Ultra-Lightweight Cement

Third Quarterly Technical Progress Report

April 1 to June 30, 2001

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Issued July 18, 2001

DOE Award Number
DE-FC26-00NT40919, Tasks 1, 2, 3, and 8

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Abstract

The objective of this project is to develop an improved ultra-lightweight cement using ultra-lightweight hollow glass spheres (ULHS). Work reported herein addresses Task 1: Assess Ultra-Lightweight Cementing Issues, Task 2: Review Russian Ultra-Lightweight Cement Literature, Task 3: Test Ultra-Lightweight Cements, and Task 8: Develop Field ULHS Cement Blending and Mixing Techniques. Results reported this quarter include:

- preliminary findings from a literature review focusing on problems associated with ultra-lightweight cements
- summary of pertinent information from Russian ultra-lightweight cement literature review
- laboratory tests comparing ULHS slurries to foamed slurries and sodium silicate slurries for two different applications
- initial laboratory studies with ULHS in preparation for a field job

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Introduction

Oilwell cementing involves placing a pumpable slurry of Portland cement, additives, and water into a wellbore. The slurry is pumped into the annular space between the borehole and a steel pipe, called a casing, intended to produce a conduit from the reservoir to the surface. The cement sets in place to support the casing in the hole, to isolate various formations from one another, and to control fluid movement within the well.

Typical cement fluid density ranges from 14 to 17 lb/gal. Certain conditions can be encountered during the well construction process that necessitate application of cements with much lower density. Lower density is required to limit hydrostatic pressure exerted on formations through which the wellbore passes in order to prevent the formation from fracturing and imbibing the well fluid. This phenomenon, named lost circulation, increases time to drill and complete the well and increases construction cost due to expensive remedial treatments. Most common sections of a well in which lost circulation occurs are the upper sections: surface casings and intermediate casings. Since formations covered by these casings are relatively close to the Earth's surface, application temperatures for these low-density cements are relatively low.

The minimum practical density achievable with conventional cements and additives is roughly 11 lb/gal. At this density, the stability of the slurry and strength of the set cement are only marginally acceptable. The primary density-reducing material in these conventional cements is water. Additional water dilutes the cement, causing low strength. Lower temperature further delays strength development. Achieving density requirements lower than this or strength requirements greater than minimum necessitate use of ultra-lightweight materials mixed into the slurry.

Ultra-lightweight hollow spheres (ULHS) are excellent as candidate material for producing ultra-lightweight cements. These small, hollow, glass beads effectively encapsulate air in the slurry, thereby lowering the slurry density significantly compared to the addition of water to the slurry.

This project is designed to develop cementing systems using ULHS. The development will be achieved through a carefully designed program of modeling, design, laboratory testing, and field testing.

The phase of the project documented in this report involves review of previous investigations conducted in Russia; review of literature about potential alkali-silica reactivity; laboratory testing of cements made with ULHS, foamed cements, and cements made with sodium silicate; and preliminary laboratory testing in preparation for field testing.

Executive Summary

The third quarter of this investigation focused on laboratory testing that compared slurries containing ULHS to two other lightweight cement systems: foamed and sodium silicate slurries. In addition, literature reviews also revealed pertinent information about lightweight cement systems and some of their potential problems. Further studies examined ULHS characteristics and performance.

A review of literature dealing with potential problems of lightweight cement systems revealed a phenomenon known as alkali-silica reactivity (ASR). This is a potential problem since silica is a large component of ULHS. Reaction mechanisms, prevention techniques, and test methods for ASR are presented.

A review of Russian literature reveals some of the lightweight cement systems that are used in Russia and its neighboring countries. The review presents some performance characteristics that are common to some of the lightweight hollow spheres used in that area. In addition, the review offers information on ASR and some prevention methods.

Because of the large volume of cement required for this project, a quality-control process has been implemented. Information and data gathered for the program are presented.

Further testing evaluated the performance of ULHS. Studies investigated the effect of shear on the breakage of ULHS and the resulting change in density. A simple series of tests was also performed to confirm density measurements of slurries made with ULHS.

To test the performance of slurries made with ULHS and compare them with two other lightweight cement systems (foamed and sodium silicate slurries), the project will employ a battery of tests to simulate three cementing applications:

- deepwater conductor pipe
- land-based surface pipe
- land-based intermediate casing

This phase of the project tested six slurries on two of the three cementing scenarios/applications. The six different slurries are the three lightweight cement systems (ULHS, foamed, and sodium silicate) prepared using two different cement types (Class A and TXI Lightweight). Preliminary data indicates that the slurries made with ULHS performed as well as or better than the sodium silicate and foamed slurries based on performance criteria established thus far. Mechanical testing in the next quarter will help establish performance qualities of cement slurries made with ULHS.

Preliminary laboratory data has also been gathered in preparation for a field test that is tentatively scheduled for July.

Literature Review

Task 1 of this project is to Assess Ultra-Lightweight Cementing Issues. A portion of that task includes a literature review of lightweight cement systems. The literature review also covers the potential alkali-silica reaction that could occur with ULHS.

Siliceous admixtures combined with cement create a potential for a phenomenon known as alkali-silica reactivity (ASR). Whether this detrimental reaction occurs depends on several factors including cement chemistry and available moisture.

An aqueous liquid pore solution is present in all cements. This pore solution is alkaline (pH typically greater than 12.5) and can contain calcium cations (Ca^{++}) and alkali cations (Na^+ , K^+). The hydroxyl ions in the alkaline pore solution chemically attack silica (SiO_2) creating SiO^- species that are susceptible to reaction with the cations (Ca^{++} , Na^+ , and K^+). If large concentrations of calcium cations are in the pore solution, a calcium silicate hydrate (C-S-H) gel is formed. C-S-H gel is considered one of the safe reactions that can occur; it is not prone to swelling.¹

When the alkali concentration in pore solutions is high as compared to the calcium ion concentration, a deleterious high alkali-silica gel will be formed. The high-alkali gel has a greater capacity to swell with absorption of moisture. This swelling can lead to detrimental cracking of the cement. As might be expected, if available moisture remains low (below 80% relative humidity referenced to 70 to 75°F), there will not be enough moisture available for detrimental swelling.²

There are several methods available to limit or avoid this detrimental expansion due to ASR. One of the ways to avoid this detrimental reaction is to avoid using siliceous materials that are known to be reactive. Decreasing the alkali level of the cement (less than 0.60% equivalent Na_2O) can also limit the detrimental ASR gel formation. Another method includes the addition of specific admixtures. These admixtures impede normal ASR by reducing the steady state concentration of alkali and hydroxyl ions in concrete, or by altering diffusion rates of alkali and lime to reactions sites. Common admixtures that are used to limit ASR are fly ash, blast-furnace slag, and even silica fume.²

Because of the siliceous components of ULHS, it is important to determine if ULHS leads to ASR. There are several ASTM methods that aim to identify reactive materials that lead to ASR in cements²:

1. ASTM C 227 Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar Bar Method)
2. ASTM C 289 Test Method for Potential Reactivity of Aggregates (Chemical Method)
3. ASTM C 295 Guide for Petrographic Examination of Aggregates for Concrete

There are pros and cons to each method. As suggested by their titles, these methods are used to identify potentially reactive aggregates that are generally used in construction cement. Methods C 227 and C 289 are effective in identifying rapidly reactive materials,

but fail to identify slowly reactive materials. ASTM C 295 is a rapid way to identify potentially reactive aggregates, but is highly subjective and dependent on the capability of the petrographer examining the aggregate.²

If ULHS is affected by ASR, there is a possibility that the glass wall of the ULHS will be chemically attacked. This could in turn increase the permeability of cements made with ULHS. Hence, long-term permeability testing could reveal if ASR negatively affects cements made with ULHS.

