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Experimental Investigation of Silica Fume as a Cement Extender for Liner Cementing in Iranian Oil/Gas Wells

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Abstract

Silica fume is a by-product of silicon metal or ferrosilicon alloys in smelters using electric arc furnaces. It consists of 85% to 95% amorphous silicon dioxide (SiO_2). Each individual particle of silica fume is spherical with average diameter 0.15-0.3 μm (100 times finer than cement particle); therefore its specific surface area is high. Silica fume particles are water wet and absorb excess water in cement slurry when cement slurry is extended by water. Silica fume thickens the cement slurry, so rheological properties are controlled by dispersants. In this paper, optimal concentration of silica fume and other additives for preparing 90 pcf cement slurry for liner cementing in one Iranian oilfield is determined. The criteria of designing slurry formulation are slurry density, rheological properties, fluid loss, free water, thickening time of cement slurry, and compressive strength and permeability of set cement. Finally, based on experimental results, the preferable slurry compositions are selected. This formulation can be used for cementing of oil and gas wells where moderate and light weight cement density is needed.

Keywords: *Cement Extender, Light Weight Cement Density, Silica Fume, Cement Slurry, Permeability of Set Sement, Compressive Strength*

Introduction

Cement extenders are a routine additive used for reducing slurry density and increasing the yield of cement slurry. A reduction of slurry density reduces the hydrostatic pressure during cementing; this helps to cement oil and gas wells in low pressure or depleted reservoirs and prevent induced lost circulation because of the breakdown of weak formations. In addition, the number of stages required to cement a well may be reduced. Extenders reduce the amount of cement required to produce a given volume of set product which results in a greater economy. Different types of cement extenders additives, such as bentonite, pozzolans, microspheres and foam cement are used for preparing light weight cement slurry.

Silica fume can serve as an extender by allowing an additional 0.532 gallons of mix water to be added to the slurry per pound of

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silica fume (4.4 *cc* water/*gr.* silica fume).[1] Some other names of silica fume are microsilica and condensed silica fume.

Silica fume consists of 85% to 95% amorphous SiO_2. Its particles are very fine (95% of SiO_2 are less than 1μm), and therefore act as a microfiller in a set cement micro-structure and it is a reactive solid to pozzolanic reaction with free lime in normal temperature.[1,2] Trace elements in silica fume depend upon the type of fume; usually, these materials have no impact on the performance of silica fume.[3]

P.K. Mehta and O.E. Gjorv[2] in 1982 had compared the compressive strength of concrete containing the fly ash and silica fume. The specimens containing the silica-fume showed considerably higher strengths, even at early and 90-day ages of curing, the their compressive strength was almost twice as much as the control.

At a fixed slurry density, increasing the silica fume concentration improves the early compressive strength development and reduces the free water, but it slightly increases the slurry viscosity.

Silica fume particles act as particulate materials in filter cake to reduce the fluid loss of slurry into the permeable formation. Silica fume particle size is very small (less than 0.5 μm), and therefore can enter the filter cake and lodge between the cement particles, block the narrow passage of fluid, and finally, decrease the permeability of cement cake.

Increase of water to cement ratio (WCR) of cement slurry causes the cement permeability to increase [4]. In water extended cement slurry the WCR is high, but Golapudi et. al.[5] had observed significant reduction in gas permeability when silica fume/fly ash was blended in cement. In addition, silica fume prevent the chloride and sulfate ions penetration.

Blomberg et. al.[6] and Grinrod et al.[7] observed that silica fume has a gas migration control effect. If static gel strength of cement slurry quickly increase from 100 $lb/l00ft^2$ to 500 $lb/l00ft^2$ (often referred to as the cement slurry transition), then it is viewed as favorable for restricting gas percolation through the unset cement. In gas-tight cement, a transition time less than 30 min is recommended. A key ingredient in the cement design is high fineness amorphous silica that produces several important gas-tight properties.[8] In this paper, the optimal concentration of silica fume and other additives for preparing 90 pcf cement slurry for liner cementing in one Iranian oilfield is determined. The criteria of designing slurry formulation are slurry density, rheological properties, fluid loss, free water, thickening time of cement slurry, and compressive strength and permeability of set cement. The following section describes the procedures of formulating the slurry compositions 90 pcf cement.

Material and Method

Silica fume is obtained from Iran Ferrosilice Company. Table 1 shows the Physico-chemical properties of silica fume (SF). Class G type HSR Dyckerhoff cement is used as Portland cement and its chemical analysis is given in Table 2. Distilled water is used for making cement slurry.

Cement Slurry Design Parameters

Before cement slurry is pumped into a well, various laboratory tests can be conducted to ensure proper placement and to assist in predicting the performance and behavior of the slurry as it is pumped and after its placement. The following factors will affect cement slurry design[9]:

- Well depth and temperature
- Max. allowable pumping or thickening time
- Strength of cement required to support casing
- Quality of available mixing water
- Type of drilling fluid and its additive
- Slurry density
- Filtration control

Two basic affecting factors on the downhole performance of cement slurries are temperature and pressure.

In this study, 90 pcf cement slurries are designed for 7 in liner cementing in one Iranian oilfield. Cement slurry density is reduced by increasing WCR and adding silica fume as an extender additive.

Liner shoe depth is approximately 2400-2500 m and liner length is about 400-500 m. The temperature gradient (TG) of this field is 1.15-1.2 °F/100ft. The bottom hole static temperature (BHST) is about 170-180 °F and Liner lap static temperature is approximately 160 °F. According to Table 4 of API Recommended practice 10B[10], TG and liner shoe depth, schedule 9.18 is selected. Also, Bottom Hole Circulating Temperature (BHCT), Heat up rate, initial pressure and final pressure are 132 °F, 1.6 °F/min, 650 psi and 5000 psi, respectively. Liner lap is the critical point of liner; therefore liner lap static temperature is used for compressive strength and water permeability tests. Minimum allowable pumping time for 7 in liner is approximately 180 (min), therefore the minimum designed slurry Thickening Time (TT) should be 180 min. The API fluid loss of cement slurries must be controlled to 70 cc/30min.

Table 1. Physicochemical properties of the used silica fume.

Chemical composition, wt%					
SiO_2	Fe_2O_3	CaO	Al_2O_3	MgO	C
85-95	0.4-2	2-2.3	0.5-1.7	0.1-0.9	0.6-1.5
Average grain size			0.2-0.3 μm		
Specific surface area			14000 m^2/kg		
Specific gravity			2.2		
Bulk density			200 kg/m^3		
Color in bulk			Light gray		
Color in slurry			Black		

Table 2. Chemical composition of class G Dyckerhoff cement.

Chemical composition, wt%							
CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	Na_2O	K_2O
67.7	22.91	3.89	4.75	1.8	0.74	0.10	0.64

Well Cement Tests Procedures

American Petroleum Institute (API) has presented "*Recommended practice for testing well cements*", *API recommended practice 10B*[10], attempting to unite the well cement test procedures worldwide. These tests were devised to help drilling personnel to determine if a given cement composition will be suitable for given well conditions. API RP-10B[10] is used to determine the cement slurry and set cement specification.

Slurry Preparation

Cement slurry was prepared according to section 5 of API RP-10B [10]. The mixing method strongly affects slurry and set cement properties. Cement additives can be dry blended or wet blended in cement slurry. When additives are added to mixed water before cement, it is called wet blending. Sequence of adding additive and mixing time are important in the wet blending method. The solution of cement slurry is prepared at low rpm of mixer (4000 ± 200 rpm).

Slurry Density Determination

Slurry density was determined according to section 6.5 of API RP-10B.[10]

Rheological Properties

Rheological properties and the gel strength of cement slurry are measured according to sections 12.6 and 12.7 of API RP-10B[10], respectively. Before conducting tests, cement slurry was conditioned for 35 to 40 min in an atmospheric consistometer to BHCT. During these tests, the temperature of the slurry was kept roughly fixed. Rheological properties of cement slurry are determined by "Chan35 V-G meter" of Chandler Engineering Company.

Well Simulation Free Water Test

Free water test of cement slurry is performed according to sections 15.2, 15.3, 15.4.1 and 15.5 of API RP-10B[10]. Cement slurry was conditioned for 35 to 40 *min* in an atmospheric consistometer to BHCT before the test. According to section 15.4.1 of API RP-10B[10] the graduated cylinder must be immersed in a water bath with a vibration free condition, but in this study, due to lack of an appropriate water bath, the graduated cylinder was put in the circulated water bath. According to section 15.5 of API RP-10B[10] the graduated tube must be static in ambient temperature. The graduated cylinder was held in vertical position because the well was assumed vertically.

Static Fluid Loss Test

Fluid loss test was performed according to section 10 of API RP-10B[10] and some of them are based on standard API filter press test in which filtration pressure is 100 psi and double layer screen 325 mesh on top and support screen 60 mesh are used as the filtration area. Cement slurry was conditioned for 35 to 40 min in an atmospheric consistometer to BHCT before test.

For tests that "blowout" before 30 min in the standard API filter press, the following equation is used for calculating standard API fluid loss.

$$\text{Calculated standard API fluid loss} = Q_t \frac{5.477}{\sqrt{t}}$$

For tests that "blowout" before 30 min in API fluid loss (filtration pressure is 900 psi), the following equation is used.

$$\text{Calculated API fluid loss} = 2\,Q_t\,\frac{5.477}{\sqrt{t}}$$

Q_t is the volume (cc) of filtrate collected at the time t (min) of the blowout.

