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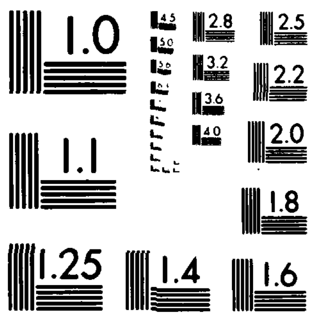

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RESINS AND NON-PORTLAND CEMENTS FOR CONSTRUCTION IN THE COLD

Robert Johnson

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A laboratory investigation was conducted to assess the potential of some resins and non-portland cements for structural concrete at low temperatures. The resins investigated were urethane (non-hydrophilic), epoxy and polyester, as well as a polysulfide polymer. Two non-portland (modified) cements were also tested. The curability of the resins, when mixed with fine aggregate, showed that they had potential for low temperature use in the following decreasing order: urethane, polyester, and epoxy. Of the non-portland cement

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20. Abstract (cont'd)

materials, mixed as individual neat slurries, one showed potential for low temperature use at -10°C (using 3.9°C water).

PREFACE

This report was prepared by Robert Johnson, formerly Research Civil Engineer, Geotechnical Research Branch, Experimental Engineering Division, U.S. Army Cold Regions Research and Engineering Laboratory. The study was funded under DA Project 4A762730AT42, Design, Construction and Operations Technology for Cold Regions, Task A2, Soils and Foundations Technology for Cold Regions, Work Unit 001, Design Criteria for Foundations.

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INTRODUCTION

Increasing development in Alaska has focused attention on the use of conventional construction engineering methods in permafrost areas and other northern regions. Because of the short summers in these regions most construction engineering must be carried out under freezing conditions or at low temperatures. Inexperience with such climatic conditions results in concrete curing problems for construction engineers, concrete design engineers, and manufacturers of concrete construction materials. This leads to concern in using concrete for general construction purposes, including structural foundations, in permafrost or cold environments.

When portland cement concrete is to be placed in environments where the ambient temperatures are below freezing and where the ground is permafrost or contains a high ice content, the concrete mix (slurry) must be allowed to set soon enough and strong enough to resist the forces of expanding and freezing water. For foundations below grade, the hydration heat evolved by the curing concrete must not melt the permafrost.

These conditions have led engineers to the familiar solutions of protecting fresh concrete with construction enclosures, heating the concrete materials, or heating the concrete after placement. These add cost for extra equipment, materials, heat, and labor. Considering all aspects, current cold-weather construction is costly not only for engineers, contractors, and their clients, but also for the nation, at a time when fuel supplies are dwindling.

Previous work at CRREL on this problem included a survey by Sayward (1974a) on the potential use and curability of synthetic resins (plastics) at low temperatures, as for mortar, binder, etc., for construction or repairs. His report indicated that the thermosets were potentially most adaptable, particularly epoxy, polyurethane, and polyester. This survey led to limited further investigation of thermoset resins for construction and repair in the cold (Kovacs et al. 1974, Sayward 1974b). Smith and Kahl (1975) tested a liquid activator solution and binder/aggregate repair mix for concrete, and concluded that "Darex 240 Concrete has properties which favor its use over regular portland çement concrete at colder temperatures." Test results were given down to -11.1°C (12°F).

In a previous investigation (Johnson 1979) of grouting soils using low viscosity synthetic resins, materials were used at 0.5°C that produced unconfined compression strengths as high as 11,200 kPa (1624 psi) with good workability at that temperature as well as at -6.6°C (see Appendix A). This indicated potential usefulness in cold environments for foundations, patch work, and placement of construction materials, as cementitious binders, mortar, etc.

The purpose of this investigation was to extend the above work to gain further information on non-portland cements, including quick setting cement compounds as well as synthetic resins that have potential as construction materials at low temperatures. Tests included "neat" materials (binder alone) and binder-aggregate mixes. Five resin solutions and two cement type materials (Table 1) were examined as to binder application in the cold. The fine aggregate used was Ottawa graded standard sand.

Table 1. Chemical solution or cement material tested

Resin or solution	Type
EP Systems	Urethane resin
Epotuf 37-130 and 37-052	Epoxy resin
Ancamine AD, LT and MCA	Curing agents (used with epoxy resin)
Gold Label	Polyester resin
J-27	Polyester resin
F-181	Hydraulic cement compound
Sonopatch	Hydraulic cement compound
Thiokol	Polysulfide polymer

MATERIAL PREPARATION AND FABRICATION OF SAMPLES

Resin binders

The resin binder materials were prepared according to formulas suggested by the manufacturers. The solutions or components were first mixed by hand using a clean wooden rod and a 1000-mL batch vessel. All materials were mixed well (2 to 10 minutes) before being molded.

Resin binder and sand

The resin-bound materials were prepared by mixing 40% resin solution (mixed as above) plus catalyst with 60% sand by volume.

Sample fabrication

The mixes (resin alone, resin-binder, or non-portland with binder) were placed in 5.6-cm-diameter by 11.2-cm-long waxed cardboard cylinder molds to set or cure for 24 hours, unless otherwise stated. After the curing period, each sample was trimmed using a lathe or a miter box and hand file.

Ambient temperatures

All the equipment and materials were maintained, used, and stored in a coldroom where the temperature could be set for each test (3.9°C and below). All materials were tempered at the test temperature for a minimum of 24 hours before being mixed.

