IMPROVING OIL-WELL CEMENT INTEGRITY USING A SYNTHETIC FIBER

BY

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Dedication

This thesis is dedicated to my parents, my brothers, my sisters and my fiancé for their continuous support and prayers

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LIST OF ABBREVIATIONS

μ_p	:	Plastic Viscosity
API	:	American Petroleum Institute
ASTM	:	American Standards for Testing and Measurement
Bc	:	Bearden Consistency Unit
BHCT	:	Bottomhole Circulating Temperature
BHP	:	Bottomhole Pressure
BHST	:	Bottomhole Static Temperature
BWOC	:	By Weight of Cement
BWOW	:	By Weight of Water
HPHT	:	High Pressure High Temperature
HSR	:	High Sulphate Resistant
ISO	:	International Organization for Standardization
MSR	:	Moderate Sulphate Resistant
MW	:	Mud Weight
OSR	:	Ordinary Sulphate Resistant
PCF	:	Pound per Cubic Feet
PPF	:	Polypropylene fiber
PV	:	Plastic Viscosity
RPM	:	Rotation per Minute
SEM	:	Scanning Electron Microscope
TRB	:	Time to Reach Bottom

TVD	:	Total Vertical Depth
UCA	:	Ultrasonic Cement Analyzer
WOC	:	Wait on Cement
XRD	:	X-ray Diffraction
YP	:	Yield Point

ABSTRACT (ENGLISH)

NAME: Anas Alsiddig Yousif Mohammd Ahmed

TITLE: Improving Oil-Well Cement Integrity using a Synthetic Fiber

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The high global growth of oil demand is significantly notable with the oil/gas companies searching for the unexplored areas, the task is more difficult in terms of depth, pressure and temperature. In deeper wells, high temperature and pressure and post cementing operations put extreme stresses on the cement sheath and dramatically affect the cement integrity. So, the design of the cement slurry is very important to ensure the durability and long term integrity of the cement sheath. The design should lead for a better zones isolation and strengths the bonding with the casing. Furthermore, it should help to shield the casing from corroding and unpredictable shock loads in deeper zones and sustain plugs to control the lost circulation in fractured and high permeability zones.

Many techniques have been implemented within oil cementing history application to enhance the properties especially those relate to the strength and toughness. Among these techniques are using small-sized particles such as polymers, fibers, and bio-medicine in concrete history. Because of their large surface areas and small size, those particles are highly being used in petroleum industry in the latest decades.

In this research, the usage of polypropylene fibers (PPF) is evaluated for oilwell cements at high pressure and high temperature (HPHT). The polypropylene fibers were added into

cement admixture with discrete amount to evaluate its influence on enhancing the performance of cements especially those relate to the toughness. Experimental studies were performed using distinct cement slurry characteristics such as rheological properties, thickening time, compressive strength, free water content, density, porosity and permeability. The Scanning Electron Microscope (SEM), and X-ray powder diffraction (XRD) was also performed for analyzing the microstructure of the cement.

The best slurry design is obtained based on the results of different percentages of fibers used in experiments. From the experimental tests, it is observed that addition of fibers improves the mechanical properties, accelerates the thickening time, reduces the free water contents, reduces the density and helps in designing a new cement slurry which withstands the HPHT conditions and gives good results.

ملخص الرسالة

الاسم: **أنس الصديق يوسف محمد أحمد**

عنوان الرسالة: **تحسين كمالية إسمنت حفر أبار النفط باستخدام الألياف الصناعية**

التخصص: **هندسة البترول**

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الزيادة الكبيرة للطلب العالمي للنفط أصبح ملحوظاً بشكل كبير وذلك بالإلحاح الكبير لشركات النفط والغاز بالبحث عن المناطق غير المكتشفة، عملية البحث تلك تشكل تحدي في حالة الظروف العالية للضغط والحرارة. في الأبار العميقة، عمليات السمنتة تتطلب جهوداً عالية لتصميم الغلاف الإسمنتي وتوثر بشكل ملحوظ على كمالية الإسمنت. لهذا، تكون عملية تصميم الخليط الإسمنتي بالغة الأهمية لجلب متانة وكمالية أمثل للإسمنت وعلمية السمنتة. وذلك بعزل أفضل للطبقات داخل التكوين الصخري للأرض وأيضاً لربط أفضل للبطانة. وحماية أقوى للبطانة من التآكل، ومحافظة أكمل للبطانة من الصدمات أثناء الحفر بأعماق بعيدة، وعملية تسديد أفضل للطبقات الضعيفة ذات المسامية العالية من فقدان دورة السائل داخل التكوين الصخري.

تستخدم بعض التقنيات في العديد من التطبيقات وفي مختلف الصناعات في عملية تحسين متانة وجودة وخواص تلك المواد كصناعة البوليمرات و الألياف الإلكترونيات والأدوية الحيوية وصناعة الخرسانات. تمتاز تلك المواد بأنها ذات مساحة سطح عالية وحجم صغير، هذه الميزات جعلتها محبذة الإستخدام في صناعة النفط. أحرزت إستخدام تلك التقنيات في صناعة النفط تقدماً في العقود الأخيرة.

في هذه الرسالة، سندرس تأثير إضافة الألياف البولمرية (PPF) في إسمنت أبار النفط تحت ظروف الضغط ودرجة الحرارة العالتين (HPHT). هذه الألياف البولمرية ستضاف بنسب مختلفة لخليط الإسمنت المستخدم حالياً في عملية سمنتة الأبار في المملكة العربية السعودية. ستجرى دراسات مخبرية لمعرفة خصائص خليط الإسمنت كزمن التثخين، خاصية فصل الماء الحر، الخواص الريولوجية (الخواص المتعلقة بتشوه المادة)، القوة الإنضغاطية، الكثافة، دراسة المجهر الإلكتروني الماسح (SEM)، دراسة البلورات بالأشعة السينية (XRD) ودراسة الرقائق.

وفقاً لنتائج تلك التجارب، يتم إختيار أفضل تصميم للخليط الإسمنتي مع نسب مختلفة من الألياف البولمرية، من هذه التجارب، وجد أن إضافة تلك الألياف تحسن الخواص الميكانيكية للإسمنت، تقلل من خاصية فصل الماء الحر، تسرع عملية التثخين وتقلل من كثافة الإسمنت، وتساعد أيضاً في تصميم إسمنت جديد يقاوم ظروف الضغط والحرارة العاليتين بإعطاء نتائج جيدة.

Chapter 1

INTRODUCTION

1.1 OVERVIEW

Most of the oil/gas drilled wells in the world need to be cased to withstand the overburden pressure that comes from the formation. The casing set in the wellbore and then surrounded by cement to add more pressure stability to the wellbore. It helps to achieve the zonal isolation in the wellbore, to prevent the fluids communication behind the casing with the formation and to withstand the drilling shock, during perforating and fracturing operation.

The cementing technologies have been developed over the ages from 1920's by conducting laboratory tests which are equipped with strength-measuring devices and string equipment to identify the cement slurries fluidity and pumpability at HPHT conditions. The American Petroleum Institute (API) have assigned many regulations to utilize and standardize those tests.

The chemical composition of those cement slurries have been investigated to identify its properties and how they react with the formation fluids and rocks. Those components include shales, clays, silica sands, blast furnace slag, iron ores and pyrite ashes, making it classified according to API standards to nine distinct classes (From A to G), Classes G and H are the common used ones in the worldwide cementing procedures. The hydration process is quite important to determine the cement stiffening, hardening and strength development results, those parameters are proportionally directed with the settling time and temperature. Proper laboratory job simulations needed to select the best composition of the cement slurries. (Calvert *et al.* 1990)

The producing well performance depends largely on the quality of primary cementing job, starting to know the wellbore geometry, mud weight and type, cement-column height and formation types, all which planned before the start of drilling job. The hydraulic seals must be set well to ensure the quality of the cementing job, those procedures are aimed mainly to reduce the drilling days and mud costs. (Murtaza *et al.* 2013)

The fluid-loss rate control plays a major role for selecting the appropriate additives that added to the cement slurries, and ensuring more quality control will exclude the endurable errors or mistakes which can happen in the cementing job.

The primary cementing equipment selection depends on the geological constructions as well as the wells requirements to improve the mud removal from the wellbore after drilling process. That equipment consists of the floating equipment which been determined by either durability under downhole conditions or differential-pressure capability, the multistage cementing collars which may completed by perforating distinct sections of the casing and pumping the cement slurries with multi-stage process, the cementing plugs which designed to withstand high landing forces. And other attachments for the casing which make the cementing jobs more proper like centralizers to uniform flow of the cement and scratchers to minimize the sticking casing off problems. Finally, the inner-string cementing methods to counter the problems of pumping high volumes of the cement slurries through the small-diameter pipes the casing outside and to minimize the cement volume that drilled out of conventional cemented large casing diameters. (Ford 1997)

1.2 POLYPROPYLENE CEMENTING FIBER

As known that the cement during the curing process, some cracks will be developed in the cement if the cement is brittle and weak in tension, or due to thermal expansion over a period if it is not designed properly. Many recent developments of secondary reinforcement in cement sheaths in different fields like constructions and wells has provided improvement in the deficiencies for the cement sheath. One of those developments that have been conducted in construction field is introducing the polypropylene fibers by Reliance Technology Centre, the cutting edge technology which a specialized secondary reinforcement material developed after long-term extensive research process. **Figure 1.1** shows the schematic illustration of the polypropylene fiber which has a triangular shape structure that provides higher surface area and higher flexural strength than the ordinary rounded shape structure. (Liu et al. 2012, Park et al. 2003).

Figure 1.1: Polypropylene fiber structure

1.2.1 Features and Benefits

- Improves resistance to plastics and drying shrinkage cracking.
- Inhibits growth of cracks and micro-cracks and provides stability to the cement set.
- Improves flexural toughness and increases split tensile strength.
- Enhances abrasion resistance and increases energy absorption of cements thereby improving the impact resistance.
- Acts as a pumping aid in making the cement more homogenous.
- Reduces the surface water absorption and the cement permeability
- Improves durability and enhances longevity of cement.

Figure 1.2 illustrate the main mechanism of the fiber for creating a mesh network for improving the tensile strength of the cement as well as further control of lost circulation.

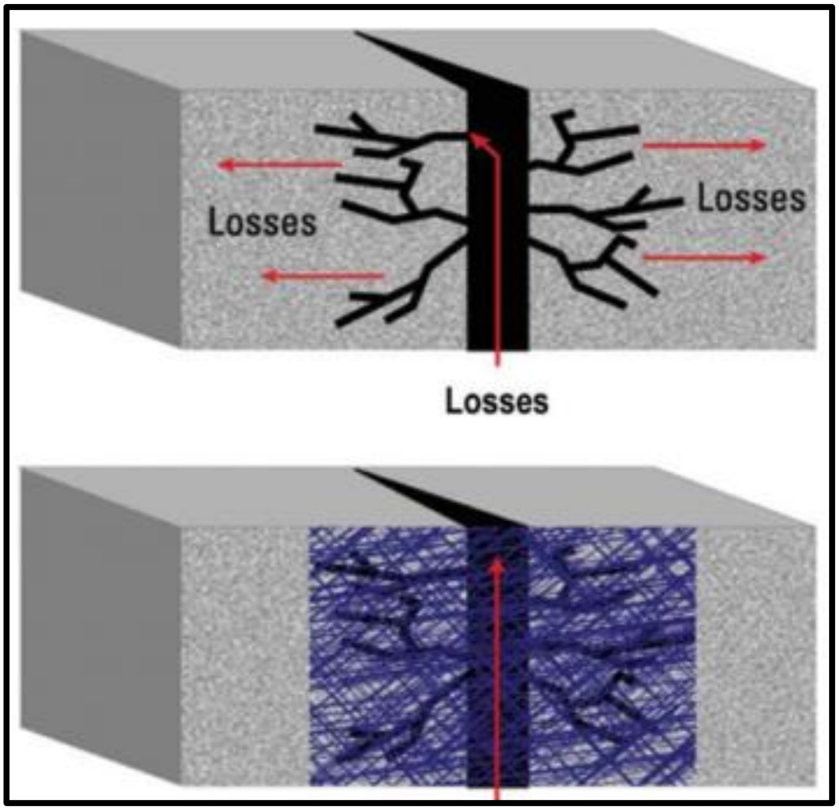

Figure 1.2: Fiber mechanism on creating the mesh network to control the lost circulation. After (Messier et al. 2003)

Table 1.1 shows the Polypropylene fiber specification that is preferable for usage in this work because of the cost efficiency as well as high temperature withstanding properties.

Table 1.1: Commercialized polypropylene fiber (Recron 3s) specifications (Patel et al. 2015, Madhavi et al. 2015)

Property	**Value**
Cut length	6 – 12 mm (High surface area)
Fiber shape (Triangular cross section)	Special for aggregated cement holding improvement
Tensile strength	4000 – 6000 kg/cm^2 (56000 – 85000 psia)
Melting point	Greater than 250°C (482°F)

1.3 RESEARCH MOTIVATION

The petroleum industry has been expanded worldwide in the late decades. This growth has been associated with a jump in exploring more undiscovered areas, and targeting deep zones in hope to find new reservoirs and trying to put them into production. However, the industry has faced several challenges in most of these exploration zones which require more research to be conducted in order to get over these challenges, and come up with improved and developed techniques. In fact, well cementing is considered one of the difficult challenges faced during drilling as well as well completion. In other words, the poor cementing job might result in serious problems and eventually threaten the success of the cementing job or ruin the cement well.

Poor cementing jobs took place in HPHT wells might cause serious problems, for example, gas migration, communication between formation zones, fluid contamination, and strength retrogression, and quick solution need to be applied to handle this problem property. Thus, oil companies and universities are continuously conducting researches and projects in a hope to come up with a new cement mix design or new chemical materials that can improve the cementing process in oil or gas wells (Messier *et al.* 2003) (Al-Yami et al. 2006). From the literature review it can be conclude that using polypropylene fiber with the cement has not been investigated for oil well cementing application at HPHT conditions. Because of this, more investigation in this area may open a new door for a cheaper and more effective material which can help in solving many cementing problems.

1.4 PROBLEM STATEMENT

The polypropylene fiber has been used for decades in several ways in the concrete job operations which was first introduced an admixture in 1965 (Madhavi et al. 2015) (Husain & Aggarwal 2015). However, from the literature review, it has been seen that polypropylene fiber has not been investigated for application in oil/gas well cementing. So, investigating polypropylene fiber with cement may open a new door to cheap additives of cement. Their ability to improve concrete properties and to sustain cement strength in high temperature application has made them a favorable mixture with cement. Few literature reports are available mentioning use of polymer fibers in the petroleum industry (Heinold et al. 2002) (Ramirez-Vazquez et al. 2014). Their results showed improvement in compressive and flexural strength compared to plain cement mortar.

When cement is pumped to the well, properties such as rheology, thickening time, gas migration, water loss, shrinkage, development of slurry strength with time and porosity and permeability of cement are as critical as high compressive strength developed after cement setting. This research investigates the effect of polypropylene fiber on the cement properties such as density, fluid loss, thickening time, rheology, density, compressive strength, tensile strength, porosity and permeability under HPHT conditions.

1.5 RESEARCH OBJECTIVES

The main objective of this study is to investigate the effect of adding the polymer fibers on Class G cement at HPHT conditions as well as to determine the optimum design of cement mixture with polymer fibers. To achieve these objectives, several cementing tests were conducted at reservoir conditions to study the cement properties as follows:

a. Thickening time
b. Fluid loss test.
c. Density
d. Rheology
e. Free water separation
f. Compressive strength test
    - Compressive strength by “Crushing” method
    - Compressive strength by “Sonic” method
g. Brazilian tensile strength test
h. Microstructural analysis
    - XRD (X-ray Diffraction)
    - SEM (Scanning Electron Microscope)

The following are the specific objectives of this study:

1. Investigate the effect of polymer fiber usage on the cement integrity.
2. Evaluate the usage of fiber with and without additives to enhance the cement integrity.
3. Identify the applicable and optimum cement mixture with fibers for the well cementing application.
4. Study the microstructural characterization of the cement with fiber.

1.6 THESIS ORGANIZATION

This research is organized with respect to the rules identified by the Deanship of Graduate Studies of King Fahd University of Petroleum & Mineral. It has been divided into five chapters as follows:

Chapter 1: starts with a brief overview about the oil well cementing and its concepts, and an introduction of the polypropylene fiber that was used in this study with its features and properties. The problem statement, research motivation and the objectives are introduced as well in this chapter.

Chapter 2: discusses the main principle of oil well cementing process as well as the physical and chemical properties related to cementing. It also explains the types of oil well cements used in the industry, and cement additives with their main function. In addition, it presents a literature review, which has been implemented in the same area of oil well cementing and also the chemical additives used during cementing process development.

Chapter 3: describes the steps and the procedure that has been followed for conducting the cement experiments according to the API standards. In this chapter there are several cement tests that explained in details along with use of each test to address certain cement properties.

Chapter 4: explains the results and discussions of all experimental tests conducted to analyze the behaviour of cement.

Chapter 5: explains the conclusion and highlights the major research outcomes with recommendations for future work.

Chapter 2

LITERATURE REVIEW

2.1 INTRODUCTION

Oil well cementing is described as the process where the cement is placed in the annulus space in between the formation wellbore surface and the casing. In general, cement is defined as a binder material which sets after a certain time, and hardens resulting in bonding of other materials together. A cement system used in oil well cementing, is called a cement slurry, where it contains different materials that give the cement favourable properties, and helps in improving its performance. The cement system consists of three main components, cement powder, water, and cement chemical additives. The first component is the cement powder (Portland land cement), where it consists of calcium silicate, calcium aluminate, in addition to other oxide components. Water is the second element in the cement system, and it gives the cement its fluidity and works as a hydrating agent in the cementing operation. The amount of water added to the cement is considered a critical issue, and the water cement ratio is usually picked and optimized carefully to provide appropriate cement slurry. In other words, lower percentages of water cement ratio might result in high viscosity cement and cause quick setting, whereas higher percentages might lead to the presence of free water and a drop in the final cement density. Finally, cement chemical additives are the last component in the cement mix, and used to enhance cement behaviour by giving the cement new favourable properties.

Oil well cement was firstly started at the end of 1920s, with the main objective of supporting the casing and later prevent shocks when resumed drilling further in the same well. It also prevents the casing from getting corroded and stops salt water from flowing into oil producing areas (Shahriar 2011). Furthermore, cement is used to stop fluid migration between fracture zones, where the cement acts as either a plug to stop lost circulation problems, or as solution for shutting down the wells in the abandonment period. (Bourgoyne et al. 1991).

Oil well cement should be designed in a way to adapt with porous or weak formation, corrosive formation fluids, and high pressurized regions. In addition, other parameters like well bore geometry, drilling mud, casing, formation safety, as well as mixing of cement must be taken into consideration during the designing process of the cement. Later, when cement is placed, cement mechanical properties, and long-term durability must be controlled, and precisely identified to avoid future cement failure, especially under severe conditions. Therefore, there are a number of oil well cement classes authorized by the American Petroleum Instate API, and each has different implementation functions depending on the encountered conditions (API spec. 10, 2012). Also, a number of chemical additives are added to the cement to change cement chemical, and physical properties ensuring good fluidity, pumpiability, and long term performance of the cement. Extensive work has been done for enhancing the effectiveness of the production zones through improving the mechanical and physical properties of oil well cements. In this chapter, the basic concepts in oil well cementing has been addressed, the commonly used cements, as well as physical and chemical properties exerted by these cements. Good understanding of the cement additives might help in selecting the correct cement additives, and their correct percentages for the cemented well. Furthermore, cement additives can alter the behaviour of the cement systems, provide a successful placement cement job through the wellbore, rapid development of compressive strength, and sufficient zonal isolation throughout the life of the wells is obtained.

2.2 WELL CEMENT CONCEPT

Generally, a normal depth of oil or gas well might extend to a few thousand meters with a diameter of around one meter. The oil well can be constructed by putting a metal casing through the well bore, and then bound it using a particular cement slurry mix in which it seals the annular space and then provide a good bonding between the casing and the hole wall surface. Sometimes cement slurries are pumped into deep wells, where depths might exceed 20000 ft. In these deep wells, the temperature might increase up to 205^{o}C (400 ^{o}F), and might affect the properties of the produced cement sheath. When Portland cement is subjected to temperatures higher than 230°F, significant changes in cement phases will take place, and eventually could influence the compressive strength, increase in the permeability, and cause strength retrogression due to crystalline structure break down within these conditions (Shahriar 2011). So, a special treatment should be applied to Portland cement so it can perform well under these conditions.

Later, when the desired depth is reached, the drill pipe is pulled out the drilled hole, and the selected casing is run in the bottom of the well. After that, drilling mud has to be cleaned off and replaced with a strengthened cement. To make sure that a good bonding is obtained between the formation and the casing, appropriate amount of cement slurry is pumped into the bore hole through the casing and then enforced through the annular space outside between the formation well bore surface and the casing with the help of two-plug during the cementing process (Oilfield Glossary, 2009). The pressure is applied on the top of cement plugs using an aqueous liquid which is used to push any cement remains inside the casing. There are two types of cement plugs used in oil well cementing, the top and the bottom plug. The objective of these plugs is to help in pushing the cement through the casing, as well as reduce cement contamination by the other liquids remaining inside the casing before the pumping process of the cement slurry (Oilfield Glossary, 2009).

Normally, when cement is pumped into the well, it is placed at much higher depth than the production zones, which resulted in reduction of undesirable fluids, corrosion of the casing, and prevent fresh water zones (Calvert et al. 1990). Once the cementing process is finished, the cement is left for a certain time to cure and to get hardened before drilling is resumed to a deeper horizon. In addition, the hard cement yields a low permeability annulus, which isolates the productive zones from other neighbouring zones in the wellbore.

2.3 CLASSIFICATION OF OIL WELL CEMENT

Oil well cement slurries are normally prepared from cement clinker or hydraulic mixed cements, these materials are considered as the main components in the cement slurry mix. The cement is pumped into the annular space, and then left to set and harden so as to provide good bonding between the casing and the borehole wall surface (Chatterji et al. ,1983). In the early ages of well cementing, there were two types of cement available in the market which are the clay and the lime. With the increasing demand for oil, more new deep oil and gas wells has been drilled, and cement types have to be improved to cope with this speed of development. So, well cementing using the previous two kinds of cement was resulted in unsatisfactory performance of the cement sheath which resulted due to the change in the environmental conditions within these deep wells. After that, the API committee was founded in 1937, and this was followed by extensive research on oil well cements to produce a better functionalities cement classes suitable for different environmental conditions (Smith 1989). Oil well cements are divided into eight cement class range from class A to H. This classification is done depending on its content of tricalcium aluminate (C_3A) which is listed as the moderate and the high sulphate resistant (MSR, HSR) as well as ordinary (O) cement. In addition, each type of cement classes is selected depending on certain factors such as well depth, temperature, pressure, and the presence of sulphate within the well. Furthermore, the most frequently used well-cement classes in the world are class A, G, and H. In fact, class A cement is usually used in a milder well, where the conditions are unchallenged, whereas class G and H, are used in deeper wells where higher pressure and temperature conditions are faced (Shahriar 2011). **Table 2.1** shows the API cement class, and their properties (API Specification 10A, 2002; Shahriar, 2011).

Table 2.1: API cement class and properties (API Specification 10A, 2002; Shahriar, 2011)

Cement Class	**Recommended w/c%**	**Recommended range of depth, ft**	**Availability**	**Cost**	**Other features**
A	46	0 – 6000	O class, suitable to be used with ASTM C 150, Portland Cement Category I	Lower cost	Used when no special properties needed
B	46	0 – 6000	HSR*** and MSR** classes, close to that of ASTM C 150, Category II		1. Used when moderate or high sulphate resistance needed. 2. C3A content is lower compared to cement Class A.
C	56	0 – 6000	MSR** & HSR***, O' classes, almost identical to ASTM C 150, Category III	More Expensive compared with usual Portland cement	1. Used when high early strength needed. 2. High content of C3S. 3. High surface area.
D	38	6000 – 10000	HSR*** and MSR** classes		1. Used in conditions of moderate to high temperatures and pressure 2. Decreasing the amount of C3S and C3A cause cement retardation. 3. Cause a rise in the cement grain particle size.
E	38	10000 – 14000			1. Needed in high pressure temperatures wells. 2. Decreasing the amount of C3S and C3A cause cement retardation. 3. Cause a rise in the cement grain particle size.
F	38	10000 – 16000			1. Used in extremely high pressure and temperature conditions 2. Decreasing the amount of C3S and C3A cause cement retardation. 3. Cause a rise in the cement grain particle size.
G	44	0 – 8000		–	1. Normal used oil well cement. 2. Cement thickening time is controlled by using additives and aimed to stop the loss of circulation till 250°F.
H	38	0 – 8000		–	1. Normal used oil well cement. 2. The outside areas are coarser comparing with Class G cement. 3. Cement thickening time is controlled by using additives and aimed to stop the loss of circulation till 450°F.

* Ordinary cement ** Moderate Sulfate Resistant *** High Sulfate Resistant

Cement is also classified by another organization called the American Society for Testing and Materials (ASTM), where they divided it into eight cement classes depending on the application conditions. **Table 2.2** shows ASTM cement classification, and their application parameters. Cement class type I and II are mostly used cements in the United State where its consumption percentage can reach 92% for the whole Portland cement used (Mobeen, 2013).

Table 2.2: ASTM cement Classification

ASTM Cement Class	Properties
I	1. Equivalent to cement API class B 2. Normal cement uses, where no mitigating conditions are presented.
II	1. Equivalent to cement API class B 2. Moderate sulfate resistance is provided.
III	1. Equivalent to cement API class C 2. Used in conditions where high early strength is needed.
IV	Used in conditions where heat of hydration low is required.
V	Used in conditions where high sulfate resistance is needed.
IA	1. Used as entraining agent 2. Consists of cement class I with integral air.
IIA	1. Used as entraining agent 2. Consists of cement class II with integral air.
IIIA	1. Used as entraining agent 2. Consists of cement class III with integral air.

2.4 OIL WELL CEMENT ADDITITVES

There are eight types of cement additives added to the cement to improve the cement performance for a successful cementing job. These cement additives are accelerators, retarders, weighting agents, extenders, dispersants, lost circulation control agents, fluid-loss control agents, and other additives like fibers, and antifoam agents. Retarders and accelerators are used as cement additives and aimed to control the setting property of the cement, whereas the main function of the cement weighting agents is to raise the cement density by incorporating it with light-weight systems to obtained the cement mix. Extenders are used to reduce the cement density, so a light weight cement is produced associated with improvement in the cement yield. Furthermore, the addition of cement dispersants provides more control in the viscosity of the produced cement mix. In the same way, there are other cement additives that work as viscosifiers such as fluid-loss control agents which help in controlling the fluid loss, and minimizing the penetration of the cement aqueous phase into the formation so a constant cement water ratio is obtained within the pumped cement. On the other hand, lost circulation control agents help in stopping the penetration of the cement into regular or weak formation. Extensive literature review regarding cement additives was presented by Nelson. In addition to the previous cement chemical additives, there are other types of cement additives that are mixed with the cement and caused improvement in cement mechanical properties. Example for these materials are fly ash, powdered coal, diatomaceous earth, silica, and gilsonite (Nelson et al., 1990, 2006).

2.4.1 Accelerating and Retarding Agents

Cement thickening time is a critical issue, and it differs from well to well depending on the wellbore conditions. The main objective of these cements thickening time additives is to control the cement thickening time, so the cement remains pumpable through the cementing process until it reaches the target and placed in position between the formation wall surface and the casing. Therefore, in the case of cementing shallow wells, where the pressure and temperature are considerably low, the need for long cement thickening time is not required, and as a result, accelerators are added to the cement mix. Examples of these accelerators are calcium chloride, and sodium chloride. On the other hand, in the case of deep wells, where the conditions of both high pressure and temperature are faced, the cement thickening should be extended, so the cementing job can be finished without of encountering early hardening problems of the cement paste. In this case retarders played the main role, and examples of these retards used commonly in oil well cementing are calcium lignosulfonates and borax.