Well Simulation Compressive Strength Test

Set cement compressive strength is achieved according to section 7 of API RP-10B[10] at atmospheric pressure after 24 hr curing in 160ºF. Some of the cement slurries were conditioned to BHCT before molding to provide better simulation of the well condition.

Permeability Test

Water permeability of set cement is determined according to section 11 of API RP-10B[10]. Cement slurry was molded in a special mold sample holder and cured at 160ºF at atmospheric pressure. Some of the cement slurries were conditioned to BHCT before molding for better simulation of well condition. After removing permeability molds from the curing water bath and detaching the top and bottom plates, permeability molds are placed in a cooling bath for four hours. Curing time of permeability mold in water bath is 24hr.

Well Simulation Thickening Time Test

Slurry thickening time test was performed according to section 9 of API RP-10B[10] at atmospheric and pressurized consistometer consistent with schedule 9.18.

Slurry Solution Stability

After mixing the wet blend additives with mixed water, the solution is poured in a 250(cc) graduated cylinder and put in rest and free-vibration condition at ambient temperature. Accumulated free water on top of the slurry solution is monitored and recorded.

Results and Discussion

Experimental results of 90 pcf cement slurry tests are graphed in this section. This data consist of cement slurry composition, density, rheological properties, fluid loss, free water, thickening time test of cement slurry, compressive strength, water permeability of set cement and solution stability.

According to the additives concentration, different cement slurry compositions are designed in this study. Every cement slurry composition has a number used in the discussion.

90 pcf water extended cement slurry is prepared by using class G Dyckerhoff cement. 15% and 20% BWOC Silica fume are used to hold excess water in cement slurry and to obtain stable cement slurry. D145A is a dispersant additive used to decrease the cement slurry rheological properties. Also, D112 is a fluid loss control for high water to cement ratio (WCR) cement slurry[11] used to decrease the fluid loss of cement slurry. At last BA-100S and NaCl are used in cement slurry. BA-100S is a bonding agent and anti gas migration additive of BJ Service Company and NaCl is a salt used to increase the compressive strength of cement.

Weight of cement slurry is decreased by increasing the WCR of the slurry. The WCR of 100 pcf and 90 pcf cement slurry are 0.81 and 1.22, respectively. Cement slurry and set cement properties of 100 and 90 pcf cement slurry are compared in Table 3.

Table 3. Cement slurry and set cement properties of 100 and 90 pcf cement slurries.

Properties	100 *pcf*	90 *pcf*
PV (*cp*)	4.875	3.75
YP (*lbf/ 100ft2*)	4.125	2.25
10 *sec* GS (*lb/ 100ft2*)	3.5	2
10 *min* GS (*lb/ 100ft2*)	12	9.5
Std Fluid Loss (*cc/30min*)	1415.4	1741
Free Water (*%*) at 80°*F*	9.6	27.6
Compressive Strength (*psi*)	1220	150
Water Permeability (*md*)	0.01585	0.3391

When the WCR of slurry increases from 0.81 to 1.22, the rheological properties of cement slurry are decreased, but the free water of cement slurry is increased drastically from 9.6% to 27.6%. Standard FL of 100 and 90 pcf cement slurry are 1415.4 and 1741 (cc/30min), respectively. By increasing WCR, the solid fraction of cement slurry is decreased; hence the thinner cement cake is created when a certain volume of cement slurry is in the filtration chamber, whereupon the fluid loss of cement slurry is increased.

WCR has a detrimental effect on Compressive Strength (CS) and water permeability of set cement. When the WCR is increased, the compaction of set cement is decreased. Water of slurry occupies the pore volume in set cement.

Pore volume acts as a stress concentrator in set cement and causes a decreases in the compressive strength.[12] Set cement pore volume is directly related to WCR. Thickening time of high WCR slurry is long; long thickening time causes the the CS of set cement to decrease. More excess water in 90 pcf neat cement creates a wider channel in set cement, so water permeability of 90 pcf neat cement is 21.4 times more than the water permeability of 100 pcf neat cement.

A WCR of 90 pcf cement slurry is 1.22, whereas the API WCR is 0.44. High WCR cement slurry is unstable and its free water percentage is high. According to D.T. Mueller and R.L. Dillenbeck[1] the recommended 5 %BWOC and 10 %BWOC silica fume do not provide stable slurry therefore, 15% and 20% BWOC silica fume are used to prepare cement slurry.

PV and YP of 90 pcf cement slurry with 0%, 15% and 20% BWOC silica fume (SF) are shown in Fig. 1. SF increases the PV and YP of slurry. It is obvious that YP and GS of slurries are more affected than PV. SF absorbs the water in the solution and increases the gel of the slurry, making a strong network of cement and SF particles. PV is the result of friction between solid to solid and solid to liquid and liquid viscosity. SF does not increase the viscosity of the liquid phase of the slurry and its spherical particle shape acts as a ball bearing between the cement particles[13], hence it does not have a significant effect on the PV of cement slurry with respect to other rheological properties. Also, 10 *sec* and 10 *min* gel strength of 90 *pcf* cement slurry are increased the same as PV and YP (see Fig. 2).

Standard Fluid Loss (FL) and Free Water (FW) of the mentioned cement slurry are shown in Fig. 3. FL of 90 pcf neat cement slurry decreases from 1741 to 537 and 490.7 (cc/30min) when 15% and 20% BWOC SF are added to the cement slurry. Note that the FL of 15% and 20% BWOC SF is close together. The mechanism of fluid loss controlling of SF is bridging and blocking the pores in the cement cake.

Regarding the recommendation of D.T Mueller and R.L Dillenbeck[1], 15% BWOC SF has 24 (cc) excess water, but the FW of 15% and 20% BWOC SF is zero.

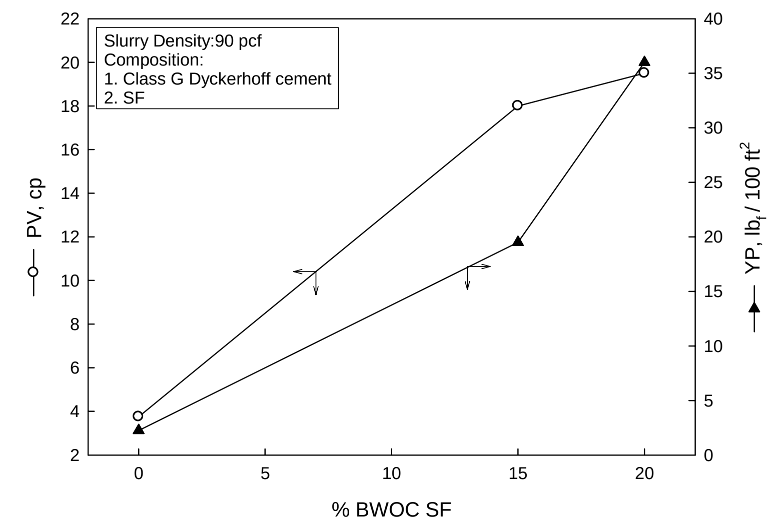

Figure 1. Effect of SF on PV and YP of 90 pcf cement slurries.

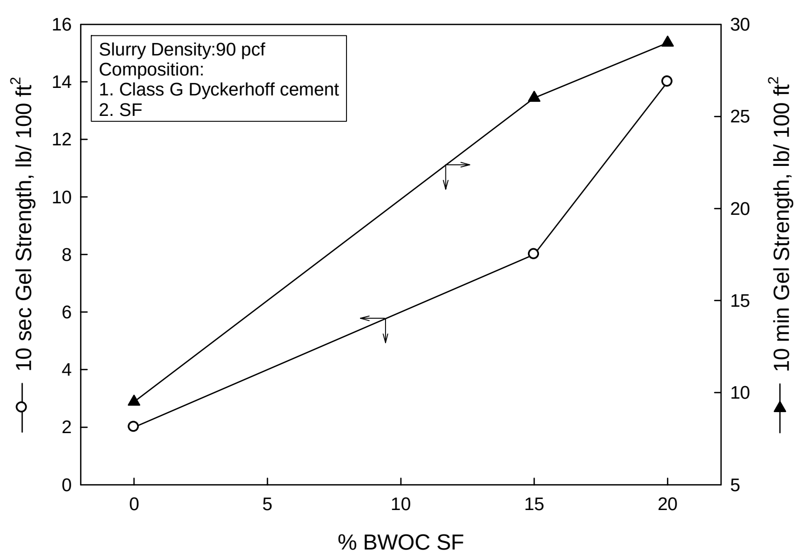

Figure 2. Effect of SF on 10 sec and 10 min Gel Strength of 90 (pcf) cement slurries.

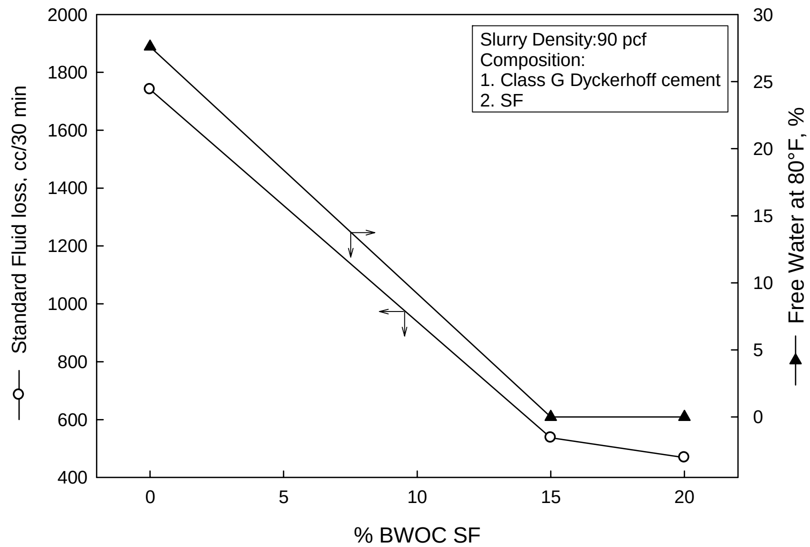

Figure 3. Effect of SF on Standard Fluid loss and Free Water of 90 pcf cement slurries.