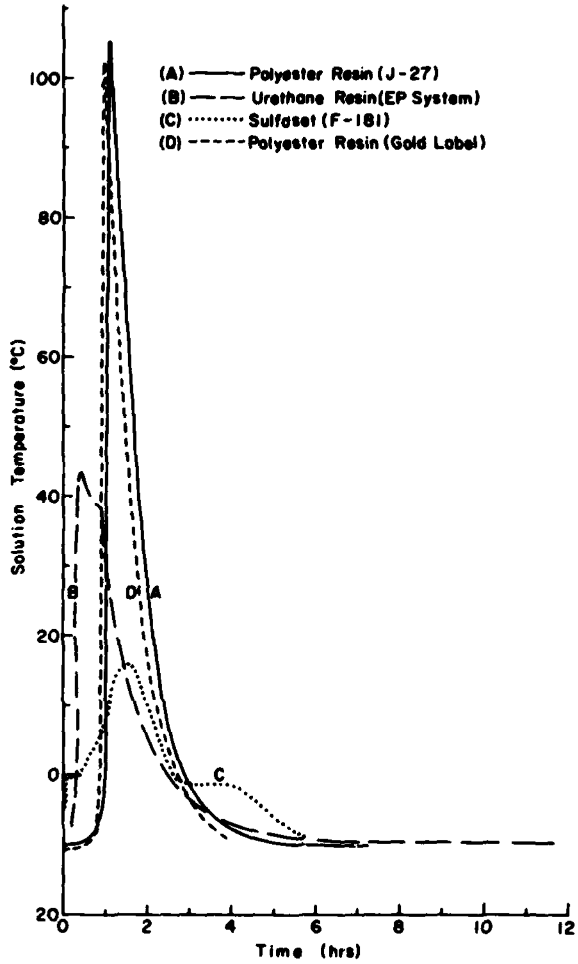

Figure 1. Set test results of neat mixes at -10°C.

TEST METHODS

Set tests

Set tests had been performed on the urethane and epoxy resins at 3.9°C and below in a previous study (Johnson 1979; see Appendix A, Fig. A1). The current tests were performed using 5.6-cm-diameter by 11.2-cm-long cylindrical molds with a thermocouple centered in each of the mixed solutions. The thermocouples were wired to a data retrieval system that recorded at 1-minute to 1-hour intervals. The results of the set tests appear in Figure 1.

Unconfined compression strength tests

Unconfined compression strength tests were conducted after a 24-hour curing period, unless otherwise stated. All samples investigated were strained at 1.3 mm/min. The test results are presented in Table 2 for the non-portland cements and in Table 3 for the resin-based materials.

Table 2. Properties and test results of non-portland type cements.

	Cement type slurries*	
	Sonopatch (Repair compound)	Sulfaset F-181 (Anchoring compound)
Manufacturer-suggested curing temperature	Not given	Not given
Set information from manufacturer	Set not controlled	Set not controlled
Air temperature (°C)	-10	-10
Cement temperature (°C)	-10	-10
Water temperature (°C)	3.9	3.9
Reaction		
Time to max temp (minutes)	†	90
Max temperature (°C)	†	16
Unconfined compressive strength in 24 hr (psi)		4705
Cleaning solution	Water before set	Water before set
Approximate cost (1977)	--	$0.38/lb
Manufacturer	Sonneborn, Contech, Inc., Minneapolis, Minn. 55435	Randustrial Corp., Cleveland, Ohio 44210

*Neat slurry mixes

†Did not set at -10°C and mix water froze. Sample did not produce temp-time curve.

Table 3. Properties and test results of resin materials.

	Urethane resin	Type solution Polyester resin		Epoxy resin
	EP series	J-27	Gold Label	Epotuf
Manufacturer-suggested minimum curing temp (°C)	Experimental product	-40	-17.7	-1.1
Set information from manufacturer (°C)	Conrolled set	7 hr at -23.3 24 hr at -40	6 hr	Controlled set
Air temp (°C)	-10	-10	-10	-10
Resin or solution component temp (°C)	-10	-10	-10	-10
Reaction				
Time to max temp (min.)	25	65	60	174
Max temp (°C)	40	105	102	-5
Unconfined compressive strength in 24 hr (psi)	4715	2045	See remarks	See remarks
Cleaning solution	Not given	Rad-Clean (chlorinated)	Naphtha (high flash-point solvent)	
Approximate cost (1978)	See App. B	$30.70/gal	$10.84/gal.	
Manufacturer	Ashland Chemical Co., Columbus, Ohio 43216	Radiation Technology Inc., Rockaway, N.J. 07866	Preco Industries, Plainview, N.Y. 11803	Reichhold Chemicals Inc., White Plains, N.Y. 10702
Remarks	Remains low in viscosity as temperature decreases, good workability.	Shrinkage cracks form upon cooling. One of three samples split during curing.	Shrinkage cracks split all four samples during curing, therefore strength tests could not be performed.	Did not set at -10°C Poor workability, viscosity increases significantly as temperature decreases.

TEST RESULTS

Urethane resin system

An EP urethane resin system of low viscosity was used. This system was mixed 50 parts EP 65-12 to 50 parts EP 65-17 and used a catalyst (EP 65-18) added in amounts of up to 4%. Tests conducted in previous low temperature work used the above urethane system as well as a less concentrated form of the same system (65-92 and 65-93) with the same catalyst (65-18) and catalyst no. A-11. The low temperature set test results of both systems are shown in Figure 1 and Appendix A.

