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Development of an Improved Cement for Geothermal Wells

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Keywords

Geothermal cement, zeolites, self-healing, pozzolans, acid-resistance, ferrierite, intergrind, FlexCem®

ABSTRACT

Geothermal cements are subjected to thermal and mechanical shocks, high temperatures and aggressive environments that can cause cement damage and compromise well integrity, contaminate underground waters and, if not repaired in time, lead to the well collapse. Beginning in 2010, Trabits Group and subcontractor the University of Alaska Fairbanks undertook a Department of Energy Geothermal Technologies Office (GTO) funded project to develop an improved cement for geothermal wells. The goal of the research was to develop a zeolite-containing lightweight, high temperature, high pressure geothermal cement which would provide operators with an easy to use, flexible cementing system that saves time and simplifies logistics. For the GTO research jet mill processing was used to micronize test zeolite types. Jet milling, while effective for research sized batches, is not economic for commercial cement manufacture. Alternatives were investigated to reduce the high cost of micronizing and dry blending. The most economic option identified was to intergrind cement clinker and zeolite which can be done at the cement plant or at a standalone grinding facility. The intergrinding approach to manufacturing and choice of zeolite results in a variable density cement with a working range of 11.5 to 14.3 ppg. The intergrind method was tested at four U.S. cement plants. Each of these full scale, large tonnage intergrinds were compared for resulting cement chemical properties and cement performance. All four intergrinds were essentially identical in properties and performance proving the method provides a consistent and repeatable cement product. Trabits Group applied for and was issued a Trademark for "FlexCem" by the U.S. Patent and Trademark Office. FlexCem® cement retains the original GTO target qualities of thermal stability, resistance to carbonation and additionally exhibits the unique ability to self-repairing in a wide range of temperatures. FlexCem® was demonstrated to possess self-healing (strength-recovery) properties both at high hydrothermal temperature of 300□C and moderate temperature of 100^{o}C. The blend also strongly outperformed common high-temperature well cement formulation in acid resistance tests (pH 0.2, 90^{o}C, 28 days).

1. Introduction

In early 2010, Trabits Group submitted a geothermal cement development research proposal to the U.S. Department of Energy, Geothermal Technologies Office (GTO). The stated goal of the research program was "Development of a zeolite-containing lightweight, high temperature, high pressure geothermal cement which would provide operators with an easy to use, flexible cementing system that saves time and simplifies logistics". Nine target criteria were established. These were:

- Thermal stability with little strength retrogression to 300° C.
- Tensile strength to withstand temperature and pressure changes.
- Low-density, low-viscosity slurries with low equivalent circulating densities (ECD) without the need for air or nitrogen foaming.
- A single cement blend allowing density adjustments without adversely affecting slurry properties to eliminate the need for separate blends for lead and tail slurries.
- Resistance to carbonation.
- Accurate downhole densities throughout cement placement without significant changes in viscosity.
- Water absorption capacity without retaining free water.
- Good bonding to casing and formation.
- Adequate compressive strength.

During the initial screening, five different types of zeolite, each micronized to 5, 10 and 44µm, were used at three replacements of Class G and Class H cement at 15%, 27.5% and 40%. The initial screening samples totaled 180 individual tests in a heuristic process.

Trabits Group selected one best zeolite/cement blend for detailed testing and development. The zeolite used in this best blend was Ferrierite which exhibited excellent thermal stability. This best blend was subjected to long-term testing in geothermal brine from the Ormat Brawley field for a 3 month period after which it was tested for compressive strength, modulus of elasticity and permeability. This best blend met all nine target performance criteria.

Throughout the project Trabits Group conducted internal peer review and participated in the annual GTO Peer Review program. The project was completed in 2015 and after considering multi-year Peer Review comments Trabits Group reached the conclusion that commercial development of the technology was warranted.

Reviewer 25041

"This project develops a novel, zeolite-based light weight, high temperature, and high pressure geothermal cement."

Reviewer 23537

"It appears that the required criteria were met via this zeolite compound and that the Quality of this work and the Productivity standards were all met to a high degree."