2.4.2 Fluid Loss Control Agents

The cement slurry filtrate is one of the important aspects, particularly during cementing productive as well as high permeable formations, where precaution or proper handling should be taken, in order to keep the quantity of this filtrate as low as possible. This problem is called fluid loss, and to solve it, several additives are added to the cement to control the rate of filtration loss, and to keep it in the acceptable ranges authorized by the industry. An example of these fluid loss additives is cellulose derivatives, and organic polymers.

2.4.3 Expanding Agents

In the cementing process, the cement is pumped through the casing and reversed to fill the annular space between both the formation wall surface and the casing. After cementing the well, the cement is left to set and harden prior to resuming drilling or completion jobs. On the other hand, the produced cement column might suffer from other problems, such as shrinkage due to severe conditions of both high pressure and temperature. Thus, expansion agents are added to the cement to ligament the casing or liner with the well bore, so a healthy well is obtained without shrinkage problems. In addition, gas and fluid migration problems are reduced, and a better zonal isolation as well as enhancement in well productivity is accomplished.

2.4.4 Anti-foaming Agents

The problem of air entrapping is normally faced during the mixing of the cement. This entrapped air might cause a few problems such as pump failure resulted from the damage caused by the air, unreliable or wrong cement density as well as a high porous produced cement sheath. Anti-foaming agents are available in liquid or powder state, and the ultimate objective of these deformers is to reduce foaming and are usually used with most of cement systems.

2.4.5 Free Water Control Additives

The main objective of the free water cement additives is to bind or hold the water that is used in the extended cements together with the cement particles, so no free water appeared in the mix. Free water is a critical problem, and action should be taken, since water in the cement might be absorbed by the adjacent formation, and might cause a change in the properties of the produced cement sheath. Free water is also a problem in the case of cementing horizontal well, where water will accumulate at the top of the cemented section and will cause problems in future. An example of the free water cement additives is aluminum chlorohydrate.

2.4.6 Lost Circulation Control Additives

Lost circulation is a critical issue in which more attention should be paid during the cementing operation. Lost circulation is usually encountered during cementing high permeability zones, natural or induced fracture, vugs, and weak formation. This problem can be solved by either lowering the cement density, or by introducing special materials to the cement that form a bridge like material around these openings, and end up in plugging these high permeable or fracture formation. Examples of lost circulation control agents such as fibrous agents (nylon), flake type (cellophane), and granular type (gilsonite).

2.4.7 Weighting Agents

During the cementing process of deep wells, cement density must be increased in order to control the formation pressure. As a result of this, cement weighting agents are added to the cement to raise the cement density, and overcome the pressure. Examples of these weighting agents are hematite, and barite.

2.4.8 Dispersing Agents

The main objective of these dispersion additives is to improve the rheological properties of the cement, so good pumping and mixing cementing properties are obtained. These dispersion materials work by reducing the friction forces between the cement particles, and results in reduction of the water/cement ratio so more compressive strength is gained. Furthermore, turbulent flow is observed when added to the cement mix, which resulted in better mud removal through the wellbore. On the other hand, these dispersants can result in extending of the cement thickening time, so caution should be taken while using them. Also, these cement additives are enhanced by various oil companies, and are provided in liquid or powder phase.

2.4.9 Strength Retrogression Agents

In severe conditions, where the temperature might exceed 230^{o}F, the strength retrogression problem appears within this range of temperature and it affects the productivity as well as the integrity of the well. Hence, to get rid of this strength retrogression problem, any silica products, for example, silica sand, or silica flour is added to the cement mix. Silica flour is added to the cement mix at percentages ranged between 35–40% to maintain compressive strength and prolong life time of the well. (Iverson et al., 2010).

2.5 CEMENTING DESIGN PROCESS

Millions of dollars are usually spent in the drilling and well completion, hence it is also an obligation to come up with a good design cement program that can avoid remedial cementing jobs which would put extra cost to the project. The cement is designed according to the conditions of the particular well/formation, and this is usually followed with lab tests to evaluate the properties of the cement.

(**Ravi & Xenakis 2007**) explained the steps needed in the case of designing the cement (see **Figure 2.1**). The first step in the cement design process is having a detailed engineering analysis report. This step needs first defining the nature of formation, whether the formation is loose or hard one? Taking into consideration all the forces that might appear when the well is put on production. Also taking in mind if the cemented well is a type of high pressure or high temperature. In addition, the instruction illustrated in step one also includes stress analysis in order to see if the produced cement sheath can sustain the series of cyclic loads the well might face through its lifetime. Knowledge of the above parameters would lead to the second step which is the designing of the cement based on those factors. Cement properties such as Poisson's ratio, Young's modulus, tensile strength, shrinkage/expansion during hydration, plasticity parameters as well as post-cement slurry hydration which needs to be selected carefully, so it can match the well bore conditions. As a result, laboratory tests should be conducted on the proposed designed cement. After that, the results obtained from the laboratory tests in addition to that collected from the first step are analyzed together to evaluate the cement performance. The last step includes known the best drilling and cementing practices, for example, cleaning the well bore from the drilling mud, centering of casing during the cementing process, in addition to well monitoring.

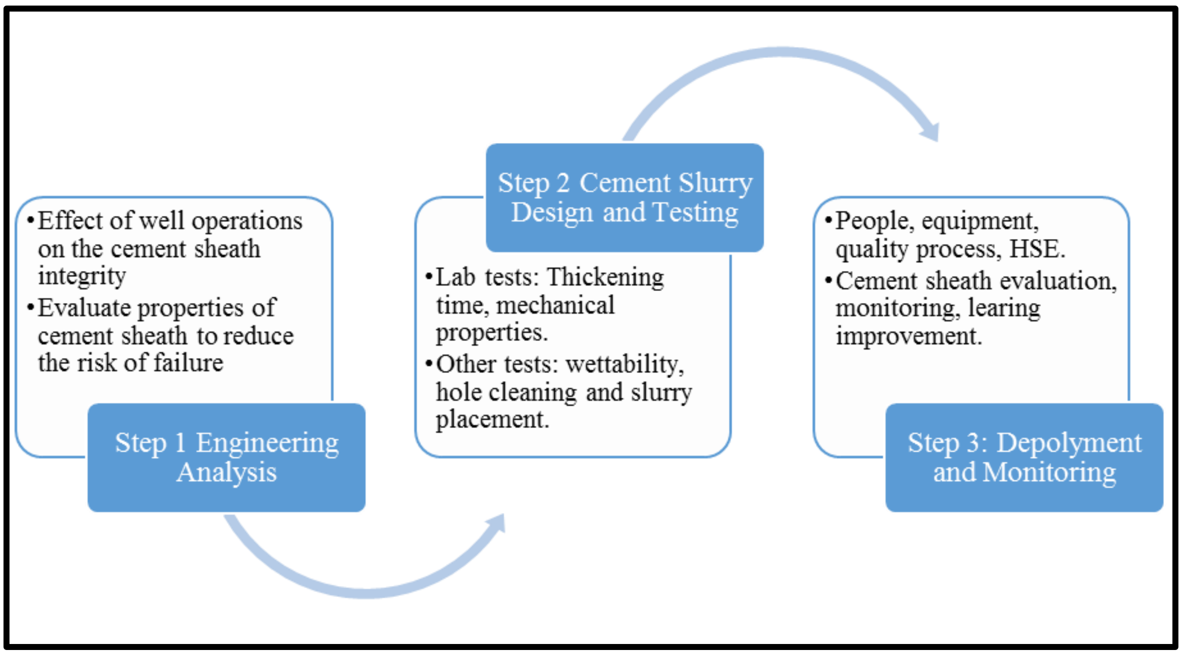

Figure 2.1: Cement slurry design basic steps (Ravi et al. 2007)

2.6 LITERATURE REVIEW

(Parker *et al.* 1965) presented an efficient technique of replacing the mud from the wellbore while placing cement into the wellbore. Cement when contacted with mud reacts instantly and creates a mass of very high gel strength having properties different from cement and mud. This is a major factor in the success of cementing job. Field results and laboratory studies suggested that low pumping rates for cement helps in effective displacement of mud through the wellbore.

(Parcevaux & Sault 1984) investigated the cement bonding properties under confined pressure and temperature curing conditions. It is found out that cement bonding properties were straightly proportional to cement shrinkage and elasticity. Laboratory and field experiments were implemented to compare the bonding performance of the standard cement compositions with that of modified cement composition with bonding agent.

(Baret 1988) presented a study on two types of fluid loss behavior during cementing i.e dynamic and static fluid loss. This study concluded that the fluid loss in dynamic behavior is dependent on the upper density limit. It is also presented formulations for calculating acceptable limits of fluid volume lost and mud cake build up during the respective behaviors. All the problems encountered due to the fluid loss during cementing were also discussed in this study.

(Shi *et al.* 1995) carried out a study of modifying the cement performance to improve the cementing job quality by analyzing many parameters that affect the cementing job, the first one is the cement stability which is controlled by many factors like the cement volume, the acceleration of cement shrinkage in the high temperature conditions especially for improving the cement resistance to the high temperature and the effect of curing humidity for cement volume shrinkage. The second parameter is the mechanical breaking properties effects for the cement sheath, by using an organic fiber the addition of 0.55% of that fiber the cement toughness increased up to 10% and with adding more up to 1.25%, the cement toughness increased by 40%. Also, they carried out the work with adding polymer to the cement which increases its toughness by 50% and they claimed that if an appropriate amount of fiber is added into the system, the cement toughness will improve straightly. Furthermore, the fiber-polymer compounded cement shows good results for improving the breaking resistance properties by conducting certain perforation simulation tests which was observed in enhancing the stress-stain properties as shown in **Figure 2.2.**

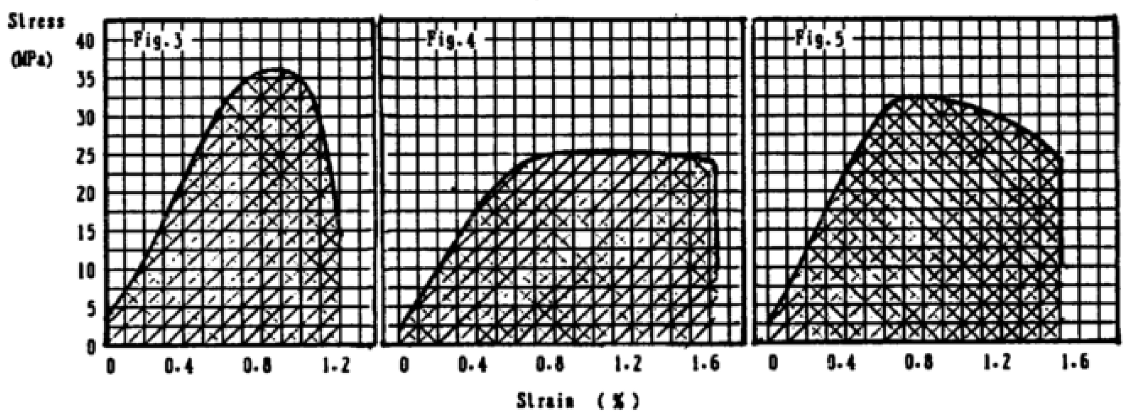

Figure 2.2: Stress-strain properties improvement for different types of cements (Right: Net cement, Middle: Fiber cement, Left: Polymer cement). After (Shi et al. 1995)

(Elmoneim *et al.* 2000) conducted a study for evaluating the cementing job for a deepest 20" casing in Gas Khuff basin in UAE using a blend of novel light-weight cement (10 ppg) slurry with adding fiber to reduce the lost circulation problems during the cementing job execution. This study claimed that the fiber concentration design depends on the losses type and severity, and for their case they did the job with concentration of 2 lbm/barrel cement slurry by adding them slowly to ensure that the fibers dispersed properly throughout the slurry. Using Schlumberger's LiteCRETE technology to develop the light-weight cement integrity.

(Gambino *et al.* 2001) evaluated the interactions between the fluids and rock using formation rock from Venezuela. Optimum criteria for cement design was established. X-ray diffraction, SEM, permeability and formation wettability were analyzed to evaluate the rock changes, in addition gas chromatography, GC-MS, NMR and IR were used to analyze the formation fluids. It is found that, a huge reduction in the formation permeability can be produced from the filtration of mud. Two different spacers were used to remove the formation damage and improve the permeability. It was stated that the rock formation global mineralogical composition did not change dramatically. Furthermore, clay minerals distribution changes, mainly kaolinite is the most oversensitive of the clay minerals to surfactants presence. The overall results will allow more achievement for design of the cement approach.

(Heinold *et al.* 2002) addressed the impact of cement additives for improving the mechanical properties of normal density cement sets such as flexural and tensile strength, it is claimed that the appropriate choice of those additives will strongly impact the enhancement of the cement mechanical performance, some of them could increase the tensile properties and not flexural properties and other do the contrary. The experiments were applied using monogrammed class G cement slurries with adding some polymers like Polyviyl Alcohol (PVA) for permeability reduction, Silica Fuma (SF) for compressive strength enhancement and CO_2 resistance increasing, Hydroxeythyl cellulose (HEC) for lost circulation control for high temperature conditions. And adding a fiber which called Wollastonite in high temperature conditions at 400^{o}F with 50% BWOC for enhancing the flexural and tensile properties as well as sulfate resistance. Each of those additives were added to cement individually per run test to see how those additives would react for improving those properties. By analyzing their tests, it is found that in case of HEC, Wollastonite and PVA, it exhibited an improvement for tensile strength in all temperature conditions, while in SF's case, it showed an enhancement in tensile strength at lower temperatures but exhibited a damaging effect on tensile strength at high temperature at 200^{o}F. It was concluded in the end by mixing those additives with appropriate ratio might improve the whole mechanical properties with excellent levels at all temperature conditions.

(Shaughnessy *et al.* 2002) addressed the procedures and philosophy of the cementing operation by using reverse circulating methods. It was stated that, successful cementing operations are critical to economically completing the high temperature and high pressure wells. More than 15 wells were studies to demonstrate that, the records of production rates can proof the success of cement job. It was concluded that, the successful keys for optimum cement work are; the best known conditions for the downhole and drilling mud should be used to test the cement slurry comprehensively, a simulation model should be used to simulate the cement displacement prior to the job, then the cement test and the simulation model should match the field experience, and an overall attention should be given to details from testing and simulation.

(Messier *et al.* 2003) applied the usage of silicic fibers in the cementing process through shallow coal seams which predicted to have unpredictable lost circulation while cementing in shallow gas wells in Eastern Irrigation Block in southern Alberta. Those fibers were added into cement slurries at concentration of 2.1 lbm/bbl which results in efficient reduction in the lost circulation by observing the increment of returned cements volume as well as decreasing in the drilling operation costs more than 50% of the original cost. Those fibers also reduce the environmental impact by limiting the cement volumes for surface disposal.

(Morris *et al.* 2003) designed a high tough cement sets for effective well isolation for improving the mechanical properties of the cement by conducting mechanical tests, perforator tests and field tests using logs. All those tests were carried out using two scenarios. The first one is without adding any admixed fibers which shows some enhancement for all mechanical properties and the second one is with adding those admixed fibers which observed to be useful for improving those properties in addition to the elastic behavior of cement as well as toughness performance which increased by eight times than those in the normal cements. Also, it improves the bonding strength between casing and the formation which provides a good isolation for those formations. Moreover, those fibers help to control the cement deterioration and cracking around the perforator hole which was confirmed by running sonic logs to check their integrity before and after the perforation for both cases.

(Mahmoudkhani *et al.* 2008) developed new environmental friendly cement for cementing operation in oil and gas wells, their slurries reduce greenhouse gas significantly in comparison with the conventional cement slurries. The slurries sets consist of geopolymeric materials that exhibit superior chemical and mechanical characteristics at a low cost. Aluminosilicate materials were used to replace the weight of cement by up to 60%, which lead to lightweight slurries with high desirable elasticity and compressive and flexural strengths. The cement can be used in a wide range of density and provides predominant cement bonds and long-term performance.

Figure 2.3 shows the reduction of CO_2 amount per cubic meter of geopolymer-cement slurries based on neat cement content, CO_2 amount can be reduced to the half when the cement contains 60 % of the geopolymer materials. **Figure 2.4** compares the amount of required water per cubic meter for geopolymer-cement slurries and neat cement, using cement slurry with 60% of the geopolymer materials leads to reduce the water requirement by 15%.

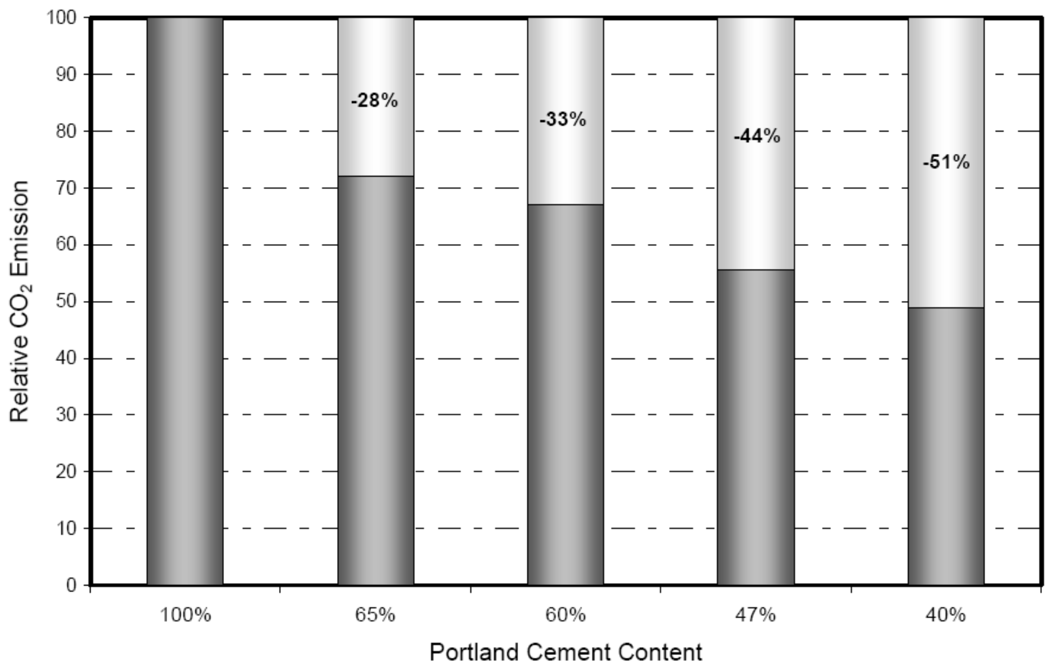

Figure 2.3: CO_2 Emission reduction per cubic meter of geopolymer-cement slurries. After (Mahmoudkhani et al. 2008)

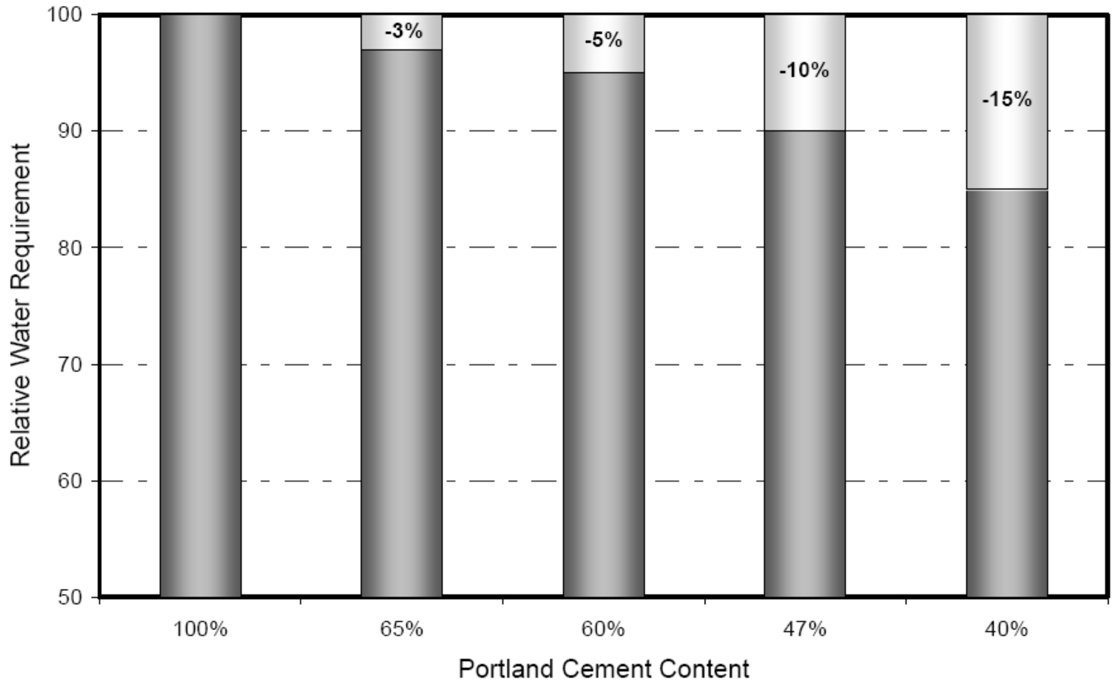

Figure 2.4: A comparative sketch of water requirement per cubic meter for neat cement and geopolymer-cement slurries. After (Mahmoudkhani et al. 2008)

(Teodoriu *et al.* 2010) investigated the theoretical background and construction of a new testing instrumentation to study the cement long-term sealability. The testing facilities were designed to simulate the load variations and their effect on the cement set. Finite element method (FEM) analysis was used to adjust the loads during the tests, which allows stresses generation in the test instrumentation equal to those at the interface between cement and casing in the wellbore. The results show that, the micro annulus and radial cracks in the cement set can be identified from the measured test gas pressure drop, radial and axial strains and axial displacement.

(Yalcinkaya *et al.* 2011) studied experimentally the changes inside the internal fractures of cement when exposed to acidic brine through an artificial fracture, by conducting core-flood with 12-in cement core and CO_2 saturated brine at flow-rate of 2 ml/min in a coreflooding device with low overburden and injection pressure system (600 psia respectively). The tests are repeated with high overburden and injection pressure system (1800 and 600 psia respectively) in ten days to evaluate the cement degradation process pressure. High resolution X-Ray and CT scans were conducted. It showed that the total porosity decreased from 26% to 22% after one month of exposure in the low pressure system. While in the high pressure system, no changes in total porosity were observed because of the duration of test, but the micro-porosity of this system was changed during that duration. It was observed that porosity decreased with exposure of the acidic brine for the low pressure system which is higher than high pressure system as seen in **Figure 2.5.**

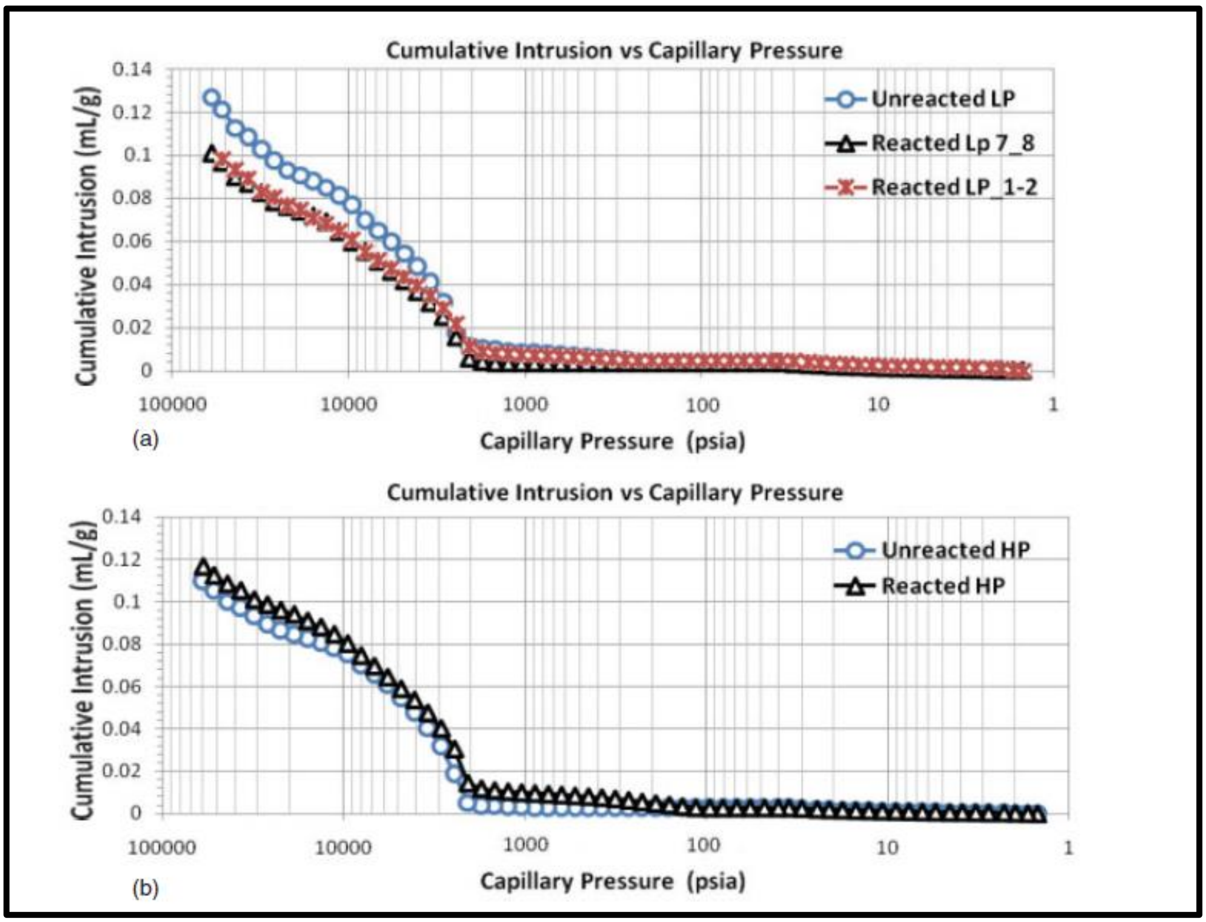

Figure 2.5: The cumulative intrusion vs. mercury injection pressure for the both high and low pressure systems. (After Yalcinkaya et al. 2011)

(Neuville *et al.* 2012) studied the class G cement paste properties exposed to brine at HPHT conditions, showing that the pressure has major impact on kinetics of the paste degradation especially for the modifications of the cement mineralogy and microstructure can be expected in the cement external zones. The calcium carbonate precipitation at 70 and 200 bars shows that the material transport properties could be improved. Also the results observe that the diffusion rate controls the kinetics slower than the chemical reaction rate which result in lowering the mechanical strength.

(Brandl *et al.* 2012) proposed a modified multifunctional polymer to improve the well cementing quality. A modified cellulose-based polymer which is an environmentally friend polymer was used to control the fluid loss at temperature up to 170°F. They found that, this single additive works as a foam stabilizer and gas control agent in cement slurries, which was not observed for any other cellulose-based polymers tested. In addition, the modified cellulose-based polymer exhibits less slurry viscosification and therefore, facilitates the surface mixing and pumping during the cement operations. This single additive can replace many additives in a cement set to adjust the required cement slurry performances for optimum placement. A case study of deepwater cementing operation was accomplished to show the successful application of the multifunctional polymer.

