THE CURING AND STRENGTH CHARACTERISTICS OF COLD SETTING CIMENT FONDU GROUT

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Abstract

Several mix designs of a Ciment Fondu based grout were tested in the laboratory to develop a grout which would perform adequately as a pile backfill material in permafrost as cold as -10°C. The test procedure and results of thermal, compressive strength, and workability performance of the different mix designs are described in detail.

Résumé

Plusieurs mélanges de coulis à base de «Ciment Fondu» ont étés expérimentés en laboratoire afin de développer un coulis pouvant se comporter de façon adéquate comme matériel de remplissage pour des pieux implantés dans le pergélisol jusqu'à -10°C. L'expérimentation ainsi que les résultats en terme de performance thermique, de résistance en compression et de maniabilité pour les différent mélanges sont discutés en détail.

Introduction

The Department of National Defence (DND) is currently proceeding with the construction of the North Warning System (NWS) to replace the aging DEW Line System. The NWS includes 35 unmanned Short Range Radar (SRR) sites which span the Canadian mainland coastline from Alaska to Labrador. These sites encounter foundation conditions varying from ice-rich fine-grained soils to bedrock. The SRR facility foundation design was to be based on 100 mm pipe in 165 mm holes. The hole size of 165 mm was governed by the capability of an airtrack drill which would be used on most of the remote sites to bore the pile holes. One of the options which was examined was that of using a grout backfill which was required to cure with the surrounding soil as cold as -10°C.

There are essentially four ways to ensure that grout will cure adequately in a sub-zero environment: 1) the temperature of the grout may be artificially maintained above 0°C using external heat sources (this method is expensive and impractical for sub-surface cementing in frozen soils), 2) cements with rapid rates of hydration (evolving heat at a high rate) may be utilized to maintain the temperature of the grout above 0°C, 3) salts may be added to the cement to depress the freezing point of the mixing water and accelerators utilized to decrease setting time, and 4) utilize grouts with very low water contents which are designed to set quickly.

High alumina cements (such as Ciment Fondu) utilize their high rates of hydration, evolving heat very quickly to prevent freezing of the grout. Johnston and Ladanyi (1972) report successful use of Ciment Fondu for grouted anchors in warm permafrost (T > -1°C). Biggar and Sego (1989) report successful use of Ciment Fondu grout in soil and rock at temperatures between -5° and -7°C.

Testing program and procedure

General

This research program involved the testing of different grout mixes prepared using differing amounts of admixtures while keeping the cement: sand: water ratio the same. The objective was to produce a mix which would cure adequately at temperatures of -10°C yet be workable for field placement around piles. Cylinders of grout were cast in a frozen soil mold maintained at -10°C. Temperatures of the grout and surrounding soil were monitored during the first 24 hours of curing. Subsequently compressive strengths of the cylinders were measured at regular time intervals. In addition, strength tests were performed on cubes cast and cured at room temperature. Flow cone and vicat needle tests were carried out to assess grout workability.

The program consisted of three phases:

I) the first phase involved comparing the performance of a soil mold using a 600 mm mold (which had been used on tests conducted previously by Geocon (1988), Lafarge (1988(3)) and a constant temperature bath cell (CTBC) to determine if the CTBC would provide comparable curing conditions,