Reviewer 23478

"The cements produced appear to be relatively easy to manufacture, they have far fewer additives or other chemicals, and are not complex. This probably means that they can be obtained easily for use in remote sites and that they can be emplaced without sophisticated equipment or procedures."

While the developed cement was successful, the method of making it was not. In the GTO research, jet mill processing was used to micronize test zeolite types. Jet milling, while effective for research sized batches, is not economic for commercial cement manufacture. The research clearly documented that a zeolite particle size of 15 to 20µm was optimal for performance at high temperatures and at low temperatures as well. In addition to the cost of micronizing zeolite there was the cost of dry blending with a finished API cement to make the final composite zeolite-containing cement.

2. Intergrinding Cement Clinker and Zeolite

Alternatives were investigated to reduce the high cost of micronizing and dry blending. The most economic option identified was to intergrind cement clinker and zeolite which can be done at the cement plant or at a standalone grinding facility. Intergrinding zeolite and cement clinker provides a bimodal particle size distribution given that zeolite is a “softer” material than clinker with the zeolite preferentially grinding finer. This preference for zeolite grinding finer results in the zeolite being “micronized” as the cement product is made.

Advantages of Intergrinding

- Economic gain of replacing a higher cost clinker with a lower cost zeolite.
- Increased physical properties of the zeolite intergrind product being improved strength, lower permeability and resistance to carbonation.
- Lowering the resulting interground cement environmental cost associated with greenhouse gases.

2.1 Interground Cement Design

When considering making a high temperature cement for geothermal application it becomes quickly apparent that the geothermal cementing market is small. Returning to the GTO research, base API Class cements were “enhanced” by the addition of zeolite along with additives for retrogression and retarders to ensure cement performance at high temperature. The zeolite provided performance benefits beyond what API Class cement alone could provide at high temperature.

In actual practice, none of the cementing service companies own cement plants. All buy cement whether it be Class G or H or some non API such as Type III. All use proprietary additives to create cement slurries with properties supposedly unique to a particular brand name. Geothermal application is no different than that of oil and gas wells. Temperatures are certainly higher and conditions are harsh but so are conditions in oil steam assisted gravity drainage wells. Cement slurries are designed for the conditions of the particular well or the conditions of the field.

Accordingly, the interground cement can be designed to serve as a base cement for geothermal well application and as a cement for the wider market of oil and gas well application.

Concept Goal

"Manufacture an interground lightweight variable density well cement with high performance that is economic with multiple applications from a single intergrind."

2.2 Initial Trial Intergrind

Unfortunately it is essentially impossible to duplicate full scale cement plant grinding mill performance in a laboratory bench scale grinding mill. Accordingly, Trabits Group contracted with a western U.S. cement plant to make a 1,000 ton intergrind run to test the basic parameters of milling rate, particle size distribution, resulting Blaine and final cement chemistry. For this first intergrind a Class G clinker and the zeolite Clinoptilolite were used in a design ratio of 55% clinker, 40% zeolite and 5% gypsum. The manufactured cement had an Optimum Density of 12.73 ppg, which was good, and had required strength at 24 hours but lacked the target of high early strength.

2.3 Second Test Intergrind

Taking what was learned in the Trial Intergrind we revisited the earlier GTO work and made the decision to use the zeolite Ferrierite (FER) as the replacement zeolite. We also looked at the cement clinker properties and made the decision to switch to a Type I/II clinker rather than Class G clinker. We also changed the cement/zeolite ratio. This reevaluation and redesign resulted in exactly the performance standards we had set for the interground cement.

2.4 Commercial Production

In keeping with the Concept Goal of "multiple applications from a single intergrind" Trabits Group applied for and was issued a Trademark for "FlexCem" by the U.S. Patent and Trademark Office. Now with a method, formulation and a product name the cement was ready for commercial production.

What FlexCem® is:

FlexCem® LVD is a lightweight variable-density well cement with a density range of 11.5 to 14.3 ppg. It is manufactured using patented technology in which cement clinker and proprietary zeolite are interground in specific ratios to maximize set-cement properties. FlexCem® LVD can be used in all types of cement applications, as a lead or tail slurry, and it is compatible with most currently-used cement additives.