At 3.9°C ambient temperature, set test temperature graphs (Appendix A, Fig. A-1b, curves B-1 and B-2) show that with a "fast" catalyst (A-11) the less concentrated urethane set test produced a maximum temperature of 53.8°C in 20 minutes, and that with a "slow" catalyst (EP 65-18) the sample reached a maximum temperature of 52.3°C in 18 minutes.

At 0.5°C ambient temperature, set tests were performed using the above formula with 1.5% catalyst (65-18). The set time was the same as that at the 3.9°C temperature and the viscosity appeared to be the same, indicating no significant temperature effect. This set test was conducted using 300-mL cups without thermocouples. This material changes color upon setting.

Further set tests, at temperatures below 0°C, were conducted, since the resin solution would freeze as would "aqueous" mixes. In these samples, thermocouples were used to monitor temperatures below 0°C (Fig. A1a, Appendix A).

At -10°C ambient temperature, using the more concentrated system (65-12 and 65-17) and 1.5% catalyst 65-18, a neat urethane solution was mixed. The material was easy to mix, indicating good workability. At the end of the 24-hour period, an unconfined compression strength test gave 33,400 kPa (4845 psi).

In view of the success in curing and testing at -10°C, a urethane solution was used to make a sand slurry (see Material preparation). Mixed by hand, this slurry had good workability and the 24-hour unconfined compression strength measured 18,800 kPa (2725 psi).

This urethane formula (using 1.5% catalyst) was also used to mix a sand slurry at -20°C. Workability was good and the sample cured without problems. The unconfined compression strength measured 15,230 kPa (2210 psi).

An attempt to mix a urethane resin sand slurry by adding the catalyst after the sand was mixed with the urethane solution resulted in a very stiff mix (poor workability). The samples did cure but were not tested because it was feared that prepared in this manner they were not mixed consistently, as evidenced by color variation. The addition of the catalyst before the solution was mixed with the fine aggregate resulted in better workability. The catalyst produces a heat of chemical reaction that is needed for good workability when mixing at low temperatures.

At -30°C the urethane component EP 65-17 became more viscous than at high temperatures, but it flowed easily. Workability remained good, but a little more effort was needed to mix and pour the sand mix solution into the cylindrical molds. Four percent catalyst was used and a thermocouple was placed in the center of one of these samples. It showed that during curing the temperature rose from -30°C to -11°C before the rate of heat generation began to fall, in approximately 50 minutes (see Fig. A1a, curve ES). Curve E in Figure A1a is for the same urethane and catalyst solution cured at a similar temperature but without the sand. The sand served as a heat sink and retarded the liberation of heat and the chemical reaction of hardening. This is evident in the results of 24-hour and 48-hour unconfined compression tests. The 24-hour test gave 13,815 kPa (2005 psi) and the color of these samples did not change (change in color indicates setting), as was usually the case at higher temperatures. Therefore, the remaining samples were left for 24 additional hours (total 48 hours) at room temperature (20°C) before another unconfined compression strength test was conducted. This test gave 17,810 kPa (2585 psi), indicating additional chemical reaction and strength gain. Interestingly, the samples stressed with plastic yielding (see Discussion).

Epoxy resin system

The Epotuf resin system used was Epotuf 37-130 (a modified diglycidyl ether of bisphenol A - epoxy resin family) and Epotuf 37-052 (previously 37-149, a butyl glycidyl ether - aliphatic monepoxide family). The curing agents used with these epoxy resins were: 1) Ancamine AD, a curing agent for liquid and semi-solid epoxy resins that is a blend of amines and hardening accelerators, and reportedly is capable of curing under cold, damp conditions; 2) Ancamine LT, a curing, amine-based agent designed to operate down to -5°C, effective under water as well as in air; 3) Ancamine MCA, a modified amine of high reactivity that reportedly cures at low temperatures (down to 5°C) and under water.

Epotuf was formulated as recommended by Pacific Anchor Chemical Corporation. However, the 37-052 component was a very low viscosity solution (approximately that of water) and was added to the 37-130 component (20% by weight) to lower the overall viscosity of the solution, as recommended by Reichhold Chemicals, Inc.

In tests at 3.9°C, the Epotuf components mixed readily. Within five minutes, the temperature was 6.3°C, and within one hour 24°C. The sample then began to liberate heat at a high rate and produced a maximum temperature of 172°C within the next 26 minutes. The temperature-versus-time graph is presented as curve A-1 in Figure A1a of Appendix A. An epoxy pressure injected sand sample resulted in an unconfined compression strength of 3370 kPa (490 psi) (Fig. A2, Appendix A).

At 0.5°C ease of mixing was affected. The set test showed no setting within 48 hours.

At -10°C, after being stored for a month, Epotuf resin 37-130 became viscous and a white, brittle, easily crumbled material settled out. The 37-052 remained low in viscosity (like water). The AD hardener became very viscous and poured very slowly; the MCA hardener was more fluid and easy to pour. Because of the high viscosity of 37-130 at -10°C, the 37-130 and 37-052 were taken to a 20°C room and mixed 20 parts (by weight) 37-052 to 80 parts 37-130 (the manufacturer's suggested formula to lower the viscosity of 37-130). This mix, without the hardener, was returned to -10°C and tempered. In hand mixing with its hardeners this resin was very stiff (poor workability). The sample did not cure at this temperature, which is not surprising in view of its slowness at 0.5°C.

