PERFORMANCE OF SAUDI BENTONITE AND NANO CLAY ADMIXED CEMENT FOR HPHT CONDITIONS

BY

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Dedication

To My Parents,

My brothers Anwar and Jalal, My Wife and Lovely Son Abdullah

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LIST OF ABBREVIATIONS

API	:	American Petroleum Institute
ASTM	:	American Standards for Testing and Measurement
Bc	:	Bearden Consistency Unit
BHCT	:	Bottom Hole Circulating Temperature
BHP	:	Bottom Hole Pressure
BHST	:	Bottom Hole Static Temperature
BWOC	:	By Weight of Cement
EOR	:	Enhanced Oil Recovery
HCS	:	Hollow Ceramic Spheres
HPHT	:	High Pressure High Temperature
HSR	:	High Sulphate Resistant
MSR	:	Moderate Sulphate Resistant
MW	:	Mud Weight
N_C	:	Nano Clay
OSR	:	Ordinary Sulphate Resistant
OWC	:	Oil Well Cements
PCF	:	Pound per Cubic Feet
PV	:	Plastic Viscosity
RPM	:	Rotation per Minute
SEM	:	Scanning Electron Microscope
TRB	:	Time to Reach Bottom
TVD	:	Total Vertical Depth
UCA	:	Ultrasonic Cement Analyser
W/C	:	Water to Cement Ratio
WOC	:	Wait on Cement
XRD	:	X-ray Diffraction
YP	:	Yield Point

ABSTRACT

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In order to keep up with the world's energy demands, oil and gas producing companies have taken the initiative to explore offshore reserves or drill deeper into previously existing wells. The consequence of this, however, has to deal with the high temperatures and pressures encountered at increasing depths. For a well, whether oil or gas, to maintain its integrity and produce effectively and economically, it is pertinent that a complete zonal isolation is achieved during well completion. Hence, the cement slurry must be designed in a way that the produced cement sheath has specific properties to assure its durability, and robustness to accommodate those conditions.

Bentonite is used extensively as an extender additive when mixed with the cement. During cementing weak formation, mixing bentonite with the cement results in a reduction in the cement slurry density, therefore a successful cement job is achieved. Bentonite also works as fluid loss additive when mixed at lower percentages with the main objective to get rid of fluid loss problem during cementing permeable formations. Moreover, bentonite develops high gel strength cement, and shows thixotropic behavior when used to control gas migration.

Saudi bentonite is available in big quantities in Khulays area, 70-km north Jeddah, Saudi Arabia. Saudi bentonite is a calcium-Based, which contains large percentages of silica and lesser of sodium compared with commercial imported bentonite. As a result, Saudi bentonite has a poor swelling behavior when it is hydrated in water. Recently, Nano-materials have proven their effectiveness in most of life fields ranges from textiles, defense to aerospace and energy. Also Nano-materials have shown their superior when they are added to the cement in the construction industry, where small amounts of these materials have obvious effect and enhancement in the mechanical properties of the produced cement sheath.

In this study, the investigations focused on evaluation of mechanical and rheological properties of the cement mix which was conducted on a typical well in Saudi oil field under high pressure and high temperature (HPHT) conditions by adding (1) the untreated Saudi bentonite added at 1, 2, and 3% by weight of cement (bwoc), (2) Nano clay at 0.5, 1, and 1.5% admixed with 1% untreated Saudi bentonite bwoc, and (3) treated Saudi bentonite developed by KFUPM researchers used at percentage of 1.9% bwoc to produce a low density cement. The investigations conducted on the above mixes under HPHT conditions include thickening time cement test, plastic viscosity and yield point, the evolution of compressive strength by ultrasonic method, crushing strength, free water, fluid loss, permeability, porosity, and microstructural characterization.

Results indicate that under HPHT conditions, untreated Saudi bentonite and Nano clay exhibit pozzolanic activity resulting in a significant enhancement of compressive strength, lower the porosity and the permeability, allow control on thickening time, reduce fluid loss with no free water, and prevent settling. Results obtained from the treated Saudi bentonite fulfill all the requirements needed from the bentonite when used as an extender in oil well cementing application. In fact, it reduces the cement density and provides good dispersion and suspension of the particles when compared with the commercial bentonite. This similar properties exposed by the treated Saudi bentonite proved that it can be used as an alternative to the commercial bentonite in the oil well cementing applications.

ملخص الرسالة

الاسم الكامل : أمجد سعيد أحمد بن ثعلب

عنوان الرسالة : دراسه تأثيرخلط البنتونايت السعودي والنانو كلاي مع الإسمنت تحت ظروف درجة الحراره و الضغط العاليين

التخصص : هندسة بترول

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من أجل مواكبة العالم من حيث الطلب على مادة البترول الخام, أخذت الشركات المنتجه للنفط والغاز على عاتقها عمليات إستكشاف و استخراج النفط الخام والغاز الطبيعي من مناطق جديدة لم تستكشف من قبل أو عن طريق حفر آبارعميقة في أماكن يعرف تواجد النفط فيها مسبقاً. ونتيجة لذلك توجب على هذه الشركات مواجهة بعض الظروف متمثلة في الحرارة والضغط العاليين في تلك المناطق ذات العمق الكبير. بالنسبه للبئر المحفور سوا كان بترولي او غاز طبيعي, ومن أجل ضمان سلامة البئر وتحقيق إنتاج نوعي ذو جدوى إقتصادية, يجب أن يكون البئر مغلف بغلاف أسمنتي جيد يضمن الفصل الجيد بين طبقات البئر ويحافظ على فترة إستمرارية أكبر للبئر. لذا فإن الخليط الإسمنتي يجب أن يصمم بحيث يحتوي الغلاف الإسمنتي على خصائص معينه مثل المتانة والمقدره على التغلب على هذه الظروف.

يستخدم البانتونايت كماده إضافية موسعه عند خلطها بالإسمنت. وعند سمنتة الآبار ذات الطبقات الارضيه السفلى الضعيفة والعالية المساميه, فإن خلط البانتونايت مع الإسمنت يساعد في تخفيض كثافة الخليط الإسمنتي وبالتالي يؤدي إلى الحصول على عملية سمنتة ناجحة نتيجة إستخدام الإسمنت المقترح. كذلك يستخدم البانتونايت كمواد مانعة لفقدان السوائل عند خلطها بنسب قليله حيث انها تعمل على التخلص من مشكلة فقدان السوائل عند سمنتة التكوينات الي تمتاز بنفاديه كبيرة. من ناحية أخرى يساعد البانتونايت على تكوين طبقة هلامية أسمنتية عالية القوة تعمل على تكوين جل يتصلب عند دخوله الفتحات وانخفاظ سرعة دخوله حيث ان الهدف الرئيسي له هو منع هروب الغاز من خلال الطبقه الإسمنتيه.

يتوفر البانتونيات السعودي بكثرة في منطقة الخليص والتي تبعد 70 كم شمال جده في المملكة العربية السعودية. ويعد البانتونايت السعودي من الأنواع التي تحتوي على الكالسيوم, حيث أنه يتميز بإحتوائه على نسبة كبيرة من السيليكا ونسبة قليلة من الصوديوم مقارنة بالبانتونيات التجاري المستورد مما يجعل البانتونايت السعودي ذو قابلية أقل للتضخم عندما يتعرض للماء لأكثرمن ليلة. في الآونة الأخيرة، أثبتت المواد الدقيقة الحجم، أو ما يعرف بالمواد النانونية، فعاليتها في مختلف المجالات، كالنسيج و الدفاع و الفضاء و الطاقة، و قد أثبتت هذه المواد جدارتها عند إضافتها إلى الإسمنت في مجال صناعات البناء والتشييد , حيث أن إضافة كميات قليلة من هذه المواد تساعد على تحسين الخواص

الميكانيكية للخرسانه الإسمنتية المنتجه. كما إن إستخدام النانوكلاي في الصناعات البترولية أحرز تقدماً ملحوظاً في الآونه الأخيرة وساعد في تحسين العمليات الإسمنتية وصلابة الإسمنت الناتج.

إن الإختبارات التى تمت في هذه الدراسه قد تركزت حول تقييم خصائص الإسمنت الفيزيائيه (الميكانيكيه) وخصائص جريان الإسمنت المستخدمة لسمنتة بئر في حقل نفطي في المملكه العربية السعوديه تحت ظروف الضغط والحراره العاليتين وذلك عن طريق إضافه (1) البنتونايت السعودي بالنسب التاليه (1%,2%,3%) بالنسبه لوزن الإسمنت (2) النانو كلاي بالنسب (0.5%,1%,1.5%) لأفضل نسبه تم الحصول عليها عند اضافه البنتونايت السعودي للاسمنت من التجارب السابقه (3) البنتونايت السعودي المعالج الذي تم تطويره عن طريق باحثين في جامعة الملك فهد للبترول والمعادن عند أستخدامه بنسبة 1.9% من وزن الإسمنت لأنتاج خليط اسمنتي له كثافه منخفضه. إن الاختبارات التي تم اجرائها للإسمنت المنتج تحت ظروف الضغط والحرارة العاليتين تشمل زمن تصلب الإسمنت, لزوجة الإسمنت, وتقييم قوة وصلابة الإسمنت باستخدام اجهزة قياس الصلابة بالموجات الصوتية أو عن طريق سحق المكعب الإسمنتي, إختبار فقدان السوائل, إختبار قياس النفاديه والمساميه, إضافه الى تحليل المكونات النهائيه للصخرة الإسمنتية الناتجة.

وتشير النتائج التى تم الحصول عليها الى أن إضافه البنتونايت السعودي و النانو كلاي إلى الخليط الإسمنتي تحت الظروف من ضغط وحراره عاليه يؤدي الي سرعة التفاعل الكيميائي وتحسن كبير في صلابة الإسمنت المنتج وتقليل الفجوات مما يسمح في زيادة التحكم في وقت التصلب للإسمنت وتقليص كمية الماء المفقود ومنع ترسب الإسمنت. كما تشير النتائج التي تم الحصول عليها الى أن إضافه البنتونايت السعودي المعالج إلى الخليط الإسمنتي تحت ظروف الضغط والحرارة العالتيين يعمل على تغطية كل المتطلبات التي يجب توفرها في البانتونيات الذي يستخدم كماده موسعه لتقليل الكثافه وجعل الإسمنت الناتج في حالة الإنتشار المتجانس بدون حصول ترسب للحبيبات الإسمنتية مقارنة بالإسمنت الناتج من خلط البانتونيات التجاري. التشابه في هذه الخواص للبانتونيات السعودي المعالج مع البانتونيات التجاري يوكد إمكانية إستخدام البانتونايت السعودي كبديل للبانتونايت التجاري في تلك التطبيقات المتعلقه بسمنتة الآبار النفطية.

CHAPTER 1

INTRODUCTION

1.1 Overview

As the industry revolution expands, the request for oil has been increased significantly, which in turn, make a notable change in the global economic structure. Statics showed that the growth in oil consumption has been raised by 171 % all over the world through the time between 1965 and 2008 (Yahaba, 2010). For the last twenty years, the quantity oil reverses discovered was less than the amount of oil consumed each year. Thus, to meet growth in demand for oil in the industry, more production wells should be discovered, and added to avoid the problem of oil production shortage with time. In fact, the amount of oil stored underground is not exactly well known and predicting the ultimate drop in oil production is difficult to say. Currently, oil companies are trying to search in new locations and targeting deep zones hopping to hit the oil.

During drilling of oil or gas wells, we usually face the problems such as wellbore instability through drilling salt or shale formations, and fluid movement between layers. To solve these problems, steel casing is run to support the well bore, and minimize the damage. This steel casing is bonded to the wellbore by using cement. In fact, casing cementing is a critical issue, which must be done in a perfect way, so a healthy well is obtained without problems in the future. Also well cementing is aimed to stop fluid migration between zones, provide good cement bonding which support the casing, and later prevent shocks when drilling operation pursued. In addition, cement sheath stops the casing from getting corroded, and used as a plug to stop fluid loss to the thief zones (Bourgoyne et al., 1986).

When cement is placed in a wall, after a certain time the cement starts to set and cause an increase in the compressive strength, which is important in the case of further drilling operation. Early strength development is also a critical issue, since it supports the casing, and provide mechanical and hydraulic isolation for down hole intervals. Hence, to achieve this early strength, a certain amount of time is needed, which is called wait on cement (WOC). Since strength development is a key factor, accelerators are used to enhance the overall compressive strength without resulting in unwanted gelation.

As we mention above, Oil well cementing is a critical issue, and the improper cement job might put the oil production at risk. An example is the Gulf of Mexico oil spill that happened 20 April 2010, in the deep water horizon, and resulted in a tremendous loss of oil. Although the Mexico oil spill caused economic losses, it also resulted in big environmental hazards such as marine organism's damage due to toxic materials in oil. Billions of dollars from the oil industry have been spent in developing equipment and materials to help in reducing oil and gas loss and to improve oil production. On the other hand, it is still almost difficult to resolve every problem that might appear (Shadravan and Amani, 2012).

In the case of HPHT, the cement sheath might suffer some problems due to these severe conditions. For instance, after the cement is set and placed, strength retrogression problems might come up at the end of two weeks (Ogbonna and Iseghohi, 2009). The main reasons can be related to either the water in the cement sheath is lost to the formation, or the change in the cement structure. Calcium silicate hydrate is the main produced compound during the hardening process of the cement. When the well temperature is close to 250 ^{o}F, the compound calcium silicate hydrate is transformed into alpha-di calcium silicate which is a porous and weak structure compound that lead to the problem of strength retrogression. In addition, the occurrence of this porous compound in the produced cement heavily depends on the faced temperature (Ogbonna and Iseghohi, 2009).

In the last years, new developed chemical admixtures have been introduced to the cement to give the cement specific properties. Examples of these cement additives are retards, accelerators, strength enhancement materials, fluid loss, etc. which are added in an identified percentages to help in improving the early hardening as well as other properties of the cement (Nelson, 1990). Therefore, the effect of these cement additives heavily depends on the cement chemical and physical properties. The addition of these cement additives can affect the properties of normal Portland cement to achieve the general purposes of cementing. On the other hand, in the case of cementing underground holes, a special type of cement permitted by the API should be used so a successful cement job is achieved (API spec. 10, 2012). Thus, in the case of high pressure high temperatures, obtaining the best chemical additives that are mixed with the cement to produce a good well integrity cement to overcome these severe conditions still need more research.

Cement extenders are divided into three types, water extender, gas, and low density aggregates. Water is considered the most common and cheapest extender, where the more water added to the cement slurry, the greater the slurry yield and volume obtained, and for sure the more reduction in density is achieved. When water is added to the cement slurry, the cement particles must be in suspension to prevent the any settling of the cement. To do that, extenders like clays, or water viscosifying agents are added to the cement system to allow the extra addition of water so slurry extension can be accomplished. These extenders are known of forming a homogenous slurry, and stop the appearance of excessive water within the slurry. The second type of extender is gas, and an example of it is nitrogen or air. Low density cement is obtained by using gas without the need for adding more water the cement system (Samsuri et al., 2001). The third type of extenders is the low density aggregates which are defined as materials that has a density lower that Portland cement. These materials can be obtained from diatomaceous earth, volcanic ash, and fly ash. Thus, a noticeable reduction in the cement density is observed, when big quantities of extenders are present within the cement system (Nelson, 1990). Despite the benefits of adding these extenders to the cement mix, there are some drawbacks associated with the use of the above extenders. The main disadvantage of using extenders is the reduction in the final compressive strength, and the increase in the permeability of the harden cement sheath. In fact, bentonite, microspheres, and foamed cement decrease the density as well as the compressive when cured at 100 ^{o}F for one day (Samsuri et al., 2001).

Although clays are considered one well known minerals spread wide, defining them precisely is a difficult job. Clays are said to be a colloidal type material or close to colloidal particle size, in which it consists basically of hydrous aluminium silicates. Bentonite is naturally occurring clay in which montmorillonite mineral forms the biggest part of its mineral composition. Through industry, bentonite is considered as the most frequent and common used extender during cementing operation. In addition, bentonite has an expanding lattice which allows water molecules to incorporate and combine in and around its structure. This extending property can be enhanced by first mix bentonite with water and leave it to hydrated before mixing it with the cement (Dowell Schlumberger, 1989).

Bentonite can be classified depending on the domain element, such as sodium, potassium, and calcium bentonite. Saudi bentonite is a calcium based type which contains big percentages of silica and lesser of sodium compared with commercial bentonite. It works as fluid loss materials and can perform well when added to cement. In addition, bentonite offers the cement thixotropic property in which gels as soon as shearing stopped and form high gel strength that plug the formation.

Saudi bentonite was found in the Khulays area, 70-km north Jeddah, close to the Makkah Madinah road. Khulays bentonite deposit has reserves ranging from 420 thousand tonnes (proven) to 28.9 million tonnes (indicated) and 38.9 million tonnes (possible). Al-Homadhi showed that bentonite can be enhanced with the addition of certain materials and can perform well compare with commercial bentonite (Al-Homadhi, 2007). Samples were brought from Jeddah and then grinding into 75 micron particle size and prepared for testing.

Nano materials have been used in several fields such as catalysis, polymers, electronics, and biomedical, due to their superior where they have smaller size and high surface area. Also, we have used Nano-alumina in constructions to develop high early strength paste. In other words, it helps in improving the final compressive strength and reduces the fluid loss. For example, mechanical properties of mortar and concrete composites that prepared by mixing of cement and aggregate are affected by the properties and the arrangement of each component in it. Introducing Nano materials into a matrix to enhance the mechanical properties appeared as a promising area. Unlike polymers which have a dense structural matrix, cement is quite different since the area of cement matrix compounds has a relative loose structure. Nano sized air voids are existing between the cement and aggregates and these voids may have a significant effect on the Nano composite mechanical properties because their presence give a room for inserting Nano materials into the cement matrix (Li et al., 2006).

From the literature we found that addition of Nano materials resulted in enhancement in cement mechanical properties through the construction industry application. For instance, Zhenhua (2006) investigated the effect of adding Nano-alumina in the cement mechanical properties such as elastic modulus and compressive strength (Li et al., 2006). Campillo also proved that the addition of Nano alumina in belite cement caused enhancement in the compressive strength of the produced cement (Campillo et al., 2007). In addition, Santra (2012) described the effect of certain Nano-materials in oil well cement hydration and mechanical properties. He found that mixing small-size Nano-silica with the cement mixture resulted in an improvement in concrete compressive strength compared with the base mix (Santra, Boul, and Pang, 2012).

It was proven that Saudi bentonite is available in commercial stocks and has been tested for drilling fluid application (Al-Homadhi, 2007) (Musaab, 2014). However, usage of Saudi bentonite as a cement additive has not been implemented till now. Likewise, Singh explained that using nanotechnology helped in solving completion problems (Singh, Ahmed, and Growcock, 2010). Also Mobeen (2013) proved that adding Nano clay to the cement improved the mechanical properties of the cement in conditions of HPHT (Mobeen, 2013). Up to date, no report has been provided discussing the use of untreated Saudi bentonite, or untreated Saudi bentonite admixed with Nano clay as well as treated (upgraded) Saudi bentonite with other additives on the cement properties, and whether or not there is improvement resulted from this addition.

The ultimate object of this work is to first see the effect of untreated Saudi bentonite, and then untreated Saudi bentonite with Nano clay on the cement mechanical properties. Also, the effect of untreated Saudi bentonite, and untreated Saudi bentonite with Nano clay on the cement performance under high pressure and high temperature is reported. Finally, a comparison between upgraded (treated) Saudi bentonite and commercial bentonite for the low cement density of 101 PCF under high pressure and high temperature is reported.

Specific well in Saudi Arabia has been selected for this study, and the effect of untreated Saudi bentonite, Nano clay, and the treated Saudi bentonite on the cement properties is reported to come up with the optimum cement suitable for the well.

1.2 Motivation

The petroleum industry has been increasingly expanded among most of the world. 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 researches 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, Stiles, and Morgan, 2003) (Al-Yami et al., 2006).

Saudi bentonite is available in huge commercial stocks in the Khulays area in Saudi Arabia. Al-Homadhi investigated the use Saudi bentonite as drilling fluid (Al-Homadhi, 2007). Musaab treated Saudi bentonite and investigated using it as drilling fluid too (Musaab, 2014). From the literature review, we found that Saudi local bentonite has not been investigated for application in oil/gas well cementing. So, investigating of Saudi bentonite with cement may open a new door to cheap additives of cement.

Nano materials have proven their effectiveness in most of life fields starting with textiles, defense and up to aerospace and energy. For example, Nano silica (Senff et al., 2009), and Nano Alumina (Li et al., 2006) improved the cement mechanical properties in the construction industry. Addition of Nano clay might provide us with a better understanding of the fluid, and rock chemical behavior, reducing the fluid loss, and improve the well bore stability as well as drilling efficiency (Zhang Xu, and Yan, 2008).

From the previous work we conclude that using Saudi bentonite with the cement has not been investigated for oil well cementing application. Also the combine effect of Saudi bentonite, and Nano clay for application in oil well cementing at HPHT condition has not been looked at up to now. As a result of this, more investigating in this area may open a new door for a cheaper and more effected material which can help in solving many cementing problems.

1.3 Knowledge Gap

From the literature review, it was found that Saudi local bentonite has not been deeply investigated. Work done by Al-Homadhi had been just an enhancement to the Saudi bentonite for using it as drilling fluid and comparing its performance with the commercial bentonite (Al-Homadhi, 2007). Musaab also treated Saudi bentonite and investigated its usage as a drilling fluid (Musaab, 2014). Using of Saudi bentonite with the cement has not yet been looked at up to now. Preliminary experiment using untreated Saudi bentonite showed that bentonite does not swell too much compared with commercial bentonite. Investigating addition of untreated Saudi bentonite to the cement might open a new door to cheap additives of cement, since it Saudi bentonite is available in big quantities in the al Khulays area. Investigating other cement properties can clarify if this Saudi bentonite can provide any how improvement to the cement properties.

Also the treated Saudi bentonite developed by Musaab (2014) showing good swelling and dispersion, and some preliminary tests showed that this treated bentonite exposed similar properties to that exerted by commercial bentonite (Musaab, 2014).

Nanotechnology is considered an interesting and superior area of research due to enhancement achieved when added to the materials. Applications of Nano-materials resulted in enhancement to the cement composites, especially in improving mechanical, and chemical properties of the prepared Nano composites (Tjong, 2006). High performance materials are gained when nanomaterial used for construction application. Nano silica (Senff et al., 2009) and Nano Alumina (Li et al., 2006) improved the cement mechanical properties in the construction industry. Furthermore, (Mobeen, 2013) proved that the addition of Nano clay to the cement improved its mechanical and rheological properties under high pressure high temperature conditions.

The use of nanomaterial in oil well cementing at HPHT conditions was investigated in MS Thesis work at KFUPM. Nano-silica was investigated by Sami, 2012. Nano-clay was investigated by Mobeen, 2013. In both, significant improvement in mechanical and rheological properties has been reported.

From the previous work we conclude that the addition of Nano clay with cement has improved the properties of the cement. Using this Nano clay admixed with bentonite will be something new and potential area of work. Nano clay can help in giving more strength to the cement and may reduce the fluid loss, which is preferred when we face a loss zones.

As the cement slurry is pumped down hole, properties such as free water, fluid loss, rheology, thickening time, and strength development with time are considered as critical as compressive strength development, placement, and setting of the cement slurry.

This research will investigate the effect of untreated Saudi bentonite and its combination with Nano clay on the cement properties such as density, fluid loss, thickening time, and rheology etc. under HPHT condition. Also treated Saudi bentonite developed at KFUPM (Musaab, 2014) will be tested and compared with the commercial bentonite.

1.4 Problem Statement

From the literature review, we found that Saudi local bentonite has not been investigated for application in oil/gas well cementing. So, investigating of Saudi bentonite with cement may open a new door to cheap additives of cement.

From the previous work we conclude that the addition of Nano clay with cement has improved the properties of the cement. Using this Nano clay admixed with bentonite will be something new and potential area of work. Nano clay can help in giving more strength to the cement and may reduce the fluid loss and thickening time, which is preferred when we face a loss zones.

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 cement are as critical as high compressive strength developed after set. This research will investigate the effect of first untreated Saudi bentonite and then its combination with Nano clay on the cement properties such as density, fluid loss, thickening time, rheology, etc. under HPHT condition. Also treated Saudi bentonite will be tested and compared with the commercial bentonite in oil and gas well cementing in Saudi Arabia.

1.5 Objective

The ultimate objective of this investigation is to:

1. To study the effect of untreated Saudi bentonite admixed with Saudi cement class G oil well cementing under HPHT Conditions.
2. To study the influence of Nano-clay on the cement slurry composed of Saudi cement class G and untreated Saudi bentonite for the optimum mix.
3. To compare the performance of cement slurry with 1.9% treated Saudi bentonite and 1.9% commercial bentonite for cement slurry with a density of 101 PCF.

1.6 Research Methodology

To study the effect of untreated Saudi bentonite, untreated Saudi bentonite with Nano clay, and treated Saudi bentonite on the properties Saudi cement class G at HPHT conditions.

1. The Saudi local bentonite will be added at 1, 2, and 3% bwoc to the cement mixture and the effect of it on cement properties will be documented.
2. Nano-clay will be added at a percentage of 0.5, 1, and 1.5% to the optimum cement mixture with Saudi bentonite above, and its effect on the properties will be reported.
3. A Comparison of the performance of cement slurry with 1.9% treated Saudi bentonite and 1.9% commercial bentonite for cement slurry to produce a cement with a density of 101 PCF.

The following tests would be conducted in the experimental program to achieve our objective:

a. Thickening time cement test.
b. Fluid loss test.
c. Free water separation test.
d. Density.
e. Rheology test.
f. Compressive strength.
    - By "crushing method".
    - By "sonic wave method".
g. Microstructure analysis:
    - XRD
    - SEM

1.7 Thesis Organization

This research is organized with respect to the rules identified by the Graduate Studies at King Fahd University of Petroleum and Minerals. The thesis is divided into five chapters as follows:

Chapter 2: this chapter 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 survey, 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 during conducting the cement experiments. In this chapter there are several cement tests that explained in details in which each test is used to address certain cement properties.

Chapter 4: shows all the experimental results for all the cement tests with a comprehensive analysis and explanation for them.

Chapter 5: gives the summary of this work associated with conclusions depending on the applied experimental program. At the end a few recommendations are presented for the further work.

CHAPTER 2

LITERATURE REVIEW

2.1 Cement Systems and Applications

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 in which after a certain time it sets, and hardens resulting in bonding of other materials together. A cement system used in oil well cementing, is called a cement slurry design, 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 during the end of 1920s, with the main objective of supporting the casing and later prevent shocks when resumed drilling deeply 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 abandoning solution when shutting down well is needed (Bourgoyne et al., 1986).

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 kit, 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 frailer, 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, pumpility, 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. During this chapter, the basic principles in oil well cementing will be 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 of compressive strength, and sufficient zonal isolation throughout the life of the wells is obtained.

2.2 Cementing Principles

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 sending 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 systems are pumped into deep wells, where depths might exceed 20000 ft. In these deep wells, the temperature might increase up to 205 ^{o}C, 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 performed on the top of cement plugs using an aqueous liquid which is used push any cement remain inside the casing. There are two types of cement plugs used in oil well cementing, at the top and the bottom. 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 a much higher than the production zones, which resulted in reduction of undesirable fluids, corrosion of the casing, and prevent fresh water zones (Calvert, 2006). 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. **Figure 2-1** shows an example of a cemented well.

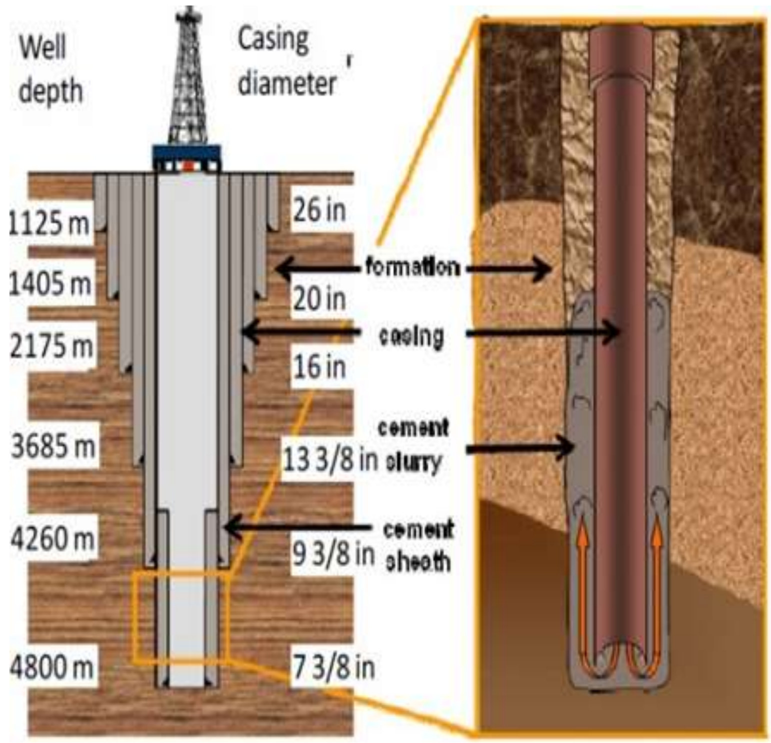

Figure 2-1 Casing design of cemented well (Plank, 2011)

2.3 Classification of Oil Well Cements

Oil well cement slurries are normally prepared from cement clinker or hydraulic mixed cements, in which these materials are considered 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. 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 changeable 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, 1987). 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), moderate and high sulphate resistant (MSR, HSR) as well as ordinary (O). 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 (Nelson, 1990, API Specification 10A, 2002; Shahriar, 2011)

Cement class	Recommended w/c, %	Recommended rage of depth, ft	Availability	Cost	Other features
A	46	0 to 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 to 6000	HSR''' and MSR'' classes, close to that of ASTM C 150, Category II	Lower cost	1. Used when moderate or high sulphate resistance needed. 2. C_3A content is lower compared to A cement Class.
C	56	0 to 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 C_3S. 3. High surface area.
D	38	6000 to 10000	HSR*** and MSR** classes		1. Used in conditions of moderate to high temperatures and pressure 2. Decreasing the amount of C_3S and C_3A cause cement retardation. 3. Cause a rise in the cement grain particle size.
E	38	10000 to 14000			1. Needed in high pressure temperatures wells. 2. Decreasing the amount of C_3S and C_3A cause cement retardation. 3. Cause a rise in the cement grain particle size.
F	38	10000 to 16000			1. Used in extremely high pressure and temperature conditions 2. Decreasing the amount of C_3S and C_3A cause cement retardation. 3. Cause a rise in the cement grain particle size.
G	44	0 to 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 ºF.
H	38	0 to 8000		--	1. Normal used oil well cement. 2. The outside areas are coarser comparing with Class G cement. . Cement thickening time is controlled by using additives and aimed to stop the loss of circulation till 450º F.

Cement is grinded into a very fine powder depending on the conditions needed. As a matter of fact, a cement which is too fine grinded such as micro-fine and also ultra-fine cements (where the blain surface is lower 9000 square cementer per gram) is not recommend for using in primary oil well cementing operation. The reason for this limited application is that the compressive strength developed from these cements is insufficient to hold the casing under bottom hole conditions, and also the inappropriate sulphate resistance acquired by these types of cements. On the other hand, during the oil well repairing operation which is called remedial jobs, using of micro-fine cements can be considered the best choice due to their small particle size, which give the cement the advantage to penetrate through minor cracks compared with ordinary oil well cements (Kumar et al., 2002).