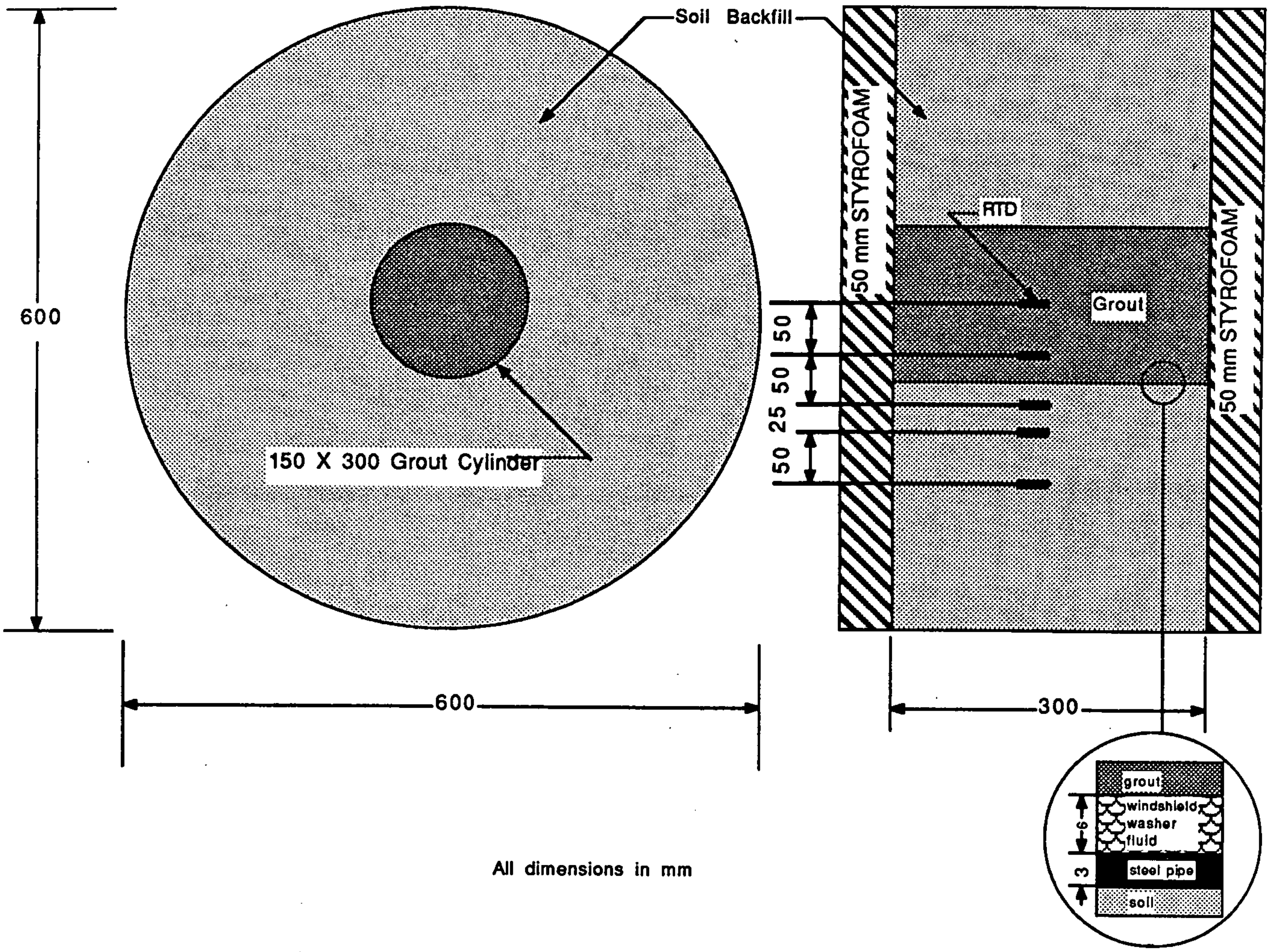

Figure 1. 600 mm, 200 litre cell.

II) the second phase involved varying the admixture proportions to produce an optimum mix design which was completely cured at -10 °C while providing adequate workability, and

III) the third phase involved utilizing the optimum mix design and varying the environmental conditions under which it was produced and cured.

CELL DESCRIPTION

A schematic diagram of the 600 mm mold used is shown in Figure 1. It consists of a 200 litre drum with a 50 mm thick layer of Styrofoam in the bottom, and a 300 mm thick by 600 mm diameter soil "donut" surrounding a steel pipe 160 mm in diameter by 300 mm long by 3 mm thick into which the 150 mm diameter grout cylinder mold could be placed. A 50 mm layer of Styrofoam was placed directly on top of the soil after the cylinder had been placed. Resistance Temperature Devices (RTD's) were placed in the soil to measure temperatures at distances of 25, 50 and 100 mm from the edge of the cylinder and in the centre and near the edge of the grout cylinder. The small annular space between the grout cylinder mold and the steel pipe was filled with antifreeze to ensure good thermal contact between the grout cylinder and the soil.