A static fluid loss control at high temperatures was conducted to evaluate the performance of the modified cellulose-based polymer in controlling the fluid loss, as shown in **Figure 2.6**. A very good API fluid loss control (<50 ml/30 min) was found for the dosage of 0.2% by weight of blend (bwob) up to 170°F. At temperatures above 170°F, the API fluid loss significantly increases, which can be attributed to poor water solubility of the modified cellulose-based polymer at higher temperatures. Therefore, the modified polymer is an effective cement fluid loss additive at temperatures up to 170°F.

To achieve successful and safe cementing operations, the cement slurry should be designed to withstand the gas flux during and after placement in highly pressured formation (Rogers et al. 2004). The performance of the modified cellulose-based polymer as a gas control additive in cement slurries was evaluated using a gas flow model at predicted bottom hole circulation temperature (BHCT). **Figure 2.7** shows the outputs of gas flow model test at high temperature (86°F) for class G cement mixed with 0.6 gps of the modified cellulose-based polymer, 1.0 gps of a sodium silicate and 12.0 ppg of sea water. The cement matrix permeability was found to be very low at the end of the test, which prevents the gas mitigation. As a result, the modified cellulose-based polymer has successfully passed the gas flow model test.

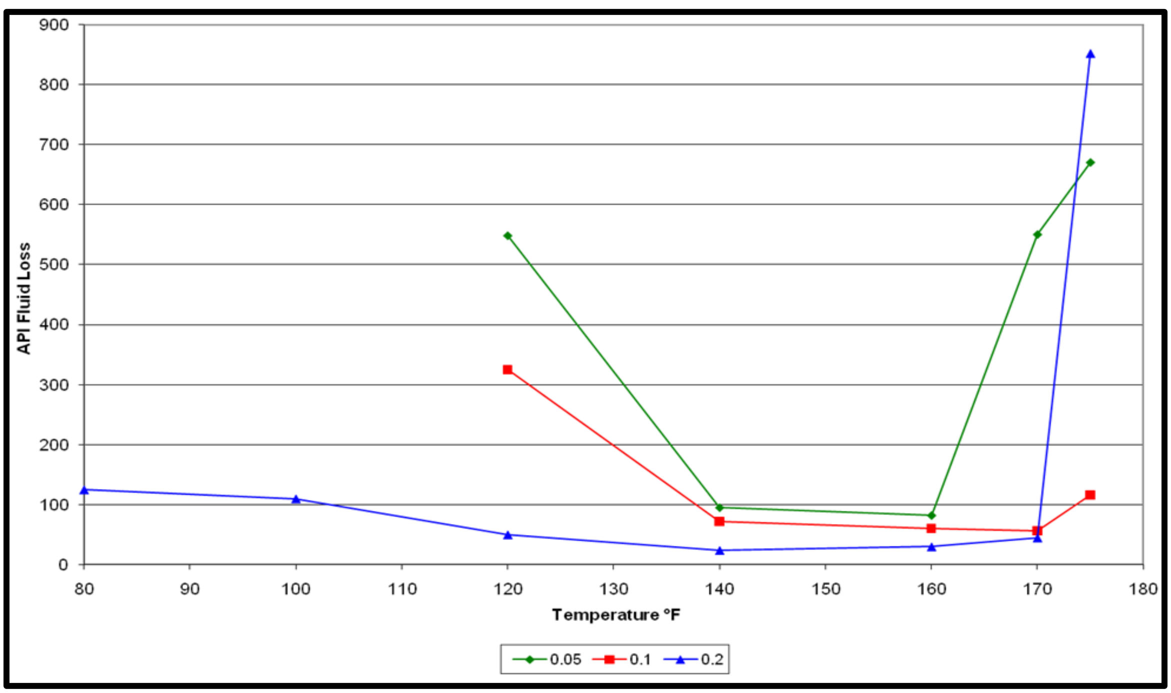

Figure 2.6: Fluid loss control for cement slurries (15.6 ppg) containing the modified cellulose-based polymer (% bwob). (After Brandl et al. 2012)

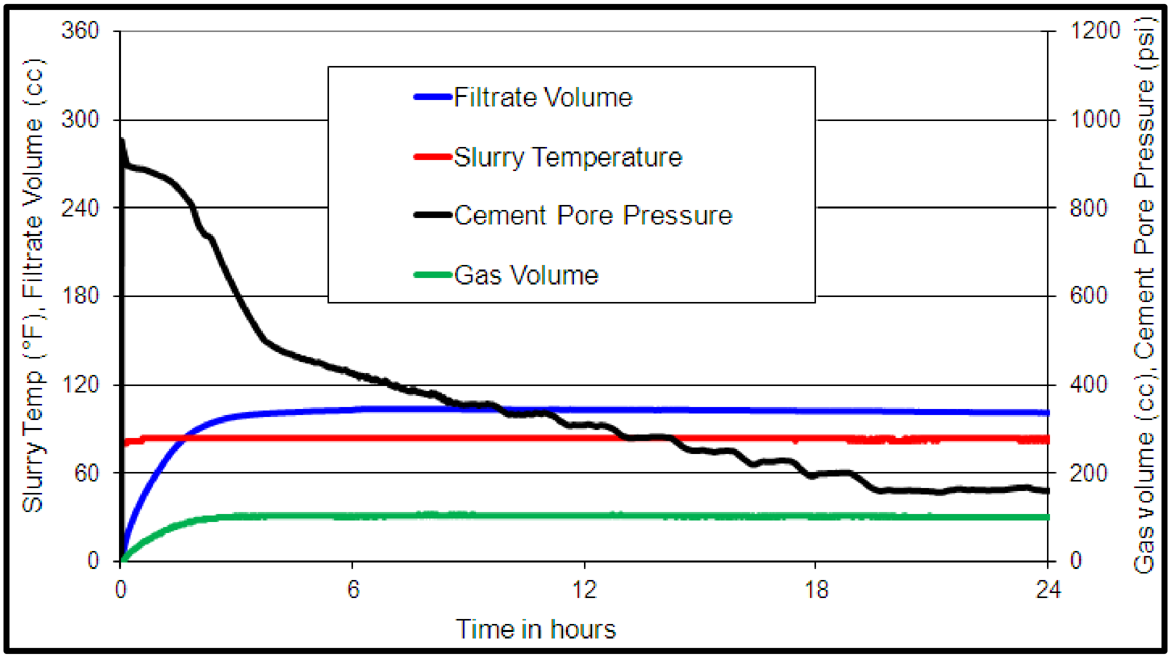

Figure 2.7: Output of a gas flow model test at 86°F BHCT. (After Brandl et al. 2012)

(Ilyas *et al.* 2012) carried out an extensive study for how to improve the cementing process in the deep wells to provide good zonal isolation in a field in Pakistan, they used a fiber to achieve further lost circulation control by consolidating the loss zones as they shown in the cement bond logs and variable density logs.

(Arshad *et al.* 2014) developed a set of engineered fiber base lost circulation control pills to encounter the lost circulation problems, and it showed that they were impactful in curing losses during drilling and cementing allowing to increase the mud weight up to 17 lbm/gal. It shows successful results in reducing the non-productive time (NPT), minimizing the quality, health, safety and environment concerned to the well control and achieving more zonal isolation. Those pills use a combination between the physical properties and fibers and solids to plug fractures, mini-cracks that occur during the drilling and cementing process as seen in **Figure 2.8**. Those pills could distribute the fibers and solid evenly and they are well engineered to improve the performance robustness and optimize the fiber mesh network. When they had been applied in the field cases in northern Pakistan for cementing procedure by adding 75 bbls of 16.5 lbm/gal of those pills, it is shown that there is increment of surge pressure due to poor particles induced the fractures. It also provides a good zonal isolation and good cement coverage as seen in the cement logs.

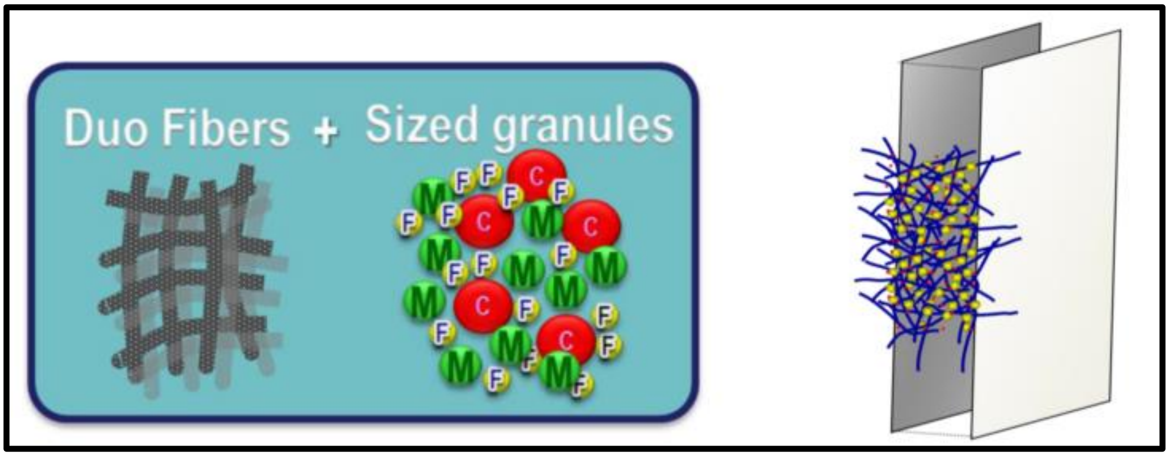

Figure 2.8: Mechanism of engineered fiber system plugging. (After Arshad et al. 2014)

(Brandl *et al.* 2014) implemented a study for how to combat the severe lost circulation problems by with improving the cement quality in an offshore carbonate formation in Malaysia. A sealing spacer with 14.5 ppg thixotropic cement was used with conventional synthetic fibers for further lost circulation control. But due to high temperature conditions (275^{o}F) that exceeded the maximum application temperature of those fibers, they modified the cement to be light weighted of 11.5 ppg to control the cement stability in that high temperature conditions as well as avoiding further induction of fractures alongside the carbonate formation.

(Maskary *et al.* 2014) conducted a study for improving the cementing job by using monofilament polypropane fibers to encounter and cure the lost circulation problems, in the laboratory tests it shows the effectiveness of fiber in plugging different sizes and it was improved when combined high-performance light-weight cement slurries were used selecting the optimum particle size distribution system.

(Ramirez-Vazquez *et al.* 2014) developed some technologies to carry out a successful cementing job in steam injection wells in southern Mexico by using optimized and high performance thermal cement sets in addition to usage of a new sets of resin-based centralizers to improve the casing/cementing placement integrity. Also it leads to reduce the lost circulation problems and improve the bonding strength of the cementing system. A 3mm glass fibers was used to increase the tensile strength without significantly decreasing the compressive strength, prevent the lost circulation problem and enhance the cement thixotropy by forming a fiber-mesh network. Also a latex is used for acid resistance during the steam injection processes, fluid-loss control and high temperature suspension properties. All those additives together mixed with slurries leads to prevent the lost circulation problem, good results in the cementing bond logs (CBL), no annual channels avoiding the trapped fluids to escape, the cements becomes thermally and mechanically stable during the production life.

Chapter 3

METHODLOGY

Cement laboratory tests are the key factors of understanding actual cement behavior under specific conditions. Prior to any field cementing job, engineers always do those tests so as to evaluate, and enhance the properties of cement system, so that it can match cement actual behavior in both high pressure and temperature down hole conditions.

In this study, a number of cement tests were conducted according the American Petroleum Institute standard procedures (API spec. 10, 2012), where each of these cement tests is conducted to study specific cement properties.

Based on those tests, the effect of polypropylene fiber on Portland cement has been investigated, the cement properties addressed in this study are:

a. Thickening time cement test.
b. Fluid loss test.
c. Free water separation test.
d. Density
e. Rheology
f. Compressive strength test
    - By “Crushing” method
    - By “Sonic” method

g. Microstructural analysis
    - X-Ray Diffraction (XRD)

- Scanning Electron Microscope (SEM)

3.1 WELL SPECIFICATION

A typical well in Saudi Arabia is picked to see the effect of these materials on the cement. Cement system design with different percentages of polypropylene fiber has been prepared and tested to see cement performance of these materials under these conditions. **Table 3.1** shows well specification of the typical Saudi well. The job is to cement a 7” inch liner casing.

Table 3.1: Well specifications

Well Parameters	**Values**
Depth of well (TVD)	14000 ft
Bottom hole pressure (BHP)	8265 psi
Bottom hole static temperature (BHST)	292°F
Bottom hole circulating temperature (BHCT)	228°F
Time to reach bottom (TRB)	49 min
Surface pump pressure	1050 psi
Mud weight (MW)	85 PCF

3.2 CEMENT SLURRY DESIGN

A high pressure and temperature deep well was selected for this study, and as a result, an exceptional cement system must be design and prepared for cementing the well. Various materials are used in preparing the cement system which is contributing to the improvement of the chemical and physical properties of the cement, so a successful cement job might be obtained. **Table 3.2** shows the cement slurry design without adding polypropylene fiber to the mix.

Table 3.2: Cement slurry design without polypropylene fiber

Class G cement powder + 35% silica flour + 1% expanding agent + 0.4% Dispersant + 0.2% Fluid loss control agent + 0.5% Fluid loss control agent + 1% Retarder + 0.25gm Defoamer	
Expected Slurry Density	125 PCF
Water Cement Ratio	0.44
Slurry Yield	1.367 cuft/sack
Thickening Time	4-5 hours

At the beginning, a series of cement tests were conducted to the cement system design as explained in the experimental program without the addition of polypropylene fiber. These cement test results were considered as the base case or as a reference in all the following cement results. Next, the polypropylene fiber was added to the base mix cement design with the percentages of 0.25, 0.5, and 0.75% by weight of the cement and the results were reported. **Table 3.3** illustrates the cement slurry design when the polypropylene fiber was added.

Table 3.3: Cement slurry design with polypropylene fiber

Class G cement powder + 35% silica flour + 1% expanding agent + X% polypropylene fiber + 0.4% Dispersant + 0.2% Fluid loss control agent + 0.5% Fluid loss control agent + 1% Retarder + 0.25gm Defoamer	
Expected Slurry Density	Unknown
Water Cement Ratio	0.44
Slurry Yield	Unknown
Thickening Time	Unknown
Where X represents the percentages of polypropylene fiber used in the cement.	

3.3 CEMENT COMPOSITION

Cements used in oil well cementing are divided into eight classes (A to H) depending on the depth and the chemical composition (varying degrees of sulfate resistance) (Nelson, 1990). The class G cement used in this study is manufactured using high sulfate resistance with a specific gravity of 3.14. **Table 3.4** shows the chemical properties of simple class G cement. All cement systems have been prepared using tap water. **Table 3.5** shows cement additives from Halliburton and the percentages used in the mix along with their fraction.

Table 3.4: Chemical composition of Class G cement

Chemical Component (%)	
Silica (SiO_2)	21.6
Alumina (Al_2O_3)	3.3
Iron Oxide (Fe_2O_3)	4.9
Calcium Oxide, Total (TCaO)	64.2
Magnesium Oxide (MgO)	1.1
Sulphur Trioxide (SO_3)	2.2
Loss on Ignition	0.6
Insoluble Residue	0.3
Equivalent Alkali (as Na_2O)	0.41

Phase Composition (%)	C_3A	<1
	C_3S	62
	C_2S	15
	C_4AF+2C_3A	16

Table 3.5: Commercialized additives with their functions and percentages

Additives	Functions	Concentration (%BWOC)
SSA-1	Strength stabilizing agent	35
MBHT	Extender	1
HR-12	Retarder	1.5
CFR-3	Friction reducer	0.4
Halad-344	Fluid loss controlling agent	0.2
Halad-413	Fluid loss controlling agent	0.5
DA-3000	Anti-foaming agent	25 gm

Figure 3.1: Additives used in experiments

3.4 CEMENT SLURRY PREPARATION

Cement mixing is an important issue since it affects the rheological properties as well as other primary properties of the cement slurry, for example, thickening time, compressive strength, porosity, and fluid loss (Orban et al, 1986). A variable speed high-shear blender is used to prepare the cement mix which contains blades as per the API specification as shown in **Figure 3.3**.

Mixing the cement is an important step which should be implemented according to the API procedure. In general, there are two methods for mixing the cement, dry mixing and wet mixing depending on well conditions and locations. In the dry mixing, the cement and all the additives are dry blended before mixing with water as shown in **Figure 3.2**, whereas in wet method, the additives are blended with water and then with cement. Tap water is used in all the experiments.

Figure 3.2: Blending of cement with silica products prior mixing

Figure 3.3: High speed blender

The wet mixing procedure has been implemented for all the experiments. Mixing steps are described as follows:

1. Cement, water, and additives are weighted according to the cement design.
2. Cement and silica flour are dry blended.
3. Water and the dry additives are blended at low speed of 4000 RPM.
4. Cement and the dry mixed powder are poured into water additive mixture within 15 seconds.
5. The whole mixture is then blended, at high speed 12000 RPM for 35 seconds.

6. After that, the cement slurry is conditioned in an atmospheric consistometer for 20 minutes at a temperature of 194°F as shown in **Figure 3.4**.

Figure 3.4: Atmospheric consistometer

3.5 THICKENING TIME

Thickening time cement test determines the time period in which cement remain pumpable under certain conditions (Dwight, 1990). In this test, the cement remains in liquid form for a long time during pumping, and then it turns into solid when pumping stops. During cementing, there are several factors affects slurry pumpability as well as the thickening time such as fluid loss, fluid contamination, and also a sudden stop in the cement pump, but cannot be included in the laboratory cement thickening time test. **Figure 3.5** shows high pressure high temperature consistometer. This equipment contains a vessel capable of holding pressure and temperature similar to well conditions. In this vessel, there is a rotational cylinder where the cement sample placed and also equipped with stationary paddle assembly (API Spec.10, 2012). The rotating cylinder runs at a speed of 150 RPM. Cement thickening time is determined by measuring the consistency of the cement, which known as Bearden units of consistency (Bc), is determined by measuring the torque imposed from the cement against the paddles.

The steps of the mixing are as follows:

1. Tap water is weighted and put in blender.
2. Cement and silica flour are dry blended.
3. Water and dry additives are blended at low speed of 4000 RPM.
4. Dry mixture is poured in water additive mixture within 15 seconds.
5. The whole mixture is then blended at high speed of 12000 RPM for 35 seconds.
6. Cement slurry is poured in to consistometer slurry cup then put into a HPHT equipment.
7. Increase the temperature and pressure of the cement slurry in the cement container in the agreement with suitable well simulation-test schedule which 228^0F, and 9315 psi.
8. Slurry consistency is monitored until consistency of 100 Bc is obtained.

9. After that, the consistometer is cooled before releasing the pressure from the vessel.
10. The potentiometer and the slurry container are removed and cleaned and made ready for the next test.

Figure 3.5: HPHT consistometer

3.6 CEMENT FLUID LOSS TEST

The cement fluid loss test measures the amount of the fluid lost when the cement is subjected to a differential pressure. Most of the cement loss is occurred during cementing the high permeability or sensitive formation. **Figure 3.6** shows high pressure high temperature fluid loss cement apparatus. The temperature used is 194^{o}F with differential pressure of 1000 psi.

The steps of the mixing are as follows:

1. Tap water is weighted and put in a blender.
2. Cement and silica flour are dry blended.
3. Water and dry additives are blended together at low speed of 4000 RPM.
4. Dry mixture is poured in water additive mixture within 15 seconds.
5. The whole mixture is then blended at high speed of 12000 RPM for 35 seconds.
6. After that, the cement slurry is cured in an atmospheric consistometer and at a temperature of 194^{o}F for about 20 minutes.
7. Cement slurry is poured into the testing cup and positioned in a HPHT fluid loss tester, then tested under a pressure of 1000 psi and a temperature of 194^{o}F for 30 minutes.
8. The volume of the fluid loss is collected, and measured using gradual cylinder.

Figure 3.6: Fluid loss tester

3.7 CEMENT DENSITY

Density is a key factor during drilling or through cementing, where using the appropriate density will result in a successful cement job without further problems. It also shows the hydrostatic head exerted by the cement in the well. If the improper cement density used, this might result in either destroying the well formation or blowing out of the well, especially during cementing deep wells where high density is needed.

Cement density is measured using a pressurized fluid density balance. The pressurized fluid density balance is preferred over normal density balance since it minimizes the entrapped air during the pressurizing of the cell as shown in **Figure 3.7**.

The steps of the mixing are as follows:

1. Tap water is weighted and put in blender.

2. Cement and silica flour are dry blended.
3. Water and dry additives are blended together at low speed of 4000 RPM.
4. Dry mixture is poured in water additive mixture within 15 seconds.
5. The whole mixture is then blended at high speed of 12000 RPM for 35 seconds.
6. After that, the cement slurry is cured in an atmospheric consistometer and at a temperature of 194ºF for about 20 minutes.
7. Cement slurry is poured into the density cup and closed.
8. Cement is pumped using syringe until no more cement enter the cell.

   Finally, the rider is moved back and front till the bubble in the glass is balanced, and the reading is recorded.

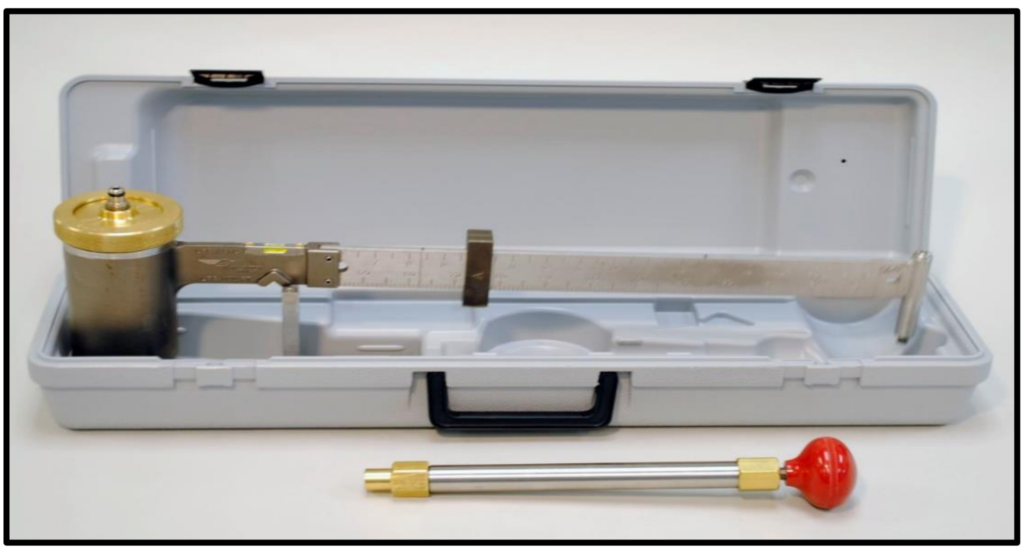

Figure 3.7: Pressurized density balance

3.8 FREE WATER SEPARATION TEST

Water is added to the cement at a fixed water cement ratio to set required cement density. Also water gives fluidity to the cement and works as a chemical agent through hydration process. When an excessive amount of water is added to the cement mix, water will be accumulated at the top, whereas cement settles at the bottom. Keeping this in mind will help in the case of stopping fluid separation in static condition during and after cement placement. **Figure 3.8** shows a gradual cylinder for free water cement test.

The steps of the mixing are as follows:

1. Tap water is weighted and put in blender.
2. Cement and silica flour are dry blended.
3. Water and dry additives are blended together at low speed of 4000 RPM.
4. Dry mixture is poured in water additive mixture within 15 seconds.
5. The whole mixture is then blended at high speed of 12000 RPM for 35 seconds.
6. After that, the cement slurry is cured in an atmospheric consistometer and at a temperature of 194^{o}F for about 20 minutes.
7. Next, cement slurry is poured into a gradual cylinder, and then is covered with an aluminum foil at the top.
8. Cement slurry is aged for two hours under room temperature.
9. Lastly, free water is collected from the top using a syringe and the amount of free water is reported.

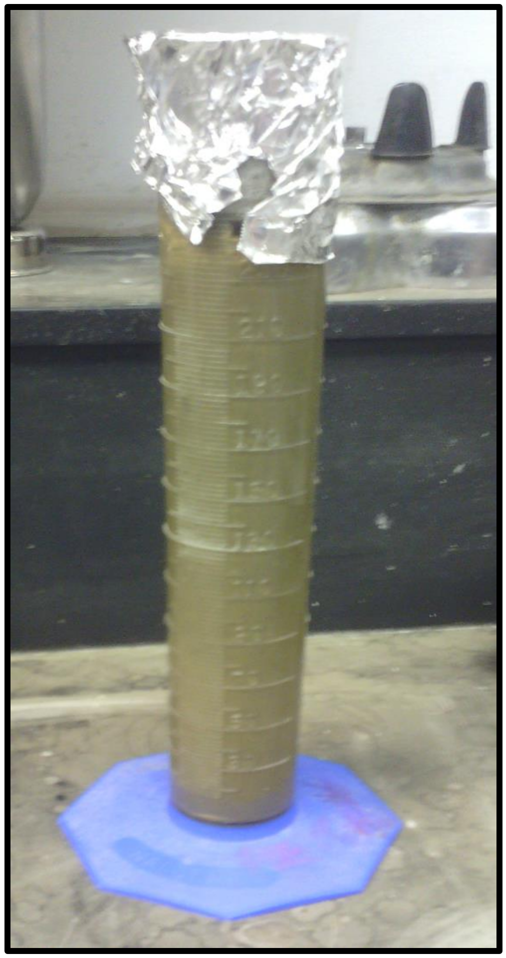

Figure 3.8: Graduated cylinder

3.9 RHEOLOGICAL PROPERTIES

Rheological properties are used to describe the quality of the final cement product and also used to predict the future performance at work environment as well as its physical properties during and after processing. In addition, it gives a quick guess of the frictional pressure losses as well as the required pump pressure needed during pumping.

Cement rheological properties like plastic viscosity, yield point, and gel strength are determined under high temperature conditions. Variable speed rheometer is commonly used to measure the rheology as shown in **Figure 3.9**.

Figure 3.9: Rotational viscometer

The steps of the mixing are as follows:

1. Tap water is weighted and put in blender.
2. Cement and silica flour are dry blended.
3. Water and dry additives are blended together at low speed of 4000 RPM.
4. Dry mixture is poured in water additive mixture within 15 seconds.
5. The whole mixture is then blended at high speed of 12000 RPM for 35 seconds.
6. After that, the cement slurry is cured in an atmospheric consistometer and at a temperature of 194^oF for about 20 minutes.
7. After that, cement slurry is put into rheometer cup in which it also conditioned at a temperature of 194^oF.

8. The cement slurry is stirred first ascending at a speed of 3, 6, 100, 200, 300, RPM and then descending order with 10 seconds for each speed and the readings are recorded.

For the gel strength, it is measured immediately after measuring the rheological properties.

- For 10-sec gel

The cement must be stirred for one minute at 300 RPM, and then stop for 10 seconds, and the max reading is recorded at a speed of 3 RPM.

- For 10-min gel

After 10-sec gel reading is recorded, the cement is left in a static condition for 10 minutes, and then max reading is recorded at a speed of 3 RPM.

3.10 COMPRESSIVE STRENGTH OF CEMENT

The purpose of compressive strength test is to calculate the ability of the cement to resist axial pushing forces (Adam, 1986). The compressive strength is determined by using two methods, direct method "crushing" or by using the ultra-sonic cement analyzer. (API Spec.-10A, 2012).

3.10.1 Compressive strength by crushing method

The crush strength cement test describes the cement integrity and long-term bearing ability after being pumped and allowed to set static in well. The cement slurry is prepared and subjected to pressure and temperature similar to bottom hole conditions or any point along the cement column.

