

DEVELOPING A CEMENT FORMULATION FOR GAS MIGRATION CONTROL
DURING CEMENTING FOR HIGH-PRESSURE/HIGH-TEMPERATURE WELLS

A Thesis

by

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Submitted to the Office of Graduate and Professional Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

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December 2019

Major Subject: Petroleum Engineering

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ABSTRACT

Cementing high-pressure/high-temperature (HP/HT) gas wells is a challenging and expensive job in the oil and gas industry. Gas migration through the cement column has major safety, environmental, and economic concerns. Problems such as weighting material segregation, excessive fluid loss, and shortages of water available for cement hydration can produce high-porosity cement structures that are prone to progressive gas migration. This work aims to increase hydration efficiency, develop sufficient gel strength, reduce fluid loss, and enhance mechanical properties to produce gas-migration-resistant HP/HT cement slurries. This work developed an HP/HT cement slurry with high resistance to gas migration by optimizing a new additive, Maxcrete, which replaces multiple common gas-migration-control additives. The experimental slurry was prepared and evaluated following the API RP 10B-2 procedures. Fluid loss was optimized through various iterations of additive combinations at different concentrations. The cement hydration process was monitored over 24 hours using computed tomography (CT) scans and compressive-strength analysis.

The optimized cement slurry with the new additive showed superior enhancement in the main properties that control the gas migration process. A class G cement slurry with 2.5 lb/100 lb by weight of cement (BWOC) of the new additive showed an enhancement in the cement hydration capacity, fluid loss control, and compressive strength compared to a slurry without the additive. These improvements resulted from coating the cement particles with a layer that is capable of attracting more

water molecules, which prevented quick free-water separation and provided for better and faster hydration. The suggested mechanism was supported by gas migration evaluation and rheology assessment. The results of these tests showed rapid development of an initially low gel strength that mitigated the initial induction of gas channels in the cement body during the setting time. The CT-scans showed that the cement blocks were more hydrated and, with time, demonstrated a reduction in the cube porosity.

This work introduces and evaluates a new additive with the capability to replace multiple additives in cement slurries for better gas-migration control. The suggested chemistry has the potential to work synergistically with fewer additives and deliver superior cement properties for HP/HT wells.

DEDICATION

To my family, teachers, and friends who gave me understanding, patience, and strength
to produce this work.

ACKNOWLEDGEMENTS

I thank my committee chair, Dr. Hisham Nasr-El-Din, for his constant support, guidance, and supervision. I also extend my gratitude to my committee members, Dr. Jerome Schubert and Dr. Mahmoud El-Halwagi, for their guidance, support and wisdom. I thank Gia Alexander for helping me with my thesis writing. I also thank all Petroleum Engineering department staff members who helped me with my research. I thank Dr. Tom Harper and the team at Maxflo Chemicals for trusting me with an important role with their project.

I also thank my colleagues and friends who helped me with my education and research and made my learning experience at Texas A&M memorable.

Finally, I thank my family for being my backbone and my inspiration.

CONTRIBUTORS AND FUNDING SOURCES

Contributors

This work was supervised by the thesis committee chair Dr. Hisham Nasr-El-Din of the Harold Vance Department of Petroleum Engineering at Texas A&M University.

All other work conducted for the thesis was completed independently by the student.

Funding Sources

This work was funded by Maxflo Oilfield Chemical Solutions.

NOMENCLATURE

API	American Petroleum Institute
BWOC	By Weight of Cement
CaCO ₃	Calcium Carbonate
CT	Computed Tomography
HU	Hounsfield Unit
Mn ₃ O ₄	Manganese Tetroxide
PPG	Pounds per Gallon
RP	Recommended Practice
RPM	Rotations per Minute

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1. INTRODUCTION

Gas migration is the most frequent, critical, and dangerous problem in well cementing and is more prominent in deep wells across gas formations. This gas communication causes pressure buildup in the annular region between the production path and the intermediate casing that can lead to cement failure, which, in turn, creates more paths for gas migration (Carter and Slagle 1972). Cement hydration is a series of complex chemical process between water and cement that provides it the strength. Hydration process is associated with volume shrinkage. The volume of the hydration process has a smaller volume compared to the volume of the reactants. This reaction leaves intergranular porosity in the set cement (Bois et al. 2011). In gas-producing deep formations, gas can migrate from the rock formation through the pore spaces and fractures in the cement column all the way up to the surface. These pathways are shown to be the primary cause for gas migration into ground aquifers. Cement failures are more common than casing failures, which leave even more open pathways for gas migration (Jacobs 2014). If this gas leaks into the atmosphere, it can damage the environment and contribute to the greenhouse effect (Dusseault, Gray, and Nawrocki 2000). If this gas contains sulfurous compounds, it can make the groundwater non-potable. Methane can also be harmful as it can enter household system when the natural gas taps are turned on. Gas migration can also cause economic and safety hazards such as higher annular surface pressure, high water cuts or gas cuts, blowouts, poor zonal isolation, loss of gas to nonproductive zones, poor stimulation, low producing rates, etc. The period where

cement develops gel strength is when the cement loses its ability to transmit hydrostatic pressure. During this period, gas can invade the cement structure and cause problems. Other mechanisms like high fluid loss, high cement permeability, and formation of micro annulus due to poor bonding between cement and casing/formation (Pour and Moghadasi 2007).

Cementing is an essential step in well completion because it mitigates gas migration. The cementing process serves two very important purposes: supporting the casing and providing separation between different zones. Cement helps the casing stay in its place in the wellbore and provides a support to the casing string. Without the cement, the casing string would just hang from the surface. To keep fluids from one zone entering into another, a seal must be provided in the annulus in the form of a competent cement job. This seal must be provided during the initial settling phase when the cement is in slurry form, as well as when it develops strength and becomes a hard structure (Nelson 1990). Primary cementing also protects the casing from plastic formations and corrosive formation fluids. Without the cement, plastic formations like salt structures will deform and may cause damage to the casing. Corrosive fluids, such as water containing salts such as sodium, calcium, magnesium, or water with dissolved CO₂, can damage the casing (Shakirah 2008).

Cementing is a complex process, especially for deep wells with extreme conditions. Gas migration becomes more evident in deep-well formations across gas intervals, where the gas causes pressure buildup in the annulus between two casings (Carter and Slagle 1972). Various conditions affect the ability of cement to control gas

migration, including improper hydration, fluid loss, lack of mechanical strength, etc. These problems in the field must be dealt with to achieve a successful cement application.

Salt required in cement formulations to meet drilling fluid concentrations may also cause reduced effectiveness of additives and might cause polymers with ionizable hydrophilic groups to be coiled due to charge shielding (Brothers and de Blanc 1989). This may cause the cement slurry to develop high viscosity and settling issues.

Cementing deep wells with high-pressure and high-temperature (HP/HT) conditions can be an even more difficult job. Changes in pressure and temperature conditions can damage the cement structure and cause it to fail. The cement and/or the casing can expand or shrink with the pressure and temperature changes according to their mechanical properties and may develop shear failure, tensile failure, or micro-annulus (Al Yami et al. 2018). This failure of the primary cement can create migration paths for fluids from a higher-pressure reservoir zone to move to another zone or even the surface. Secondary cement jobs to rectify the primary cement job failure can be challenging and expensive.

Various studies have investigated what causes cement to allow gas migration. For example, variation of downhole pressure and temperature can induce stresses in the cement structure. This induced stress can cause damage to the cement structure and may lead to the creation of paths for gas to migrate through the cement column (Thiercelin et al. 1998). Creation of a micro-annulus or micro-fractures is caused by the inability of the cement to maintain overbalance pressure over gas formations when the slurry

changes from a liquid to a solid phase. During this phase change, the slurry will behave like a porous media with the non-reacted water in the pore space. The pressure exerted would be equivalent to the hydrostatic pressure by the water column (Talabani and Hareland 1995).

As the cement and matrix water continue to react, micro-cracks will connect to each other and allow the gas to leak from the cement structure over time. This process takes about a year. The distribution of hydrostatic pressure in the annulus depends on the cement gel strength. With higher gel strength, the distribution ability of the slurry reduces (Wilcox, Oyenein, and Islam 2016). Fluid loss from the cement slurry causes volume loss; this volume loss can allow pressure loss in the cement column. The shear resistance of the cement slurry sets a maximum pressure loss limit. The permeability of the cement allows the volume loss in the cement to be transmitted through the entire column (Sabins and Wiggins 1997). This volume loss is caused by fluid loss or hydration volume loss. When the hydrostatic pressure exerted by the cement column is higher than the formation pressure, lack of a proper fluid-loss control causes the slurry to lose water and develop a filter cake across the permeable zone, blocking the annulus. Below this point the effective hydrostatic pressure is reduced due to the filter cake. The pressure loss in the cement allows gas to flow from the formation to the cement column. A differential pressure of only 1 psi between the formation and the cemented annulus can initiate gas migration (Christian et al. 1976).

Fluid loss can also lead to premature dehydration of the cement, which is the primary reason for gas migration. Mobility of the fluids in the pore space of the cement

after it has begun to take load is also an important factor. As long as the fluid inside is mobile, it can be eventually displaced by the formation gas (Cheung and Beirute 1985). Poor hydration can also lead to a cement structure with low mechanical strength that is prone to develop fractures that create paths for the gas to flow. The recommended fluid loss for high-density cement should be less than 50ml/30 min according to the API standard test to achieve desirable cement properties (Al Yami 2015). However, fluid loss might offer some benefits such as increased density, reduction in thickening time, and improvement in compressive strength as long as the fluid loss is within a reasonable limit (Daccord and Baret 1994).

The cement for deep wells must be of high density to withstand high formation pressure. There are two ways to achieve high density: reduce the amount of water or increase the amount of high-density solids. Manganese tetroxide, hematite, calcium carbonate etc. are primarily used in the cement formulation to provide higher density. Silica is also used with the cement to reduce the required volume to prepare a given slurry, at the same time preventing strength retrogression at high temperatures. However, when the calcium-to-silica ratio is reduced, higher permeability is introduced in the crystalline structure, which is detrimental. The homogeneity of the initial dry blend of the solids in the field can also be related to the compressive strength and hardness of the cement (Noik and Rivereau 1999). Using either method, the water-to-solids ratio is smaller for high-density cement slurries. Cement properties are sensitive to this water-to-solid ratio, and any decrease in the ratio can impair the cement placement operations. (Baret 1988).

Paths for fluid migration can be created if cement slurry is not placed in the entire annulus, and/or if the cement sheath fails, either due to shrinkage or loss of structural integrity (Reddy et al. 2009). Reducing the amount of water gives rise to an even smaller quantity of water available for cement hydration, as part of it is also absorbed by the solid additives. Increasing the solid portion of the slurry also causes problems like settling and formation of a cement structure with high permeability. High-density weighing materials tend to settle and create density difference throughout the column. This leads to a cement structure with less mechanical strength. A higher amount of solid weighing material can also cause the cement structure to have high permeability which can provide paths for gas to migrate through the cement column (Al Yami 2015). In addition to the design of cement slurry, practices like mud conditioning, spacer design, casing centralization, proper displacement efficiency, etc. are also required to achieve a proper seal in the annulus and prevent gas migration (Mata, Diaz, and Villa 2006).