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** ASTM cement classification, and their application parameters. Cement class type I and II are mostly used cements in the United States where its consumption percentage can reach 92% for the whole Portland cement used (Mobeen, 2013).

Table 2-2 ASTM cement classification

ASTM Cement Types	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 Additives Used in Oil Well Cements

Well cementing is a critical issue and is totally different from the conventional cementing jobs. In fact, the oil well cementing should be designed in a way the produced cement sheath must have certain properties such as expectable set time, low viscosity and free water, fluid loss control, sufficient compressive strength, adequate sulphate resistance in addition to long-term durability. Generally, oil well cementing slurry is essentially requires a cement which has a lower viscosity, so the process of pumping the cement into the deeper zones can be easily achieved without any problems. Also deep wells are known of both high pressure and temperature, so the setting behavior of the cement should be adjusted in a way it can sustain those harsh environments. Furthermore, these cement systems need to be designed in a way that sufficient physical properties associated with no compatibility problems encountered with the geological formation under these conditions of high pressure and temperature. On the other hand, there are other cases where the produced cement needs to be adapted with the corrosive formation fluids, weak formations, porous, and high pressurized zones. This problem has been solved by using cement additives which are nowadays available, and aimed to improve the properties of the produced cement slurry such as rapid compressive strength improvement, and excellent zonal isolation through the life time of the cemented well. Cement additive performance has heavily depended on oil well cement parameters, such as the chemical composition of the cement, particle size, phase hydration activity, silicate and aluminate phase distribution, gypsum/hemihydrate ratio, free alkali content, total sulphate content, and the specific surface area of the primary hydration outcomes. In addition, there are other parameter that has a big effect on the behavior of the oil well cement such as pressure, temperature, blender or mixing equipment, mixture quantity, mixing steps, and the used water to cement ratio (Nelson et al., 1990, 2006).

2.5 Types of Additives Applied in Oil Well Cements

There are eight types of cement additives added to the cement to improve the cement performance so a successful cement job is obtained. 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 light-weight systems to 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 gives us 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).

2.5.1 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.

2.5.2 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.

2.5.3 Lost Circulation Control Agents

Lost circulation is a critical issue in which more attention should 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 solves 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.

2.5.4 Free Water Control Agents

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.

2.5.5 Defoaming Agents

This problem of air entrapped is normally faced during the mixing of the cement. This entrapped air might cause a few problems such as pump frailer resulted from the damage caused by the air, unreliable or wrong cement density as well as a high porous produced cement sheath. Defoaming 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.

2.5.6 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.

2.5.7 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 exited between the cement particles, and resulted in reduction the used 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.

2.5.8 Expansion 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.

2.5.9 High Temperature Agents

In the case 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 up to 40 percent to maintain compressive strength and prolong life time of the well.

2.6 Cementing Design Process

Millions of dollars are usually spent in the drilling and completion of oil wells, 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, and this is usually followed with lab tests to evaluate the properties of the cement.

In fact, Ravi and Xenakis, (2007) explained the steps needed in the case of designing the cement (see **Figure 2-2**) (Ravi et al., 2007). 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 us 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 (step three) 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.

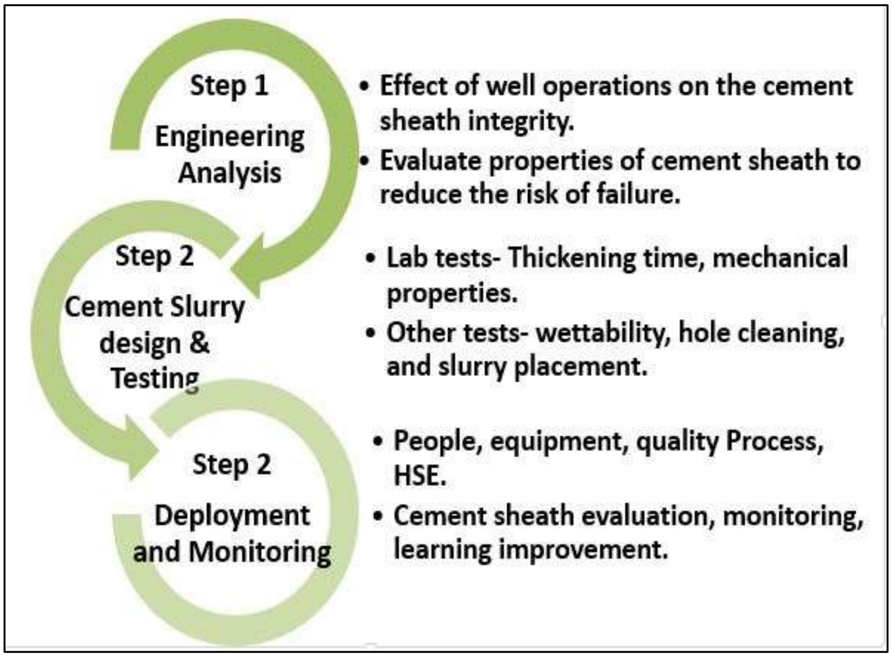

Figure 2-2 The basic three steps in designing the cement slurry (Ravi et al., 2007)

2.7 Density of Oil Well Cement Slurries

When cement is mixed with water, this mixture is called neat cement, and its density might range from 110 lb/ft^3 (14.7 Ib/gal) to123 lb/ft^3 (16.4 Ib/gal) depending on the used percentage of water to the cement, and also it depends on the cement class used. In both high pressure and temperature wells, it is a necessary for cement slurry to have a higher cement density to achieve excellent controlling of well fluids under these conditions. It is also advisable to raise the cement density since it assists in reducing the diffusion and helps in the removal of dense drilling fluids remained in the wellbore. Sometimes, materials such as bentonite or organic gums are added to the cement mix in order to help with stopping separation of the dense constituents occurred within the oil well cement (Ramachandran, 1984).

Extenders and weighting agents are the main cement additives used to alter cement density, so the required cement density needed for a certain job is achieved. As a matter of fact, extenders are materials with a low specific gravity, and are applied to decrease the density of the produced cement as well as to raise cement slurry yield, where cement weighting agents are added to increase the cement weight, so a more dense cement is achieved. Barite, sand, and micro sand are examples of weighting materials used as additives in both oil well cementing, and drilling mud, and aimed to increase the density since they are a smoothly distributing solid materials with a relatively high specific gravity. During oil well cementing, barite the most commonly used materials as weighting additive with a particle size distribution ranged between 3 to 74 microns (Ariffin, 2009; Barite, 2009), and a minimum specific gravity of 262 lb/ft^3 (35 Ib/gal) (Oilfield Glossary, 2009). Hematite, carbonate, tetra-oxide, calcium, siderite, manganese, ilmenite are also other kinds of weighting additives (Nelson et al., 2006), and barite and hematite are considered a related API/ISO standards (Oilfield Glossary, 2009). **Table 2-3,** and show the regular weighting agent's properties as well as extenders that are used in oil well cementing (Nelson et al., 2006; Oilfield Glossary, 2009; Saasen and Log, 1996). From the literature, Barite was documented as weighting agent known for its lower efficiency compared with other materials such as manganese tetra-oxide, hematite or ilmentite (Nelson et al., 1990, 2006).

Table 2-3 Regular weighting agent's properties which are used in oil well cementing (Nelson et al., 2006; Oilfield Glossary, 2009; Saasen and Log, 1996).

Weighting material	Density g/cm^3	Additional water requirement (Liter/kg)	Highest cement density gm/cm^3
Barite	4.33	0.20	2.28
Hematite	5.05	0.019	2.64
Ilmenite	4.45	0.00	Less than 2.40
Manganese tetra-oxide	4.84	---	2.64
Siderite	3.80	---	

Table 2-4 Properties of extenders used in in oil well cementing (Nelson et al., 1990, 2006)

Extender	Obtained density (lb/gal)	Other features
Bentonite (clay extenders)	11.5 up to 15	1. Lower resistant to sulphate as well as corrosive fluids due to the rapid increase in permeability of the produced harden cement. 2. Assists fluid loss control.
Fly ash	13.1 up to 14.1	Protect the cement against corrosive fluids.
Sodium Silicates	11.1 up to 14.5	1. Lower percentages are needed. 2. Excellent choice when mixed with sea water. 3. Appropriate viscosity is obtained helped in using large amounts of water without excessive free water separation
Microspheres	8.5-15.0	1. Better compressive strength values are obtained. 2. Lower permeability. 3. Good isolating properties associated with thermal stability.
Silica Fume	≥11	1. Low density cement systems could be obtained. 2. Enhances fluid loss control. 3. Higher rates of compressive strength growth are achieved.
Foamed Cement	6.0 up to 15.0	Lower the permeability and enhances the strength

Saudi bentonite is available in huge commercial amounts in Khulays area, 70-km north Jeddah, and close to the Makkah Madinah road. It is a calcium base bentonite and with the poor swelling property. Adding this untreated Saudi bentonite to the cement mix may open a new door for cheap cement weighting agent.

Nanomaterial proven their superior in construction application. Also nanomaterial such as Nano-clay or Nano-silica are used to design a light weight cement since they reduce the cement density.

2.8 Cement Hydration

The setting and hardening behavior of oil well cement might be related to the continuing series of chemical reactions that take place between both the constituents of the cement and the added water. Vlachou, 1997 investigated the chemical and physical changes on cement class G from the start of adding cement until the cement starts to set and solidify. SEM and XRD tests were conducted on the cement slurry, and their results showed that the structure and the appearance of the final hydration outcomes were depended on the experimental conditions. Examples of these experimental conditions are the hydration time at the start of the mixing process, stirring conditions (Vlachou and Piau, 1997), temperature, chemical composition of cement as well as the used additive (Shahriar, 2011). Furthermore, constant viscosity in addition to sufficient fluidity are observed after several hours in the case when the cement slurry is hydrated under continuous stirring. The hydration particles are formed in a shape of small spheres of aluminate phases, and these produced spheres did not affect the flow and solidification of the cement. The reason for this behavior is that formed particles do not connect or bond with the particles present there, which led the particles to move more freely within the inter-particle spaces. After that, the cement starts to thicken so quickly, and the amount of the hydrated crystals is increased rapidly giving the sign that the setting process of the cement has been initiated. However, on the other experimental conditions (the rest), the hydrated slurries are showing a vast increase in the viscosity during the first hours, after that, this growth in the hydration process is slowed down (Vlachou and Piau, 1997). Also under these conditions, the appearance of aluminate hydrated crystals of colloidal shape was also observed due to the excessive increase in ion saturations in the grain region. (Vlachou and Piau, 1997). After that, the grain surface is covered by the crystals and resulted in a slowdown of the hydration process. However, when the hydrated cement slurry is under stirring condition, the ions of the cement mix will be scattered all over the mixed cement, and dissolution will be continued until the whole mix is saturated. In fact, chemical composition development of the liquid cement phase is usually affected by the cement chemical composition as well as the additives used in the cement admix (Vidick et al., 1989; Michaux et al., 1989).

At temperatures higher than 110 ^oC, test results from XRD and SEM showed that main compounds in the neat cement slurry are converted from CSH (II), C_2SH_2, $C_3S_2H_3$ into the chemical compound C_2SH (dicalcium silicate hydrate), and the microstructure of the produced set cement were transformed into a three-dimensional fiber network which seals or complete block at various curing temperature. However, when silica sands were added to the cement, the compounds in the final harden cement were converted into $C_5S_6H_5$,

C_6S_6H when temperature is less than 150 ^oC, or converted into any type of calcium silicate hydrate such as $C_5S_5A_{0.5}H_{5.5}$, $C_3.2S_2H_{0.8}$ at high temperature. Also the microstructures were converted into fiber a fiber network, mass block structure a small -parallel-needle or coarse structure network (Zhang et al., 2008).

When additives are added to the cement mix, differential sensitivity is exhibited with various cement classes (Jupe et al., 2007; Vidick et al., 1989). Justnes explained that for the basic cement class G, where the water cement ratio is about 0.5, nearly 10% of the hydration process is needed for the cement to preserve its shape under atmospheric conditions (Justnes et al., 1995). Although, C_3S hydration changes during pumping time of the cement might be connected, it was observed that most of the crystals transformed through pumping of the cement as a function of temperature, which was appeared clearly in the temperature interval in both ettringite/monosulphate degrades, and crystalline hydro garnet were occurred as well (Jupe, 2005).

The produced crystals accumulated on the outside surface of the grains and resulted in a reduction of the hydration reaction. However, ions are scattered along the sample volume and disintegration resumes until the whole mixture is saturated especially in mixtures hydrated below stirring.

Portland cement consists generally from five main compounds as shows in **Table 2-5** and lower amounts of other compounds (De la Roij, Egyed, and Lips, 2012).

Table 2-5 Chemical composition present in Portland cement and the weight (De la Roij, Egyed, and Lips, 2012)

Components	Symbol	Weight %	Chemical formula
Tricalcium silicate	C_3S	50	$3Cao.Sio_2$ or Ca_3SiO_5
Dicalcium silicate	C_2S	25	$2Cao.Sio_2$ or Ca_2SiO_4
Tetracalcium aluminoferrite	C_4AF	10	$3CaO.Al_2O_3$ or $Ca_3Al_2O_6.Al_2O_3$
Tricalcium aluminate	C_3A	10	$4CaO.Al_2O_3.Fe_2O_3$ or $Ca_4.Al_2Fe_2O_{10}$
Gypsum	--	5	$CaSo_4.2H_2o$

2.9 High Pressure High Temperature Cement Slurry Properties

The cement slurry used in severe conditions of both high pressure and high temperature must contain certain properties. The API presented some limitation in the proposed cement used to cement these well. **Table 2-6** summarized the important parameters for HPHT wells.

Table 2-6 Important parameters for HPHT wells

Property		limit
Fluid Loss	Casing Across Productive or Medium Permeability Zones	< 50 ml/30 min
	Liner	< 50 ml/30 min
	Squeeze	50-100 ml/30 min
	Test Temperature - All above Applications	BHCT
	Gas Prevention	< 50 ml/30 min
	Test Temperature – Gas Prevention Jobs	BHST
Strength Retrogression	BHST or Producing Fluids above 230°F (110°C), whichever is less	Minimum 35% BWOC Silica Flour (For Normal Viscosity Slurries)
		Minimum 35% BWOC Silica Sand (To Reduce Viscosity)
Thickening Time	Readings At	POD, 40Bc, 70Bc, 100Bc
Compressive Strength	Drillout/ Pressure Test	>500 psi
	TOL	>500 psi
	KOP	>3000 psi
Settling Test (BP)	Density Difference - Top to Bottom / Shrinkage	0.05 g/cm3 (0.4 ppg) / <5 mm
Free Water	Normal/Low Porosity Slurries	0 ml/250 ml
	Test Cell Angle - Vertical Wells (<20°)	0°
	Test Cell Angle - Deviated Wells (>20°)	45°

2.10 Use of Bentonite with Cement

During the cementing and the development process, extensive work has been done on many proposed cement systems in order to come up with a better cement system suitable for cementing different wells without future problems. The main function of oil well cement is first to provide good bonding of the cement to the wellbore as well as preventing and supporting the casing from collapse. Also to stop fluid migration between zones, reduced the annular space occurred outside casing, and the induced vags appeared in the formation. In addition, the cement is also used as a plug to stop losing circulation or to seal an abandoned portion in the well (Bourgoyne et al., 1986).

Morgan et al., (1951) discussed the need for a low strength cement composition which can be used in oil well cementing. Results from laboratory and field experience showed that modified cement gave control of the final tensile strength of around 200 psi. The modified cement can be prepared by either high early strength or regular Portland cement mixed admixed with bentonite and appropriate agent used for dispersing and adjusting the setting time of the slurry. Furthermore, laboratory-scale test results also proved that modified cement had good pumpability and permeability properties which satisfactory oil well cementing. Moreover, modified cement was easier to be penetrated by bullets, especially in thick cement and stop loss of seal due to shattering of cement. What's more, low density of modified cement was more beneficial in solving lost circulation problems, reducing the time and expense needed for the stage cementing operation. In the end, low cost of the cement gave it an economic advantage compared with the conventional cement (Morgan et al.,1951).

Morgan and Dumbauld (1953) showed that cements which contain 8 to 12 percent of bentonite has performed successfully on several thousand wells where temperature ranged from 100 to 250 ^{o}F. Good placement and performance of cements containing bentonite was due to using calcium lignosulfonate in concentrations from 0 to 0.75 percent aimed to control setting properties of the cement. Produced cement had a low density, low water loss, and low set strength of around 200 to 250 psi tensile. In addition, using bentonite with cement resulted in savings of nearly 15 to 25 % compared with jobs done without bentonite. Furthermore, extra savings can be gained because of reduction in time and shortage of stage jobs due to using of light weight slurries which prospered of cement and bentonite. Moreover, success was observed because of these low set strength of cements gave good penetration by perforators and in stopping loss of seal due to shattering of the cement upon perforation (Morgan and Dumbauld 1953).

George et al., (1959) discussed the preparation of cement with the colloidal clays such as bentonite. They said that premixing of cement and clay results in uniform slurries, whereas mixing of clay and cement separately produces a non-uniform cement slurries. A retarder such as sodium, lithium and potassium preferred in a range from 0.75 to about 4 % to delay the swelling of the clay. On the other hand, if a small amount of clays used, smaller percentages lower than 4 are needed to maintain fluidity. At the start, sodium is dissolved in water, then followed by calcium or equivalent lignosulfonate salt used between 0.1 and 1 %, bwoc added to mix. The objective is to raise plasticity or workability of the wet cement by delaying the cement setting time. Then clay is added in the aqueous and followed with the cement. **Table 2-7** shows the amounts used for one liter of cement with 0.5 % calcium lignosulfonate (Lunsford, 1959).

Table 2-7 Amount used for one liter of cement with 0.5 % calcium lignosulfonate (Lunsford, 1959).

Test	Bentonite (%) (gram)	Portland cement	Water ml	Density (lb/gal.)
Blank	(4%)(43)	1077	609	14.6
Blank	(12%)(98)	815	675	13. 4
Blank	(25%)(137)	547	763	12.0

Harrison and Blount (1986) proved that a use of a non-rig Coiled Tubing Unit (CTU) requires no need to kill the well when a squeeze cement technique is implemented. In-situ contamination of cement remains is used when the squeeze cement job stopped. After the squeezed cement had set, the contaminated cement was circulated or inverted out of the well bore, so that drilling was no longer needed. The advantages of this method were to block channels in the primary cement to either the aquifer or the gas cap, get rid of undesirable production perforations, and also to change injection profiles. In addition, work over (remedial work) had decreased by 85% in these wells. In the placement of the cement in the required level, nearly six fluids were prepared which include water, brine, bentonite gel, polymer, semi-solid polymer plug, and a mixture of brain with HEC. In the case of water, the cement did not start moving up unless the hole was full of cement. In the case of the bentonite, some of cement channels down among the bentonite gel with some bentonite contaminated with cement and moved up. The contamination systems were aimed to stop the cement from getting solidifies and keeps the solids for a minimum of seven days at 190 ^{o} F and atmospheric pressure (Harrison and Blount, 1986).

Sugama et al., (1987) studied the mechanism of in situ transformation of the drilling fluids that were used as a water based bentonite into a cementitious material used to stop losing circulation in wells where the temperature exceeds 300 °C. This was achieved by using a mixture contains a cement, a borax blend, and a fiberglass used as a linking material. In addition, the plugging mechanism of these materials depended on the fibre size and concentration used. In other words, if the bridging materials were not used, a large amount of cement could be lost to the formation before the cement sets and plug the fracture. Bridging materials could effectively decrease the flow and seal the fractures till the LCMs solidify (Sugama et al., 1987).

Nelson et al., (1990) discussed the use of bentonite with cement. He explained that bentonite can be used as an extender to reduce density and the percentage used can be increased up to 20 % bwoc. Bentonite can also be used with cement to develop a high gel strength and it exhibits thixotropic behaviour. Concentration of bentonite in cement ranged from 0.05 to 2% bwoc (Nelson, 1990).

Wilson et al., (1990) discussed the conversion of mud used as drilling into cement slurry. They confirmed that modern development in using copolymer technology enabled the introduction of Portland cement into the drilling fluid and at the same time sustained control over its rheology. New accelerator was also added to the cement which provided design flexibility and rise in strength development. In addition, converting drilling fluid into cement decreased the drilling cost because of reduction in disposal cost. It also enhanced annular displacement efficiency by adjusting the flocculation that occurred due to mud and cement contact. In addition, it also provided full utilization of manpower, tools, time and resources through enhancing services at rig sit. Moreover, the conversion mud to cement was environmentally safe, economic, and promising method which meets cementing requirements (Wilson, Carpenter, and Bradshaw, 1990).

Jones & Carpenter (1991) came up with a unique cementing system which consists of a combination of (latex and thixotropic) that enhances cement bonding as well as zonal isolation in wells where the bottom hole static temperature is less than 175 °F. Also, laboratory experiments and case histories showed that using the proposed system during well cementing can result in good primary and cause minimizing of the remedial cementing by lowering invasion through permeable unconsolidated zones as well as stop gas migration. Furthermore, this system provides excellent zonal isolation, decreases the filtration loss, accelerates the compressive strength growth, and reduces the wait on cement time (Jones and Carpenter, 1991).

Samsuri et al., (2001) have studied the effect of using Malaysian bentonite collected from Sabah when used in oil well cementing application, and then compared his results with that obtained using Wyoming bentonite. The Malaysian bentonite had a 75μm particle size and the results obtained from XRD, XRF, and methylene blue tests showed that this bentonite had a lower values cation exchange capacity. It also showed lower amount of montmorillonite mineral with other impurities such as quartz, muscovite, hematite, kaolinite, and illite. In addition, bentonite was also mixed with cement class G in the ranged between 2% and 16% BWOC so as to consider the free water and density performance. Unlike Wyoming bentonite, the more increase in bentonite concentration, more free water present in the cement mix. This was because the presence of the powerful calcium cation that could connect the structural plates of the montmorillonite together and resist water entry. Also the dry Malaysian bentonite processed samples showed a good control of the free water compared with wet ones. However, after treatment, free water was reduced as bentonite concentration increased. Furthermore, before treatment, addition of Malaysian bentonite increased the density unlike Wyoming bentonite. This was due to the extremely existence of hematite that indicated the presence of quartz the structural composition. This was enhanced after treatment, where the density reduced by the addition of the Malaysian bentonite (Samsuri, Junin, and Osman, 2001).

Heinold et al., (2002) investigated and analysed the effect of cement additives on the mechanical properties of a standard cement density. In their experiments, organic and non-organic materials were admixed with oil well cements at a water cement ratio fraction ranged from 0.5, to 0.66. After that, unconfined compressive strength as well as flexural strength tests was implemented on cement samples in order to observe and see the impact of these additives on mechanical properties of ordinary cement density. Finally, they reported that individual using of these cement additives did not result in any improvement in the cement flexural and tensile strength properties in the case of high density mix. In addition, these additives were known to enhance the tensile and flexural strength properties in standard cement density, but might not work well in both high density cement systems, and higher temperatures (Heinold, Dillenbeck, and Rogers, 2002).

Jason, and Barry (2003) started a research on abandoning a number of wells by using compressed sodium bentonite (Zonite) as a replacement for the cement. The objective of this work was to come up with a way to reduce the cost of plugging the wells. Pilot tests had conducted through 500 wells across the USA using this compressed bentonite and results showed nearly 100 wells had been marked as possible abandonment candidates on Barrow Island in Australia. A minimum differential of 750 psi was achieved when this Zonite plug was applied in wells. The exacted reservoir pressure was inaccurately known, but it might be considered around 1000 psi. Finally, the abandonment cost had been reduced by 50% of Barrow Island (Clark and Salsbury, 2003).

Jennings (2005) performed long term high temperature cement tests to evaluate the physical properties of cement placed in gas wells. He also tested the durability of this Saudi cement when used in oil well cementing. Three cement compositions consisted of normal density class G, bentonite extended, and optimized particle packing of a low weight cement system containing hollow ceramic spheres (HCS) were tested. Cement composition subjected to several experiments to measure the rheology, permeability, compressive strength, shrinkage, settling time, Poisson ratio, and Young modulus. Results showed that the compressive strength of HCS admix, reduced by 81% through 11.75 months at 300 ^{o}F and 3000 psi, and the shrinkage of the mix after initial setting was unacceptably high (see **Figure 2-3,** and **Figure 2-4**). Also, the permeability of the HCS was improved by 7.7 fold within one year, while Young’s Modulus was reduced by 81% for more than a year as shown in **Figure 2-5,** and **Figure 2-6** (Jennings, 2005).

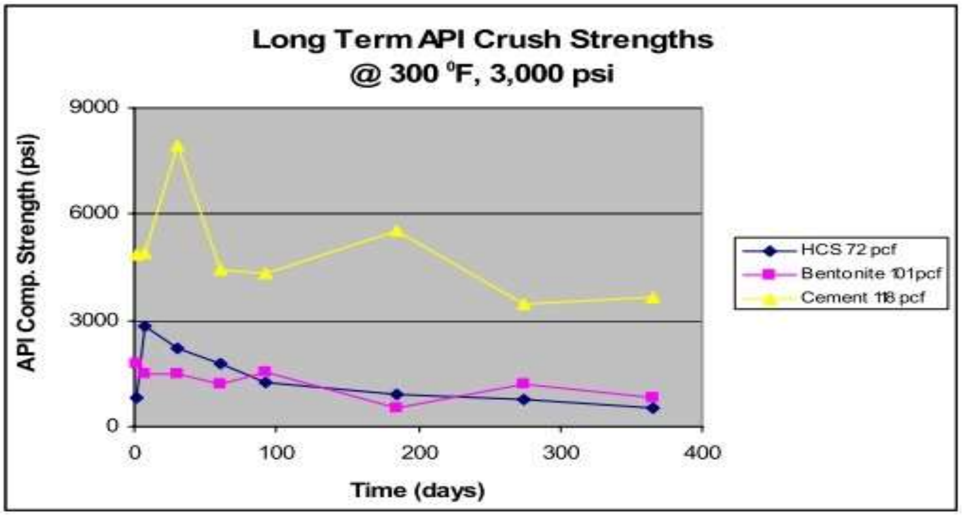

Figure 2-3 Compressive strengths by crushing of Saudi cement at 300 ºF and 3000 psi (Jennings, 2005)

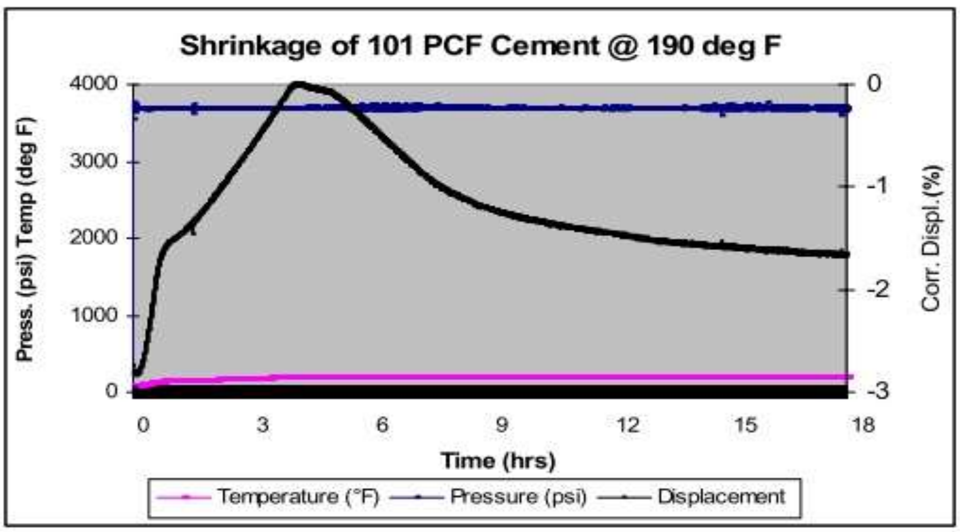

Figure 2-4 Shrinkage of 101 pcf cement of cement at 190 ºC (Jennings, 2005)

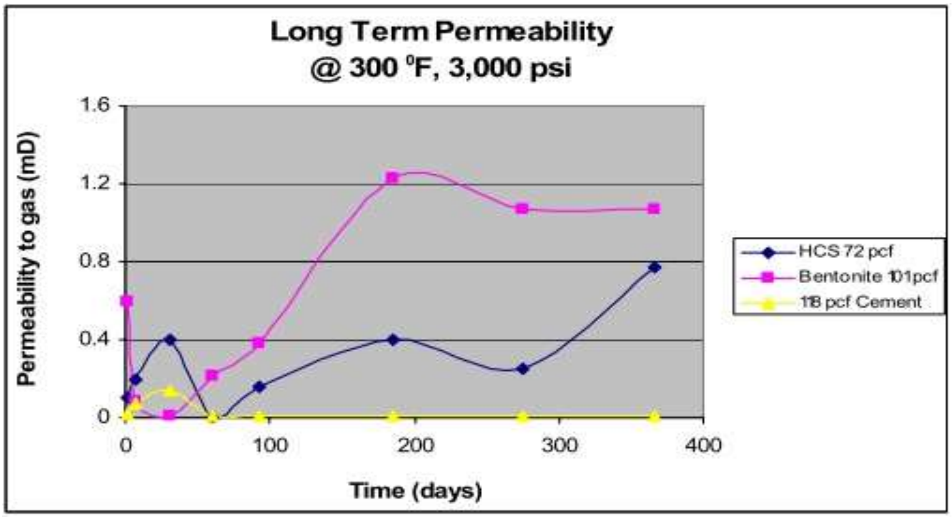

Figure 2-5 Permeability of cement at 300 ºF and 3000 psi (Jennings, 2005)

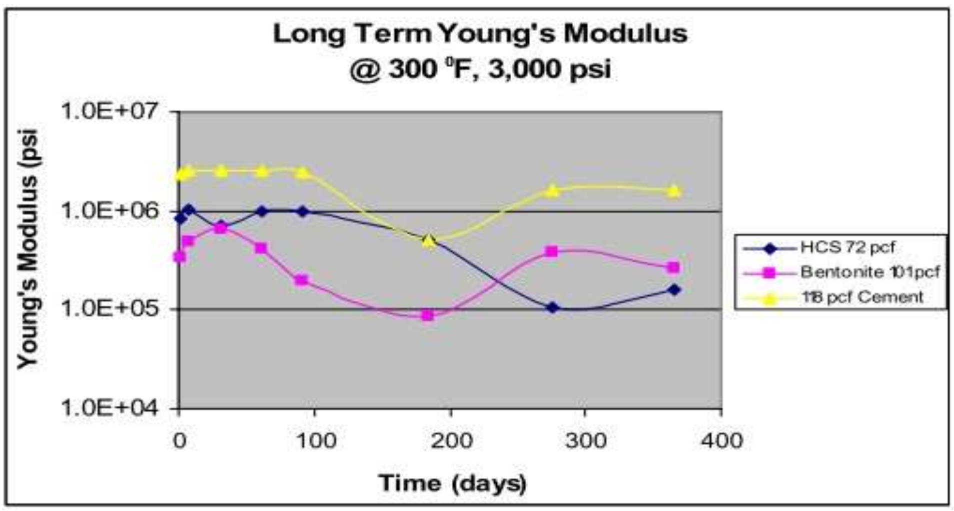

Figure 2-6 Young's Modulus of cement at 300 ºF and 3000 psi (Jennings 2005)

Fasesan et al., (2006) showed that the use of sodium Metasilicate when admixed in cement mixtures decreased the cost and improved the quality compared with bentonite. Mixtures of 50:50 Class C or H: Pozzalon with bentonite used in percentage of 2 have been performed well in the past time. The objective of using 2% bentonite in cement was to enhance the water cement ratio, and to decrease the density with no free water present in the produced cement. The decision to replace bentonite was due to two reasons: the presence of bentonite decreased the efficiency of a specified concentration of the most commercial existing fluid loss additive. The second reason was the cost of transfer this material over long time periods. Test results indicated that little amounts of sodium

Metasilicate 0.5% bwoc have performed well in replacing 2% bentonite. Benefits of the proposed system showed a reduction in the total fluid loss additives, a better improvement in fluid loss, and lower chances of wellbore water and formation water contamination. The slurry density of this system was in the accepted range and did not cause nor plugging or suspension problems. The economic savings of using cement system of class C and H when mixed to give a density of 14.2 lb/gal showed 9.5% differential savings compared with cement system free of SMS (Fasesan, Heinze, and Tesalonika, 2006).