The first commercial production was conducted at a cement plant in South Dakota and targeted the Bakken field in North Dakota. FlexCem® was used as lead cement for intermediate completions in the Bakken. Table 1 shows Bakken well conditions and slurry design. A total of fifteen wells were cemented with no problems and good results reported by the service company.

Table 1: Bakken well conditions and FlexCem® slurry design

Date	10/21/2017
Fluid Type	Lead
Job Type	Intermediate
TMD	11211 ft
TVD	10904 ft
BHCT	220^{o}F
BHST	245 ^{o}F
Temperature Gradient	1.51 ^{o}F/100ft
Surface Temperature	80 ^{o}F
Density	11.50 lb/gal
Yield	2.42 ft^{3}/sk
Water Requirement	14.20 gal/sk
Sack Weight	85.00 lbs
Fluid SG	1.38
Service District	Dickinson

Although successful in the Bakken, commercial production of FlexCem® was moved to a cement plant in Oklahoma and a cement plant in Texas. The market in the Bakken is limited while markets in what is referred to as "MidCon" and the Permian Basin are much larger. In these markets FlexCem® is being used as a lightweight lead cement at 11.0 ppg and 11.5 ppg for most applications.

2.5 Quality and Repeatability

Cement plants take great care to ensure that cements manufactured are the same from one batch to another and meet specific quality parameters. Quality is also paramount in the manufacture of FlexCem® with a few added conditions. For example, in the finish mill cement plants use grinding aids to improve mill efficiency. Because certain grinding aids interfere with additives FlexCem® is interground without the use of grinding aids. The intergrind also has to be repeatable, not just from one batch to another in the same plant, but also repeatable at other plants. The logic here is geographic flexibility and taking advantage of the readily available Type I/II clinker which is made at most cement plants.

The following two Tables illustrate Repeatability of the same intergrind design at four different cement plants.

Table 2: Chemistry comparison for four different cement plants

	Plant A	Plant B	Plant C	Plant D
SiO2	35.42	36.51	36.16	34.34
Al2O3	6.25	6.50	6.56	6.09
Fe2O3	3.33	2.53	2.47	2.76
CaO	44.87	44.26	48.55	46.68
MgO	1.92	1.59	1.26	1.07
SO3	2.81	3.04	2.94	2.71
Na2O	0.43	0.69	0.58	0.58
K2O	1.90	1.71	1.35	1.60
Free CaO	0.630			
LOI	3.97	3.93		4.2
Alk. Eqv.	1.67	1.81	1.46	1.63
S. G. (C188)	2.715	2.740		2.763
Blaine	7420	7598	7140	7270

In Table 2 above, the chemistry is about the same but Blaine is lower for Plants C and D. As shown in Table 3 below the effect of lower Blaine can be seen in final cement performance where lower 24 hour compressive strength appears to directly correlate with lower Blaine.

Table 3: Blaine relationship to 24 hour compressive strength

	Plant A 13 lb/gal	Plant B 13 lb/gal	Plant C 13 lb/gal	Plant D 13 lb/gal
Initial Viscosity	9	17	31	3
Time to 30 Bc	66	79	0	87
Time to 70 Bc	94	95	91	106
Time to 100 Bc	104	105	104	116
Free Fluid (ml)	0	0	0	0
S.G.	2.71	2.76	2.85	2.85
24 HR	1720	1997	1209	1529
Viscometer Readings				
300	76	88	83	87
200	68	81	77	81
100	58	76	68	72
6	29	23	25	26
3	19	18	17	20

3.0 Special Attributes for Geothermal Wells

Brookhaven National Laboratory completed research on the ability of cements to self-heal. One of the cement formulations tested was FlexCem®.

Ability of cements to self-heal attracted significant attention in the recent years (Huang *et al*., 2013; Huang *et al*., 2014; Talaiekhozan *et al*., 2014; Sahmaran *et al*., 2008; Termkhajornkit *et al*, 2009; Sahmaran *et al*., 2013; Qian *et al*., 2010). The idea of cements that can recover their properties after a damage is of great interest for geothermal cements which are subjected to continuous thermal, mechanical and chemical stresses throughout the life of wells.