The steps of the mixing are as follows:

1. Tap water is weighted and put in blender.
2. Cement and silica flour are dry blended.
3. Water and dry additives are blended together at low speed of 4000 RPM.
4. Dry mixture is poured in water additive mixture within 15 seconds.
5. The whole mixture is then blended at high speed of 12000 RPM for 35 seconds.
6. After that, the cement slurry is cured in an atmospheric consistometer and at a temperature of 194^{o}F for about 20 minutes.
7. Next, cement slurry is poured into the chamber of prepared moulds (see **Figure 3.10**) to produce the cubes (2 inch × 2 inch).
8. After that, moulds are placed in the HPHT curing vessel as shown in **Figure 3.11** with an initiating temperature of 27^{o}C. After that, test schedule is applied to reach the target temperature and pressure of around 292^{o}F and 3000 psi respectively. The cubes are cured in the machine for 24 hours.
9. At the end of the test, samples are removed from the curing vessel, and cubes are detached from moulds as displayed in **Figure 3.12** and **Figure 3.13**.
10. Finally, cement cubes are crushed in a compressive strength tester (see **Figure 3.14**) and values are reported.

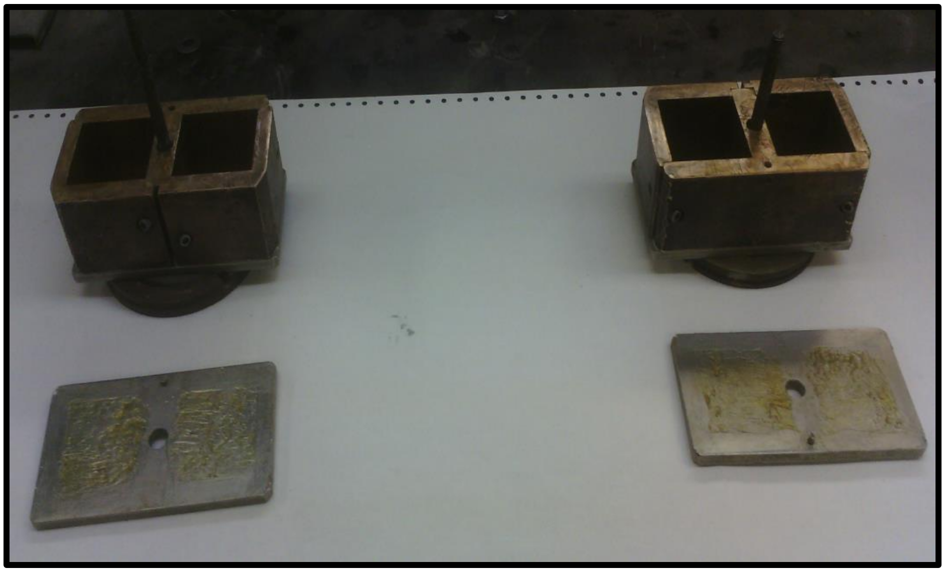

Figure 3.10: Cement moulds

Figure 3.11: HPHT curing chamber

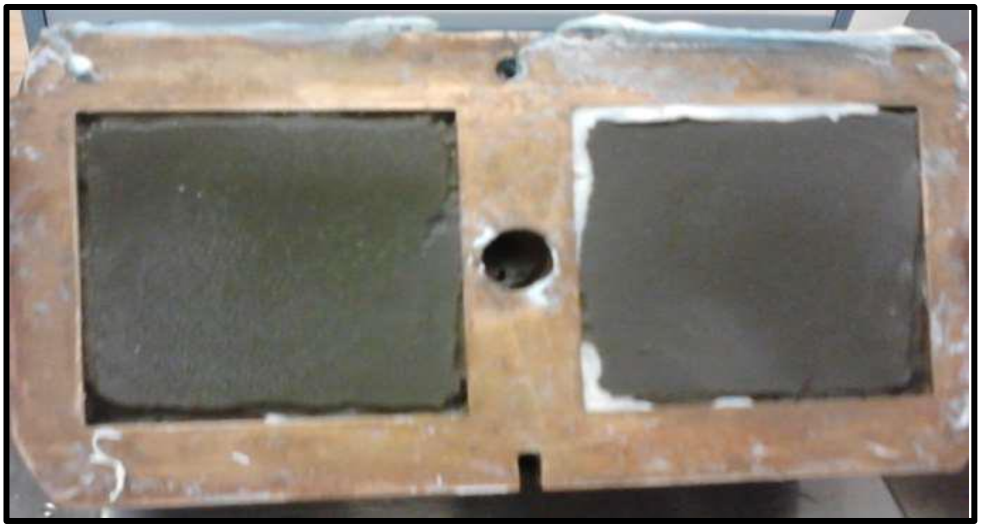

Figure 3.12: Cured cement moulds

Figure 3.13: Cured cement cubes

Figure 3.14: Crushing of cement cubes using compressive strength tester

Figure 3.15: The samples after crushing

3.10.2 Compressive strength by sonic method

Ultra-sonic cement analyzer (UCA) is a non-destructive strength test use the sound waves to measure the cement strength. In the device, an average compressive strength values are developed depending on the time the ultrasonic signal takes when passed through the cement composition as it starts to set and solidify. Compressive strength values obtained from sonic and by crushing can diverge significantly depending on the cement test condition and the used composition. **Figure 3.16** shows the ultra-sonic cement analyzer.

The steps of the mixing are as follows:

1. Tap water is weighted and put in blender.
2. Cement and silica flour are dry blended.
3. Water and dry additives are blended together at low speed of 4000 RPM.
4. Dry mixture is poured in water additive mixture within 15 seconds.
5. The whole mixture is then blended at high speed of 12000 RPM for 35 seconds.
6. After that, the cement slurry is cured in an atmospheric consistometer and at a temperature of 194^{o}F for about 20 minutes.
7. Next, cement slurry is poured into the cement chamber before putting the UCA device.
8. At the start, the test is fixed at bottom hole circulating temperature of 228^{o}F during the first 49 minutes, after that the temperature raised until reaching the static temperature of 292^{o}F, while the pressure is 3000 psi.
9. The test has been operated for 48 hours, then it stopped and the cooling process started.
10. Cement sample is removed and cell clean for the next test.

Figure 3.16: Ultra-sonic cement analyzer (UCA)

3.11 TENSILE STRENGTH OF CEMENT

The tensile strength properties determine the integrity of cement and its ability to withstand the perpendicular loading stresses. The Brazilian tensile strength test method was used during the experiment.

The steps of the mixing are as follows:

1. Tap water is weighted and put in blender.
2. Cement and silica flour are dry blended.
3. Water and dry additives are blended together at low speed of 4000 RPM.
4. Dry mixture is poured in water additive mixture within 15 seconds.
5. The whole mixture is then blended at high speed of 12000 RPM for 35 seconds.

6. After that, the cement slurry is cured in an atmospheric consistometer and at a temperature of 194ºF for about 20 minutes.
7. Next, cement slurry is poured into the chamber of prepared moulds to produce the cubes (2 inch × 2 inch).
8. After that, moulds are placed in the HPHT curing vessel with an initiating temperature of 27^{o}C. After that, test schedule is applied to reach the target temperature and pressure of around 292^{o}F and 3000 psi respectively. The cubes are curried in the machine for 24 hours.
9. At the end of the test, samples are removed from the curing vessel, and cubes are detached from moulds.
10. Cement plugs 1 inch × 1 inch are drilled out of the cubes as in **Figure 3.20**.
11. The cement plugs are cut into 0.5 inch three disc-shape specimens as in **Figure 3.17**.
12. Every specimen is loaded by diametrically across the circular cross for testing the tensile failure by the crushing machine as seen in **Figure 3.18** and **Figure 3.19**.

Figure 3.17: The disc-shape specimens cut from the cement plugs

Figure 3.18: Crushing the specimens by the crushing machine

Figure 3.19: The crushed specimens

3.12 POROSITY AND PERMEABILITY OF CEMENT

Permeability is described as the ability of a fluid to flow at different pressures, and helps in determining the long term performance of cement sheath. In general, cement sheath is used to seal the formation zones so that no fluid can migrate between layers. Thus, low permeability of the cement sheath is needed to obtain a good cementing especially under HPHT.

Porosity is defined as a void space in the cement sheath where fluids are stored in, and later can affect the long term durability of the cement sheath.

Steps in determining the permeability and porosity of the cement samples:

1. Cement plugs 1 inch × 1 inch are drilled out of the cubes as in **Figure 3.20**.
2. Cement plugs are dried in an oven for one day.
3. Porosity and gas permeability tests are conducted using automated porosimter/ permeameter (see **Figure 3.21**), with confining pressure of 500 psi is used.

The automated porosimter /permeameter (AP-608) is a device that measures the porosity and gas permeability under true reservoir condition. This apparatus AP-608 installed with manually loaded hassle type core holder and the permeability ranges from 0.001 up to 500 md.

Figure 3.20: Cement plugs

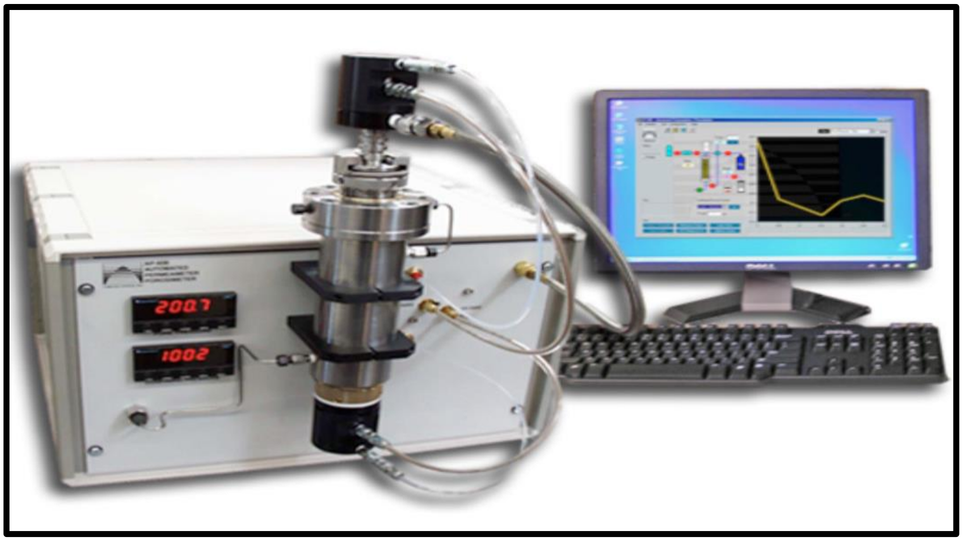

Figure 3.21: Automated porosimeter/permeameter

3.13 MICROSTRUCTURAL ANALYSIS

The microstructure of the cement can be analyzed by subjecting the cement to structural tests such as SEM and XRD. In general, the SEM cement test is used to define the composition, topography, and the pore structure of the final cement product, whereas XRD is commonly used to study the cement composition as well as cement hydration.

When cement is mixed with water, the hydration process takes place and a lot of compounds are generated in the cement paste, for example, alite (C_3S), belite (C_2S), ettringite (Aft), calcium hydroxide (CH, portlandite), and calcium silica hydrate C-S-H which can be measured and showed clearly in the XRD spectra.

Cement must be crushed into powder and then XRD test can be applied, but for the SEM cement test, a small piece of cement rock is needed to run the proposed test.

Chapter 4

RESULTS AND DISCUSSION

This chapter shows the results of adding the polypropylene fiber to Portland Saudi cement class G under both high pressure and temperature conditions. This chapter presented well parameters and experimental program that were followed during the experiments. Each experimental test results are discussed thoroughly for selected well cement system.

4.1 EFFECT OF POLYPROPYLENE FIBER ON THICKENING TIME

Thickening time cement test gives an indication about the time period the cement remains pumpable under certain conditions. In fact, thickening time cement test is an important property which needed to clarify before starting cementing operation. This test is conducted to know the time up to which the cement remains in the liquid form and will indicate time to stop the pumping, as cement turns into solid form. Hence, the knowledge of this thickening time property helps us in determining whether it's suitable to use this cement in different work circumstances. High pressure and temperature condition has been applied to the cement slurry as provided in the API specification 10B in 2012. The maximum pressure and temperature used in the schedule were 9300 psia, and 228^{o}F. The maximum temperature was reached in 42 minutes. Four cement systems containing the polypropylene fiber with varied percentages of 0, 0.25, 0.5, and 0.75% BWOC have been subjected to the cement thickening time test, and the times where the cement slurries needed to reach a value of 100 BC were reported.

Figure 4.1, Figure 4.2, Figure 4.3 and **Figure 4.4** illustrate the thickening time for cement systems containing polypropylene fiber with varied percentages 0, 0.25, 0.5, and 0.75%.

The four cement systems having polypropylene fiber percentages of (0%, 0.25%, 0.5% and 0.75%) BWOC were subjected to thickening time test and time of cement slurries to reach a consistency of 100 Bc were recorded. As known that at the start of tests the consistencies become very high and when the test conditions are implemented, the viscosities are reduced and stabilized for some period of time until 100Bc consistency achieved where they are considered unpumpable.

At the start of the test, the cement slurries have consistencies of (40, 55, 56, and 65) Bc respectively. When the conditions of the test applied, this value reduced and remain stable for a certain time until the 100 Bc value reached, which is an indicator that the cement is now unpumpable. It is clear that the addition of polypropylene fiber to the cement resulted in decreasing in the thickening period time from five hours down to one and half hour as shown in **Figure 4.5**, and **Figure 4.6**.

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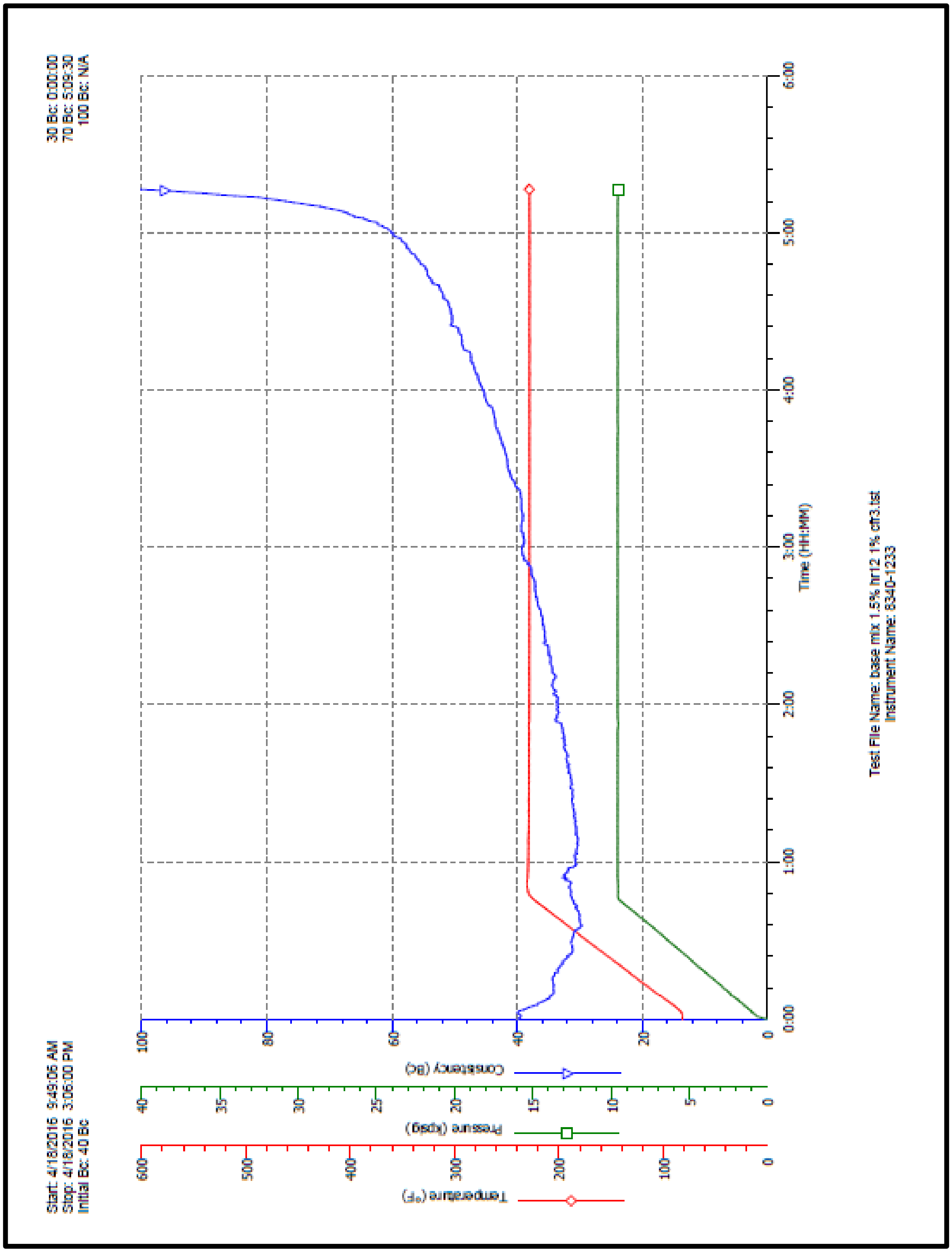

Figure 4.1: The thickening time for the base mix (0% polypropylene fiber) cement slurry

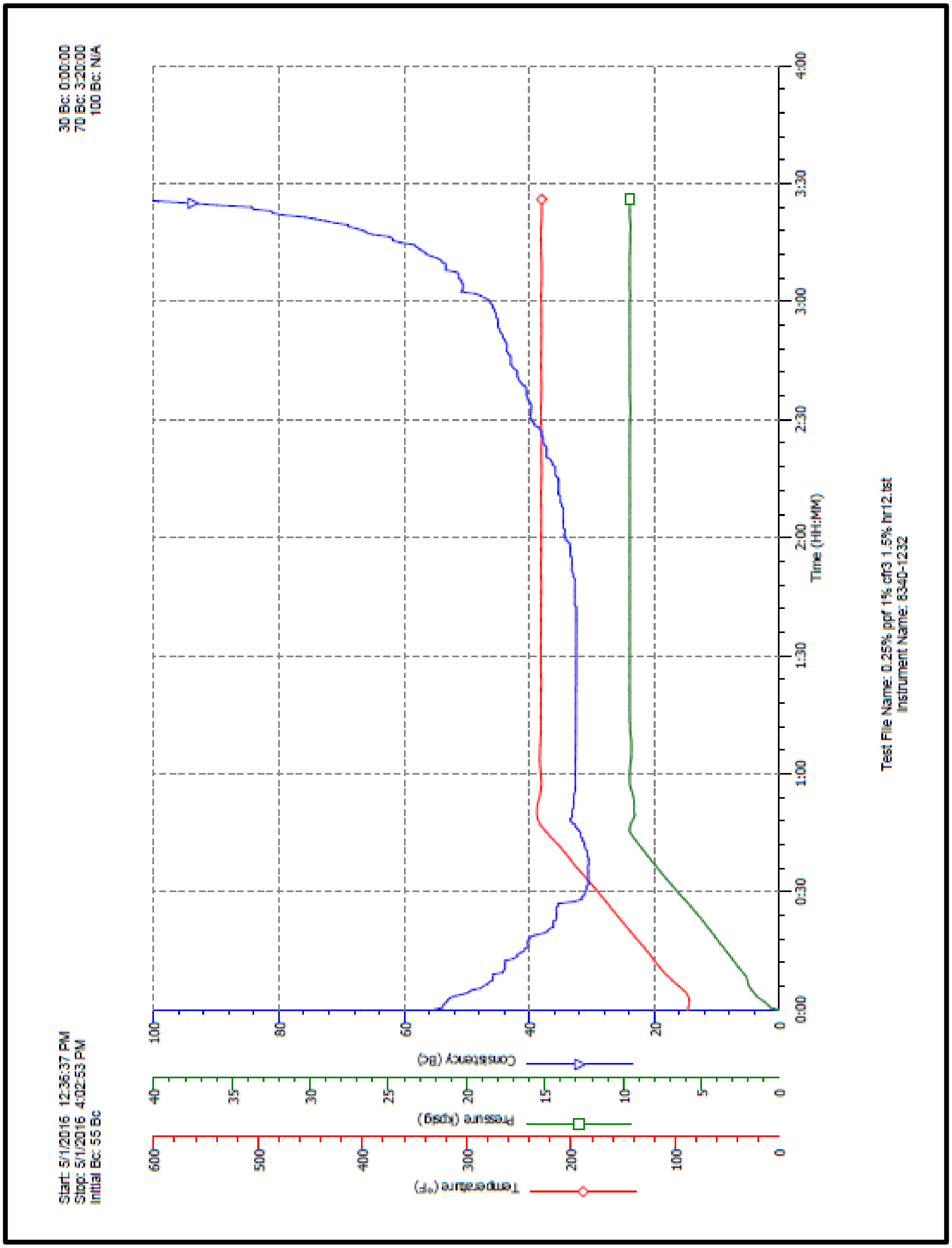

Figure 4.2: The thickening time for 0.25% polypropylene fiber cement slurry

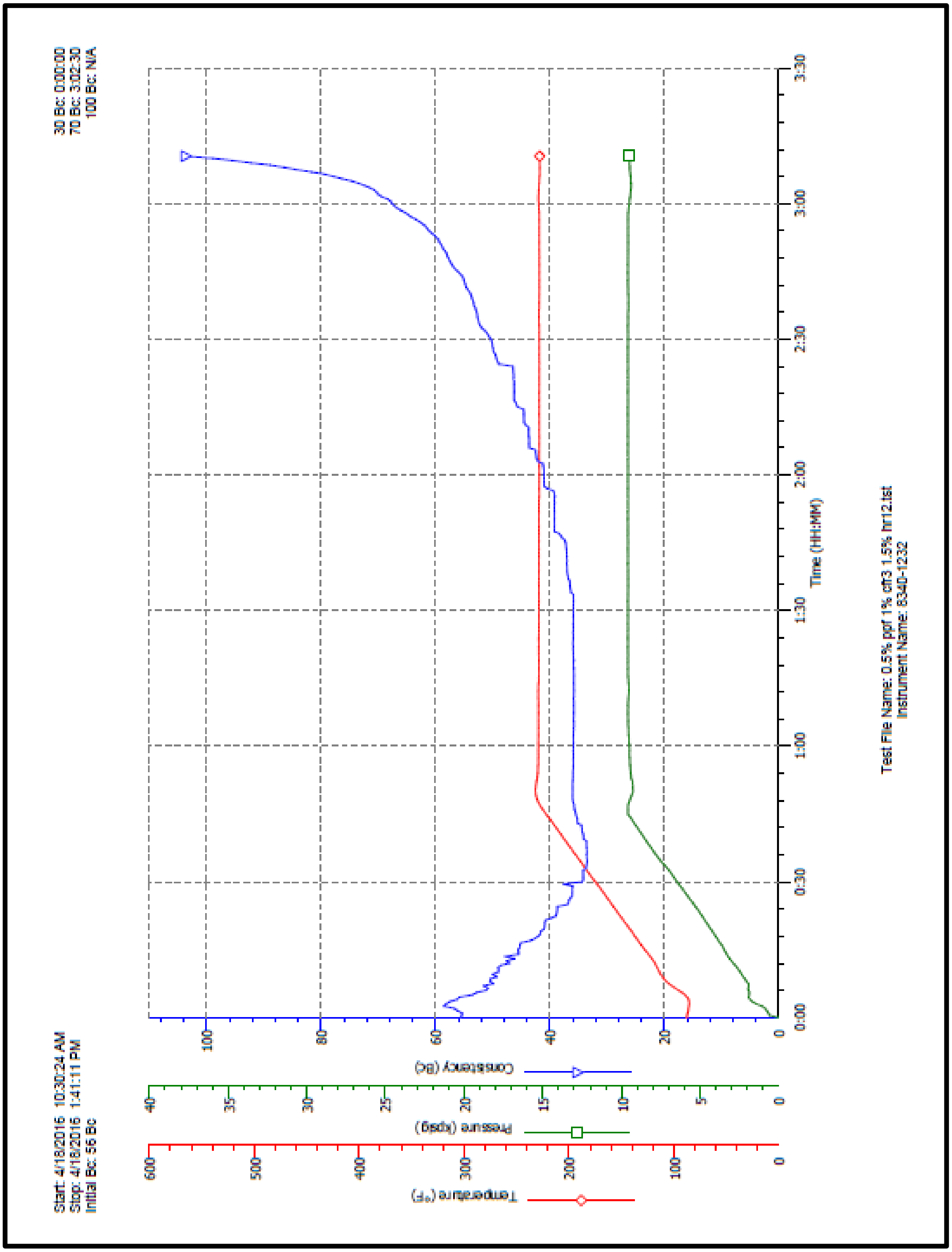

Figure 4.3: The thickening time for the 0.5% polypropylene fiber cement slurry

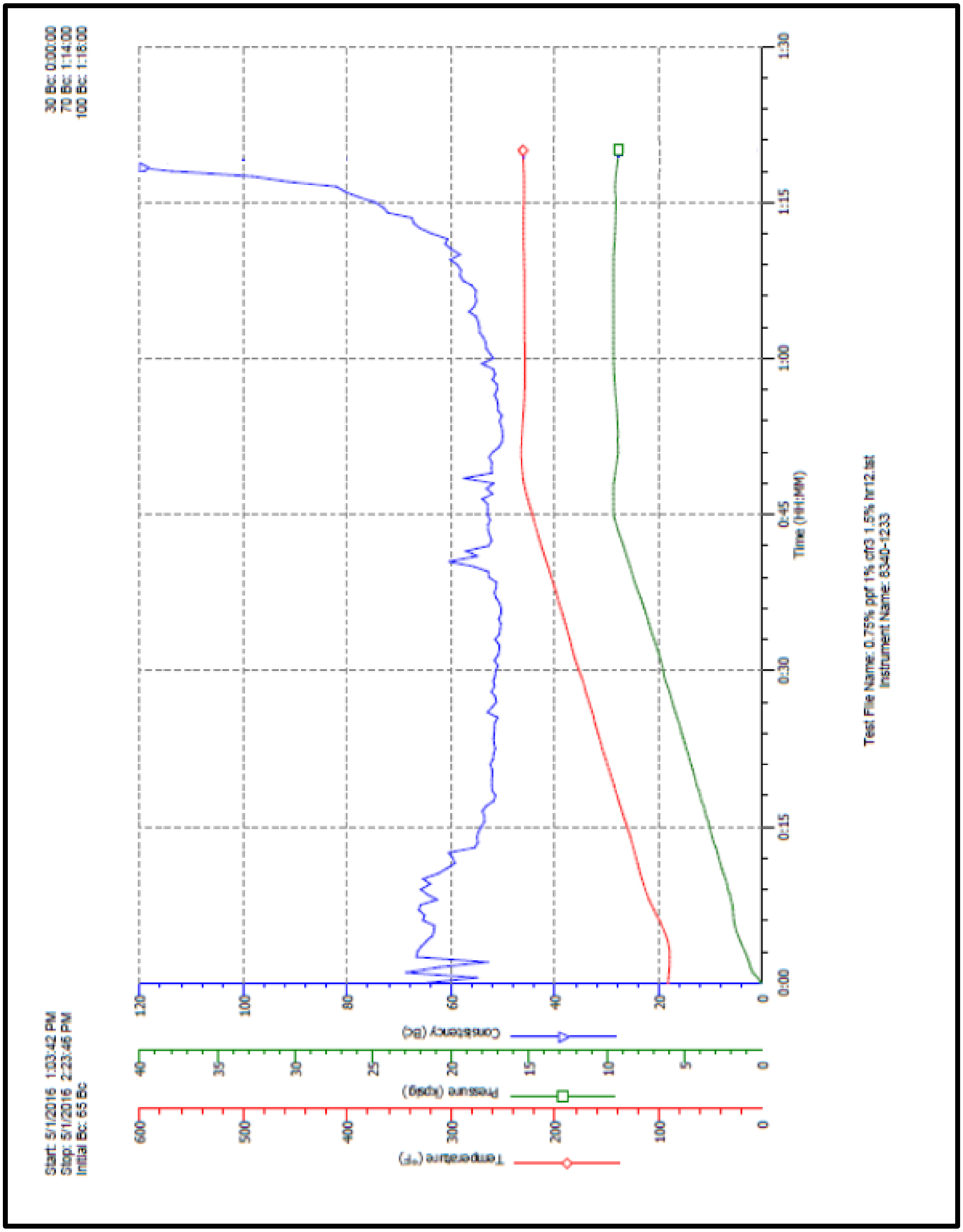

Figure 4.4: The thickening time for the 0.75% polypropylene fiber cement slurry

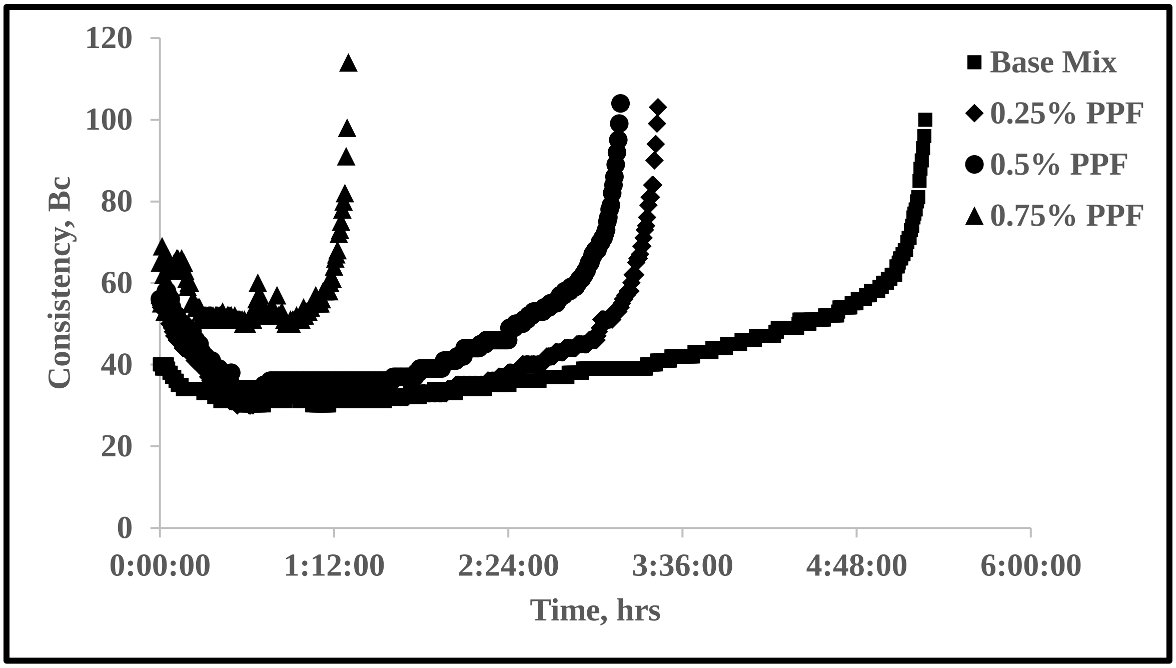

Figure 4.5 The thickening time for the cement slurry compositions for different fiber concentrations (0%, 0.25%, 0.5% and 0.75%)