The project steering committee agreed to test for ASR by testing ULHS as outlined by ASTM C 289 as well as testing for long-term permeability of cements made with ULHS.

Review of Russian Lightweight Cement Literature

Task 2 of this project is to Review Russian Lightweight Cement Literature. The first quarterly progress report for this project contained an appendix with a report of the Russian literature review conducted by Dr. Vladimir Sledkov. This review reveals that there are several types of hollow microspheres used in Russia. A group of microspheres with the trade name Plaminol is made from phenol-formaldehyde and carbamide formaldehyde tars and filled with nitrogen. These Plaminol microspheres are generally expensive and have limited hydrostatic collapse strength (less than 2,900 psi).^{3,4}

Most of the hollow glass microspheres from Russia have low hydrostatic collapse strengths (less than 1,500 psi). However, there are Russian companies that produce aluminum sodium borosilicate glass hollow microspheres with collapse strengths of 1,450 to 2,900 psi. The collapse strength of 2,900 psi is still below most of the ULHS under study in this project.

This literature review also points out that sodium glass is susceptible to attack by alkaline medium. To counteract the effects of lime, it is recommended that materials such as pozzolan or slag be added to the cement slurry.⁵ Protective materials, such as boron and aluminum silicates, are effective in boosting the chemical resistance of glass microspheres. Some of the products from initial reaction with these materials help to create a protective barrier that slows silicate glass destruction.⁶ Boron and aluminum silicate glass microspheres generally have hydrostatic collapse strengths in excess of 4,000 psi.

Cement Quality-Control Program

An extensive quality-control program was initiated because of the large quantity of cement used over the course of this project. Each bucket of cement is labeled with a materials log number and date upon receipt. When a bucket is first opened for use, the date of opening is recorded in the materials log. This log number will be referenced on the laboratory sheets for each test performed. Where applicable, tests according to API Specification 10A are conducted. Additionally, several other tests tailored specifically for the test conditions and materials (rheology and low-temperature compressive-strength development) are included in this QC Program.

The Class A and Class H cement performance requirements are presented in API Specification 10A. Because the Lightweight Oilwell cement is not an API cement, it is tested according to QC procedures developed by the manufacturer.

Initially, the testing lab received five 5-gallon buckets of API Class A cement (analogous to ASTM Type I cement), ten 5-gallon buckets of API Class H cement, and nineteen 3-gallon buckets of Lightweight Oilwell cement from TXI. Both API Specification tests and tests recommended by advisory board members are being performed for the cement quality-control program. The physical requirements used for testing each of the cements are listed in Table 1. To accelerate the rate of compressive-strength development at low temperatures, calcium chloride (CaCl_2) is being used with the cements according to

Table 2. Calcium chloride was selected because it is one of the most effective and commonly used cement accelerators.

Table 1—Cement Slurry Compositions for Quality-Control Testing Program

Cement	Mix Water (%)	Density (lb/gal)	Cement (g)	Water (mL)	Test
Class A	46	15.6	772	355	API
TXI LW	75	13.2	541	406	TT and CS
TXI LW	105	12.1	426	447	FW
Class H	38	16.4	860	327	API

Table 2—Percent CaCl_2 for Low-Temperature Compressive Strengths

Temperature (°F)	CaCl_2 (%)
80	0.0
60	1.0
45	2.0

All of these tests have been run to provide a baseline for each type of cement. This QC data will provide a comparison when examining other data for this project. The complete set of tests will be conducted periodically throughout the DOE project.

Appendix A shows the data gathered for the quality-control program through the third project quarter. These data track well with performance data that were provided by the vendors and presented in the previous report. The test results exceeded the required specifications, and this process will be repeated when new cement supplies are received. Data will also be presented in the appendices of future reports.

Tests of Lightweight Cements

Task 3 of the project encompasses testing of ultra-lightweight cement slurries made with ULHS. In the second quarterly progress report, the specific gravity of the different kinds

of ULHS were investigated. One part of the investigation studied the specific gravity of the beads at different applied pressures.

The preliminary comparisons found that the 5K beads (supplied in November, 2000) did not perform as well as expected. In the third project quarter, a new sample of 5K beads was tested; these tests showed similar performance results. The 5K bead sample sent to the principal investigator in November, 2000 was typical production material. The bead manufacturer analyzed that specific lot for survival strength and found it to be a statistical normal lot and to fall within a normal distribution curve, albeit slightly below the targeted mean. The strength testing that is performed by the bead manufacturer is very similar to the way that the principal investigator tests the slurries with the beads at different applied pressures. The results observed with the 5K beads may be related to the lower-than-average strength of the specific lot tested. The bead manufacturer plans to submit to the principal investigator a new lot of 5K beads for re-evaluation in lightweight cement slurries. The bead manufacturer anticipates that the new lot will fall in line with the expected strengths of the 4K and 6K beads.

Additional studies have been performed on the ULHS slurries to determine the effect of blending shear on bead breakage. One way to analyze possible breakage is by monitoring slurry density changes after shearing ULHS slurries. The slurries for these shear effect studies were first prepared as follows.

1. Weigh out the appropriate amounts of the cement sample and additives, water, and ULHS into separate containers.
2. Mix the cement slurry according to Section 5.3.5 of API RP 10B.⁸
3. Pour the slurry into a metal mixing bowl and slowly add ULHS while continuously mixing by hand with a spatula. Mix thoroughly.

The density of this (unsheared) slurry is then measured with a pressurized mud balance and recorded. This same slurry is then placed in a Waring blender and mixed at 4,000 RPM for 35 seconds. The density of the slurry is again measured and recorded. The slurry is again transferred to the Waring blender and mixed at 4,000 RPM for an additional 15 seconds (50 seconds total). The density of the slurry is again measured and recorded. Table 3 shows the results from these experiments.

