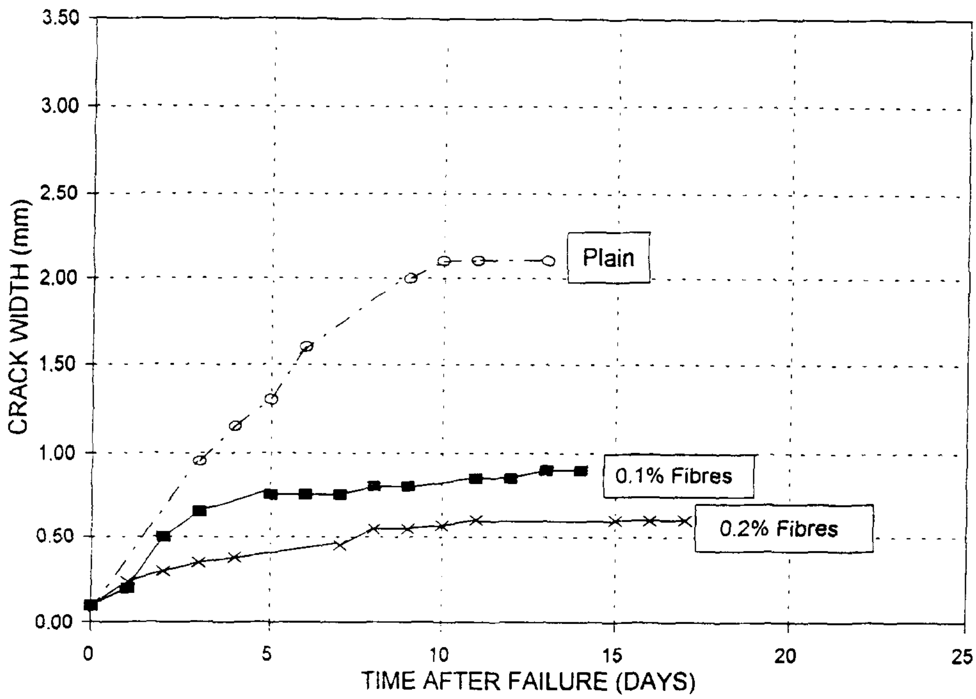

FIG. 4.
Crack Width versus Time After Failure for Mix 4.

TABLE 2. Residual Compressive Strength and Ultrasonic Pulse Velocity for Wet-Dry Specimens

Mix	Residual Strength (MPa)	% Change	Initial UPV (km/s)	Residual UPV (km/s)	% Change
1	18.1±3.2	-55.1	3.43±0.09	3.34±0.06	-2.6
1 0.2F	26.2±1.0	-25.5	3.14±0.10	3.40±0.04	+8.2
2	31.4±2.1	-10.8	3.45±0.05	3.45±0.03	0
2 0.1F	32.9±1.9	-3.2	3.29±0.06	3.42±0.02	+3.9
2 0.2F	33.7±1.3	-3.2	3.33±0.03	3.35±0.19	+0.6
3	37.2±2.2	+13.4	3.58±0.05	3.57±0.10	-0.3
3 0.1F	35.3±1.7	-8.8	3.46±0.11	3.48±0.21	+0.6
3 0.2F	34.5±2.0	-6.7	3.49±0.15	3.33±0.10	-4.6
4	38.6±4.1	+7.8	3.45±0.04	3.59±0.01	+4.0
4 0.2F	40.0±4.8	+14.3	3.63±0.05	3.69±0.00	+1.6

unreinforced and fibre reinforced grout beams subjected to freeze-thaw cycles displayed slight surface pock marks.

TABLE 3. Residual Cube Compressive Strength After Freeze-Thaw Cycles

Mix	Control Strength (MPa)		Residual Strength (MPa)	
	Mean	Standard Deviation	Mean	Standard Deviation
2	37.4	3.8	30.3	3.0
2 0.2F	39.1	4.1	34.7	3.5

Discussion

Compressive and Flexural Strength

The unreinforced grouts had similar compressive strengths when wet cured as indicated in Figure 1. The unsanded cement grout (Mix 1) shows the highest strength and this is a reflection of the low water/cement ratio (w/c). Failure occurred by splitting parallel to the direction of load application. The fibre reinforced grout remained contiguous after maximum compressive load was achieved due to fibre bridging across cracks whereas the unreinforced grouts fragmented into two or more separate pieces. In general, addition of fibres did not influence compressive strength. A two-tailed *t*-test was conducted at the 5% significance level to compare the sample means of the plain and fibre reinforced grout compressive strengths. It was assumed that the strengths were normally distributed with equal variances. The only significant differences were a decrease in strength for the wet cured fibre reinforced version of Mix 1 and an increase in strength for the wet cured fibre reinforced version of Mix 3. These differences are possibly due to batch variations. An increase in compressive strength due to incorporation of fibres was not expected since the elastic modulus of the fibres is low relative to the grout matrix and bond strength, while adequate, is not high.

Fibre reinforcement did not significantly influence the compressive strength of the soil cured grouts, as shown in Figure 2. Comparison between Figures 1 and 2 shows the expected decrease in compressive strength associated with reduction of hydration for soil curing.

The mean flexural strength of Mix 2 was increased at the 5% significance level by incorporation of polypropylene fibres for both 0.1 and 0.2% fibres, as indicated in Figure 3. Mix 3 showed an increase in mean flexural strength at the 5% significance level for 0.2% volume fraction of fibres. Flexural strength of the other grout mixes was not significantly altered by fibres. It was not obvious why Mix 2 should show greater improvement in flexural strength when fibres were added compared with the other grouts. It is possible that the fine sand in Mix 2 results in better distribution of fibres throughout the grout matrix. The high standard deviation associated with flexural strengths for the plain and fibre reinforced unsanded grout (Mix 1) is possibly due to presence of shrinkage cracks within the beams.

