The use of admixtures are encouraging. Air entraining admixtures give accelerating effects to the strength of the CLSM, and higher compressive strength at 28 days is expected. This effect comes from the higher bleeding water expelled from the CLSM in plastic state because of the migration of the bubbles to the surface. Decreasing water content pulls the particles closer together, therefore a better compact structure of CLSM is obtained.

FREEZING AND THAWING DURABILITY

Characteristic of Ground Temperature

The properties of CLSM are comparable to those of granular soil. In soil there are two basic patterns of ground temperature change. The daily patterns - which follow the diurnal heat exchange, and the seasonal patterns - which follow the annual heat exchange at the earth-atmosphere interface. The maximum depth of daily change in ground temperature for most Indiana areas is approximately 50 cm (20 inches). In coarse textured sand and sandy loams, the diurnal change in soil temperature often occurs at depths greater than 50 cm (20 inches).

At depths below the diurnal heat exchange penetration, only seasonal and annual patterns of soil temperature changes exist. This implies that for some CLSM applications in Mid-West regions (mostly back-filling), the top 50 cm (20 inch) portion is the most important part that should resist freeze and thaw deterioration. In Indiana, typical sandy soil with bare cover has an extreme minimum temperature during the winter of -6°C (21.2 °F) at a layer of 20.3 cm(8 inch) depth and -10°C (14.0 °F) for 10.2 cm (4 inch) depth [2].

The Freezing Process

Presumably the freezing process in CLSM is closely related to that of concrete and granular soil. There are three conditions required for freezing damage to occur: (a) there must be a cold surface to propagate freezing; (b) there must be a source of water to feed the ice growth; and (c) the physical composition of the material that promotes the migration of the moisture to the freezing zone [3]. When the air temperature drops below freezing, the moisture in the upper layer of the CLSM will freeze in place because of those three conditions. In some CLSM mixes because of high water content, the layer exists because of segregation of particles.

The upper layer of the segregated CLSM particles will consist of finer materials, which in turn give the CLSM smaller pores and fine texture. If the conditions described above exist, the free moisture from below will migrate along the thermal gradient toward the colder surface up to a space below the upper layer. When it reaches the frost line, it will freeze and expand right at the line where the physical composition is different. This condition causes deterioration at the surface of the CLSM in the form of scaling.

In most highway applications, CLSM is placed below the pavement. Although the pavement is thick enough and acts like a "blanket" for the CLSM, the thermal conductivity of concrete is higher than that of sandy soil. Especially when the concrete is saturated with water, the freeze line on the top layer of the CLSM is unavoidable.

Materials for the tested CLSM samples

ASTM C-150 Type I cement was used for all of the mixes except for the regulated set cement CLSM. Table 1 shows the analyses of the Type I cement provided by the cement manufacturer. For the regulated set CLSM, a regulated set Portland cement was used, provided by the cement manufacturer. Table 2 shows the analyses of the regulated set Portland cement.

Fly ash from Schahfer Station was obtained directly from a precipitation hopper as a result of burning Eastern bituminous coal. The sample for this research was available from previous projects. This ASTM C-618 class F fly ash from Schahfer has a unique color, light gray, very close to that of silica fume. Table 3 shows the chemical analyses of Schahfer class F fly ash. It has a low content of calcium oxide (CaO) but a high content of silicon oxide (SiO_2). The SO_3 content is also high; about 80% is immediately soluble while the alkali contents are low and insoluble. However, the most interesting analyses are shown in Table 4. The mean particle size is extremely low; 74% of the particles are smaller than 5 microns. That is why the measurement of Blaine fineness is very high -- 710 m^2/kg. A Pozzolanic Index Test with cement (ASTM C-311) of this type of fly ash gives a very high value -- 144% with a specific type of cement. This Pozzolanic Index Test gives an indication of the relative reactivity of that fly ash with cement.

Stout Station fly ash is available commercially and marketed as ASTM C-618 class F fly ash. The color of this fly ash is very typical of class F fly ash, which is olive gray. The fly ash was the result of burning a mixture of several Illinois Basin or Eastern coals. Table 5 shows the chemical analyses of Stout fly ash. This is a very common fly ash with the total SiO_2 + Al_2O_3 + Fe_2O_3 content more than 90%. The SO_3 content is small and only 42% of it is immediately soluble. Table 6 shows the physical analyses of Stout fly ash. The particle size mean diameter was 17 microns and the distribution of particle sizes was almost normally distributed, but the coarser particle portions from 75 to 115 microns seems to be high. In addition, the total percentage of particles larger than 45 microns is a little bit high, 24%. The specific surface area for Stout fly ash is low as shown in Table 6, 220 m^2/kg.