Al-Yami et al., (2006) came up with new cement that works at a slow rate of penetration and aimed to help in drilling sidetrack wellbores. Experimental results showed that an increase in the cement density caused a slight decrease in the penetration rate for the side tracking drills. His results also showed a reduction in the rate of penetration as well as improvement in the compressive. Furthermore, this cement system could perform well from any of the present cement designs for side track drilling and also had a big chance to improve sidetrack angle builds up (Al-Yami et al., 2006).

Al-Homadhi (2007) explained that Saudi Arabia usage of clay bentonite alone can reach over 100 thousand tons a year and all of it is Wyoming bentonite from USA. He also discussed the availability of huge commercial clay bentonite stocks in Khulays area, 70 km north Jeddah, Saudi Arabia. He showed that local bentonite can be enhanced economically by adding some cheap materials to mud to enrich its viscosity and filtration loss such as Drispac polymer, and bentonite extenders. Local bentonite enhanced by adding a salt extender (Soda Ash) and Drispac polymer at a concentration of 5 %, 0.5 % respectively. In addition, the viscosity and the filtration loss of the 7% wt imported bentonite gave almost the same as the 8 % wt enhanced local bentonite mud. Finally, the cost of the improved local bentonite, which gave a yield higher than 90 bbl/ton, was 46% lower than Wyoming bentonite cost (Al-Homadhi, 2007).

Mussab (2014) investigated the using of local Saudi bentonite as a water base drilling fluid mud. Two phases of treatment has been used: purification, and thermal treatment. Results showed improvement in Saudi bentonite properties similar to the commercial oil well drilling fluid application. This behaviour proved that upgraded Saudi bentonite can be used as an alternative to the commercial bentonite in the oil well drilling fluid industry application (Mussab, 2014).

2.11 Use of Nano Clay with Cement

Zhenhua et al., (2006) discussed the effect of Nano-alumina on cement mechanical properties of the cement, for example, elastic modulus, and compressive strength. In their experiment, cylindrical samples (Φ20×40 mm) of cement mixed with various volume fractions of Nano-alumina were tested and cured for 3, 7, and 28 days. They found that adding Nano-alumina resulted in a rise in the elastic modulus and compressive strength of the finial produced cement sheath. Mixing 5 % of Nano-clay caused an increase in the elastic modulus by 143% at 28 days. Also, after 7 days, a rise of 30 % in the compressive strength was observed when 7% of Nano-alumina added to the cement mixture. This enhancement in the compressive strength, and elastic modulus of cement composite was due to an increase in the compactness of interfacial transition zone since it acts as an excellent fine aggregate that filled this zone and also work as porosity reducer. **Figure 2-7** illustrates the results of elastic modulus and compressive strength (Li et al., 2006).

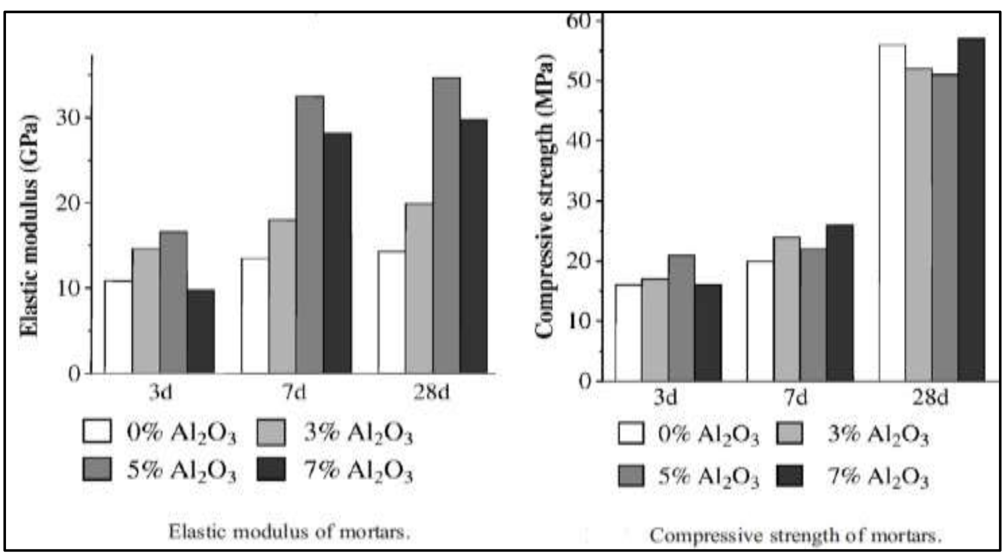

Figure 2-7 Results of elastic modulus and compressive strength (Li et al., 2006)

Campillo et al., (2007) investigated the effect of Nano-alumina in belite cement. The cement samples tested at an early age (7 days), the compressive strength grows remarkably when two kinds of Nano alumina were added to the cement. In addition, colloidal alumina performs well compared with agglomerated alumina, and also when the smaller alumina weight used, almost identical strength rise is reached. Furthermore, the Nano alumina can work as a reactive agent that increases the hydraulic activity of the slow reactive belite phase existed in cements. This caused an improvement in the microstructure of cement, and provided the development of the mechanical strength at early ages of the curing process (Campillo et al., 2007).

Senff et al., (2009) discussed and analyzed the effect of Nano-silica particles on the fresh state behavior when added to the cement with a percentage ranged between 0 and 2.5 by weight of cement. They conducted rheological experiments and their results displayed that after 75 minutes from the beginning of mixing, the grout containing 2.5 wt% Nano-silica showed inadequate flowability. Also admixing of Nano-silica with the cement resulted in an increase in both yield stress, and the plastic viscosity by 66.5, 3.6%, respectively. Moreover, the setting time and the moment to extend to the highest temperature was reduced by 60% and 51.3%, respectively, when compared with samples that used without Nano-silica. Finally, results from X-ray diffraction indicated that calcium hydroxide appeared after 9 hours in the cement samples containing 2.5 wt% Nano-silica. What is more, the addition of Nano-silica caused an increase in air content by 79%, and a reduction in the density by 2.4% (Senff et al., 2009).

Pourafshary et al., (2009) explained that nanotechnology provided answers to some of the upstream and downstream challenges the industry has encountered during the past few years. They did a full study of the uses of nanotechnology in the upstream oil industry, and decided to choose some of investment zones to be involved in the AHP survey. In addition, looking at the field of EOR from economic and technical points, investment in the enhancement of nanotechnology in chemical and water injection to be used in the EOR operation gained big attention from our pool of experts (Pourafshary et al., 2009).

Singh et al., (2010) presented a literature review showed that using nanotechnology played a big role in resolving drilling and completion issues. They also explained the introducing of nanotechnology in the oil/gas industry still needs to be totally explored (Singh, Ahmed, and Growcock, 2010).

Santra et al., (2012) described the effect of certain Nano-materials in oil well cement hydration and mechanical properties. They found that enhancement resulted from the addition of small-size Nano-silica to the cement mixture led to an increase in concrete strength compared with silica fume, even though the less amount is used than for silica fume. It was clear that adding Nano-materials to the cement mixture resulted in strong bonding of the cement paste aggregates. It also resulted in a rise in both tensile and compressive strength, reduced the permeability and caused an increase in resistance to calcium leaching and different types of chemical attack, and more uniform microstructure associated with reduction in pore size and volume. On the other hand, the difficulties with the equal dispersing of Nano-silica come up with its amount and the specific surface area in the case where less water to cementitious materials ratio is utilized (Santra, Boul, and Pang, 2012).

Sami (2012) investigated the effect of adding Nano silica on the Saudi cement class G under HPHT conditions. His results showed significant improvement in mechanical and rheological properties of the final cement sheath. The optimum percentage used was 1% Nano silica with significant improvement in the final produced cement sheath compare with cement base mix (control mix) (Sami, 2012).

Mobeen (2013) investigated the effect of adding Nano clay on the Saudi cement class G under HPHT conditions. His results showed a substantial enhancement in mechanical and rheological properties. The optimum percentage used was 1% Nano clay with significant improvement in the final produced cement sheath compare with cement base mix (control mix) (Mobeen, 2013).

CHAPTER 3

EXPERIMENTAL PROGRAM

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

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

The cement properties addressed in this study are:

a. Thickening time cement test.
b. Fluid loss test.
c. Free water separation.
d. Density.
e. Rheology.
f. Compressive strength
    - By “crushing method”.
    - By “sonic wave method”.
g. Microstructure analysis:
1 XRD
2 SEM

Therefore, the effect of Saudi bentonite on Portland cement will be investigated.

3.1 Well Specifications

Atypical well in Saudi Arabia is picked to see the effect of these materials on the cement. Cement system design with different percentages of Saudi bentonite 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 specification

Parameters	Values
Depth	14000 ft
Bottom hole pressure	8265 psi
Bottom hole static temperature (BHST)	290 ºF
Bottom hole circulating temperature (BHCT)	228 ºF
Surface pump pressure	1050 psi
Time to reach the bottom (TRB)	49 min
Mud weight (MW)	85 PCF

3.2 Cement System Consideration

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 untreated Saudi bentonite to the mix.

Table 3-2 Cement slurry design without Saudi bentonite

Cement Class G (powder) + 35% silica flour +1% dispersant + 0.2% fluid loss additives + 1% expanding agent + 0.5% fluid additives + 1% retarder + 0.25 gm defoamer.	
Expected slurry density	125 PCF
Water cement ratio	0.44
Slurry Yield	1.367 ft^3/sack
Expected thickening Time	4-5 hours

At the beginning, a series of cement tests will be conducted to the cement system design as explained in the experimental program without the addition of untreated Saudi bentonite. These cement test results will be considered as the base case or as a reference in all the following cement results. Next, the untreated Saudi bentonite will be added to the base mix cement design with the percentages of 1, 2, and 3% by weight of the cement and the results will be reported. **Table 3-3** illustrates the cement slurry design when untreated Saudi bentonite added.

Table 3-3 Cement slurry design with Saudi bentonite

Cement Class G (powder) + 35% silica flour + **X%** Saudi bentonite +1% dispersant + 0.2% fluid loss additives + 1% expanding agent + 0.5% fluid loss additives + 1% retarder + 0.25 gm Defoamer.	
Expected slurry density	unknown
Water cement ratio	0.44
Slurry Yield	unknown
Expected thickening Time	unknown

Where X in the table represents the percentages of Saudi bentonite 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 (Mobeen, 2013)

Component	Percent %
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
C_3A	<1
C_3S	62
C_2S	15
$C_4AF + 2C_3A$	16

Table 3-5 Cement additives and the percentages used in the proposed system

Additives	Function	% BWOC
SSA-1	High temperature strength stabilizer	35
MBHT	Extender	1
HR-12	Retarder	1
CFR-3	Friction reducer	1
Halad-344	Fluid loss additives	0.2
Halad-413	Fluid loss additives	0.5
DA-3000	Anti-foaming agent	0.25/10bbl

3.4 Saudi Bentonite Properties

In general, bentonite is a clay type material, and it is defined as a natural, earthy material which develops plasticity when mixed with a limited amount of water. In addition, clay fraction known with the term of particle size, as size fraction composed of the smallest particles. Clay is composed essentially of silica, alumina, smaller amount of iron, magnesium, sodium and potassium in addition of water. The composition of untreated Saudi bentonite used in this study is shown in **Table 3-6**.

Table 3-6 Composition of Saudi bentonite by XRF

Elements	Concentration
Si	72.9903
Na	2.3406
Fe	15.7427
Mg	2.0248
Ca	2.1638
Ti	1.9577
K	2.3365
Zn	0.1142

In addition, XRD test was also conducted on the samples of the untreated Saudi bentonite, and the results showed that the phases were quite similar to that of commercial bentonite as shown in **Figure 3-1**.

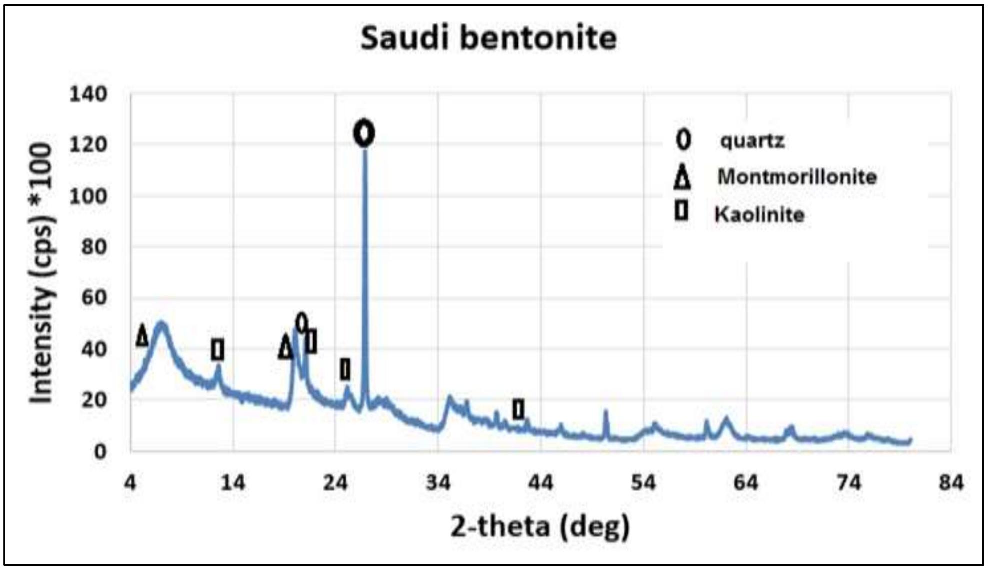

Figure 3-1 Composition of Saudi bentonite by XRD

It is clear that local untreated Saudi bentonite contain higher percentages of quartz as showed in XRD spectrum and lower of Kaolinite compared with that of commercial bentonite. SEM analysis tests were also conducted on the samples of untreated Saudi bentonite and the images as well as elements produced from this test are shown in **Figure 3-2**, and **Figure 3-3**. From the images we confirm the presence of higher percentages of silicon in the tested samples.

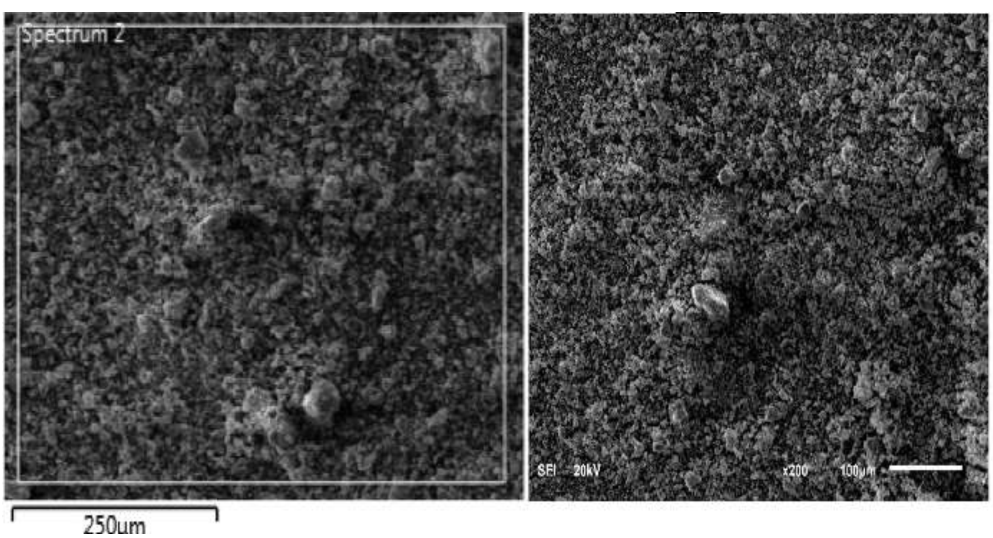

Figure 3-2 Saudi bentonite image by SEM test

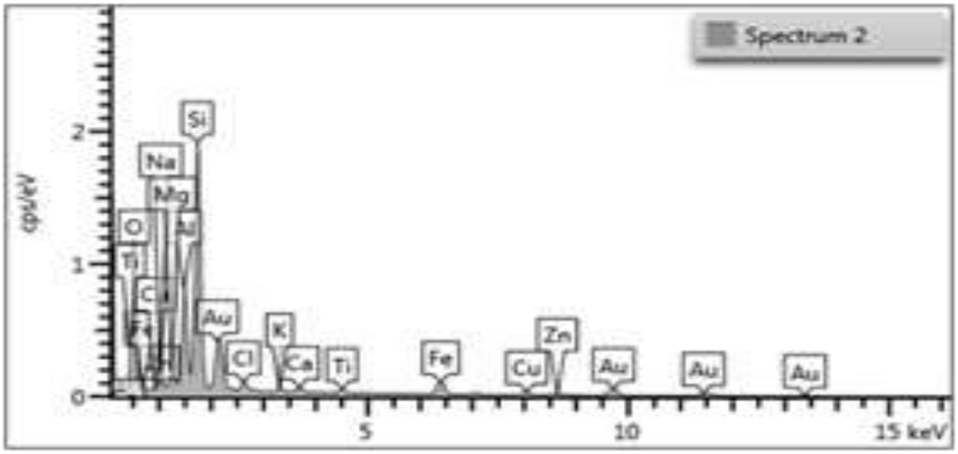

Figure 3-3 EDX test for Saudi bentonite

3.5 Nano Clay Properties

Clays are defined as an earthy, natural occurring material which it develops plasticity when mixed with a specific amount of water. As we mention above, clay fraction defined with respect to particle size, as size fraction composed of the smallest particles. **Table 3-7** represents Nano clay element concentration which is particularly consists of silica, alumina, and smaller amount of iron, potassium, magnesium, and sodium in addition of water. It can be said that Nano clay is essentially formed from chlorite clay mineral since Mg, and Fe elements found in the composition. This chlorite clay is considered non expendable clay type, where Fe or Mg are the central cations in appeared on the octahedral sheets as in the XRD results as in **Figure 3-4**. **Table 3-7** summarized the elements concentration of Nano clay material obtained from the XRF test analysis. It was obvious from the XRF test that this type of Nano clay is silicon-aluminium based since it contain higher percentages of silicon and aluminium.

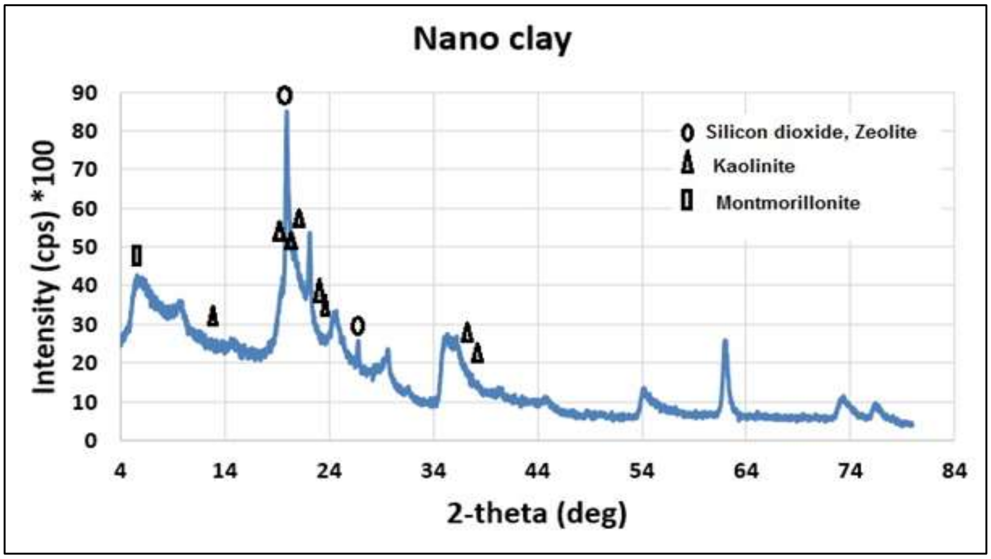

Figure 3-4 Composition of Nano clay by XRD

Table 3-7 Composition of Nano clay by XRF

Elements	Concentration
Si	31.82
Al	11.82
Fe	5.84
Mg	1.04
Ca	0.42
Cl	0.58
Ti	0.12
S	0.04
K	0.04
Cr	0.02
Zn	0.02
O	48.89

Figure 3-5, and **Figure 3-6** displays the chemical structure for typical Nano clays. Nano clay is considered a layered material, where it has a thickness 10 A° and its width can be expanded up to 1000 nm. As a matter of fact, the one gram of Nano clay powder might have billions of Nano clay particles with a surface area of many square meters (Uddin, 2008). Also, the knowledge of the particle size as well as understanding how the average particle size in the Nano clay is being distributed is playing an important role in predicting the outcome of these Nano composites. The usage of these Nano clays in oil well cementing

can result in a significant enhancement in the final cement sheath, since addition of it causes an increase in the cement strength, stiffness, and heat resistance which gives it credit for using in these fields (Morsy et al., 2012). On the other hand, there are some disadvantages in using it such as moisture absorption reduction, decrease flammability as well as permeability to gas and water. Use of Nano clay can result in a significant enhancement in the mechanical properties of the cement sheath. **Figure 3-7** shows the powdered Nano clay used in the experimental work.

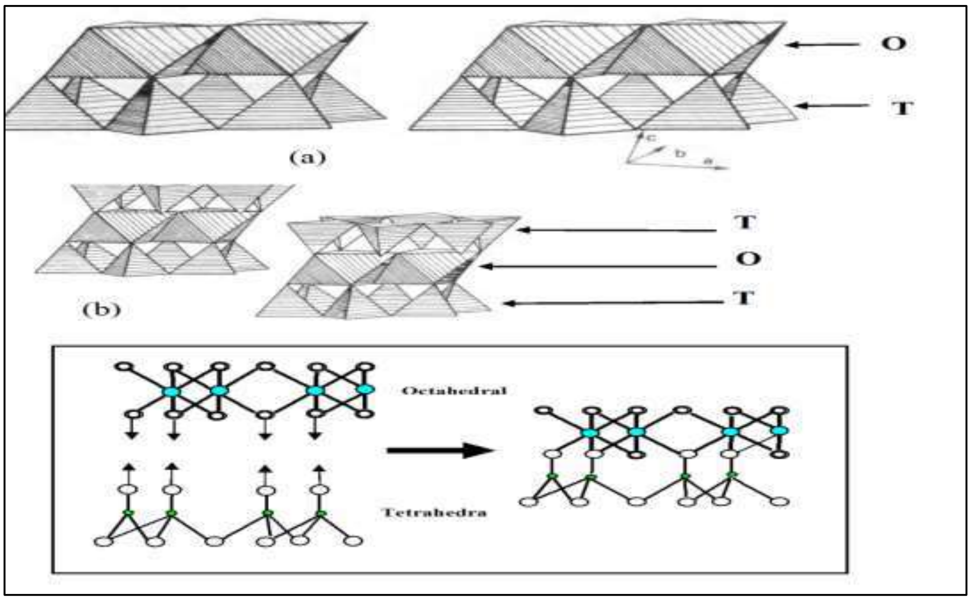

Figure 3-5 Structure of clay

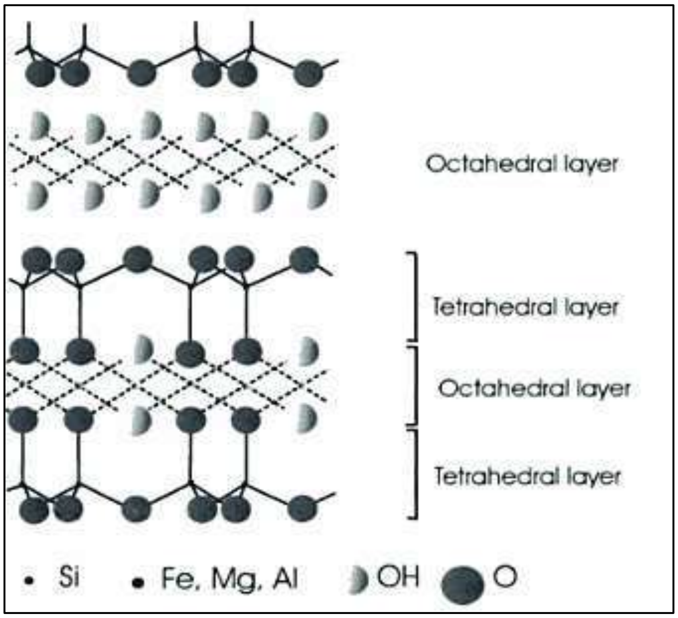

Figure 3-6 Chlorite clay structure

Figure 3-7 Nano clay powder

3.6 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 3-8**.

Figure 3-8 High speed blender

Mixing the cement is an important issue 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, whereas in wet method, the additives are blended with water and then with cement. Tap water is used in all the experiments.

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

- Procedure of mixing:
    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 ^{o}F as shown in **Figure 3-9**.

Figure 3-9 Atmospheric consistometer

3.7 Thickening Time Cement Test

Thickening time cement test determines the time period in which cement remain pumpable under certain conditions. In this test, the cement remains liquid 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 cement test, fluid contamination, and also a sudden stop in the cement pump, but cannot be included in the laboratory cement thickening time test. **Figure 3-10** 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.

- Procedure of mixing:
    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 to consistometer slurry cup and put in a HPHT equipment.
    7. Increasing temperature and pressure in the machine is done in accordance with suitable cement test schedule until 228 ^{o}F, and 9315 psi is gained.
    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 prospered for the next test.

Figure 3-10 Pressurized HPHT consistometer

3.8 Fluid Loss Cement Test

The cement fluid loss test measures the amount of the fluid lost when this cement is subjected to a differential pressure. Most of the cement loss is occurred during cementing the high permeability or sensitive formation. **Figure 3-11** shows high pressure high temperature fluid loss cement apparatus. The temperature used is 194 ^{o}F with differential pressure of 1000 psi.

- Procedure of mixing:
  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. Cement slurry is poured into the tasting 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 as showed below.

Figure 3-11 HPHT fluid loss cement tester

3.9 Density Measurement

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 3-12.**

- Procedure of mixing:
  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. Then for about 20 minutes, cement slurry is cured at a temperature of 194 ^{o}F using an atmospheric consistometer.
  7. Cement slurry is poured into the density cup and closed.
  8. Cement is pumped using syringe until no more cement enter the cell.
  9. Finally, the rider is moved back and front till the bubble in the glass is balanced, and the reading is recorded.

Figure 3-12 Pressurized density balance

3.10 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 3-13** shows a gradual cylinder for free water cement test.

- Procedure of mixing:
  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 cured at a temperature of 194 ^{o}F in an atmospheric consistometer 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 3-13 A gradual cylinder for free water cement test

3.11 Rheology

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 3-14.**

Figure 3-14 Variable speed rheometer

- Procedure of mixing:
  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. Then for about 20 minutes, cement slurry is cured in an atmospheric consistometer and at a temperature of 194 ^{o}F.

7. After that, cement slurry is put into rheometer cup in which it also conditioned at a temperature of 194 ^{o}F.
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.

3.11.1 Gel Strength

Gel strength 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.

3.12 Compressive Strength

The purpose of compressive strength test is to calculate the ability of the cement to resist axial pushing forces. The compressive strength is determined by using two methods, direct method "crushing" or by using the ultra-sonic cement analyser.

3.12.1 Compressive Strength by the 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.

- Procedure of mixing:

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 cured in an atmospheric consistometer 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 3-16**) to produce the cubes (2 inch × 2 inch).
8. After that, moulds are placed in the HPHT curing vessel as shown in **Figure 3-15** with an initiating temperature of 27 ^{o}C. After that, test schedule is applied to reach the target temperature and pressure of around 290 ^{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 as displayed in **Figure 3-17**.
10. Finally, cement cubes are crushed in a compressive strength tester (see **Figure 3-18**) and values are reported.

Figure 3-15 HPHT curing machine

Figure 3-16 Cement moulds

Figure 3-17 Cured cement cubes

Figure 3-18 Compressive strength tester

3.12.2 Compressive Strength by Sonic Method

Ultra sonic cement analyser (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 3-19** shows the ultra-sonic cement analyser.

- Procedure of mixing:
  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 cured in an atmospheric consistometer 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 290 ^{o}F, while the pressure is 4666 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 3-19 Ultra sonic cement analyzer

3.13 Porosity and Permeability Tests

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 × 1.5 inches 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 AP-608 installed with manually loaded hassle type core holder for 1 and 1.5 inch cores, and the permeability ranges from 0.001 up to 500 md.

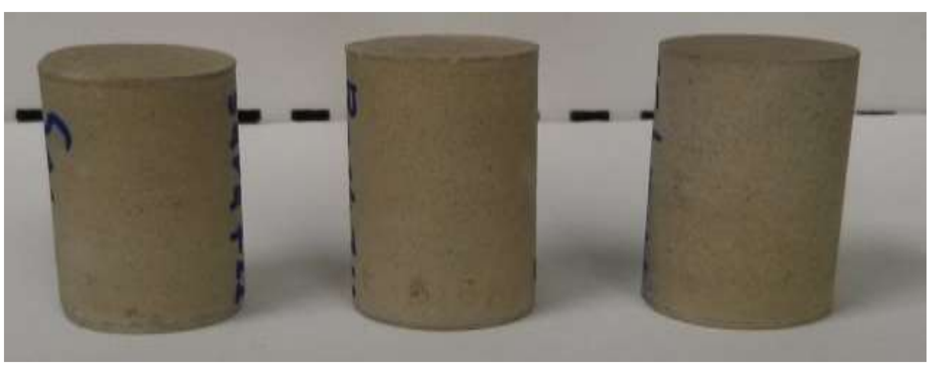

Figure 3-20 Cement plugs

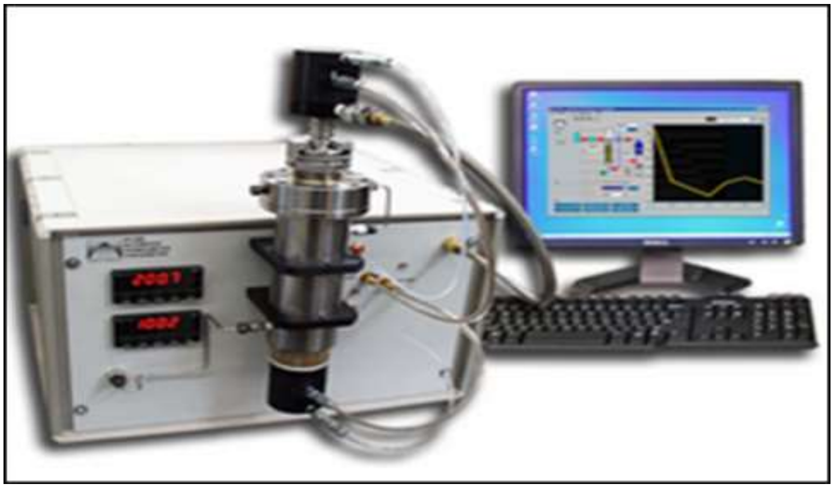

Figure 3-21 Automated porosimter/permeameter

3.14 Microstructure Analysis

The microstructure of the cement can be analysed 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 studying the effect of adding untreated Saudi bentonite to Portland Saudi cement class G under both high pressure and temperature conditions. In this chapter, we presented well parameters and experimental program that we followed during conducting the experiments. Each experimental test results are discussed thoroughly for selected well cement system.