The fibre reinforced grouts were more ductile in flexure than their unreinforced counterparts. As was the case for the compressive strength tests, the fibre reinforced beams tested in flexure remained contiguous due to fibre bridging and the unreinforced beams split into two halves. Unreinforced grout completely failed at peak load, and load returned to zero. Fibre reinforced grout exhibited some load-bearing capacity after peak load was reached, and thus were pseudo-ductile. Some fibre pullout occurred, but enough fibres remained soundly bonded to prevent total separation.

Restrained Shrinkage

An important potential benefit of fibres is the reduction of shrinkage cracking and the reduction of any crack widths. This would be due to toughening of the grout that improves the resistance to crack growth. The fibres did not prevent formation of drying shrinkage cracks, but did reduce the crack widths. All specimens showed one radial crack at failure, except for Mix 3 which had two radial cracks.

The crack width at failure was typically 100 to 150 μm for unreinforced grouts and was approximately 100 μm when fibres were present. The time to cracking was not found systematically depend on the presence of fibres. Crack widths increased with time when the shrinkage rings were removed from soil and left to dry and shrink in air as depicted in Figure 4 for Mix 4. At a given time after failure the crack width of the fibre reinforced grout was less than the companion plain grout due to fibre bridging. It was also evident that the crack width at a given time decreased as the volume fraction of fibres increased. The reduction of crack width in shrinkage rings by fibres has also been demonstrated for carbon fibres in cement composites (5) and for cellulose and polypropylene fibres in concrete (8,9).

Wet-Dry Cycling

All of the specimens subjected to wet-dry cycling developed visible surface cracks by the second drying cycle. The unsanded grout, Mix 1, had the greatest tendency to crack and the surface crack widths were approximately 100 μm. The sanded grouts had surface crack widths lower than 100 μm and a lower visible surface crack density. The poor performance of the unsanded grout demonstrates the necessity to incorporate sand in grout formulations for barriers to reduce shrinkage and improve durability. Addition of fibres did not eliminate the appearance of surface cracks. The greatest improvement by addition of fibres was observed for Mix 1 where both the surface crack density and the crack widths were visibly reduced.

Visible surface cracking is not necessarily indicative of internal cracking. However, permeability tests on grouts subjected to wet-dry cycling have shown an increase in permeability that is attributable to interconnected microcracks within the specimens (11). Fibrillated fibres have been found to decrease the extent of permeable microcracking in wet cured grout after wet-dry cycles (11).

All grouts except Mixes 1 and 1 0.2 F showed a decrease in visible surface crack width several cycles after initial cracks had formed. The cracks appeared to be partially filled with white deposits. It is presumed that these deposits were calcium carbonate that led to partial crack healing. Calcium hydroxide probably leached into the cracks during the wetting cycle and this was converted to calcium carbonate by reaction with atmospheric carbon dioxide during the drying cycle. Such autogenous healing is frequently observed on concrete and has been reported in grout (12).

Sealing or partial sealing of cracks by this process would reduce permeability. For the proposed subsurface service conditions of the grouts it is uncertain as to whether crack healing would occur in the same manner as that during the wet-dry tests. This is because the availability of moisture for leaching of $Ca(OH)_2$ and of CO_2 for reacting with $Ca(OH)_2$ to form $CaCO_3$ would be reduced in comparison. Thus, crack healing by this mechanism may be slower and less extensive.

The greatest compressive strength losses were measured for Mixes 1 and 1 0.2 F which corresponded with the visual observations of the highest degree of surface cracking. Fibres did reduce the strength loss for Mix 1. Some mixes showed an increase in strength over that measured at 28 days, and this is probably due to ongoing hydration. The overall effect of fibres on the residual strength was masked by the increases in strength. It is also possible that an error is introduced by comparing 28 day and residual strength data from different batches. Thus, low changes in strength are probably negligible.

The measurement of ultrasonic pulse velocity was unable to distinguish the degree of cracking as indicated in Table 3. This is because the microcracks did not cause a significant increase in transit time. Addition of fibres did not result in a significant change in initial pulse velocity of specimens.

Freeze-Thaw Durability

The freeze-thaw performance of the unreinforced and fibre reinforced air entrained Mix 2 grouts was compared using a two-tailed *t*-test of the residual cube compressive strengths. The mean strengths of the control beams with and without fibres were not significantly different at the 5% level. For the beams exposed to freeze-thaw cycles, the mean residual strengths for the plain and fibre reinforced grouts were also not significantly different at the 5% level. Therefore, it appears that addition of 0.2% volume fraction of fibres did not significantly change the residual cube compressive strength. The lack of influence of fibrillated polypropylene fibres on the freeze-thaw durability of grouts concurs with other findings for this type of fibre in air entrained concrete (13). If grout or concrete does not contain an air entraining agent fibres may be able to reduce deterioration to a degree, but are unlikely to substitute for air entrainment.

Conclusions

Addition of fibrillated polypropylene fibres at volume fractions of 0.1 and 0.2% to cementitious grouts reduces width of cracks developed under restrained shrinkage conditions. Fibres do not significantly or invariably alter compressive strength, flexural strength or freeze-thaw durability of tested grouts but improve ductility. The wet-dry durability of unsanded grout is enhanced by incorporation of fibres. Although fibres do not significantly improve strength, the reduction of number of cracks and crack widths by fibres improves the performance of grouts used as containment barriers or in other applications where integrity is required.

Acknowledgement

This work was performed under the auspices of the U.S. Department of Energy, Washington, DC, under Contract No. DE-AC02-76CH00016. Prepared for the Department of Energy Office of Technology Development, Office of Environmental Management.

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