Among the most common strategies applicable for high-temperatures of geothermal wells is addition of slow-reacting cementitious materials that contribute to the development of mechanical properties at later times when exposed to well fluids through cracks and fractures in damaged cements. Such materials can be economical, slow reacting pozzolans, including slag, fly ash, silica in different forms, zeolites etc. Another self-healing effect occurs when reactions between environmental and cement constituents form new phases favorable for recovering cement properties. This, for example, includes initial carbonation of cements with formation of dense calcium carbonate or formation of calcium-magnesium-silicate minerals that, generally, noticeably contribute to strength recoveries (Schlangen, 2010). At longer curing periods under hydrothermal conditions phase transitions may play an important role in cement healing properties. A recent study of Roman cement structures in sea water demonstrated that the cement formulated with pozzolanic volcanic ash successfully sealed cracks formed through its long life. The healing was a combined effect of slow pozzolanic reactions and favorable phase transitions (Jackson *et al*., 2014).

Although advantages of using pozzolanic materials in cementitious blends are widely recognized, their use in geothermal wells is limited. Fly ash is a part of Halliburton's ThermaLock® cement that was specifically developed for applications in geothermal wells with high carbon dioxide concentrations (Sugama, 2006). However, it has some drawbacks associated with the presence of calcium-aluminate cement in its composition. Among the problems of using calcium-aluminate-based cementitious blends are difficult control of their set and incompatibility with Portland cement commonly used for subterranean cementing jobs, which requires a separate mixing and pumping equipment for calcium-aluminate blends and complicates logistics.

The lightweight variable density well cement developed by Trabits Group with the Department of Energy Award DE-EE0002785 and trademarked as "FlexCem®" (FlexCem) based on Portland cement chemistry and including a pozzolanic zeolite, ferrierite, in its composition is of particular interest. As described above, Trabits Group modified the high temperature geothermal cement formulation for use as a lightweight variable density cement targeting Bakken and Permian shale development and other completions where depth or fragile formations require a lightweight cement. The variable density aspect allows FlexCem to be used as lead and tail cement eliminating multiple blend requirements at the wellhead.

The intergrind production of FlexCem gives a bimodal particle size distribution between the harder clinker and the softer zeolite resulting in increased strength properties. The replacement of clinker with zeolite results in a final cement product that has 30% less greenhouse gas emissions compared with Portland cement. Since FlexCem is of interest both for geothermal and oil and gas wells its self-healing performance was evaluated at high (300^{o}C) and moderate (100^{o}C) temperatures.

3.1 Self-healing properties

We reported in our previous work that FlexCem demonstrated outstanding strength recoveries after repeated compressive damage and short healing periods of 5 days at 300^{o}C (Pyatina, Sugama, Trabits, & Jordan, 2018). A combination of OPC with another zeolite CLIN was tested in that work and the recoveries of that blend in water at 300oC were significantly lower than for the intergrind FlexCem samples. Figure 1 shows compressive strength of tested formulations after repeated damage in water or carbonate. Zeolite-modified cements had a lower initial compressive strength than OPC with silica flour. All the formulations recovered some compressive strength both after the fist and the second crush tests and additional 5-day curing at 270-300^{o}C so that their final strength was more than 1000 psi except for OPC/CLIN one cured in water (760 psi). Surprisingly, the strength of FlexCem cured in water increased after the two crush tests and repeated healing of 5 days and increased after the first crush test before decreasing after the second crush test when healed in carbonate. The strength of OPC/SiO_2 and OPC/CLIN in water decreased consecutively in two crush tests with two healing periods. The strength of OPC/CLIN formulation cured in carbonate stayed around 2000 psi throughout the testing. FlexCem demonstrated very high recoveries of >160% (1st break) and >130% (2nd break); its recovery in carbonate was 170% (1st break) but only 70% after the 2nd break. OPC/SiO_2 recoveries stayed constant between ~60 and 65% in two damage tests and two healing periods (Figure 1). The recoveries of OPC/CLIN were around 100% after both crush tests when cured in carbonate but only 80/56% after the first and the second crush tests followed by healing in water. Low strength recoveries were generally associated with the formation of large, long cracks. Young's modulus mirrored the compressive strength behavior with especially conspicuous increase in it for FlexCem cured in water (Figures 2).