Various studies have been done to improve the gas migration control of cement formulation for deep wells. Al Yami (2015) worked with different weighing material concentrations and combinations to develop a formulation that gives the least fluid loss and achieved the lowest fluid loss with using hematite, magnesium tetroxide, silica sand and silica flour. A range of cement additives are required to achieve the desired fluid loss properties. Al Yami (2015) in his study also focused on the cement in its slurry phase and did not combined it with the mechanical strength testing. Haidher (2008) suggested the use of silica flour and fumes as bonding agent to fill the gap between

cement pores. Al-Saeedi et al. (2011) suggested the use of only manganese tetroxide as a weighing material and liquid fluid loss additive for deep well application. The liquid fluid loss agents has to be mixed with water and can be difficult to mix at high concentration. A non-viscosifying fluid loss additive is required which might increase the cost of the additive. Roshan and Asef (2010) showed that use of a cement formulation using CMC at optimum concentration can reduce the fluid loss while maintaining a good compressive strength. However, CrCl_3 is required in this blend to achieve the acceptable fluid loss level which is toxic and non-environmentally friendly. The CMC can remain undissolved and reduce the compressive strength of the cement. A simple approach in cement formulation is required that can produce a cement formulation that has a good gas migration control and is economical and field applicable at the same time.

It can be summarized from the above literature review that to achieve a gas migration control; improved fluid loss control of the cement slurry and high mechanical strength of the set cement is required. This can be achieved together with improvement in hydration reaction between water and cement. A novel chemical additive Maxcrete is introduced that can improve the hydration reaction. Computed tomography scan is used to visualize the hydration improvement by correlating it with increased specific gravity of the cement formulation. Additives are required in the cement to achieve desirable cement properties. At high densities, this requirement might increase, which can also add to the cost. A better cement formulation that can avoid gas migration problems without adding too much cost is needed in the oil and gas industry. This study aims at observing

the effect of this new additive on the cement formulation and the interaction between Maxcrete, cement, water, and additional cement additives. The study also aims at optimizing the cement formulation to achieve gas migration control by reducing fluid loss and improving mechanical strength for high density cement formulation.

2. OBJECTIVES

Gas migration through cement is a serious issue that has challenged the petroleum industry for a long time. The literature has developed a primary understanding of how different factors play a role in cement properties and ways to improve the cement formulations. A simple solution is required that improves the cement properties. To work toward this simple solution, the present research has the following objectives:

1. Understand the effect of cement additives on the primary cement properties such as fluid loss, compressive strength, free water, cement rheology, etc.
2. Observe the effects of adding Maxcrete on the cement properties and understand the limitations.
3. Observe the effects on the hydration of the cement using Computed Tomography (CT) scans.
4. Improve and optimize the cement formulation to improve its properties and allow for better gas migration control.

3. EXPERIMENTAL STUDIES

3.1. Mathematical Foundations

This research tested the effects of a new chemical additive, Maxcrete on the cement properties that promotes gas migration in oil and gas wells. The primary experimental study was divided mainly into three steps. First, different properties of a cement slurry were evaluated, second the mechanical strength of the set cement was evaluated, and finally a CT scan analysis was performed to visualize the hydration effects of the new additive within the cement. To maintain a standard in the testing, API RP-10b 2 was followed. From these primary studies, this research aims to optimize the cement formulation to improve the performance of the prepared slurry.

To distinguish the results of the additive, a base-case cement formulation without Maxcrete was tested as a control. Variation in cement slurry density was taken into account to understand its effect on cement properties. The cement density was calculated by following the API method (API RP 10b-2). All the additives were measured as a percentage by weight of cement (% BWOC), and then the amount of water required to meet the density required is calculated as follows (Eq. 1).

$$cement\ density = \frac{(mass\ of\ cement\ and\ additives)+(mass\ of\ water)}{(volume\ of\ cement\ and\ additives)+(volume\ of\ water)} \dots\dots\dots (1)$$

The mass of the water required can be calculated by substituting the known relation of (Eq. 2), as follows:

$$volume = \frac{mass}{density} \dots\dots\dots (2)$$

With the known values of the cement mass and the concentration of different additives in the form of % BWOC, the mass values for every additive except water can be determined. The density for each additive was known and the desired cement density was a design input, leaving only the mass of water as an unknown variable to be solved from the following simplified equation (Eq. 3):

$$M_w = M_A * \frac{\rho_w}{\rho_A} \left(\frac{\rho_A - \rho_T}{\rho_T - \rho_w} \right) \dots\dots\dots (3)$$

where, M_w is the mass of water, M_A is the sum of the mass of the cement and additives, ρ_w is the density of water, ρ_A is the average density of cement and additives (sum of mass/ sum of volume), and ρ_T is the desired total cement slurry density

The units for mass, volume, and density should be consistent so that the unit of density is given as a unit of mass divided by a unit of volume. This research used three different densities- 16, 17.4, and 18.7 ppg. Cement formulations at these high densities require some additional additives to achieve the desirable cement properties.

3.2. Materials

3.2.1. Cement

Class G cement is the basic cement class used for a wide range of applications. It was used in this study as it is the most common cement class used in the oil field. The specific gravity of the class G cement is 3.14.

3.2.2. Silica Sand and Silica Flour

Silica is used in the cement formulation to serve two purposes. First, it prevents strength retrogression in the cement structure at higher temperature (Nelson 1990). Second, silica is cheaper than cement, and using silica instead of cement lowers the cost of the cementing job by reducing the amount of cement needed to produce a certain volume of slurry. Silica sand of 100 mesh size and silica flour of 15 μm average particle size was used in this research.

3.2.3. Fluid Loss Additive

Fluid-loss additives prevent the aqueous phase of the slurry from filtering out and escaping into the formation. A desirable fluid-loss level is <50 ml/30 min. This low fluid loss ensures that the formulated cement has enough water to hydrate the cement. Special industrial water soluble-polymers are used that increase the slurry viscosity and reduce permeability of the filter-cake.

3.2.4. Dispersant

Cement slurry is a suspension of solid particles in water. When the solid particle concentration is high, a dispersant is used to break up the solids and get a cement slurry with low viscosity which improves the pumpability of the cement. The rheology of the cement slurry depends on the properties of the water. Dispersants adjust the particle surface charge. Sodium lignosulfonate is used, which is a polyanion. Cement slurry has positively and negatively charged particles that interact with each other to make the

slurry less pumpable. The polyanions attach to the positively charged particles and suppress the particle interactions to make the slurry more pumpable (Nelson 1990).

3.2.5. Calcium Carbonate

Calcium carbonate powder reduces the cement requirement in a formulation and improves the economy of the slurry. This powder was used for this purpose in the present research. Calcium carbonate has a specific gravity of 2.71.

3.2.6. Manganese Tetroxide

Manganese tetroxide has a high specific gravity (4.88) and is a small, (average particle size of 1 micron) spherical weighting material that is used to achieve high density required for the cement application. The size and shape of manganese tetroxide particles help to reduce friction, resulting in a slurry that has lower viscosity (Al Yami 2015). The inertness of manganese tetroxide makes it more suitable for the cement application.

3.2.7. Hematite

Hematite (Fe_2O_3) is a common weighing material in oilfield cement that has a high specific gravity (4.6) and is supplied in fine particle form. For the hematite sample used, 99% of the particles passed through a 325 mesh sieve (44 micron). Dispersant is required with hematite as it may cause increased viscosity (Nelson 1990).

3.2.8. Maxcrete

Maxcrete is a combination of inorganic and organic solutions that reacts with Portland cement to promote more complete hydration, thus creating a more impermeable structure. Increased hydration results in many improvements to the cement, including early setting and ultimate strength increase.

3.3. Experimental Procedure

3.3.1. Slurry Preparation

The slurry was prepared using a standard API blender. The additives were added at 4000 rotations per minute (rpm) and mixed at 12,000 rpm for 40 seconds. The order of addition of the additives also affects the cement formulation as the cement water reactions are affected. The standard practice in the field requires all the dry additives to be dry blended separately before adding into water. In the primary test, addition of the solid additives followed a step by step addition approach to observe effect of an additive on the ease of mixing of the cement slurry. For the improvement tests, a field resembling approach of dry blending the dry additives was used.

3.3.2. Fluid-Loss Test

Fluid loss measurement is required to assess the ability of the cement to hold its liquid contents. A fluid-loss test cell is prepared with a bottom cap, followed by a rubber gasket, a wire mesh, a 50-micron filter paper, and another rubber gasket in that order from bottom to top. The cell is assembled and filled with the prepared cement slurry up

to a mark that leaves ¼ inch from the top. This assembly (**Fig. 1**) is placed on the fluid-loss measurement arrangement, and a cap is used to securely close the cell from the top.



Fig. 1—Fluid loss cell assembly.

Nitrogen is injected from the top cap into the cell at 100 psi pressure. The filtrate produced is collected from the bottom. The standard test run time, as mentioned in the API RP 10b-2, is 30 min. If the gas breakthrough occurs before 30 min, a correction to the collected fluid volume is made according to API RP 10b-2.

3.3.3. Free Water Test

Free water is detrimental to the cement formulation as it is water that has not reacted with the cement. This free water can leak into the formation and create pores within the cement structure. The free water test uses a graduated cylinder of 250 ml which is filled with the cement slurry and allowed to settle for one hour. The cylinder is kept in a vertical orientation. After one hour, the amount of free water on the top is recorded. It is desired that the cement slurry have no free water. Non-zero free water can also suggest improper mixing of cement.

3.3.4. Compressive Strength Test

Cement compressive strength is a measure of cement structure's mechanical strength. A cement with higher compressive strength is less likely to develop cracks and fractures. A mold is used to prepare three casts of cement cubes with 2-in. face dimensions. After preparing the cement slurry, it is poured halfway into the mold and air bubbles are removed by a puddling rod. The same step is repeated to completely fill the mold, and excess cement is carefully removed using a spatula to create casts with uniform sides. The molds are covered using a top cap to avoid cement and water contact. The cement molds are cured in a water bath at 60°C for 24 hours. The cured cement casts are removed from the mold and crushed using a uniaxial compressive strength machine. The Ramp up rate for the machine is 67 psi/s. The breaking point is achieved when the compressive load reduces to 50% of the peak load. The peak load is measured as the compressive strength. The average compressive strength of the three casts is taken as the compressive strength of the batch.

3.3.5. Computed Tomography Scan (CT Scan)

CT scan is a useful tool to visualize the hydration process of the cement slurry. CT number that is measured in Hounsfield Units or HU gives a measure of a particle's specific gravity. A hydrated cement has higher CT number compared to improperly hydrated. Cement formulation was molded using a cylindrical mold with 6 in height and 1 1/2 in diameter. The cement was kept at 60°C for 24 hours to allow the slurry to solidify. After 24 hours, the cement casts were scanned using a CT scan machine. The

data generated from the CT scan was then analyzed using ImageJ and 3Dslicer software and visual images were processed. The results were in the form of CT numbers that are directly correlated with the specific gravity of the scanned material. The higher the CT number, the higher the specific gravity. The CT scan image was then processed using ImageJ software and a CT number window corresponding to a color range was applied to get the final results.

4. PRIMARY RESULTS AND DISCUSSION

The primary tests were performed on the cement formulation to understand the cement formulation and the effect of the chemical additive Maxcrete on the cement properties. Different tests were performed with the cement slurry as well as with hydrated cement to understand the primary working mechanism of the material. The results show improved cement properties with the addition of the Maxcrete additive for the selected cement formulation scheme.

4.1. Slurry Preparation and Order of Mixing

Experiments revealed that the order of inclusion of the additives affects the slurry mixing and hydration (**Tables 1-4**). It was observed that cement should be added right after the water to allow for better hydration of the cement and make the slurry more mixable. The silica sand, silica flour, and calcium carbonate powder can be added after the cement as they do not have a very high specific density. A dispersant was required before adding the manganese tetroxide to reduce viscosity and allow the solid particles to be dispersed in the cement slurry. Fluid-loss additive was the last ingredient that was added in the slurry. The polymeric nature of the fluid-loss additive increased the viscosity of the slurry, which made it difficult to mix (**Fig. 2**). Therefore, the final slurry was prepared to have density of 18.71 ppg. The correct order of mixing was determined by a series of tests, and the following two formulations were selected, one without the

Maxcrete and one with Maxcrete. The Maxcrete added was at 2.5% BWOC for all formulations.