Table 3—Effect of Shear on Slurry Density

Measurements for 10.0 lb/gal Slurry Design				
	ULHS Type			
	3K	4K	5K	6K
ULHS Concentration (%bwoc)	20.89	21.71	21.71	29.13
Water Concentration (gal/sk)	8.5	8.5	8.5	8.5
Density Before Shear (lb/gal)	10.0 [†]	10.0	10.0	10.0
Density After 35 Seconds of Shear (lb/gal)	10.3 [†]	10.2	10.5	10.1
Density After 50 Seconds of Shear (lb/gal)	10.4 [†]	10.3	10.5	10.1
Measurements for 11.0 lb/gal Slurry Design				
	ULHS Type			
	3K	4K	5K	6K
ULHS Concentration (%bwoc)	13.24	13.74	13.74	18.18
Water Concentration (gal/sk)	8.5	8.5	8.5	8.5
Density Before Shear (lb/gal)	11.0 [†]	11.0	11.0	11.0
Density After 35 Seconds of Shear (lb/gal)	11.3	11.2	11.4	11.0
Density After 50 Seconds of Shear (lb/gal)	11.4	11.3	11.4	11.1

[†]Density confirmed with three samples.

As evidenced by an increase in slurry density, the 3K, 4K, and 5K beads were distinctly damaged by the shear. The 6K beads suffered the least from shearing.

The cross (†) in Table 3 marks four slurries for which the density was checked three times. Of these slurries, the slurry was divided into three different samples and the density for each sample was measured with a pressurized mud balance. For each slurry, the measured density for all three samples was the same. These additional density measurements were performed to confirm that the density measurements are repeatable.

The shear effects studied to this point have focused on laboratory mixing procedures. Future work will investigate the effect that field mixing equipment and procedures have on bead breakage.

Application Scenarios

One way to gauge the performance of cements made with ULHS is to compare them to other ultra-lightweight cements. Members of the project steering committee decided to compare ULHS cements with a foamed cement and with a conventional sodium silicate slurry.

Three different application scenarios were chosen to test the performance of the lightweight cements. Application 1 simulates deepwater cementing. Application 2 simulates a typical land-based surface pipe cementing job. Application 3 simulates a typical land-based cementing job for intermediate casing.

Table 4 shows the pressure and temperatures associated with the three different applications. The conditions for Application 1 (deepwater cementing) came from typical applications as experienced by the principal investigator. The values for the surface pipe and intermediate casing applications (2 and 3) came from average values attained from *Worldwide Cementing Practices*⁷ as detailed in the first quarterly progress report. For the three different applications, the slurries were designed to try to achieve certain performance standards (although they could not always be achieved). These standards are also listed in Table 4.

Table 4—Simulation Conditions for Comparison of Lightweight Slurries

Application	BHCT (°F)	BHST (°F)	Pressure (psi)	Desired Thickening Time	Desired Free Fluid	Desired Fluid Loss
1	65	45	5,300	4 to 6 hours	<1%	NA
2	78	96	1,000	4 to 6 hours	<1%	NA
3	128	174	5,000	4 to 6 hours	<0.5%	250cc/30min

Of the different classes of ULHS being studied in this project, the 6K beads were chosen because of their ability to withstand the hydrostatic pressures associated with the applications.

Most of the tests were performed as specified in API RP 10B⁸. Some preparation and testing methods were modified to adapt for the ULHS and foamed slurries. The mixing procedures were modified for the ULHS slurries to minimize bead breakage that can occur due to high shear from API blending procedures. The following blending procedure was used for the ULHS slurries.

1. Weigh out the appropriate amounts of the cement sample and additives, water, and ULHS into separate containers.
2. Mix the cement slurry according to Section 5.3.5 of API RP 10B.⁸
3. Pour the slurry into a metal mixing bowl and slowly add ULHS while continuously mixing by hand with a spatula. Mix thoroughly.
4. Pour this slurry back into the Waring blender and mix at 4,000 RPM for 35 seconds to mix and evenly distribute the contents.

Testing methods for foamed slurries were also modified. For example, thickening time is performed on unfoamed slurries only. Because the air in the foam will not affect the thickening rate, the slurry is prepared as usual per API RP 10B⁸ and then the foaming surfactants are mixed into the slurry by hand without foaming the slurry. This and the other non-standard procedures for testing foamed cement are presented in Appendix B.

Stability of foamed slurries has recently been getting more attention and discussion. The foamed slurries in these laboratory tests appear thus far to be durable and stable. After the slurries were mixed and foamed in the blender, the foam did not expand while opening

the container; many people in the industry have experienced foam expansion once the container is opened. The densities of the foamed slurries were measured using a volumetric container and a scale and were found to be within 0.1 lb/gal of the designed foamed density.

Also, cubes that have were cured for compressive strength testing showed no visual volumetric change. The cubes were the same size after curing as they were when they were poured into the molds. Other indications of foamed cement stability will be examined and presented as the project progresses.

Table 5 presents the slurry compositions for Applications 1 and 2. Additives (slurry extenders, accelerators, and foaming agents) were added to the different slurries in order to adapt each slurry to the application's testing conditions as specified in Table 4. The additives are widely available, generic additives, and are not specific to any particular service company.

Table 6 presents rheology data for the slurry designs specified in Table 5. The data was taken using a Chandler Chan 35 direct indicating viscometer.

Table 5—Slurry Compositions for Applications 1 and 2

Application 1				
Cement Type	Slurry System	Measured Density (lb/gal)	Water Content (gal/sk)	Additive Concentrations
Class A	ULHS	11.7	8.00	2.0%bwoc CaCl ₂ ; 14.9%bwoc (6K) ULHS
	Foamed	15.6 [†] (11.5 [‡])	5.20	1.0%bwoc CaCl ₂ ; 0.03 gal/sk Witcolate; 0.01 gal/sk Aromox C-12
	Sodium Silicate	11.5	16.87	3.0%bwoc Sodium Silicate
TXI Lightweight	ULHS	11.5	6.65	2.0%bwoc CaCl ₂ ; 11.5%bwoc (6K) ULHS
	Foamed	13.1 [†] (11.4 [‡])	7.00	2.0%bwoc CaCl ₂ ; 0.03 gal/sk Witcolate; 0.01 gal/sk Aromox C-12
	Sodium Silicate	11.6	12.11	3.0%bwoc Sodium Silicate
Application 2				
Cement Type	Slurry System	Density (lb/gal)	Water Content (gal/sk)	Additive Concentrations
Class A	ULHS	11.5	7.09	16.2%bwoc (6K) ULHS
	Foamed	15.6 [†] (11.5 [‡])	5.20	0.03 gal/sk Witcolate; 0.02 gal/sk Aromox C-12
	Sodium Silicate	11.5	16.87	3.0%bwoc Sodium Silicate
TXI Lightweight	ULHS	11.5	6.50	2.0%bwoc CaCl ₂ ; 11.9%bwoc (6K) ULHS
	Foamed	13.0 [†] (11.5 [‡])	7.00	0.03 gal/sk Witcolate; 0.02 gal/sk Aromox C-12
	Sodium Silicate	11.5	12.11	3.0%bwoc Sodium Silicate