Compressive strength and water permeability of 0%, 15% and 20% BWOC SF are shown in Fig. 4. When the 15% and 20% BWOC silica fume are added to 90 pcf neat cement slurry, the compressive strength of set cement is increased and water permeability decreases drastically. Compressive strength of set cement is improved by the micro filling effect of silica fume particles and their pozzolanic reaction.

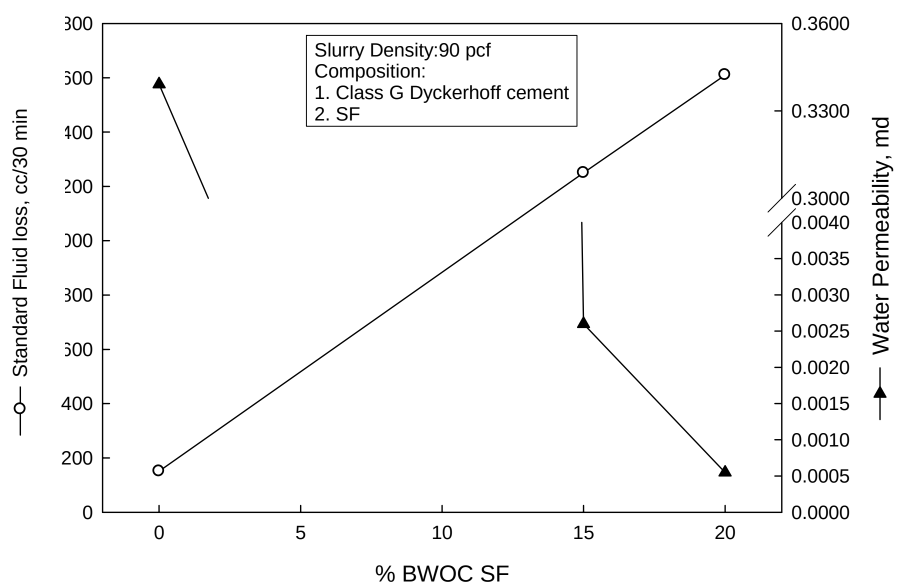

Figure 4. Effect of SF on Compressive Strength and Water Permeability of 90 pcf cement slurries.

Height reduction of neat cement in permeability mold is equal to 5 (mm). 20% height reduction of neat cement slurry in the permeability mold leads to cement particles getting close together, but the height reduction of 15% and 20% BWOC is zero. Silica fume particles hold the excess water of the cement slurry creating a narrow channel and increasing the pores tortuosity of set cement by its small size and pozzolanic reaction. Although 15% and 20% BWOC SF slurries have preferable characteristics, 15% BWOC SF is selected based on economical concern. The summary of 15% BWOC SF slurry and set cement properties are shown in Table 4.

Table 5 shows the composition number, cement slurry composition, mixing method and density of the cement slurries that contain 15% BWOC silica fume.

Table 4. Summary of composition No. 56 properties.

Composition No. 56	Mixing method	
2 *cc* AF IT G Dyckerhoff cement 15%BWOC SF	Wet blended	
Compressive Strength (*psi*)	1250	
PV (*cp*)	18	
YP ($lb_f/100ft^2$)	19.5	
10 *sec* GS ($lb/100ft^2$)	8	
10 *min* GS ($lb/100ft^2$)	26	
Free water (%) at 80°*F*	0	
Standard FL (*cc/30min*)	537	
Water Permeability (*md*)		
50 *psi*	100 *psi*	150 *psi*
0.00351	0.00582	0.0065

Table 5. Composition No., cement slurry composition, mixing method and density of 90 pcf and 15% BWOC SF cement slurry.

Comp. No.	Composition	Mixing method	Density (*pcf*)
55	3 *cc* AF IT G Dyck. cement 0% SF	After cement	90
56	2 *cc* AF IT G Dyck. cement 15%BWOC SF	Wet Blend	85-
57	2 *cc* AF IT G Dyck. cement 15%BWOC SF 0.05 GPS D145A	3 mix 1 *min* 1 W.b. 1 *min* 2 W.b. 30 *sec*	85.5+
58	0 *cc* AF IT G Dyck. cement 15%BWOC SF 0.1 GPS D145A	1 W.b. 1 *min* 2 W.b. 30 *sec*	90
59	2 *cc* AF IT G Dyck. cement 15%BWOC SF 0.15 GPS D145A	3 mix 1 *min* 1 W.b. 1 *min* 2 W.b. 30 *sec*	87
60	0 *cc* AF IT G Dyck. cement 15%BWOC SF 0.2 GPS D145A	1 W.b. 1 *min* 2 W.b. 30 *sec*	90
66	1 *cc* AF G Dyck. cement 15%BWOC SF 0.2 GPS D145A 1%BWOC D112	4 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 30 *sec* 2 W.b. 1 *min*	IT AF: 87.5 FP-6L AF: 89+
67	1 *cc* AF FP-6L G Dyck. cement 15%BWOC SF 0.2 GPS D145A 1.25%BWOC D112	4 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 30 *sec* 2 W.b. 1 *min*	89
68	1 *cc* AF G Dyck. cement 15%BWOC SF 0.2 GPS D145A 1.5%BWOC D112	4 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 30 *sec* 2 W.b. 1 *min*	IT AF: 87+ FP-6L AF: 89
69	1 *cc* AF FP-6L G Dyck. cement 15%BWOC SF 0.2 GPS D145A 1.75%BWOC D112	4 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 30 *sec* 2 W.b. 1 *min*	89-
70	1 *cc* AF FP-6L G Dyck. cement 15%BWOC SF 0.2 GPS D145A 2%BWOC D112	4 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 30 *sec* 2 W.b. 1 *min*	89- Using 1.5 *cc* AF: 89+
71	1.5 *cc* AF FP-6L G Dyck. cement 15%BWOC SF 0.25 GPS D145A 2%BWOC D112	4 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 30 *sec* 2 W.b. 1 *min*	89
72	1.5 *cc* AF FP-6L G Dyck. cement 15%BWOC SF 0.25 GPS D145A 2.25%BWOC D112	4 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 30 *sec* 2 W.b. 1 *min*	89.5-
73	1.5 *cc* AF FP-6L G Dyck. cement 15%BWOC SF 0.2 GPS D145A 2%BWOC D112 1%BWOW NaCl	5 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 1 *min* 2 W.b. 1 *min* 4 W.b. 30 *sec*	89-
74	1.5 *cc* AF FP-6L G Dyck. cement 15%BWOC SF 0.2 GPS D145A 2%BWOC D112 3%BWOW NaCl	5 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 1 *min* 2 W.b. 1 *min* 4 W.b. 30 *sec*	89
75	1.5 *cc* AF FP-6L G Dyck. cement 15%BWOC SF 0.2 GPS D145A 2%BWOC D112 5%BWOW NaCl	5 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 1 *min* 2 W.b. 1 *min* 4 W.b. 30 *sec*	90-
76	1.5 *cc* AF FP-6L G Dyck. cement 15%BWOC SF 0.2 GPS D145A 2%BWOC D112 8%BWOW NaCl	5 mix 1 *min* 1 W.b. 1 *min* 3 W.b. 1 *min* 2 W.b. 1 *min* 4 W.b. 30 *sec*	89.5-

According to D145A data sheet, D145A is a liquid dispersant additive with low retarding effect on the setting time of slurry. D145A is recommended for use in silica fume and microcement slurries. Fig. 5 and 6 show the effect of D145A on PV, YP, 10 sec and 10 min GS of 15% BWOC silica fume cement slurry. PV, YP, 10 sec and 10 min GS of cement slurry are decreased with D145A concentration. The dispersant additives are the polyanions or polycations that are absorbed on the cement grain surface and affect the electrical charge on the cement surface, converting the electrostatic attraction of cement particles to electrostatic repulsion. Fig. 7 shows the effect of D145A on standard FL and FW of cement slurry. D145A decreases the standard FL of 15% BWOC SF from 537 to 328.2 (cc/30min) when 0.2 (GPS) D145A is used.

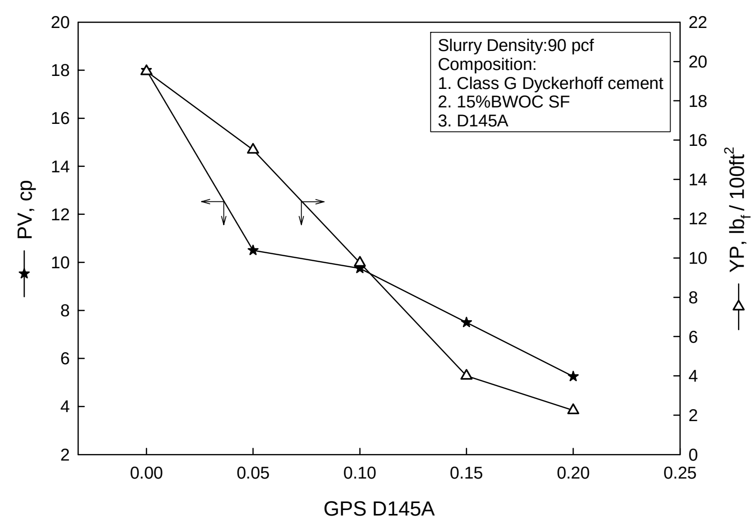

Figure 5. Effect of D145A on PV and YP of 90 pcf cement slurries.