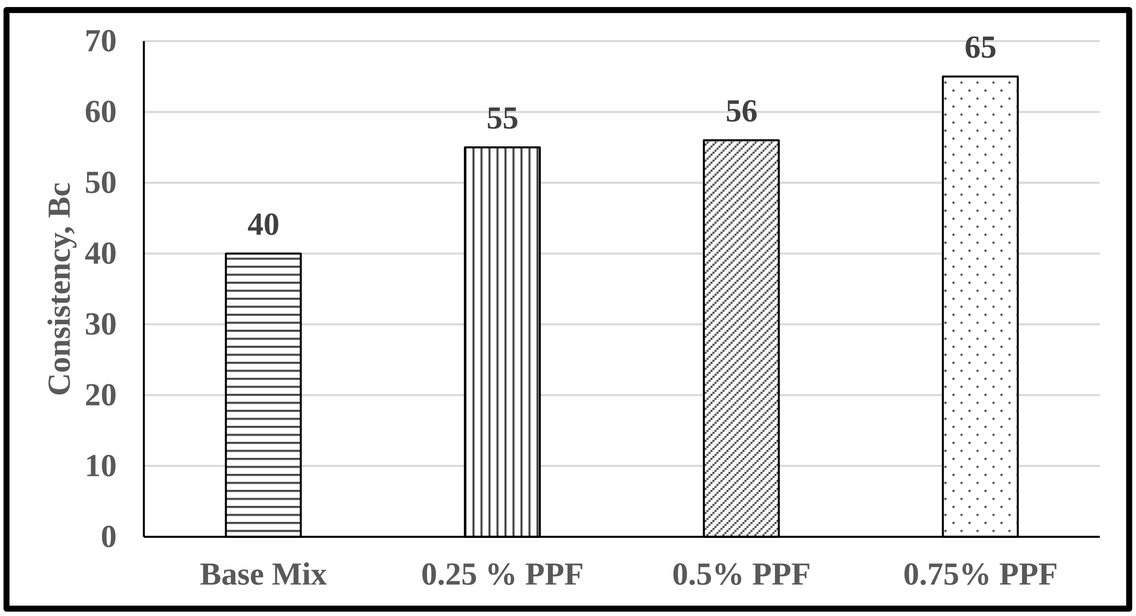

Figure 4.6: The consistencies at the start of test

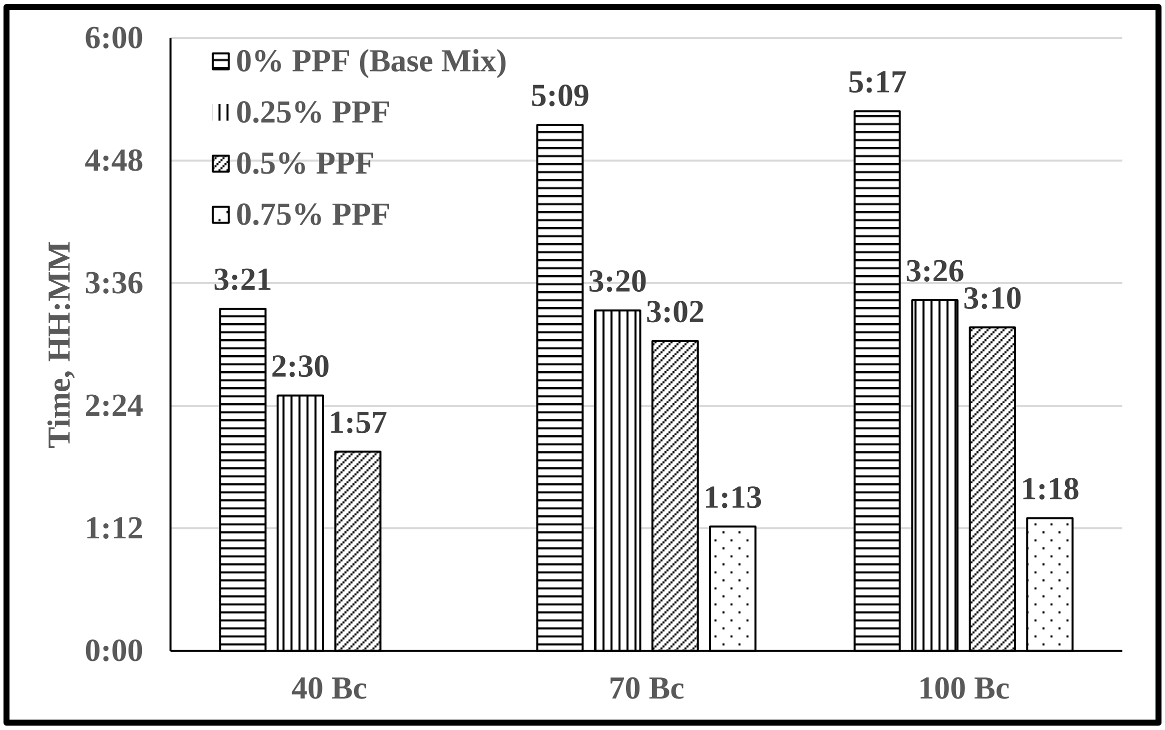

Figure 4.7: The time to reach 40Bc, 70Bc and 100Bc consistencies

From **Figure 4.5**, it was observed that the addition of the polypropylene fiber to the cement resulted in decreasing in the thickening period time as explained above. The reason for this acceleration is that the addition of the polypropylene fiber causes a quickening in the hydration process of the cement, and in turn, it shrinks the time the cement takes to thick.

When 0.25% of polypropylene fiber added to the base mix, the cement thickening time decreased from 5:17 minutes down to 3:26 minutes. Further addition of higher percentages polypropylene fiber shortens down the thickening time to the period of 1:18 minutes in the case of 0.75%. Accelerating the cement is one of the biggest concerns when it comes to drilling shallow wells, therefore, adding 0.5% of polypropylene fiber maybe the best practice, since it gave sufficient time while the minimum thickening time needed for the proposed well was three hours according to the given data that come from the proposed well.

When the tests started, higher consistencies of (40, 55, 56, and 65) Bc respectively, were observed (see **Figure 4.6**), then followed by a reduction in the consistency due to test conditions and remain stabilized. It was also observed that it takes long time to reach 40 Bc for all cement slurries. **Figure 4.7** displays the time to reach 40, 70, 100 Bc consistencies. All the cement slurries take long time to reach a consistency of 40 Bc, and only leaving a short period of time to reach 100 Bc consistency. This short time is known as a right angle set of the cement and it takes the minimum time to reach 100 Bc (API spec. 10, 2012). In other words, it can be said that reaching 70 Bc is an indicator that the cement is becoming unpumpable since the cement has only a short time to reach 100 Bc.

4.2 EFFECT OF POLYPROPYLENE FIBER ON CEMENT SLURRY FLUID LOSS

The purpose of the cement fluid loss test is to measure the amount of the fluid lost when the cement is subjected to a differential pressure within the well. Most of the cement losses are occurring during cementing the high permeability or sensitive formation. A lot of cement fluid loss additives are mixed with the cement to minimize the amount of fluid lost from the cement as much as possible. The polymer fiber is considered one of the fluid loss additives that added to the cement to reduce this problem.

The typical well which selected for this study had a depth of 14000 ft. In this case, the required cement system needed must have an acceptable fluid loss so a successful cement job is obtained.

When polypropylene fiber added to the cement mix, it resulted in a reduction in the amount of the fluid loss as shown in **Table 4.1**. **Figure 4.8** shows the trend of the fluid loss. All the results obtained from these tests were almost the same of around 55 ml, where slight reduction of around 5 ml was observed when 0.25% polypropylene fiber was added compared with that obtained from the base mix. On the other hand, all the results were in the acceptable range of the fluid loss since the industry permitted fluid loss is around 100ml/30 min.

Table 4.1: Fluid loss of cement with 0, 0.25%, 0.5% and 0.75% of the polypropylene fiber

Fluid loss (API), ml			
0% (Base mix)	**0.25% PP Fiber**	**0.5% PP Fiber**	**0.75% PP Fiber**
65	60	55	50

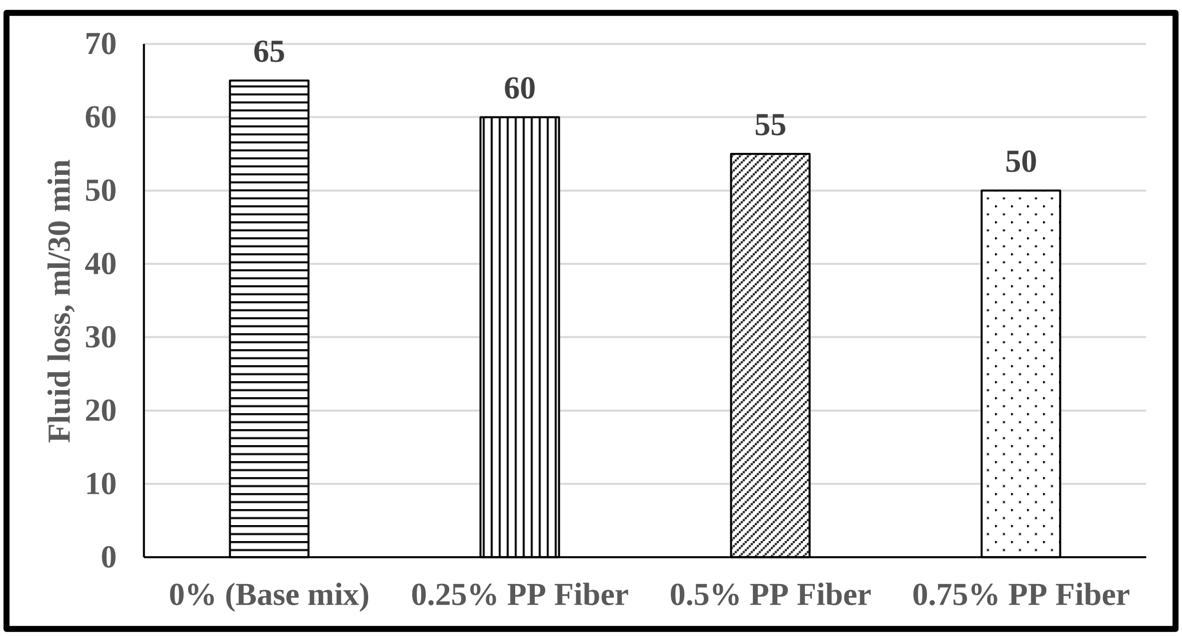

Figure 4.8: The fluid loss trend for polypropylene fiber admixture

4.3 EFFECT OF POLYPROPYLENE FIBER ON FREE WATER SEPERATION

Normally, cement slurry is produced by mixing cement, water, and additives. Water which is used in the cement gives the cement its fluidity, and mainly work as main agent in the chemical hydration activity. Water is added to the cement at a fixed water cement ratio to give the cement its appropriate density. If excessive amounts of water added to the cement, water will accumulate at the top, and the cement settles at the bottom.

In this test, four cement systems containing polypropylene fiber with percentages 0, 0.25, 0.5, and 0.75% BWOC have been tested for the free water separation. The cement samples were aged for two hours under room temperature and atmospheric pressure and the amount of free water accumulated at the top was measured.

It is well known that using Class G cement produce a considerable amount of free water, especially in both high pressure and temperature wells, where the cement settles at the bottom and cause improper cementing. Also the presence of free water during the cementing of horizontal wells leaves a gap between the cement and the casing allowing the fluids to move through it. Thus the need for adding cement additives to the cement mix design is necessary for HPHT. Also adding 35% silica flour resulted in no free water, and the cement stays in suspension, since silica flour showed the ability to absorb more water. **Table 4.2** shows the results of a cement free water test of polypropylene fiber with 0, 0.25, 0.5, and 0.75% BWOC. It is clear that the addition of polypropylene fiber to the cement slurry resulted in no free water separation at the top of the cement.

The reason of no free water being observed when polypropylene fiber added to the cement because the polypropylene fiber blocks the capillaries and prevent water separation. Also, the polypropylene fiber did not cause any distribution in the particle suspension property in the cement.

Table 4.2: The free water separation results at atmospheric pressure and room temperature

Separated free water, ml/250ml			
0% (Base mix)	**0.25% PP Fiber**	**0.5% PP Fiber**	**0.75% PP Fiber**
0	0	0	0

4.4 EFFECT OF POLYPROPYLENE FIBER ON DENSITY

Well control is one of the vital issues that engineers should carefully consider about during drilling and cementing. Density takes a main and heavy part in the case of drilling using the suitable drilling fluid density or through cementing. Neglecting this part might result in either destroying the well formation, or leading to well blowout, especially when cementing deep wells where high density is required.

Controlling cement density can be obtained by any of two methods, either adjusting the water cement ratio or by the addition of weighting agents (Oilfield Glossary, 2009). As mentioned above, well conditions play an important role in selecting the cement density. In deep wells, where high cement density is required, class G cement cannot perform well to control the well pressure, as a result, different types of cement additives are added to the cement to give it special properties, and help in handling specific conditions. Furthermore, heavy cement density is needed to flush the heavy cement fluids and decrease their diffusion in the well. A pressurized cement balance is normally used to measure the cement density in the field as well as in the laboratory. Here the designed cement density after putting cement additives is to be around 16.6 lb/gal. Cement systems with different percentages of polypropylene fiber 0, 0.25, 0.5, and 0.75% BWOC were prepared, and the density of the produced cement was measured using a pressurized cement balance. **Table 4.3** show the density of cement with different percentages of polypropylene fiber 0, 0.25, 0.5, and 0.75% mixed with the cement.

Table 4.3: The density of cement slurries having (0% 0.25%, 0.5%, 0.75%) polypropylene fiber

Density, lb/gal			
0% (Base mix)	**0.25% PP Fiber**	**0.5% PP Fiber**	**0.75% PP Fiber**
16.63	16.61	16.58	16.56

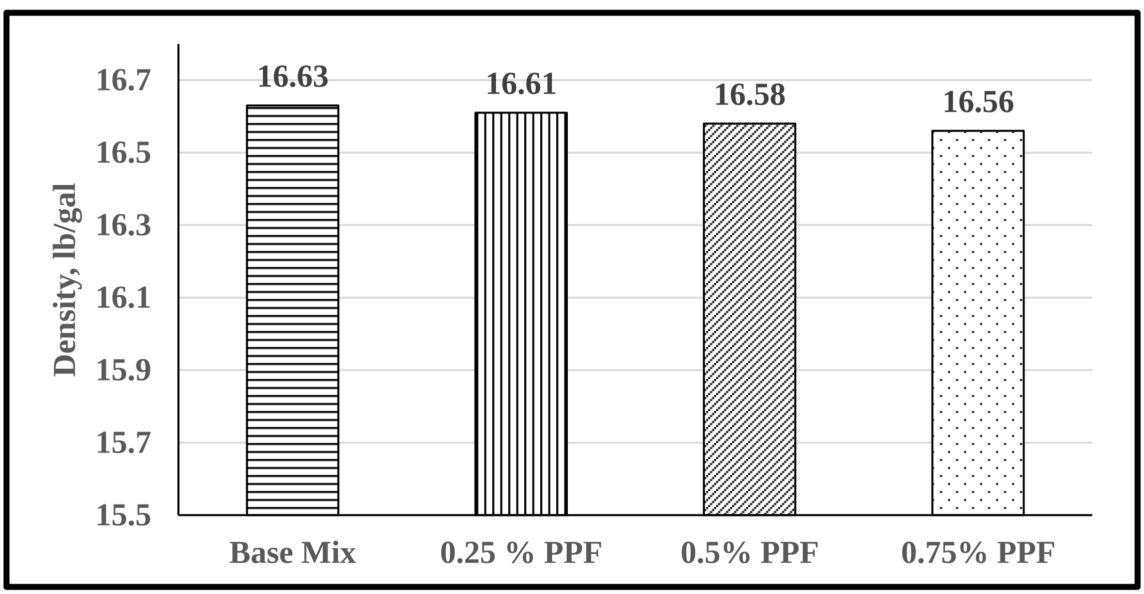

Figure 4.9: The cement slurry density trend for polypropylene fiber admixture

The cement system without polypropylene fiber gives the density of 16.63 lb/gal value. When polypropylene fiber is incorporated in base cement design, it decreases the density of cement slurry. But the addition of polypropylene fiber does not reduce the density value appreciably as the density of cement slurry having 0.75% polypropylene fiber is just reduced by 0.4% from the base cement. In short, incorporating polypropylene fiber to the cement does not affect the cement density and the results were almost the same as shown in **Figure 4.9**. This change in density explains that polypropylene fiber being lightest material helps in reducing the density of cement and can be used in designing the light weight cement slurries.

4.5 EFFECT OF POLYPROPYLENE FIBER ON COMPRESSIVE STRENGTH

The compressive strength properties determine the integrity of cement and its ability to bear long term imposed stresses. Cement slurry is supposed to develop the compressive strength early and make strong bond with walls of wellbore. The compressive strength tests are conducted to evaluate the development of cement strength with time utilizing the ultrasonic cement analyzer (UCA) as well as to determine cement bonding stability after set utilizing the conventional compressive strength test (destructive approach).

4.5.1 Non-Destructive Compressive Strength by Ultrasonic Cement Analyzer

Four cement system containing polypropylene fiber with different percentages of 0, 0.25, 0.5, and 0.75% have been tested for compressive strength using an ultra-sonic cement analyzer under conditions of both high pressure (3000 psi), and high temperature (292^{o}F) cured for 48 hours.

In this test, the cement slurry is placed in the UCA chamber, and the conditions of high pressure and temperature are applied to the cement. After that, the compressive strength of the tested cement starts to develop with time, and is simultaneously measured using acoustic waves. It is well known that as the compressive strength increased, transit time required for these waves to travel through the cement is reduced, and its acoustic impedance began to increase.

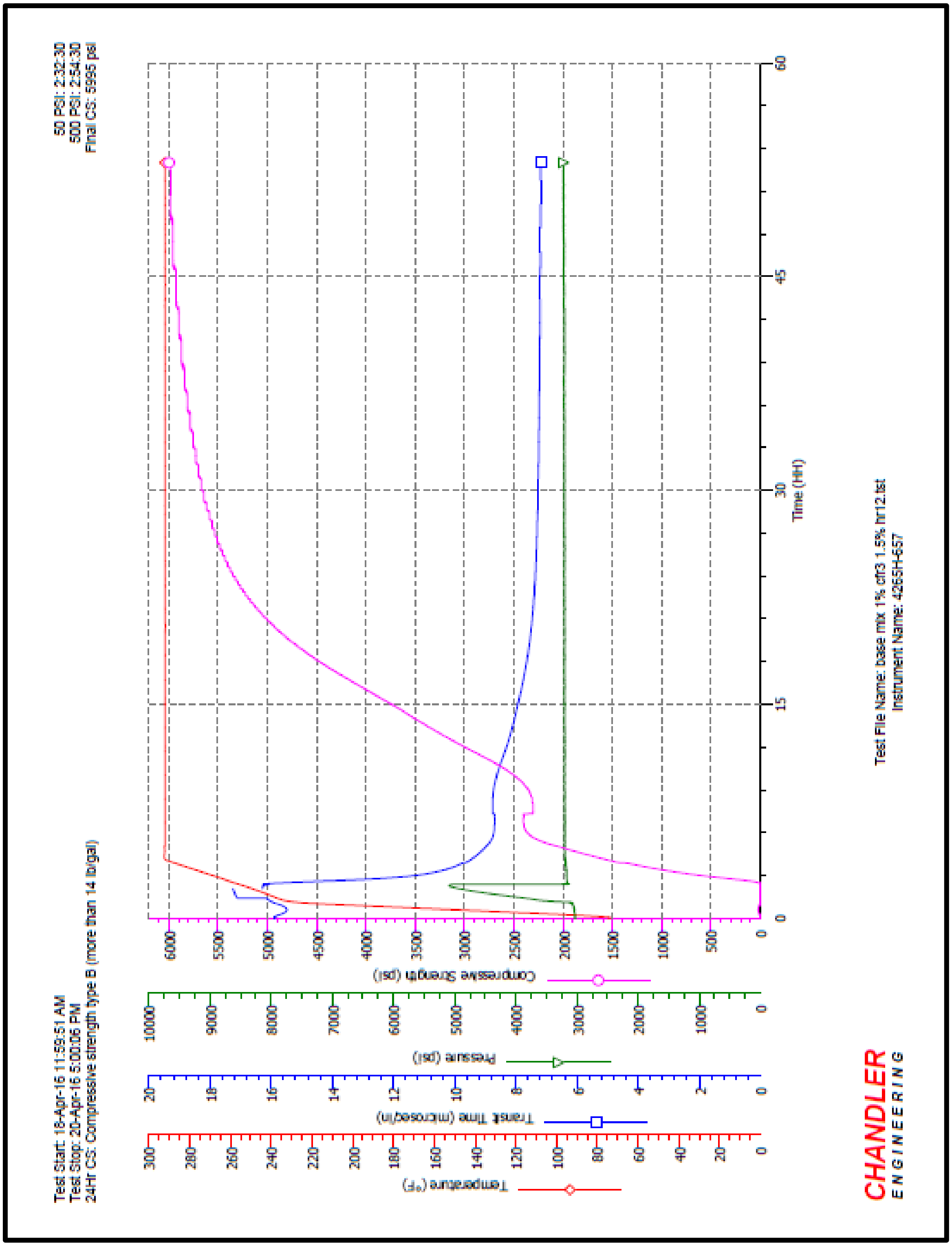

Figure 4.10: The compressive strength development for the base mix (0% polypropylene fiber) cement slurry at HPHT

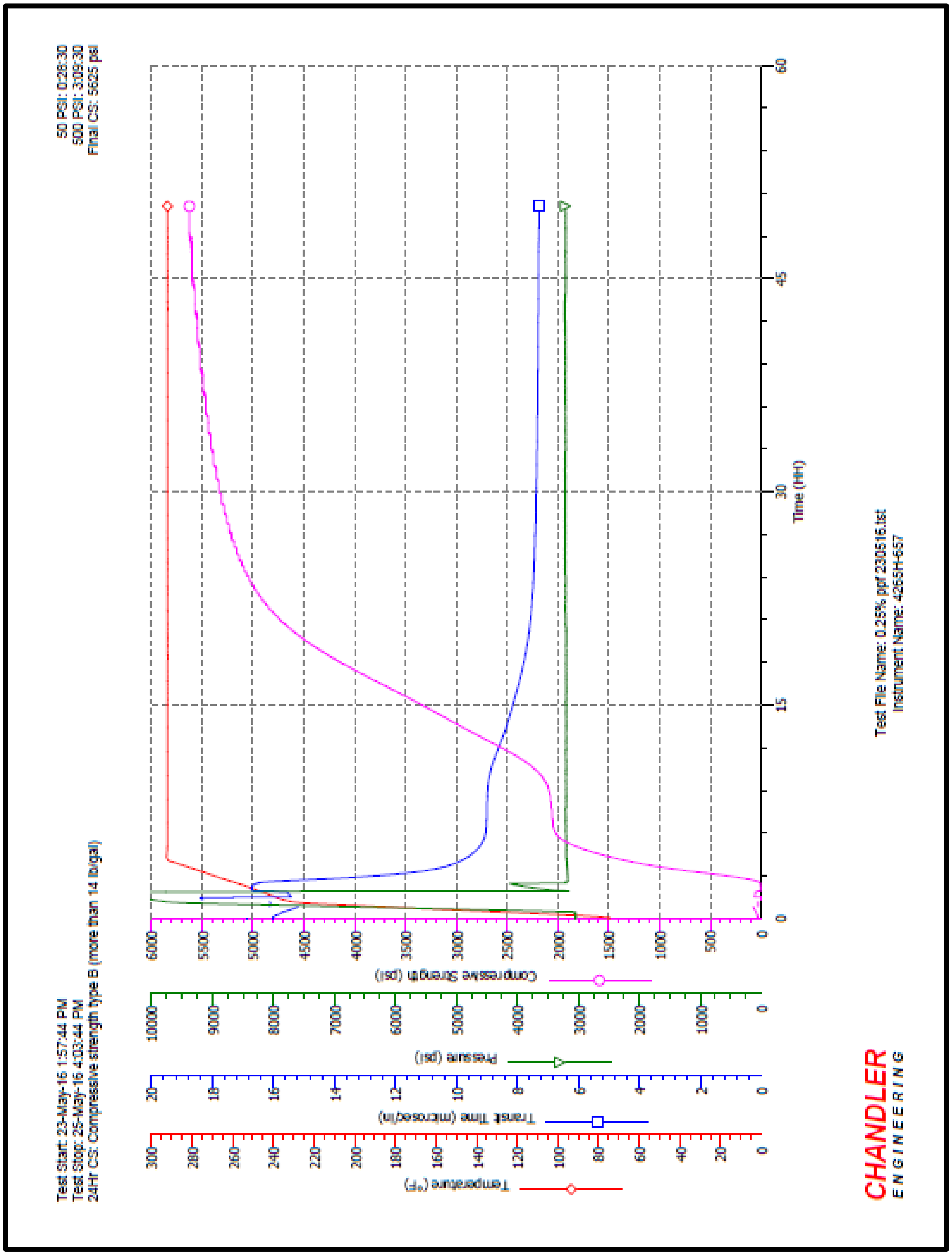

Figure 4.11: The compressive strength development for 0.25% polypropylene fiber cement slurry at HPHT