This low value is a result of a higher portion of coarser particles. The result from the Pozzolanic Index Test with particular cement gives 71% that of the control mortar. However, 30% weight straight replacement of cement to the same volume of fly ash without adjusting the water content gives the Pozzolanic Index of 105%.

Table 7 provides the analyses of the Tippecanoe sand. Moisture content of that air dry sand was 0.15% and the specific gravity using a picnometer indicated a value of 2.67. The fineness modulus of this sand was 2.87.

Freeze and Thaw Test Arrangement

Specimens were concrete prisms 7.62 x 7.62 x 38.1 cm (3 x 3 x 15 inches) and cylinders 7.62 x 15.24 cm (3 x 6 inches). Both specimens were contained in Styrofoam boxes to simulate the actual condition in the field. The temperature of the containers was carefully monitored with thermistors so that the cooling process took about six hours from 23 °C (74 °F) to 0 °C (32 °F). The freezing in air process took about twelve hours and the minimum temperature for the specimens was -5 °C (23 °F). The temperature in the inside of the specimens was carefully monitored by embedded thermistors.

The thawing process took five hours, and was accomplished by immersing the specimen into 2 °C (35.6 °F) water to eliminate the temperature shock. The temperature inside the specimen was monitored so that at the end of the thawing process, the specimens were at about room temperature 23 °C (73.4 °F).

To measure the effect of scaling, the specimens were brushed on each side three times with a soft baby brush. Then the specimens were placed into the Styrofoam boxes to let the water drip off them. By this time, the specimens were ready to be weighed and the pulse velocity measured. The pulse velocity measurements were made by using cellulose as a couplant material and a "bench" with a constant pressure to the transmitter and receiver. This eliminates damage on surfaces of measurement and error in weight loss and pulse velocity measurements.

The following properties were examined:

(a) Concrete strength: To investigate the influence of strength in a slow rate of freezing and thawing environment. The strength is based on water to cement ratio. Compressive strength at 28 days for W/C about 8 was 0.552 MPa (80 psi) as Mix 1 and for W/C about 6 was 1.090 MPa (158 psi) as Mix 2.

(b) Air contents: To investigate the effectiveness of air entrainment to prevent surface deterioration during freeze and thaw. The total air content for the freshly mixed CLSM was 8% by volume. Since the air content prior to the addition of air bubbles was 2.5%, 5.5% of the air voids were generated. The air bubbles were generated by foam generator and mixed with the CLSM during mixing.

The specimens were demolded three days after placement and were placed in the moist room for curing until the day they were tested. The CLSM cylinder specimens were covered with a rubber membrane to prevent disintegration because the residual strength of the specimens were going to be measured at a certain freeze-thaw cycle. The cylinders were capped with sulfur for the compressive strength test.

Results

Tables 8 thru 13 and Figures 1 thru 5 show the result of the three mixes for the CLSM: Mix 1 in Table 8, with a compressive strength of 0.55 MPa (80 psi) and W/C at about 8; Mix 1A in Table 10, which was the same as Mix 1 but was air entrained; and Mix 2 in Table 12, with a compressive strength of 1.09 MPa (158 psi) and W/C at about 6.

1. Table 8 shows the design of Mix 1 with corresponding freeze-thaw test in Table 9. It can be seen that during the first cycle the weight loss was small but the pulse velocity suddenly dropped from 1.97 to 1.39 km/second (1.22 to 0.86 mil/second). Figure 1 shows that the weight loss was increasing but the decrease in pulse velocity was small beginning with the fourth cycle. It shows that the scaling on the surface of the CLSM was continuing while the inside deterioration was diminishing. The inside deterioration was diminishing because of open channels already existing so that the freezing water could escape. It seems that once the surface was opened additional scaling was promoted.

2. Samples from Mix 1 after freezing and thawing showed the deterioration mostly on the top surfaces and edges of the prisms and the cylinders. This came from the fact that the top layer of Mix 1 had more fine materials, mostly fly ash, because of segregation. In addition, the top surface has less air voids compared to the lower part which has coarser materials making the upper layer more compact.

3. Table 11 shows the results of Mix 1A in Table 10. It has 8% total air content in freshly mixed CLSM. Figure 2 shows that although the weight loss was high enough at 9 cycles, the pulse velocity was almost constant in all cycles. This indicates no significant deterioration on the inside. It may come from a bit stronger compressive strength at the test date, 0.65 MPa (94 psi) compared to Mix 1 with 0.55 MPa (80 psi), and distributed voids in the mix.

