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Cement Technology for Plugging Boreholes in Radioactive-Waste-Repository Sites: Progress Report for the Period October 1, 1978, to September 30, 1979

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Contents

PREFACE

This is the last in a series of annual progress reports summarizing the research and development efforts on cement technology relative to borehole plugging. The work was conducted in the Chemical Technology Division of the Oak Ridge National Laboratory (ORNL) during the period October 1, 1978, to September 30, 1979. Previous work in this field has been described in the following progress reports:

1. *Cement Technology for Plugging Boreholes in Radioactive Waste Repository Sites: Progress Report for the Period October 1, 1977, to September 30, 1978*, ORNL-5524 (August 1979).
2. *National Waste Terminal Storage Program Progress Report for the Period October 1, 1976 to September 30, 1977*, Y/OWI-9 (April 1978).
3. *National Waste Terminal Storage Program Progress Report for the Period April 1, 1975 to September 30, 1976*, Y/OWI-8 (November 1976).

CEMENT TECHNOLOGY FOR PLUGGING BOREHOLES IN RADIOACTVE WASTE REPOSITORY SITES: PROGRESS REPORT FOR THE PERIOD OCTOBER 1, 1978, TO SEPTEMBER 30, 1979

J. G. Moore, M. T. Morgan, E. W. McDaniel,
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SUMMARY

Laboratory evaluations were made of several borehole plug formulations proposed for the Bell Canyon field test. Measurements included compressive strength, permeability, density, and thermal conductivity. A few preliminary tests with saltcrete formulations showed no significant difference in physical properties of the solid as a function of fly ash or cement composition. The saltcrete proposed for the field test gave acceptable pushout strength and permeability values using miniature borehole plugs in anhydrite. Similar laboratory tests made with a freshwater formulation indicated high permeability. Electron micrographs showed dissolution cavities or cracks at the plug-wall interface. These studies showed that the reactions occurring between the borehole plug and the adjacent rock wall are an important factor in obtaining a good seal and that laboratory tests are useful to indicate the possibility of success or failure of field tests.

Abstracts of separate reports on permeability experiments and systematic studies of pozzolanic cement and saltcretes are included.

1. INTRODUCTION

In the selection of a site for use as a permanent repository for radioactive wastes, provision must be made to seal (plug) all holes drilled in the area to ensure the integrity of the site. These plugs must form leakproof bonds with the wall rock and must have lifetimes comparable to the surrounding rock formations. An evaluation of previous work indicated that one of the more viable solutions involves the use of cementitious materials.[1]

The Cement Technology for Borehole Plugging Program was established to assist the Office of Waste Isolation [now the Office of Nuclear Waste Isolation (ONWI)] in devising and testing cementitious mixtures suitable for plugging drill holes that may represent a hazard to a radioactive waste repository.

During this report period, systematic studies of pozzolanic (fly ash)—cement and salt—cement mixtures were continued. Because of its major importance, special emphasis was placed on the development and use of techniques for permeability measurements in the microdarcy range. In addition, the formulations proposed by Sandia Laboratories for a borehole plugging field test were evaluated by using core specimens from the actual site; this operation was designated as the Bell Canyon test (BCT).[2] Sandia Laboratories also planned and conducted the BCT to verify that a cement-grout plug could be installed and would withstand hydrostatic pressures of about 13.8 MPa (2000 psi) in a borehole. After placement of the plug, permeability measurements were made for comparison with laboratory determinations.

Data from the laboratory evaluation tests made for the borehole plugging field test are discussed in detail. However, since reports were recently published describing all the systematic and permeability studies, only the summaries from these reports are presented.

2. EFFECT OF FLY ASH AND SALT ON CEMENTITIOUS MIXTURES

Results of the initial studies of a systematic investigation undertaken to determine the effects of fly ash and salt on the physical properties of pozzolanic concretes and saltcretes have been reported.[3] The objective of this program was to develop cementitious mixes that will form thermodynamically stable solids suitable for plugging boreholes in various geologic media. In the first phase of the investigation, some of the problems and major effects in such cementitious systems were identified, and laboratory techniques were developed.

The addition of fly ash to mortars decreased the set time and bleed characteristics, increased the compressive strength and permeability, but had very little effect on the density or the thermal conductivity of the solid. The magnitude of these effects was only slightly related to the lime content of the fly ash.