4.1 Cement System Validation

As we discussed earlier in chapter 3, there were two cement systems proposed for the experimental program, and the main difference between the two systems was the addition of untreated Saudi bentonite with percentages of 0, 1, 2, and 3% of the cement weight. After we started the experiments we observed the following:

At the beginning, we used a cement system as explained in the previous section without untreated Saudi bentonite and with 0.4% BWOC dispersant. After that, the viscosity of the produced cement from this cement design was measured using fann rheometer, and the reading was out of range, which indicating that the produced cement was too viscous and might lead to higher pressure losses throughout the cementing process. As a result of this, the used percentage of the dispersant was increased up to 0.8% and then to 1% bwoc, so as to produce an easy mixing cement with a reduced viscosity. Therefore, this cement mix design was kept stable through all the conducted experiments.

When untreated Saudi bentonite added to the cement, the viscosity of the cement was increasing rapidly, and as a result, the produced cement became thicker compared with a free cement mix. It was also observed that by increasing the amount of untreated Saudi bentonite percentage, the viscosity of the cement mix increased as well, and more water should be added to reduce the viscosity. Furthermore, with 1, and 2% Saudi bentonite, the produced cement viscosity was acceptable and close to each other. On the other hand, higher percentages like 3% added, it causes an increase in the viscosity, but still pumpable. The higher percentages added, the more thick cement is produced, and the more water cement ratio is needed to obtain a flowable fluids.

Here we also presented a comparison between upgraded (treated) Saudi bentonite (Musaab, 2014) and the commercial bentonite using 1.9% bwoc to produce a cement slurry with a density of 101 PCF with a cement design provided from Saudi Aramco.

First, we will study the effect of untreated Saudi bentonite on the cement, and the optimum mix design will be identified. Next, Nano clay will be added to the optimum mix and cement properties will be studied, and the optimum mix will be reported. Finally, treated Saudi bentonite will be added at 1.9% to a cement to produce a cement with a density of 101 PCF, and the results will be compared with that of commercial imported bentonite.

4.2 Effect of Saudi Bentonite (SB) on the Cement Properties

Here the effect of adding untreated Saudi bentonite to the cement mix will be investigated and the following properties will be studied:

4.2.1 Effect of SB on the Cement Slurry Thickening Time

Thickening time cement test gives us 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. In this test the cement remains liquid for a long time during pumping, and then it turns into solid when pumping stops. 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 64 MPa, and 228 ^{o}F. The maximum temperature was reached during 42 minutes. Four cement systems containing untreated Saudi bentonite with varied percentages of 0, 1, 2, and 3% 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 untreated Saudi bentonite with varied percentages 0, 1, 2, and 3%.

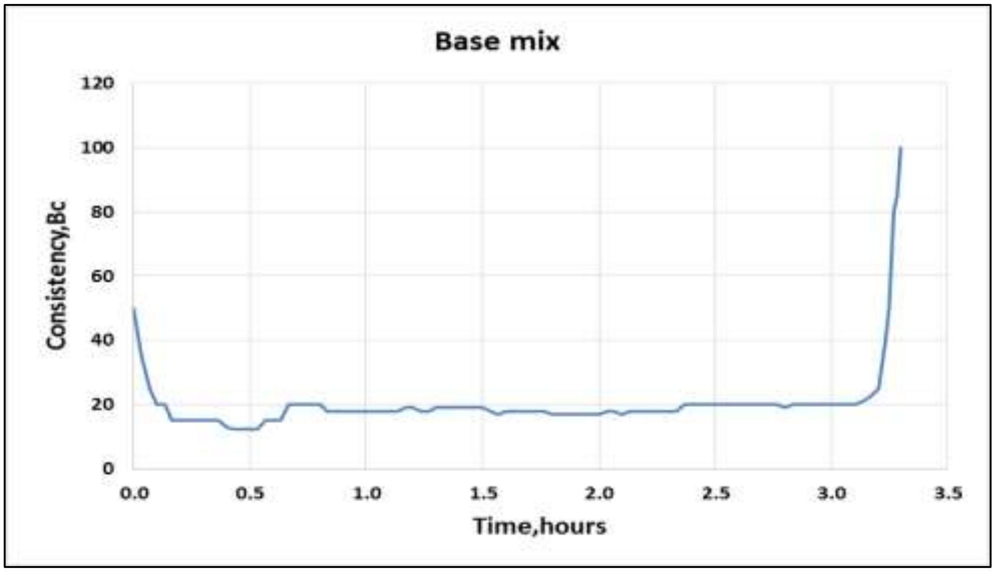

Figure 4-1 Thickening time cement test plot with 0% untreated Saudi bentonite (base mix)

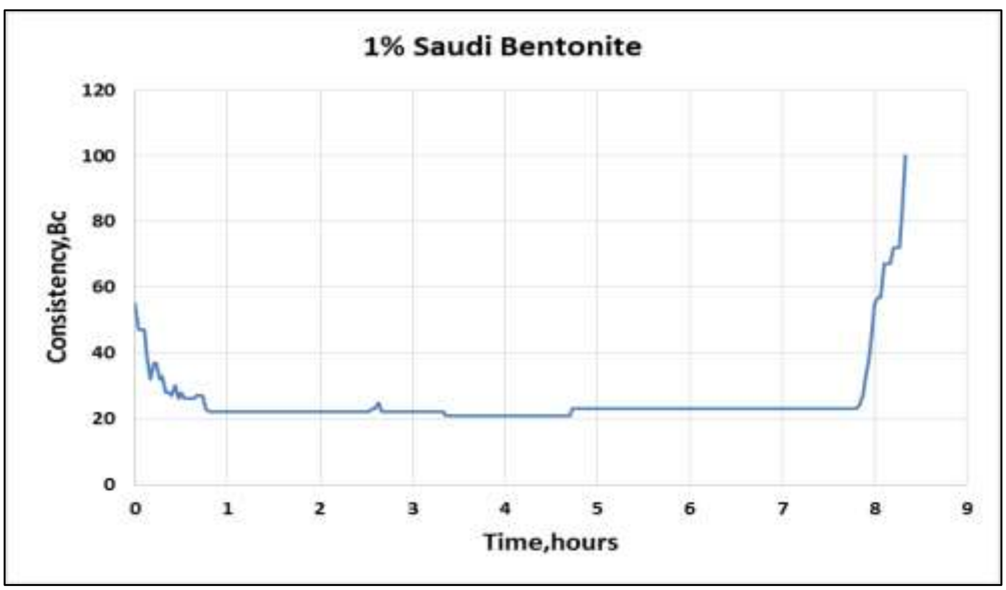

Figure 4-2 Thickening time cement test plot with 1% untreated Saudi bentonite

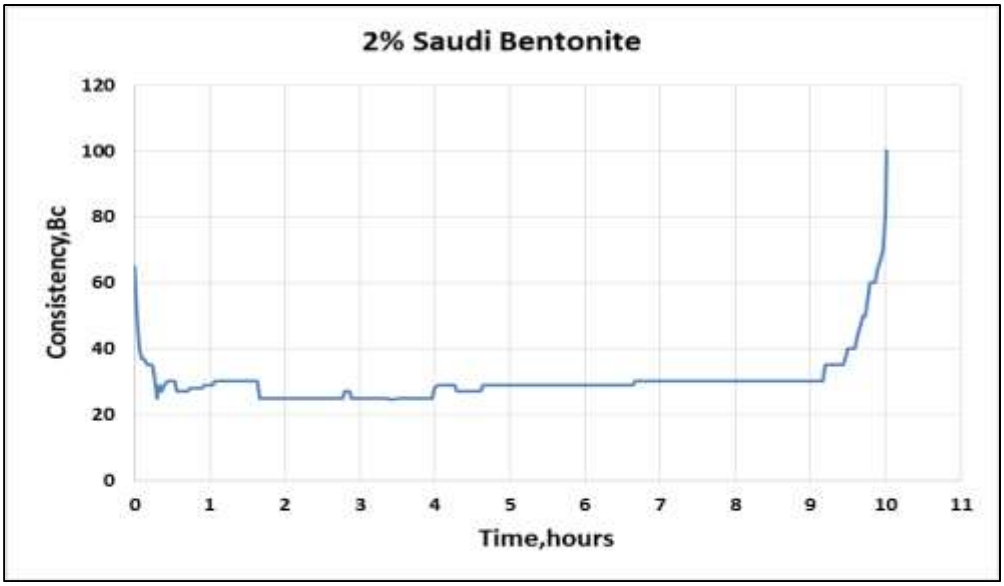

Figure 4-3 Thickening time cement test plot with 2% untreated Saudi bentonite

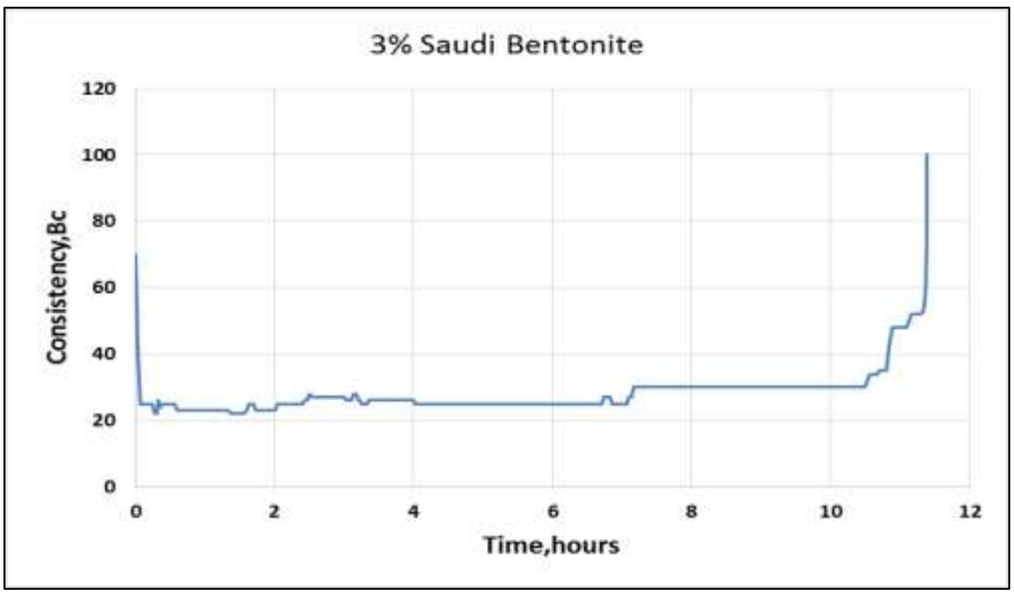

Figure 4-4 Thickening time cement test plot with 3% untreated Saudi bentonite

At the start of the test, the cement slurries have a consistencies of (55, 60, 65, and 70) 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 untreated Saudi bentonite to the cement resulted in increasing in the thickening period time from three hours up to eleven hours as shown in **Figure 4-5**, and **Figure 4-6**.

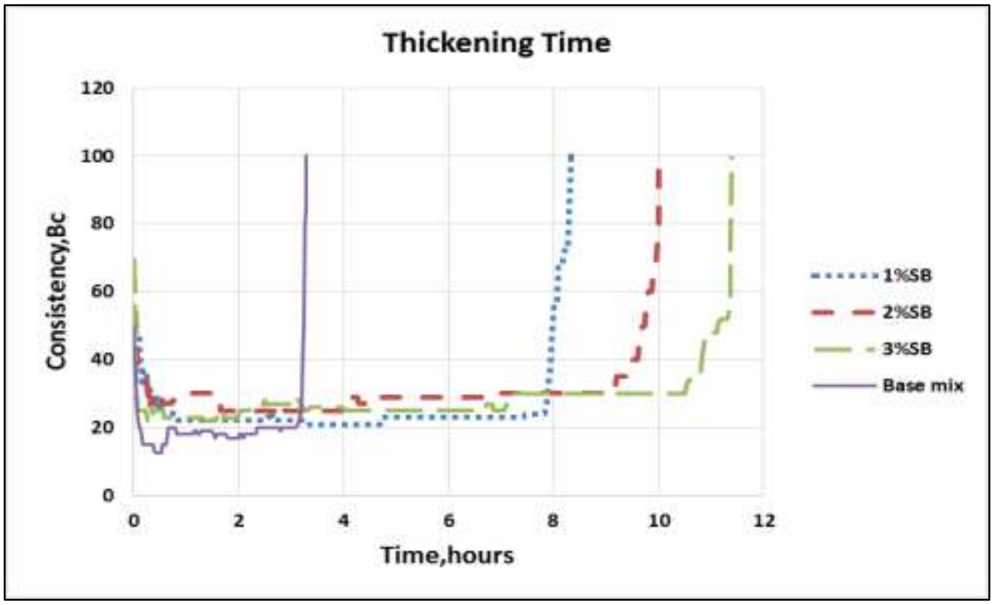

Figure 4-5 Thickening time for 0, 1, 2, and 3 % SB

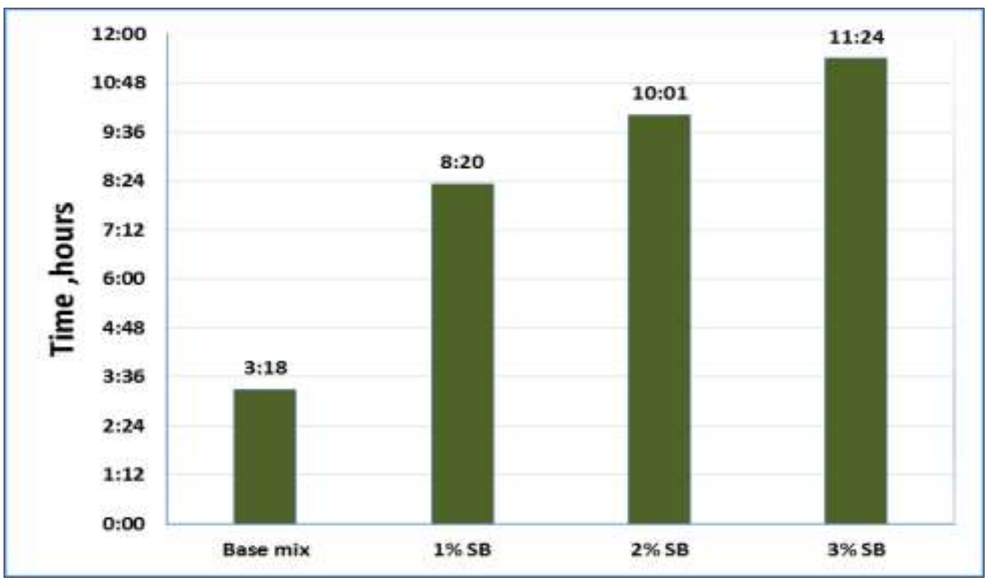

Figure 4-6 Comparison of cement thickening time with 0, 1, 2, and 3% of SB

It is quite obvious that the addition of untreated Saudi bentonite to the cement resulted in increasing in the thickening period time as explained above. The reason for this delay is that the addition of untreated Saudi bentonite causes a slow in the hydration process of the cement, and in turn, it extends the time the cement takes to thick.

When 1% of untreated Saudi bentonite added to the base mix, the cement thickening time increased from 3:18 minutes up to 8:20 minutes. Further addition of higher percentages untreated Saudi bentonite pushed up the thickening time to the period of 11:24 minutes in the case of 3%. Retarding the cement is one of the biggest concerns when it comes to drilling deep wells, therefore, adding 1% of untreated Saudi bentonite maybe the best practice, since it gave sufficient time where the minimum thickening time needed for the proposed well was five hours.

When the tests started, higher consistencies of (55, 60, 65, and 70) Bc respectively, were observed (see **Figure 4-7)**, 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 4-8** 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. In other words, it can be said that reaching 40 Bc is an indicator that the cement is becoming unpumpable since the cement has only a short time to reach 100 Bc.

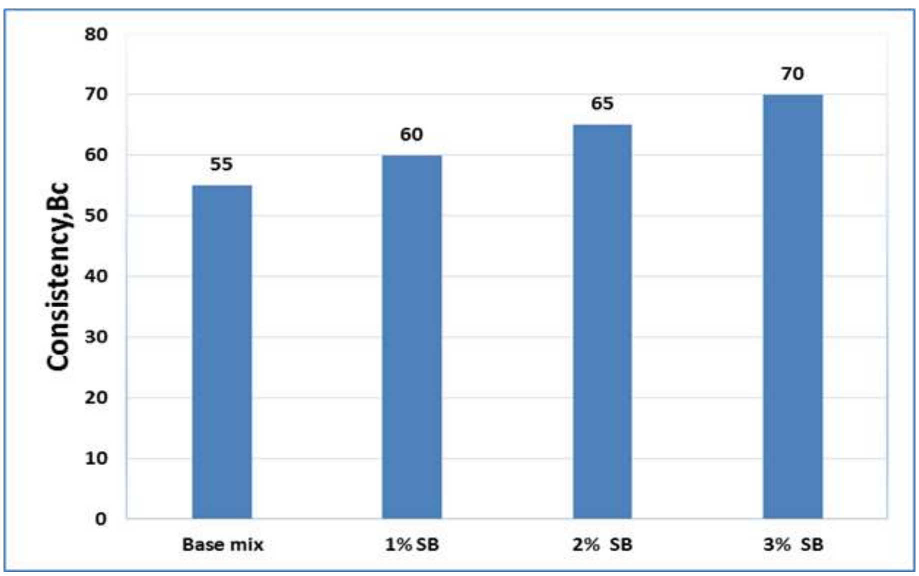

Figure 4-7 Consistency at the beginning of thickening time cement test

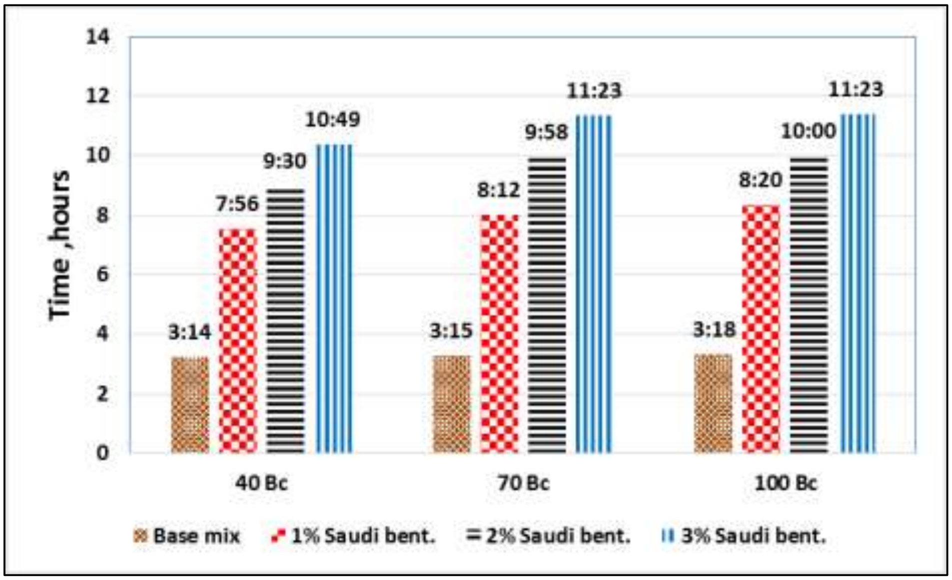

Figure 4-8 Time to reach 40, 70, 100 Bc cement consistencies

4.2.2 Effect of SB 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. Bentonite 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 untreated Saudi bentonite added to the cement mix, it resulted in a reduction in the amount of the fluid loss as shown in **Table 4-1**. **Figure 4-9** 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 1 percentages untreated Saudi bentonite 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 100 cc/30 min.

Table 4-1 Fluid loss of cement with 0, 1, 2, and 3% of SB

Fluid loss (API)	Base mix	1% SB	2% SB	3% SB
ml	65	60	56	52

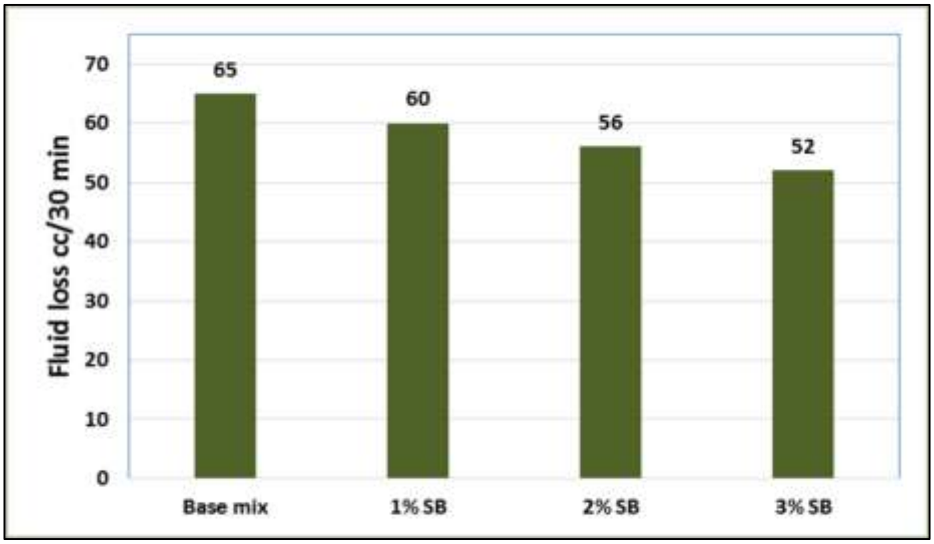

Figure 4-9 Fluid loss of cement with 0, 1, 2, and 3% of SB

4.2.3 Effect of SB on Cement Slurry Free Water Separation

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 untreated Saudi bentonite with percentages 0, 1, 2, and 3% as well as simple class G cement 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 untreated Saudi bentonite with 0, 1, 2, and 3% bwoc. It is clear that the addition of untreated Saudi bentonite to the cement slurry resulted in no free water separation at the top of the cement as observed in **Figure 4-10**, and **Figure 4-11.**

The reason of no free water being observed when untreated Saudi bentonite added to the cement because bentonite is a clay type material which absorbed the water and swells. So, the water is trapped and blocked between the layers of bentonite clay resulting in disappearing of the water separation problem. Also, untreated Saudi bentonite did not cause any distribution in the particle suspension property in the cement.

Table 4-2 Free water content with 0, 1, 2, and 3% SB mixed with cement

Free water	Class G	Base mix	1% SB	2% SB	3% SB
ml/250ml	2.1	0	0	0	0

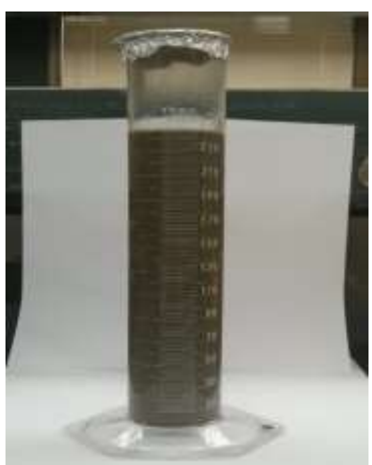

Figure 4-10 Cement slurry at the start of the free water cement test

Figure 4-11 Free water cement test after 2 hours aging

4.2.4 Effect of SB on Cement Slurry Density

Well control is one of the most important 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 we mention 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. One more thing, 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 we designed the cement density after putting cement additives to be around 16.6 Ib/gal. Cement systems with different percentages of untreated Saudi bentonite 0, 1, 2, and 3% bwoc were prepared, and the density of the produced cement was measured using a pressurized cement balance. **Table 4-3** shows the density of cement with different percentages of untreated Saudi bentonite 0, 1, 2, and 3% mixed with the cement.

Table 4-3 Density of cement with 0, 1, 2, and 3% of SB

Cement slurry	Density (Ib/gal)
Class G	15.8
Base mix	16.65
1% SB	16.52
2% SB	16.37
3% SB	16.27

It is clear that the utilizing cement class G in cases of deep well cannot be functional, since the produced density might not be able to bear the formation pressure as well as it is poor mechanical properties. As a resulted, cement additives are inserted to improve its density and enhance its mechanical properties. From the table above, cement with the additives has a density of 16.65 Ib/gal, which indicates improvements. On the other hand, admixing untreated Saudi bentonite with the cement has not a big influence in the on the resulted density since it is calcium base ,and the density with 3% SB was around 16.27 Ib/gal with a reduction of 2.28 percent from the cement base mix containing a zero percent of Saudi bentonite. In short, incorporating untreated Saudi bentonite to the cement does not affect the cement density and the results were almost the same as shown in **Figure 4-12**.

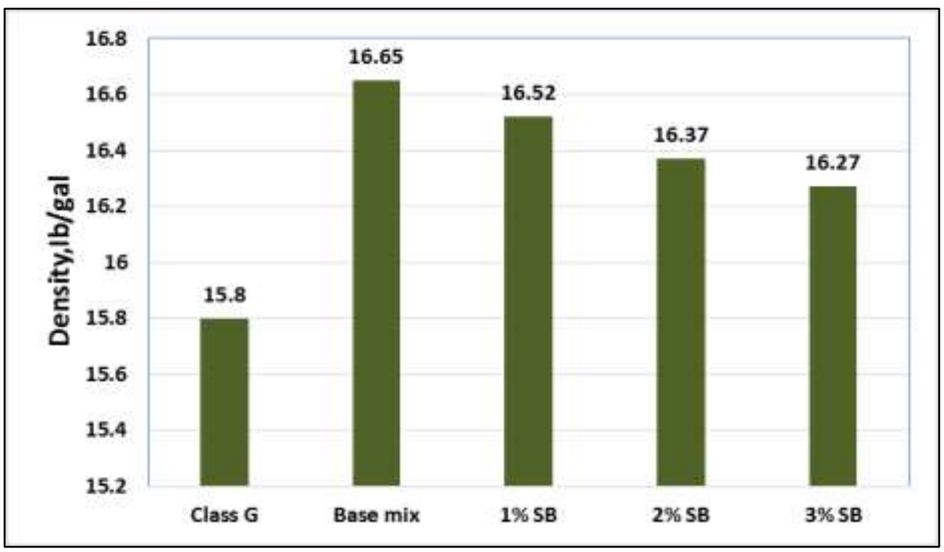

Figure 4-12 Density of cement with 0, 1, 2, and 3% of SB

4.2.5 Effect of SB on Cement Rheology

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 4-4 shows plastic viscosity and yield point of simple class G, and cement base mix with various percentages 0, 1, 2, and 3% of untreated SB. It was clear that simple class G cement has the lowest values of plastic viscosity which gave an indication that using the cement system cannot improve mud displacement in deep wells. For these particular wells, cement additives must be added to the base mix to improve its rhetorical properties. At the end, we added untreated Saudi bentonite with different percentages and see its effect on cement rhetorical properties.

It is obvious that the addition of untreated Saudi bentonite to the base mix resulted in enhancement in the rheological cement properties like plastic viscosity and yield point. Plastic viscosity measures the shear rate and solid particles present in the mix as well. **Figure 4-13** shows the plastic viscosity trend of the cement with different percentages of Saudi bentonite. Apparently, adding untreated Saudi bentonite caused an increase in cement solid particles which in turn increased the plastic viscosity and caused enhancement in cement viscosity. Unlike this enhancement growth observed in plastic viscosity, yield point trend has been slightly improved (see **Figure 4-14)**. The addition of 1% untreated Saudi bentonite resulted in 8.27 $Ib_f/100\ ft^2$ yield point. As the percentage of untreated Saudi bentonite increase, the value of yield point slightly increased, for instance, 3% untreated Saudi bentonite gave around 12 $Ib_f/100\ ft^2$. Here with 3% we observed an increase in the trend which gives an indicator that further addition of untreated SB will result in a rapid increase in both yield point and plastic viscosity. Thus, Yield point has some side effects on cement properties, so completion engineers take this parameter into consideration when designing the optimum cement slurry for the operation.

Table 4-4 Plastic viscosity and yield point of cement with 0, 1, 2, and 3% of SB

Properties	class G	Base mix	1% SB	2% SB	3% SB.
Plastic viscosity, cp	32.2	240.3	250	258	284.7
Yield point Ib/100 ft^2	16.4	7.18	8.27	9	11.49

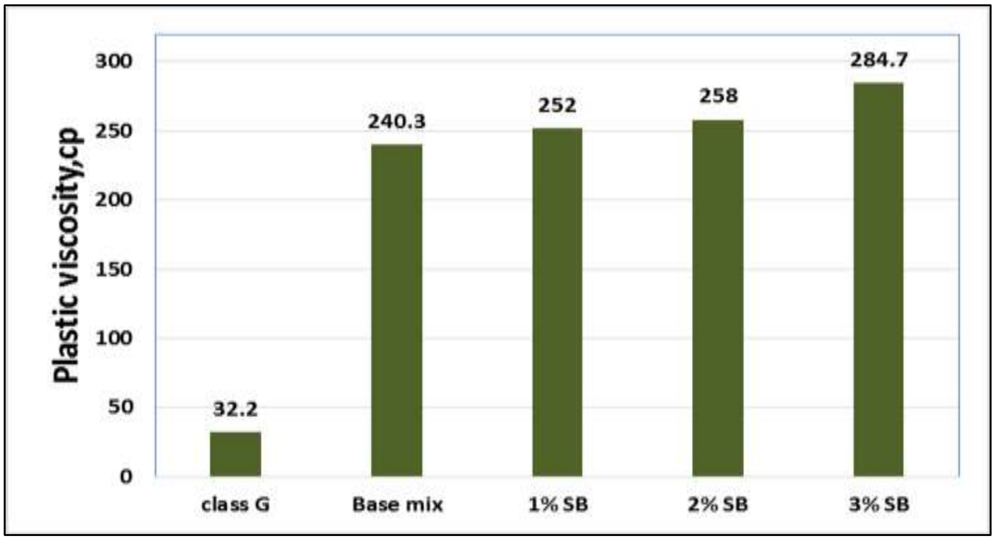

Figure 4-13 Plastic viscosity of cement with 0, 1, 2, and 3% of SB

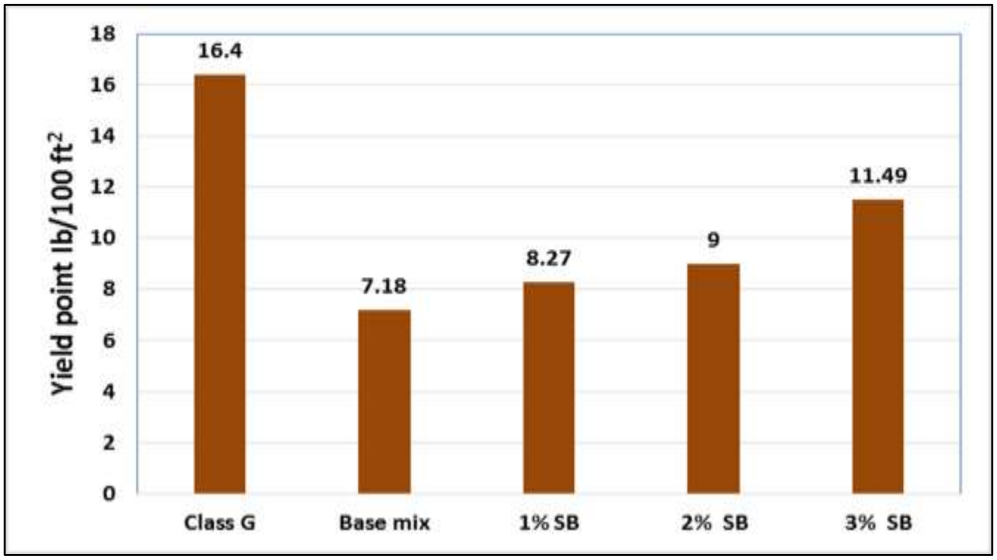

Figure 4-14 Yield point of cement with 0, 1, 2, and 3% of SB

4.2.6 Effect of SB on the Gel Strength of the Cement

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.