[†]Unfoamed slurry density

[‡]Foamed slurry density

Table 6—Rheology Data for Applications 1 and 2

Application 1											
Cement Type	Slurry System	Temperature (°F)	Dial Reading (RPM)								
			300	200	100	60	30	20	10	6	3
Class A	ULHS	80	59	48	24	29	25	23	19	14	10
		65	72	57	40	32	26	24	20	16	11
	Foamed†	80	96	82	65	56	49	45	33	23	15
		65	80	68	54	47	41	37	28	18	12
	Sodium Silicate	80	31	28	24	23	21	20	19	17	14
		65	31	27	23	21	19	19	18	17	11
TXI Lightweight	ULHS	80	92	80	65	58	50	46	33	25	17
		65	112	94	74	64	55	50	38	26	18
	Foamed†	80	40	35	29	26	23	22	19	16	11
		65	54	47	38	34	29	27	23	19	12
	Sodium Silicate	80	43	38	33	30	27	25	23	20	11
		65	40	36	31	28	26	23	22	20	12
Application 2											
Cement Type	Slurry System	Temperature (°F)	Dial Reading (RPM)								
			300	200	100	60	30	20	10	6	3
Class A	ULHS	78	107	87	64	53	40	33	20	14	9
		96	122	102	79	67	47	36	22	14	9
	Foamed†	78	80	68	53	47	39	36	24	16	9
		96	102	90	74	67	56	48	28	19	10
	Sodium Silicate	78	34	30	27	25	23	22	20	19	10
		96	29	26	23	21	19	18	17	13	8
TXI Lightweight	ULHS	78	115	98	78	69	58	52	35	26	17
		96	144	124	101	88	72	59	38	29	19
	Foamed†	78	42	37	30	26	23	21	19	16	9
		96	61	54	46	41	35	31	25	18	10
	Sodium Silicate	78	40	35	32	29	26	25	24	20	12
		96	45	39	33	30	27	25	23	18	12

[†]Rheology data from unfoamed slurry

Table 7 shows an overview comparison of the performances of each of the slurries in Applications 1 and 2. The thickening time for each slurry is recorded as the time that the consistency reached 70 Bc. Table 7 also presents the point of departure for the sodium silicate slurries. The point of departure indicates when the slurry consistency begins to noticeably increase. This is sometimes taken as the thickening time for highly extended slurries such as sodium silicate because the mechanical agitation of the thickening time test may “break” hydrates that are forming but which have low consistency (Bc) because of the large amount of mix water. The values for the crush compressive strengths in Table 7 are the averages of three cube samples that were crushed.

Table 7—Summary of Data for Applications 1 and 2

Application 1						
Cement Type	Slurry System	Thickening Time to 70Bc (Hr:Min)	Point of Departure (Hr:Min)	Percent Free Fluid	Crush Compressive Strength	
					24-Hour (psi)	7-Day (psi)
Class A	ULHS	7:00+	---	0.0	215	995
	Foamed	3:55*	---	0.6*	170	1,125
	Sodium Silicate	6:40+	4:40	1.1	Not Set	240
TXI Lightweight	ULHS	6:40	---	0.0	155	450
	Foamed	5:35*	---	0.4*	85	280
	Sodium Silicate	7:00+	4:35	0.4	65	175
Application 2						
Cement Type	Slurry System	Thickening Time to 70Bc (Hr:Min)	Point of Departure (Hr:Min)	Percent Free Fluid	Crush Compressive Strength	
					24-Hour (psi)	7-Day (psi)
Class A	ULHS	5:05	---	0.8	975	2140
	Foamed	5:29*	---	1.0*	780	1,180
	Sodium Silicate	5:30+	3:50	0.0	235	350
TXI Lightweight	ULHS	5:12	---	0.0	935	3,075
	Foamed	5:53*	---	0.2*	385	1615
	Sodium Silicate	7:00+	2:55	0.0	200	605

*Thickening time and free fluid tests performed on unfoamed slurry

+Thickening time tests with (+) were terminated at indicated time

Because of the low temperatures associated with Applications 1 and 2, calcium chloride (CaCl_2) was used with the foamed and ULHS slurries to accelerate hydration. Calcium chloride was not used with the sodium silicate slurries due to viscosity issues specific to this slurry. The only viable accelerators for sodium silicate slurries are company-specific and not widely available in a generic form.

Application 1. At low temperatures, thickening times for cements can be quite long. In Application 1 (see Table 7), the Class A cement with ULHS was still below 40 Bc when the thickening time test was taken off after seven hours, even though it contained CaCl_2 . The Class A cement with the foaming surfactants had a thickening time of 3 hours and 55 minutes. The Class A cement with sodium silicate was below 40 Bc when its test was taken off after 6 hours and 40 minutes, but it had a point of departure of 4 hours and 40 minutes. Even with the differences in thickening times for the ULHS and foamed slurries, the 24-hour and 7-day compressive strengths for the two slurries were comparable.

With the TXI Lightweight cement, the thickening time for the ULHS slurry was longer than that for the foamed slurry. However, the compressive strengths of the ULHS slurry were higher than those for the foamed slurry.

Application 2. In this application, the compressive strengths of the sodium silicate slurries were lower than those of the foamed or ULHS slurries. At this temperature (78°F), the thickening times for the foamed and ULHS slurries are relatively close. The compressive strengths for the ULHS slurry were above those for the other slurries.

Comparison of Compressive Strengths Using Ultrasonic and Crush Methods

In the previous quarterly technical report, a comparison study was presented of six slurries using 3K ULHS. The study compared compressive strengths determined using ultrasonic cement analyzer (UCA) with those using the traditional crush method. There were varying degrees of agreement between the UCA and crush values.

The two slurries that showed the least agreement between UCA and crush were tested again. Table 8 shows the results obtained from the previous project quarter along with the results from this quarter. The difference between the UCA and crush values are presented in Table 8 as a percent difference between the UCA and crush value while using the

crush value as the reference value. In other words, $\% \text{Difference} = \frac{(UCA - \text{crush})}{\text{crush}}$.

Table 8—Comparison of Ultrasonic and Crush Compressive Strengths

Project Quarter		2				3			
Cement Type		TXI LW		TXI LW		TXI LW		TXI LW	
3K ULHS (%bwoc)		27.6		6.1		27.6		6.1	
Density (lb/gal)		9.0		11.5		9.0		11.5	
		UCA	Crush	UCA	Crush	UCA	Crush	UCA	Crush
12 Hours		900	500	1,220	830	616	590	1,118	968
18 Hours		1,300	850	1,700	1,280	970	1,088	1,776	1,563
24 Hours		1,400	1,050	2,000	1,600	1,038	1,228	1,973	2,048
48 Hours		1,470	1,250	2,150	2,000	1,132	1,475	2,169	2,116
		Percent Difference=(UCA - Crush)/Crush							
% Difference	12 Hours	80%		47%		4%		15%	
	18 Hours	53%		33%		-11%		14%	
	24 Hours	33%		25%		-15%		-4%	
	48 Hours	18%		8%		-23%		3%	
	Average	46%		28%		-11%		7%	

(-) Indicates that the crush value is higher than the UCA value

Table 9 compares the values of Table 8 in a different way. Table 9 presents differences between readings from the two different project quarters. This is one way to look at the

repeatability of the tests. In Table 9 the percent difference is presented as the difference between second quarter and third quarter values divided by the third quarter value. The 2 and 3 used as subscripts indicate the project quarter that the value came from.