Polyester resin systems

J-27: J-27 is a polyester resin freezer floor patch. According to the manufacturer, it cures within 8 to 48 hours and may be used to -40°F. Its hardener is methyl ethyl ketone peroxide.

At -10°C the neat liquid resin was easy to mix. The mix rose to 105°C at 65 minutes (see Table 3 and Fig. 1). One of three samples molded was lost due to shrinkage cracking. The sample tops showed bubbles, as from gases produced by the chemical reaction. An unconfined compression strength test of this neat polyester resin gave 14,115 kPa (2045 psi).

A sample prepared after test mixing this resin with the Ottawa graded standard sand did not present any problems. However, the sand mix had lower strength (5325 kPa (770 psi) after 24 hours at -10°C).

At -30°C, the liquid was easy to mix with sand, but within 24 hours the samples were sticky with solution and, perhaps, hard from the low temperature and cold sand mixture, and not properly cured. Also, the bubbles were larger on the sand mix samples. These samples were moved to a 20°C room; at 30 minutes they became very warm, indicating that chemical reaction was proceeding.

Gold Label: A modified version of Gold Label is recommended for use below -6.6°C. It consists of a liquid polyester resin and a liquid catalyst, unlike the standard product that consists of a liquid resin and a powdered catalyst. According to the manufacturer, up to 40 lb of sand can be added to 1 gal. of this system and aggregate may also be added with the sand up to approximately 25 lb.

At -10°C, the neat liquid resin was easy to mix. The set test gave 102°C in 60 minutes (see Fig. 1 and Table 3). All four samples mixed were lost, hence there were no compressive tests. In mixing this resin with sand at this temperature, workability was good. The sand mixes showed an unconfined compressive strength of 7025 kPa (1020 psi).

At -20°, samples were mixed using sand, and workability was still good. However, at 24 hours the samples were sticky with solution and not properly cured, as with J-27.

Polysulfide polymer

Thiokol is a two-component liquid polysulfide polymer. At -10°C the Thiokol component became too stiff to mix and the hardener became hard. No further testing was conducted.

Non-portland cement type materials

F-181 Bolt Anchor Sulfaset: According to the manufacturer, F-181 is a fast-setting, super strength anchoring compound, reinforced with albatite for greater strength and resistance to water, oil, and chemicals. It is mixed with water.

At -10°C, with the temperature of the water 3.9°C and the cement tempered to -10°C, mixing a neat slurry was attempted using 16% (by weight) water. This formula made the cement compound a very stiff paste at this temperature, with poor workability. Therefore, the water was increased to 24%, giving good workability. The 24-hour unconfined compression strength measured 32,445 kPa (4705 psi) (see Table 2).

Sonopatch: According to the manufacturer, Sonopatch is a fast-setting, non-shrinking concrete repair compound that resembles mortar. At -10°C this material was mixed with 16% by weight of 3.9°C water. The compound hardened with ice crystals across the top of the samples and did not liberate any heat that could be measured by thermocouples. After 24 hours, the samples were moved to a 20°C room. They became soft, indicating that they had frozen (see Table 2). This was a neat mixed slurry.

DISCUSSION

The present results provide qualitative information on viscosity, curing, and workability of the materials tested as temperatures were decreased. It was found that the urethane gave a lower heat liberation rate and lower maximum temperature when mixed with fine aggregate (sand), as the temperature was lowered. The aggregate served as a heat sink. In Figure A1a, curve E is shown the temperature-time curve of the urethane without fine aggregate. For similar conditions, the curve for this urethane with sand is, of course, much lower (curve ES). Test results showed that curing the polyester was nulled by the heat sink effect of the aggregate at -10°C.

The workability of the materials is a direct function of ambient temperature and aggregate temperature. At lower temperatures workability is poorer, due to viscosity increase. In the case of the epoxy, at least the retarding of chemical reaction and heat liberation may have also contributed to poor workability. This was evident with the decrease in workability of the epoxy resin solution due to a significant increase in viscosity, as the temperature was lowered to -10°C. When set tested at 3.9°C, the epoxy resin without fine aggregate rose to a maximum temperature of 172°C (see Fig. A1b, Appendix A). The viscosity and workability of the polyester resin solutions (both J-27 and Gold Label) were not significantly sensitive to the temperature change, and they did cure properly in 24 hours at -10°C as a sand slurry. At -20°C viscosity and workability were not noticeably changed; however, the sand mix did not cure properly. Without fine aggregate at this temperature, the Gold Label and J-27 solutions rose to maximum temperatures of 102°C and 105°C within 60 and 65 minutes, respectively. These were more than twice the maximum liberation heats of others at this temperature (Fig. 1). The F-181 Anchor Bolt Sulfaset (-10°C) displayed good workability only after additional water (3.9°C) was added. This is a non-portland cement type quick set anchoring compound that absorbs water quickly.