Element	Order of Adding in the Mixture	Mass, g	Density, ppg
Cement G	6	500	26.3
Mn₃O₄	7	450	40.6
Silica Sand (100 mesh)	4	50	22.1
Silica Flour	5	125	22.1
Dispersant	3	4	4.2
Fluid Loss Additive	2	1.5	6.3
Water	1	318.6	8.3
Total		1449.1	

Table 1—Difficult-to-mix additive order.

Element	Order of Adding in the Mixture	Mass, g	Density, ppg
Cement G	2	500	26.3
Mn ₃ O ₄	5	450	40.6
Silica Sand (100 mesh)	3	50	22.1
Silica Flour	4	125	22.1
Dispersant	6	4	4.2
Water	1	318.6	8.3
Total		1447.6	

Table 2—Another difficult-to-mix additive order.

Element	Order of Adding in the Mixture	Mass, g	Density, ppg
Cement G	2	350	26.3
CaCO ₃	5	140	22.6
Mn ₃ O ₄	7	210	40.6
Silica Sand (100 mesh)	3	35	22.1
Silica Flour	4	87.5	22.1
Dispersant	6	2.8	4.2
Fluid Loss Additive	8	0.525	6.3
Water	1	197.9	8.3
Total		1023.7	

Table 3—Cement formulation without addition of Maxcrete.

Element	Order of Adding in the Mixture	Mass, g	Density, ppg
Cement G	3	350	26.3
CaCO ₃	6	140	22.6
Mn ₃ O ₄	8	210	40.6
Silica Sand (100 mesh)	4	35	22.1
Silica Flour	5	87.5	22.1
Dispersant	7	2.8	4.2
Maxcrete Additive	2	8.75	8.3
Fluid Loss Additive	9	0.525	6.3
Water	1	189.1	8.3
Total		1023.7	

Table 4—Cement formulation with addition of Maxcrete.



Fig. 2—A difficult-to-mix slurry in the blender.

In the field however dry additives are blended together and mixed with water. This study was conducted to study the effect of different additives on the cement slurry by adding them one at a time. A correct order of slurry mixing allows cement to hydrate properly as it is added initially, followed by silica that does not react with the cement slurry. The next is the dispersant to allow addition of the weighing material to reduce the viscosity. Fluid loss additive is added at the end to make sure the cement formulation is pumpable. The interaction between the additives and the cement can be observed in the case of adding the additives one after the other.

4.2. Fluid-Loss Test

Fluid-loss tests were performed for the formulations mentioned above, one without Maxcrete and one with Maxcrete. The tests were performed according to the API standards as mentioned before (**Table 5**). The formulation containing Maxcrete showed 18.84% less fluid loss compared to the formulation without Maxcrete. Fluid loss calculated for the cement formulation for 30 minutes was 79.19 ml. The gas breakthrough happened in 15 min compared to the 30-min time recommended in the API standard tests. For both cases the fluid loss volume in 30 minute (V30) was calculated using API suggested method, as follows (Eq. 4):

$$V_{30} = V_t \sqrt{\frac{30}{T}}, \text{ ml} \dots\dots\dots (4)$$

Where V_t = volume of filtrate collected during the test before breakthrough, ml, and T = elapsed time, min.

API fluid loss is determined by multiplying the volume of filtrate collected in 30 minutes by 2, as follows (Eq. 5):

$$\text{API fluid loss} = 2 * V_{30}, \text{ ml} \dots\dots\dots (5)$$

Additive Status	Vt, ml	T, min	V30, ml/30 min	API Fluid Loss, ml
Without Maxcrete	69	15	97.58	195.16
With Maxcrete	56	15	79.19	158.38

Table 5—Fluid-loss test measurement for the cement formulations.

The Maxcrete additive improves the hydration between cement and water. This allows cement to react with more water and less water is left free within the cement structure. This increased hydration reaction allows cement to have a better fluid loss control. The recommended fluid loss is <50 ml/30 min. Here we observe that the fluid loss is higher than 50 ml. Also, in an ideal cement slurry- no gas breakthrough should be observed during the 30 minute of test time. In our formulation we do observe gas breakthrough at 15 minute. This is due to the fact that Maxcrete is not a fluid loss additive. It only improves the hydration that can help in achieve a better fluid loss control. The higher than recommended fluid loss can be attributed to the performance of the fluid loss additive. To optimize the cement formulation, fluid loss additive should be changed.

4.3. Compressive-Strength Test

For the compressive-strength test, cement slurries were prepared according to the two formulations noted above, one without Maxcrete and one with Maxcrete (Table 3,

4). For each formulation, three cubes of 2-in. face dimensions were molded and cured for 24 hours in a water bath at 60°C. After 24 hours, the cubes were crushed using a uniaxial loading machine to measure the compressive strength. **(Figs. 3 and 4)** The addition of Maxcrete in the cement showed a 70.56% increase in compressive strength.

(Table 6)

Additive Status	Test 1 Compressive Strength, psi	Test 2 Compressive Strength, psi	Test 3 Compressive Strength, psi	Average Compressive Strength, psi
Without Maxcrete	2446	2410	2233	2363
With Maxcrete	3365	4381	4345	4030.3

Table 6—Compressive strength test for the cement formulations.



Fig. 3—Crushed cement molds (without Maxcrete).



Fig. 4—Crushed cement molds (with Maxcrete).

The improved compressive strength of the molds prepared with the Maxcrete can be attributed to the improved hydration. Cement particles develop strength and structure as they react with water. Improved hydration process allows the cement particles to develop a stronger cement structure. The improved structure can withstand higher compressive load. This improved compressive strength shows the effect of addition of Maxcrete on improving the mechanical strength of the cement structure. Mechanically stronger cement structure is less prone to crack development and does not allow gas to migrate easily.

4.4. Rheology and Gel Strength

The viscosity measurement is difficult for the formulation containing Maxcrete since for higher rpm, the dial reading is more than 300, which is the limit for the instrument. A better method to measure the viscosity is to be planned in future work. But from the results obtained, it was evident that the effective viscosity of the cement formulation with the Maxcrete is higher than the formulation without Maxcrete. Viscosity was measured for the case without Maxcrete (**Fig. 5, Table 7**) A quantitative comparison is yet to be obtained, and is also planned for future work. The gel strength as measured at low rpm according to API standard is 29.03% higher for cement formulation with the addition of Maxcrete, which reduces the segregation of weighting material. An improvement in the Maxcrete additive that can keep the viscosity of the cement formulation low is recommended to optimize the performance.

RPM	Shear Rate, 1/s	Reading 1	Reading 2	S Factor	Effective Viscosity, cp
3	5.11	-	60	100	6000
6	10.21	100	110	50	5250
100	170	210	200	3	615
200	340	245	240	1.5	363.75
300	511	275	277	1	276
600	1021	>300	>300	0.5	NA

Table 7—Rheology and gel strength for the formulation without Maxcrete.

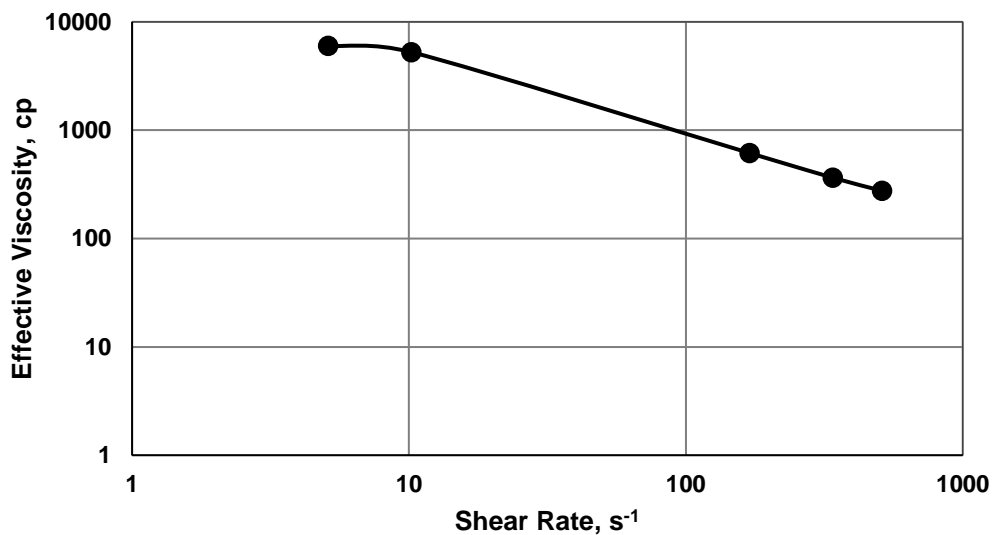


Fig. 5—Effective viscosity vs shear rate for the formulation without Maxcrete.

4.5. Free Water Test

The free water test for both the cement formulation resulted in 0 ml of free water at the top of the 250 ml graduated cylinder after 1 hour (**Figs. 6 and 7**). This is desired for a cement slurry as free water can be caused by improper mixing as well as excess of

water in the cement formulation. The difference between both the formulations was evident during the cleaning of the graduated cylinders as the one with the cement slurry without Maxcrete had a lot of settled solid particles, mostly weighing material whereas the graduated cylinder with the cement slurry including Maxcrete had no such settling observed. This settling can cause two major issues. A cement with non-uniform density and a cement zone with high permeability. As the weighing material settles at the bottom, the upper zone of the cement column has lower density. This may cause weaker cured cement structure that can develop cracks. As the weighing material settles downwards, it creates a layer of solid particles that has high porosity and high permeability. This can act as pathways for gas to migrate through the cement column.

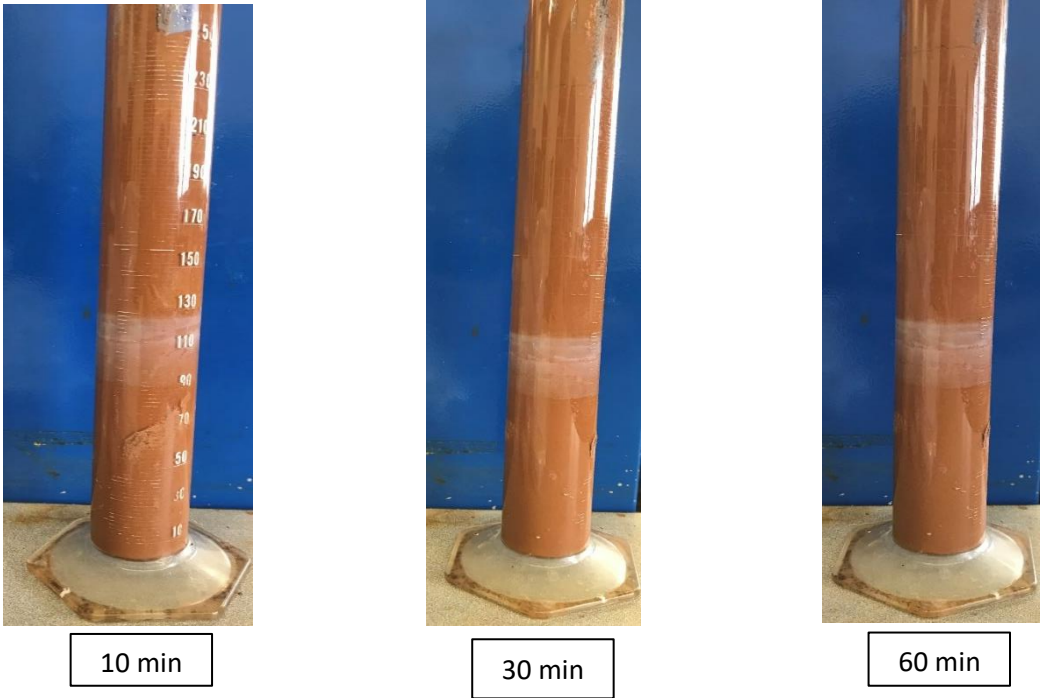


Fig. 6—Free water test for cement formulation without Maxcrete.