Table 9—Comparison of UCA and Crush Strengths by Quarter

Cement Type		TXI LW		TXI LW		TXI LW		TXI LW	
3K ULHS (%bwoc)		27.6		6.1		27.6		6.1	
Density (lb/gal)		9.0		11.5		9.0		11.5	
UCA or Crush		UCA	UCA	UCA	UCA	Crush	Crush	Crush	Crush
Project Quarter		2	3	2	3	2	3	2	3
12 Hours		900	616	1,220	1,118	500	590	830	968
18 Hours		1,300	970	1,700	1,776	850	1,088	1,280	1,563
24 Hours		1,400	1,038	2,000	1,973	1,050	1,228	1,600	2,048
48 Hours		1,470	1,132	2,150	2,169	1,250	1,475	2,000	2,116
% Difference =		$(UCA_2 - UCA_3) / UCA_3$				$(Crush_2 - Crush_3) / Crush_3$			
% Difference	12 Hours	46%		9%		-15%		-14%	
	18 Hours	34%		-4%		-22%		-18%	
	24 Hours	35%		1%		-14%		-22%	
	48 Hours	30%		-1%		-15%		-5%	
	Average	36%		1%		-17%		-15%	

(-) Indicates that the UCA or crush value from the 3rd quarter is higher than the UCA or crush value from the 2nd quarter.

In general, for the 48 hour time period examined in these studies the agreement between the UCA and crush values tends to improve with time and with strength development as seen in Table 8. The same general trend can be seen in Table 9 when comparing UCA strengths to each other or crush strengths to each other. The agreement of these compressive strength values is relatively close considering that such differences are not uncommon even when crushing three samples of the same slurry within the same test.

Preliminary Data for Field Job

Tasks 8 and 9 of this project focus on preparing and testing ULHS slurries for possible field applications. A potential candidate well has been identified in Webb County, Texas. The cementing application is for intermediate casing with a total depth of 6,100 feet, BHCT of 135°F, and BHST of 178°F. The cement that will be used is TXI Class A. The slurry design will also consist of 22.9% bwoc ULHS (4K), 0.6% bwoc proprietary retarder, and 8.5 gal/sk water for a design density of 10.0 lb/gal. Table 10 presents the preliminary laboratory tests that have been completed for this candidate well. The field job is scheduled for July.

Table 10—Preliminary Laboratory Data for Field Job

Slurry Density (lb/gal)					
Surface/Atmospheric: 9.9					
Pressurized at 3,200 psi: 10.1					
Rheology					
Fann Readings					
RPM	300	200	100	60	30
80°F	158	120	67	45	27
135°F	95	70	40	27	19
Thickening Time (Hr:Min)					
40 Bc		70 Bc		100 Bc	
2:56		3:06		3:29	
Free Fluid					
4.6 mL (1.8%) after 2 hr at 135°F					
Crush Compressive Strength (psi)					
Cure Temperature (°F)		80	127	178	
Strength - 24 Hours		310	490	945	
Strength - 48 Hours		1,075	1,550	1,630	
UCA Compressive Strength (psi)					
Cure Temperature (°F)		80	127	178	
Hours to 500 psi		18	7	6	
Strength - 24 Hours		730	1,490	1,630	
Strength - 48 Hours		1,260	1,970	1,740	

Conclusions

- Laboratory mixing procedures for 6K ULHS have been established and are acceptable.
- Field versus laboratory mixing will be verified with large-scale testing.
- Short-term laboratory testing shows mixable, pumpable slurries are possible. However, mechanical testing will help verify long-term viability.
- Testing for possible alkali-silica reactivity will be performed using tests outlined in ASTM C 289 as well as testing for long-term permeability of cements made with ULHS.
- Conventional testing data has been generated and acceptable test variabilities established.

List of Acronyms and Abbreviations

API—American Petroleum Institute
ASR—alkali-silica reactivity
ASTM—American Society for Testing and Materials
avg—average
Bc—Bearden units of consistency
BHCT—bottomhole circulating temperature
BHST—bottomhole static temperature
bwoc—by weight of cement
CaCl₂—chemical formula for calcium chloride
gal/sk—gallon(s) per sack of cement
H₂O—chemical formula for water
lb/gal—pound(s) per gallon
POD—point of departure
psi—pound(s) per square inch
QC—quality control
RPM—revolution(s) per minute
TXI—Texas Industries
TXI LW—lightweight cement available from TXI
UCA—ultrasonic cement analyzer
ULHS—ultra-lightweight hollow (glass) spheres
3K—3,000-psi designation
4K—4,000-psi designation
5K—5,000-psi designation
6K—6,000-psi designation

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Appendix A—Specification Testing

TXI Information			CSI Information				Free Fluid			Rheology									
Cmt Type	Production Date	Date Received	CSI Log #	Bucket Opened	Test Date	Temp (°F)	mL	% by volume	H ₂ O (%)	Temp (°F)	300 RPM	200 RPM	100 RPM	60 RPM	30 RPM	6 RPM	3 RPM	10 sec G.S.	10 min G.S.
LW	09/18/00	11/06/00	C-108 B-1	11/07/00	11/15/00	105	2	0.8	75	80	57	50	42	38	33	22	12	13	120
LW	09/18/00	11/06/00	C-108 B-2	12/05/00	01/04/01	105	2	0.8	75	80	58	52	45	38	32	21	13	13	125
LW	09/18/00	11/06/00	C-108 B-11	06/24/01	06/26/01	105	2	0.8	75	80	60	55	47	39	21	20	15	15	127

CSI Information			Water		45°F Strengths			60°F Strengths			80°F Strengths			120°F Strengths			Thickening Time			
Cmt	CSI	Test	Conc.		Time	CaCl ₂	CS	Time	CaCl ₂	CS	Time	CS	Time	CS	Time	Sch	Initial	Time to	Time to	
Type	Log #	Date	(%)		(Hr)	(%)	(psi)	(Hr)	(%)	(psi)	(Hr)	(psi)	(Hr)	(psi)	(Hr)	#	Bc	70 Bc	100 Bc	
LW	C-108 B-1	11/15/00	75.0		24	2.0	166	24	1.0	254	24	523	24	1579	24	5	6	2:02	2:20	
LW	C-108 B-2	01/04/01	75.0		24	2.0	161	24	1.0	221	24	441	24	1225	24	5	5	2:00	2:17	
LW	C-108 B-11	06/26/01	75.0		24	2.0	158	---	---	---	24	501	---	---	---	5	9	1:57	2:18	