A schematic diagram of the CTBC is shown in Figure 2. It consists of a 300 mm thick by 300 mm diameter soil "donut" surrounding a steel pipe 160 mm in diameter by 300 mm long by 3 mm thick into which the 150 mm diameter grout cylinder mold could be placed. The soil "donut" was surrounded by a bath of antifreeze which contained coils through which glycol maintained at -10 °C was circulated. A 50 mm layer of Styrofoam was placed beneath the soil and cylinder and directly on top of the cell. Resistance Temperature Devices (RTD's) were placed in the soil at distances of 25 and 50 mm from the edge of the cylinder, one was placed in the fluid bath, and others were placed in the centre and near the edge of the grout cylinder. The moisture content of the soil was approximately 15 % with dry densities between 1650 and 1850 kg/m^3.

The operation of the CTBC should be described in more detail. By maintaining a near constant temperature 75 mm from the edge of the grout cylinder a steep temperature gradient is maintained in the frozen soil and a large heat sink

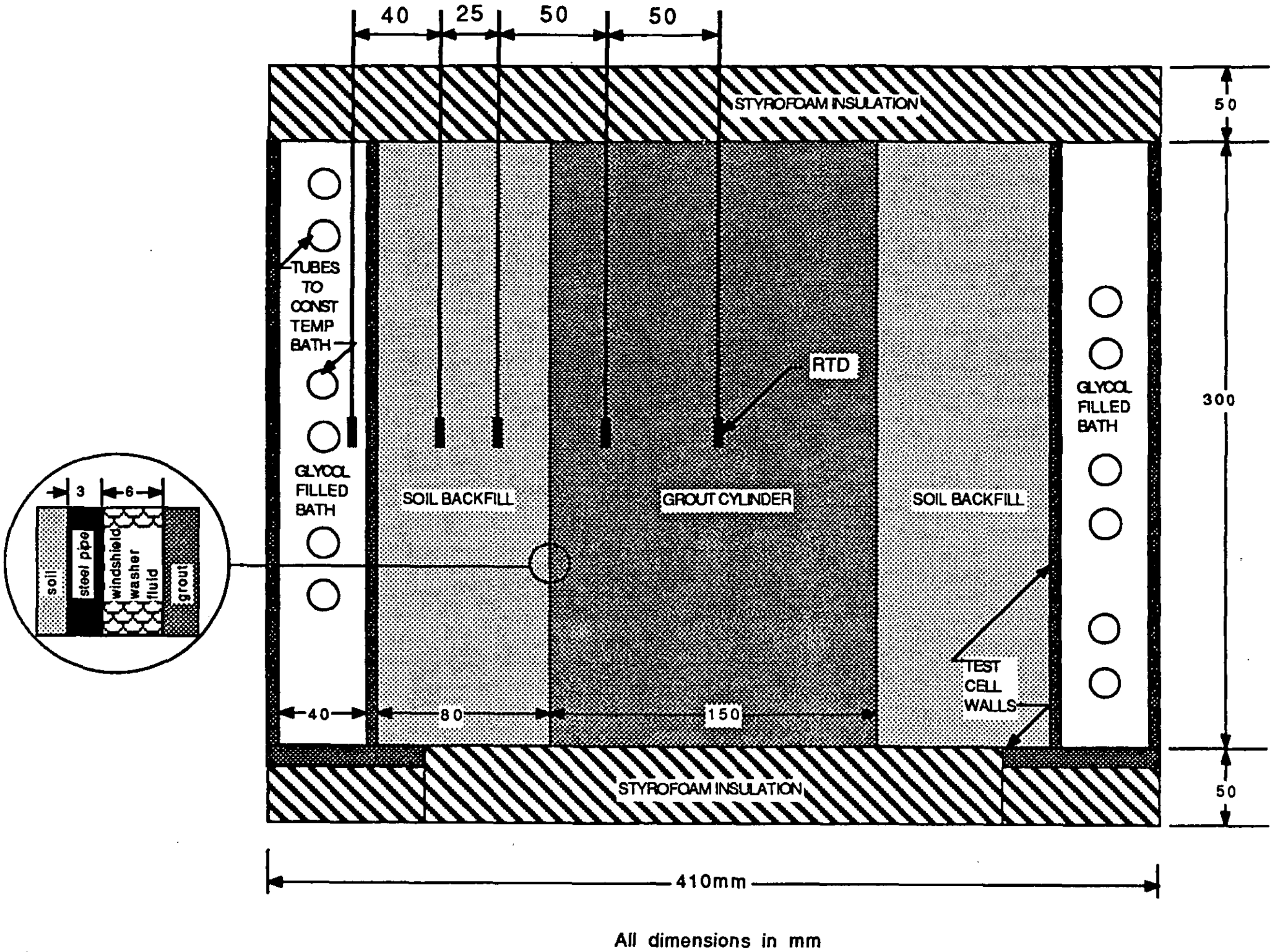

Figure 2. Constant temperature bath cell (CTBC).

is more closely modelled than by using the 600 mm cell which has its outer surface exposed to the ambient air temperature in the cold room. Studies of data from models similar to the 600 mm drum indicate that the temperature of the entire soil mass surrounding the grout cylinder may increase such that the temperature at the outer edge of the soil cell rises above the initial soil temperature (Geocon, 1988). The air in the cold room is a poor heat conductor, consequently, field conditions are poorly modelled. Thus the CTBC is believed to more closely represent field conditions. In any case, the temperature conditions in the CTBC will be more severe than with the 600 mm cell so that if the grout performs adequately in the CTBC it will certainly perform adequately in the 600 mm cell, and should perform adequately under field conditions.

Sample preparation