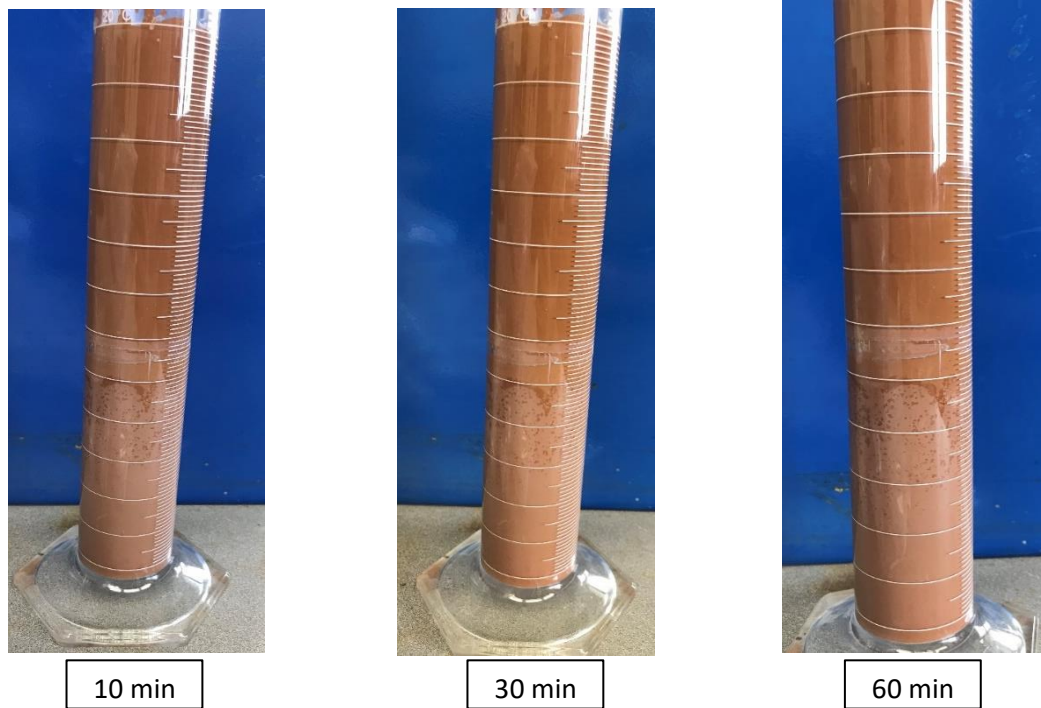


Fig. 7—Free water test for cement formulation with Maxcrete.

4.6. Computed Tomography (CT) Scan

The CT number is dependent on the specific gravity of the particles. Lower Hounsfield Units (CT number unit) shows lower specific gravity for the section scanned and vice versa. A lower CT number suggests more low-density free water/air bubble and less hydrolyzed cement. **Fig. 8** shows a comparison between the CT number of the scanned cylindrical molds of the prepared cement molds with and without the additive Maxcrete. The dashed line in the plot represents the case without Maxcrete, which peaks at a lower CT number compared to the solid line plot which represents the case with Maxcrete. The lower CT number in the cement mold without Maxcrete represents the presence of free water, which has a lower specific gravity compared to the hydrated

cement grains. This plot shows that the cement formulation without Maxcrete has developed lower density compared to the Maxcrete case which can be related to a better hydration and density development in the cement formulation due to the addition of the Maxcrete.

Fig. 9 presents case results for cement without the Maxcrete additive. It depicts different sections from top to bottom of the cylindrical mold. Each plot is peaked at relatively different CT number which shows that the specific gravity distribution within the mold is not uniform throughout the cylindrical mold. This uneven distribution can lead to poorer mechanical strength which is observed in the compressive strength test. Cement with different density in different section can react differently under a stress condition and is prone to develop cracks which can allow pathways for gas migration.

Fig. 10 is a similar representation for the case of cement with the Maxcrete additive. Each section of the cylindrical molds peak at relatively uniform CT number, which represents a uniform specific gravity distribution within the cylindrical cement mold. A cement structure with similar specific gravity distribution is less prone to developing fractures as they react similarly under a given stress condition and has no weaker section that develops cracks. This kind of cement structure will have higher mechanical strength which is represented through the higher compressive strength.

To generate CT scan images of the cement mold, a set of parameters had to be defined in the analysis. The Hounsfield Units (HU) that are a measure of X-ray attenuation can be related to the specific gravity or density of the object. A dense object such as set cement will have a higher density and therefore a higher CT number in HU.

Unreacted water within the cement will have a lower specific gravity and therefore a lower CT number in HU. For a given cement structure, higher the CT scan number in HU represents higher developed specific gravity. A higher CT number also signifies less free water within the cement pores and more hydrated cement. To analyze this phenomenon, a CT scan threshold and a window was defined within the software tool. A CT scan threshold is the range of HU that the image shows. For our study we are focused on the 2000-4000 HU which is typical for high density solids. Any object below 2000 HU such as Air bubbles or free water would be represented as red color. The next parameter to be defined is the window. This window is used to set a filter of colors in the scanned image. A window width was defined to be 900 HU which is the total width of the CT numbers where the color filter would be applied. A window level of 2550 HU was defined which is the midpoint CT number value of the window. These parameters results in a green color for CT number of 2550 HU. For CT numbers between 2100 and 2550, the colors red, orange and yellow are assigned. For CT numbers between 2550 and 3000, the colors blue, indigo, and violet are assigned.

Figs. 11 and 12 are the CT-scan images for the cores without Maxcrete and with Maxcrete respectively. These images provides a direct visual correlation between the addition of the Maxcrete and improved cement hydration. The CT scan image clearly shows a lower specific density zone in the middle marked by yellow-red color for the case of without Maxcrete. This image shows an uneven hydration in the center of the sample, which can lead to lower cement strength. However, the CT scan image for the case with Maxcrete shows a higher CT number and, therefore, a higher specific gravity

distribution marked by green and blue color. The hydration is more even, which provides a better strength to the cement structure.

The CT scan images provides a confirmation to the fluid loss and compressive strength results. With the addition of Maxcrete, the fluid loss is reduced and compressive strength is improved. This is due to the working mechanism of Maxcrete as it improves the hydration between water and cement to formulate a better cement slurry that is less prone to developing cracks. The CT scan images provided a visual proof that the hydration is improved as the cement developed a higher and more uniform density. A cement formulation with better hydration, higher developed density, uniform density distribution, fluid loss control, and mechanical strength will provide a better gas migration control.

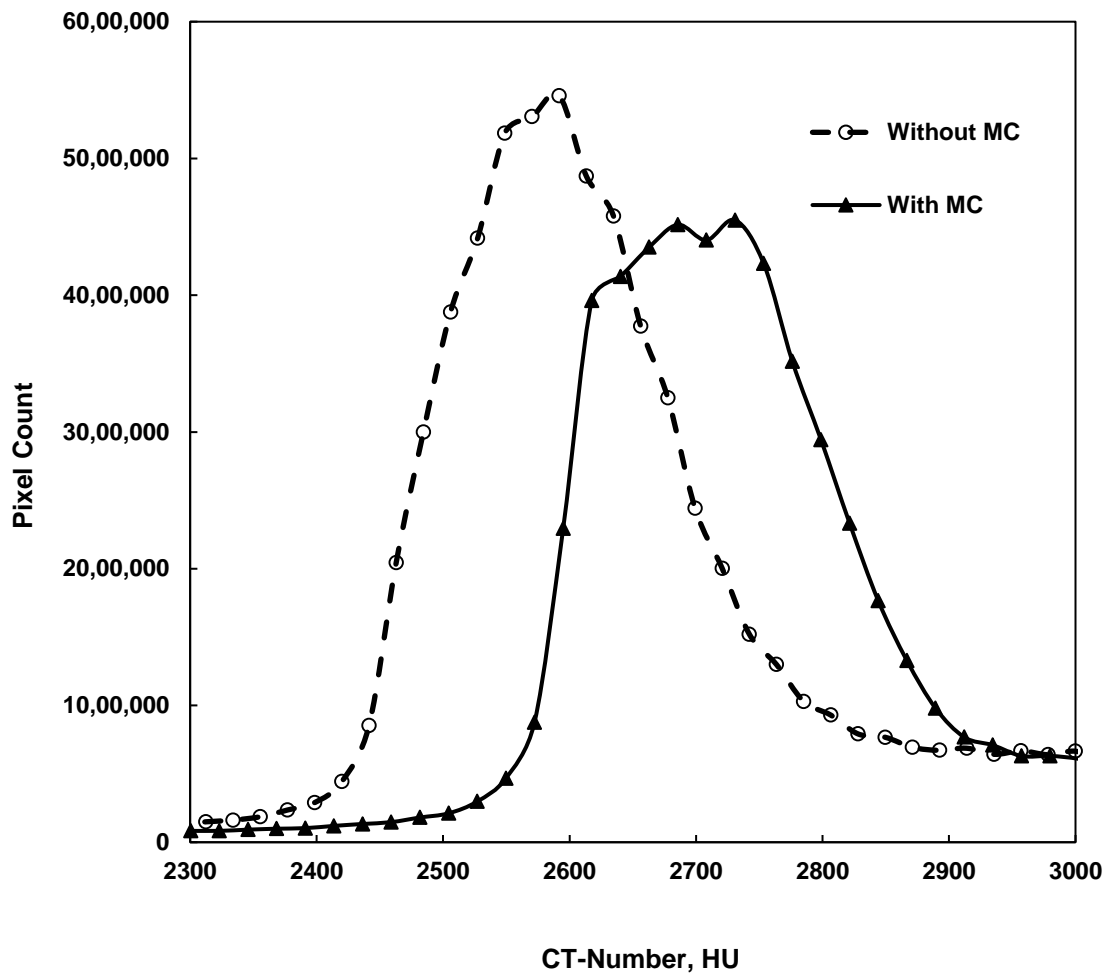


Fig. 8—Pixel count vs CT-number (HU) plot for cement formulations with and without Maxcrete.