Appendix A—Specification Testing

TXI Information		CSI Information			Free Fluid		Rheology									
Cmt Type	Prod. Date	Date Received	CSI Log #	Bucket Opened	Test Date	mL	% by volume	Temp (°F)	300 RPM	200 RPM	100 RPM	60 RPM	30 RPM	6 RPM	3 RPM	10 min G.S.
Class H	09/27/00	11/06/00	C-108 A-1	11/06/00	11/07/00	1.9	0.8	80	89	72	53	45	37	16	10	---
Class H	09/27/00	11/06/00	C-108 A-1	11/06/00	11/21/00	3	1.2	80	85	70	53	45	38	14	8	19
Class H	09/27/00	11/06/00	C-108 A-2	11/06/00	01/11/01	3	1.2	80	95	80	60	51	42	20	12	26
Class H	09/27/00	11/06/00	C-108 A-3	02/22/01	---	---	---	---	---	---	---	---	---	---	---	---
Class H	09/27/00	11/06/00	C-108 A-4	06/26/01	---	---	---	---	---	---	---	---	---	---	---	---

CSI Information			Water		100°F Strengths		140°F Strengths		Thickening Time		
Cement Type	CSI Log#	Test Date	Conc. (%)	Time (Hr)	CS (psi)	Time (Hr)	CS (psi)	Sch #	Initial Bc	Time to 100 Bc	
Class H	C-108 A-1	11/07/00	38.0	8	524	8	2088	---	---	---	
Class H	C-108 A-1	11/29/00	38.0	8	480	8	2249	5	12	2:03	
Class H	C-108 A-1	02/22/01	38.0	---	---	8	1617	---	---	---	
Class H	C-108 A-1	02/22/01	38.0	---	---	8	1550	---	---	---	
Class H	C-108 A-2	01/11/01	38.0	8	1201	8	1964	5	7	1:54	
Class H	C-108 A-2	01/12/01	38.0	8	1039	8	1756	---	---	---	
Class H	C-108 A-2	02/12/01	38.0	8	424	8	1717	---	---	---	
Class H	C-108 A-3	02/22/01	38.0	---	---	8	1567	---	---	---	
Class H	C-108 A-3	02/22/01	38.0	---	---	8	1580	---	---	---	
Class H	C-108 A-4	06/29/01	38.0	---	---	---	---	5	17	2:00	

Appendix A—Specification Testing

TXI Information			CSI Information					Rheology							
Cmt Type	Production Date	Date Received	CSI Log #	Bucket Opened	Test Date	Temp (°F)	300 RPM	200 RPM	100 RPM	60 RPM	30 RPM	6 RPM	3 RPM	10 sec G.S.	10 min G.S.
Class A	09/27/00	11/06/00	C-113 A-1	11/09/00	11/17/00	80	75	62	48	40	33	17	10	10	19
Class A	09/27/00	11/06/00	C-113 A-2	11/20/00	11/20/00	80	80	65	49	40	34	17	10	10	19
Class A	09/27/00	11/06/00	C-113 A-3	12/04/00	12/05/00	80	78	64	50	40	32	16	10	10	19
Class A	09/27/00	11/06/00	C-113 A-4	01/09/01	01/09/01	80	80	65	52	40	33	17	11	10	20
Class A	09/27/00	11/06/00	C-113 A-5	11/09/00	---	---	---	---	---	---	---	---	---	---	---
Class A	03/10/01	03/19/01	C-189 A-1	03/20/01	03/20/01	80	77	63	51	39	32	17	10	10	20
Class A	03/10/01	03/19/01	C-189 A-5	06/01/01	06/29/01	---	---	---	---	---	---	---	---	---	---

CSI Information				Water Conc. (%)	100°F Strengths				45°F Strengths				60°F Strengths				80°F Strengths				Thickening Time			
Cmt Type	CSI Log #	Test Date	Time (Hr)		CS (psi)	Time (Hr)	CS (psi)	Time (Hr)	CaCl ₂ (%)	CS (psi)	Time (Hr)	CaCl ₂ (%)	CS (psi)	Time (Hr)	CS (psi)	Test Date	Sch #	Int. Bc	Time to 100 Bc					
Class A	C-113 A-1	11/17/00	---	---	---	---	---	---	---	---	---	---	---	---	---	11/15/00	4	9	2:17					
Class A	C-113 A-2	11/27/00	8	754	24	2607	24	2	737	24	1	1194	24	1689	01/05/01	4	8	2:21						
Class A	C-113 A-2	02/12/01	---	---	---	---	---	---	---	---	---	---	---	2022	---	---	---	---	---					
Class A	C-113 A-3	12/05/00	---	---	24	2585	---	---	---	---	---	---	---	---	---	---	---	---	---					
Class A	C-113 A-4	01/17/01	8	729	24	2527	24	2	1042	---	---	---	---	---	---	01/17/01	4	4	2:09					
Class A	C-113 A-5	02/15/01	8	931	24	2568	---	---	---	---	---	---	---	---	---	---	---	---	---					
Class A	C-113 A-5	02/22/01	---	---	24	2334	---	---	---	---	---	---	---	---	---	---	---	---	---					
Class A	C-113 A-5	02/22/01	---	---	24	2590	---	---	---	---	---	---	---	---	---	---	---	---	---					
Class A	C-113 A-5	02/22/01	---	---	24	2250	---	---	---	---	---	---	---	---	---	---	---	---	---					
Class A	C-189 A-1	03/20/01	8	916	24	2364	---	---	---	---	1	1238	24	1737	03/20/01	4	12	1:42						
Class A	C-189 A-5	06/29/01	---	---	---	---	---	---	---	---	---	---	---	---	---	06/29/01	4	10	1:37					

Appendix B—Laboratory Mixing, Testing Procedures, and Testing Scope for Foamed Cement

The working draft of ISO 10426-4¹ outlines the recommended practices for the atmospheric generation and testing of foamed cement slurries and their corresponding unfoamed base slurries. The procedures discussed in this appendix and used for this project were borrowed from ISO 10426-4.

B.1 Preparing Unfoamed Base Slurry

B.1.1 Calculation of Base Cement With and Without Surfactants

Because the final slurry for foamed cement contains surfactant(s), these materials cannot be added to the base slurry for initial mixing. This will require that the density of the base slurry be adjusted to compensate for the later addition of the surfactant(s) prior to foaming.