The mix designs of the grouts tested are shown in Table 1. The cement: sand: water ratio was maintained constant and only the proportion of the admixtures was changed. Only one accelerator (lithium carbonate) was used and one water reducing admixture, or superplasticizer (SPN), (Sulphinated Napthelene Formaldehyde Condensate) was used as well.

Phase I: A spatula mixer was utilized for this small batch size. Twenty kg of cement were added to the water over the first minute of mixing and the sand was added over the second minute. The temperatures of the components was 21 ±1 °C prior to mixing. The grout was mixed for a total of four minutes. The cylinders were prepared and placed within 20 minutes of starting the mixing.

Phase II: The mixes used 50 kg of cement (0.042 m^3) and proportionate amounts of the other components. All components of the mix were maintained at a temperature of 21 ± 1 °C prior to batching. The batch provided enough grout for 6 cylinders, the cubes, the flow cone, and the vicat needle tests. The grout was mixed in a 0.10 m^3 mortar mixer. The cement was added to the water over the first minute and the sand was then added over the second minute of batching. The grout was allowed to mix for approximately 1 minute at which time the admixtures were added. The grout was mixed for a total of 4 to 5 minutes. The flow cone test was carried out immediately after the mixing stopped and then the cylinders were cast and placed in the cold room. The grout could be poured directly from the mixer into the cylinders. Generally the cylinders were in the cold room within 10 minutes of mixing and in the cells within 20 minutes. The cubes were cast immediately after the flow cone test was carried out.

Table I: Grout Mix Desing

Mix#	Ciment Fondu (kg)	Sand (1) (kg)	Water (kg)	Accelerator (2) (g/%)	SPN (3) (g/%)
PHASE I					
I-A	20	8.9	7.0	0	150/.75
PHASE II					
II-1	40	17.8	14	0	300/.75
II-2	50	22.6	17.5	2.5/.005	0
II-3	50	22.6	17.5	7.5/.015	375/.75
II-4	50	22.6	17.5	2.5/.005	375/.75
II-5	50	22.6	17.5	5.0/.010	375/.75
II-6	50	22.6	17.5	5.0/.010	375/.75
II-7	50	22.6	17.5	2.5/.005	0
II-8	50	22.6	17.5	2.5/.005	375/.75
PHASE III					
III-9	50	22.6	17.5	5.0/.010	375/.75
III-10	50	22.6	17.5	5.0/.010	375/.75
III-11	50	22.6	17.5	5.0/.010	375/.75
III-12	50	22.6	17.5	5.0/.010	375/.75