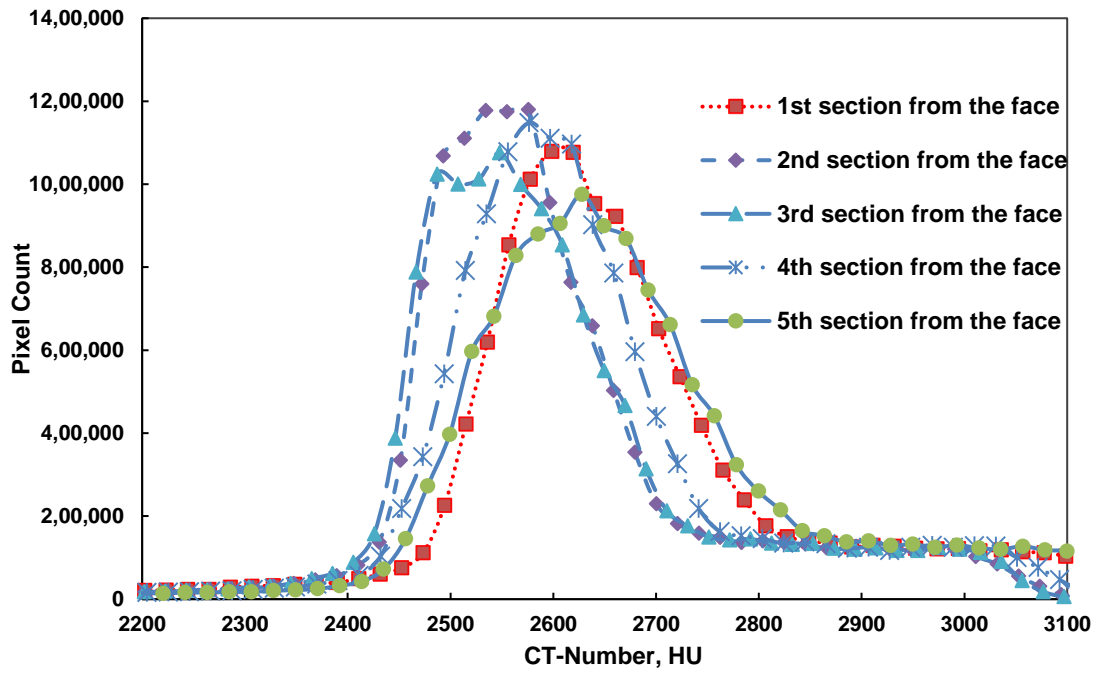


Fig. 9—Pixel count vs CT-number (HU) plot for cement formulation without Maxcrete at different sections from face to bottom.

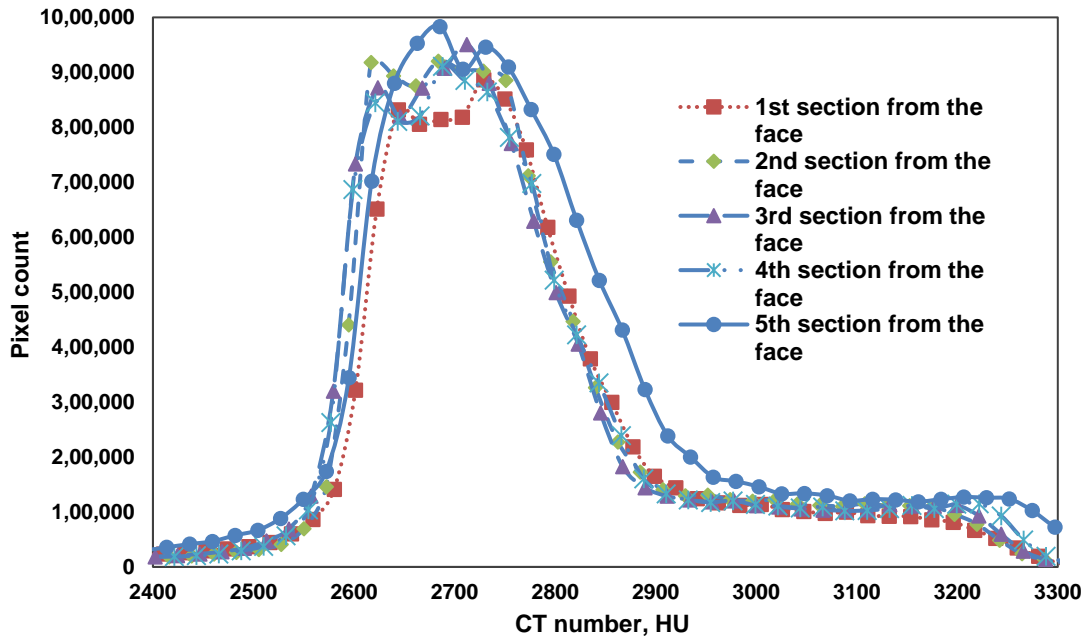


Fig. 10—Pixel count vs CT-number (HU) plot for cement formulation with Maxcrete at different sections from face to bottom.

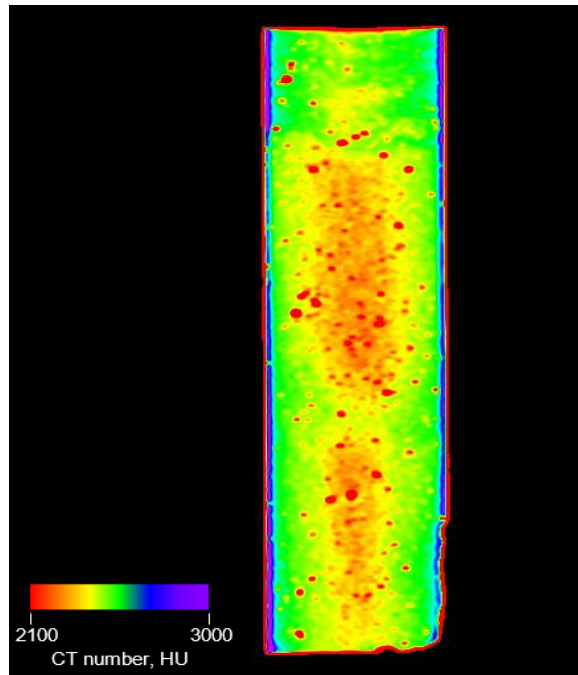


Fig. 11—CT scan image for cement formulation without Maxcrete.

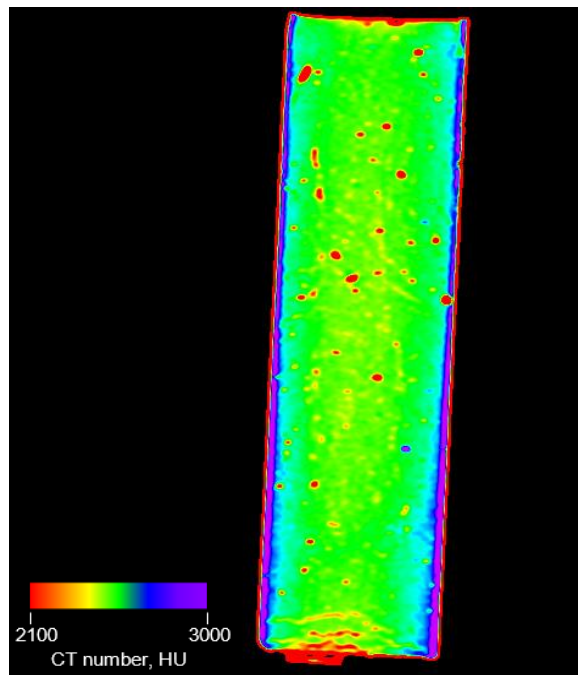


Fig. 12--CT scan image for cement formulation with Maxcrete.

5. IMPROVEMENT IN THE CEMENT FORMULATION

The primary cement tests showed positive effect with the introduction of the new chemical additive, Maxcrete. The next step in the study was to improve the cement formulation by understanding how to improve the new chemical additive and find possible limitations. New chemicals or materials were introduced in the formulation to improve the fluid-loss control and mechanical strength of the cement.

5.1. Effect of Hematite on Fluid Loss

One of the primary aims of a cement formulation is to reduce fluid loss to an acceptable level (<50 ml/30 min). According to Al. Yami, 2015 hematite powder was introduced along with Mn_3O_4 to reduce the fluid loss. Haidher (2008) suggested that hematite without other weighing materials can settle at the bottom of the cement creating a higher permeable structure. Sieve analysis was also done to observe the effect of the particle size distribution on the fluid loss. From the available two hematite grades, the one with finer particles showed a better result and was preferred for the future tests. The aim of the cement slurry formulations investigation in this research was to produce acceptable fluid loss before measuring the other properties such as permeability or mechanical strength.

Three different systems including 1) Mn_3O_4 with silica flour and silica sand; 2) Mn_3O_4 with $CaCO_3$, silica flour, and silica sand; and 3) Mn_3O_4 with hematite, silica flour and silica sand were compared. The density for each cement system was 18.71 ppg. The

concentration of fluid-loss additive and dispersant was also kept the same to study the effect of the system on the fluid-loss property. We found the system with only Mn_3O_4 and silica to have a very high fluid loss. Introduction of $CaCO_3$, reduced the fluid loss but not to the accepted limits. Addition of hematite significantly reduced the fluid loss. **Fig. 13** shows comparison of fluid loss between these three cement systems.

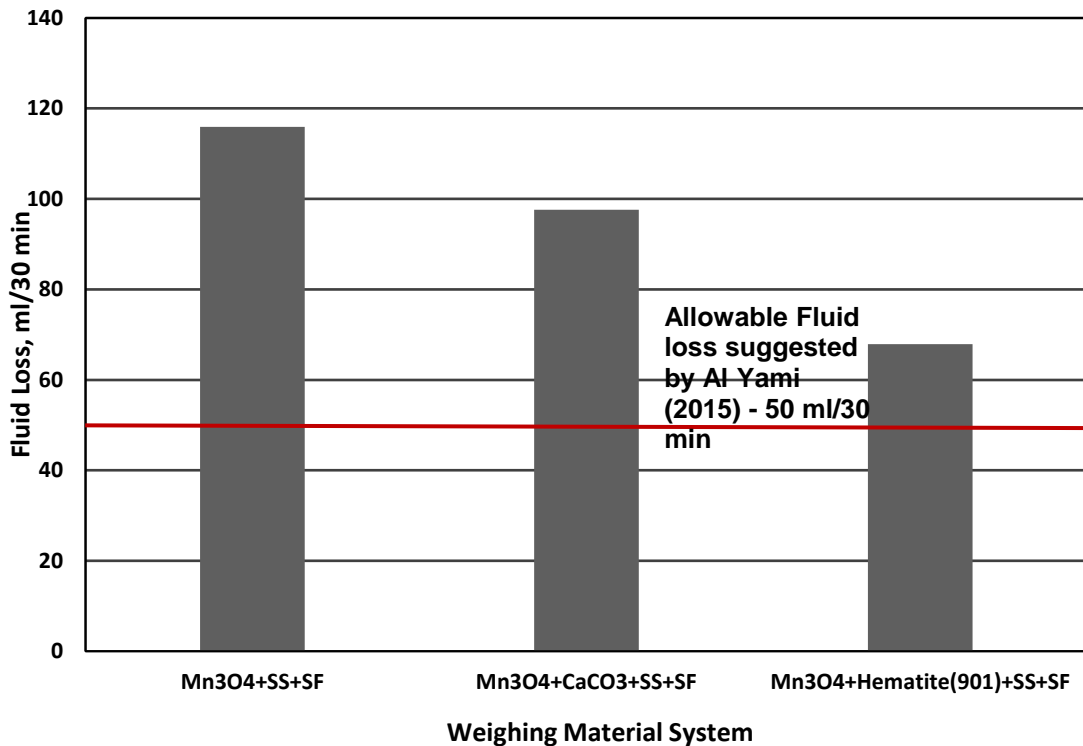


Fig. 13—Fluid loss for different cement systems.

Hematite has a higher specific gravity and has similar size to the manganese tetroxide particles. Hematite introduction makes the higher cement density achievable. The combination of manganese tetroxide and hematite allows to create a low permeability and high density structure. Although the viscosity of the resulting cement is important as hematite can increase the viscosity and make the cement non-pumpable.

5.2. Change in Fluid Loss Additive

The fluid loss improvement was required to improve the cement formulation. A test with change in fluid loss additive concentration was conducted to understand the effect of the fluid loss additive and a requirement to change it. Three concentrations 0%, 0.15%, and 0.3% BWOC fluid loss additive was used to compare the change in fluid loss at 18.7ppg density cement. It was observed that the fluid loss additive did not have much effect on the fluid loss control and provided similar performance over different concentrations (Fig. 14).