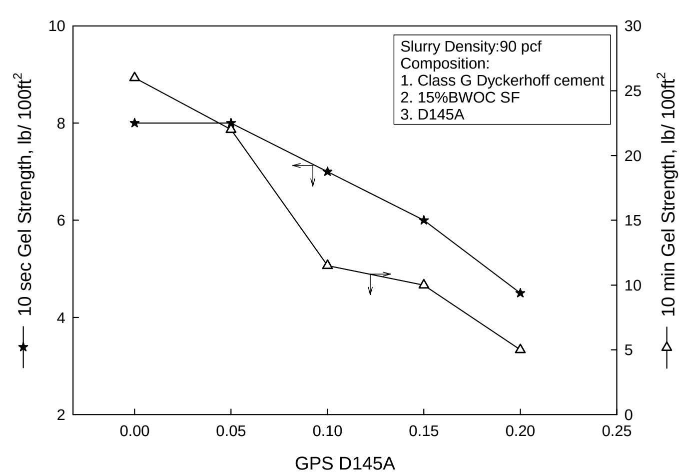

Figure 6. Effect of D145A on 10 sec and 10 min Gel Strength of 90 pcf cement slurries.

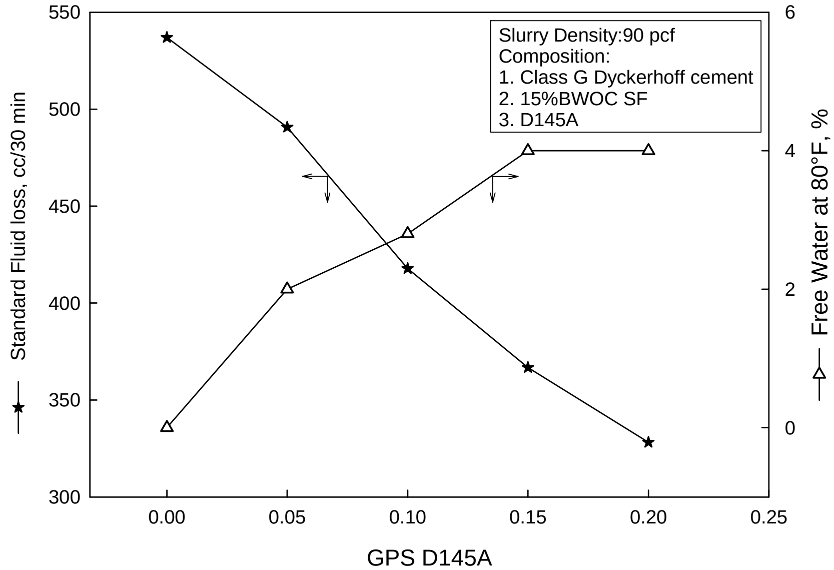

Figure 7. Effect of D145A on Standard Fluid loss and Free Water of 90 pcf cement slurries.

When the cement slurry is not dispersed, the cement particles that attract to each other (aggregate) create a permeable cement cake, however they cannot cover the open holes in the cement cake perfectly. Negative or positive charge on the surface of dispersants neutralize the attractive force between the cement particles and disperse them to slurry.[14] The dispersion of cement and SF particles improves the cement cake permeability and reduces the fluid loss volume as shown in Fig. 7.

Dispersant additives break the cement aggregate and free the entrapped water, increasing the freed water volume with dispersant concentration. Note that D145A increases the free water of 15% BWOC SF cement slurry from 0% to 4% when 0.2 GPS of D145A are used.

D145A decrease the CS and water permeability of 15% BWOC SF (see Fig. 8). One side effect of D145A is retarding, hence CS is decreased from 1250 to 1000 psi when 0.2 GPS D145A is used. Fluctuation in the compressive strength curve is small with respect to 1050 psi. By increasing D145A, the distribution of cement and SF particles become uniform and water permeability decreases to zero. In higher concentrations of D145A, the retarding effect of D145A is dominant and water permeability of 0.2 GPS of D145A increases.

Based on the experimental results shown in Fig. 5 to 8, 0.2 GPS of D145A (composition No. 60) is selected for the next step of tests. The summary of cement slurry and set cement properties of this composition are shown in Table 6.

Fig. 9 shows the effect of D112 on PV and YP of cement slurry. PV and YP of slurries are increased with D112 concentration. The effect of D112 on PV is more than the YP of slurry. D112 is a water soluble polymer that is solved in the continuous phase of cement slurry and increases the PV of cement slurry.

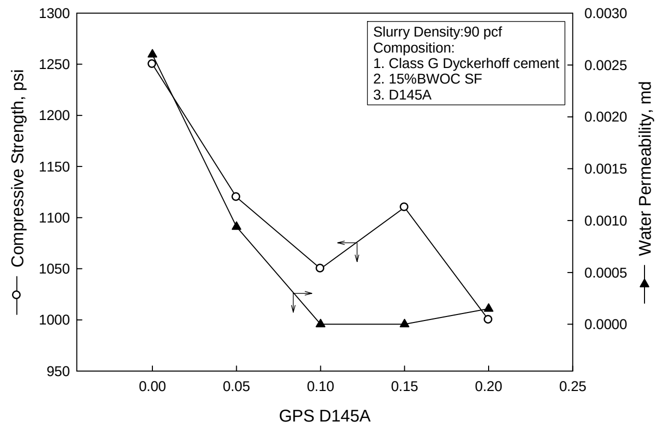

Figure 8. Effect of D145A on Compressive Strength and Water Permeability of 90 pcf cement slurries.

Table 6. Summary of composition No. 60 properties.

Composition No. 60		**Mixing method**
0 *cc* AF IT G Dyckerhoff cement 15%BWOC SF 0.2 GPS D145A		1 Wet blended 1 *min* 2 Wet blended 30 *sec*
Compressive Strength (*psi*)		1000
PV (*cp*)		5.25
YP ($lb_f/100ft^2$)		2.25
10 *sec* GS ($lb/100ft^2$)		4.5
10 *min* GS ($lb/100ft^2$)		7
Free water (%) at 80°*F*		4
Standard FL (*cc/30min*)		328.2
Water Permeability (*md*)		
50 *psi*	100 *psi*	150 *psi*
No flow	0.00019	0.00027

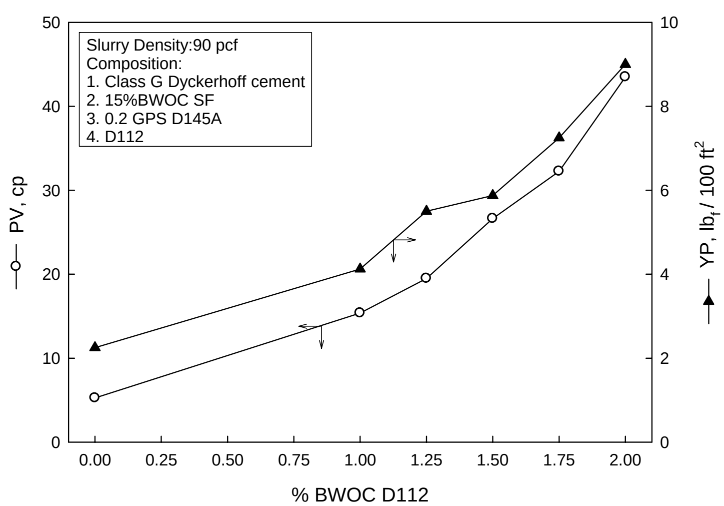

Figure 9. Effect of D112 on PV and YP of 90 pcf cement slurries.

10 sec and 10 min GS of cement slurry versus D112 concentration are shown in Fig. 10. Gel strengths of D112 slurries are increasing with the concentration of D112. Comparing Figs. 10 and 11 reveals the differences of interaction between D112-D065 and D112-D145A in the gel strengths of cement slurries. 10 sec and 10 min GS of 1.75% BWOC D112 in Fig. 10 have fluctuations; this may be the result of laboratory error.

Standard FL and FW of D112 slurries are reduced as shown in Fig. 12. FW of slurries are decreased to zero by adding 1% BWOC D112 and only a very thin, white and hard layer are formed on top of the cement column of free water test. Standard FL are decreased drastically by adding 1%BWOC D112 from 328.2 to 92.2 (cc/30min), but controlling the FL rate declines in higher concentrations of D112.

D112 is a water soluble fluid loss controller. This additive increases the viscosity of the continuous phase of cement slurry and it can form weakly bonded colloidal aggregates in solution, which are sufficiently stable to become wedged in filter-cake constrictions. Such polymers may also adsorb onto the cement grain surfaces, thus reducing the size of the pores. More likely, a superposition of these two phenomena, adsorption plus aggregation, is the true mechanism of action of polymeric fluid-loss agents.[14]

Standard fluid loss of composition No. 72 (0.25 GPS D145A and 2.25% BWOC D112) is 18.8 (cc/30min), but set cement cubes of CS test are soft after 24 hr curing in 160 ˚F. Note that the HPHT fluid loss of 1.5% D112 and 2% D112 slurry are 100 and 60 (cc/30min), respectively.