In the case of saltcretes, the addition of a low-lime fly ash slightly decreased the set time and the bleed characteristics of the wet mix. However, a high-lime fly ash doubled the set time and decreased the bleed characteristics to essentially zero. The compressive strength of saltcretes was increased by the addition of fly ash and was independent of the lime content. Such additions had little effect on the thermal conductivity or density.

The thermal conductivities of cement pastes containing fly ash showed a near-linear relationship with the density of the resulting solids. In the case of mortars, the thermal conductivity decreased with increasing temperature and showed some hysteresis in the initial heating cycle. After the first cycle, the thermal conductivity decreased from about 1.32 W/m·K at 350 K to 1.27 W/m·K at 475 K.

These interim results have shown the need for more detailed studies on the role of water and fly ash composition in these formulations and the necessity to develop a more accurate means of measuring time-dependent volume changes that occur in cementitious specimens.

3. PERMEABILITY OF CEMENTITIOUS SOLIDS

Techniques were developed to measure gaseous and liquid permeabilities in the microdarcy range. The abstract from the publication describing these techniques and presenting data obtained from measurements involving cementitious solids and plug-wall rock junctions follows.[4]

> The permeability of borehole plug solids and plug-wall rock junctions is a property of major interest in the Borehole Plugging Program. This report describes the equipment and techniques used to determine the permeabilities of possible borehole plugging materials and presents results from tests on various cementitious solids and plug-rock combinations. The cementitious solids were made from mixtures of cement, sand, salt, fly ash, and water. Three different types of cement and four different fly ashes were used. Permeabilities ranged from a high value of 3×10^{-4} darcy for a neat cement paste to a low of 5×10^{-8} darcy for a saltcrete containing 30 wt % sodium chloride.
>
> Miniature boreholes were made in the following four different types of rock: Westerly granite, Dresser basalt, Sioux quartzite, and St. Cloud granodiorite. These small holes were plugged with a mix consisting of 23 wt % Type I Portland cement, 20 wt % bituminous fly ash, 43.2 wt % sand, and 13.8 wt % water. After curing for 91 days at ambient temperature, the permeability of the plug-wall rock junctions ranged from 3×10^{-5} to $< 1 \times 10^{-8}$ darcy. Three of the four miniature plugged boreholes exhibited permeabilities of < 10 microdarcys.

4. EVALUATION OF FIELD-TEST PLUG FORMULATIONS

A borehole plugging program was initiated several years ago by Sandia Laboratories as part of the project concerned with the Waste Isolation Pilot Plant (WIPP).[5] Work was sponsored at the U.S. Army Corps of Engineers Waterways Experiment Station (WES) to develop pumpable cementitious mixtures that would form durable plugs.[6] The BCT was undertaken during this report period to verify the ability to place a grout in a borehole and to determine the short-term plugging effectiveness of one of the grout formulations developed by WES. In addition, the BCT presented an opportunity to compare laboratory and field data and to possibly indicate the course of future laboratory studies. The BCT was conducted in an abandoned exploratory site about 5 miles northeast of the proposed WIPP shaft. At the request of ONWI, ORNL evaluated several WES proposed plug formulations and assisted in the selection of a formula suitable for field operations. Core sections of the anhydrite wall rock from the test site were furnished by Sandia so that plug-wall rock bonding and permeability studies could be made.

The techniques for sample preparation and the determination of the physical properties of the wet mix and cured solids were described previously.[7] In addition, the push-out strengths and permeabilities of plug-rock combinations were determined experimentally. Miniature boreholes were formed by coring 2.54-cm-diam holes in 10.16-cm-diam anhydrite samples. These anhydrite samples were part of core No. 10, boxes 178 and 179, and were obtained at a depth of 1332.9 ± 0.5 m in AEC well No. 7. Slices of the cored samples, 2.54-cm thick, were filled with the plug mix for the push-out tests. The 2.54-cm anhydrite cores left from the above operation were bored to make still-smaller plugged boreholes, 1.27 cm diam by 2.54 cm long, for permeability measurements.