Table 4-5 illustrates the gel strength results of cement with various percentages of untreated Saudi bentonite conducted using ofite and fann Viscometer. From the table, we observed that the gel strength has increased with increasing the amount of untreated Saudi bentonite. It is obvious that the addition of untreated Saudi bentonite does not produce a noticeable effect on the 10-sec gel, and the values are almost close to each other as in percentages of 0, 1, and 2%. However, higher percentages of untreated Saudi bentonite cause a rise in the 10-sec gel, for instance, 3% produce 13 $Ib_f/100ft^2$ (see **Figure 4-15)**.

The same trend is observed when the cement slurry is subjected to 10-min gel strength cement test. When lower percentages of untreated Saudi bentonite of 0, 1, and 2% of added to the cement mix, the 10-min gel results were almost the same of around 25 $Ib_f/100ft^2$. Though, further addition of Saudi bentonite, for instance, 3%, ended in a jump on the 10-min gel strength up to 32 $Ib_f/100\ ft^2$. This gel strength trend behaviour was also observed when Nano clay was mixed with the cement slurry (Mobeen, 2013). On the other hand, adding Nano silica with small percentages caused a rapid increase in the gel strength compared with Saudi bentonite and Nano clay (Sami, 2012). The reason for this gel strength development is that Nano silica develops gel strength so quickly and helps in reducing particle settling problems. In other words, adding untreated Saudi bentonite to the cement system helps in developing early gel strength and reducing the settling problems.

Table 4-5 Gel strength of cement with 0, 1, 2, and 3% of SB

Gel strength $Ib_f/100\ ft^2$	Class G	Base mix	1% SB	2% SB	3% SB
10-sec	7	7.5	8	9.5	13
10-min	20	25	25.5	28	32

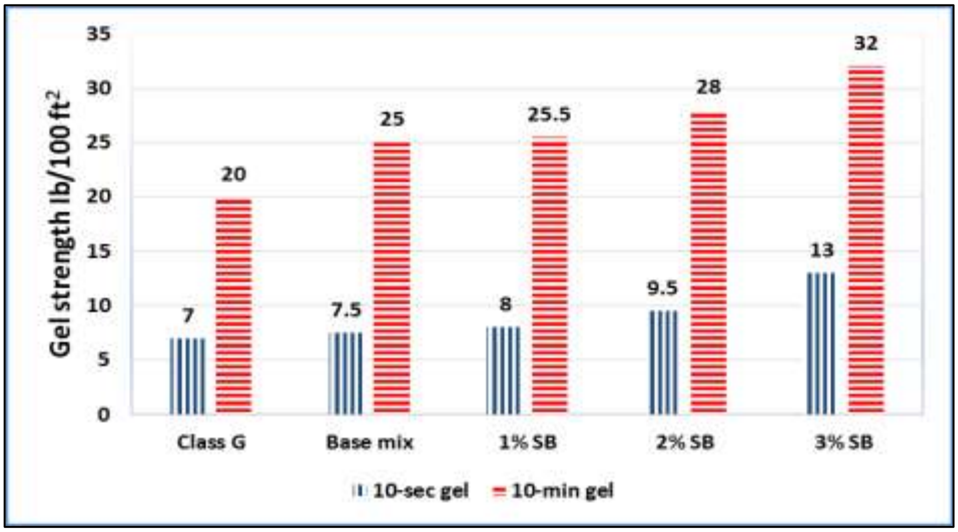

Figure 4-15 Gel strength of cement with 0, 1, 2, and 3% of SB

4.2.7 Compressive Strength of Cement

4.2.7.1 Effect of SB on Cement Compressive Strength by Crushing Method

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 untreated Saudi bentonite with different percentages of 0, 1, 2, and 3% as well as simple class G cement 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 290 ^{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**: it is clear that adding untreated Saudi bentonite with percentages of 1% 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 1% untreated Saudi bentonite improved the compressive strength, which might be a consequence of high percentages of silica in the untreated Saudi bentonite that accordingly induced pozzolanic reaction. Maximum compressive strength was achieved with 1% untreated Saudi bentonite, however, adding untreated Saudi bentonite with percentages beyond the 1% resulted in a reduction in the compressive strength property as observed clearly in **Figure 4-16**. Experiments conducted with 2% and 3% of untreated Saudi bentonite gave closer values of base cement. This reduction in compressive property might be related to the low density of cement since adding untreated Saudi bentonite resulted a lower cement density. As a result, 2% and 3% untreated Saudi bentonite are not recommended in the cases where highly compressive strength cements are needed.

Nano clay (Mobeen, 2013), and Nano silica (Sami, 2012) were also added to the cement admix and the results showed that after 24 hours curing, 1% of Nano clay as well as 1% Nano silica gave the highest compressive strength of 6878, and 6967 psi respectively. In our experiment, 1% untreated Saudi bentonite also gave a similar value of compressive strength like Nano clay and Nano silica of around 6846 psi.

Table 4-6 Compressive strength of cement with 0, 1, 2, and 3% of SB by crushing after 24 hours

Sample	Class G	Base mix	1% SB	2% SB	3% SB
1	3377.2	7250	7641.5	5887	5959.5
2	3197.2	5800	6307.5	6351	6568.5
3	3230.6	5365	6592	7105	5930
Average	**3268.3**	**6138**	**6846.9**	**6447.66**	**6152**

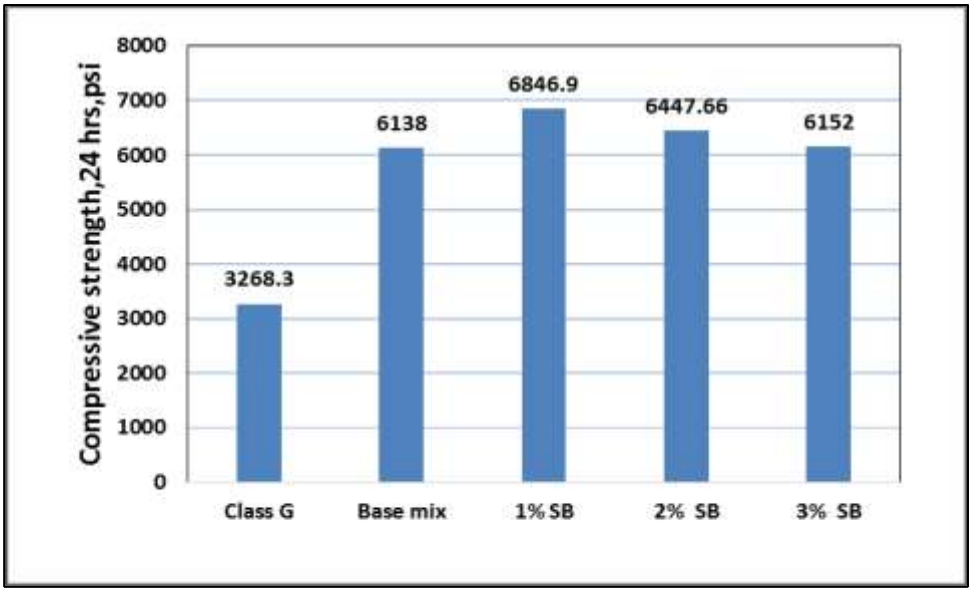

Figure 4-16 Compressive strength of the cement with 0, 1, 2, and 3% of SB by crushing after 24 hours

4.2.7.2 Effect of SB on the Compressive Strength by Sonic Waves

Four cement system containing untreated Saudi bentonite with different percentages of 0, 1, 2, and 3% in addition to simple class G cement have been tested for compressive strength using an ultra-sonic cement analyser under conditions of both high pressure (4666 Psi), and high temperature (290 ^{o}F) curried 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. From the UCA cement test results, class G cement showed fast hydration reaction, where the cement sets quickly compared to the cement base mix, or with cement admixed with untreated Saudi bentonite as shown in **Figure 4-17**. This increase in the speed of strength development is almost slows down after 12 hours, and then come to stabilize at the end of the 24 hours. Also, it can be observed that in severe conditions where the temperature is above 230 ^{o}F, enormous phase changes took place within the structure of class G cement, and resulted in a significant reduction in the strength of the finial produced cement. Furthermore, the problem of strength retrogression appeared in severe conditions, where the temperature might exceed 230 ^{o}F. To solve this problem, silica flour is added in percentages ranged from 35 to 40% bwoc to the cement mix to produce a cement sheath with an enhancement in the permeability and strength (Iverson, Maxson, and Bour, 2010).

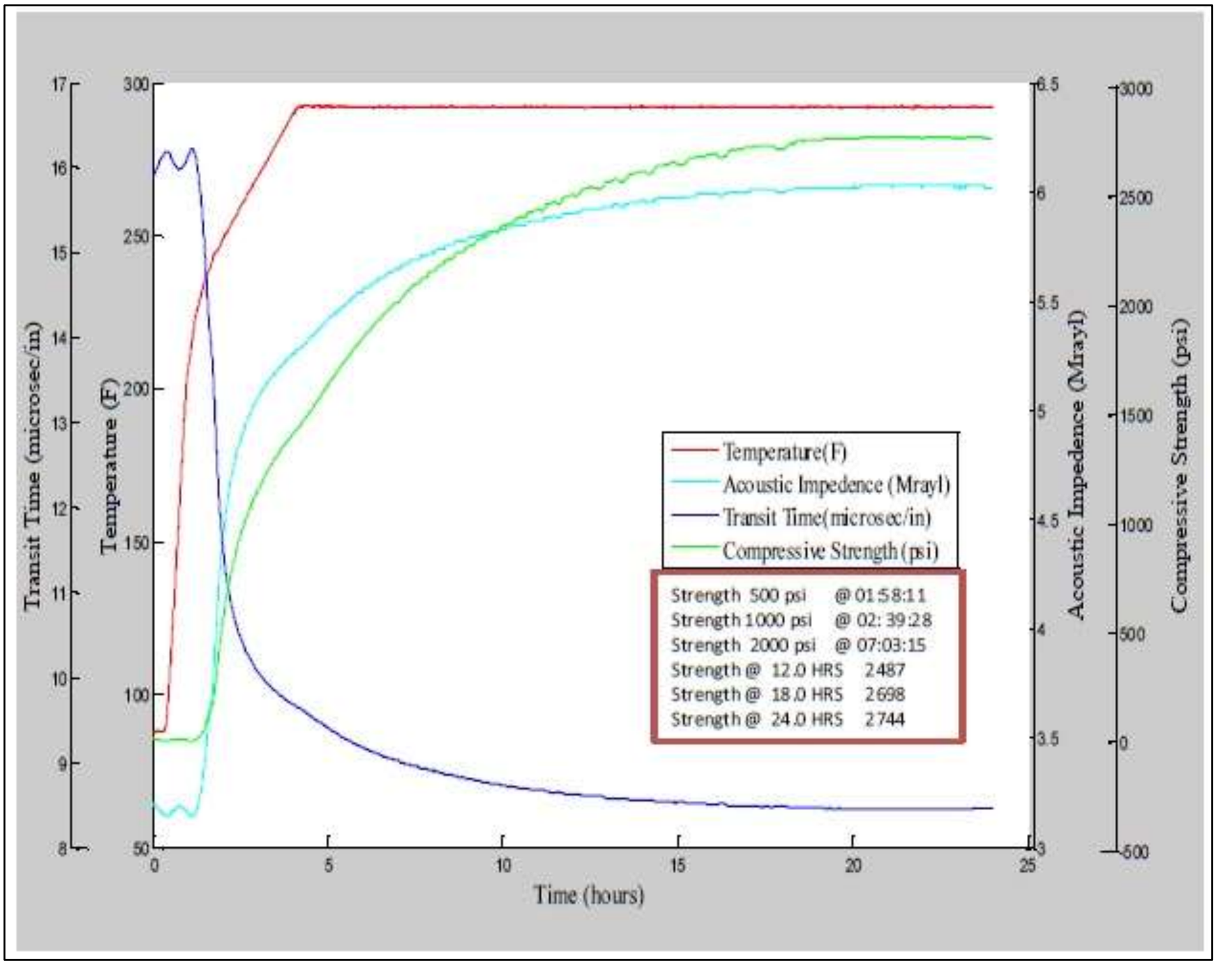

Figure 4-17 Strength development of cement class G using UCA apparatus through 24 hours

The compressive strength is a key factor, especially in cases of cementing and drilling ahead. In fact, during the oil well cementing, exact known of the time period to reach a compressive strength of 50 and 500 psi are important, and this time period should be minimized. The 50 psi is the minimum gel strength required and the 500 psi is the minimum compressive strength required for resuming drilling. Consequently, trying to reduce the time spent in waiting for the cement to solidify and hardening (WOC) time prior to resuming and drilling ahead is needed (Coker, Harris, and Williams, 1992). It was observed that cement class G showed the smallest transient time to reach up the compressive strength of 500 psi which was around 2 hours. Although this cement type class G showed enhancement in the early compressive strength development, this cement is not a good choice in the case of cementing high pressure high temperature wells. **Figure 4-18**, **Figure 4-19**, **Figure 4-20**, and **Figure 4-21** show the compressive strength results from UCA for the cement base mix, and the cement with 0, 1, 2, 3% bwoc of untreated Saudi bentonite.

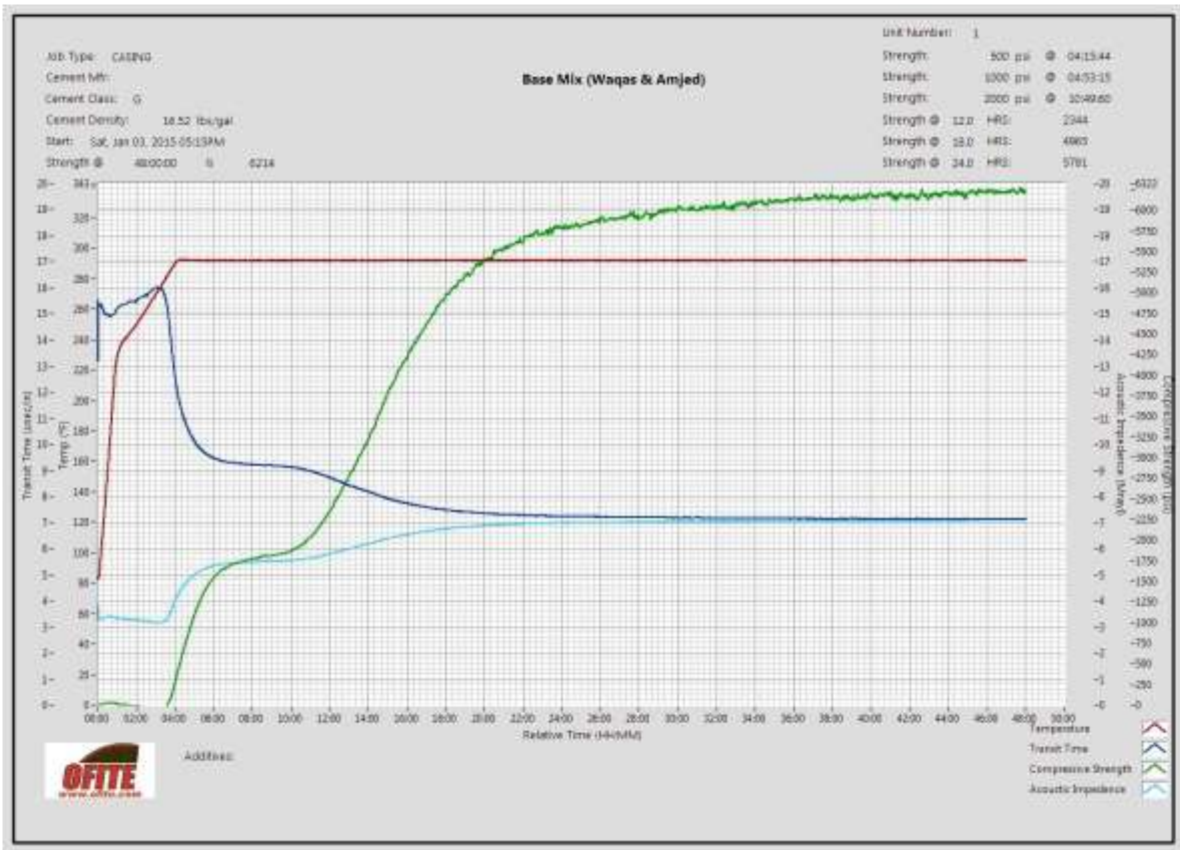

Figure 4-18 Strength development of cement base mix by UCA for 48 hours

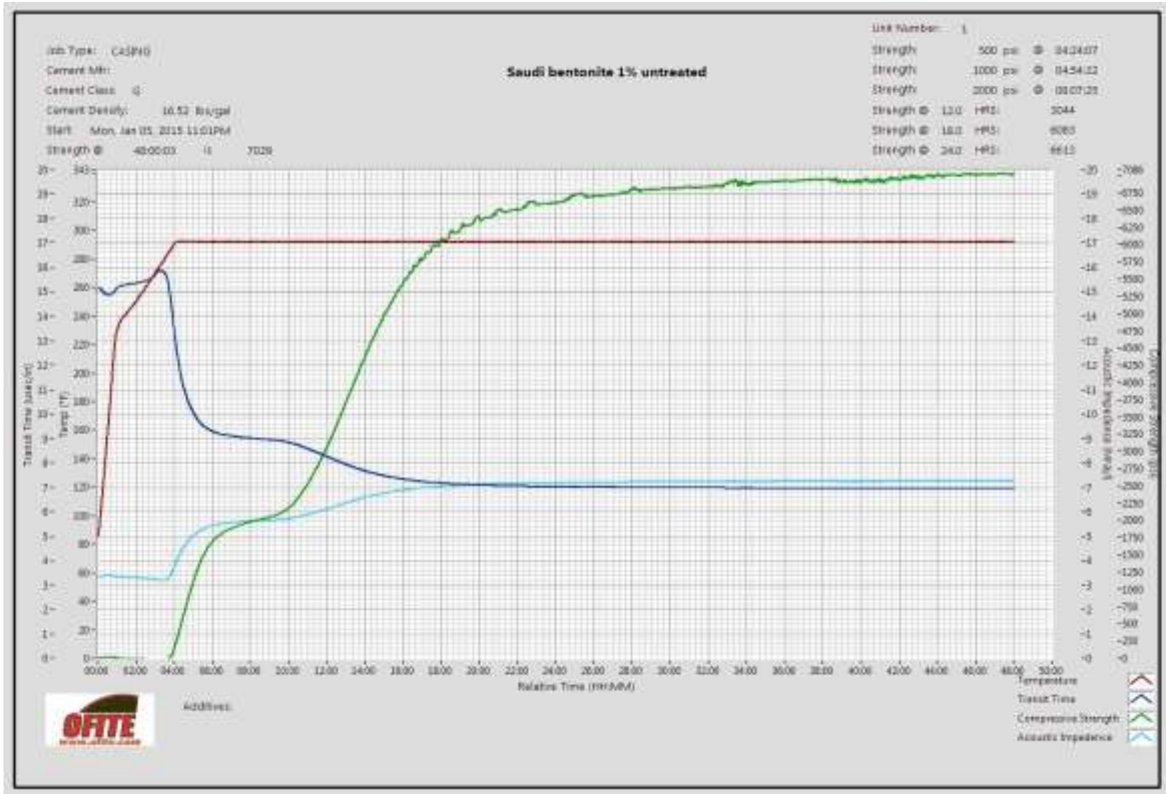

Figure 4-19 Strength development of a cement mixed with 1% SB by UCA for 48 hours

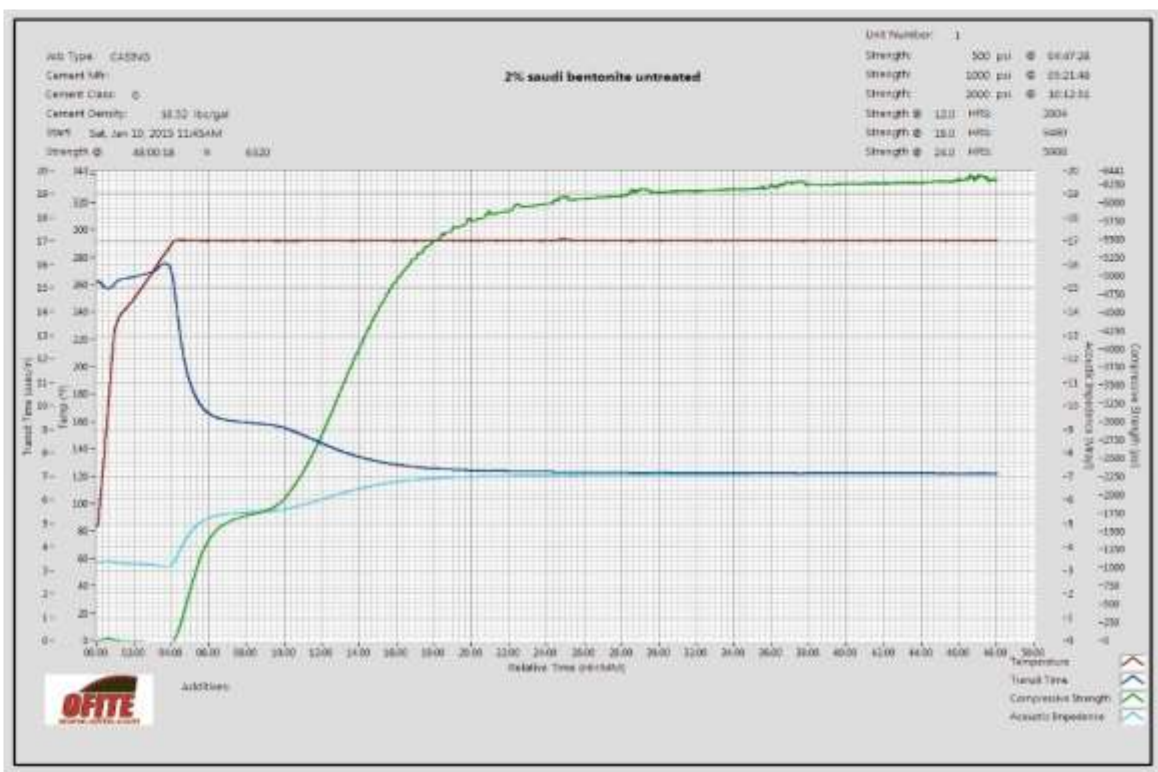

Figure 4-20 Strength development of a cement mixed with 2% SB by UCA for 48 hours

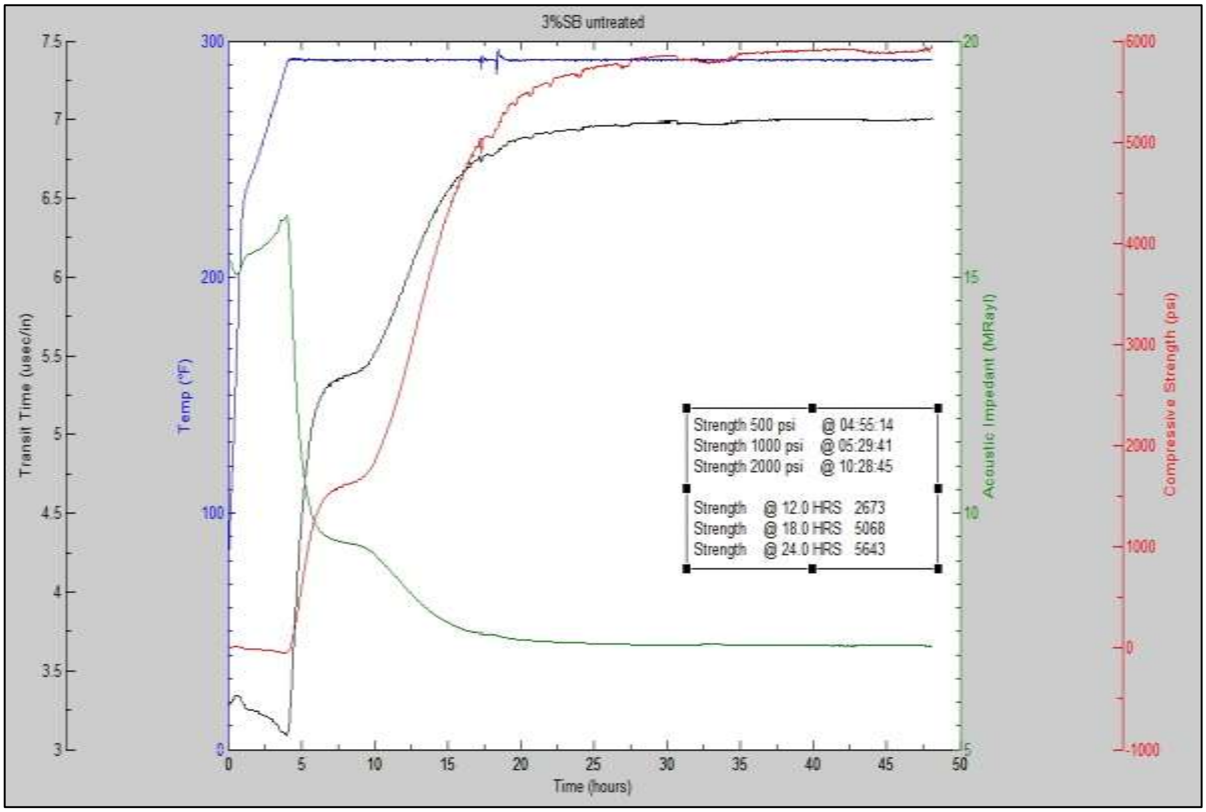

Figure 4-21 Strength development of a cement mixed with 3% SB by UCA for 48 hours

Also, strength retrogression is one of the most challenging problems that are faced, especially during dealing with high temperature conditions. As we mention above, the addition of silica flour with 35% bwoc will terminate this problem. Untreated Saudi bentonite was added to the cement mix, and the effect of adding this material on the mechanical properties of the produced cement was observed. **Table 4-7** shows compressive strength results of class G cement as well as cement with different percentages 0, 1, 2, and 3% of untreated Saudi bentonite. Untreated Saudi bentonite 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 4-22**, and **Figure 4-23**. It is obvious that the addition of 1% untreated Saudi bentonite resulted in the highest compressive strength of around 7000 psi after 48 hours aging. **Figure 4-24** proves that increasing the amount of untreated Saudi bentonite added resulted in a reduction in the final values of the produced compressive as well as the cement density.

Table 4-7 Compressive strength results of cement with SB at various times

Time, hours	Compressive strength, Psi				
	G class cement	Base mix	1% SB	2% SB	3% SB
12:00	2487	2344	3044	2804	2673
18:00	2698	4965	6083	5480	5068
24:00	2744	5781	6613	5988	5643
48:00	-	6274	7030	6390	5940

Table 4-8 Time to achieve strength of 50, 500, and 2000 psi.

Compressive Strength(psi)	Time to reach 50, 500, 2000 psi strength				
	Cement class G	Base mix	1% SB	2% SB	3% SB
50	1:25	3:43	3:53	4:13	4:21
500	1:58	4:16	4:24	4:47	4:55
2000	7:03	10:52	8:23	10:13	10:29

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 4-8**).

UCA cement tests are conducted on a cement containing various percentages of untreated Saudi bentonite, and from the results we noticed that adding 1% untreated Saudi bentonite bwoc to the cement mix gave the smallest time period of 31 minutes in the case of strength development from 50 till 500 psi. In contrast, cement mix containing (2, and 3%) Saudi bentonite gave a transient time of (33, 34, minutes) respectively, which is higher compared with 1% untreated Saudi bentonite as shown in **Figure 4-22**.

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 we observed that 1% untreated Saudi bentonite gave the lowest time to reach 2000 psi compressive of around

8:23 minutes compared with the base as well as other cement mixes with higher percentages of untreated Saudi bentonite as shown in **Figure 4-23**.

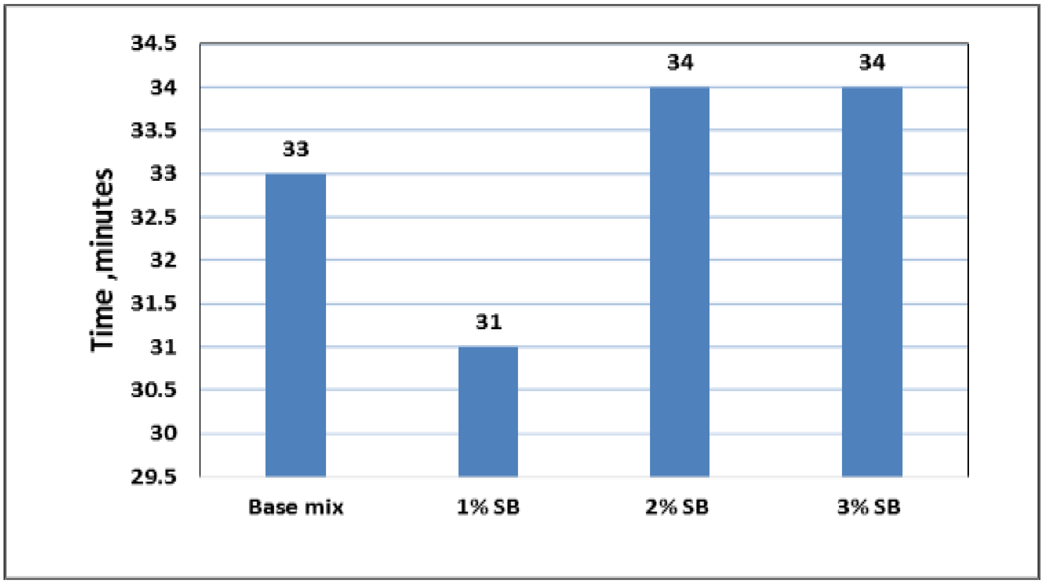

Figure 4-22 Transition time of the strength development between 50 and 500 psi for SB

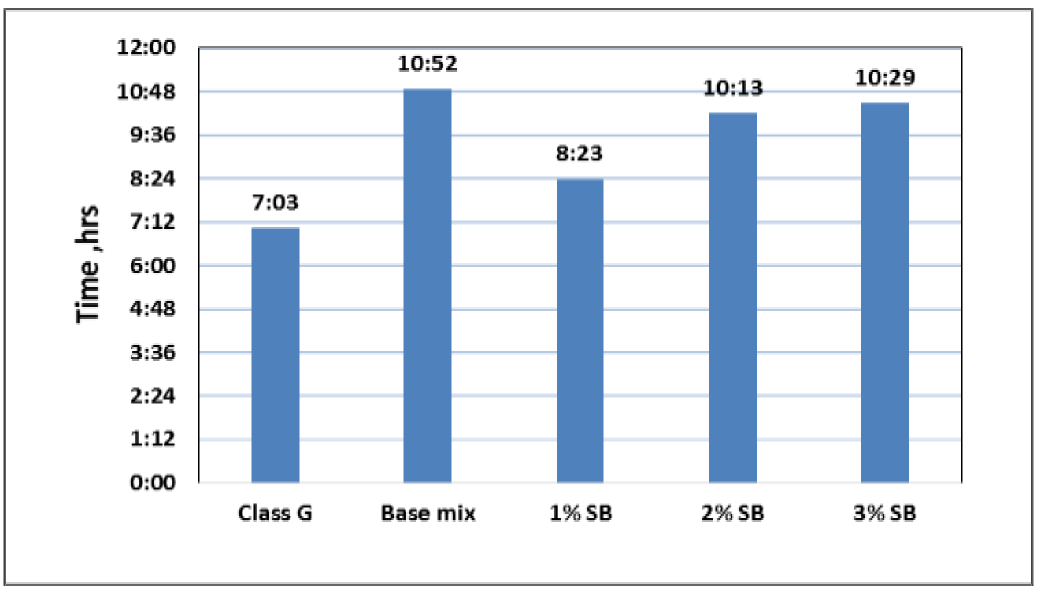

Figure 4-23 Time to achieve 2000 psi for SB

In short, the addition of 1% untreated Saudi bentonite to the cement mix resulted in an improvement in the early strength development, and gave the maximum compressive strength of 7030 psi after 48 hours curing (see **Figure 4-24**). After that we observed a reduction in the final compressive strength as the amount of untreated Saudi bentonite increased to reach a strength of around 6000 psi in the case of adding 3% SB.