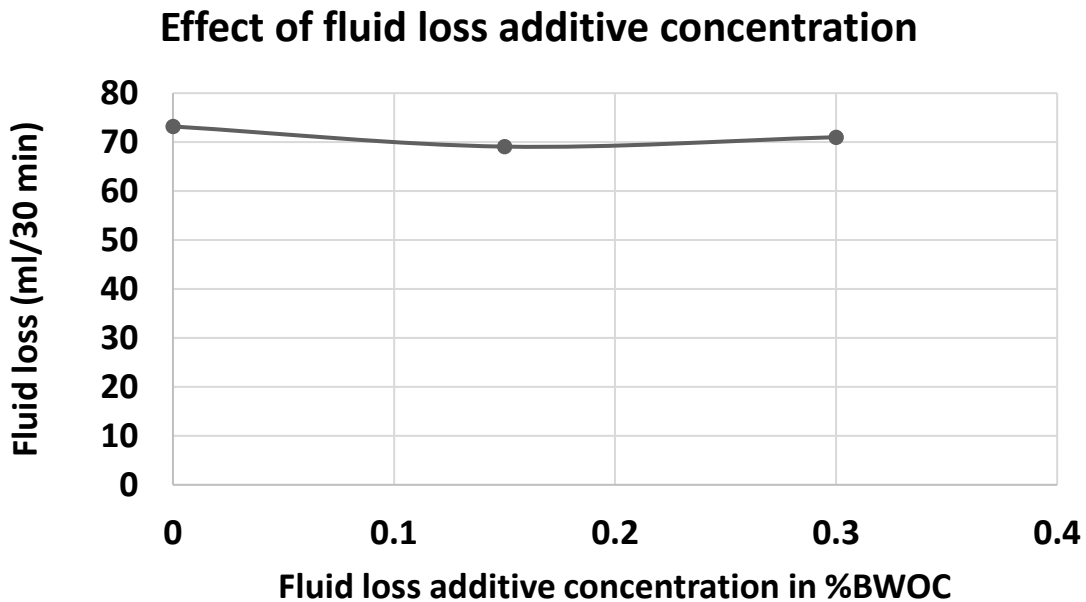


Fig. 14—Effect of fluid loss additive concentration on fluid loss.

To reduce the fluid loss from the cement formulation to an acceptable level below the advised limit of 50 ml/30 min according to the API standards, a new fluid-loss

additive was used. The new fluid-loss additive, CH showed improved fluid loss control without any gas breakthrough for the 30-minute test for the case without Maxcrete (**Table 8**). However, the mixing of this new fluid-loss additive was more difficult as it increased the viscosity of the cement formulation which adversely affected the performance of the cement formulation with Maxcrete.

Increased viscosity caused additional fluid loss in the slurry containing the new fluid loss additive and Maxcrete. The solid particles were not dispersed effectively and thus the fluid loss increased. Maxcrete actually increased the fluid loss by reducing the time required for the gas to break through.

Fluid loss additive system	Fluid loss, ml	Breakthrough time, min	Fluid loss, ml/30 min
Fluid loss additive with no Maxcrete	52.0	17.0	69.1
New fluid loss additive (CH) with no Maxcrete	46.0	-	46.0
New fluid loss additive (CH) with 2.5 % BWOC Maxcrete	42.0	10.0	72.7

Table 8—Effect of change in fluid-loss additive on fluid loss.

5.3. Effect of Cement Density with Maxcrete Concentration

To determine whether the Maxcrete application was limited to a certain density; three different cement densities, 18.7, 17.4, and 16 ppg, were tested at the following four different Maxcrete concentrations: 0%, 0.625%, 1.25%, and 2.5% BWOC (**Table 9, Figs. 15-18**). Fluid loss declined with increasing cement density, which was expected due to decreasing water/solid ratio. However, for the same cement density, increasing

the Maxcrete concentration also increased the fluid loss, again due to decreasing gas breakthrough time.

Cement Density, ppg	Maxcrete concentration %BWO	Fluid loss, ml/30 min	Gas breakthrough time, minute
18.7	0	46.00	-
	0.625	51.80	28.00
	1.25	63.60	15.00
	2.50	72.70	10.00
17.4	0	91.10	28.00
	0.625	102.80	23.00
	1.25	127.30	15.00
	2.50	143.50	14.00
16.0	0	145.80	21.00
	0.625	178.40	19.00
	1.25	189.50	15.00
	2.50	206.60	13.00

Table 9—Effect of density and Maxcrete concentration on fluid loss.

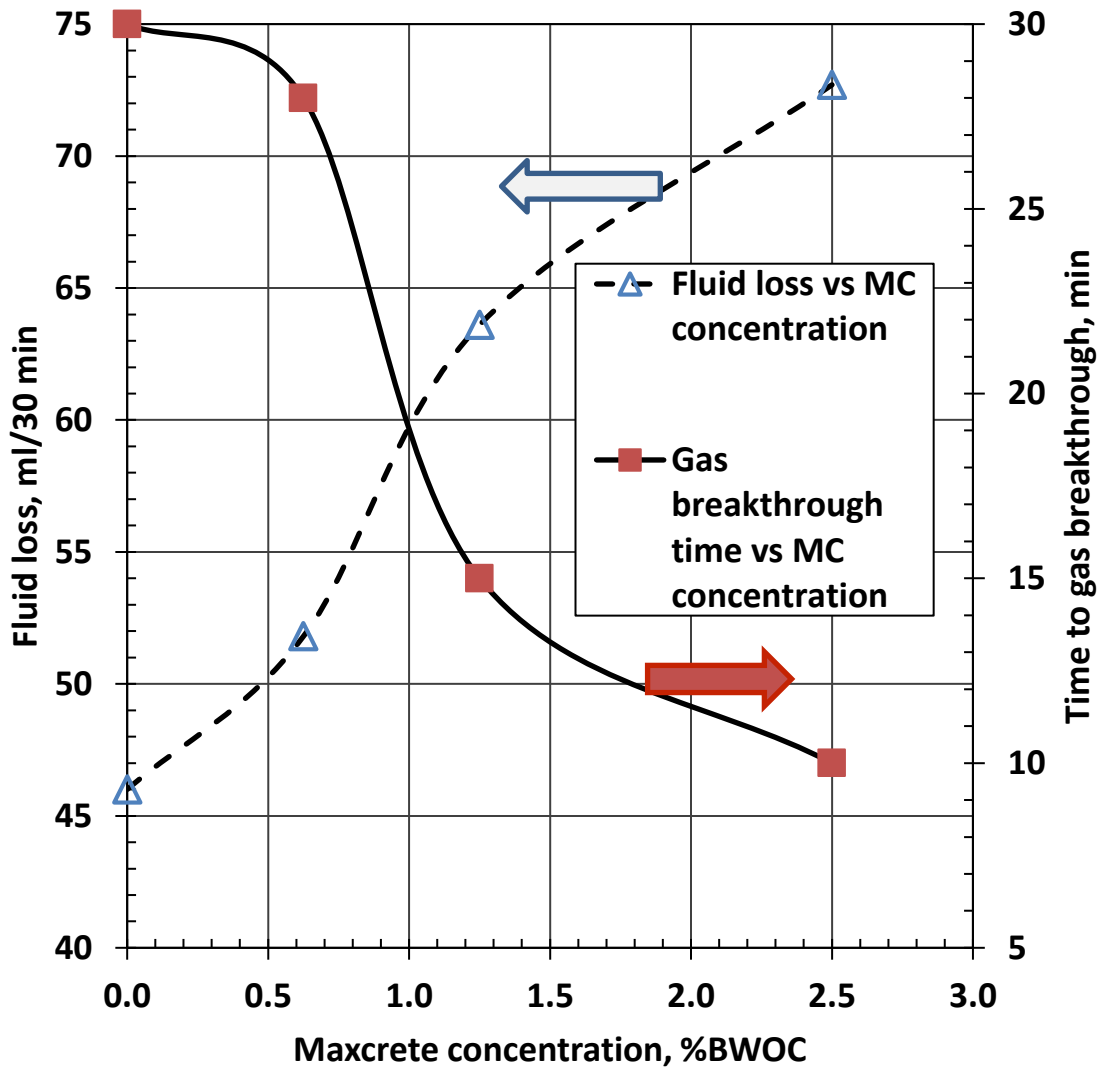


Fig. 15—Maxcrete additive concentration vs fluid loss and breakthrough time for 18.7 ppg cement.

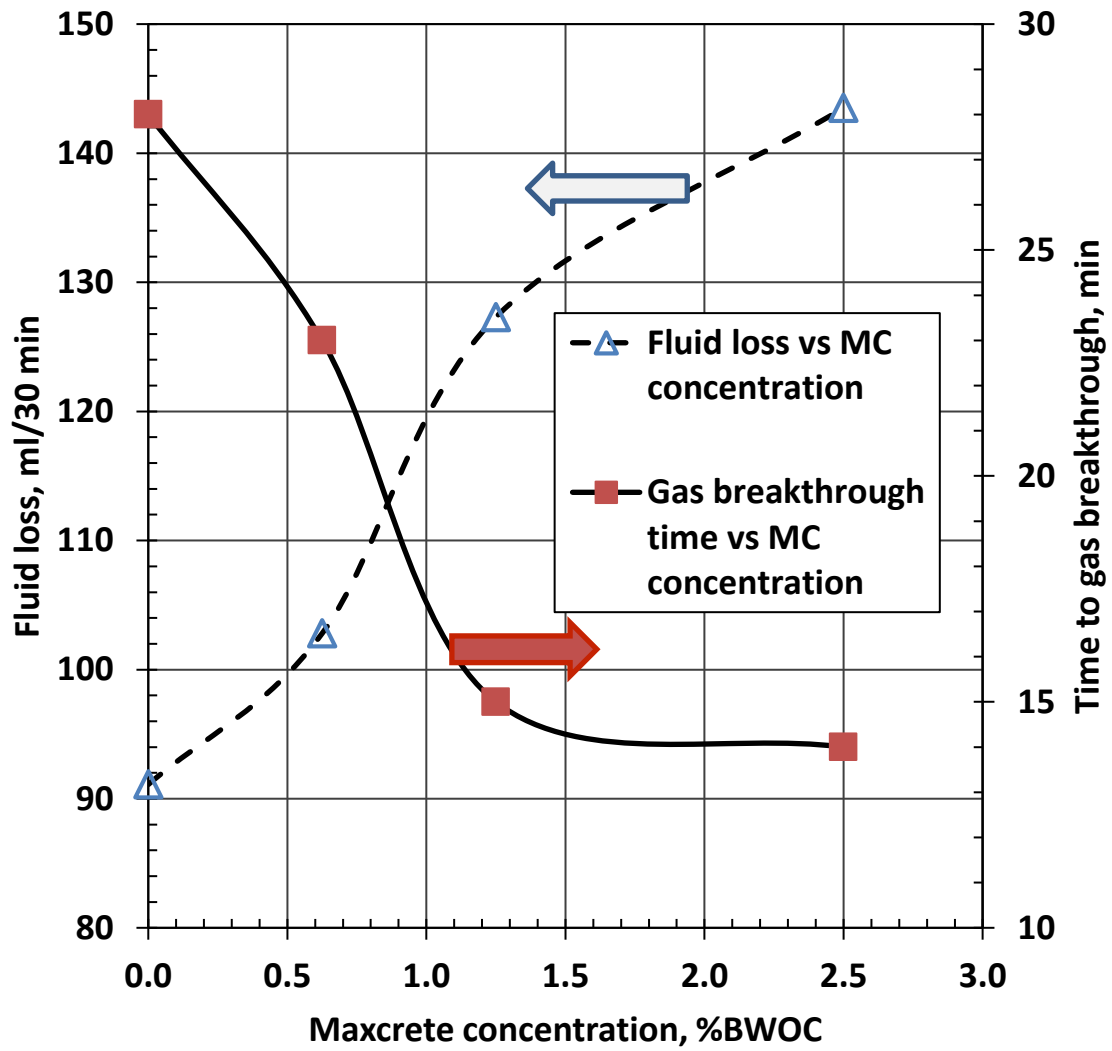


Fig. 16—Maxcrete additive concentration vs fluid loss and breakthrough time for 17.4 ppg cement.