The grout specimens and/or plug-rock samples were made with several test formulas (Table 1). Most of the grouts, which were suspended over a small amount of excess water in the bottom of sealed Mason jars, were cured at specified temperatures. Specimens of the final BCT mix (Formula 6) were cured in preheated simulated ground water (Table 2). Physical

Table 1. Composition of borehole plug formulations

	Formula number					
	1[a]	2	3	4	5[b]	6
Class H cement, wt %	50.8	46.7	57.2	57.8	48.6	52.2
Fly ash						
Type	P20	P20	P20	P20	P22	P22
Wt %	17.2	16.5	19.4	19.6	16.6	17.2
Salt, wt %	7.4	7.4	8.3	8.4	6.[illegible]	
Dowell additives, wt %						
Expander A		4.9			6.6	7.0
Plasticizer D-65				0.1[c]	0.2	0.2
Defoamer D-47					0.017	
Water, wt %	24.6	24.6	15.1	14.0	21.6	23.0
Ratio of water/cement	0.48	0.53	0.26	0.24	0.44	0.44

[a] 0.1% Plastiment was used in some mixes with this formula.

[b] Measured bleed rate = 1.535×10^{-6} $cm^3/cm^2 \cdot s$, bleed capacity = 3.59 x 10 cm^3/cm^2, initial set time = 42 h, and final set time = 47 h.

[c] Super plasticizer used was Plastiment made by the Sika Corp.

Table 2. Simulated WIPP-site ground water composition[a]

Compound	Concentration (g/ℓ)
NaCl	303.22
$NaHCO_3$	0.32
$MgSO_4$	0.62
$NaSO_4$	9.33
$CaCl_2$	0.47

[a] Composition supplied by Sandia Laboratories.

properties of the grout specimens and plug-rock samples were determined after specified curing times. Established procedures were used to measure permeabilities.[4]

The temperatures of the BCT borehole were originally estimated to range from ambient at ground level to ~55°C at a depth of 4800 ft (1460 m); actual temperature measurements were 34°C at the bottom.[8] Thus some samples were cured at 40 and 55°C in addition to ambient (22 to 25°C) and several specimens were cured at 85°C, because it was assumed that increasing the curing temperature would accelerate any reactions taking place. This should produce the same changes in concrete in a short time that would occur normally over an extended period. The test criteria would be to compare the microstructures, properties, and ratios of components produced by normal and accelerated reactions. If identical results were produced by accelerated reactions over several months as compared to a few years of normal reaction, it would be possible to extrapolate accelerated results to simulate extended periods of normal conditions.

4.1 Preliminary Studies

Preliminary studies were made with some earlier WES formulations[6] or modifications thereof to set up the necessary test procedures. The ingredients used in these tests and the final field test formulations were selected on the basis of the initial development studies at WES. The mixes contained Dowell fly ash (designated Lite-Poz 3), Class H cement (from the Incor Cement Co. plant at Maryneal, Texas), and an expansive agent. The cement provided the 5% C_3A content required for the expansive agent to achieve the desired level of expansion. In addition, the coarseness of this cement should allow the use of a lower water/cement (W/C) ratio, which would result in a higher density and a lower permeability. The use of brine drilling mud and the possibility of the presence of halite lenses in the wall rock suggested that salt should be present in the formulations, which would improve the compatibility of the grout mix with the surrounding environment. However, as the investigation proceeded, no halite lenses were found, which eliminated part of the need to use a brine mix.

The initial series of mixes was made to determine the effect of fly ash composition, cement type, and water reducing agents. Materials used in this study were two samples of lignite fly ash (P17 and P20, Table 3) from Dowell with different compositions; Type I Portland cement; class H cement; and Plastiment, a superplasticizer or water reducing agent. Mixes containing these ingredients were prepared by Formula 1 and cured for 1 to 28 days. The compressive strengths were measured on single 2-in. cubes and are plotted vs curing time in Figs. 1 and 2.

These results show that no significant changes in strength were produced by the water reducing agent or by the difference in cement or fly ash composition. The curves, which are typical for cementitious mixes, show a rapid increase in strength during the first 3 days and a slower increase over the remaining time.

The mixes prepared using Formula 1 were very thin and watery; therefore, additional mixes (Formulas 3 and 4) were made in which the water content was reduced. Plastiment was added in Formula 4. The compressive strength of these mixes at 28 days of curing time is compared in Table 4. As anticipated, the mixes with the reduced water (lower W/C ratios) show a definitely higher strength.

Formula 2 (Table 1) is the same as Formula 1 but contains an expansive agent that caused no change in permeability, density, or thermal conductivity. Specimens of the two formulations cured 28 and 91 days at various temperatures showed only the usual experimental variation (Tables 5 and 6). The compressive strength, however, was higher at 91 days curing time or with higher curing temperatures.