4. As expected, scaling on Mix 1A occurred mostly at the bottom edges of the prisms and bottom parts of the cylinders because they are the weakest section of the samples. The surface layer of the

prisms and the cylinders were almost perfect; the same as the initial condition even after 9 cycles. This is because the voids on the top surface appeared thoroughly distributed. Despite the existence of segregation and plane of weakness, there was no scaling on the top surface of the prisms.

5. Table 13 shows the result of Mix 2 in Table 12. The compressive strength was 1.09 MPa (158 psi). The weight loss for the first cycle was 0.43%, which was almost the same as Mix 1 and Mix 1A. The pulse velocity measurements for Mix 2, during the test, were almost constant into 12 cycles. Figures 3 shows the results. The deterioration came mostly in the form of scaling on the top surface. Since the top surface was void deficiency, the scaling was unavoidable despite the fact that Mix 2 had a higher compressive strength compared to Mix 1A.

6. During the test, compressive strengths at specific freeze-thaw cycles were conducted. The initial compressive strength for Mix 2 was 1.09 MPa (158 psi). After 5 cycles (which was 33 days old) the compressive strength was 1.21 MPa (175 psi). The compressive strength after 12 cycles was 1.17 MPa (169 psi). This indicates that there was no significant effect from freeze-thaw to the Mix 2 except scaling. Tests for Mix 1 and Mix 1A could not be conducted because the cylinder samples were already damaged.

Conclusions

The permeable voids (ASTM C-642) for the tested CLSM in this study is high (27.5%). Basically, these voids can provide escape paths for the expanding freezing water. However, for Mix 1 samples, it is not the case. Additional voids should be added to distribute them inside the samples to provide more escape paths for the freezing water and decrease the hydraulic pressure. Both increasing the strength and providing enough voids overcame the freeze and thaw deterioration of the tested CLSM samples. A combination of air voids and strength, for some typical designs and a freezing rate condition, can create a CLSM product that resists freeze and thaw deterioration. The only single problem is consistency and accuracy of the mix. If later excavation is a major concern, the compressive strength in later days can not exceed 1.04 MPa (150 psi). CLSM applications in the freeze and thaw environment should be tested prior to using the mix. Different types and proportion of fly ash will not only affect the void content, but also the strength and the gradation of the particles.

Except for Mix 1A, scaling mechanism in CLSM is the same as that of in concrete. The surface of the scaled CLSM has moisture channels on it because of segregation of coarser particles that leave fine particles on the surface. This mechanism creates a dense surface layer on the top with a less dense layer immediately beneath it. Mix 1A had higher bleeding water compared to Mix 1 which indicates that Mix 1A was well compacted. Since Mix 1A had air entrainment, the effect of compacted surface to the freeze and thaw deterioration was eliminated. This higher bleeding might come from the fact that larger voids from the foam went to the surface and created paths to the bleeding water and then compacted the mix.

If scaling occurred on the top surface, settlement after freeze-thaw cycles in CLSM will to be minor. When saturated CLSM is frozen with no change in moisture content, the CLSM expands in volume by an amount equal to the phase change for the water. When this CLSM is thawed, it returns to its original volume and consolidates further. This additional consolidation will be insignificant because the CLSM was in a relatively dense state before freezing, especially the upper layer. However, to avoid the formation of this weak surface, the CLSM can be designed to be more flowable by proper proportioning of the materials rather then adding more water.

The sudden decrease in pulse velocity for Mix 1 may come from the creation of additional paths for escaping freezing water. When the sample was resaturated, water occupied these paths. Freezing after this condition formed ice lenses in the additional paths. Therefore, the inside deterioration should come from the formation of ice lenses. To create a design mix durable enough to withstand a slow rate of the freeze and thaw condition, the additional paths at the first cycle of freezing should not be formed. There are three ways to control the additional path from being formed. They are: (a) add more bubbles, (b) increase the strength to about 1.04 MPa (150 psi), and (c) a combination of both.

EARLY SET AND STRENGTH DEVELOPMENT OF CLSM

Behavior of Slurry

CLSM that contains fine aggregates (sand) as a filler will behave as Coulomb materials. It has been tested rheologically that flow stress is very much dependent on the high friction between the solid grains. The ability of wet sand to support weight is highly sensitive to the coefficient of friction between particles. In CLSM the material contains fly ash. During mixing and placing, the fly ash will improve flowability and provide self-consolidation. However, in the presence of fly ash the friction between grains is reduced and the ability of the material to support weight during the first hours up to the plastic state decreases [4].