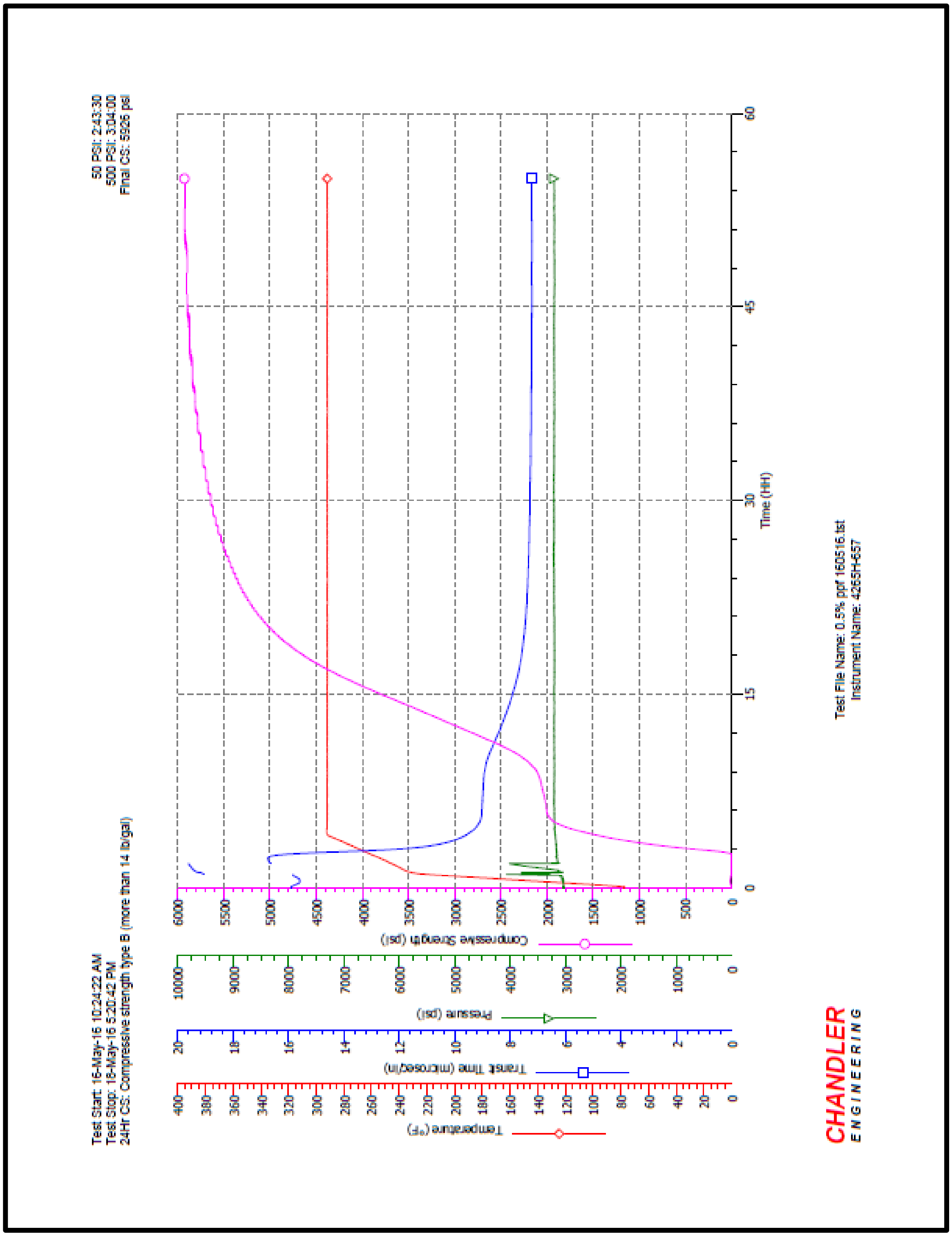

Figure 4.12: The compressive strength development for 0.5% polypropylene fiber cement slurry at HPHT

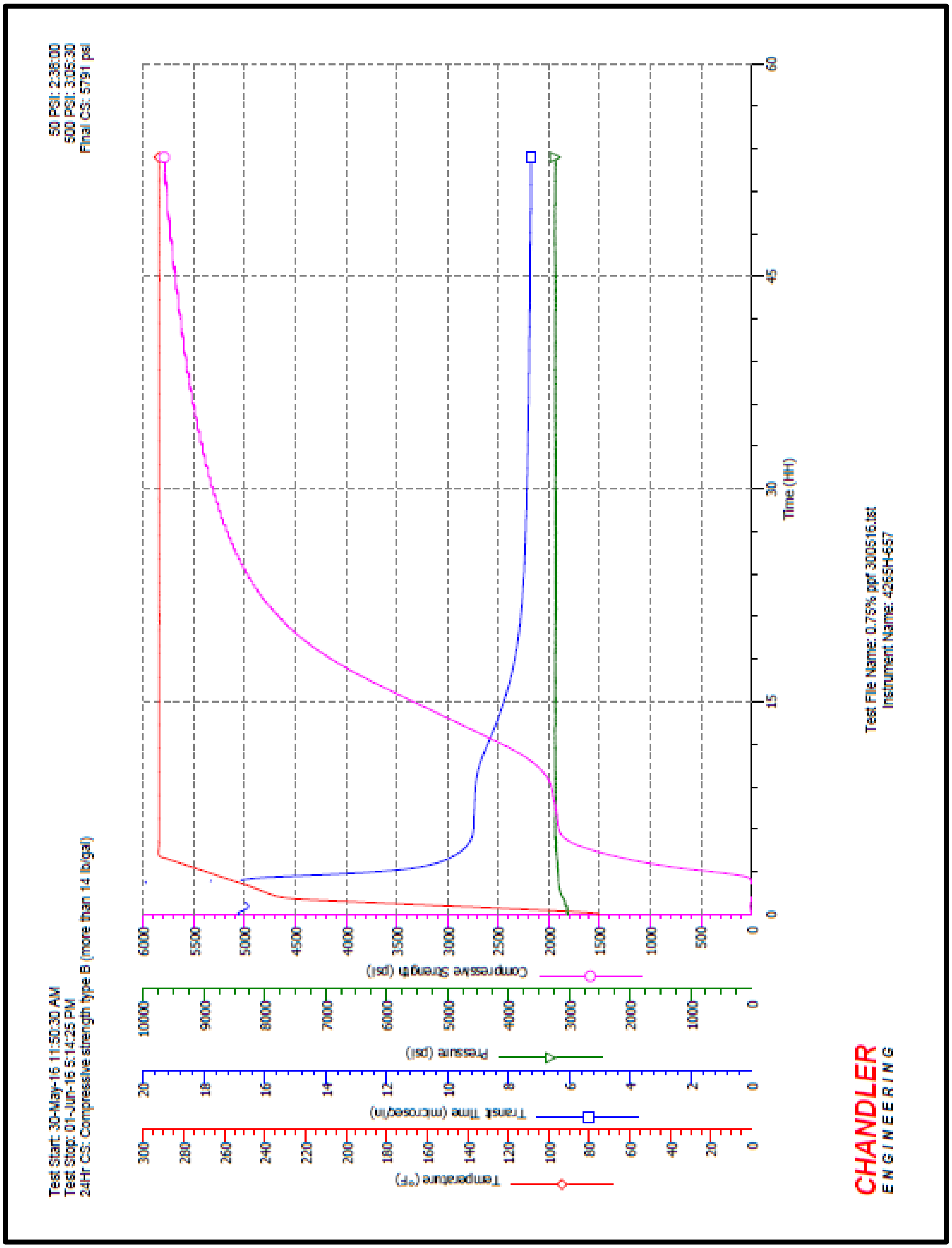

Figure 4.13: The compressive strength development for 0.75% polypropylene fiber cement slurry at HPHT

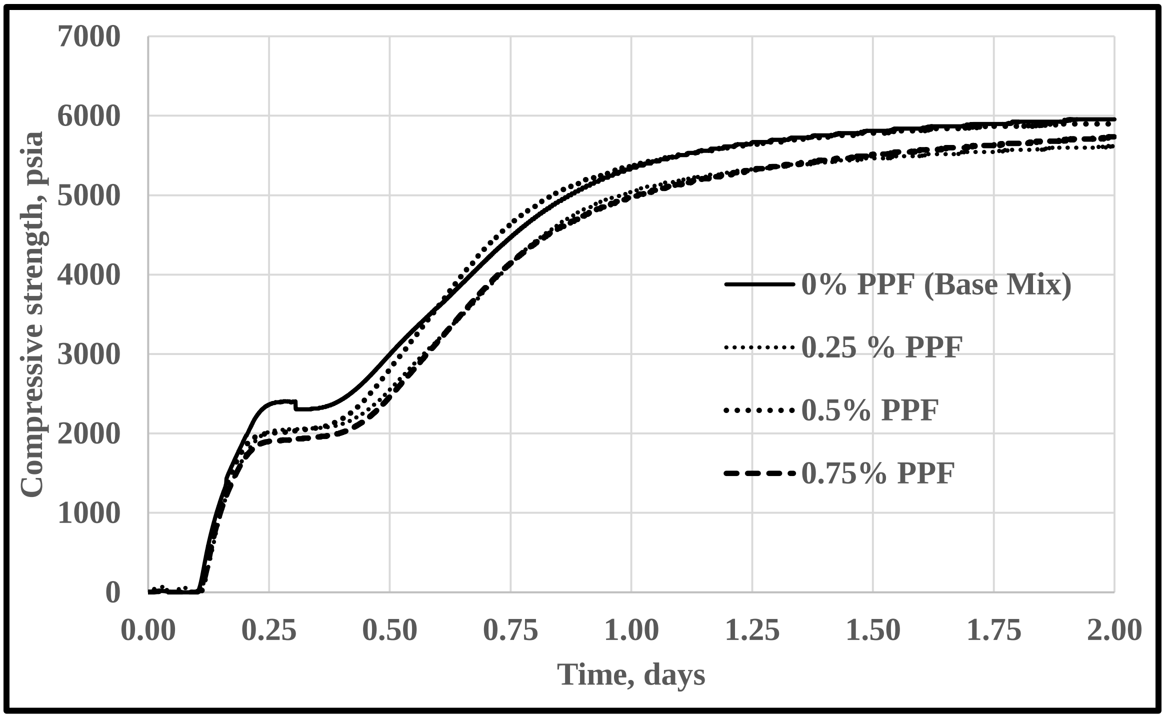

Figure 4.14: The compressive strength development for various compositions of cement slurry at HPHT

Different polypropylene fiber percentages are mixed in base slurry to study the super mechanical potential of polypropylene fiber as **Table 4.4** explains the compressive strength tests results. Strength retrogression is one of the most challenging problems that are faced, especially during dealing with high temperature conditions. As known that the addition of silica flour with 35% BWOC will terminate this problem. Polypropylene fiber has an accelerating effect, since it showed a rapid as well as early strength development compared with the cement base mix as illustrated clearly in **Figure 4.14**. It is observed that the addition of 0.5% polypropylene fiber resulted in the highest compressive strength of around 6000 psi after 48 hours aging. **Figure 4.15** proves that increasing the amount of polypropylene fiber added resulted in a reduction in the final values of the produced compressive as well as the cement density.

Table 4.4: The sonic method compressive strength results at 292^{0}F and 3000 psia for all the cement compositions

Time period	Compressive strength, psia			
	0% (Base mix)	**0.25% PP Fiber**	**0.5% PP Fiber**	**0.75% PP Fiber**
12 hours	2995	2547	2806	2462
18 hours	4462	4140	4634	4142
24 hours	5373	5038	5373	4966
48 hours	5955	5611	5897	5735

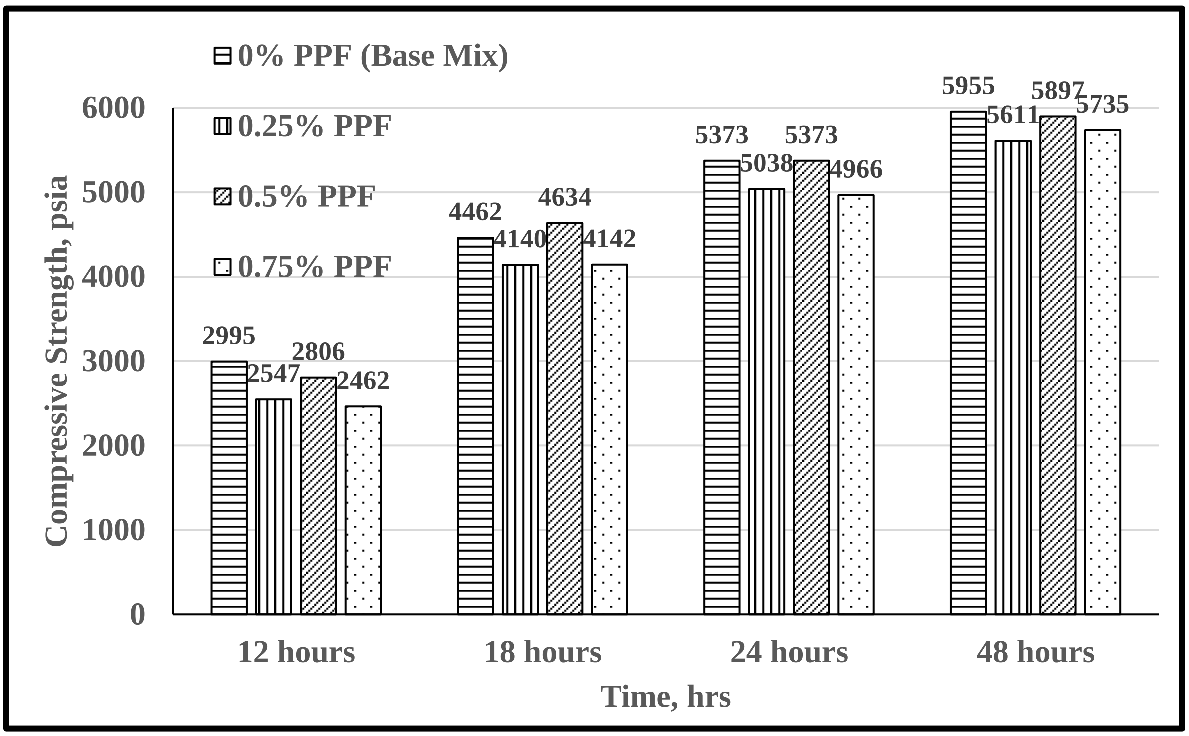

Figure 4.15: The compressive strength trend by sonic method

Table 4.5: The time to gain various compressive strength values at 292^{0}F and 3000 psia for all the cement compositions

Compressive strength, psia	**Time, hh:mm**			
	0% (Base mix)	**0.25% PP Fiber**	**0.5% PP Fiber**	**0.75% PP Fiber**
50	2:33	2:38	2:43	2:38
500	2:55	3:09	3:03	3:05
2000	4:57	5:49	6:06	9:30

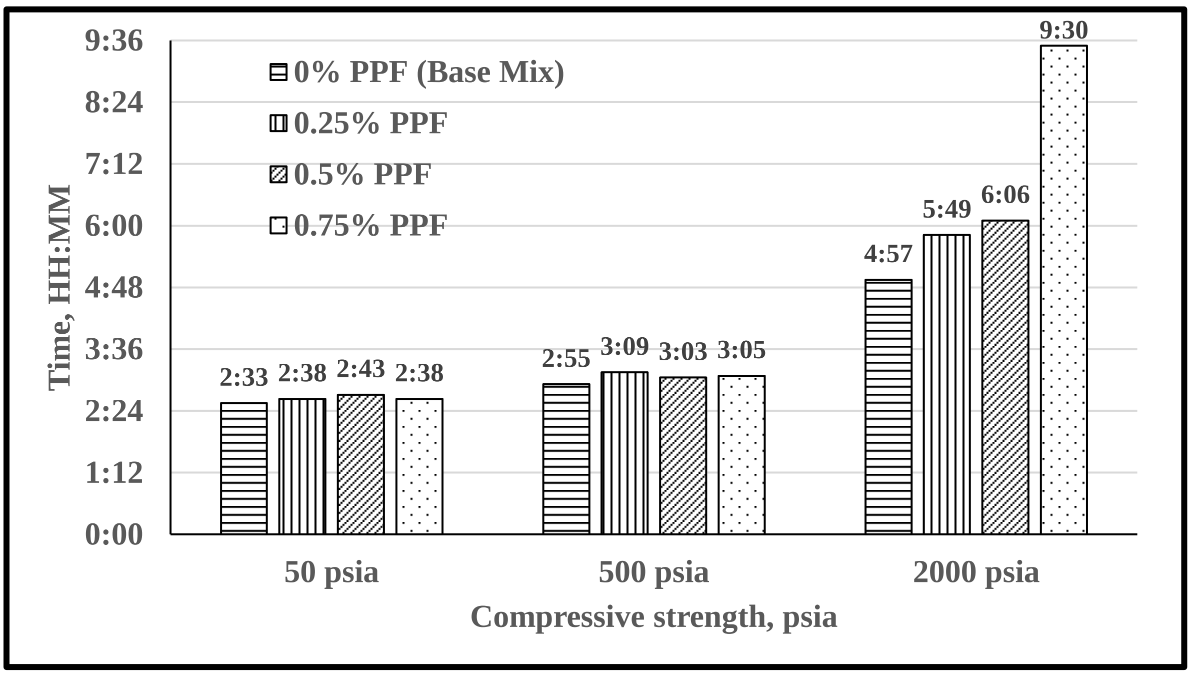

Figure 4.16: The time trend to reach 50, 500 and 2000 psia compressive strength

In addition, one of the important aspects in the case of well cementing is the strength development to reach the value of 50, 500, and 2000 psi, where they are needed to specify before starting the drilling and completion operations (see **Table 4.5**).

UCA cement tests are conducted on a cement containing various percentages of polypropylene fiber, and from the results we noticed that adding 0.5% BWOC polypropylene fiber to the cement mix gave the smallest time period of 20 minutes in the case of strength development from 50 till 500 psi. In contrast, cement mix containing (0.25, and 0.75%) polypropylene fiber gave a transient time of (31, 27, minutes) respectively, which is higher compared with 0.5% polypropylene fiber (see **Figure 4.17**).

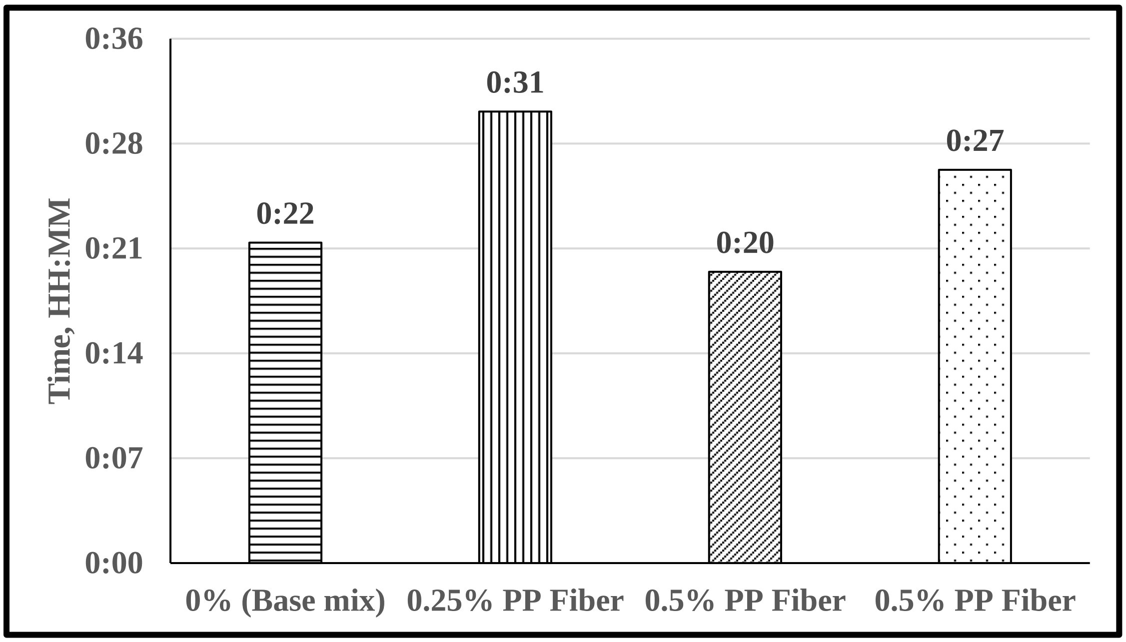

Figure 4.17: The transition time trend of achieving 50 to 500 psia compressive strength

In addition to that, knowing the required time to achieve a compressive strength of 2000 psi is essential in the case of perforation and stimulations jobs. Here gain was observed that 0.25% polypropylene fiber gave the lowest time to reach 2000 psi compressive of around 5:49 minutes compared with the base as well as other cement mixes with higher percentages of polypropylene fiber as shown in **Figure 4.16**.

In short, the addition of 0.5% polypropylene fiber to the cement mix resulted in an improvement in the early strength development, and gave the maximum compressive strength of 5897 psi after 48 hours curing. After that a reduction in the final compressive strength was observed as the amount of polypropylene fiber increased to reach a strength of around 5735 psi in the case of adding 0.75% polypropylene fiber.

4.5.2 Effect of Polypropylene Fiber on Destructive Compressive Strength

Compressive strength is an important issue, where drilling engineers carefully consider before resuming any drilling operation. In fact, cement integrity and long-term bearing ability are determined by the compressive strength property. Four cement systems, consists of polypropylene fiber with different percentages of 0, 0.25, 0.5, and 0.75% were subjected to API compressive strength test at both high pressure and temperature conditions. These cement systems were mixed and placed in moulds of cement, and then subjected to a pressure of 3000 psi and a temperature of 292^{o}F, and after that left in the curing machine for 24 hours. As the test completed, the cubes were detached and removed from the moulds, then subjected to axial increasing load until they crack and the compressive strength values reported.

From **Table 4.6** and **Figure 4.18** it is clear that adding polypropylene fiber with percentages of 0.25% BWOC caused an increase in the compressive strength compared with other cement systems or cement system with higher percentages of it. In other words, adding 0.25% polypropylene fiber improved the compressive strength, which might be a consequence of high percentages of silica in the polypropylene fiber that accordingly induced pozzolanic reaction. Maximum compressive strength was achieved with 0.5% polypropylene fiber, however, as mentioning that the polypropylene fiber lessens the density and does not affect the compressive strength appreciably if the polypropylene fiber increases.

Table 4.6: The destructive compressive strength results at 292^{0}F and 3000 psia for all the cement compositions

Compressive strength, psia			
0% (Base mix)	**0.25% PP Fiber**	**0.5% PP Fiber**	**0.75% PP Fiber**
6246	6726	8434	7709

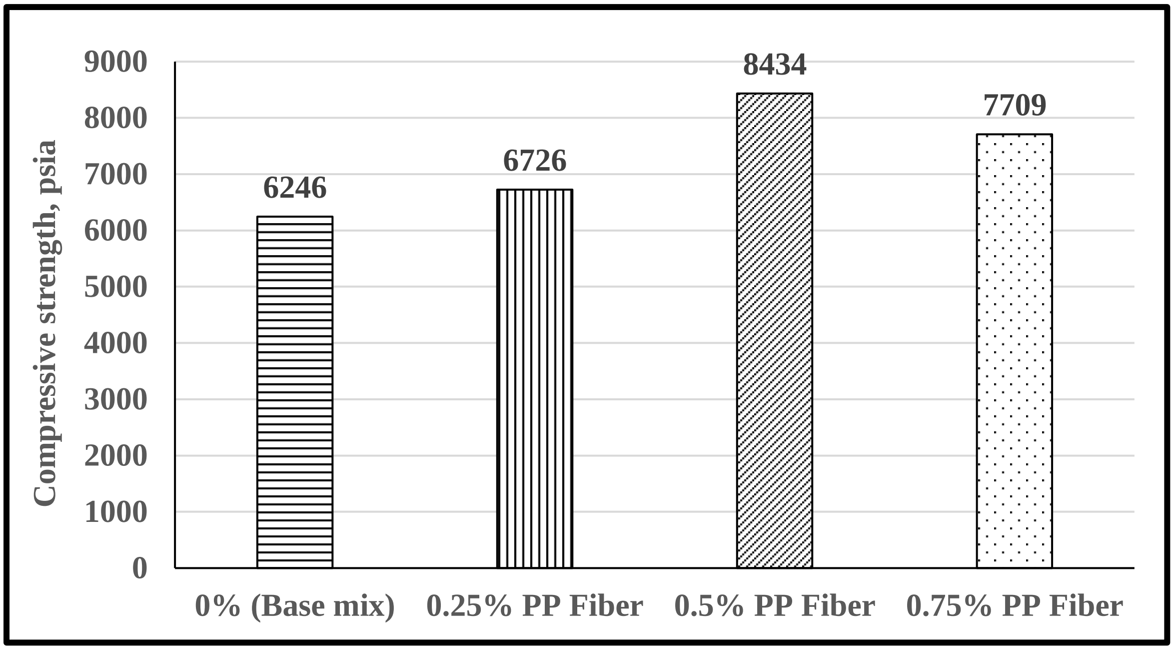

Figure 4.18: The compressive strength trend by the crushing approach

To confirm that this increasing of the compressive strength come from the polypropylene fiber contribution in the cement. Another experiment has been performed by preparing the cube moulds of Class G cement, Class G with silica flour and Class G with 0.5% polypropylene fiber, those cubes are conditioned for a day at 200^{o}C and 3000 psia. The cubes are removed from moulds and kept in water for next 24 hours. At the end of curing period, the cubes crushed to get compressive strength. **Table 4.7** explains the results of compressive strength for different percentages of polypropylene fiber conducted at ambient conditions.