Table 3. Class C fly ash analytical data[a]

	Fly ash No.		
	P17	P20	P22
Free CaO, mg/g	8.00	0.60	4.70
Surface area, cm^2/g	1.71	1.20	1.16
Density, g/cm^3	2.62	2.64	2.68
Particle size,[b] μ	12.36	10.89	10.26
Moisture, %	0.03	0.05	0.07
SiO_2, %	29.40	41.00	33.60
CaO, %	32.50	26.70	23.20
Fe_2O_3, %	4.36	4.97	5.98
NaO, %	0.76	1.27	1.51
P_2O_5, %	0.901	1.39	1.00
TiO_2, %	0.88	1.33	1.50
LOI,[c] %	0.93	1.23	0.68
MgO, %	2.67	3.24	4.66
Al_2O_3, %	11.80	17.30	18.10
SO_3, %	2.28	1.57	2.32
K_2O, %	0.44	0.76	0.53
C, %	0.29	0.22	0.23

[a]Analytical data are given in wt %.

[b]Maximum size of 50 wt % of the particles.

[c]Loss on ignition.

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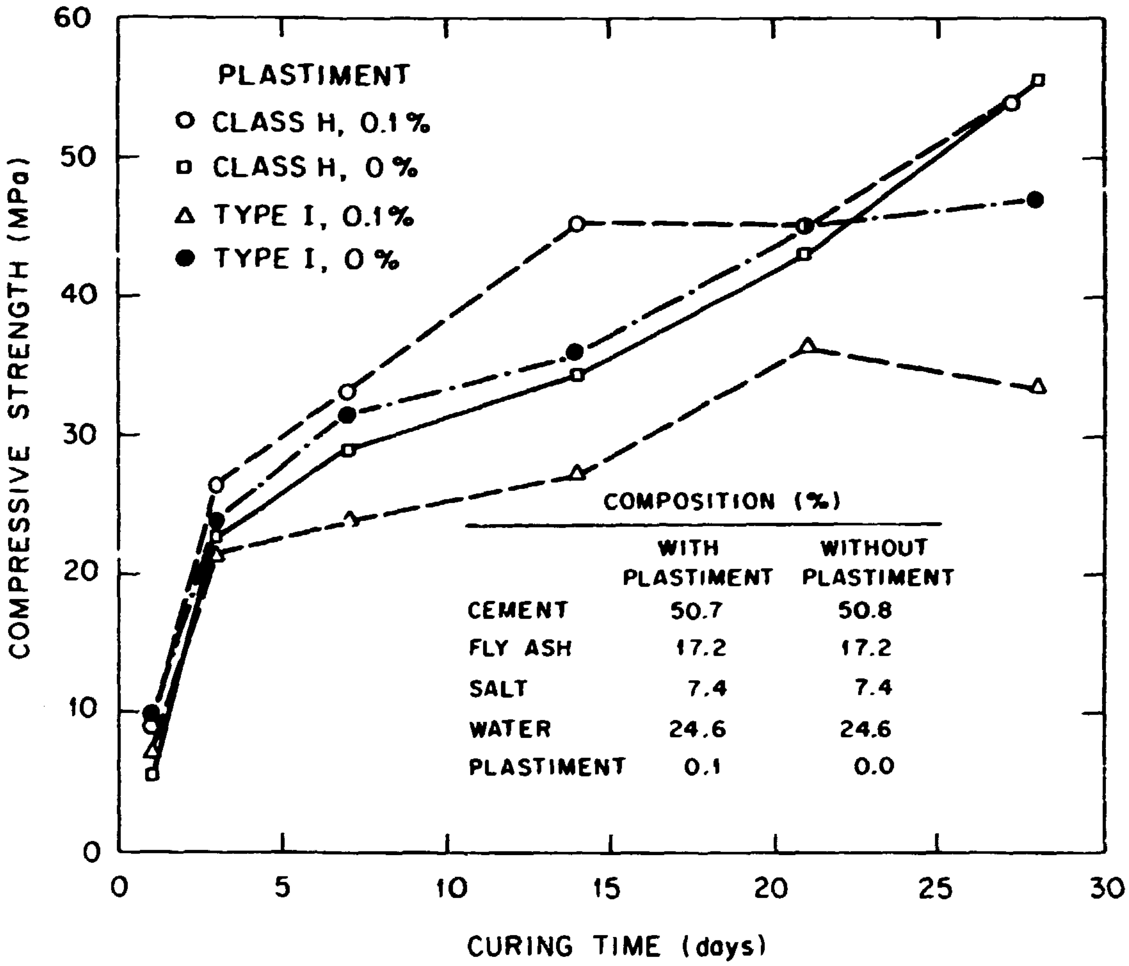