The early strength development of CLSM is very dependent upon the cohesion between the particles. The cohesion can come from two different sources. First, cohesion occurs from early hydration of cement particles. Notably, gypsum and C_3A is the first to react and hydration of C_3S. Second, inter particle forces aids cohesion. Solid grains in the liquid medium are attracted toward each other by surface forces and come into contact to form an elastic connected network through the fluid. The particles flocculate into a gel structure. All the grains are in static contact with other grains. At the point when cohesion is already present, the CLSM enters the plastic state and the contribution of inter particle friction to the strength of CLSM increases.

Stiffening and Hardening Stages

There are two different stages defined in early strength development. (a) Stiffening-stage, which indicates that the CLSM is beginning to develop cohesion, and (b) Hardening stage, which indicates that the CLSM has hardened to the point at which it can sustain some load. The stiffening-stage measures the early cohesion of the CLSM. This happens after the cement is in contact with water when the role of inter particle forces is small because of the lubrication effect from fly ash and water. The hardening-stage is determined when the bleeding after penetration resistance is subsided, and the slope of penetration resistance versus time suddenly becomes larger after a certain penetration number already exceeded. The hardening-stage is based on: (i) CLSM mixes had a higher penetration resistance because of a disappearance of the lubrication effect (no more excess water surrounded the particles) and the appearance of inter particle forces; (ii) the cement particle is already hydrated entering acceleration period indicated by a suddenly higher strength gain.

Strength Development Test Arrangements

The containers for the specimens in stiffening-stage were six small Styrofoam cups (measured 1 cup) where the diameters of the upper portions are larger than the lower portions. The container for the penetration resistance test (ASTM C-403) was a 15.24 x 27.94 x 15.24 cm (6 x 11 x 6 inch) steel container that can accommodate ten penetrations with a 6.45 cm^2 (1 $inch^2$) penetration rod and a 3.81 cm (1.5 inch) perimeter to perimeter clear distance between two penetrations.

After completing the mixing, the CLSM was placed in the six cups and the penetration container. Depending on the mix design, at about 45 minutes the first cup was opened by peeling off the upper half portion of the cup. If the CLSM can support itself in the cup without a significant slump and there are few cracks on the top surface, then the stiffening process has already begun.

The penetration resistance was done by penetrating the surface of the CLSM with a penetration rod at a speed of 0.254 cm (0.1 inch) per second to a depth of 2.54 cm (1 inch). The clear distance between two tests was at least 3.81 cm (1.5 inches) to avoid interferences from other penetration holes. Except for regulated set cement, all specimens consisted of ASTM C-150 Type I cement and ASTM C-618 class F fly ash.

The following properties were examined:

(a) W/C ratio: To investigate the effect of lower water to cement ratio to the early strength development. Mix 1 had W/C of about 8 and Mix 2 had W/C at about 6. Mix 2 was designed by adding more cement to the design of Mix 1 to preserve the same W/(C+P+FA), W/(C+P), and flowability.

(b) Fly ash: To investigate the effects of different kinds of fly ashes. Although it was class F fly ash, the smaller particles may be available to react at early periods. Fly ash from Stout station has 17 µm mean particle size with Pozzolanic index of 71% and fly ash from Shahfer station has 3 µm with Pozzolanic index of 144% [5].

(c) Calcium chloride as admixture: To investigate the effect of adding calcium chloride to the hydration process of the CLSM and its influence to stiffening and hardening stage.

(d) Regulated set cement: To investigate the effect of using regulated set cement to shorten the time to reach the stiffening-stage and hardening-stage.

All of the 7.62 x 15.24 cm (3 x 6 inch) cylinder specimens for later days strength (3, 7, and 28 days) were demolded three days after placement except Mix 2 which can gain higher strength. Mix 2 was demolded one day after the placement. The specimens were placed in the fog room until the day of the test. The cylinder samples were capped with sulfur for compressive strength test. The compressive strength test was conformed with ASTM C-39 procedures and the rate of loading was 0.24 MPa/second (35 psi/second).

Results

Table 14, Figure 6 and Figure 7 show the results of the penetration test and compressive strength at 3, 7, and 28 days. The compressive strength was conducted to monitor the later day strength for all the different mixtures.

The CLSM in a Styrofoam cup sample indicates that the stiffening-stage has a penetration resistance value of 10 to 20 psi. Most of the stiffening-stage of the tested CLSM mixes occurred at about 1 to 2 hours except for the Mix 2 Regulated-Set Shahfer which has shorter time. At this time the bleeding had already ceased to zero. However, bleeding after penetration was present up until the beginning of the hardening stage.