Second-time damage recoveries of FlexCem in carbonate were improved by adding silica flour to FlexCem (Pyatina et al., 2018). For silica-modified FlexCem second-time recoveries after the second damage increased to 111% in carbonate environment (Figure 2).

Since many geothermal and oil and gas wells are at lower than 300^{o}C temperatures it was of interest to evaluate self-healing performance of FlexCem at 100^{o}C. Since the major factor of successful healing observed at 300^{o}C was alkali decomposition of FER at this high temperature it was questionable whether the FER will be effective at lower temperature of 100^{o}C.

Figure 4 shows compressive strengths before and after 5-day healing for 300^{o}C cured and healed sample and 8 or 16 days of healing for 100^{o}C cured and healed samples. The original compressive strength of the samples cured for a day at 100^{o}C was noticeably, nearly 3-times, higher than for the samples cured at 300^{o}C. This is likely the result of formation of different hydrates at the two temperatures. Lower-temperature stable amorphous and crystalline hydrates, generally, have a higher silica-to-calcium ratio than high-temperature hydrates (e.g. tobermorite vs. xonotlite) involve more water in their structure and form a denser, more compact matrix that results in higher compressive strength. High-strength samples are usually less flexible and fail forming brittle difficult to repair fractures under stresses. However, after 8 and 16 days of healing the 100^{o}C-cured and healed samples recovered their strength. Although the strength recovery was lower than that at 300°C because of the very high initial strength of the samples it was still above 100%, specifically 120% after 8 days of healing and 122% after 16 days of healing. Based on these data it is likely that the healing was completed in a short time and additional curing did not significantly change the composition of the samples so that the strength recoveries remained similar after 8 and 16 days (Figure 4).

Previous work on high-temperature performance of OPC/SiO_2 blend demonstrated significantly lower strength recoveries (60% after the first break in water at 300^{o}C). Several factors may contribute to the good self-recovery of the FlexCem. At a moderate temperature of 100^{o}C cement hydration could be only partial after a day of the initial curing. So continuous cement hydration was likely a strong contributor to the good recovery rate. Another factor that differ FlexCem from the regular OPC formulations is the presence of FER, that decomposes under alkali environment of hydrating cement, contributing ions for formation of new phases. Although FER decomposition at 100^{o}C should be slower than at 300^{o}C it still may contribute to the formation of new phases and improve recovery rates.