Example: Slurry Design: Class G Cement + 0.2 gal/sk Surfactant

Base slurry density = 14.5 lb/gal
Surfactant weight = 10 lb/gal

Base Slurry Calculations:	<u>Weight</u>	<u>Volume</u>
Cement	94 lb	3.59 gal
Surfactant	2 lb (0.2 gal * 10 lb/gal)	0.2 gal
Water	<u>55.39 lb</u>	<u>6.65 gal</u>
Total	151.39 lb	10.44 gal

Calculation of True Weight % Contributions:

Cement	62.1 %	(94/151.39)
Surfactant	1.3 %	(2/151.39)
Water	36.6 %	(55.39/151.39)

Slurry without Surfactants:	<u>Weight</u>	<u>Volume</u>
Cement	94 lb	3.59 gal
Water	<u>55.39 lb</u>	<u>6.65 gal</u>
Total	149.39 lb	10.24 gal

Slurry Density without Surfactants: 149.39/10.24 = 14.59 lb/gal

B.2 Equipment

B.2.1 Blender Container

A special blending container is required for preparing foamed cement at ambient pressure in the laboratory. (A typical blending container is shown in Figure B.1) The blending container is similar to the one used for standard slurry preparation except that it has a threaded cap with an O-ring seal. The cap has a small hole (approx. 3/4-in. diameter) in the center fitted with a removable plug that has an O-ring seal.

B.2.2 Multi-Blade Assembly

The multi-blade assembly is what is used during this project. The multi-blade or stacked-blade assembly is constructed of a series of assemblies, each blade corresponding to the requirements of ISO 10426-2², clause 5. The assembly consists of five (5) standard blades attached to a central shaft, and spaced equally throughout the mixing container. A typical assembly is shown in Figure B.1.

Fig. B.1—Example of a typical blending container

B.3 Container Volume

Accurate determination of the volume of the blending container is critical to this procedure. The calculations for slurry volume and foamed cement density are based on this volume determination. Weigh the clean, dry, blending container (including mixing assembly, screw-on lid and screw-in plug for the lid). Remove the screw-on lid from the mixing container and then remove the screw-in plug from the lid. Fill the mixing container with water and then screw the lid on tightly. Pour additional water into the hole in the lid for the plug until the container is completely filled, and then screw the plug tightly into the lid. Wipe the excess water that exits from the plug's vent hole, and then weigh the container again. The weight of the water inside the container is then divided by the density of the water to determine an accurate volume for the mixing container.

B.4 Preparing Base Cement Slurry

This method assumes that the base slurry as described in Section B.1.1 is being prepared in a separate mixing container, and this slurry is then to be weighed into the mixing container described in Section B.2.1. To prepare sufficient volume may require multiple mixes with the standard mixing procedure.

Base slurries containing all additives except foaming surfactant(s) should be prepared according to ISO 10426-2², clause 5. When possible, the temperature of the cement sample, additives, and mix water should be within $\pm 2^{\circ}\text{C}$ (3°F) of the respective temperatures recorded from the well site. The temperature of the mixing container should approximate that of the mix water being used in the slurry design. The mixing device should be calibrated annually to a tolerance of ± 3.3 rev/s (200 rpm) at 66.7 rev/s (4,000 rpm) and ± 8.3 rev/s (500 rpm) at 200 rev/s (12,000 rpm). As required, the density of the unfoamed cement slurry can be determined by methods found in ISO 10426-2², clause 6.

B.5 Determining Slurry Volumes and Weights

B.5.1 Slurry Volume

Determine the volume of unfoamed cement slurry to be mixed. The total volume of unfoamed cement slurry should include the volume of the surfactant(s) to be added to the slurry. The surfactant(s) is to be added after the initial mixing of the base slurry. The volume of unfoamed slurry to be placed in the container may be determined by the following procedure.

When it is desired to foam a slurry with a specific amount of gas per unit volume of slurry (foam quality), the resultant density of the foamed slurry must be determined. This can be calculated by Equation 1.

$$FD = (100 - \%G) \div 100 \times UFDS \quad (1)$$

Where:

FD	=	Foamed density of the slurry
%G	=	Percentage of gas in final foamed slurry
UFDS	=	Unfoamed slurry density with surfactant(s)

When a desired foamed slurry density is known or after calculating it with Equation 1, determine the grams of cement slurry including surfactant(s) that is to be placed into the foam blender to prepare the foamed slurry. This can be calculated by Equation 2.

$$GUFS = CV \times FD \quad (2)$$

Where:

GUFS	=	Grams of unfoamed slurry including surfactant(s) to be placed into the foam mixer
CV	=	Container volume of foam mixer (mL)
FD	=	Foamed density of the slurry (g/mL)

Example:

Container volume	=	1170 mL
Base slurry density	=	14.5 lb/gal (1.74 g/mL)
Foamed cement density	=	10.0 lb/gal (1.2 g/mL)
Unfoamed slurry weight	=	$1170 \text{ mL} \times 1.2 \text{ g/mL} = 1404 \text{ g}$

B.5.2 Surfactant(s) and Slurry Weight

The surfactant(s) weight is determined by taking the unfoamed slurry weight and multiplying by the percent by weight of surfactant(s). The slurry weight is determined by taking the unfoamed slurry weight and subtracting the surfactant(s) weight. This can be calculated by Equation 4.

$$GS = GUFs \times (\% \text{Surfactant} / 100) \quad (3)$$

Where: GS = Grams of surfactants (total) to place into the foam mixer with the unfoamed slurry without surfactant(s)
GUFs = Total grams unfoamed slurry prepared in B.1

$$GUSM = GUFs - GS \quad (4)$$

Where: GUSM = Grams of unfoamed slurry without surfactant(s) to be placed into the mixer.

Example: Unfoamed slurry weight = 1404.1 g
Percent by weight of surfactant = 1.3 %
Surfactant weight = $1404.1 \times 0.013 = 18.5$ g
Slurry weight = $1404.1 - 18.5 = 1385.6$ g

B.6 Preparing the Atmospheric Foamed Slurry

Based on the volume calculated in Section B.5.1, weigh the appropriate amount of the prepared slurry into the special mixing container. Add the calculated amount of surfactant(s). The final weight of the cement slurry and added surfactant(s) should be checked against the final desired base slurry density. Before foaming, verify that the total weight of the slurry and added surfactant(s) corresponds to the weight calculated in Section B.5.2.

B.6.1 Generating a Foamed Cement

Make sure the mixing container is sealed. Using the blade assembly described in Section B.2.2, the slurry should be mixed at the 12,000 rpm setting for 15 seconds. Because of the increase in slurry volume and viscosity, the maximum rpm of the blender could be less than 12,000 rpm. The maximum attainable rpm will depend on the power of the blender, slurry density, and foam quality. Record and report the final rpm of the mixer.

During the mixing, there will be a noticeable change in the sound (pitch) from the blender. After mixing, there may be some slight pressure in the mixing container because of temperature increases and energy imparted to the foam during the foaming process. Be careful when removing the top of the mixing container. After mixing, open the sampling port or container lid, and verify that the slurry completely fills the slurry-mixing container. If the slurry does not fill the mixing container at the end of the 15-second

mixing, it is doubtful the slurry will foam properly under field conditions. The slurry should be redesigned.

B.7 Atmospheric Testing of Foamed Cement Slurries

Because of the high air entrainment in a foamed cement slurry, it is necessary to modify some of the standard testing procedures to prevent obtaining erroneous test results.