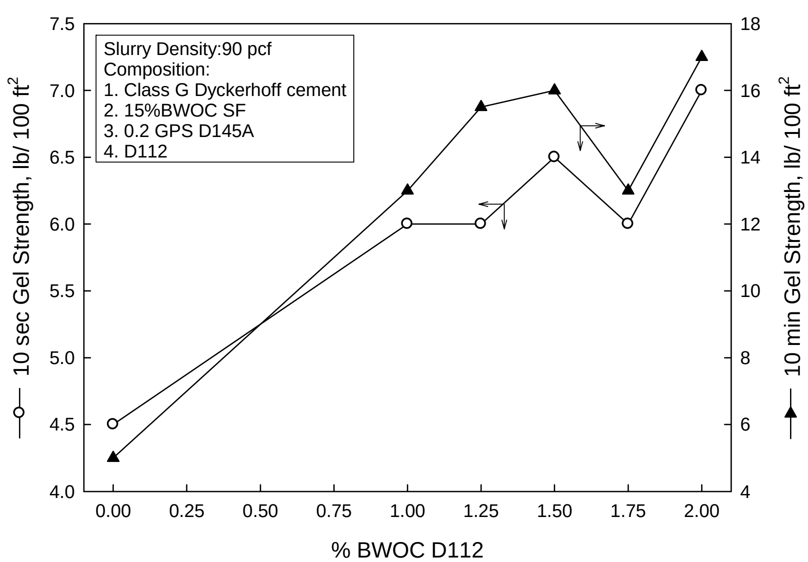

Figure 10. Effect of D112 on 10 sec and 10 min Gel Strength of 90 pcf cement slurries.

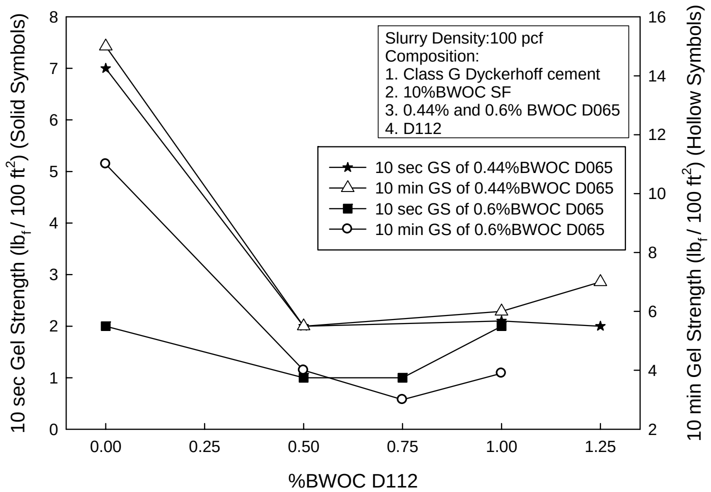

Figure 11. Effect of D065 concentration with different concentrations of D112 on 10 sec and 10 min Gel Strength of 100 pcf cement slurries.

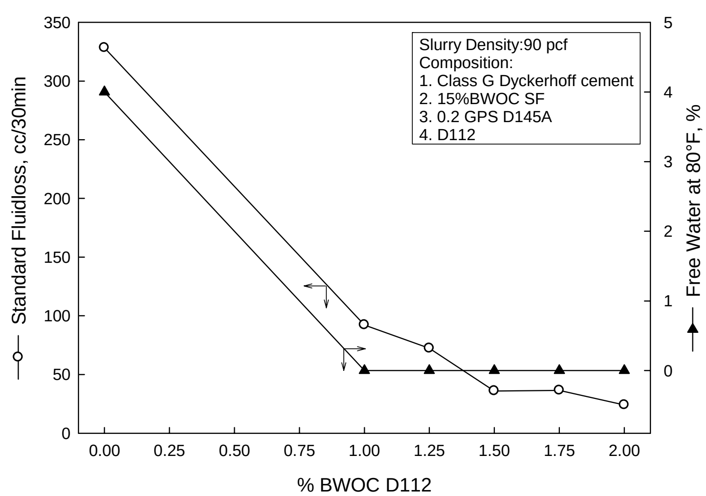

Figure 12. Effect of D112 on Standard Fluid loss and Free Water of 90 pcf cement slurries.

Retarding effect of D112 on cement slurry setting time decreases the CS of set cement from 1000 to 510 psi when 2% BWOC D112 is used in cement slurry (see Fig. 13). Water permeability, first slightly deceased from 0.00014 md to zero and then increase to 0.00524 md because of the retarding effect of D112 (see Fig. 13). Note that set cement of 2% BWOC D112 cement slurry in water permeability mold has 1 mm height reduction.

Regarding the experimental results of D112 that are shown in Fig. 9 through 13, and HPHT fluid loss of compositions No. 68 and 70, composition No. 70 is selected for the next step of tests. The only anxiety is the height reduction of cement in the permeability mold. The summary of the test results of composition No. 70 are tabulated in Table 7.

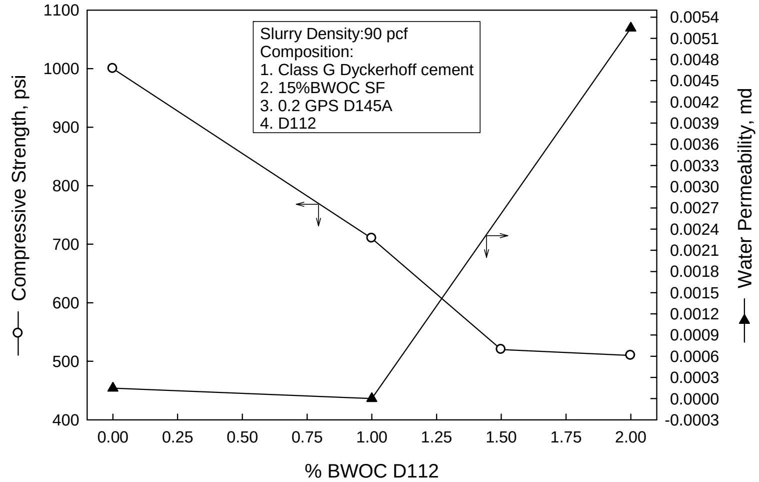

Figure 13. Effect of D112 on Compressive Strength and Water Permeability of 90 pcf cement slurries.

Table 7. Summary of composition No. 70 properties.

Composition No. 70	**Mixing method**	
0 *cc* AF IT G Dyckerhoff cement 15%BWOC SF 0.2 GPS D145A 2% BWOC D112	4 mix 1 *min* 1 Wet blended. 1 *min* 3 Wet blended 30 *sec* 2 Wet blended 1 *min*	
Compressive Strength (*psi*)	510	
PV (*cp*)	43.5	
YP ($lb_f/100ft^2$)	9	
10 *sec* GS ($lb/100ft^2$)	7	
10 *min* GS ($lb/100ft^2$)	17	
Free water (%) at 80°*F*	0	
Standard FL (*cc/30min*)	24	
HPHT FL (*cc/30min*)	60	
Water Permeability (*md*)		
50 *psi*	100 *psi*	150 *psi*
0.00487	0.00483	0.00602

Maximum NaCl usage of D145A and D112 is 37 % BWOW. It is obvious that the NaCl concentration decreases the effectiveness of these additives.

The effect of NaCl concentration on PV, YP, 10 sec and 10 min GS are shown in Fig. 14 and 15. NaCl decreases the PV, YP, 10 sec and 10 min GS of cement slurry. NaCl slightly affected 10 sec and 10 min GS in 100 pcf cement slurries, but its effect on 10 sec and 10 min GS of this slurry is higher (Fig. 15). In 100 pcf cement slurries, first the gel strengths increase then 10 sec GS holds its value. 10 min GS slightly decreased but in Fig. 15 the gel strengths of the slurries decreased uniformly. This effect of NaCl on slurry may be the result of changing D065 to D145A and increasing the WCR of cement slurry.

Fig. 16 shows the NaCl has a detrimental effect on D112 effectiveness and slightly increases the HPHT fluid loss of the cement slurry. Also, free water from the cement slurries at 80˚F is zero and NaCl has no effect on it.

According to Fig. 17, NaCl increases the CS of set cement in a limited concentration range. CS of 8% BWOW NaCl is reduced slightly to CS of 6% BWOW NaCl. NaCl has an accelerating effect to 6% BWOW NaCl in this cement slurry composition and curing temperature.