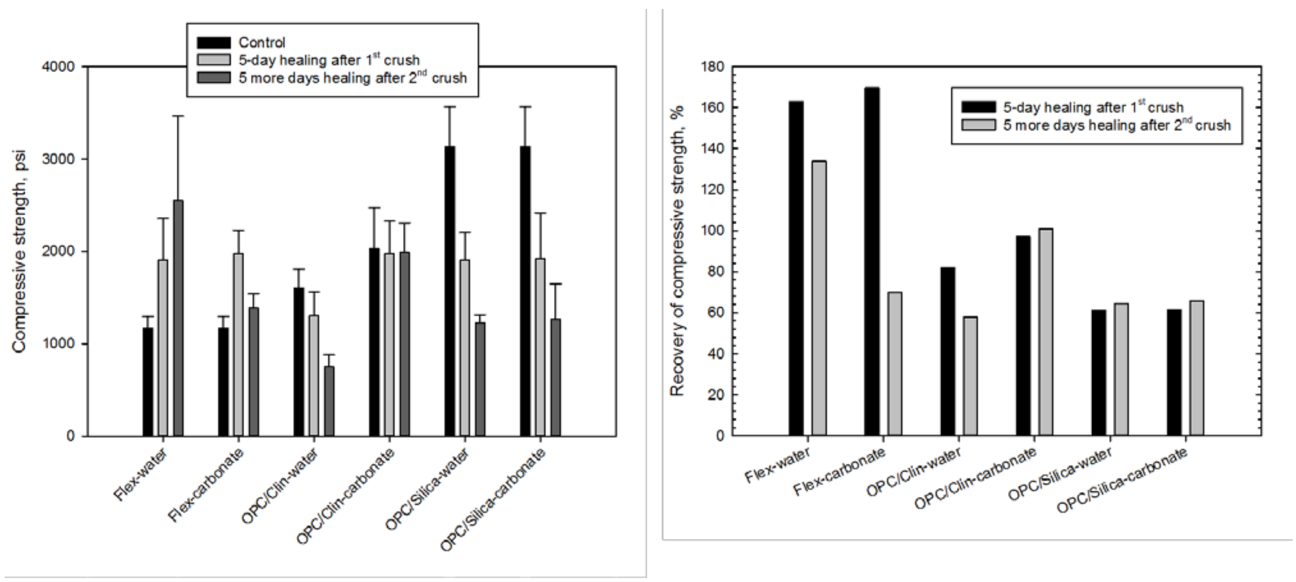

Figure 1: Compressive strength and percent its recovery in water or carbonate at 270-300^{o}C after repeated damage.

3.2 Acid resistance and acid strength recoveries

Geothermal wells are often highly acidic through H_2S dissolution with formation of sulfuric acid. Cements modified with pozzolanic materials are generally more acid resistance due to the lower calcium, particularly sensitive to acid attack, and higher aluminum and silicon contents. The data of OPC/SiO_2 and FlexCem exposure to sulfuric acid of pH 0.2 for 28 days at 90^{o}C summarized in Table 4 clearly support this demonstrating a superior performance of FlexCem. The appearance of the samples after the first 14 days of acid exposure and after a crush test and 14 more days of acid exposure is given in Figure 5.

After the first 14 days of exposure to a strong acid FlexCem lost only 9% of its strength compared against 37% loss for the OPC/SiO_2 blend and 7% of Young's modulus vs. 31% for OPC/SiO_2. There was also significantly smaller increase in samples diameter and weight indicative of scale deposition from acid reactions with cement (2.4% diameter and 12% weight increase vs. 10 and 17% increases for OPC/SiO_2 respectively). Acidification of cements accelerated after the compressive damage to the point that OPC/SiO_2 samples could not be tested anymore (see Figure 4b). FlexCem survived additional 14 days of strong-acid exposure with a residual compressive strength of 960 psi. In summary, clinker modification with FER improved its resistance to strong acid at elevated temperature.

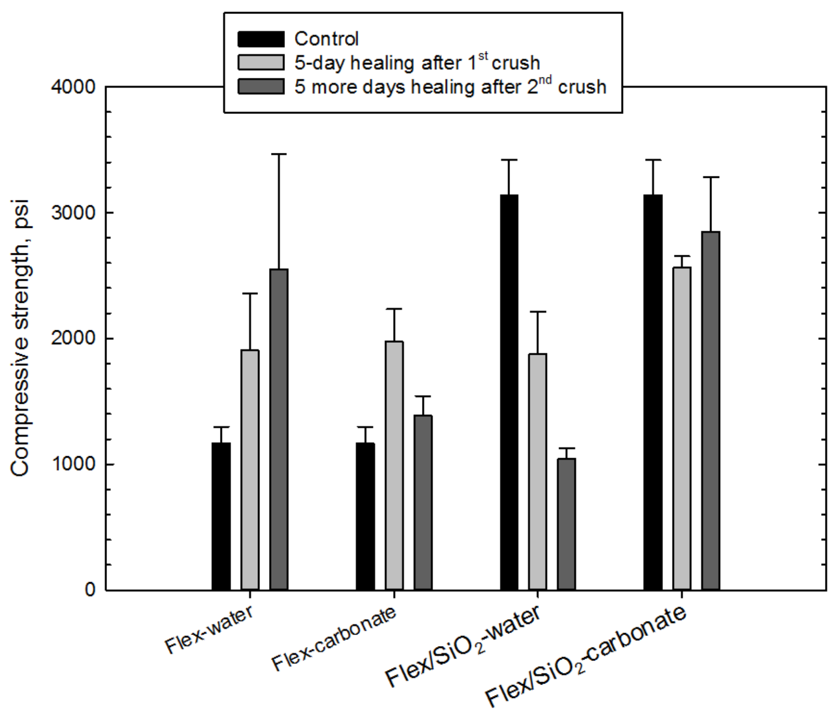

Figure 2: Comparison of compressive strength recovery for modified and non-modified FlexCem in water or carbonate at 270°C after repeated damage.