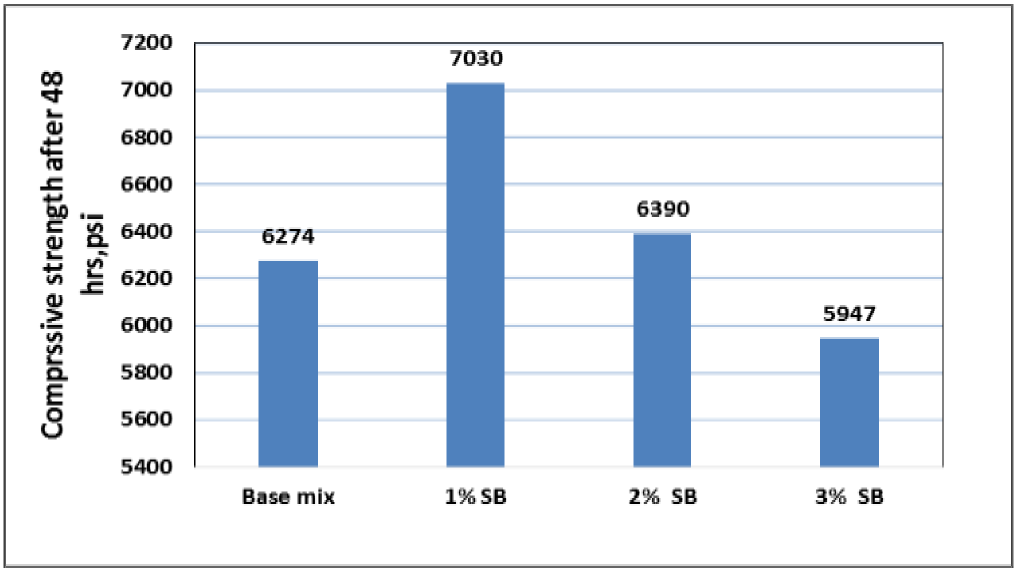

Figure 4-24 Final compressive strength after 48 hours

4.2.8 Effect of SB 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 porosimter/permeameter under a confining pressure of 500 psi.

Table 4-9 represents porosity and permeability cement results of simple class G cement, and 0, 1, 2, and 3% of SB after 24 hours curing.

Table 4-9 Porosity and permeability of cement with 0, 1, 2, and 3% of SB after 24 hours curing

Properties	Class G	Base mix	1% SB	2% SB	3% SB
Porosity %	36	31	30.795	30.372	30.006
Permeability md	0.358	0.0041	0.003	0.001	0.001

It was obvious that the addition of untreated Saudi bentonite to the cement mix resulted in a reduction the both porosity and permeability as in **Figure 4-25**, and **Figure 4-26**. Addition of untreated Saudi bentonite caused a slight reduction in the porosity, where all the results were around 30% 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 untreated Saudi bentonite were added to the cement mix compared with the cement base mix. This makes all the 1, 2, and 3% good choices with respect to permeability. In short, 1, 2 and 3% untreated Saudi bentonite percentages are all recommended for using with respect to permeability.

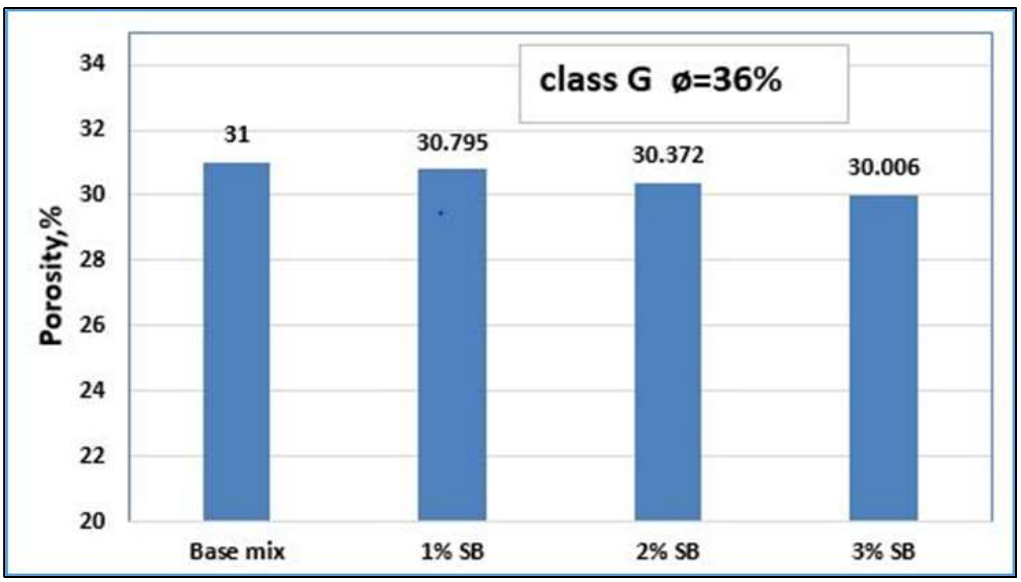

Figure 4-25 Porosity of cement with 0, 1, 2, and 3% of SB after 24 hours curing

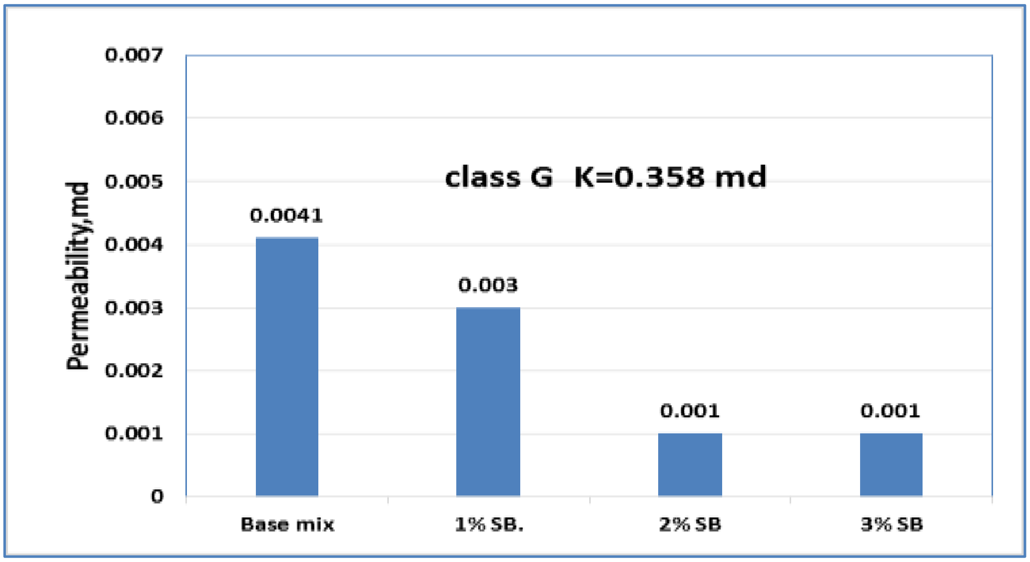

Figure 4-26 Permeability of cement with 0, 1, 2, and 3% of SB after 24 hours curing

4.2.9 Microstructural Analysis for SB Admixed with Cement

The cement composition is analysed 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 ($3CaO.Al_2O_3.CaSo_4.12\ H_2O$). **Figure 4-27, and Figure 4-28** shows the XRD patterns of neat simple class G cement cured under low and high temperature for 8 and 24 hours respectively.

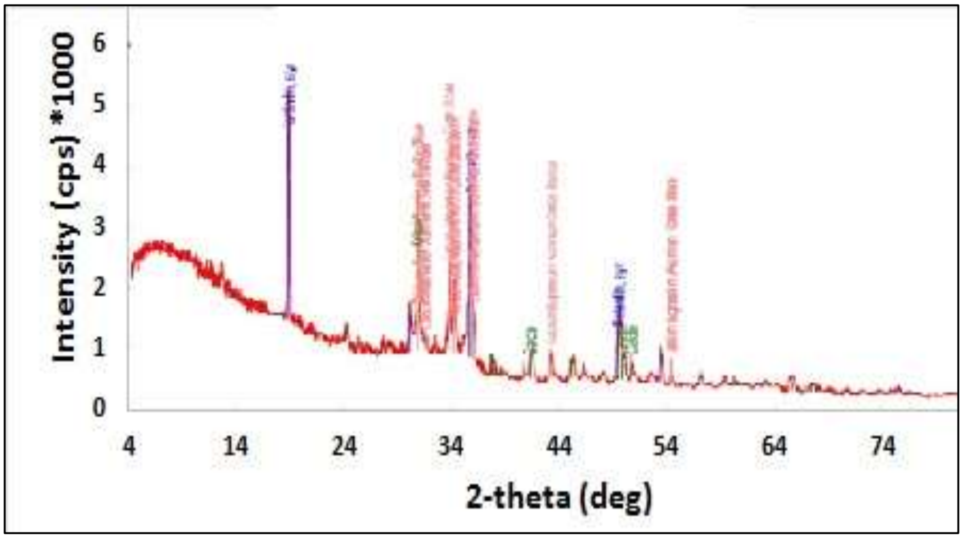

Figure 4-27 XRD spectra for simple class G cement cured ambient temperature for 8 hours

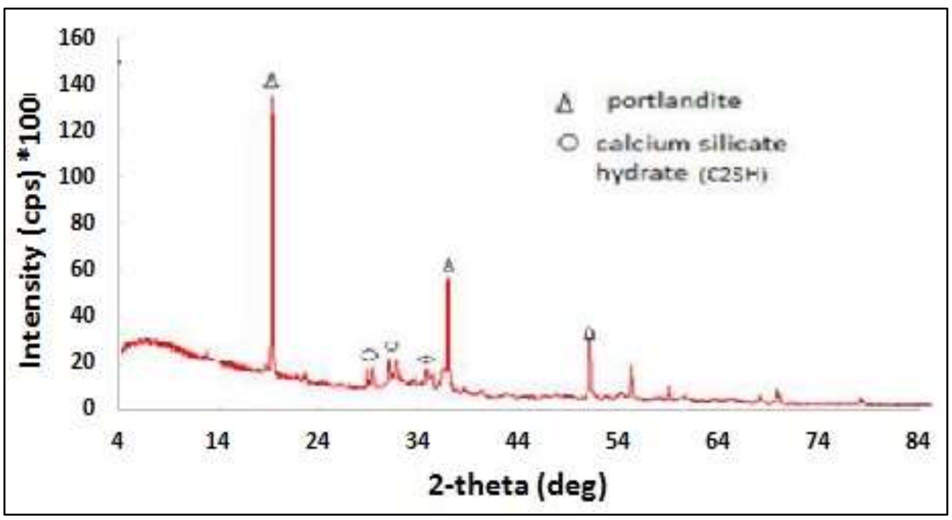

Figure 4-28 XRD spectra for simple class G cement cured HPHT for 24 hours

From XRD results above, we observed that temperature has a significant effect on the hydration of class G cement as well as its final products. When cement is hydrated at low temperature, portlandite (CH) is formed in the hydrated product, whereas calcium silica hydrate C-S-H is not detected due to amorphous behaviour under these conditions. On the other hand, when cement slurry is subjected to temperatures above 110^{o}C, C-S-H product starts to transform into C_2SH ($CaSiO_4.H_2O$), which is called α- dicalcium silicate hydrate. Under these conditions, the final product compositions were almost the same at different temperatures, but the quantity of each component can vary depending on the curing temperature used, which is indicated by a slight change in the peak intensity of the XRD spectra observed at different temperatures. In short, the major final product of cement is CH and CSH when cured at high temperature.

CSH is a fibre shaped material, where it can branch at every 0.5 µm length throughout the growth of the particles. These branches of the particles bond together and form a continuous three dimensional-network structure in the produced cement as shown in **Figure 4-29**. When the curing temperature used is below 110 ^{o}C, the main hydration products could bond together, and form the harden cement past with clear network structure characters. However, when the temperature exceeds 110 ^{o}C, the hydrated products transformed to high crystalline product C_2SH, where it has a shape of a plate-block as displayed in **Figure 4-30**. Also the microstructure of the final cement product which has a fibre network is transformed into a morphology of a pile plate block. Furthermore, C_2SH crystalline is weak and porous, and the bond stress between the blocks is weak. This results in a partial concentrate and cause a rise in the structural stress leads to a reduction in the compressive of the harden paste. Therefore, increasing the curing temperature resulted in a quick expand in the crystalline process, and cause many mass blocks to be piled up in the final composition structure.

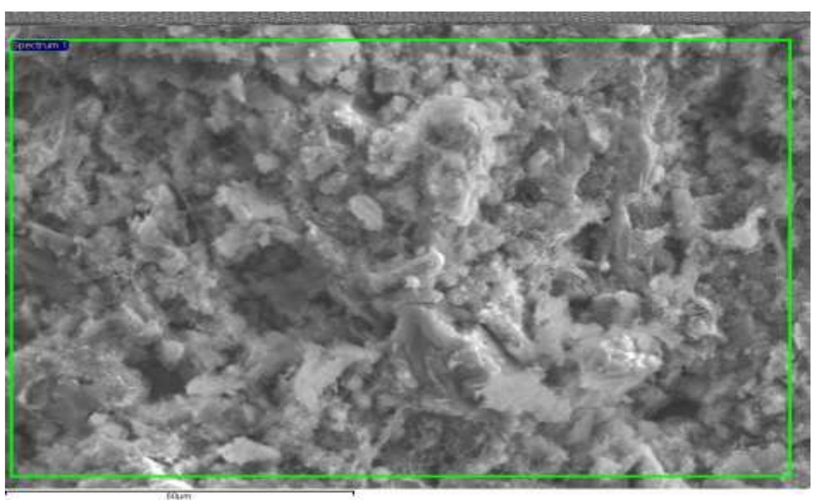

Figure 4-29 SEM photograph of hydration products of simple class G cement at ambient conditions for 8 hours

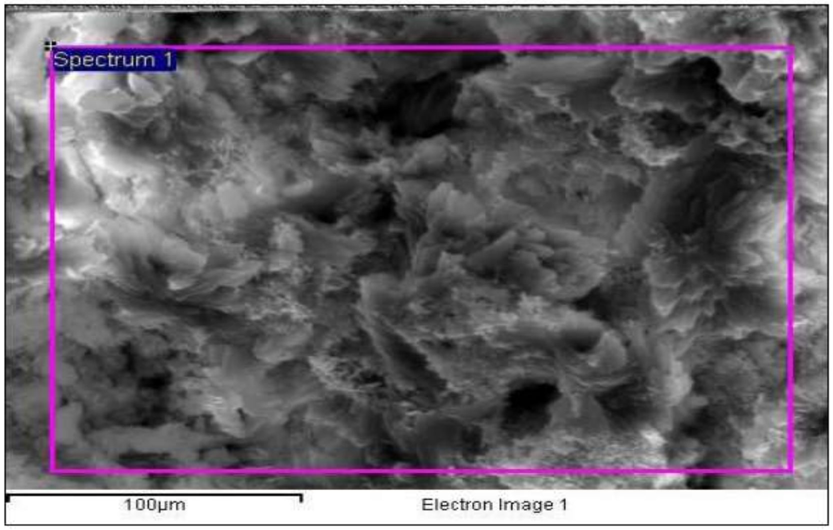

Figure 4-30 SEM photograph of hydration products of simple class G cement at HPHT for 8 hours

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 144 ^oC 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 4-31** shows XRD spectra of hydration products of cement with 0% untreated Saudi bentonite (base mix) added and cured at HPHT for 24 hours. We also observed 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.

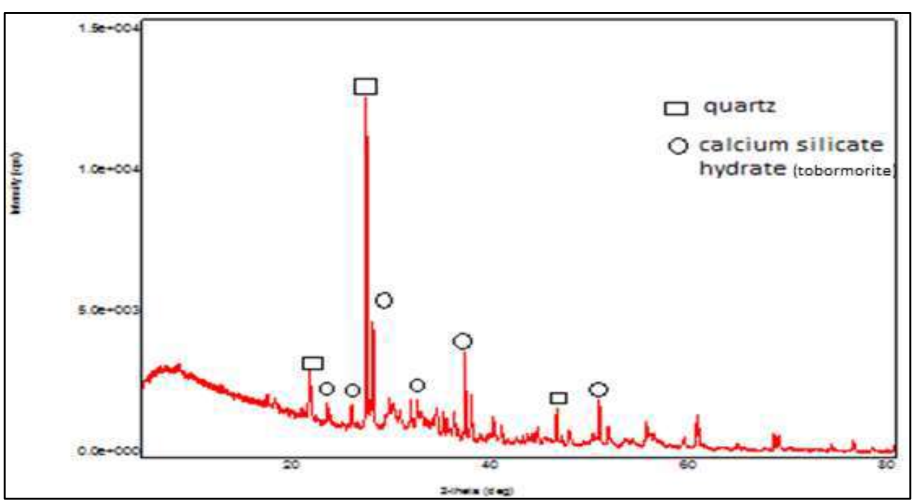

Figure 4-31 XRD hydration products with 0% untreated SB cured at HPHT for 24 hours

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 4-32** shows an SEM photograph of hydration products with 0% untreated Saudi bentonite cured at HPHT for 24 hours.

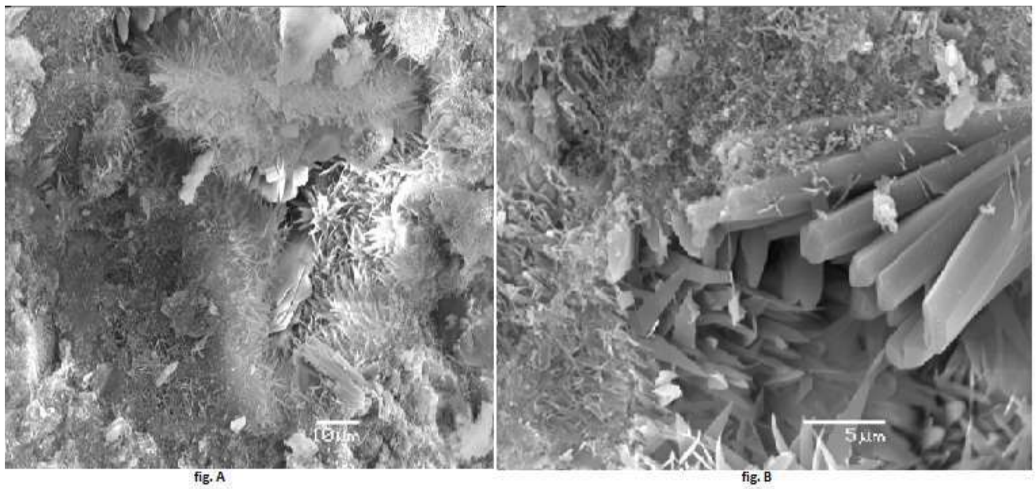

Figure 4-32 SEM photograph of hydration products with 0% untreated SB at HPHT for 24 hours

As untreated Saudi bentonite added to the cement, CH is disappearing, which indicated more polymerization occurred in the final hydrated product. Untreated Saudi bentonite is reacted in the solution and caused CH to transform into CSH as clear in the XRD pattern. When 1% untreated Saudi bentonite mixed with the cement, quartz as well as CSH appeared in high percentages in the finial cement product, and resulted in a strong cement structure. **Figure 4-33** shows XRD spectra of hydration products with 1% untreated Saudi bentonite cured at HPHT for 24 hours.

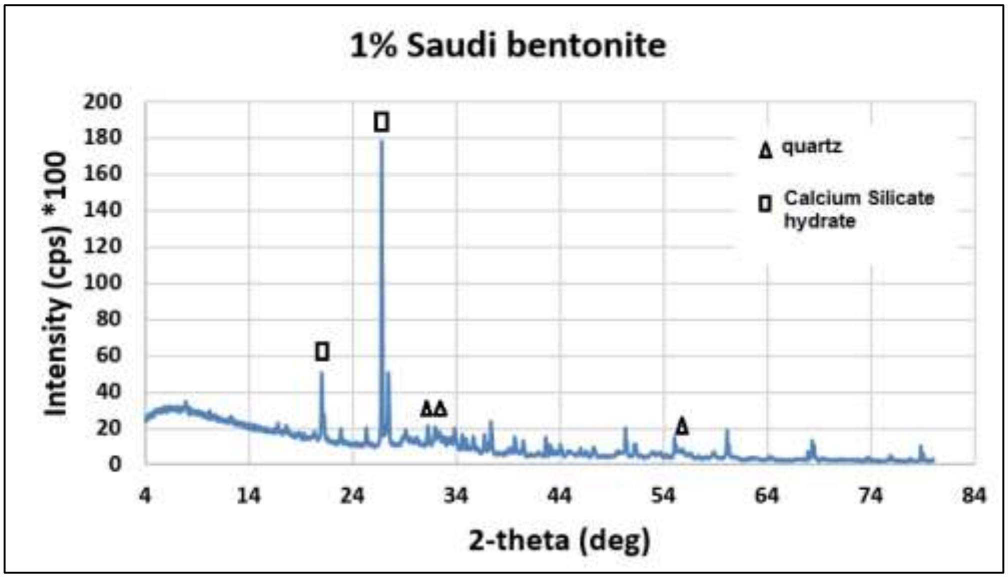

Figure 4-33 XRD hydration products with 1% SB cured at HPHT for 24 hours

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 4-34**. Cement slurry admixed with untreated Saudi bentonite resulted in big quantities of this favourable crystal due to the availability of silica in the mix. So, addition of untreated Saudi bentonite resulted in dense structure, and caused improvement in the final compressive strength. **Figure 4-35** shows SEM element analysis for 1% untreated Saudi bentonite 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.

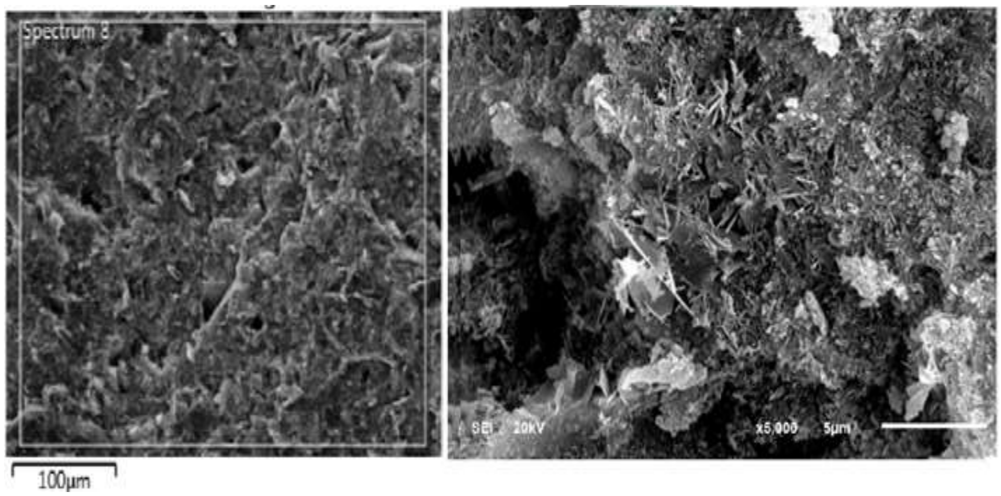

Figure 4-34 SEM photograph of hydration products with 1% untreated SB cured at HPHT for 24 hours

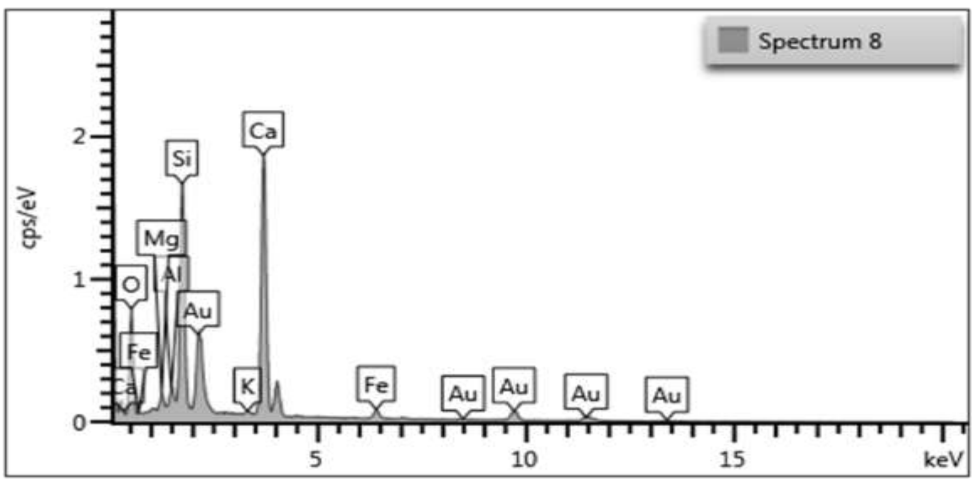

Figure 4-35 Hydration products (SEM) with 1% SB cured at HPHT for 24 hours

As we mention above, addition of untreated Saudi bentonite to the cement mix resulted in more polymerization as detected in the final products of the cement. In fact, if we compare the SEM images of a size of 5 microns for both cement systems, the cement base mix and the cement system containing 1% untreated Saudi bentonite, we observed that the cement base mix had more vugs and showed less polymerization compared with the cement system containing 1% untreated Saudi bentonite as shown in **Figure 4-36**.

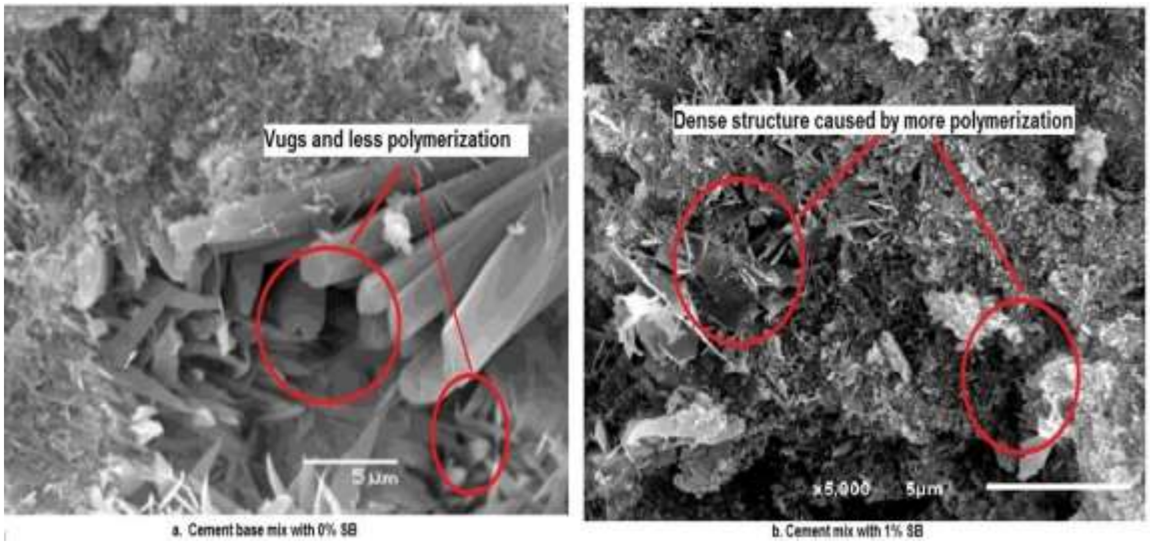

Figure 4-36 SEM 5 micron image comparing both cement base mix and cement system with 1% untreated SB

When 2% untreated Saudi bentonite added to the cement mix, the amount of producing quartz crystals was considerably high, and could be related to the hydration reaction between untreated Saudi bentonite and CSH, where huge amount of silica found in the mix as showing clearly in **Figure 4-37**. 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 4-38**). On the other hand, we observed that when 2% untreated Saudi bentonite mixed with the cement, the value of compressive strength decreased compared with 1% SB, which showed that the presence of quartz crystals in the harden cement is not the only proof of higher compressive strength. **Figure 4-39** illustrates the SEM element weight analysis for 2% Saudi bentonite cured at HPHT for 24 hours. It was obvious that the final cement product contains a higher weight percentage of silica, and calcium, which demonstrations the formation of higher percentages of CSH in the final harden cement.