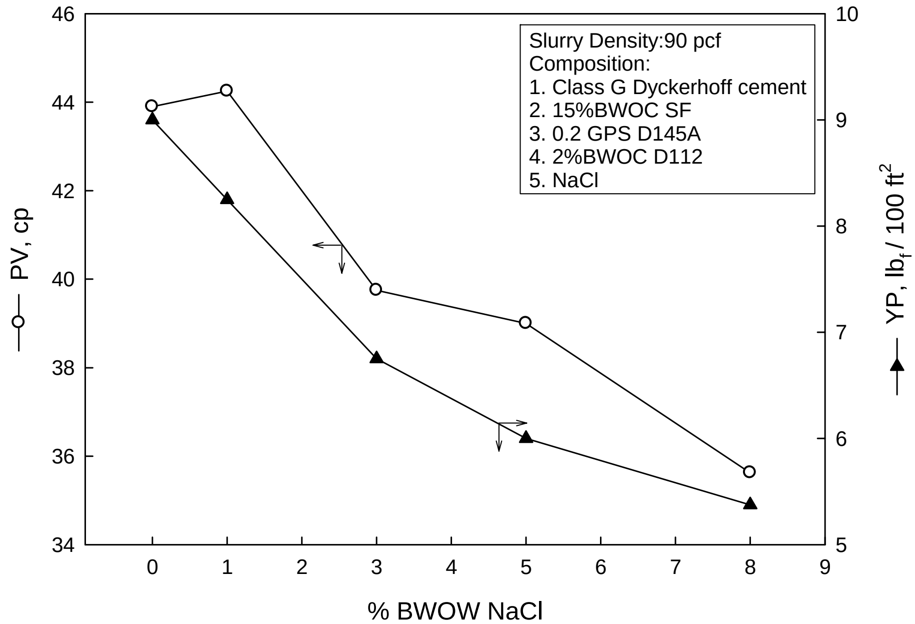

Figure 14. Effect of NaCl on PV and YP of 90 pcf cement slurries.

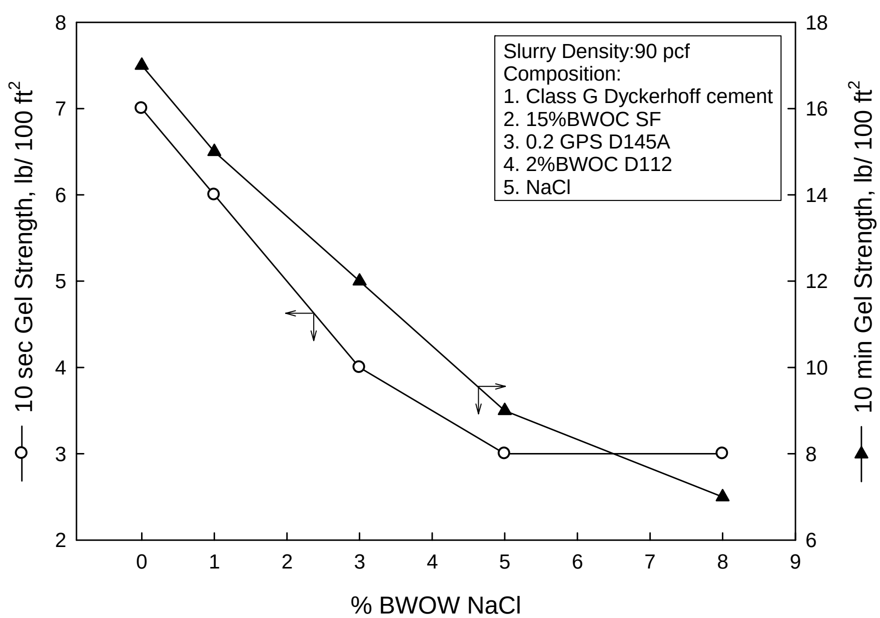

Figure 15. Effect of NaCl on 10 sec and 10 min Gel Strength of 90 pcf cement slurries.

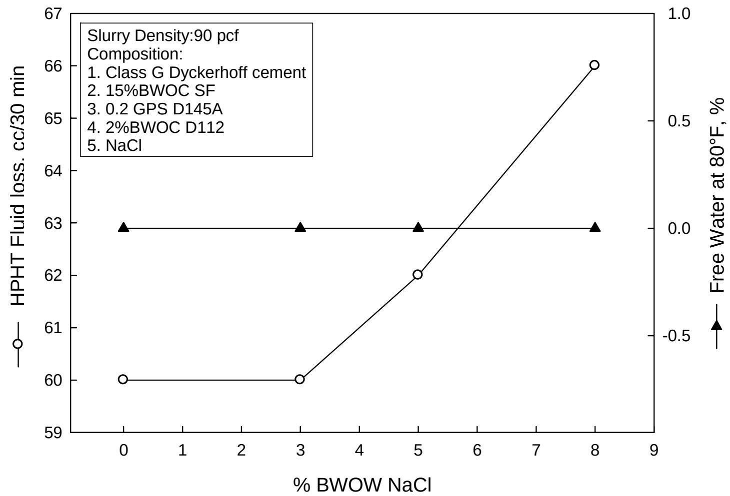

Figure 16. Effect of NaCl on HPHT Fluid loss and Free Water of 90 pcf cement slurries.

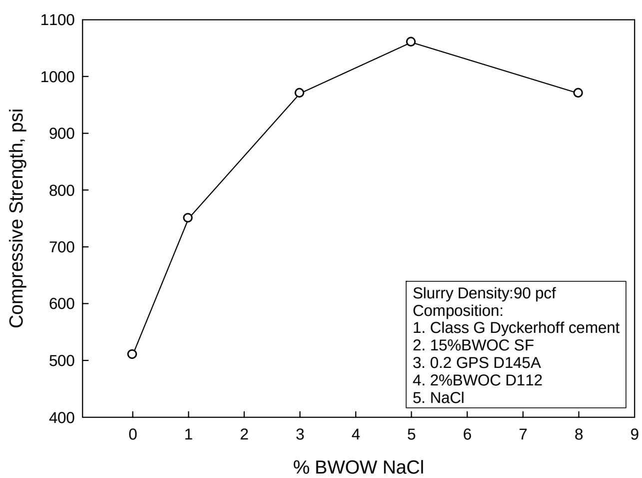

Figure 17. Effect of NaCl on Compressive Strength of 90 pcf cement slurries.

On the side wall of 1% BWOW NaCl set cement cubes of the CS test, the signs of non-hydrated cement appear and air trapping around them is observed, but the bonding between the cement and permeability mold has a good condition and the water permeability of the mentioned slurry is 0.00168 md and 1 mm of cement height in the permeability mold is reduced. Height reduction of cement in the permeability mold is a sign of FW of slurry. In higher concentrations of NaCl, the amount of air trapping and non-hydrated cement on the side wall of the set cement cube becomes more and more and bonding between the cement and the permeability mold fails, hence the cement is separated easily from the permeability mold. Around the cement plug it is full of non-hydrated cement and air voids. To remove the air traps from the slurry, the cement slurries are conditioned for 1 hr in an atmospheric consistometer at BHCT before molding, but no difference in conditions of bonding between cement and permeability mold are observed. When NaCl is added to the cement the slurry bonding problem shows itself. Because of this problem, we do not select 15% BWOC silica fume cement slurry compositions for 90 pcf cement slurry. Cement in the water permeability mold has height reduction, therefore, for eliminating this problem, the silica fume percentage is increased to 20% BWOC for the next step of investigation.

Table 8 shows the composition number, cement slurry composition, mixing method and density of the cement slurries that contain 20% BWOC silica fume.

In Fig. 18, the free water at 80 °F of 15% and 20% BWOC silica fume are compared. The FW of 15% BWOC SF is higher than 20% BWOC SF. 15% BWOC SF cement slurry has 24 cc excess water according to the recommendation of D.T Mueller and R.L Dillenbeck[1]. The FW of 15% BWOC SF and 0.2 GPS D145A is 4 times higher than FW of 20 % BWOC SF and 0.3 GPS D145A. This excess water of 15% BWOC SF cement slurry shows itself when dispersant additive is added to slurry.

After several tests on 20% BWOC SF, D145A and D112, 0.3 GPS of D145A and 1.5% BWOC D112 (composition No. 83) are selected. Experimental results of cement slurries that contain 20% BWOC SF are tabulated in Table 9. Due to the bonding failure problem of 15% BWOC silica fume cement slurry when NaCl was added; BA-100S is used in the cement slurry to solve the bonding failure.

Table 8. Composition No., cement slurry composition, mixing method and density of 90 pcf and 20% BWOC SF cement slurry.

Comp. No.	Composition	Mixing method	Density (*pcf*)
77	G Dyck. cement 20%BWOC SF	Wet blend	90
78	G Dyck. cement 20%BWOC SF 0.1 GPS D145A	1 W.b. 30 *sec* 2 W.b. 1 *min*	90
79	G Dyck. cement 20%BWOC SF 0.2 GPS D145A	1 W.b. 30 *sec* 2 W.b. 1 *min*	90
80	G Dyck. cement 20%BWOC SF 0.25 GPS D145A	1 W.b. 30 *sec* 2 W.b. 1 *min*	90
81	G Dyck. cement 20%BWOC SF 0.3 GPS D145A	1 W.b. 30 *sec* 2 W.b. 1 *min*	90
82	1 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 1%BWOC D112	4 mix 1 *min* 1 W.b. 30 *sec* 3 W.b. 30 *sec* 2 W.b. 30 *sec*	89.5
83	1.5 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 1.5%BWOC D112	4 mix 1 *min* 1 W.b. 30 *sec* 3 W.b. 30 *sec* 2 W.b. 30 *sec*	89.5
84	1.5 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 1.75%BWOC D112	4 mix 1 *min* 1 W.b. 30 *sec* 3 W.b. 30 *sec* 2 W.b. 30 *sec*	89+
85	1.5 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 2%BWOC D112	4 mix 1 *min* 1 W.b. 30 *sec* 3 W.b. 30 *sec* 2 W.b. 30 *sec*	89
87	1.5 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 1.5%BWOC D112 1%BA-100S	5 mix 1 *min* 1 W.b. 30 *sec* 4 W.b. 30 *sec* 2 W.b. 30 *sec* 3 W.b. 30 *sec*	89.5
88	1.5 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 1.5%BWOC D112 2%BA-100S	5 mix 1 *min* 1 W.b. 30 *sec* 4 W.b. 30 *sec* 2 W.b. 30 *sec* 3 W.b. 30 *sec*	89.5
89	1.5 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 1.5%BWOC D112 3%BA-100S	5 mix 1 *min* 1 W.b. 30 *sec* 4 W.b. 30 *sec* 2 W.b. 30 *sec* 3 W.b. 30 *sec*	89.5-
90	1.5 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 1.5%BWOC D112 4%BA-100S	5 mix 1 *min* 1 W.b. 30 *sec* 4 W.b. 30 *sec* 2 W.b. 30 *sec* 3 W.b. 30 *sec*	89
91	1.5 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 1.5%BWOC D112 5%BA-100S	5 mix 1 *min* 1 W.b. 30 *sec* 4 W.b. 30 *sec* 2 W.b. 30 *sec* 3 W.b. 30 *sec*	89-
92	1.5 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 1.5%BWOC D112 3%BA-100S 2%BWOW NaCl	6 mix 1 *min* 1 W.b. 30 *sec* 4 W.b. 30 *sec* 2 W.b. 30 *sec* 3 W.b. 30 *sec* 5 W.b. 30 *sec*	89.25
93	1.5 *cc* FP-6L G Dyck. cement 20%BWOC SF 0.3 GPS D145A 1.5%BWOC D112 3%BA-100S 4%BWOW NaCl	6 mix 1 *min* 1 W.b. 30 *sec* 4 W.b. 30 *sec* 2 W.b. 30 *sec* 3 W.b. 30 *sec* 5 W.b. 30 *sec*	89.5