Notes : 1) SIL SILICA, Silica sand, grade Sil-7
2) Lithium carbonate accelerator
3) Sulphinated napthelene formaldehyde condensate water reducing admixture (superplasticizer)

Phase III: The mixing of the grout in this phase of testing differed from the previous phases in that all of the dry components were blended together initially and then added to the water. The mix time remained the same (4 minutes), as did the sample preparation and testing procedure. The initial test was to compare the workability and curing temperatures of the optimum grout mix in which the dry components were preblended to the test results in which the dry components were individually added, under the initial environmental conditions (20 °C grout cured at -10 °C). Tests were then conducted with the preblended grout mix to examine the performance of placing colder grout (13 °C) cured at -10 °C, and placing 20 °C grout cured at -5 °C and +1 °C soil temperatures.

Strength testing

Constant rate of loading compression tests were conducted on 50 mm cubes cured in a moisture room at 20 °C after 6 and 24 hours which provided an index strength for the grout cured under normal conditions. Constant rate of loading compression tests were conducted on the cylinders cured at sub-zero temperatures at 12 hrs, 24 hrs, 7 days and 28 days. Prior to compression testing the cylinder was kept at room temperature until its centre was above 0 °C (approximately 6 hours) to ensure that ice bonding did not contribute to the strength.

Results

Comparison between the 600 mm cell and the CTBC

The results for mix I-A showed that temperatures at the outer edges of the cylinders in both cells reached 0 °C for approximately 3.5 hours. Figure 3 shows some of the temperature versus time curves for the grout and soil. The

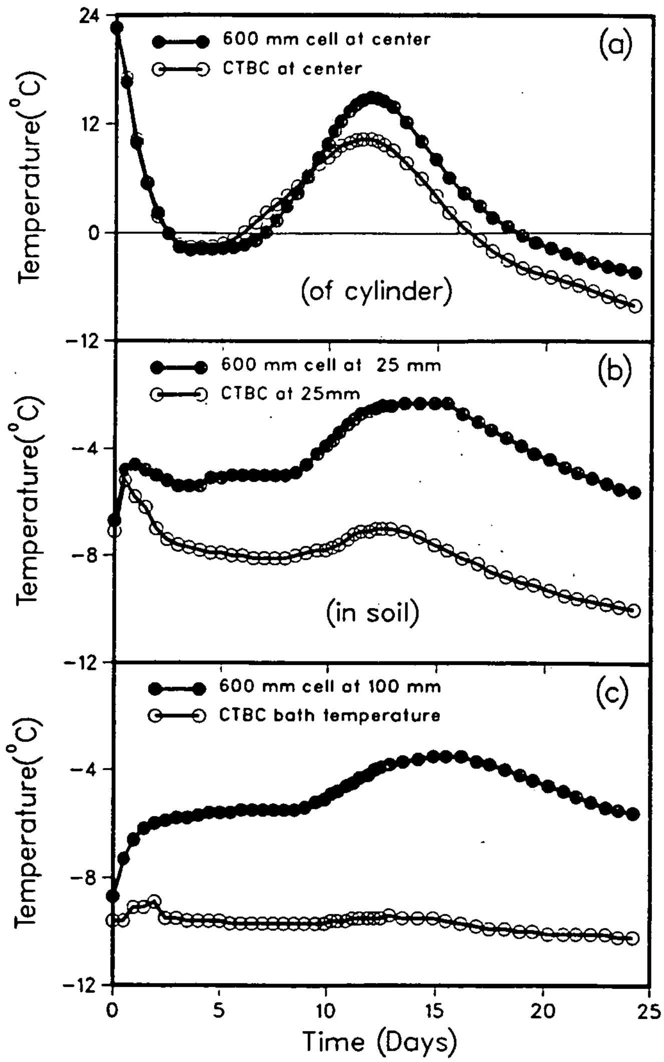

Figure 3. Comparison of thermal performance of two different test cells.