Table 4.7: The destructive compressive strength results at 200°F and 3000 psia for all the cement compositions

Compressive strength, psia		
Class G	**Class G + Silica Flour**	**Class G+ 0.5% PPF**
4198	4461	4949

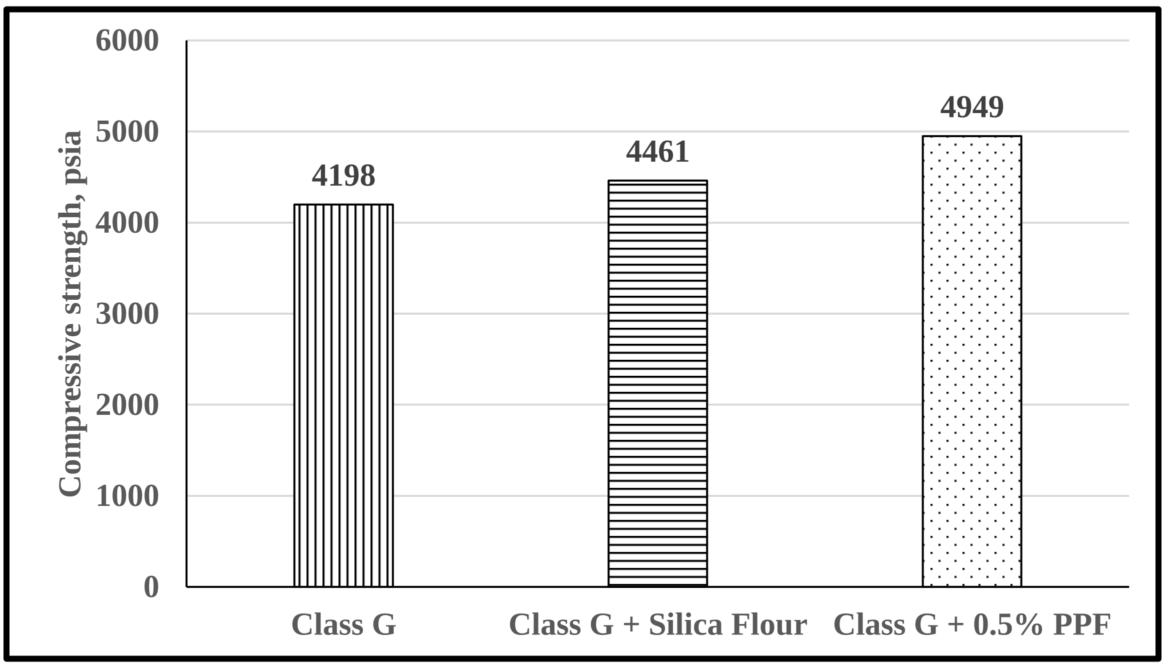

Figure 4.19: The compressive strength trend by the crushing approach

From the **Table 4.7** and **Figure 4.19** results, it is observed that addition of polypropylene fiber to cement system affects the compressive strength of cement. The rise in percentage of polypropylene fiber results in increase of the compressive strength even without the silica flour support.

4.6 EFFECT OF POLYPROPYLENE FIBER ON TENSILE STRENGTH

The tensile strength properties determine the integrity of cement and its ability to withstand the perpendicular loading stresses. The four cement systems having polypropylene fiber percentages of 0%, 0.25%, 0.5% and 0.75% BWOC have been subjected to the tensile strength test in which the molds of cement are made and cured at 292°F and 3000 psia pressure for 24 hours in HPHT curing chamber. At the end of test, the cubes are removed from molds and core plugs with 1” diameter are made from them. Those plugs are cut into three-disc shape specimens with 0.5” height and then every specimen is loaded by diametrically across the circular cross he loading causes a tensile deformation perpendicular to the loading direction, which yields a tensile failure. By registering the ultimate load and by knowing the dimensions of the specimen, the Brazilian tensile strength of the material can be computed.

Table 4.8: The Brazilian tensile strength results at 292^{0}F and 3000 psia for all the cement compositions

Brazilian tensile strength, psia			
0% (Base mix)	**0.25% PP Fiber**	**0.5% PP Fiber**	**0.75% PP Fiber**
1333	1383	1556	1574

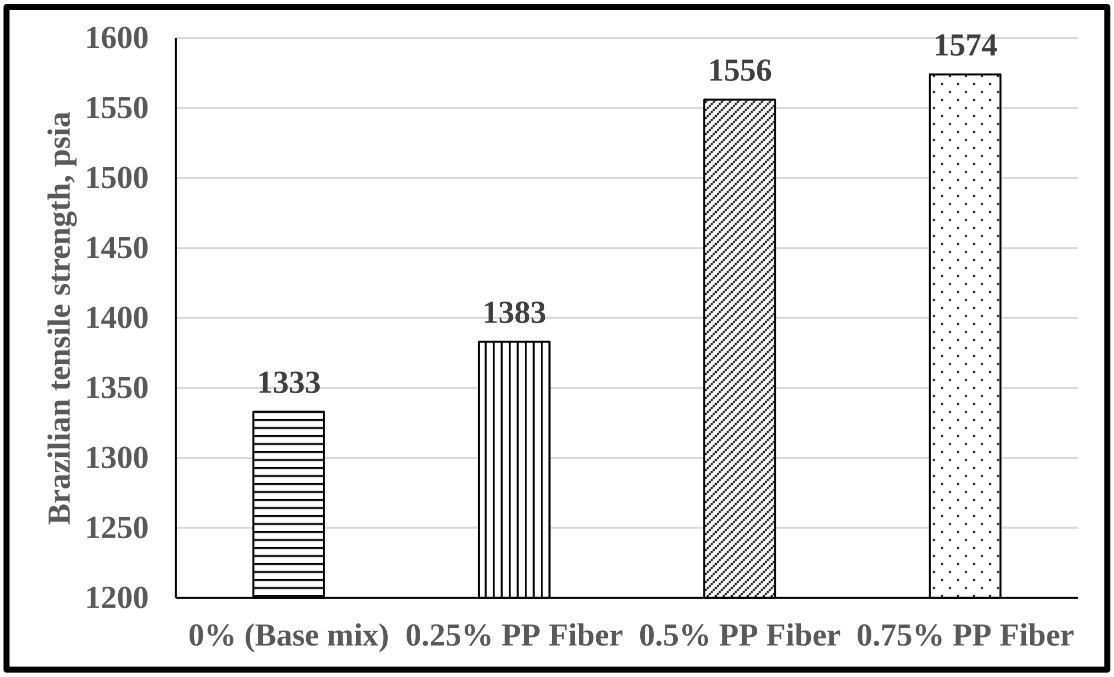

Figure 4.20: The Brazilian tensile strength trend for various polypropylene fiber concentration

From **Table 4.8** and **Figure 4.20**, it was observed that the cement with fiber has more tensile strength than the base mix cement due to the improvement that fibers provide to withstand the tensile stresses with up to 15% increment of the base mix when the high percentage of polypropylene fiber used for this case (0.75%).

4.7 EFFECT OF POLYPROPYLENE FIBER ON RHEOLOGY AND GEL STRENGTH

Rheology is a key factor in understanding the flow of fluids as well as solid deformation under stress and strain. Knowing of cement rheology can provide us with valuable information regarding which additives must be added to cement design. It also gives a quick guess of the frictional pressure losses and the required pump pressure needed during pumping. Furthermore, rheological properties are used in describing the quality of the final cement product and predicting its future performance at work environment as well as its physical properties during and after cementing processing. Cement rheology can be obtained by determining cement flow properties like plastic viscosity, yield point, gel strength, and frictional properties.

Table 4.9 shows plastic viscosity and yield point of cement base mix with various percentages 0, 0.25, 0.5, and 0.75% of polypropylene fiber. It is observed that the addition of the polypropylene fiber to the cement does not put prominent effect on the rheological properties because that the fibers does not significantly alter the plastic viscosity of the cement which means that the fibers solid particles would not contribute to enhance the viscosity. In contrast, the fiber does not have effect on changing the rheological properties.

The cement system is also subjected to gel strength cement test. Gel strength can be defined as a measure of the attractive forces between the particles of the produced cement, which cause gelation development when the flow stopped. It can also give the field operator a quick idea of cement gelation and if there is settling within the produced cement.

Gel strength test is conducted on Fann Viscometer and results are provided in **Table 4.9**. It is evident from the results that the addition of polypropylene fiber in base cement results in change in gel strength. The polypropylene fiber addition does not put prominent effects on the initial gel strength, 10- sec, as the results are almost same in 0, 0.25%, 0.5% and 0.75% polypropylene fiber cement systems.

When the polypropylene fiber cement systems are subjected to 10-min gel strength, they slightly affect the gelling behavior of cement slurries. It can be evaluated from the results that up to 0.75% BWOC polypropylene fiber cement systems the 10-min gel strength results are almost similar. In other words, adding the polypropylene fiber to the cement system helps in developing early gel strength and slightly reducing the settling problems

Table 4.9: The rheological properties results for all the cement compositions

Property	**0%**	**0.25% PP Fiber**	**0.5% PP Fiber**	**0.75% PP Fiber**
Plastic viscosity, cp	263	261	260	253
Yield point, lb/100sqft	6	6	7	7
Gel strength @ 10 sec	6	6	5	5
Gel strength @ 10 mins	22	23	23	25

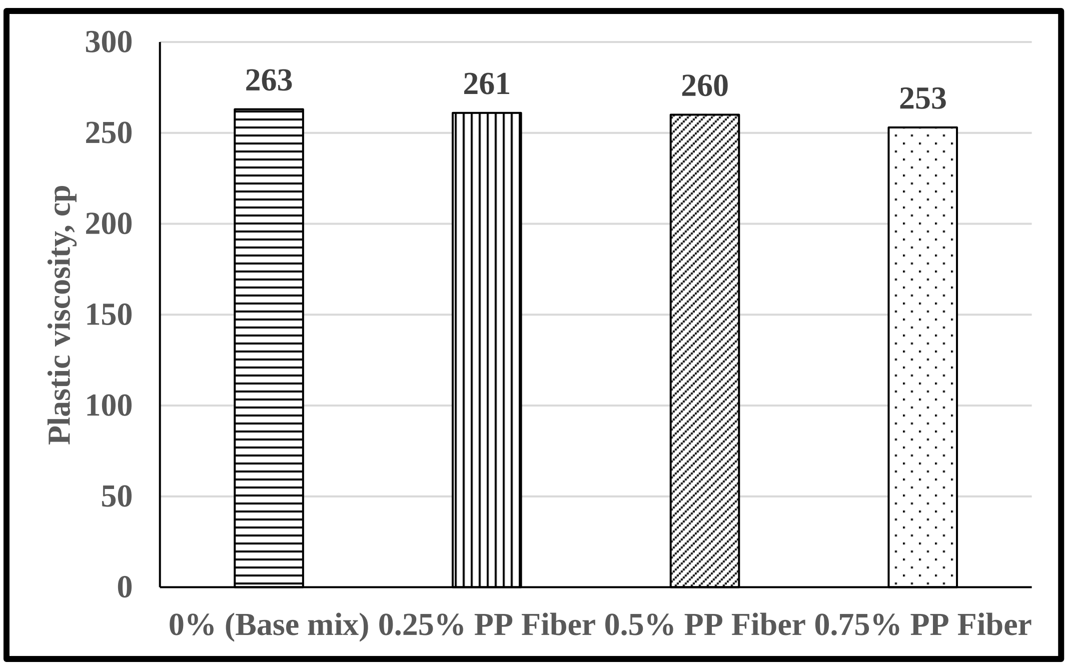

Figure 4.21: The plastic viscosity trend for various polypropylene fiber concentration

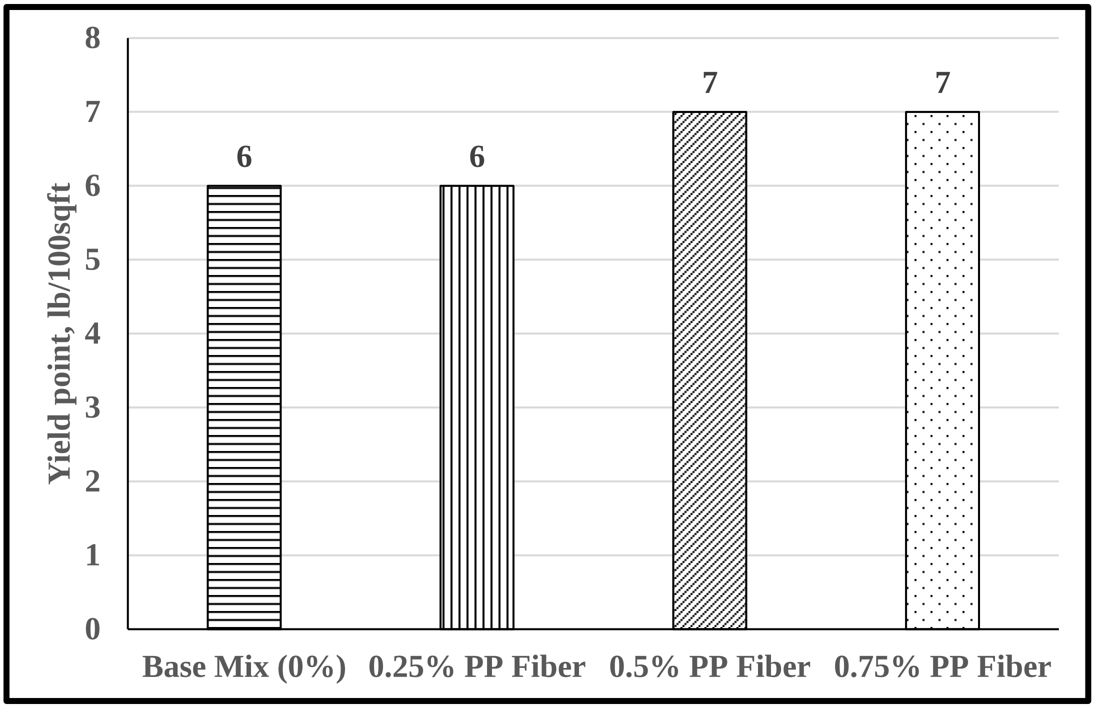

Figure 4.22: The yield point trend for various polypropylene fiber concentration

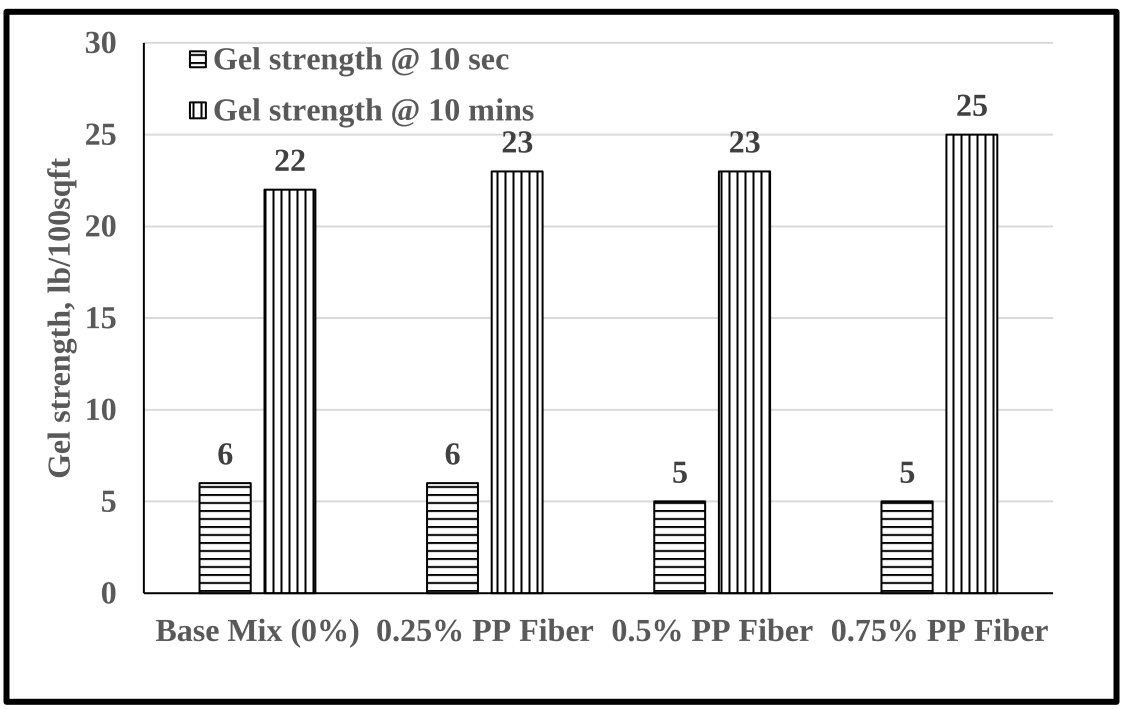

Figure 4.23: The gel strength trend for various polypropylene fiber concentration

4.8 EFFECT OF POLYPROPYLENE FIBER ON POROSITY AND PERMEABILITY

Permeability is an important property, in which it controls the ability of the fluid to flow at different pressures, and explains the long term performance of cement sheath. The main function of the cement sheath is to seal the formation zones and stop the fluid from moving between them. This can be achieved only if a lower permeability cement sheath is obtained.

Porosity is also as important as permeability, and is defined as a void space in the cement sheath where fluids are stored in, and later can affect the long term durability of the cement sheath.

In these experiments, after the cement cubes cured for 24 hours in the curing machine, cement plugs are drilled out of them. Porosity and permeability cement tests are conducted using automated porosity meter/permeability meter under a confining pressure of 500 psi.

Table 4.10 represents porosity and permeability cement results of 0, 0.25, 0.5, and 0.75% of polypropylene fiber after 24 hours curing.

Table 4.10: Porosity and permeability of cement with 0, 0.25, 0.5 and 0.75% of polypropylene fiber after 24 hours curing

Property	Base Mix (0%)	0.25% PP Fiber	0.5% PP Fiber	0.75% PP Fiber
Porosity %	27.49	25.09	24.56	22.86
Permeability, md	0.0023	0.002	0.0014	0.001

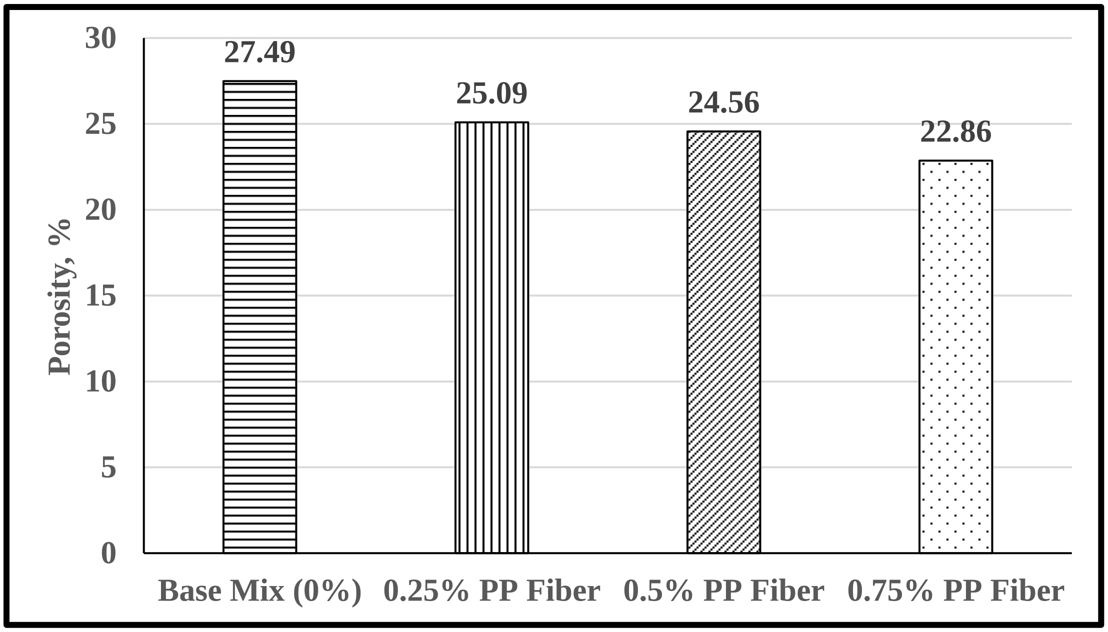

Figure 4.24: The porosity trend for various polypropylene fiber concentrations

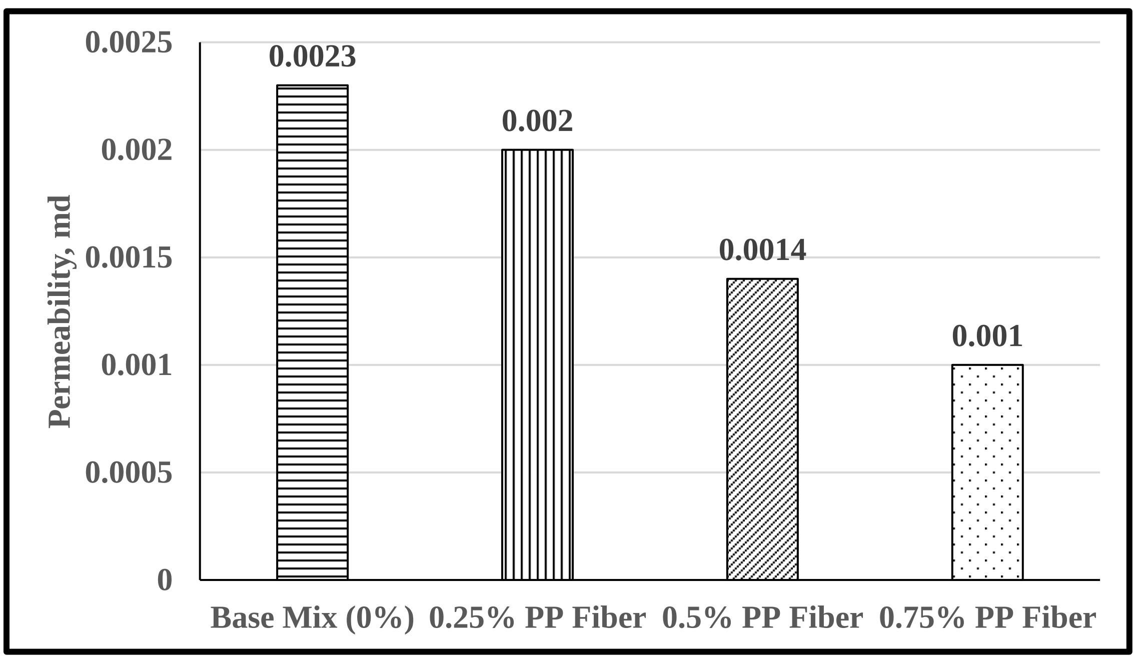

Figure 4.25: The permeability trend for various polypropylene fiber concentrations

It was observed that the addition of polypropylene fiber to the cement mix resulted in a reduction the both porosity and permeability. Addition of polypropylene fiber caused a slight reduction in the porosity, where all the results were around 26% lower by 1% from a cement base mix. On the other hand, a significant reduction in the permeability results was observed when higher percentages of polypropylene fiber were added to the cement mix compared with the cement base mix. This makes all the 0.25, 0.5, and 0.75% good choices with respect to permeability. In short, the 0.25, 0.5, and 0.75% polypropylene fiber percentages are all recommended for using with respect to permeability.

4.9 MICROSTRUCTURAL ANALYSIS

The cement composition is analyzed by exposing the cement to structural tests like SEM and XRD. The SEM cement test is used to identify the composition, topography, and the pore structure of the final cement product, whereas XRD method is the usually used to study the cement composition as well as cement hydration.

Generally, when water mixed with cement, a chemical reaction will take place, causing the cement to disintegrate and resulted in a production of hydrated compounds within the mix. The hydration process will continue because the solubility of the main anhydrous compounds comes higher than those from the hydrated products, and this process will continue till complete hydration takes place.

In general, the hydration of cement class G mostly depends on the curing temperature used. Results showed that the main hydration products in the clean cement are calcium, silica hydrate C-S-H, ($CaSiO_4.3H_2O$), C_2SH_2 ($CaSiO_4.2H_2O$), $C_3S_2H_3$ (Ca_3 $(HSiO_4)_2.2H_2O$), calcium hydroxide CH [Ca $(OH)_2$], ettringite Aft ($3CaO.Al_2O_3.CaSO_4.32\ H_2O$) Afm ($3CaO.Al_2O_3.CaSO_4.12\ H_2O$).

When silica flour 35% BWOC mixed with the cement, the hydrated products were almost different compared with the simple class G cement under curing temperature of 144ºC and 3000 psi cured for 24 hours. Also, it was quite clear in XRD spectra that specific peaks appeared with SiO_2, whereas CH was weakened by increasing the amount of silica flour added. **Figure 4.26** shows XRD spectra of hydration products of cement with 0% polypropylene fiber (base mix) added and cured at HPHT for 24 hours. It was observed also that peaks of CH and C_2SH were almost disappeared in the spectra when silica flour added, which proves that big amounts of C_2SH crystalline has been transformed to $C_5S_6H_5$ (tobormorite), and peaks of SiO_2 still appear in the XRD pattern.

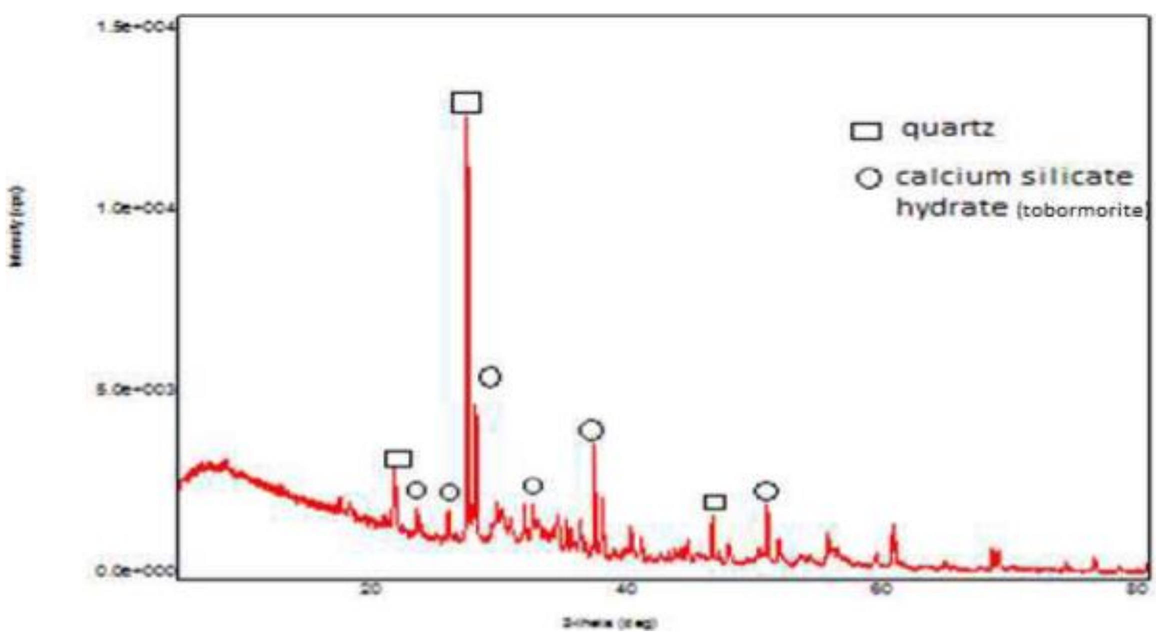

Figure 4.26: XRD hydration products with 0% polypropylene fiber cured at HPHT for 24 hours

So, when silica flour is added to the cement, $C_5S_6H_5$ crystalline would appear in the final hydration product, which is considered a good type of crystal with a needle shape. This needle shape of $C_5S_6H_5$ crystalline is combined and joined with each other to produce an ideal, and well-proportioned network structure in the hardened cement, which helps the cement to maintain high compressive strength. **Figure 4.27** shows an SEM photograph of hydration products with 0% polypropylene fiber cured at HPHT for 24 hours.