Table 9. Experimental results of cement slurries that contain 20% BWOC SF.

Comp. No.	Density *pcf*	Rheological Properties, *cp* , *lbf/100ft*^{2}				Fluid Loss, *cc/30min*		Free Water		Compressive Strength		Water Permeability, *md*			
		PV	YP	10 *sec* GS	10 *min* GS	Standard	HPHT	%	Note	*psi*	Note	50 *psi*	100 *psi*	150 *psi*	Note
77	90	19.5	36	14	29	468.5		0*		1530 1690	High air trap	No flow No flow No flow	No flow 0.00074 No flow	0.00039 0.00153 0.00086	
78	90	11.25	19.25	7.5	17	385.1		0.8*		1430	Low air trap	No flow	No flow	No flow	
79	90	7.875	8.625	7	10	319.7		0.8*							
80	90	5.625	4.875	6.5	6.5	271.2		0.8*							
81	90	5.625	1.875	4.5	6	222.5		0.8*		1150	Low air trap	No flow	0.00079	0.0008	2 mm height reduction
82	89.5	17.625	3.375	6	13	65.3		0.8	Hard, very Thin and white layer on top	600	Air trap	No flow	No flow	No flow	
83	89.5	28.125	5.375	6	16	41.8		0.8	Hard, very Thin and white layer on top	670 670	1 hr conditioning, 1 mm height decrease	No flow No flow	No flow No flow	No flow No flow	1 hr conditioning, 1 mm height reduction
84	89+					27.6									
85	89	47.25	9.75	6	16	22	47	<0.2	Hard, very Thin and white layer on top	-	Specimens have form but be soft	0.0048	0.0067	0.0062	Cement is set in the cooling bath
87	89.5	30.75	5.25	6	18		70	0.4	Hard, very Thin and black layer on top	930	1 hr conditioning,	No flow	No flow	No flow	1 hr conditioning
88	89.5	29.625	5.375	6	17		67	0.4	Hard, very Thin and black layer on top						
89	89.5-	29.625	6.625	6.5	16.5		65	0.4	Hard, very Thin and black layer on top	870 940 990	1 hr conditioning, Using grease for releasing	No flow No flow	No flow No flow	No flow No flow	1 hr conditioning
90	89							0.4	Hard, very Thin and black layer on top						
91	89-	30.375	6.125	6	17		63	0.4	Hard, very Thin and black layer on top	1000	1 hr conditioning, Using grease for releasing	No flow	No flow	No flow	1 hr conditioning
92	89.25	28.313	3.688	4	14		79	0.5	Hard, very Thin and black layer on top	960 890	1 hr conditioning, Using grease for releasing	No flow	No flow	No flow	1 hr conditioning
93	89.5	26.25	3.25	3	12			0.5	Hard, very Thin and black layer on top	1030	1 hr conditioning, Using grease for releasing.	-	-	-	1 hr conditioning, bonding failure

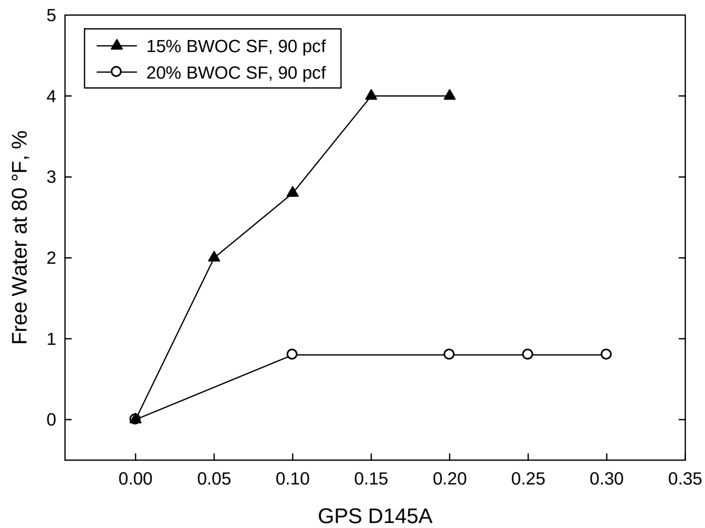

Figure 18. Effect of D145A on Free water of 15% and 20% BWOC silica fume.

Effect of BA-100S on rheological properties is shown in Fig. 19. According to the data sheet of BA-100S, BA-100S has no effect on the rheological properties. The PV, YP, 10 sec and 10 min of BA-100S cement slurries show this point.

Fig. 20 shows the effect of BA-100S on HPHT fluid loss and free water at BHCT. HPHT fluid loss of BA-100S slurries, is, initially, slightly increased when 1% BWOC BA-100S is added and then decreased to less than HPHT fluid loss of cement slurry without BA-100S. BA-100S decreases the FW at BHCT from 0.8% to 0.4%. When FW at BHCT is less than 1%, its FW at 80°F will be zero.

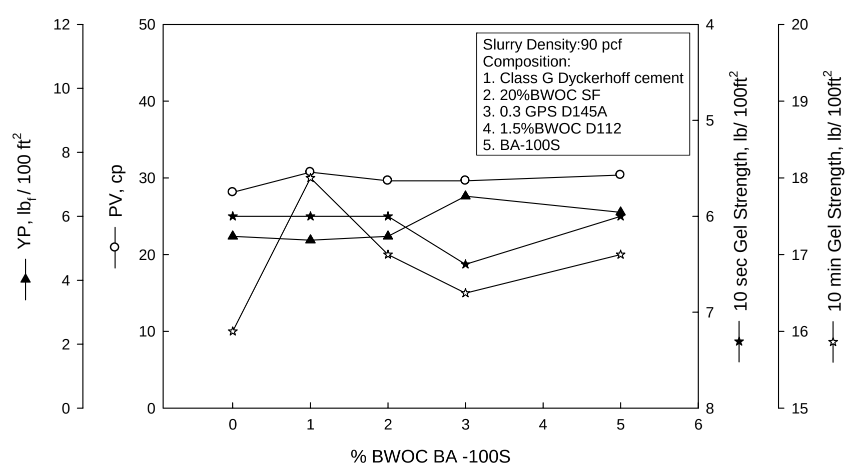

Figure 19. Effect of BA-100S on Rheological properties of 90 pcf (20% BWOC SF) cement slurries.

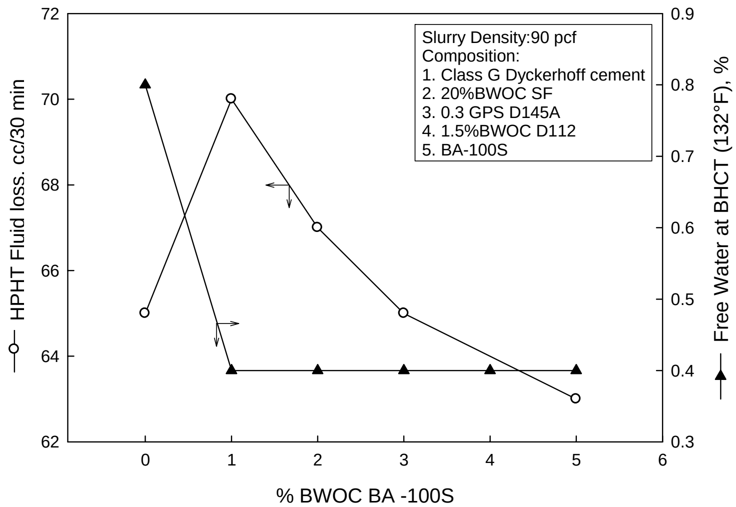

Figure 20. Effect of BA-100S on HPHT Fluid loss and Free Water of 90 pcf (20% BWOC SF) cement slurries.