COMPOSITION (%)	WITH PLASTIMENT	WITHOUT PLASTIMENT
CEMENT	50.7	50.8
FLY ASH	17.2	17.2
SALT	7.4	7.4
WATER	24.6	24.6
PLASTIMENT	0.1	0.0

Fig. 1. Compressive strength of saltcretes with Class H and Type I cements and P20 fly ash vs curing time.

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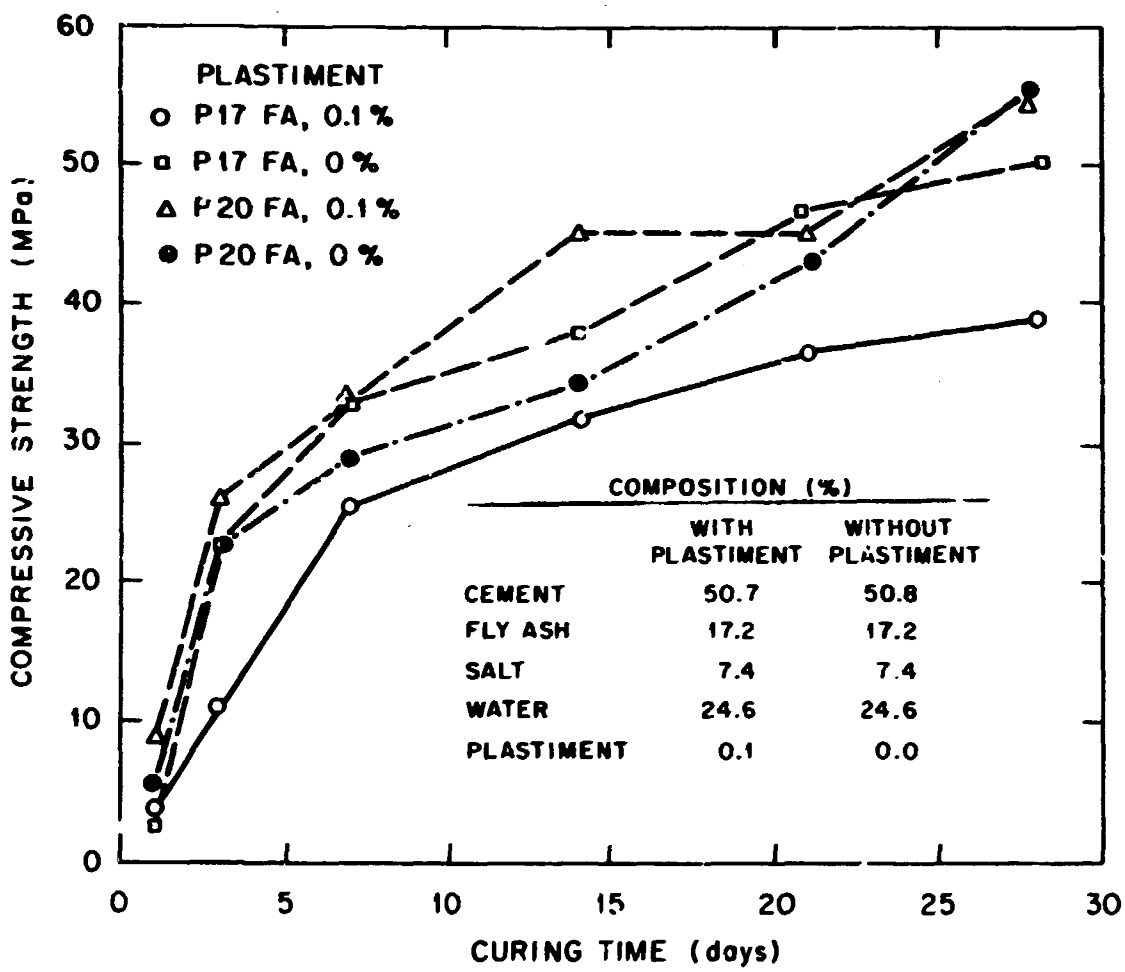


Fig. 2. Compressive strength of saltcretes with Class H cement and P17 and P20 fly ash vs curing time.