Table 2.
Grout testing summary

PHASE II

MIX	Min Temp (°C) @ time (hrs) (edge of cylinder)	Max Temp (°C) @ time (hrs) (centre of cyl)	Compressive Strengths (MPa) Cubes 6 hr	Cubes 24 hr	Cylinders 12 hr / 24 hr / 7da	Flow Cone (sec)	Vicat Needle (mins)	COMMENTS
II-1	0° @ 2.5<t<5.5	12° @ 11.5 hrs	- -	59.1	-- / 10.2 / 9.9	- -	- -	Sample had different structure 20 mm thick @ outer edge (likely frozen)
II-2	11° @ 1.0 hr	57° @ 3.0 hrs	35.0	52.7	36.8 / 32.9 / 32.6	35 (1)	100<t<135	Cardboard molds used on cylinders for temp measurement
II-3	16° @ 1 hr (see NOTE 3)	34° @ 3 hr (see NOTE 3)	26.6	36.0	29.0 / 29.6 / 38.3	too stiff	too stiff	Cardboard molds used on cylinders for temp measurement
II-4	4° @ 1.5 hr	32° @ 4 hrs	31.2	50.4	31.0 / 34.8 / 36.5	53.4 (2)	145<t<175	
II-5	2.3° @ 1.5 hr (see NOTE 3)	25° @ 4.0 hrs (see NOTE 3)	35.2	42.9	30.4 / 31.2 / 30.7	26.8	120	Cooling unit froze-up so had to defrost during test, thus temps are high.
II-6	0.4° @ 1.5 hrs	20° @ 3.5 hrs (see NOTE 5)	- -	46.5	34.2 / 36.4 / 34.2	24.1	120	
II-7	1.2° @ 1.6 hrs	14° @ 4.4 hrs	49.3	??	34.8 / 33.7 / 33.2	31.9 (2)	90<t<105	without SPN not as good a mix more lumps, and cement sticking to paddles.
II-8	0.5° @ 1.8 hrs (see NOTE 3)	11° @ 6.3 hrs (see NOTE 3)	27.4	51.4	-- / 32.1 / 36.3	33.5	169	failure with constant temp bath, temps in lab cell too high 12 mm outer layer with different structure observed (frozen)

NOTES:
1) Flow stopped at 35 seconds but the cone did not empty, estimate 50 - 75 mm left in bottom
2) Cone had to be manually shaken to complete flow as cone would not empty by itself
3) Temperatures from 600 mm cell grout cylinder
4) Compression tests done on 600 mm cell cylinder on 10 Apr
5) RTD only 38 mm from cell edge instead of 75 mm

PHASE III

MIX	Conditions Tg=Tgrout Ts=Tsoil	Min Temp (°C) @ time (hrs) (edge of cylinder)	Max Temp (°C) @ time (hrs) (centre of cyl)	Compressive Strengths (MPa) Cubes 6 / 24 hr	Cylinders 12 hr / 24 hr / 7 da	Flow Cone (sec)	Vicat Needle (mins)	COMMENTS
III-9	Tg = 20°C Ts = -10°C	3.9° @ 1.3 hrs (see NOTE 2)	25° @ 3.3 hrs (see NOTE 2)	28.0 / 42.2	31.8 / 33.7 / 37.2	15.0	84< t <104	Pump breakdown on bath #1 CTBC temps too high.
III-10	Tg = 13°C Ts = -10°C	1.2° @ 1.0 hrs	15° @ 3.9 hrs	37.1 / 51.3	-- / 28.0 / 25.8	25.8	95< t <115	3 mm thick outer layer with different structure observed (likely frozen)
III-11	Tg = 20°C Ts = -5°C	3.2° @ 1.0 hrs	25° @ 3.3 hrs	41.2 / 49.3	34.5 / 36.3 / 38.9 (see NOTE 4)	27.7 (1)	97< t <112	
III-12	Tg = 20°C Ts = +1°C	16° @ 1.3 hrs (see NOTE 3)	60° @ 2.8 hrs (see NOTE 3)	31.2 / 35.8	37.8 / 30.9 /	16.6	90 < t < 110	