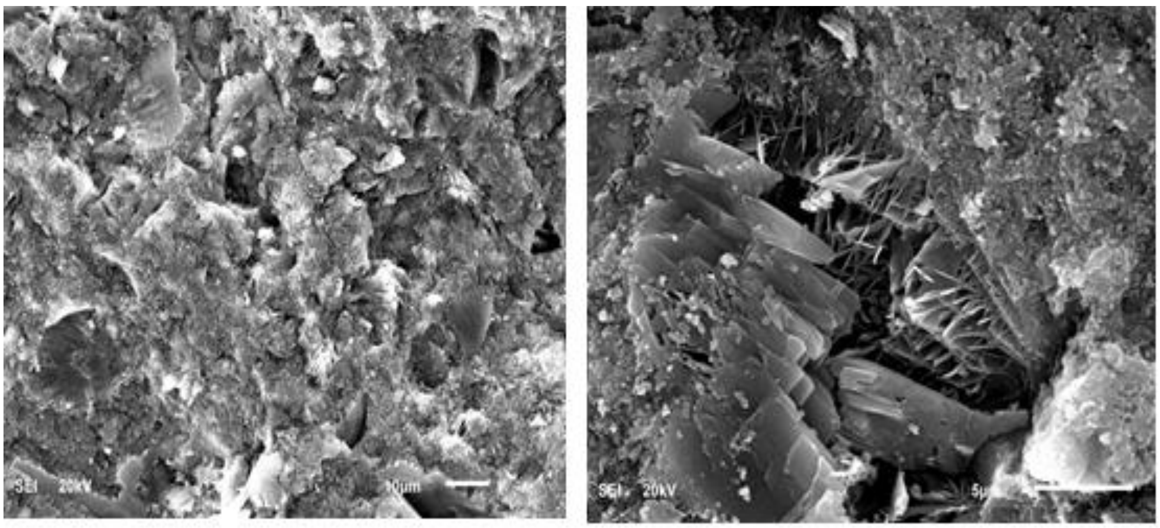

Figure 4.27: SEM photograph of hydration products with 0% polypropylene fiber at HPHT for 24 hours

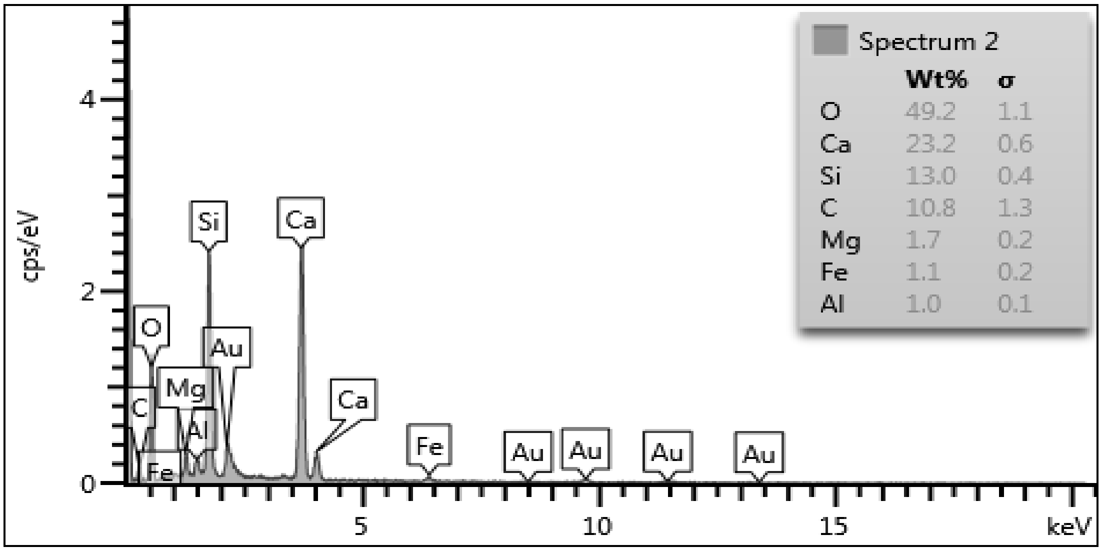

Figure 4.28: Hydration products (SEM) with 0% polypropylene fiber cured at HPHT for 24 hours

As the polypropylene fiber added to the cement, CH is disappearing, which indicated more polymerization occurred in the final hydrated product. Polypropylene fiber is reacted in the solution and caused CH to transform into CSH as clear in the XRD pattern. When 0.25% polypropylene fiber mixed with the cement, quartz as well as CSH appeared in high percentages in the final cement product, and resulted in a strong cement structure. **Figure 4.29** shows XRD spectra of hydration products with 0.25% polypropylene fiber cured at HPHT for 24 hours.

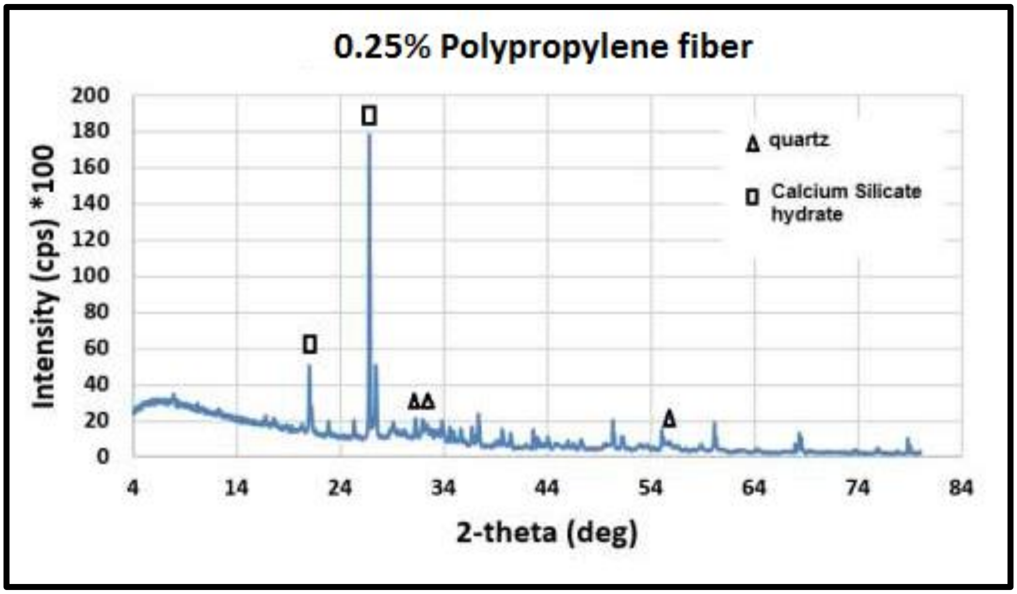

Figure 4.29: XRD hydration products with 0.25% polypropylene fiber cured at HPHT for 24 hours

As described above, the high compressive strength can be obtained when higher percentages of CSH appear in the hydrated product. This is because CSH is known as a good type of crystal, where it could interweave and bond with each other to make a perfect and well-proportioned network structure in the hardened cement as in **Figure 4.30**. Cement slurry admixed with polypropylene fiber resulted in big quantities of this favourable crystal due to the availability of silica in the mix. So, addition of polypropylene fiber resulted in dense structure, and caused improvement in the final compressive strength. **Figure 4.31** shows SEM element analysis for 0.25% polypropylene fiber cured at HPHT for 24 hours. As shown in the spectrum, it was visible that the final cement product contains higher weight percentages of silica and calcium, which confirm the formation of CSH in the final harden cement.

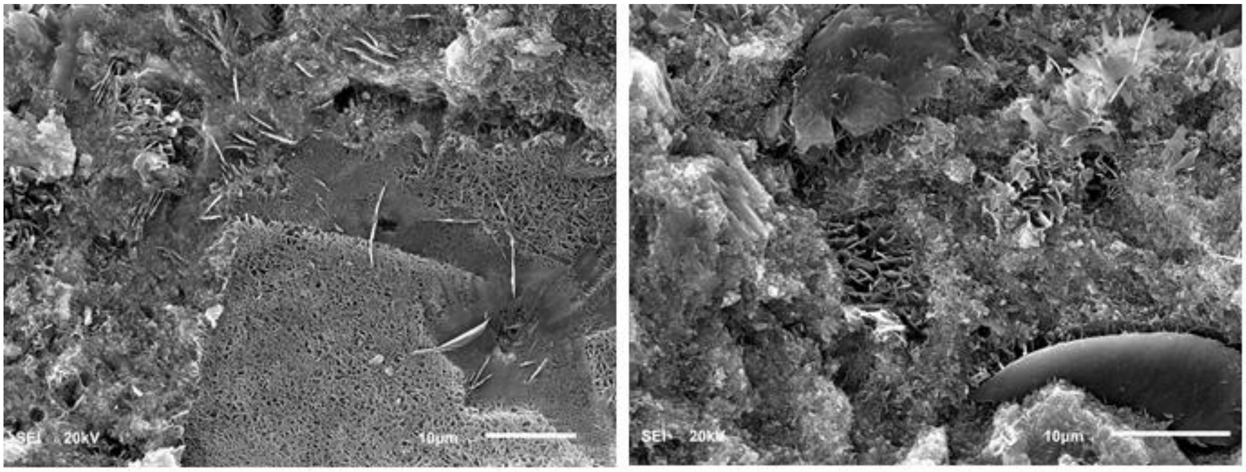

Figure 4.30: SEM photograph of hydration products with 0.25% polypropylene fiber at HPHT for 24 hours

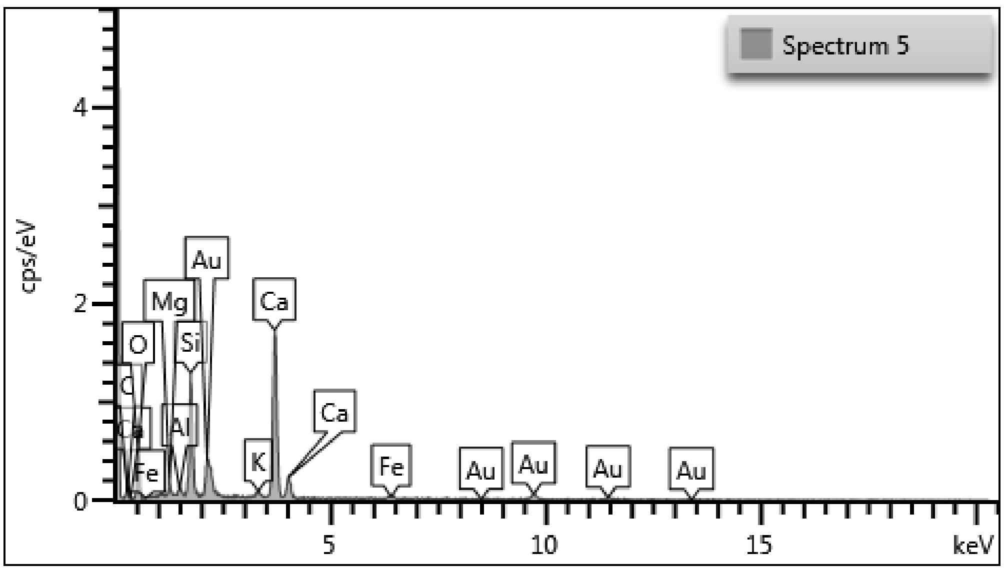

Figure 4.31: Hydration products (SEM) with 0.25% polypropylene fiber cured at HPHT for 24 hours

When 0.5% polypropylene fiber added to the cement mix, the amount of producing quartz crystals was considerably high, and could be related to the hydration reaction between polypropylene fiber and CSH, where huge amount of silica found in the mix as showing clearly in **Figure 4.32**. After that, the quartz crystals are combined and merged with each other forming an ideal network structure in the final resulted cement paste (see **Figure**

4.33). On the other hand, it was observed that when 0.5% polypropylene fiber mixed with the cement, the value of compressive strength increased compared with 0.25% polypropylene fiber, which showed that the presence of quartz crystals in the harden cement is not the only proof of higher compressive strength. **Figure 4.34** illustrates the SEM element weight analysis for 0.5% polypropylene fiber cured at HPHT for 24 hours. It was observed that the final cement product contains a higher weight percentage of silica, and calcium, which demonstrates the formation of higher percentages of CSH in the final harden cement.

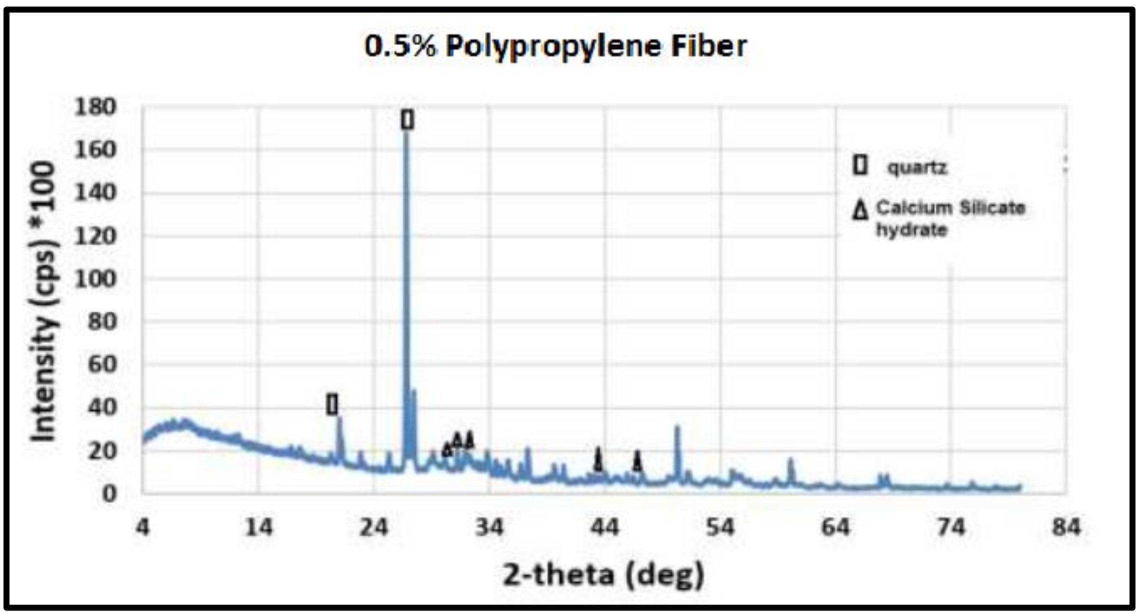

Figure 4.32: XRD hydration products with 0.5% polypropylene fiber cured at HPHT for 24 hours

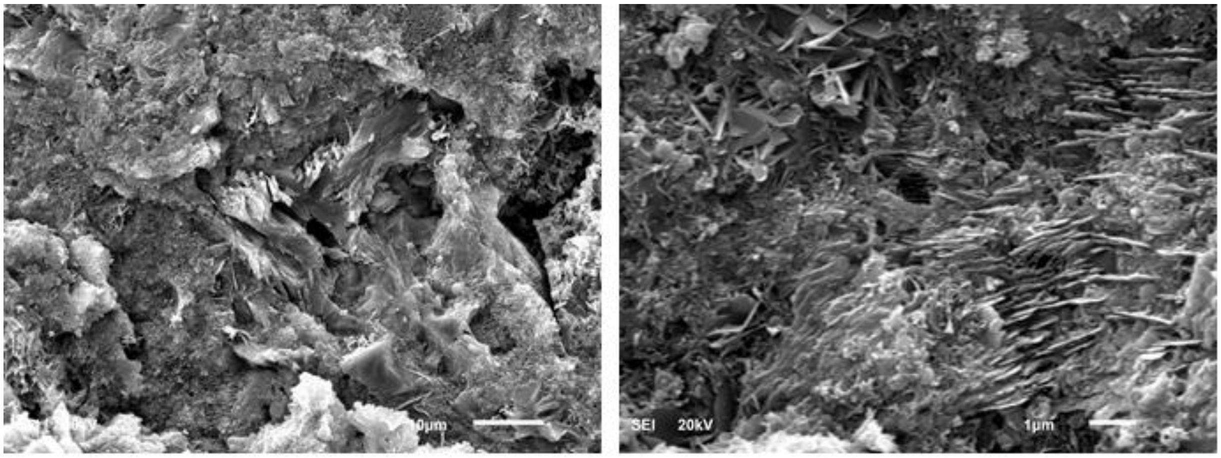

Figure 4.33: SEM photograph of hydration products with 0.5% polypropylene fiber at HPHT for 24 hours

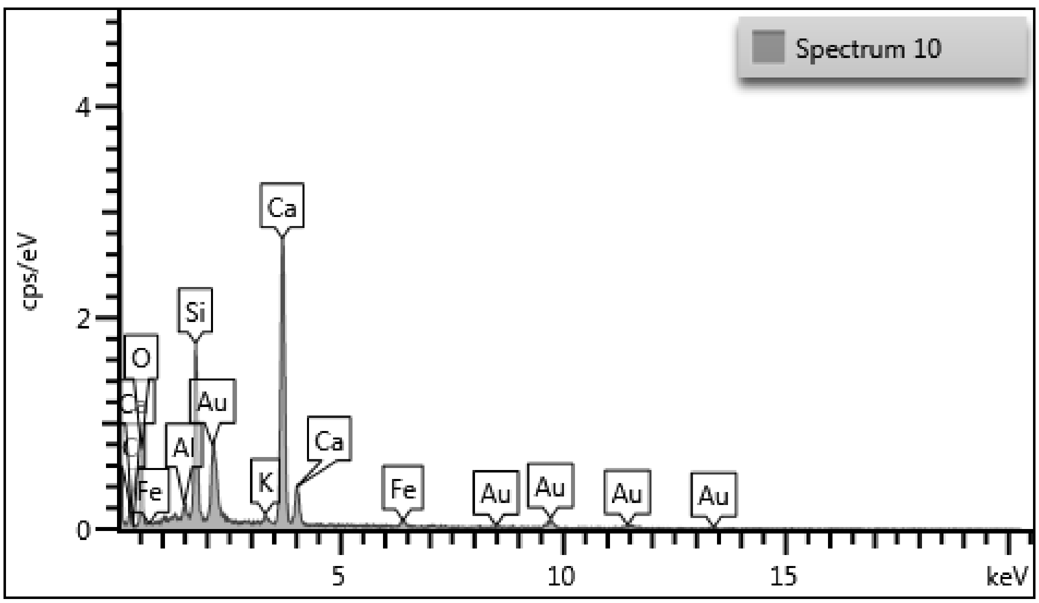

Figure 4.34: Hydration products (SEM) with 0.5% polypropylene fiber cured at HPHT for 24 hours

In the case of adding 0.75% polypropylene fiber, the same cement behavior was observed as described above. In fact, adding 0.75% polypropylene fiber caused an increase in the pozzolanic reaction and more of calcium silica hydrate CSH crystals (tobormorite) are formed in the cement past, which play an important role in the speed of compressive strength development, where big quantities of silica were found in the mix (see **Figure**

4.35). However, the compressive strength with 0.75% polypropylene fiber is higher compared with 0.5%, which might be related to the reduction of the amount quartz crystals formed in the final harden cement paste. **Figure 4.36** represents the SEM results of 0.75% polypropylene fiber cured at HPHT for 24 hours. From the SEM picture it was observed that the hard cement had gaps and voids all over the structure. **Figure 4.37** shows the SEM element weight analysis for 0.75% polypropylene fiber cured at HPHT for 24 hours. It was observed that the final cement products contain higher weight percentages of silica and calcium, which demonstrates the formation of more CSH in the final harden cement.

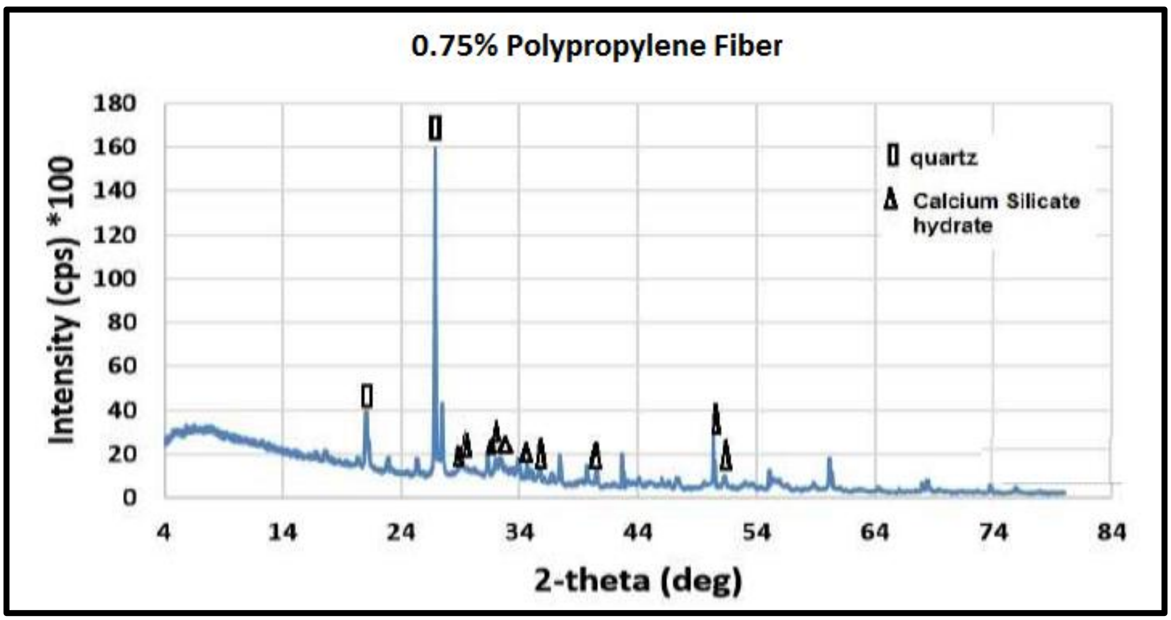

Figure 4.35: XRD hydration products with 0.75% polypropylene fiber cured at HPHT for 24 hours

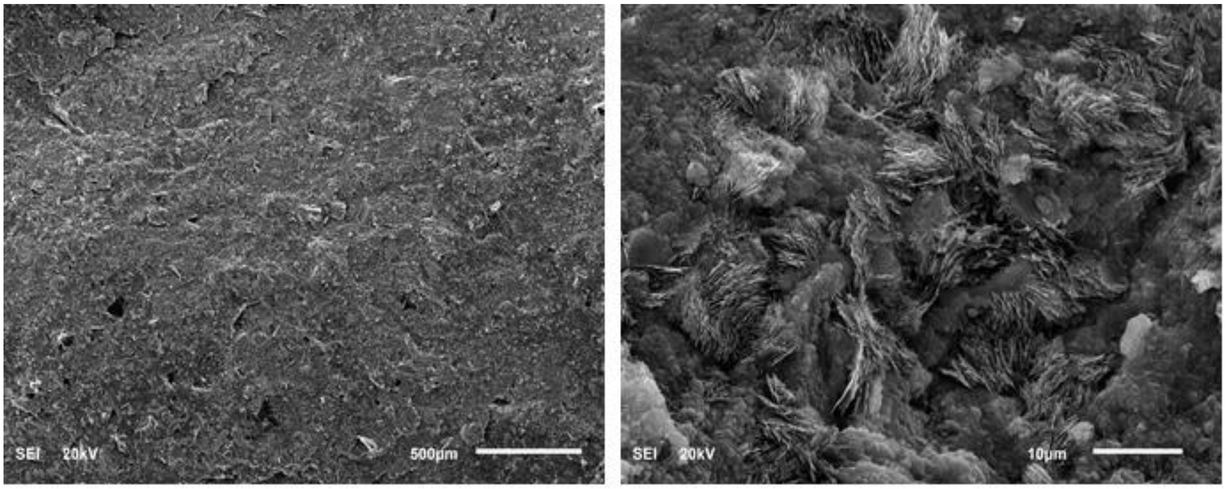

Figure 4.36: SEM photograph of hydration products with 0.75% polypropylene fiber at HPHT for 24 hours

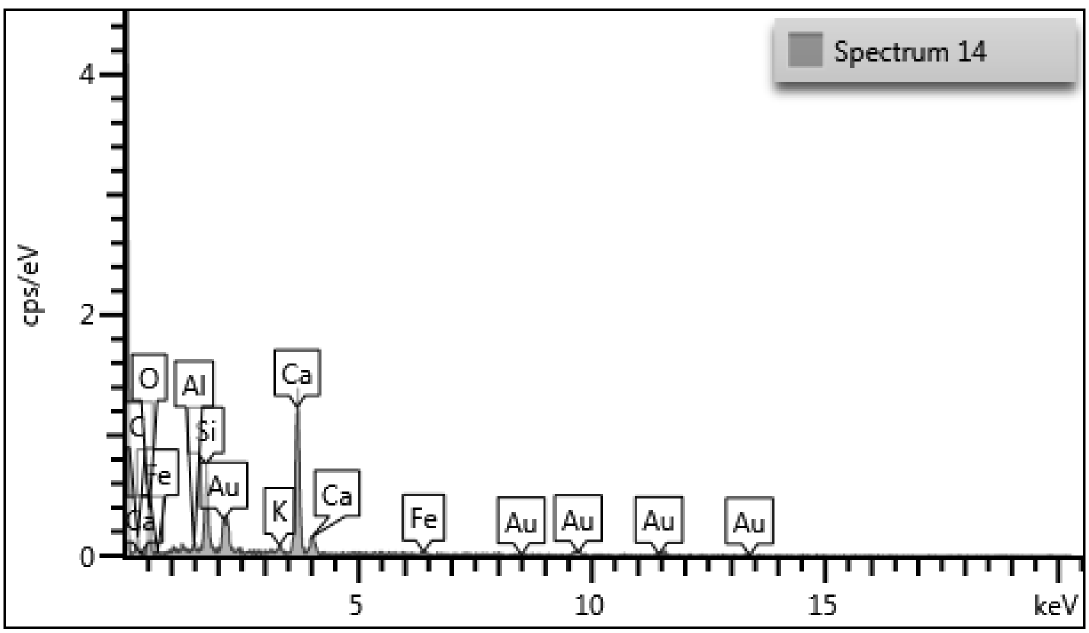

Figure 4.37: Hydration products (SEM) with 0.75% polypropylene fiber cured at HPHT for 24 hours

Chapter 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSIONS

In this work the effect of adding the polypropylene fiber were examined on the cement properties under HPHT conditions which has been investigated and reported. The conclusions obtained from this work are summarized as follows:

1. The polypropylene fiber contributes in resistance of the axial and perpendicular loads which results in improving the cement compressive strength and tensile strength.
2. The polypropylene fiber helps to decrease the porosity and permeability of the cement by filling the pores structures and block the interconnection between the pores inside the cement.
3. There is no free water separation in all polypropylene fiber cement systems after aging as polypropylene fiber blocks the capillaries and prevents the water flow.
4. It is observed that addition of polypropylene fiber reduces the density of cement slurries. But the difference in densities of all polypropylene fiber cement systems is minimal.
5. From the rheology test, it is investigated that polypropylene fiber does not have notable effects on changing the viscosity and yield point of cement slurry and the rheological properties

6. From thickening time test, it is observed that the polypropylene fiber acts as an accelerator as it speeds up the hydration reaction. It will be very helpful in cementing shallow wells. Also by adding retarders or removing accelerators will be helpful as well in deep well cementing.
7. Microstructural analysis showed that polypropylene fiber particles block the capillaries by filling the pores in the cement, so a dense cement structure is achieved.
8. The optimum percentage used was 0.5% polypropylene, and it had the following advantages:
    a. Rapid as well as early strength development was achieved with this percentage tested using sonic method, for instance, the 2000 psi strength was reached after 5 hours and 49 minutes. This behavior is advantageous in reducing wait on cement time.
    b. The high compressive strength achieved was around 6000 psi with this percentage tested using sonic method after 48 hours of the curing process.
    c. This trend was also conformed when material tested using the crushing method, where the highest compressive strength reported was 8434 psi after 24 hours of the curing process.
9. Further addition of polypropylene fiber affected the cement properties and lowered the final strength.

5.2 RECOMMENDATIONS

This work includes the findings of polypropylene fiber effects on oil well cement properties in high pressure and temperature applications but still a lot of work left to be done. In all experiments, the impact of polypropylene fiber effects in shallow applications with low pressure and temperature conditions and with different water to cement ratios should be investigated, and also with different water cement ratios.

Polypropylene fiber has potential to provide high integrity to cement sheath in high temperature applications separately. The impact of other types of fibers should be investigated.

Polypropylene fiber could be used as a good lost circulation agent especially for the light-weighted cements, so the effect of the polypropylene fiber on other types of cements should be investigated. Also, it could be used as a good fluid loss agent for the drilling fluids and that should be investigated as well.

As observed from all experiments that conducted to see the effect of adding the polypropylene fiber on the cement integrity, they were conducted in short term intervals, so the effect of polypropylene fiber on long term effect should be evaluated.

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