Table 4. Comparison of compressive strength of saltcrete with different W/C ratios[a]

Formula No.	W/C ratio	Compressive strength at 6 days (MPa)	Compressive strength at 28 days (MPa)
1	0.48	33	56
3[b]	0.26	65	81
4[c]	0.24	60	71

[a]Cured wrapped in impervious film at ambient temperature 295 to 298 K.

[b]Measured slump of K = 5.0 and workability of W = 2.25.

[c]With 0.1 superplasticizer (Plastiment).

4.2 Experiment With Final BCT Borehole Plug Formulations

The formula for the BCT field test plug was selected by Sandia Laboratories from the WES formulations. The initial Sandia selection, Formula 5 (Table 1), contained salt. Permeability measurements made at ORNL using WIPP brine showed this formula was quite promising (Formula 5, Table 6); however, tests on larger samples at WES in which tap water was used resulted in higher leakage, and salt crystals were found in scrapings from interface surfaces.[9] Salt was thus omitted in the final formula selected by Sandia Laboratories (Formula 6, Table 1). Tests at WES with this fresh water mix showed that the bond strength was higher, the permeabilities were moderate, and the expansion was approximately five times greater than that obtained with the salt mix.

The physical properties of plug and plug-rock combinations made with Formulas 5 and 6 are given in Table 7. Push-out strengths ranged from 2.5 to 5.4 MPa, with no distinct difference as a function of curing time or mix composition. Nitrogen permeabilities of the Formula-5 plug solid ranged from 1×10^{-7} to 2×10^{-6} darcy. The higher permeabilities were due to microcracks. With the exception of the sample cured for 7 days at 22°C, the liquid permeabilities of the Formula 5 plug-rock

Table 5. Physical properties of preliminary borehole plug formulations cured 28 days[a]

Formula No.[b]	Curing temperature (K)	Compressive strength (MPa)	Permeability (darcy)	Dry density (g/cm^3)	Thermal conductivity (W/m·K)
1	295	36	2.5×10^{-4}	1.66	0.67
	313	60	2.0×10^{-4}	1.70	0.70
	328	64	1.6×10^{-5}	1.70	0.72
	358	58	1.2×10^{-4}	1.70	0.67
2	295	32		1.67	0.63
	313	47	3.0×10^{-5}	1.70	0.65
	328	60	2.5×10^{-5}	1.66	0.62
	358	67	3.6×10^{-7}		0.58

[a]Compressive strengths were averages of six determinations, whereas other properties are single measurements on one sample each.

[b]Formula compositions are given in Table 1.

Table 6. Physical properties of preliminary borehole plug formulations cured 91 days[a]

Formula No.[b]	Curing temperature (K)	Compressive strength (MPa)	Permeability (darcy)	Dry density (g/cm^3)	Thermal conductivity (W/m·K)
1	295	36	8.0×10^{-5}	1.68	0.72
	313	76	1.0×10^{-4}	1.72	0.73
	328	59	2.8×10^{-5}	1.69	0.68
	358	66	8.0×10^{-5}	1.72	0.68
2	295	46	1.4×10^{-4}	1.64	0.62
	313	67			
	328	61	2.7×10^{-5}	1.68	0.61
	358	73	9.6×10^{-5}	1.66	

[a]Compressive strengths were averages of six determinations, whereas other properties are single measurements on one sample each.

[b]Formula compositions are given in Table 1.

Table 7. Results of plug-rock experiments with final BCT plug formulations

Formula No.	Curing temperature (K)	Curing time (days)	Push out strength[a] (MPa)	Compressive strength (MPa)	Permability (darcy): Plug-wall interface, Liquid	Permability (darcy): Plug-wall interface, N_2	Permability (darcy): Plug solid[b] (N_2)
5	295	7	3.7	27	1.3×10^{-3}[c]		2.1×10^{-6}
		14	5.0	36	4.0×10^{-9}		5.0×10^{-7}
	313	7	2.5	44			1.0×10^{-7}
	328	7	3.1	59			1.7×10^{-7}
		14	2.5	68	3.1×10^{-6}		1.9×10^{-6}[d]
6	295	7	2.7		2.6×10^{-5}	3.0×10^{-4}	
		14	5.4		1.1×10^{-3}	$\geq 1.0 \times 10^{-3}$	
	313	7	3.7		3.7×10^{-4}	2.1×10^{-4}	
		14	3.5		1.4×10^{-3}	$\geq 1.0 \times 10^{-3}$	
	328	7	3.8		4.0×10^{-4}	1.2×10^{-4}	
		14	3.6		2.0×10^{-5}	4.4×10^{-4}	

[a]Tests were made on 1-in. plugs in anhydrite annuli.