B.7.1 Determining Foamed Slurry Density

The density of the foamed slurry should be determined by pouring it into a container with a large open top that has a known volume when completely filled. Weigh the container, pour the foamed slurry into the container, and level the top with a straight blade. Wipe the outside of the container clean, and weigh the container with the foamed slurry. The density of the foamed slurry in the container is determined by dividing the slurry mass by the container volume and converting to the appropriate density units.

B.7.2 Determining Slurry Stability

B.7.2.1 Unset Slurry Stability

Evaluate the foam stability by pouring a sample of the foamed cement slurry into a container or graduated cylinder for 2 hours of continued evaluation. Cover or seal the top of the container to prevent drying or dehydration of the sample. Since the main purpose of this test is to check for settling and stability in the foamed slurry, the visual appearance of the foamed slurry (such as free fluid, settling, or bubbles concentrated in a specific area) must be noted. If desired, density measurements may be made of the foam at multiple locations in the cylinder after the 2-hour period. To determine the density of the slurry at various locations in the cylinder, a large syringe with a Tygon tube on it can be used to remove small portions from the top, middle, and bottom. The removed slurry can then be transferred to a smaller graduated cylinder to determine the weight of a known volume of the slurry. From there, the specific gravity and density can be determined.

Pour the foamed slurry into a standard 250-mL graduated cylinder that is used for free-fluid testing. Cover the top of the cylinder to prevent dehydration, place it onto the counter-top, and visually examine it during the 2-hour period. The cylinder cannot be cured at temperatures above the ambient temperature at which the foamed slurry was prepared because an increase in temperature will increase the bubble size and may have an effect on the slurry stability.

B.7.2.2 Set Slurry Stability

Check foam stability by curing samples until they are set for density gradient measurement throughout the sample. These may be cured in non-greased, covered 50.8-mm (2-in.) diameter, 101.6-mm (4-in.) tall cylinders or any appropriate covered container. Use of grease or other mold-release agents should be avoided as these materials may affect the stability of the foamed cement.

Cut or break the samples into sections, mark them from the top to the bottom, and

measure the specific gravity of each section. The specimen should not be cut with a saw that uses water. The use of water may cause the specimen to absorb water and change the density of the specimen. Large variations in density from sample top to bottom are an indication of instability. When determining the specific gravity by Archimedes principal, it is recommended that a beaker of fresh water be placed on a scale and tared. The specimen is placed into a loop of fine string (or thread) and suspended in the water for the first measurement for determining the volume of the specimen (V). The volume of the specimen (mL) will be equal to the weight of the water displaced by the specimen when suspended in the water. The weight of the specimen being suspended in the water must be determined quickly to prevent the specimen from absorbing water and giving erroneous results. The specimen is then lowered to rest on the bottom of the beaker of water to obtain the actual weight of the specimen (W). The specific gravity (SG) is then determined by dividing the weight, W (in grams) by volume, V (in mL). The slurry density can also be determined ($SG \times 8.33 = \text{lb/gal}$).

Signs of foam instability include the following:

- More than a trace of free fluid.
- Bubble breakout noted by bubbles appearing on the surface of the sample.
- Excessive gap at the top of the specimen. Minor meniscus effects are normal.
- Visual signs of density segregation as indicated by streaking or light to dark color change from top to bottom.
- Large variations in density from sample top to bottom.

B.7.3 Determining Compressive Strength

The foamed cement slurry is poured into a curing mold that can be sealed. The sealing lid prevents the foamed slurry from expanding out of the curing mold as it is heated. This expansion can result in an undesired density decrease. The mold can be a standard 50.8-mm (2-in.) cube mold with a cover clamped to the top.

The sealed mold containing the foamed cement slurry is then placed into an atmospheric water bath, cured, and the strength is determined as specified by API. The temperature is normally limited to approximately 65°C (149°F), but can sometimes be increased to 90°C (194°F) if there is sufficient seal to prevent the slurry from expanding out of the curing mold.

B.7.4 Determining Permeability

For determination of the permeability foamed cement slurry is poured into permeability test molds. These test specimens are then cured and tested for permeability while still in the permeability mold. If the foamed cement slurry is poured into a mold in which the specimen must be removed, cored, cut, or sealed in the permeability testing apparatus, the specimen may be damaged. Curing should be conducted under atmospheric pressure as for the compressive strengths in Section B.7.3.

The permeability testing of the cured specimens will be performed using the procedures in ISO 10426-2², clause 11.

B.8 Determining Other Tests on Base Unfoamed Slurry

A slurry that is foamed at atmospheric pressure should not be tested under pressure. Applying pressure to a foamed slurry prepared at atmospheric pressure will compress the foam, changing the density and gas ratio. This can also allow contamination when tested in a HPHT consistometer for thickening time.

For the following tests, the base unfoamed slurry without the surfactant(s) is prepared according to ISO 10426-2², clause 5. After the slurry is prepared, the mixer is stopped and the surfactant(s) added and stirred gently with a spatula to distribute it uniformly in the slurry. It is recommended the slurry be transferred gently from the mixing container to a beaker and back three times to ensure a uniform distribution. The use of a small amount of material for preventing/breaking air entrainment in slurries that are not foamed is permitted for these tests. Materials to prevent/break air entrainment should not be used in any foamed slurries.

B.8.1 Determining Thickening Time

Since the surfactant(s) will affect the thickening time, and the foam itself does not affect the thickening time of a cement slurry, the thickening time test is normally performed using a standard HPHT consistometer on the base unfoamed cement slurry containing the surfactant(s).

The thickening time test of the unfoamed slurry containing the surfactant(s) will be performed using the procedures in ISO 10426-2², clause 9.

B.8.2 Determining Fluid Loss

Fluid-loss tests performed with a foamed cement prepared at atmospheric pressure will not yield reliable results. The fluid loss values obtained from a foamed cement slurry will be slightly less than that of the base unfoamed cement slurry. The fluid loss of the base unfoamed cement is normally used as an indication of the fluid loss of the foamed cement slurry.

The static fluid-loss test of the unfoamed slurry containing the surfactant(s) is performed using the procedures in ISO 10426-2², clause 10.

B.8.3 Determining Rheological Properties

With the concentration of gas in a foamed slurry changing continuously during pumping of the job, it is impractical to perform rheological testing at all the foam quality concentrations that are needed to model the frictional pressures during pumping of a foamed slurry. Use of a rotational viscometer will result in separation of the gas from the slurry, causing erroneous results. Correlations can be used to convert the rheological properties of the base unfoamed slurry to that of a foamed cement with varying foam qualities to simulate the job.

The rheological test of the unfoamed slurry containing the surfactant(s) is performed using the procedures in ISO 10426-2², clause 12.

References

- ¹ ISO 10426-4: “Petroleum and Natural Gas Industries—Cements and Materials for Well Cementing, Part 4: Recommended Practice for Atmospheric Foam Cement Slurry Preparation,” working draft 2001.
- ² ISO 10426-2: “Petroleum and Natural Gas Industries—Cements and Materials for Well Cementing, Part 2: Recommended Practice for Testing of Well Cements,” 1998.