The curability of the materials at low temperature was affected by the addition of aggregate, which served as a heat sink. Materials that exhibited maximum temperature rise during the set test (without aggregate) did not prove to be the better materials for curability at low temperature with the addition of cold aggregate. It was found that the material which reacted and liberated heat sooner and at a higher rate, but gave a lower maximum temperature, cured better than the delayed-reaction, high ΔT types with the aggregate included in the mix at lower temperatures. Figure A1, curves A-1 to B-2, and Figure 1, curves A, B and D, show a comparison of liberation heat rates and maximum heats obtained. The curability of materials with fine aggregate included that showed the best potential for low temperature use are, in decreasing order, urethane, polyester and epoxy.

The unconfined compression strength test revealed that the J-27 neat polyester resin cured at the test temperature of -10°C and its unconfined compression strength was 14,115 kPa (2045 psi). But when mixed with sand its strength was 5325 kPa (770 psi), less than half as much. The unconfined compression strength of the Gold Label polyester resin mixed with sand measured 7025 kPa (1020 psi). Note again that the urethane resin did not give as much ΔT as the neat polyester resin did at -10°C but did react sooner (see Fig. 1). This may explain why the urethane sand mix did cure at the -20°C temperature within 24 hours, giving 15,230 kPa (2210 psi), more than twice the polyester strengths.

It is interesting that in unconfined compression strength tests at constant strain rate on the urethane (both neat and with sand) the load-deformation curve is increased to a maximum where plastic yielding occurs. Then strain continues at constant load. An investigation of the stress-strain relationships may indicate that this urethane and urethane composite (resin sand mixture) is an elastic-plastic material. After 24-hour curing at -30°C and subsequent to unconfined compression testing, the urethane was warmed (at 20°C) for 24 additional hours. Further chemical reaction apparently then occurred, which increased the unconfined compressive strength. This property could be of value in that it might allow placement of construction materials during a cold season. The material could be loaded at a high safety factor, if at all, initially, with a normal load safety factor being applied as the temperature rose and ultimate material strength developed*. Table 4 contains all unconfined compressive strength test results of the materials mixed with sand.

*Provided delayed full curing proceeded properly at the lower rates of temperature increase in the field as compared to the rapid change from -30 to +20°C in the laboratory.

Table 4. Unconfined compression strength of resin solutions mixed with fine aggregate (sand) after 24 hours.

Cure temp (°C)	Urethane (EP System) (psi)	Polyester (Gold Label) (psi)	Polyester (J-27) (psi)
-10	2726	1019	772
-20	2209	Sticky samples	Sticky samples
-30	2004 (2583*)	--	--

*This is the result after 24 additional hours (total 48 hours) of curing and testing the same sample.

Of the non-portland cement type materials, Sufaset F-181 shows promise for casting and constructing at low temperatures. The test results of curing at -10°C (using 3.9°C water) indicate that the material could be of value in situations where natural water sources are available even though the air temperature is below 0°C (as low as -10°C). For instance, the air temperature may be below freezing but a nearby well or a flowing stream may provide water for mixing. If Sulfaset F-181 is mixed with cold aggregate, the low temperature and heat sink effect may lengthen the working time (pot life) of this quick setting compound. This could prove valuable for volume use and placement, if the material generates enough heat to keep from freezing and/or can set within a reasonable time for mixing and placement (working time). Information on other non-portland cement type materials and patching compounds that include manufacturer-suggested low temperature ranges for curing may be found in a previous report (Johnson 1977).

Of the materials investigated at low temperatures in the grout study, the set test samples that included neat solutions were stored for one year. During that time, the samples were exposed to room temperature of about 20°C. Over this period many of the samples experienced shrinkage and/or syneresis (Table 5). The behavior of AM-9, with the highest shrinkage, is interesting. An AM-9 sand-injected sample had also been kept at room temperature for a year but remained hard and intact.

The attractiveness of using the materials tested in this study will depend on factors such as application temperature and curing time. Some of these materials cure more rapidly than others when used at the same temperature, as is evident in the set tests conducted. Where expedient military construction is necessary, quick setting after placement is an advantage. The successfully tested materials have the advantage of curing at low temperatures without being heated. Other available construction materials suffer the disadvantage of requiring heat, construction enclosures, extra labor and equipment. The economic effectiveness

Table 5. Shrinkage and/or syneresis of set test samples after one year (neat solution).

Cement and material or chemical solution	Type	Shrinkage (vol % loss)	Remarks
EP System	Urethane resin	0	The product remained hard and solid
XB-2403 Mud Lock	Hydrophilic urethane resin	25-30	The product remained hard and solid, and appeared to be porous
Raylig-260 L	Chrome-lignin gel	30-35	The remaining product cracked into a mass of broken parts (1.3 cm) and was very brittle
AM-9	Acrylamide gel	85-90	The remaining product was hard and appeared to be like a very hard plastic
Cyanaloc 62	Urea-formaldehyde	10-15	The product remained hard and solid
Hayward Baker Sodium Silicate	Sodium silicate	40-45	The remaining product essentially reduced to a powder
Siroc	Sodium silicate	40-45	The remaining product essentially reduced to a powder
Celtite 55 Terraset	Sodium silicate	65-70	The remaining product was a brittle solid material that crumbled easily. It remained more firm (solid) than the other sodium silicates.

*After one year (neat solution)

of the use of these successfully tested materials is then an advantage for cold regions, provided they can be placed by conventional means and have low cost, non-toxic, environmentally acceptable clean-up provisions. These higher cost materials may then become more economic in the cold than conventional concrete cement.