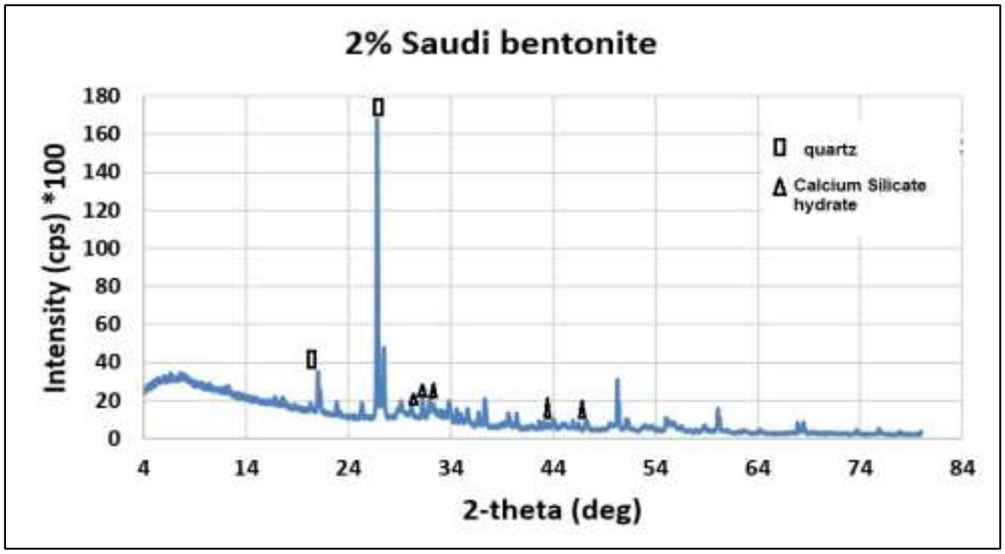

Figure 4-37 XRD hydration products with 2% untreated SB cured at HPHT for 24 hours

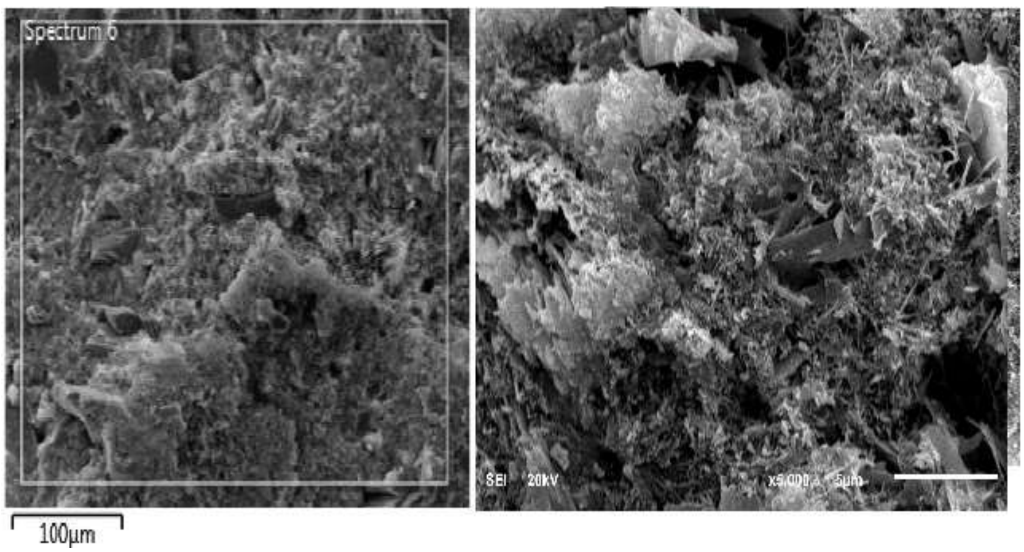

Figure 4-38 SEM photograph of hydration products with 2% untreated SB cured at HPHT for 24 hours

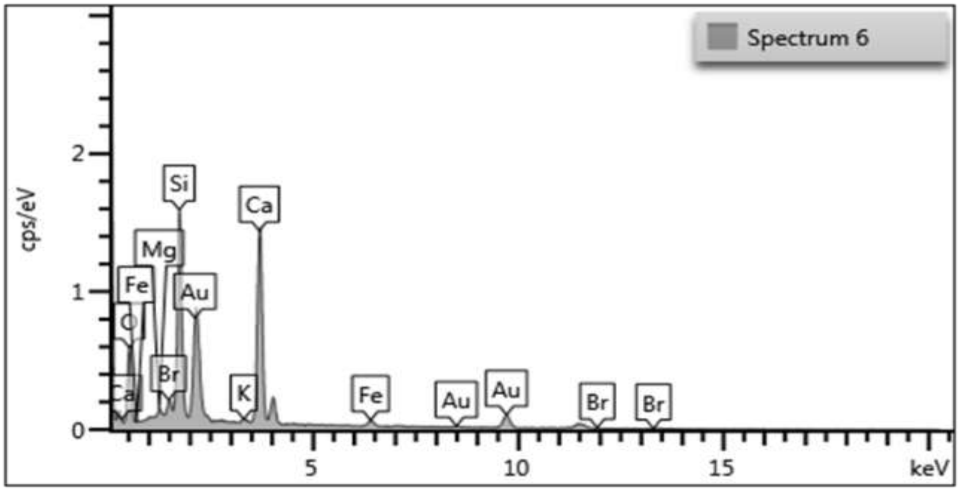

Figure 4-39 Hydration products (SEM) with 2% untreated SB cured at HPHT for 24 hours

It was obvious that the addition of 2% untreated Saudi bentonite by weight of cement caused more polymerization as detected in the final produced cement sheath. As a matter of fact, if we compare the SEM images of a size of 5 microns for both cement systems, the cement base mix and the cement system containing 2% untreated Saudi bentonite, it was observed that the cement base mix exposed more vugs, holes, and showed less polymerization compared with the cement system containing 0, and 1% as shown in **Figure 4-40**. On the other hand, vugs were started to appear with the 2% untreated Saudi bentonite addition compare with that of 1% SB which might be the reason of the compressive strength reduction measured using UCA and the crushing machine.

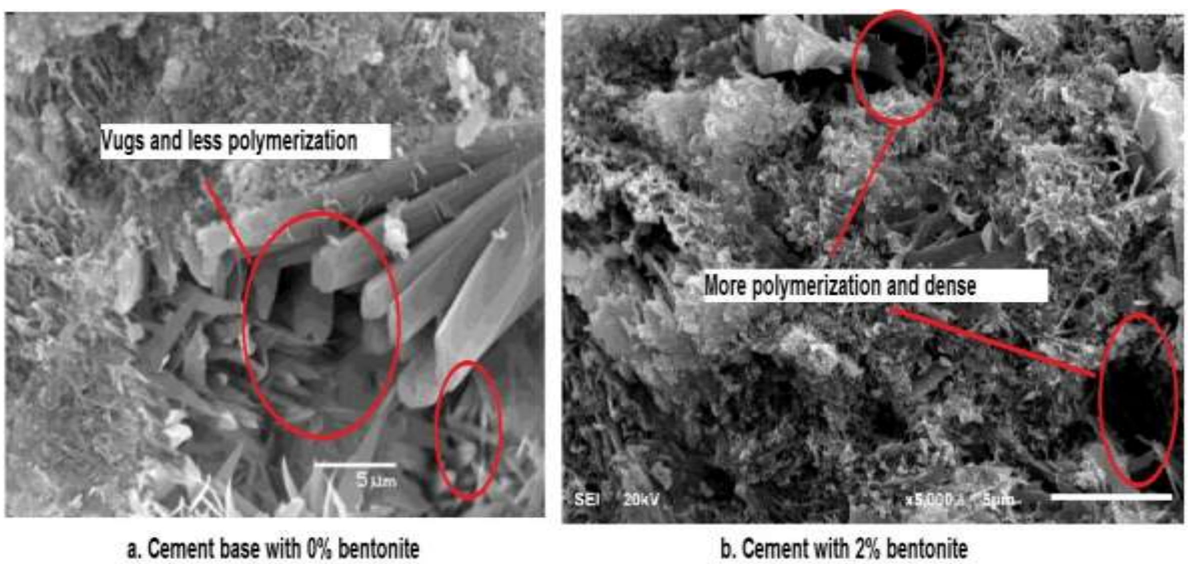

Figure 4-40 SEM 5 micron image comparing both cement base mix and cement system with 2% untreated SB

In the case of adding 3% untreated Saudi bentonite, the same cement behaviour was observed as described above. In fact, adding 3% untreated Saudi bentonite 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 4-41**). However, the compressive strength with 2% untreated Saudi bentonite is higher compared with 3%, which might be related to the reduction of the amount quartz crystals formed in the final harden cement paste. **Figure 4-42** represents the SEM results of 3% untreated Saudi bentonite 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 4-43** shows the SEM element weight analysis for 3% untreated Saudi bentonite cured at HPHT for 24 hours. It was obvious that the final cement products contain higher weight percentages of silica and calcium, which demonstrations the formation of more CSH in the final harden cement.

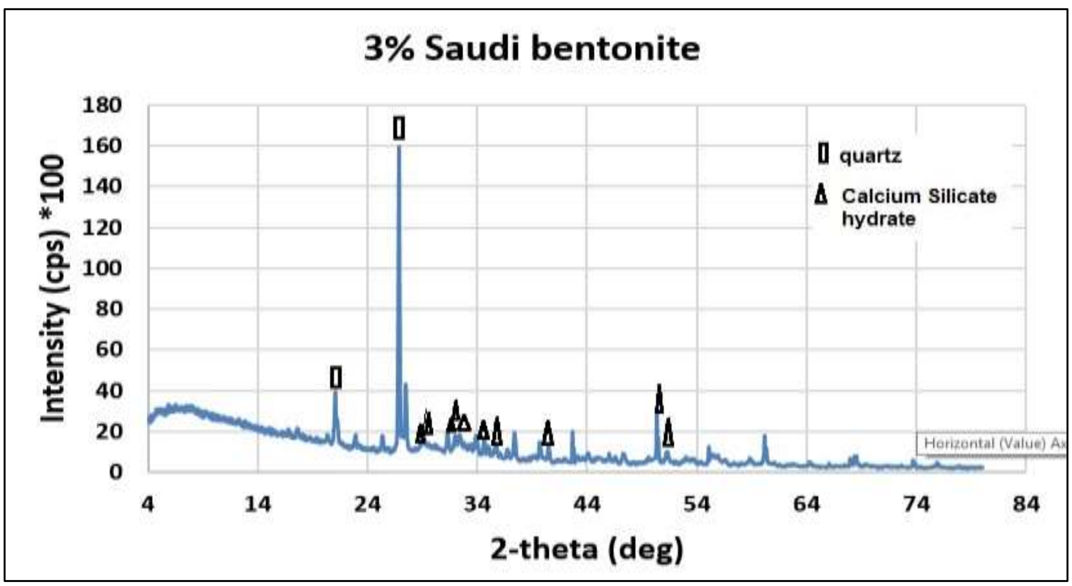

Figure 4-41 XRD hydration products with 3% untreated SB cured at HPHT for 24 hours

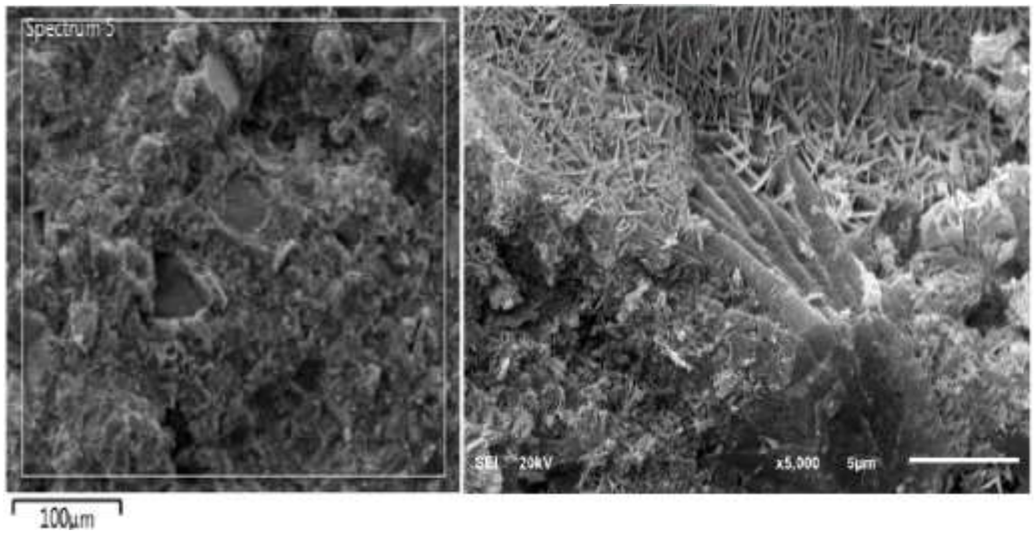

Figure 4-42 SEM photograph of hydration products with 3% untreated SB cured at HPHT for 24 hours

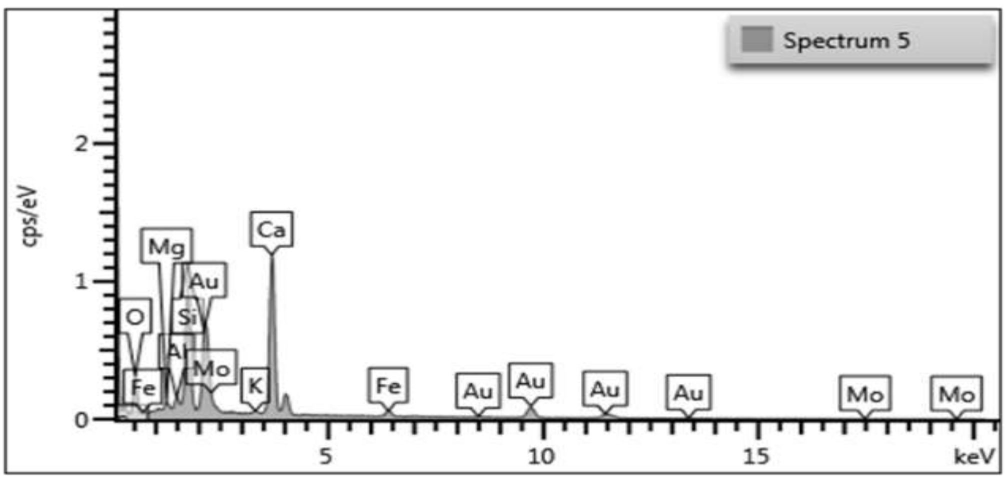

Figure 4-43 Hydration products (SEM) with 3% untreated SB cured at HPHT for 24 hours

The same trend was observed with the addition of 3% untreated Saudi bentonite to the cement mix as with the 2%. This addition caused a considerable polymerization to the cement mix as detected in the final produced cement sheath. In fact, if we compare the SEM images of a size of 5 microns for both cement systems, the cement base mix and the cement system containing 3% untreated Saudi bentonite, it was clear that the cement base showed less polymerization compared with the cement system containing 3% as shown in **Figure 4-44**. However, vugs as well as more holes were stared to appear with the 3% untreated Saudi bentonite addition compare with that of 0, 1, and 2%, which might be the reason of the compressive strength reduction.

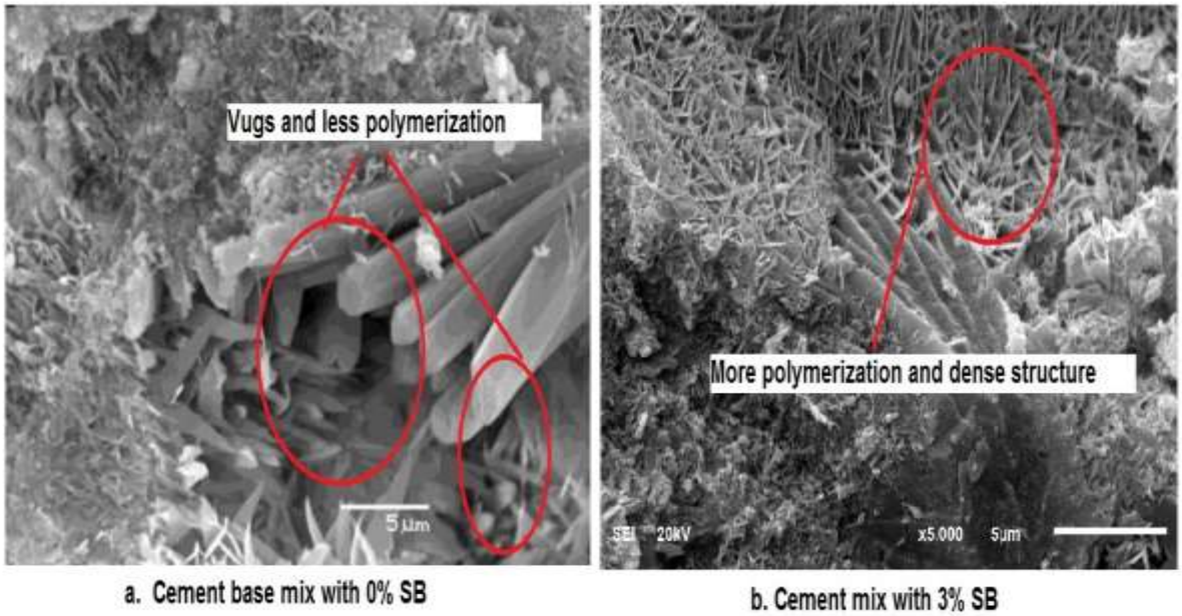

Figure 4-44 SEM 5 micron image comparing both cement base mix and cement system with 3% untreated SB

From the previous results discussed above, we found that addition of untreated Saudi bentonite to the mix resulted in extending the period of thickening from 8 hours with 1% SB up to 11 hours with 3% SB. Since the recommended thickening time for HPHT wells should be more than 5 hours, we selected 1% untreated SB as the best cement design in the case thickening time, since it satisfies the requirements. For the fluid loss, all of the results were almost the same of around 55 ml with a minor reduction in the fluid loss of the produced cement. As a result of this, 1%SB was selected as since all values are close. In the case of free water, all the systems showed zero free water. For the density, the reduction with 1%SB was 2.28% compared with 0% SB, and the more increase the amount of the added SB cause a slight reduction in the density, accordingly 1%SB is the optimum. For rheology, increasing the amount of SB caused an improvement in the plastic viscosity and yield point, but we choose 1% untreated SB, since it fulfils the requirements. Regarding compressive strength, it was obvious that 1 %SB gave the optimum strength, and showed improvement in the early as well as the rapid compressive strength compared with higher percentages of the untreated SB. This improvement was also observed in the case of a slight reduction in porosity, but significant reduction in the permeability was achieved with the 1% untreated SB. In short, 1% untreated SB is considered the optimum percentages added that give the best results compared with the higher percentages.

4.3 Effect of Optimum 1% Untreated SB with Nano Clay (Nc) on the Cement Properties

Nano clay will be added in three percentages of 0.5, 1, and 1.5 % to the optimum cement mix containing 1% untreated SB and the following cement properties will be discussed.

4.3.1 Effect of 1% SB with Various Percentages of Nc on the Cement Slurry Thickening Time

Thickening time cement test gives an indication about the time period the cement remains pumpable under certain conditions. In this case, the cement system containing 1% untreated Saudi bentonite with varied percentages of 0.5, 1, and 1.5% bwoc of Nano clay was prepared. Then, thickening time cement tests were conducted on the cement systems, and the time it took to thick was reported. **Figure 4-45, Figure 4-46,** and **Figure 4-47** display the thickening time for cement systems containing 1% SB with varied percentages 0.5 %, 1%, and 1.5 % bwoc Nano clay.

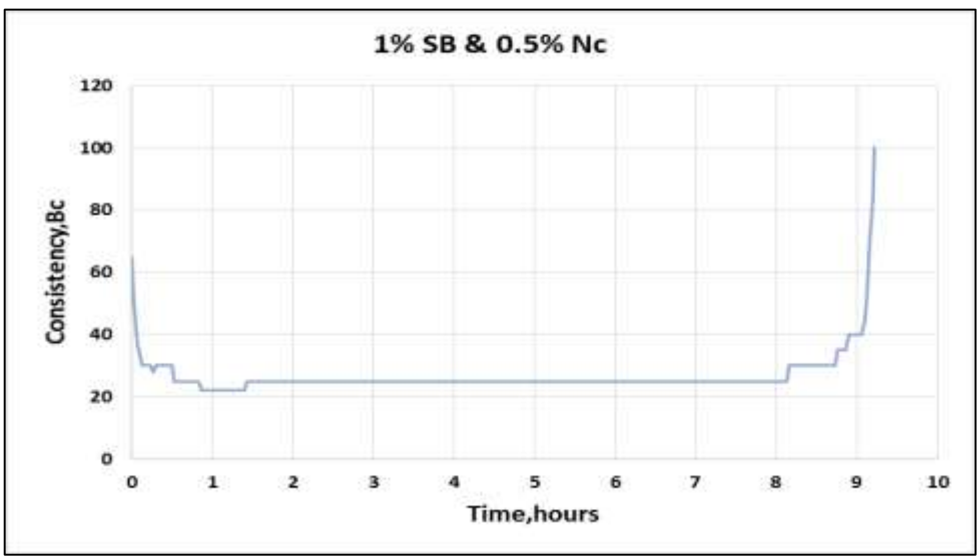

Figure 4-45 Thickening time cement test plot with 1 % untreated SB and 0.5% Nc

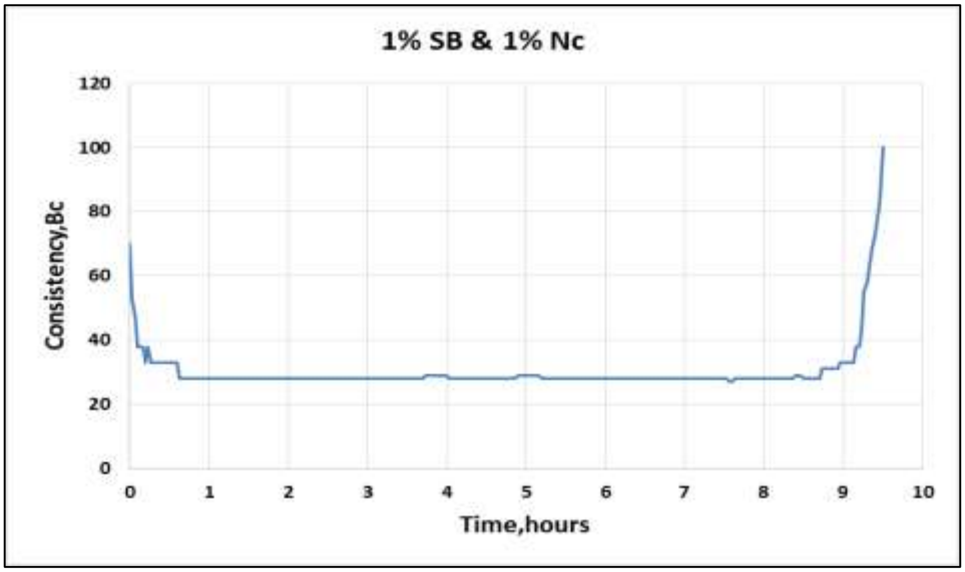

Figure 4-46 Thickening time cement test plot with 1 % untreated SB and 1% Nc

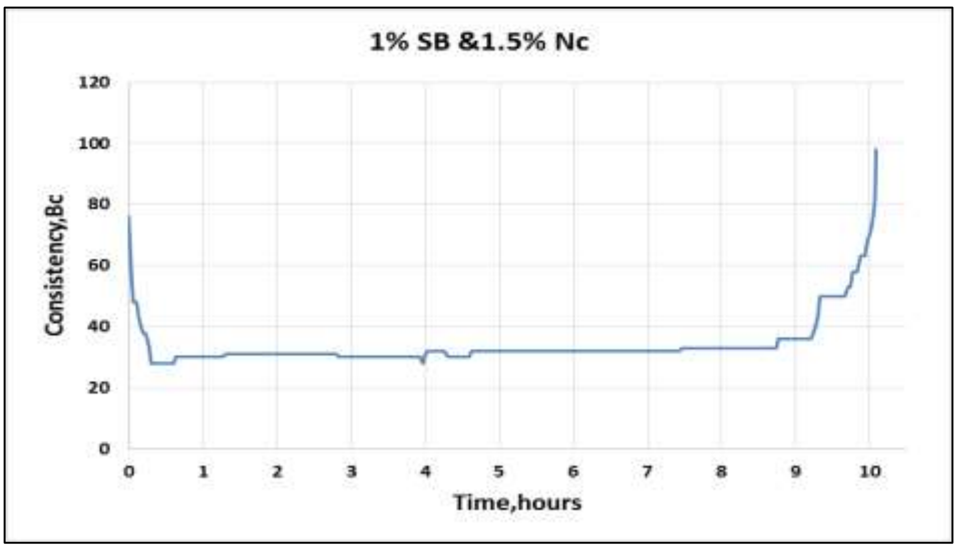

Figure 4-47 Thickening time cement test plot with 1 % untreated SB and 1.5% Nc

At the beginning of the tests, cements systems had a consistencies of 60, 65, 72, and 78 Bc for 1% untreated SB, and 1% untreated SB admixed with 0.5, 1, and 1.5% Nano clay respectively. After that, cement test conditions were applied, this value reduced and remain stable for a certain time until the 100 BC value is obtained, which was an indicator that the cement is now unpumpable. It is clear that the addition of Nano clay to the proposed cement system resulted in extending the thickening period time as shown in **Figure 4-48**, and **Figure 4-49**.

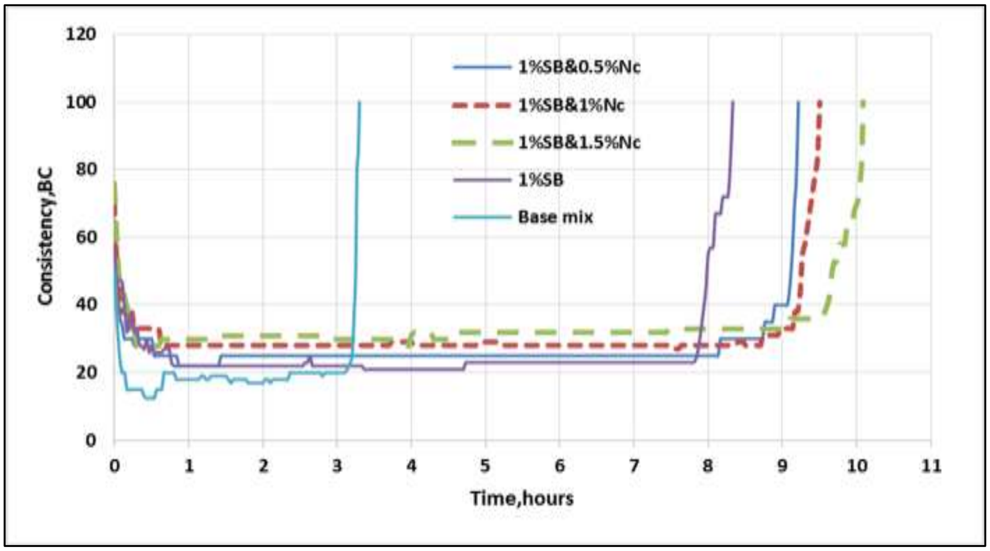

Figure 4-48 Cement thickening time of 1% SB mixed with 0.5, 1, and 1.5 % of Nc

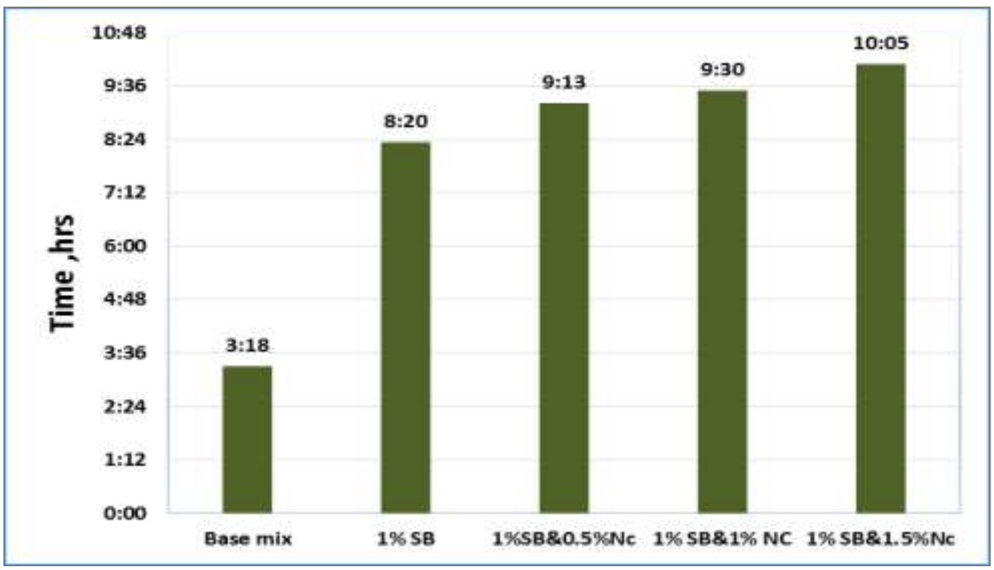

Figure 4-49 Cement thickening time of 1% SB mixed with 0.5, 1, and 1.5 % of Nc

At the beginning of the cement test, the consistencies of the tested samples were 60, 65, 72, and 78 Bc for 1% SB admixed with 0.5, 1, and 1.5% Nano clay respectively as shown in **Figure 4-50**. These consistency values were high, and then they decreased due to test conditions and finally stabilized. It was observed that it takes long time to reach 40 BC for all cement slurries. **Figure 4-51** displays the time to reach 40, 70,100 Bc consistencies. All test samples showed long time to reach consistency of 40 Bc, leaving only a short period of time to reach 100 Bc consistency and this is called the right angle set. In short, 40 Bc is an indicator showing that cement is becoming unpumpable since the cement has only a short time to reach 100 Bc.

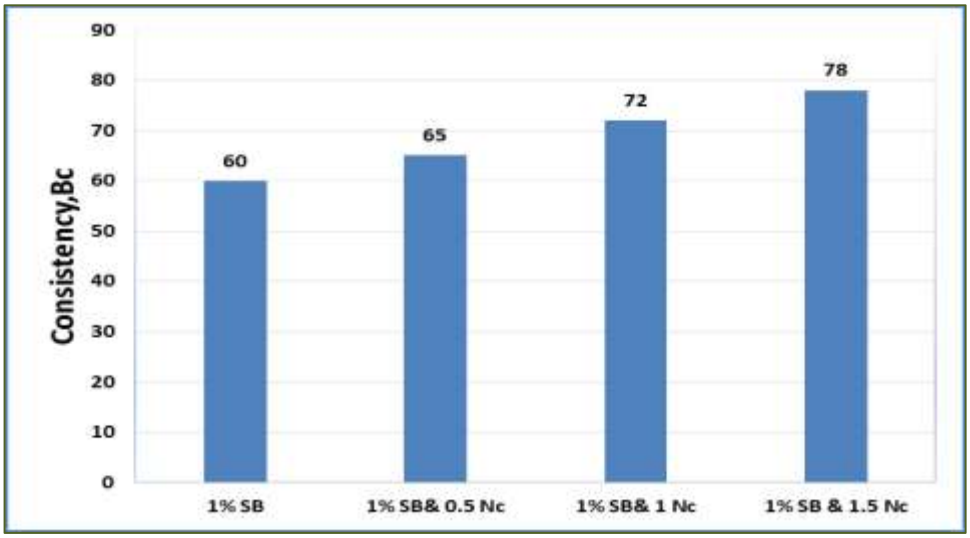

Figure 4-50 Consistency at the beginning of thickening time cement test

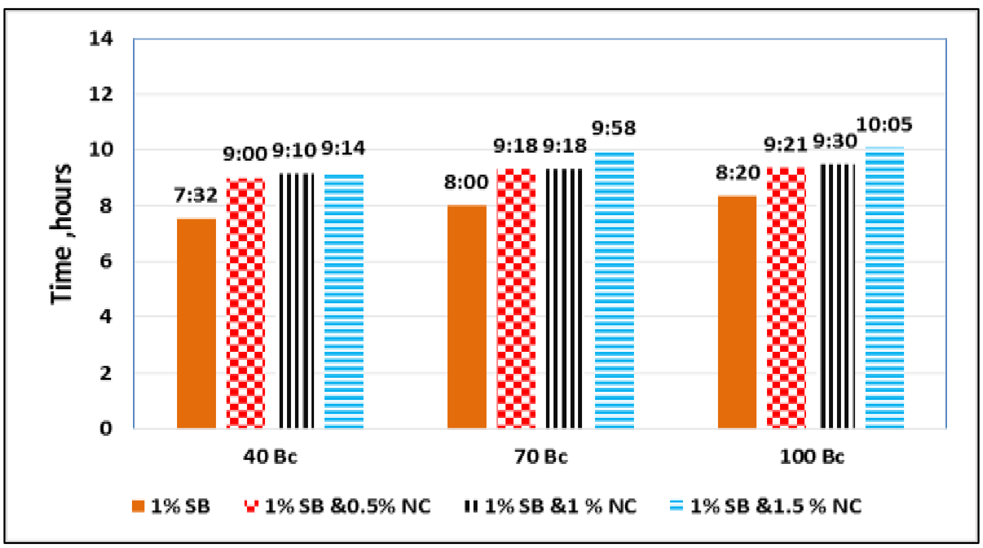

Figure 4-51 Time to reach 40, 70,100 Bc for 1% untreated SB with 0.5, 1, and 1.5 % of Nc

4.3.2 Effect of 1%SB with Various Percentages of Nc on Cement Fluid Loss

When Nano clay is added to the cement mix containing 1% SB, it results in a reduction in the amount of the produced fluid loss as shown in **Table 4-10**. **Figure 4-52** showed the trend of the fluid loss of 1% SB with 0.5, 1, and 1.5% Nc. There was a slight reduction in the cement fluid loss when Nano clay added to the cement mix having 1% untreated SB. The results obtained from these tests were almost the same of around 55 ml, and in the acceptable range of the industry. It is obvious that the higher the Nano clay percentages added, the more reduction in the fluid loss is achieved.