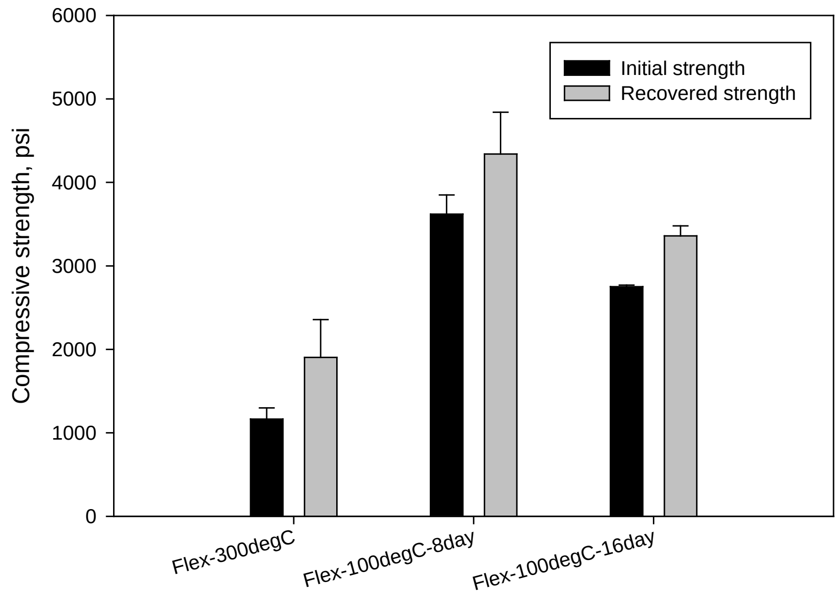

Figure 3: Comparison of compressive strength before and after healing periods of 5 days (300^{o}C), 8 days (100oC or 16 days (100^{o}C) for 1-day FlexCem samples cured at 300^{o}C or 100^{o}C respectively.

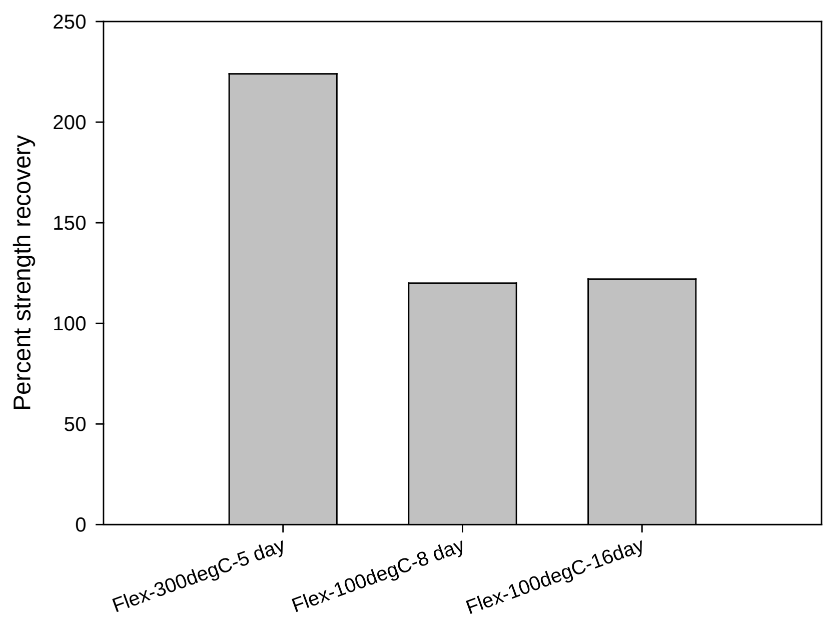

Figure 4: Strength recoveries for FlexCem samples after compressive damage and 5 days (300^{o}C), 8 days (100^{o}C) or 16 days (100^{o}C) of healing under the original curing conditions.