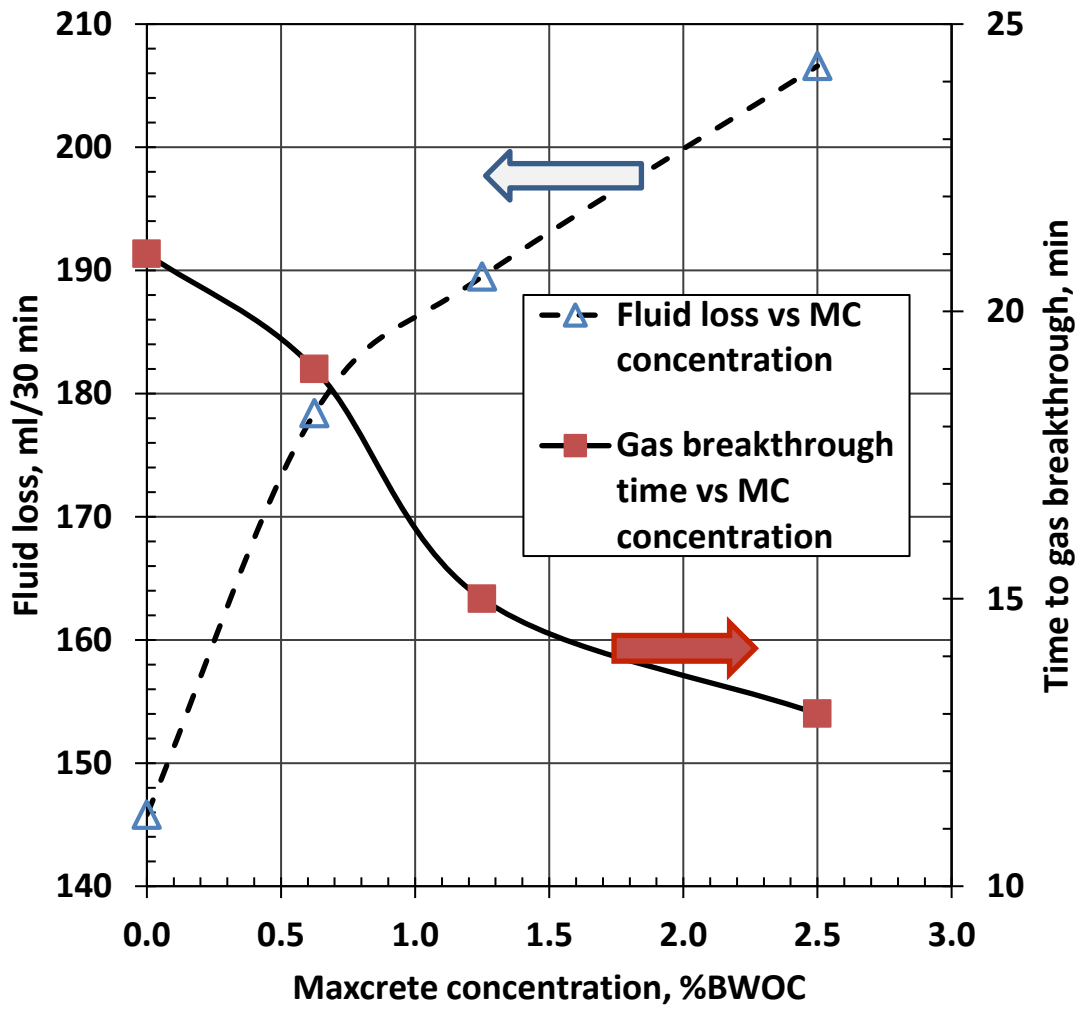


Fig. 17—Maxcrete additive concentration vs fluid loss and breakthrough time for 16 ppg cement.

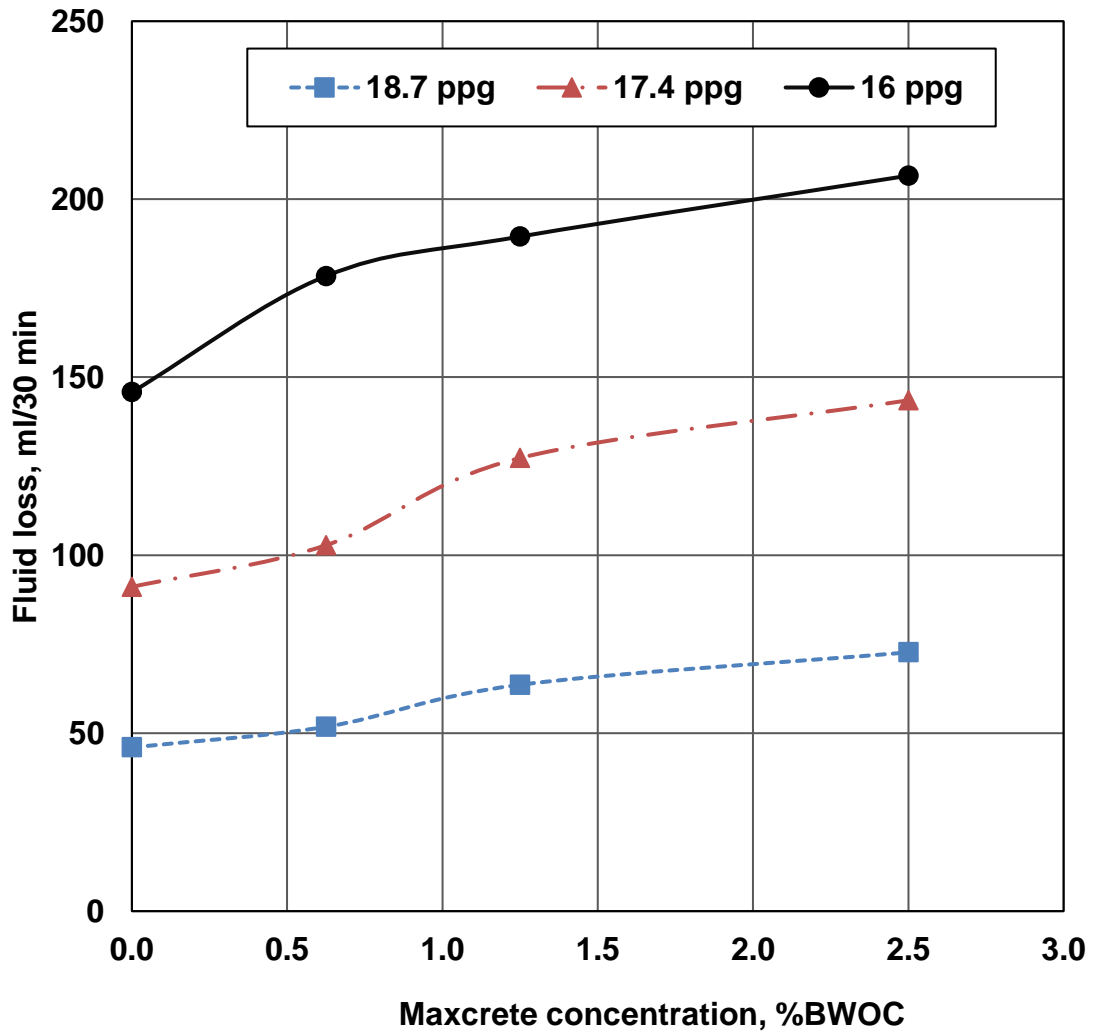


Fig. 18—Fluid loss vs Maxcrete additive concentration for 18.7, 17.4 and 16 ppg cement.

5.4. Powdered Maxcrete

Cement is dry blended with the other dry additives in the field and then mixed with water. The Maxcrete additive is in liquid form that can be added with the water to prepare the liquid blend of the cement. Dry additives can then be mixed with this liquid blend. Some operators prefer to add the water at the end and would not be able to mix any liquid additives with the water. In these cases a powdered Maxcrete can be helpful as it can be mixed with the dry blend. The powdered Maxcrete is formed by dehydrating the liquid Maxcrete. The powdered Maxcrete can be a useful addition in the applicability of the Maxcrete chemical additive.

Powdered Maxcrete was used to run fluid loss tests similar to liquid Maxcrete. The powdered Maxcrete has 2 times more active chemicals compared to the liquid Maxcrete. To compare the fluid loss effect of powdered Maxcrete with liquid Maxcrete, a base cement test was designed. The cement was formulated with only Portland class G cement, water and Maxcrete. A no Maxcrete case was also used to compare the results. Since there was no fluid loss additive, the amount of fluid lost by the cement formulation was very high and a quick gas breakthrough was also observed. The total amount of fluid loss was measured for comparison study. The breakthrough time was as low as 1-3 minutes. Since the API fluid loss calculation would have a very high error for such a small value of breakthrough time, only the absolute fluid loss is measured. The gas breakthrough for no Maxcrete was faster (1-1.5 minutes) compared to with Maxcrete case (2.5-3 minutes). 16 ppg cement was prepared with 2.5% BWOC Maxcrete in both liquid and powdered Maxcrete case. The Maxcrete powder had twice the active

ingredients and showed low fluid loss compared to the liquid Maxcrete and no Maxcrete case (**Table 10**).

Cement system	Cement, g	Water, g(% BWOC)	Maxcrete, g(% BWOC)	Fluid loss, ml
No Maxcrete	550.0	234.2 (42.6%)	0.0	112.0
Liquid Maxcrete	550.0	220.5 (40.1%)	13.8 (2.5%)	108.0
Powder Maxcrete	550.0	225.6 (41.02%)	13.8 (2.5%)	91.0

Table 10—Fluid loss test for powdered Maxcrete

Even with the improved fluid loss control offered by the Maxcrete powder, there is an application issue. The Maxcrete powder is very hydrophilic and can absorb moisture from the air. This moisture absorption can turn the powdered Maxcrete into a moist slurry. This requires the powdered Maxcrete to be stored in a no moisture or vacuumed environment. Any contact with moist air which is very common in the field can cause moisture absorption. A powdered Maxcrete that has absorbed water can cause difficulties in the dry blending. Water absorbed in the Maxcrete powder can start reacting with other additives and make the mixing of dry additives more difficult as it can stick to the blender wall. Dry cement powder can also completely become liquid for longer moisture exposure. **Figs. 19 and 20** shows the effect of moisture absorption on the powdered Maxcrete.