Most of the hardening-stage occurred varies with the mix design. Figure 6 shows that the hardening-stage, indicated by a suddenly higher slope, for regulated set cement begins as short as the first hour, while type I cement begins at the third hour.

Based on time required, the two stages in CLSM are exactly identical to the stages in hydration of cement particles. Stiffening-stage occurs after second period (end of dormant period) and while entering the acceleration period. Hardening-stage begins at the end of acceleration period through the slow down period (fourth period).

The following results are found:

1. W/C ratio, more appropriately the cement content, is an important parameter in early strength of CLSM. Mix 1 Shahfer had lower penetration compared to Mix 2 Shahfer due to the slurry behavior. A higher lubrication effect comes from the Shahfer fly ash. Table 15 shows calorimetric tests of hydration of Type I cement. Mix 1 had W/C of 8 and Mix 2 had W/C of 6. On that table, the W/C of 6 entered the acceleration period (initiation of set) in 60 minutes, the same as the W/C of 8. Figure 6 indicates that the time to the stiffening-stage for Mix 2 was earlier and the penetration number was higher than Mix 1 because Mix 2 contains more cement which means there is more paste for cohesion. Mix 2 Shahfer was in the stiffening-stage in less than 1 hour while Mix 1 Shahfer was in that stage after 1.5 hours. However, Figure 6 shows that the rate of strength gain for both mixes is almost identical (if fitted curves are provided). The difference in the penetration resistance is the result of a higher cement paste in Mix 2.

   Table 15 indicates that the W/C of 6 was at the end of acceleration period (peak) in 560 minutes while the W/C of 8 reached that point in 620 minutes, it was one hour different. It appears that the W/C does not influence the initiation of set significantly. The W/C ratio influences the time to the peak due to the hydration of C_3S is affected by the alkali concentration. It can be expected that the W/C ratio will affect the hardening stage.

2. Different properties of fly ash give different strength developments. Generally speaking, class F fly ash will affect the later days strength. However, for high Pozzolanic index and smaller diameter fly ash, the smaller particles are immediately ready to react just a few hours after the cement was in contact with water. Table 14 shows that in the first 1.5 hours the penetration resistance of Mix 1 Shahfer (Pozzolanic index 144% and mean diameter of 3 μm) was low compared to Mix 1 Stout (Pozzolanic index 71% and mean diameter of 17 μm). This is because Shahfer fly ash gave more lubricating effect to the CLSM while in its plastic state compare to the Stout fly ash due

to the smaller particles. Figure 6 shows that both the penetration number and slope of the curves between the two mixes are not identical. The slope indicate the rate of strength gain.

3. The addition of calcium chloride to the CLSM has an advantage in shortening the hardening-stage. Figure 6 shows that Mix 1 Stout 2% $CaCl_2$ had a higher penetration than that of Mix 1 Stout beginning at the third hour. This is because the calcium chloride does not react significantly with cement for a period of 2 to 6 hours and accelerates the hydration of C_3S in the accelerated period [6]. However, this admixture can give a higher compressive strength compared to a control mix.

4. The use of high early set cement is encouraging. However, there must be caution in using that product. Some of the products, when using the W/C ratio of 8, in the CLSM mix does not set at all. Low of a W/C ratio (about 6) have a funicular effect at the top layer of the CLSM. The top structure of the CLSM was weaker than that of the bottom one. This comes from the fact that the particles had not settled down yet when the regulated set cement began to react and harden. During testing a sample, it appeared that the bleeding water subsided quickly. However, the network of particles were filled with water. From two companion penetration samples in two containers, one sample was 11.03 MPa (1600 psi) in 9 hours but the other sample (without any previous penetration) was only 0.19 MPa (27 psi) and the excessive bleeding appeared after penetration.

5. Figure 7 shows that the rate of strength development is independent on the amount of cement and dependent to the reactivity of the fly ash. Because Mix 1 Shahfer and Mix 2 Shahfer have almost the same slope, this indicates that the strength development is independent of amount of cement. Mix 1 Stout has a lower slope compare to Mix 1 Shahfer because of the effect of the reactivity of the fly ash. Mix 1 Regulated Set Stout has a lower slope despite shorter stiffening and hardening stages compared to Mix 1 Stout (Type I cement). Regulated Set Cement may be not suitable for high W/C ratio CLSM mixtures.

6. Determining the hardening stage based on the penetration number alone is inappropriate. A higher cement content in the mix has a result of a higher penetration number while the rate of strength gain is the same as that of lower cement content. In addition, a mix can develop strength after passing a determined penetration number but it does so very slowly or can even stop gaining strength. For example: false setting phenomenon in concrete or setting of some high early strength cements in a very high water to cement ratio.