NOTES:
1) Appeared to be small blockage at throat of cone
2) Temperatures from 600 mm cell as pump failure resulted in high temperatures in the other cell
3) Edge temperatures are for the constant temp bath cell, centre temperatures are for the 600 mm cell
4) Compression test for 7 day strength done on different loading machine than other tests, calibration required.

maximum temperature at the centre of the grout cylinder in the CTBC reached 10.3 °C compared to 15 °C in the 600 mm cell. The soil temperatures in the CTBC can be seen to be consistently lower than in the 600 mm cell. Subsequent tests duplicated this observed behaviour, which supports the previous contention that the CTBC provides a more severe (colder) curing environment.

After curing for 24 hours in the cells the cylinders were removed and were sawn in half crosswise. Both cylinders had an outer annulus, approximately 20 mm thick with an unusual structure. This structure was needle-like but not flaky nor could it easily be scraped away with a knife. The inner portion of the cylinder which had cured appeared to be unconverted, as it was greyish in colour as opposed to the characteristic brown seen in converted Ciment Fondu (the conversion of high alumina cements is discussed subsequently).

Mix consistency

All mixes were thixotropic (with and without the SPN). Mix II-3 which had the greatest proportion of accelerator set so quickly however, that it was difficult to properly fill the six cylinder molds. The remaining mixes had to be stirred briefly after 3 cylinders had been cast in order to pour the grout for the final cylinders.

In phase III the preblending of the admixtures into the cement and sand appeared to enhance the performance of the admixtures. Initially the mix was less viscous than the mixes in phase II. However, the material began to set at an earlier time and hardened more quickly. The cold grout mix was less workable than the warm grout initially, though there was no effect on the vicat needle test results.

Flow cone tests

The tests were conducted in accordance with CSA A23.2-1B. The results are contained in Table 2. Generally flow cone times varied between 24 and 35 seconds with Mixes II-5 and II-6 giving the shortest times (i.e. least viscous). The preblended mixes had considerably shorter times, except for the grout prepared at 13 °C.

Vicat needle tests

The tests were conducted in accordance with CSA CAN3-A5-M77. In phase II the time from the start of set to complete hardening was approximately 1 hour, though in some cases it was as little as 30 minutes (Table 2). This is noteworthy when compared to the results from phase III.

In phase III generally the time to complete set was less than in phase II (100 versus 120 minutes), but the time interval from the start of hardening to complete set was considerably less, dropping from 60 minutes in phase II to 15 minutes for the preblended mixes in phase III. In other words the preblended grout started to harden later but the rate of hardening was much faster.

Thermal performance

A summary of the maximum and minimum temperatures for the samples is contained in Table 2. After Mix II-3 was completed it was realized that the use of cardboard cylinder molds was inappropriate. These molds provided an insulating layer around the grout and thus the measured grout temperatures were too high. Subsequently metal cylinder molds were used which allowed the heat to be conducted out of the grout more efficiently and resulted in lower grout temperatures. There was not however any measurable difference in cylinder compressive strength resulting from the higher temperatures during curing with the cardboard molds.

The grout cylinders in Mix II-8 had an outer annulus approximately 12 mm thick having the same structure as observed on the cylinder surface of Mix II-1. This was observed on cylinders from both the 600 mm cell and the CTBC. This phenomena was not observed in Mix II-4 which had the same admixture quantities. Consequently this mix was re-tested and the results of Mix II-8 were confirmed. In no other cases was there any structural change observed at the outer edges of the grout cylinders when an accelerator was used in a 20 °C mix. The grout mixed at 13 °C and cured at -10 °C (III10) had an outer 2 to 3 mm annulus with the needle-like structure. The thickness of this layer approached 20 mm at the bottom of the cylinder. Although the outer RTD did not indicate temperatures below 0 °C it is believed that the temperature of the outer edge of the cylinder was at 0 °C for a short period of time.