[b]Permeability of dried samples. All permeabilities of as-cured Formula-5 samples were $< 1 \times 10^{-7}$ darcy.

[c]Microfractures observed in sample.

[d]Fracture developed during drying after the permeability of the as-cured plug was measured.

interfaces were low. Much higher liquid permeabilities were measured on the Formula-6 plug-wall interfaces.

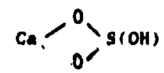
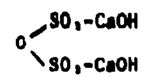
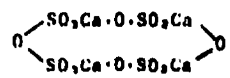
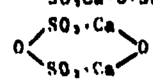
The high permeabilities in the Formula-6 plug-rock samples prompted a more detailed investigation. Optical and scanning electron microscopy revealed circumferential cavities that extended around the perimeter of the 1.27-cm-diam plug (Fig. 3). After making liquid permeability measurements using WIPP brine, the plug-rock samples were cut parallel to the axis and stored in a closed bottle for approximately 1 month before they were prepared for microphotography.

The crack-like cavity was assumed to be caused by dissolution of the anhydrite wall. Anhydrite normally dissolves very slowly in water and it has a complex hydration mechanism. Dissolution is accelerated by the presence of alkali halides and some other salts and is retarded by borax and boric acid.[10] In general, the solubility is increased by the presence of salt with no common ion. A summary of the properties of calcium sulfate is given in Table 8.[11,12] More research is necessary to determine the effect of plug-mix composition on dissolution of the adjacent anhydrite wall.

When the photomicrographs were submitted to WES for examination, it was determined that the free water remaining after the grout had hardened became saturated with sulfate by diffusion to achieve equilibrium concentration. The anhydrite that had dissolved left a roughened space in the anhydrite wall. The free water was eventually lost, leaving the space partially filled with the solids from the solution. Although the dissolved anhydrite was converted to gypsum, which occupies a larger volume than anhydrite, the volume of solids remaining in the crack was still much less than the volume of the solution which had previously filled the crack cavity. It was also suggested that an expansive grout is needed which will not dissolve anhydrite, and the mixing water should be saturated with $CaCO_4$.[13]

The results of these experiments showed that the saltcrete (Formula 5) plug-rock interfaces had considerably lower permeabilities than the mortar without salt (Formula 6); however, these preliminary studies do not explain the reasons for this difference. The solubility of both gypsum and anhydrite in water is enhanced by the presence of salt.[10] Because the

Table 9. Physical properties of calcium sulphate[a]

Form	Formula	Common names or description	Type	Crystal system	Diagram	Molecular weight	Specific gravity	Solubility (g/100 g H_2O at 25°C)
Dihydrate	$CaSO_4 \cdot 2H_2O$	Gypsum, gypsite, alabaster, selenite, satin spar, gypsum.	α β	Monoclinic Orthorhombic		172.17	2.32	0.24
Hemihydrate	$CaSO_4 \cdot 1/2\ H_2O$	Plaster of Paris - slow setting Plaster of Paris - quick setting	α β	Orthorhombic Orthorhombic		145.15		0.30
Anhydrous	$CaSO_4$	Mineral anhydrite		I Orthorhombic		136.14	2.96	0.20
		Dead burnt anhydrite	α	II Orthorhombic				
		Soluble anhydrite, Drierite	β	III Orthorhombic				

[a]Data are taken from refs. 10-12 in this report.

expansive agent, Dowell D-65, that was added to both mixes was designed for use with saltcretes, the expansion in Formula 6 mix (without salt) was more unpredictable. Both mixes were thin and watery. The use of less water would improve strength and reduce the excess water available to dissolve the anhydrite.

Although these preliminary experiments raised many interesting questions, they did show that laboratory experiments can largely predict the success or failure of a field trial. It is not practical to field test a large number of mix formulations; therefore, expanded laboratory studies are needed to completely develop an optimum mixture before field testing begins. Since boreholes can be expected to be drilled in many types of rocks, the compatability of the plug with other rocks should be investigated.

5. ACKNOWLEDGMENTS

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