BA-100S increases the CS of set cement (see Fig. 21). CS of set cement increases from 670 to 1000 (psi) when 5% BWOC BA-100S is used. Note that the water permeability of all BA-100S concentrations is zero. Based on the data sheet of BA-100S, it enhances the CS of set cement. Note that two sets of CS test failed because the cement cubes stick hard to the inner walls of the mold and the cement cubes are broken, but the cement cubes do not separate from the mold. One the third try, the mold walls are covered with a thin layer of grease to prepare an intact cubic sample for testing.

According to the experimental results that are shown in Fig. 19 through 21, 3% BA-100S (composition No. 89) is selected. The compressive strength of composition No. 89 is 960 psi and for reaching more compressive strength in 24 hr curing, NaCl will be added as an accelerator.

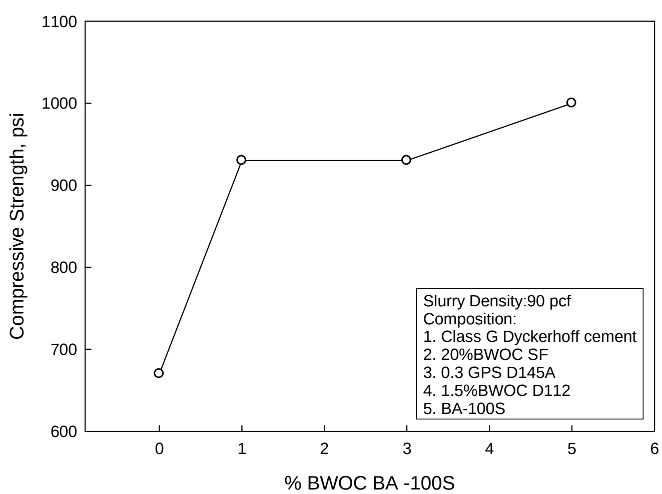

Figure 21. Effect of BA-100S on Compressive Strength of 90 pcf (20% BWOC SF) cement slurries.

Fig. 22 shows the effect of NaCl on rheological properties. Rheological properties of slurries are slightly decreased with NaCl concentration increasing. NaCl increases FW and FL of slurry because of its interaction with fluid loss control and dispersant additives. HPHT fluid loss of the mentioned slurry is increased from 65 to 79 (cc/30min) when 2% BWOW NaCl is added to the cement slurry. The FW at BHCT of slurry are slightly increased to 0.5%.

NaCl increases the CS of the set cement because it has an accelerating effect. CS of set cement is slightly increased from 930 to 1030 psi when 4% BWOW NaCl is added to the slurry. Water permeability of 2% BWOW NaCl is zero, but bonding failure occurs when 4% BWOW NaCl is used.

Regarding the detrimental effect of NaCl on FL, FW and bonding failure, NaCl is eliminated from a cement composition of 90 pcf cement slurry. At the end of this series of tests composition No. 89 is selected. Fig. 23 shows the HPHT thickening time test curves of this cement slurry composition. The thickening time of slurry is more than 270 min because its high WCR (1.38) and the side effect of the dispersant and fluid loss control additives. According to the data sheet of BA-100S; it has an anti-gas migration effect. Also, silica fume is an anti-gas migration additive[6,7,8], therefore this cement slurry composition may control the gas migration. Table 10 shows the solution stability of composition No. 89. Stability of cement slurry solution in rest condition is clear.

Rheology of cement slurry after mixing and conditioning are different. Temperature and pressure affect the rheological properties of fluids. Cement slurry is a time dependent fluid and the rheological properties of cement slurry are increased with time because of the increasing consistency of the cement slurry. According to the thickening time graph of cement slurry composition No. 89; the consistency of cement slurry does not increase due to the setting of the cement slurry after 35 min conditioning. It is obvious that the rheological properties of cement slurry decrease when the temperature is increased. Rheology curves of composition No. 89 after mixing and after conditioning in BHCT are shown in Fig. 24.

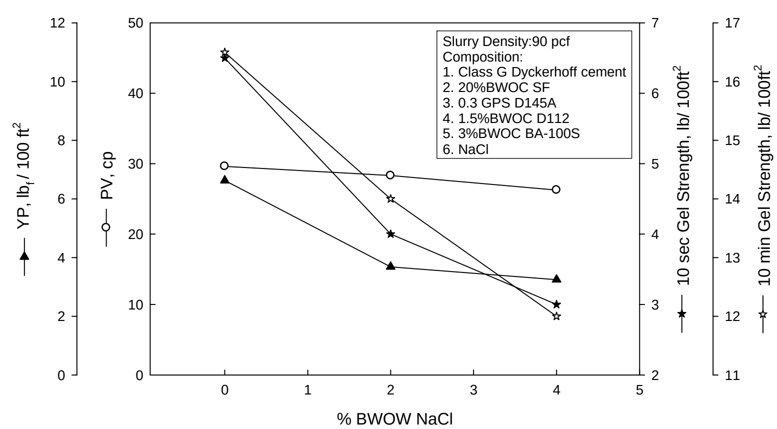

Figure 22. Effect of NaCl on Rheological Properties of 90 pcf (20% BWOC SF) cement slurries.

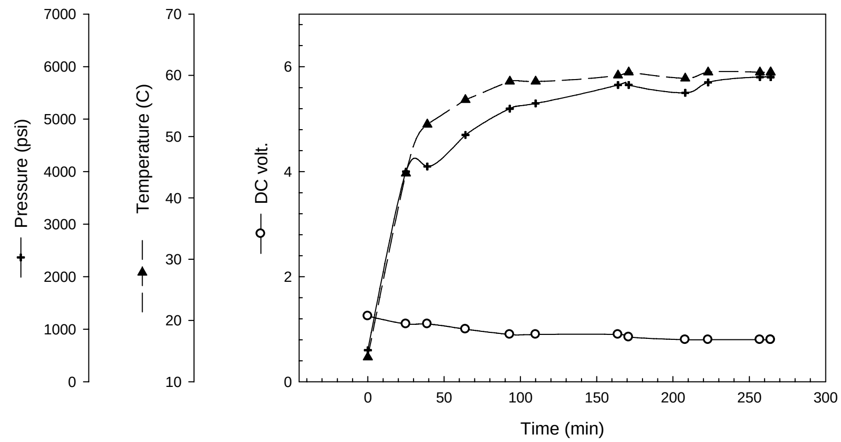

Figure 23. Thickening time test of composition No. 89 cement slurry.

Table 10. Solution stability of composition No. 89.

Time (*min*)	Separation (*cc*)
5	0
30	0
45	0
60	0

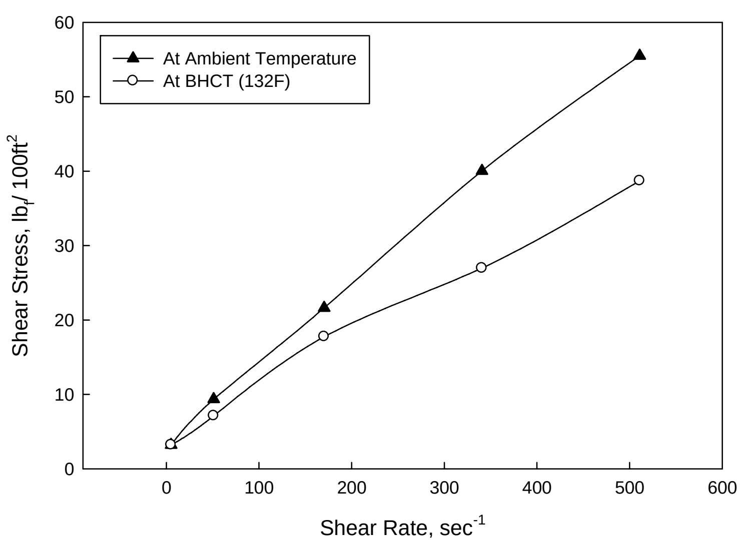

Figure 24. Shear stress versus shear rate of composition No. 89 slurry at ambient temperature and BHCT.

Conclusions

1. Silica fume has a singular effect for improving cement slurry and set cement properties, such as decreasing free water and fluid loss, increasing compressive strength and significantly decreasing water permeability in high WCR slurry. Regarding the benefits of silica fume in cement slurry, composition No. 89 is formulated for preparing 90 pcf cement slurry; its properties are tabulated in Table 9.
2. WCR increase has a detrimental effect on compressive strength, water permeability and slurry stability.
3. When WCR is high, NaCl has a detrimental effect on the bonding of the cement and permeability mold.
4. BA-100S (bonding agent) does not affect the rheological properties, but this additive improves bonding and compressive strength and decreases water permeability, fluid loss and free water.

Nomenclature

AF	Anti Foam
API	American Petroleum Institute
B_c	Burden Unit of Consistency
BHCT	Bottom Hole Circulating Temperature, °F
BHST	Bottom Hole Static Temperature, °F
BWOC	By Weight of Cement
°C	Degree Centigrade
cc	cubic centimeter
cp	Centipoise
CS	Compressive Strength, psi
°F	Degree Fahrenheit
FL	Fluid Loss, cc/30min
FW	Free Water, %
GS	Gel Strength, lb/100ft^2
gr.	gram
HPHT	High Pressure and High Temperature
HSR	High Sulfate Resistance
in.	inch
md	10^{-3} darcy
min	minute
pcf	Pound per Cubic Feet
PV	Plastic Viscosity, cp
psi	Pound per Square Inch
rpm	round per minute
sec	second
SF	Silica Fume
TG	Temperature Gradient (°F/100 ft)
TT	Thickening Time
WCR	Water to Cement Ratio
YP	Yield Point

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