In phase II there were no cases in which the measured soil temperature rose above 0 °C at 25 mm from the edge of the grout cylinder. Similar maximum and minimum temperatures, and respective times, were observed in the 20 °C grout cured at -10 °C for the preblended (Phase III) and the unblended (Phase II) grout mixes.

For the warmer soil conditions (-5 °C) in Mix III-11 the temperatures in the grout were only slightly higher than in Mix III-9 (-10 °C). The soil temperature rose above 0 °C at a distance of 25 mm from the grout cylinder but not at 50 mm. With soil temperatures above 0 °C in Mix III-12, the grout and soil temperatures were considerably higher than in previous mixes, as no energy was required to overcome the latent heat in the ice.

Compressive strengths

The cylinder compressive strengths up to 7 days were 33 ± 3 MPa for Phases I and II tests except Mix II-1 (Table 2). Mix II-3 showed lower values than the rest but this may have been due to difficulty in casting the cylinders as the grout set so quickly.

The compressive strengths of the cubes varied considerably. Six hour strengths varied between 27 and 49 MPa and 24 hour strengths between 36 and 59 MPa. Generally the lower the accelerator content, the higher the strength.

In phase III the strengths of the mixes in which the grout was placed at a temperature of 20 °C are similar to those in phase II. The compressive strengths are lower for the mix with the grout temperature of 13 °C, a result of the weaker grout structure at the outer edge of the cylinder.

High alumina cement (HAC) undergoes a change in the crystal structure of the hydrate with time when maintained at mild temperatures (20 °C)(Neville,1975). This process is called conversion, and results in a decrease in strength with time for concretes made with HAC. There was some concern regarding whether or not the grout strength would diminish if the Ciment Fondu underwent conversion at some later time. To the author's knowledge, no research has been conducted regarding the time for conversion of Ciment Fondu grouts maintained in a freezing environment. One cylinder from this testing program (Mix III-11) was immersed in a 60 °C water bath for 14 days after it had been cured for 28 days at -10 °C. Subsequent analysis (Lafarge (1989)) indicated that the grout was 68 % converted prior to this immersion and 93 % converted afterwards. The compressive strength of the cylinder was 40.0 MPa, which was within 10 % of the mean of the compressive strengths of the other cylinders of that test batch.

Discussion

Effect of test cell

The CTBC provided a more severe thermal regime than the 600 mm mold by maintaining a steeper temperature gradient in the soil adjacent to the grout cylinder. It is believed that this cell more closely models field conditions.

Effects of the admixtures

The water reducing admixture (SPN) which was used provided a more fluid mix and enhanced workability. It also retarded the set time. The lithium carbonate accelerator reduced the time interval for the grout to become exothermic preventing the temperature of the grout at the outer edge of the cylinder from dropping below 0 °C, thus preventing the water from freezing before the grout set.

The optimum mix design for the given conditions involved testing with only one type of each of the two admixtures. No attempts were made to introduce other SPN or accelerating admixtures.

The reduced set time of the preblended mix is believed to be a result of the SPN dispersing the cement more thoroughly thus allowing the accelerator to interact more efficiently, in addition to a longer period of time in which both the accelerator and cement were in contact during mixing. The reason that the cold grout mix (Mix III-10) was less workable initially may be due to the fact that the lithium carbonate accelerator dissolves better in cold water than in warm water allowing it to initiate setting more quickly.

Effects of cylinder molds

The use of cardboard cylinder molds was found to be inappropriate for thermal testing of the grout as the cardboard acted as an insulative layer around the grout. This resulted in grout temperatures being excessively high and not representative of what would occur in-situ. The use of metal cylinder molds better simulates field thermal conditions as heat is more efficiently conducted into the soil. The compressive strength of the grout was not affected by the type of mold used.

Application to field operations

The grout mix which was designed and tested was intended to cure without freezing in soil at temperatures as cold as -10 °C, which was expected to be the most severe conditions encountered in the SRR pile foundation program. The fluidity of the mix is intended to simplify grout placement in the field. With a preblended grout field preparation of the mix requires only that the proper amount of water be added and that the mixing time be adequate. The temperature of the grout prior to placement is important and will require that materials must be heated during severe weather conditions.

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