CONCLUSIONS

The urethane resin solutions tested here can be cured at temperatures as low at -30°C as a neat solution or mixed with aggregate (sand), while maintaining low viscosity and good workability for cast-in-place construction. This can be accomplished without adding heat or affording protection from freezing. The polyester resins will cure with similar handling properties and conditions at temperatures as low as -10°C, but will develop lower unconfined compression strengths.

The Sulfaset F-181 non-portland type cement can be cured at -10°C by use of water at 3.9°C. This material can be used for construction patching but may cure too fast for volume use.

RECOMMENDATIONS

Further efforts should be made to investigate other material properties as well as information from field evaluation.

Laboratory testing

The resin materials that were successfully cured here should be further tested using coarse-fine aggregate mixtures to optimize strength and cost for low temperature use.

Sulfaset F-181 (anchor bolt compound) should be mixed with low temperature aggregate to measure properties such as the heat sink effect, workability, and curability.

Other material properties that should be obtained are:

- Tensile strength
- Bonding strengths
- Stress-strain relationships
- Modulus of elasticity
- Modulus of rupture
- Modulus of shear
- Creep deformation
- Volumetric strain versus axial strain
- Durability (resistance to sulfates, acids, temperature cycle, etc.)

These will provide practical structural design information and property information to compare with other construction materials.

Field evaluation

A test slab of sufficient size and configuration should be cast in place to provide an adequate number of samples for validation of the laboratory test. It should be placed in the field at below-freezing temperatures, e.g. during winter at CRREL. This would allow an in-depth evaluation of construction procedures and materials.

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APPENDIX A

GROUT MATERIALS FOR LOW TEMPERATURE USE

INTRODUCTION

This appendix presents data extracted from results of an experimental program undertaken to develop information on grout solutions in connection with grouting soils at temperatures of 3.9°C and below (Johnson 1979). The grouts tested were organic chemicals and/or resins as well as sodium silicates that were potential products for low temperature use. Some of the same materials were recommended for further research in connection with placement of construction materials at low temperatures and were used in the present investigation. Therefore, the data in this appendix are of value for a more complete evaluation of the products tested.

The materials tested were potential chemical grout solutions as well as existing grout solutions (see Table A1).