Table 4: Changes in mechanical properties, weight, and diameter after exposure for 14 days in 90^{o}C-pH 0.2-H_2SO_4 and after compressive strength test and 14 more days in the same acid for OPC/SiO_2 and FlexCem

Formulation Conditions	**Compressive strength, psi**	**Youngs' modulus, psi**	**Changes in diameter, %**	**Changes in weight, %**
		OPC/SiO_2		
Control	3140±640	220700±83300	N/A	N/A
After 14 days in acid	2130±560 (-37%)	152000±39000 (-31%)	+10	+17
After break and 14 more days in acid	N/A	N/A	N/A	+7.2
		FlexCem		
Control	2670±60	232000±31450	N/A	N/A
After 14 days in acid	2440±250 (-9%)	216000±4900 (-7%)	+2.4	+12
After break and 14 more days in acid	960±160 (-60%)	95800±6100 (-56%)	N/A	+1.4

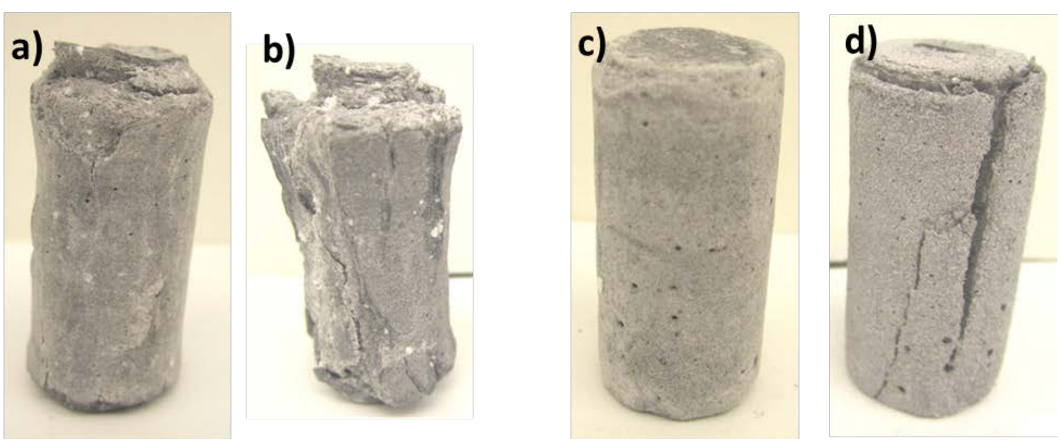

Figure 5: Sulfuric acid-treated samples (pH 0.2, 90^{o}C): a) OPC/SiO_2 after 14 days in the acid; b) OPC/SiO_2 after 14 days in the acid, break and 14 more days in the acid; c) FlexCem after 14 days in the acid; d) FlexCem after 14 days in the acid, break and 14 more days in the acid.

4.0 Conclusions

Research to develop an improved cement for geothermal wells evolved into a lightweight, variable density cement that is useful for oil and gas completions particularly where fragile formations or hydrostatic conditions require a low density cement. Formulation of the new cement does not exclude geothermal application but rather enhances such use by providing self-healing to ensure cement competence and acid resistance to geothermal environments.

The work completed by the Brookhaven study demonstrated that tested zeolites (ferrierite and clinoptilolite) decompose in blends with Portland cement or clinker at elevated temperatures contributing reactants, such as silicon, aluminum, iron, magnesium (FER) for formation of new phases. Under the tests conditions the decomposition-reaction of zeolites starts later than cement hydration resulting in lower initial compressive strength than in non-modified cement; however, the strength builds up at longer curing. Additionally, decomposition of tested zeolites, resulting in increased concentrations of silicon and aluminum in interstitial water, stabilized tobermorite at temperatures where it is usually converted to xonotlite with accompanying strength loss. Tobermorite stabilization was favorable for samples mechanical properties and acid resistance. In addition to silicon and aluminum ferrierite contributed iron and magnesium that formed such cement strengthening phases as andradite, magnesium silicates (in the matrix) and talc (at the surface).

FlexCem demonstrated self-healing ability not only at high 300^{o}C hydrothermal temperature but also at moderate, 100^{o}C temperature, recovering 120% of its strength after a short 8-day curing. FlexCem clearly outperformed OPC blend with silica in acid-resistance tests surviving 28 days at pH 0.2 and 90^{o}C. This excellent performance of FlexCem may be explained by the decreased calcium content and pozzolanic reactions of ferrierite.

5.0 Acknowledgements

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