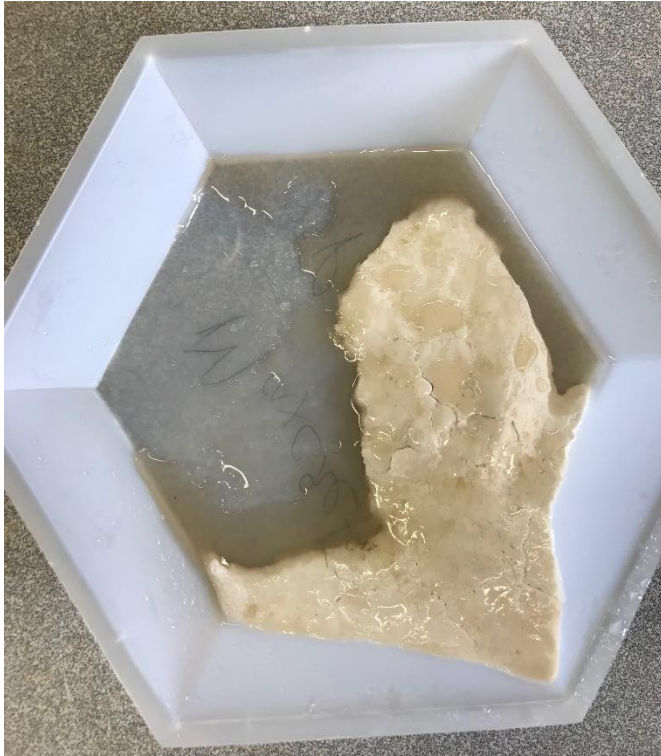


Fig. 19—Water absorption from powdered Maxcrete after 1 hour of air exposure.

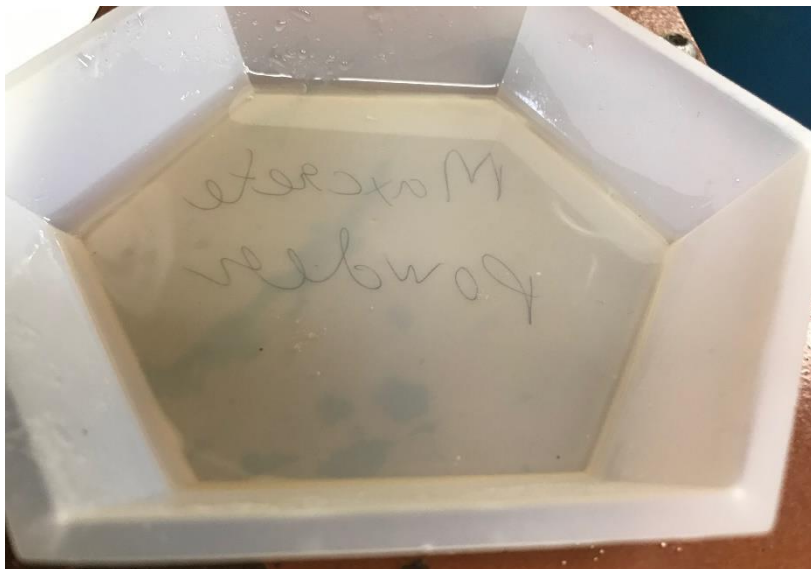


Fig. 20—Water absorption from powdered Maxcrete after 24 hour of air exposure.

5.5. Improved Chemical Additive: Maxcrete 105

With the improvement in the chemical additive’s rheological properties, the new Maxcrete 105 showed improved mixing ease with weighing material. The mixing method was changed to represent field practice in which all the solids were dry-blended first and then added into the water or the water and Maxcrete blend. The fluid loss was measured for 16 ppg cement slurry with 25% BWOC Mn₃O₄. The slurry was prepared with 2.5% BWOC old Maxcrete as well as Maxcrete-105, and also with no Maxcrete. The results showed that with improved rheological properties, the case with Maxcrete-105 performed far better compared to the other two cases in terms of fluid loss control (**Table 11**).

Cement system	Fluid loss, ml	Breakthrough time, min	Fluid loss, ml/30 min
No Maxcrete	98.0	22.0	114.4
2.5% BWOC old Maxcrete	100.0	10.0	173.2
2.5% BWOC Maxcrete 105	37.0	-	37.0

Table 11—Effect of new Maxcrete 105 on cement fluid loss.

Additive Status	Test 1 Compressive Strength, psi	Test 2 Compressive Strength, psi	Test 3 Compressive Strength, psi	Average Compressive Strength, psi
Without Maxcrete	1858	1904	1912	1891.3
With Maxcrete 105	2209	2370	2207	2262

Table 12—Compressive strength improvement with Maxcrete-105.

For Maxcrete 105, the improved fluid-loss properties also resulted in the improved compressive strength (**Table 12**). The compressive strength was performed on 2-in. cube molds that were cured at 60°C for 24 hours. The average compressive strength

was measured for 3 samples. The average compressive strength with the Maxcrete 105 is 19.6% increase compared to the no Maxcrete case. This process improved fluid loss control along with the increase in the compressive strength of the cured cement shows improved hydration.

The improved additive Maxcrete 105 reduced the viscosity of the cement formulation. A fluid-loss test was conducted to understand the effect of not adding the dispersant in the cement slurry formulation with the new Maxcrete 105 formulation (**Table 13**). A higher overall fluid loss was observed compared to using the dispersant, but the cement with the Maxcrete 105 showed better fluid-loss control compared to no Maxcrete 105 cement.

Cement system	Fluid loss, ml	Breakthrough time, min	Fluid loss, ml/30 min
No Maxcrete	123	15	174
2.5% BWOC Maxcrete 105	63	-	63

Table 13—Effect of new Maxcrete 105 on cement fluid loss without Dispersant.

6. CONCLUSIONS

The results of this study lead to the following conclusions:

1. The order of the addition of the solid additives to a cement slurry formulation affects the ease of mixing. High-density weighing materials, as well as polymers, should be added at the end of the mixing.
2. Addition of the new chemical additive, Maxcrete 105, improves fluid-loss control and compressive strength.
3. Addition of Maxcrete also improves the hydration of the cement, as observed with the CT scan results. Maxcrete coats the cement particles and helps the cement to attract more water and improve the hydration process.
4. Addition of manganese tetroxide along with hematite as weighing materials improved the overall fluid-loss control.
5. At high density and high weighing material concentration, addition of Maxcrete can reduce mixability, which can lead to higher fluid loss due to lack of uniform dispersion of the solid particles.
6. At high density and high weighing material concentrations, increasing the concentration of Maxcrete increases the fluid loss and reduces the gas breakthrough time.
7. For a given concentration of the additives, with increasing cement density the fluid loss reduces.

8. Powdered Maxcrete has improved performance with double active ingredients but has storage problem due to its tendency to absorb moisture from the air.
9. The new Maxcrete 105 with improved mixing ease shows significantly improved fluid-loss control compared to no Maxcrete and old Maxcrete, even in the presence of high-density weighing material.

This study shows the effect of the Maxcrete additive on the cement formulation. The improved hydration of cement with the addition of the Maxcrete improved the fluid loss and compressive strength. A cement with lower fluid loss and higher compressive strength is less prone to develop fracture and cracks. A cement with this improved properties can offer a better gas migration control and avoid health, safety, and environmental hazards.

REFERENCES

- Al-Saeedi, M. J., Al-Enezi, D. R., and Al Mudhaf, M. N. A. et al. (2011, September 1). Ongoing Development of Cementing Practices and Technologies for Kuwait Oil Company's Deep HP/HT Exploration and Gas Wells: Case History. Society of Petroleum Engineers. doi:10.2118/142150-PA.
- Al-Yami, A. S. (2015, October 11). An Innovative Cement Formula to Mitigate Gas Migration Problems in Deep Gas Wells: Lab Studies and Field Cases. Society of Petroleum Engineers. doi:10.2118/175194-MS.
- Al-Yami, A. S., Wagle, V., and Santagati, A. A. et al. (2018, March 20). Durable and Self-Healing Cement Systems: Lab Testing and Field Deployment. Offshore Technology Conference. doi:10.4043/28230-MS.
- API, R. 10B-2, Recommended Practice for Testing Well Cements. 2013. Washington, DC: API.
- Baret, J.-F. (1988, January 1). Why Cement Fluid Loss Additives Are Necessary. Society of Petroleum Engineers. doi:10.2118/17630-MS.
- Bois, A.-P., Vu, M.-H., Noël, K., Badalamenti, A., Delabroy, L., Théron, E., & Hansen, K. (2019, June 1). Evaluating Cement-Plug Mechanical and Hydraulic Integrity. Society of Petroleum Engineers. doi:10.2118/191335-PA

Brothers, L. E., and deBlanc, F. X. (1989, June 1). New Cement Formulation Helps Solve Deep Cementing Problems. Society of Petroleum Engineers. doi:10.2118/17181-PA.

Carter, G., and Slagle, K. (1972, September 1). A Study of Completion Practices To Minimize Gas Communication. Society of Petroleum Engineers. doi:10.2118/3164-PA.

Cheung, P. R., and Beirute, R. M. (1985, June 1). Gas Flow in Cements. Society of Petroleum Engineers. doi:10.2118/11207-PA.

Christian, W. W., Chatterji, J., and Ostroot, G. W. (1976, November 1). Gas Leakage in Primary Cementing - A Field Study and Laboratory Investigation. Society of Petroleum Engineers. doi:10.2118/5517-PA.

Daccord, G., and Baret, J. F. (1994, June 1). How Fluid Loss Influences Primary Cementing: Literature Review and Methodology. Society of Petroleum Engineers. doi:10.2118/25150-PA.

Dusseault, M. B., Gray, M. N., and Nawrocki, P. A. (2000, January 1). Why Oilwells Leak: Cement Behavior and Long-Term Consequences. Society of Petroleum Engineers. doi:10.2118/64733-MS.

Haidher, S. G. M. (2008, January 1). Best Practices in Designing HP/HT Cement-Plug Systems. Society of Petroleum Engineers. doi:10.2118/116698-MS.

- Jacobs, T. (2014, December 1). Studying the Sources of Methane Migration Into Groundwater. Society of Petroleum Engineers. doi:10.2118/1214-0042-JPT.
- Mata, F. J., Diaz, C., and Villa, H. (2006, January 1). Ultralightweight and Gas Migration Slurries an Excellent Solution for Gas Well. Society of Petroleum Engineers. doi:10.2118/102220-MS.
- Nelson, E. B. (Ed.). (1990). Well cementing (Vol. 28). Newnes.
- Noik, C., and Rivereau, A. (1999, January 1). Oilwell Cement Durability. Society of Petroleum Engineers. doi:10.2118/56538-MS.
- Pour, M. M., and Moghadasi, J. (2007, January 1). New Cement Formulation that Solves Gas Migration Problems in Iranian South Pars Field Condition. Society of Petroleum Engineers. doi:10.2118/105663-MS.
- Reddy, B. R., Xu, Y., and Ravi, K., et al. (2009, March 1). Cement Shrinkage Measurement in Oilwell Cementing--A Comparative Study of Laboratory Methods and Procedures. Society of Petroleum Engineers. doi:10.2118/103610-PA.
- Roshan, H., & Asef, M. R. (2010, September 1). Characteristics of Oilwell Cement Slurry Using CMC. Society of Petroleum Engineers. doi:10.2118/114246-PA.
- Sabins, F., and Wiggins, M. L. (1997, September 1). Parametric Study of Gas Entry Into Cemented Wellbores. Society of Petroleum Engineers. doi:10.2118/28472-PA.

Shakirah, S. (2008, January 1). A New Approach for Optimizing Cement Design to Eliminate Microannulus in Steam Injection Wells. International Petroleum Technology Conference. doi:10.2523/IPTC-12407-MS.

Talabani, S., and Hareland, G. (1995, January 1). New Cement Additives That Eliminate Cement Body Permeability. Society of Petroleum Engineers. doi:10.2118/29269-MS.

Thiercelin, M. J., Dargaud, B., and Baret, J. F., et al. (1998, December 1). Cement Design Based on Cement Mechanical Response. Society of Petroleum Engineers. doi:10.2118/52890-PA.

Wilcox, B., Oyenehin, B., and Islam, S. (2016, August 2). HPHT Well Integrity and Cement Failure. Society of Petroleum Engineers. doi:10.2118/184254-MS.