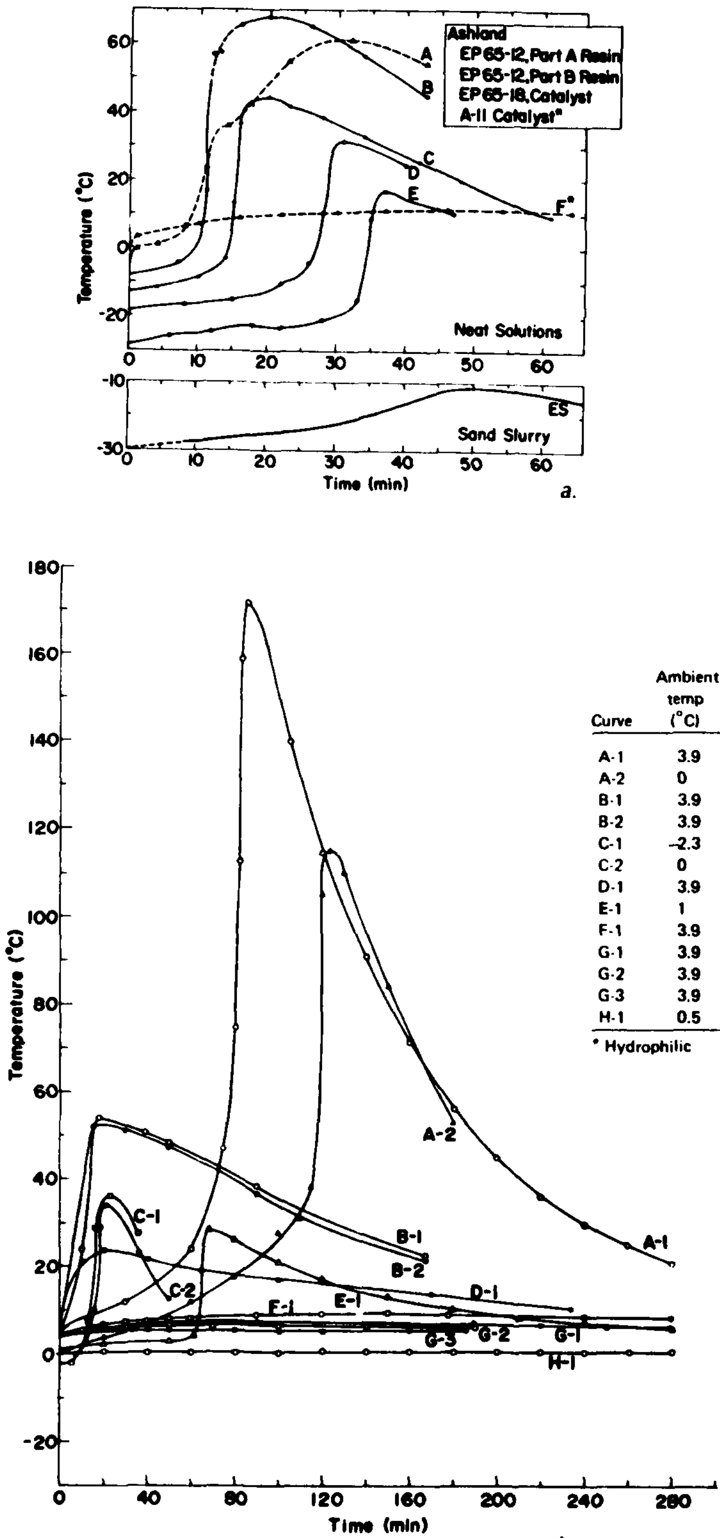
PREPARATION OF SOIL SAMPLES

Cast acrylic cylinders approximately 3-1/4 in. OD and 2-11/16 in. ID by 8 in. long were used to mold the samples. The cylinder molds were held together with threaded ready rods holding end caps and rings. The inside surface of each cylinder was coated with wax. Farrel silty sand was put into the molds at 110 lb/ft^3 dry density and Jenks sandy silt at 80 lb/ft^3 dry density before being saturated with distilled water. The dry densities were chosen as representative of frozen ground which has thawed. The cylinder molds included a porous material (similar to porous stones) which would allow the soil samples to be saturated from the bottom. This was accomplished by placing the samples held by the molds under a 1-in. head of water at 39°F or 33°F for 24 hours. All materials as well as the equipment were maintained and used in coldrooms in which the temperatures were 39 $\pm$ 1°F, 33 $\pm$ 0.9°F, and 20 $\pm$ 1°F.

The samples were injected using a pressurized tank with an agitator that held the grout solution while the pressure injection process was accomplished. The solution was pressurized into the soil sample from the top down. After the injected samples remained in the molds overnight, they were unmolded and placed in a 39°F or 33°F water-bath for the remainder of 7 days, unless otherwise stated, to determine whether the initial set product would dissolve or otherwise be affected by complete immersion of the samples.

TEST METHODS

Set tests to determine if the grout material would set or gel at 39°F were performed using 4-in.-diameter by approximately 6-in.-long cylindrical molds with a thermocouple centered in the mixed chemical



Curve	Catalyst (%)	Ambient temp (°C)
A	0.54	- 1
B	1	- 7.6
C	1	-12.9
D	1	-18.0
E	4.1	-28.4
F*	1.5	0.3
ES	4	-30

Curve	Ambient temp (°C)	Solution	Name
A-1	3.9	Epoxy Resin	EPOTUF
A-2	0	Epoxy Resin	EPOTUF
B-1	3.9	Urethane Resin	EP 65-92, 93 (1% EP 65-18)
B-2	3.9	Urethane Resin	EP 65-92, 93 (1% EP 65-94)
C-1	-2.3	Urethane Resin	EP 65-92, 93 (1.5% EP 65-18)
C-2	0	Urethane Resin	EP 65-92, 93 (1.5% EP 65-18)
D-1	3.9	Urethane Resin*	Mud Lock
E-1	1	Acrylamide	AM-9
F-1	3.9	Chrome-Lignin	Raylig 260-L
G-1	3.9	Sodium Silicate	Celtite
G-2	3.9	Sodium Silicate	Hayward Baker
G-3	3.9	Sodium Silicate	SIROC
H-1	0.5	Urea-Formaldehyde	Cyanaloc 62

* Hydrophilic

Figure A1. Temperature-time curves of set test.

Table A1. Grout materials and chemical solutions tested

Grout or chemical solution	Type
EP Systems and A-11	Urethane resin
XB-2403 Mud Lock	Hydrophilic urethane resin
Epotuf 37-130 and 37-052	Epoxy resin
Ancamine AD, LT and MCA	Curing agents (used with epoxy resin)
Raylig-260 L	Chrome-lignin gel
AM-9	Acrylamide gel
Cyanaloc 62	Urea-formaldehyde
Hayward Baker Sodium Silicate	Sodium silicate
SIROC	Sodium silicate
Celtite 55 Terraset	Sodium silicate
Portland Cement Type I	Cement slurry
Portland Cement Type III	Cement slurry
Bentonite clay	Bentonite slurry

or grout solution. The thermocouples were wired to a data retrieval system. The results are presented in Figure A1. Unconfined compressive strength tests were run on each sample after it had been immersed in 39°F or 33°F water for a period of 7 days, unless otherwise stated. The samples were trimmed, using a concrete saw, to a 6-in.-long cylinder with a 2.75- to 2.85-in. diameter. All samples were strained at 0.1 cm/min. The unconfined compression test results are presented in Table A2 and Figure A2.

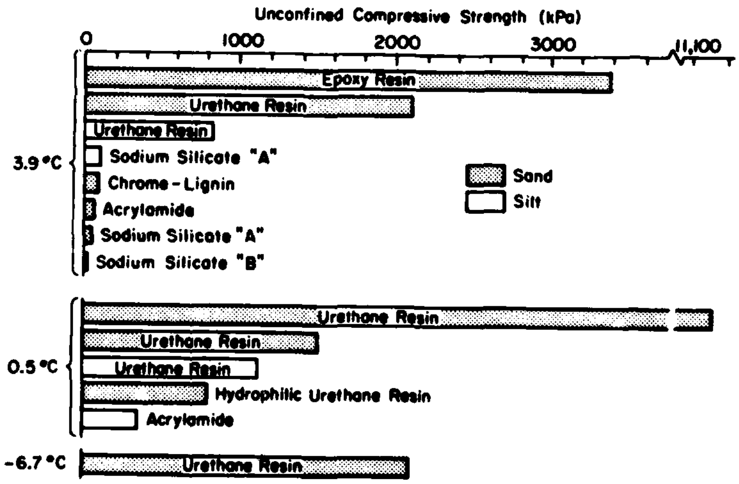


Figure A2. Unconfined compressive strength of samples.

Table A2. Properties and test results of solutions for low temperature grouting.

			Aqueous solutions				
	Urethane resin EP series	Epoxy resin Epotuf	Hydrophilic urethane resin XB-2403 Mud Lock	Acrylamide AM-9	Sodium silicate Celtite 55 Terraset	Sodium silicate Siroc	Chrome-lignin Raylig 260-L
Manufacturer-suggested cure temp, °F (°C)	Experimental product	30 (-1.1)	Down to 32 (0)	Not given	Down to 32 (0)	Not given	Not given
Set information	Controlled	Controlled	Not controlled	Controlled	Controlled	Controlled	Controlled
Air temp, °F (°C)	39 (3.9)	39 (3.9)	33 (0.5)	39 (3.9) 33 (0.5)	39 (3.9)	39 (3.9)	39 (3.9)
Resin or solution component temp, °F (°C)	39 (3.9^R)	39 (3.9^R)	33 (0.5^R)	39 (3.9^C) 33 (0.5^C)	39 (3.9^C)	39 (3.9^C)	39 (3.9^C)
Water temp, °F (°C)	No water	No water	No water	39 (3.9) 33 (0.5)	39 (3.9)	39 (3.9)	39 (3.9)
Soil type and temp before injection, °F (°C)	Sand 39 (3.9) 33 (0.5)	Sand 39 (3.9)	Sand 33 (0.5)	Sand 39 (3.9) Silt 33 (0.5)	Sand and silt 39 (3.9)	Sand 39 (3.9)	Sand 39 (3.9)
Reaction							
Time of max temp (min.)	25		23	70	40	See Fig.Ala	See Fig.Ala
Max temp, °F (°C)	100 (38) 129 (54) (with diff. catalyst)		73 (23) (resin and water 50-50)	86 (30)	41 (5)	39 (4)	46 (8)
7-day unconfined compressive strength of injected soil (psi)	306 at 3.9°C 1624 at 0.5°C	449	115	8 (Sand) 51 (Silt)	7 (Sand) 15 (Silt)	3	8
Approximate cost (1978)	*	81.5¢/lb	$13.00/gal	*	$1.26/gal W.S.	*	$20.00/short ton F.O.B. Hoquiam, Wash.
Source	Ashland Chemical Co., Columbus, OH 43216	Reichhold Chemical, Inc. White Plains NJ 10602	3M Company, St. Paul, MN 55101	American Cyanamid Wayne, NJ 04740	Celtite Inc., Cleveland, OH 44133	Raymond International, Inc. Cherry Hill, NJ 08034	ITT Rayonier Inc., New York, NY 10016
Remarks	A low viscosity (like water) solution for soil stabilization.	Viscosity increases significantly as temperature decreases below 3.9°C.	Set upon contact with water and set quickly in small amounts.	Low viscosity solution (like water), gels to a resilient product. Shows promise for stabilization of silts.	Low viscosity solution, sets to an easily crumbled material.	Set with low exotherm, low viscosity solution (like water), gels to a moist, brittle, solid gel. Shows promise for stabilization of silts.	Viscosity increases greatly as temperature decreases below 3.9°C.

* See Appendix B
R - resin; C - components (including water temp.)

APPENDIX B. GROUT OR SOLUTION COST INFORMATION.
(From Johnson 1979.)

Solution no.	Price/lb(1978)	Flash point
Urethane resin (Ashland Chemical Co.)		
EP 65-92	$ 0.4968	105°F
EP 65-93	0.5393	
EP 65-12	0.7060	130°F
EP 65-17	0.5195	120°F (PM)
EP 65-18*	4.7237	276°F (PM)
EP 65-94*	7.0350	198°F (PM)
A-11*	1.5217	130°F (TCC)
Hydrophilic urethane resin (3M Company)		
XB-2403 Mud Lock	$13.00/gal.	0°F (TCC)
Epoxy resin (Reichhold Chemical Inc.)		
Epotuf 37-130	$ 0.78 (truck load)	200°F (TCC)
Epotuf 37-052	1.08 (truck load)	130°F (TCC)
Hardeners/Reactant (Pacific Anchor Chemical Corp.)		
Ancamine AD	$ 1.60	180°F (TCC)
Ancamine LT	1.40	213°F (TCC)
Ancamine MCA	1.70	230°F (TCC)
SIROC (Raymond International)		
Silicate	$ 0.93/gal.	
Amide	4.74/gal.	
Chloride	0.30/10 lb bag	
Celtite 55 Terraset (Celtite Inc.)		
Part A	$ 0.91/gal.†	281°F (TCC)
Part B	0.91/gal.†	
Lignosulfonate (ITT Rayonier Inc.)		
Raylig-260 L	$20.00/short ton F.O.B. Hoquiam, Wash.	

* Catalyst
† Price from Paul Rausch
(PM)=Pensky-Martens Closed Tester (ASTM D93)
(TCC)=Tag Closed Tester (Cup) (ASTM D56)