Table 4-10 Effect of 1% SB with 0.5, 1, and 1.5% Nc on the cement fluid loss

Fluid loss	1%SB	1%SB and 0.5%Nc	1%SB and 1%Nc	1%SB and 1.5%Nc
ml (API)	60	58	55	53

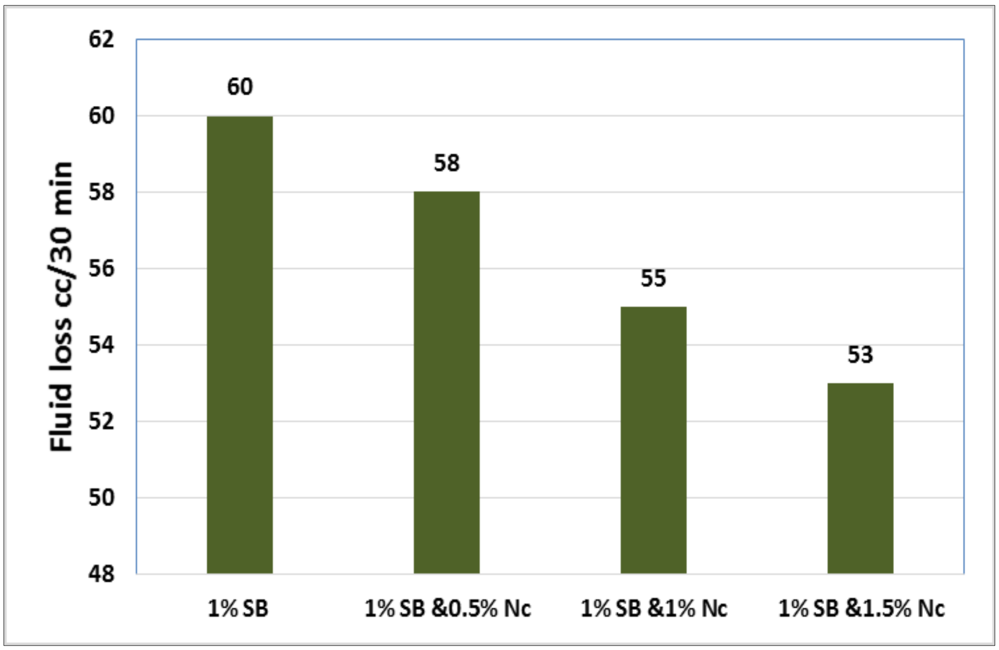

Figure 4-52 Effect of 1% SB with 0.5, 1, and 1.5 % Nc on the fluid loss

4.3.3 Effect of 1%SB with Various Percentages of Nc on Free Water Separation Test

Similar to what we did in the case of untreated Saudi bentonite, the effect of adding Nano clay was also investigated. Nano clay added in percentages of 0.5, 1, and 1.5% bwoc to a cement mixture containing 1% untreated SB, and the free water cement test values after two hours aging was reported. **Table 4-11** displays the results of a cement free water test of 1% untreated SB admixed with 0.5, 1, and 1.5% of Nano clay. It was clear that the produced cement slurry had no free water accumulated at the top of the cement as observed in **Figure 4-53**. The reason of no free water was observed when untreated Saudi bentonite and Nano clay mixed with the cement is because these materials absorb the water and swell due to their clay behaviour. Consequently, the water is enclosed and blocked between the clay layers of both untreated SB and Nano clay resulting in disappearing of the water separation problem. In addition, Nano clay as well as untreated SB did not cause any distribution in the particle suspension property in the cement.

Table 4-11 Free water of 1% untreated SB with 0.5, 1, and 1.5% Nc with cement

Free water	1% SB	1% SB and 0.5% Nc	1% SB and 1% Nc	1% SB and 1.5% Nc
ml/250ml	0	0	0	0

Figure 4-53 Free water with Nano clay after 2 hours aging

4.3.4 Effect of 1%SB with Various Percentages of Nc on the Cement Density

Well control is one of the most important issues that engineers should carefully consider during drilling and cementing practice. A pressurized cement balance is normally used to measure the cement density in field as well as in the laboratory. A cement system with 1% untreated SB admixed with 0.5, 1, and 1.5% of Nano clay were prepared and the density of the produced cement was measured using a pressurized cement balance. **Table 4-12** shows the density of cement with 1% SB admixed with 0.5, 1, and 1.5% of Nano clay.

Table 4-12 Cement density with 1% untreated SB and 0.5%, 1, and 1.5% of Nc

Cement slurry	Density (Ib/gal)
1% SB	16.52
1% SB and 0.5% Nc	16.38
1% SB and 1% Nc	16.33
1% SB and 1.5% Nc	16.25

It was clear that inserting Nano clay to the cement containing 1% untreated SB caused a slight reduction the produced cement slurry density compared with the base mix, and cement with 1% untreated SB. As we observed above, cement with 1% untreated SB and the different percentages of Nano clay had a density of around 16.3 Ib/gal. Addition of Nano clay to the cement caused a slight reduction in the density, where the 1% SB with 1% Nano clay gave a density of 16.38 Ib/gal with a drop of 0.85% from 1% SB cement admix. It can be concluded that addition of Nano clay to the cement does not have a significant effect on the cement density and all densities were almost close to each other as shown in **Figure 4-54**.

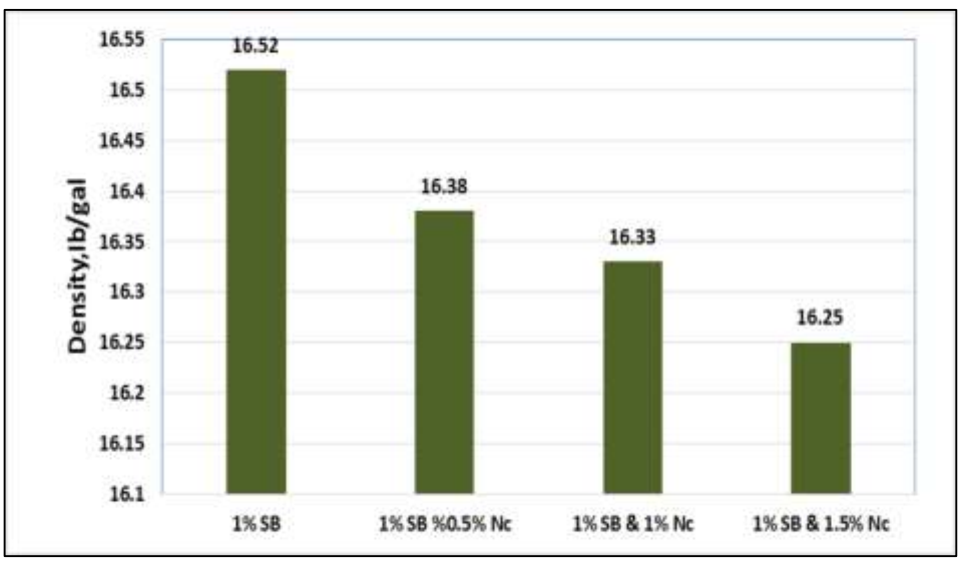

Figure 4-54 Density of cement with 1% untreated SB and 0.5, 1, and 1.5 % of Nc

4.3.5 Effect of 1% SB with Various Percentages of Nc on the Cement Rheology

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. **Table 4-13** represents the plastic viscosity and yield point of cement containing 1% untreated SB and 0.5, 1, and 1.5% of Nano clay. The table shows that adding Nano clay with 0.5% did not affect the rheological properties very much and 300 RPM speed gave a reading lower than 300 reading the rheometer gauge, in which it is recommended to use in oil industry application. However, 1 and 1.5% Nano clay resulted in an increase in the plastic viscosity of around 313, and 356 cp respectively as illustrated clearly in **Figure 4-55**. This enhancement in the plastic viscosity plays an important role in mud removal during cementing and also in the case of carrying lost circulation materials such as fibres as well as large particles. In addition, there was a slight effect on the yield point in which the results were almost close to each other as in **Figure 4-56**.

Table 4-13 Plastic viscosity and yield point of cement with 1% SB with 0.5, 1, and 1.5 % of Nc

Properties	1% SB	1% SB and 0.5% Nc	1% SB and 1% Nc	1% SB and 1.5% Nc
Plastic viscosity	252.7	283.2	313.4	356
Yield point lb/100 ft^2	8.27	8.4	9.04	10.17

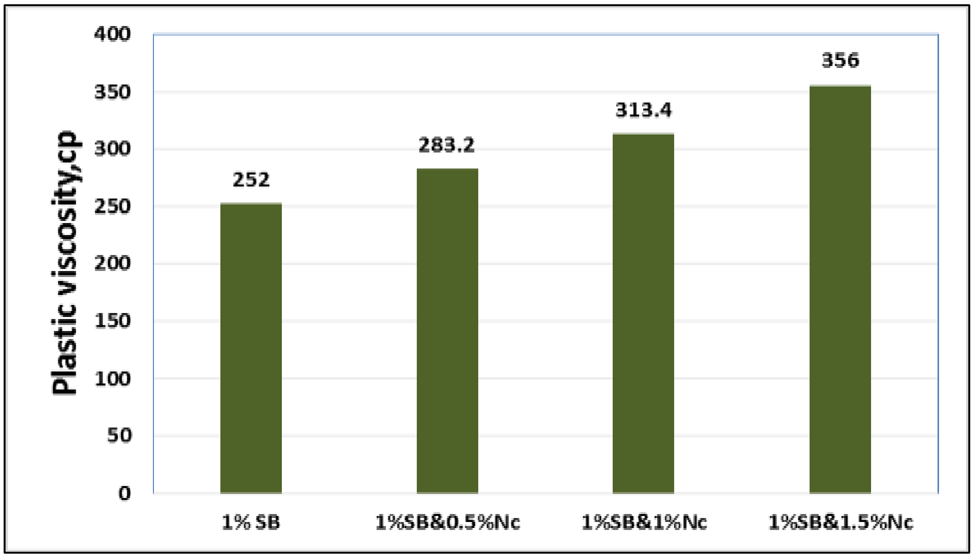

Figure 4-55 Plastic viscosity of cement with 1%SB and 0.5, 1, and 1.5 % Nc

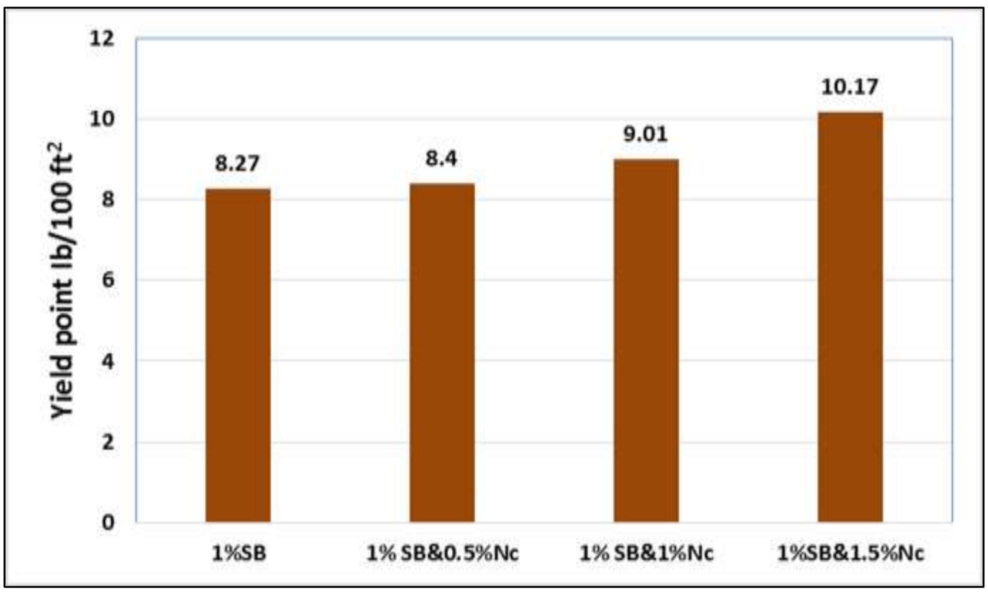

Figure 4-56 Yield point of cement with 1% SB with 0.5, 1, and 1.5 %Nc

4.3.6 Effect 1%SB with Various Percentages of Nc on the Gel Strength of Cement

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.

Table 4-14 shows gel strength of cement with 1% untreated SB mixed with various percentages of 0.5, 1, and 1.5% bwoc of Nano clay conducted using Ofite and fann viscometer. From the table, we observed that the gel strength had increased with increasing amount of Nano clay added (see **Figure 4-57**). It is obvious that the addition of Nano clay did not have a noticeable effect on the 10-sec gel, and the values are almost close to each other as in 1% untreated SB, and 1% untreated SB with 0.5, and 1% of Nano clay. However, higher percentages of Nano clay cause a rise in the 10-sec gel. For instance, 1.5% produces 11 $Ib_f/100\ ft^2$ get strength as explained below. For the 10-min gel, there was a gradual increase in the gel from 35 up to 42 $Ib_f/100\ ft^2$ with 1.5% Nc which are considered higher compared with the 1% untreated SB which have a 10-min gel of around 25 $Ib_f/100\ ft^2$.

Table 4-14 Gel strength of 1% SB with 0.5, 1, and 1.5% Nano clay in the cement

Gel strength Ib/100 ft^2	1% SB	1% SB and 0.5% Nc	1% SB and 1% Nc	1% SB and 1.5% Nc
10-sec gel	8	8.5	9	11
10-min gel	25.5	35	38	41.5

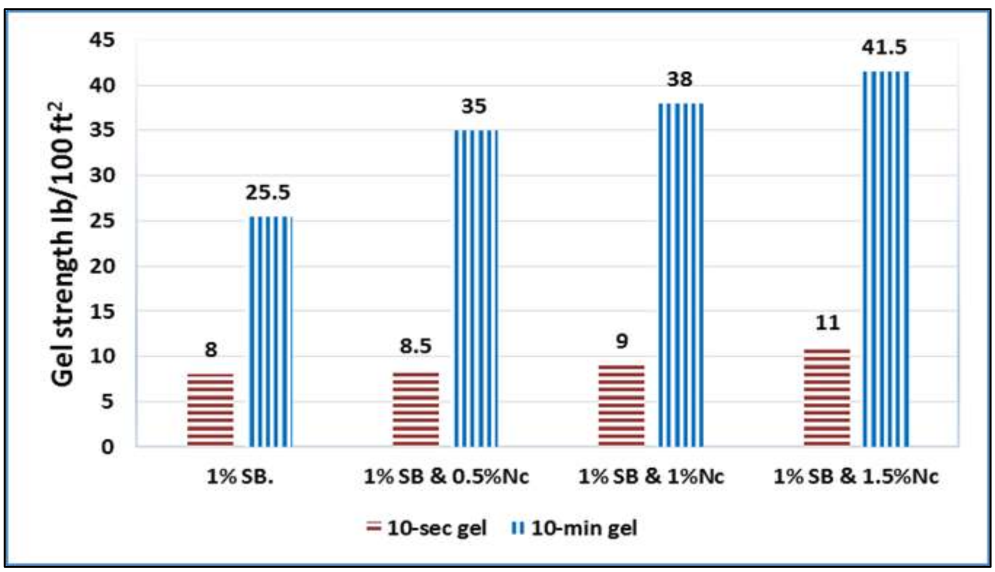

Figure 4-57 Gel strength of 1% untreated SB with 0.5, 1, and 1.5% Nc in the cement

4.3.7 Cement Compressive Strength

4.3.7.1 Effect of 1%SB with Various Percentages of Nc on Cement Compressive Strength by Crushing

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 1% untreated SB, and 1% untreated SB admixed with different percentages 0.5, 1, and 1.5% of Nano clay were prepared and tested for strength under both HPHT conditions. It was clear that adding Nano clay to the cement mix containing 1% untreated SB resulted in considerable improvement in the final compressive strength compared with the cement with 1% untreated SB as shown **Table 4-15**. At the beginning, adding 0.5% Nano clay caused significant reduction in the strength, and the recorded strength was 6300 psi. Increasing the amount up to 1% Nano clay caused a jump of strength up to 6550 compared with the other percentages when samples aged for 24 hours. Extra addition of the Nano clay to the cement mix containing 1% untreated SB caused reduction the final strength achieved. For example, 1.5% Nano clay gave compressive strength of around 5,900 psi after 24 hours. In short, 1% untreated SB admixed with 1% Nano clay caused significant improvement in strength compared with other percentages added as shown in **Figure 4-58**.

Table 4-15 Compressive strength of cement with 1% SB and 0.5, 1, and 1.5% Nc by crushing after 24 hours

Sample	1% SB	1% SB and 0.5% Nc	1% SB and 1%Nc	1% SB and 1.5% Nc
1	7641.5	5742	6713	6597.7
2	6307.5	6742	6336	5724.5
3	6592	-	-	5734.75
Average	**6846.9**	**6242**	**6525**	**5909**

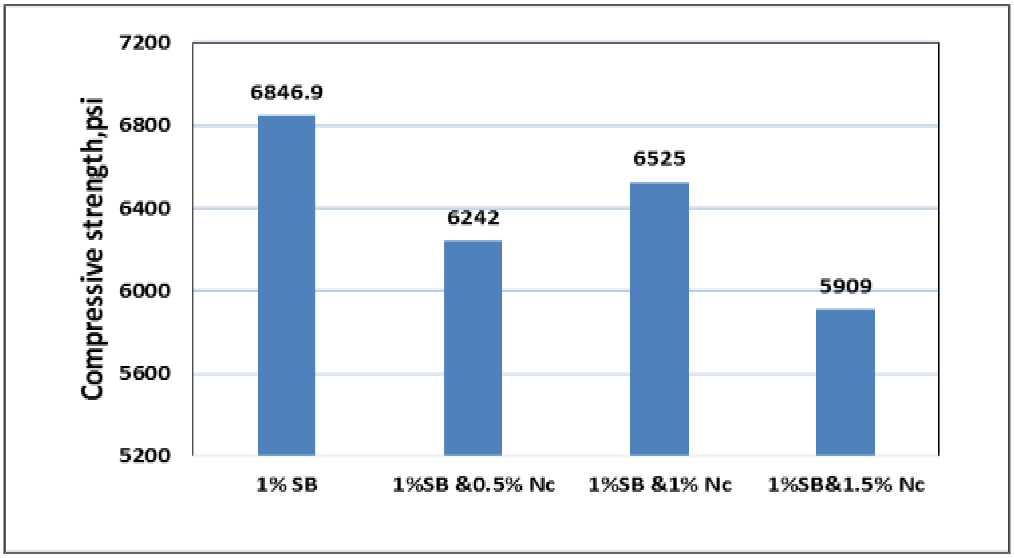

Figure 4-58 Compressive strength of cement with 1% SB and 0.5, 1, and 1.5% Nc by crushing after 24 hours

4.3.7.2 Effect of 1%SB with Nano Clay on the Compressive Strength by Sonic Waves

The compressive strength is a key factor, especially in cases of cementing and drilling ahead. Four cement systems, consists of 1% untreated SB, and 1% untreated SB admixed with different percentages i.e. 0.5, 1, and 1.5% of Nano clay were prepared and tested for strength by using a sound wave method under high pressure and temperature conditions. **Figure 4-59**, **Figure 4-60**, and **Figure 4-61** show the compressive strength results from UCA for cement containing 1% untreated SB, and 1% untreated SB admixed with 0.5, 1, and 1.5% Nano clay of the cement.

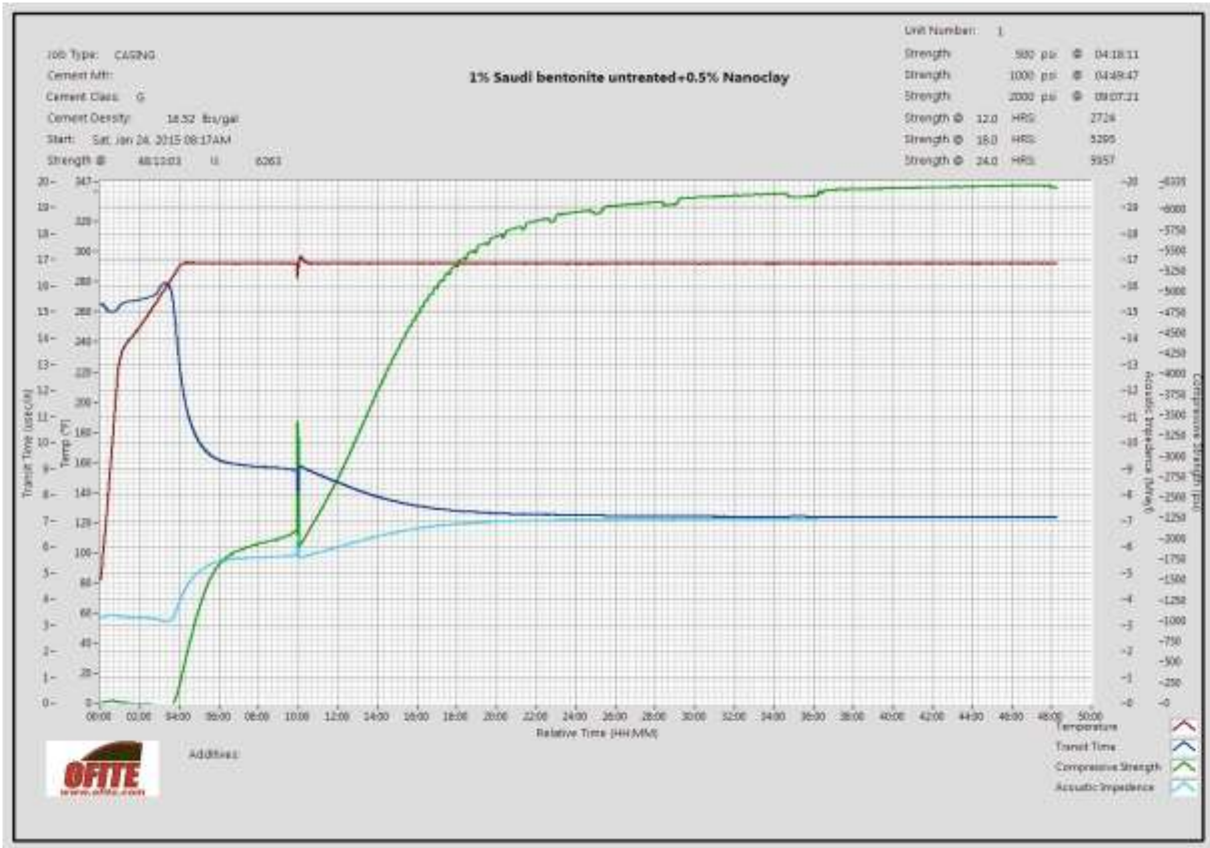

Figure 4-59 Strength development of a cement with 1% untreated SB and 0.5 % Nc by UCA for 48 hours

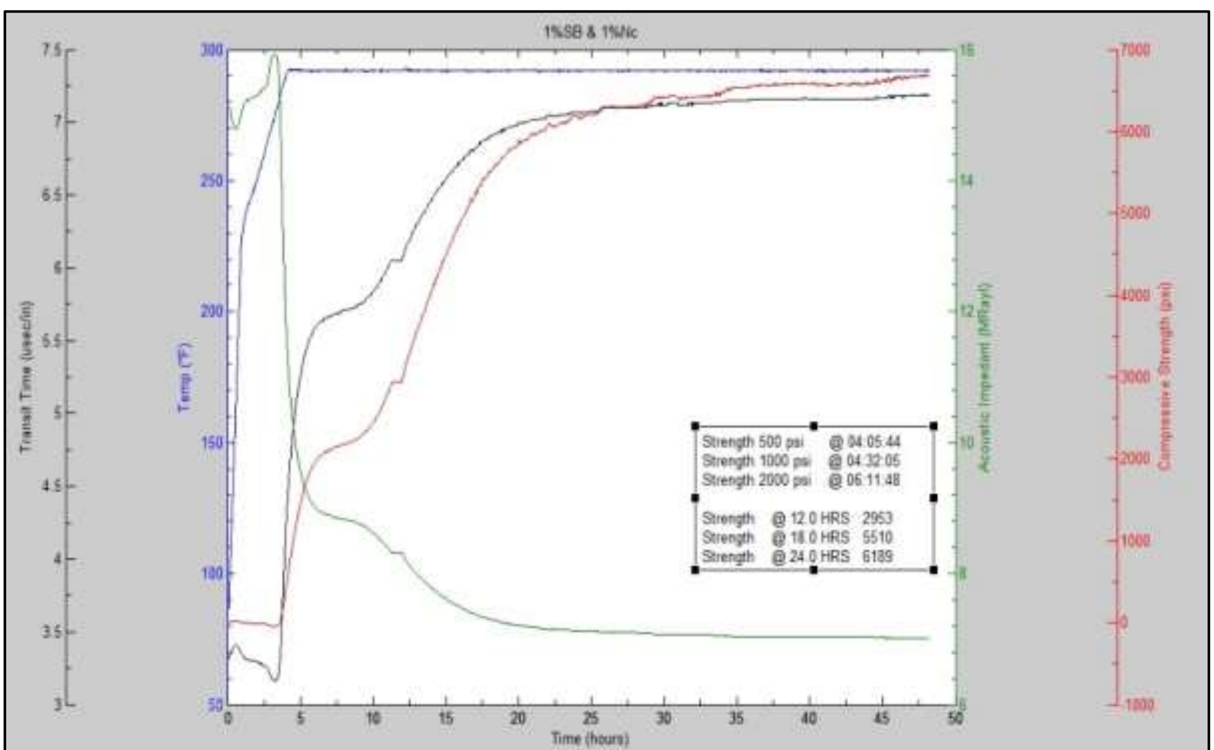

Figure 4-60 Strength development of a cement with 1% untreated SB and 1% Nc by UCA for 48 hours

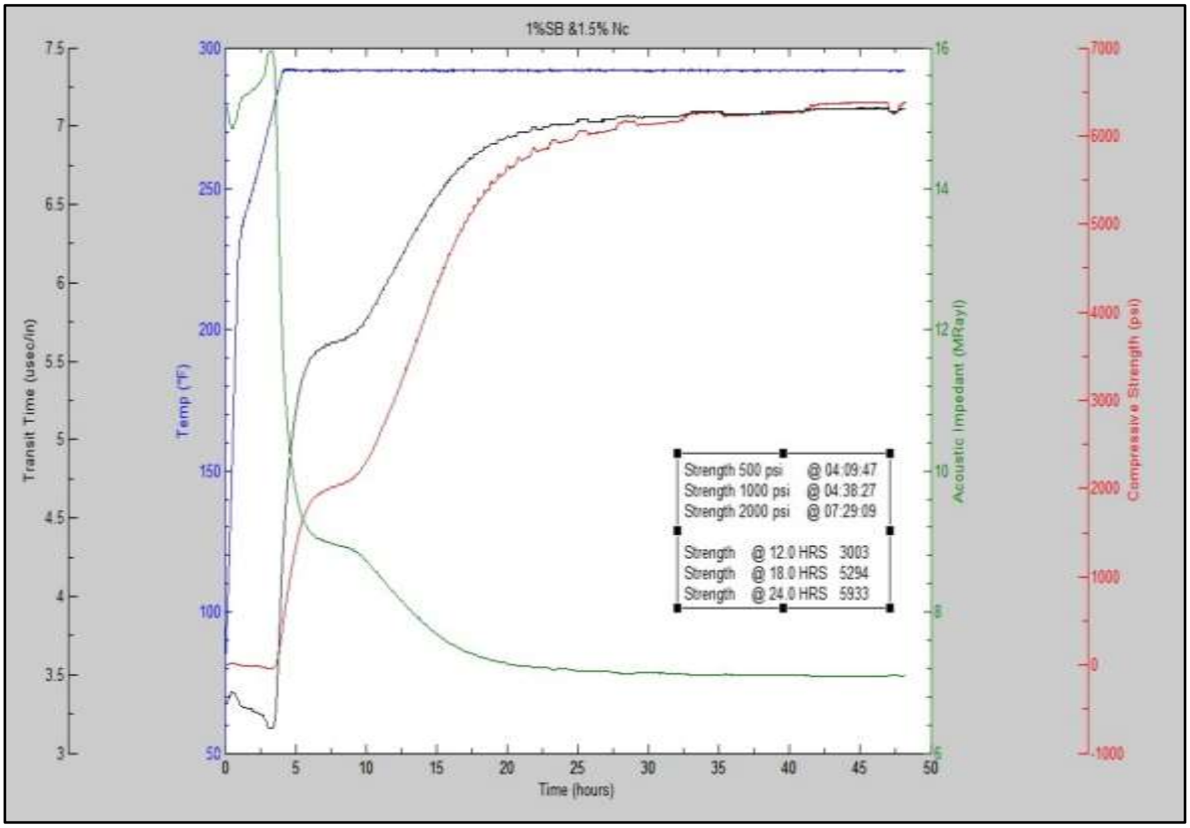

Figure 4-61 Strength development of a cement with 1% SB and 1.5% Nc by UCA for 48 hours

In fact, during the oil well cementing, exact known of the time period to reach a compressive strength of 50 and 500 psi are important, and this time period should be minimized. Consequently, trying to reduce the time spent in waiting for the cement to solidify and hardening (WOC) time prior to resuming and drilling ahead is needed (Coker et al., 1992). Nano clay was added in percentages of 0.5, 1, 1.5% bwoc to see the effect of its combination with 1% untreated Saudi bentonite on the cement. **Table 4-16** shows compressive strength results of 1% untreated SB, and 1% untreated SB admixed with of 0.5, 1, and 1.5% of Nano clay at various times. The addition of 0.5% Nano clay result in a rapid of compressive when compared with the addition of 2% untreated SB, but still not as quick as 1% untreated Saudi bentonite as shown in the table. **Table 4-17** shows the time the cement takes to achieve a strength of 50, 500, 2000 psi.

Table 4-16 Compressive strength results of 1% untreated SB with 0.5, 1, and 1.5% Nc at various times

Time, hours	Compressive strength, Psi				
	Base mix	1%SB	1%SB and 0.5%Nc	1%SB and 1%Nc	1%SB and 1.5%Nc
12:00	2344	3044	2725	2953	3003
18:00	4965	6083	5294	5510	5297
24:00	5781	6613	5957	6189	5933
48:00	6274	7030	6285	6688	6371

Table 4-17 Time to achieve a strength of 50, 500, 2000 psi.

Compressive Strength, psi	Time to reach 50, 500, 2000 psi strength				
	Base mix	1%SB	1%SB and 0.5%Nc	1%SB and 1%Nc	1%SB and 1.5%Nc
50	3:43	3:53	3:49	3:42	3:43
500	4:16	4:24	4:18	4:05	4:09
2000	10:52	8:23	9:07	6:11	7:29

The addition of 1% Nano clay to the cement mix containing 1% untreated SB showed an accelerating effect compared with the pure mix with 1% untreated SB as explained in **Table 4-17**. From **Figure 4-62**, and **Figure 4-63**, it was observed that addition of Nano clay to the cement mix containing 1% untreated SB resulted in a reduction in the transient time from 50 psi and reach 500 psi. It was clear that the best and the lowest time was obtained with 1% untreated SB admixed with 1% Nano clay which was 23 minutes. The recommend transient time in the cementing application should be less than 30 minutes to prevent gas migration. Also in the case of reaching strength of 2000 psi, addition of Nano clay caused reduction in the waiting time (WOC), and the optimum mix design was 1% untreated SB admixed with 1% Nano clay, where it reached this strength after 6:11 minutes which is considered the lowest time compared with all proposed cement systems. Furthermore, the highest final strength reported was also obtained with this cement mix design as shown in **Figure 4-64**.

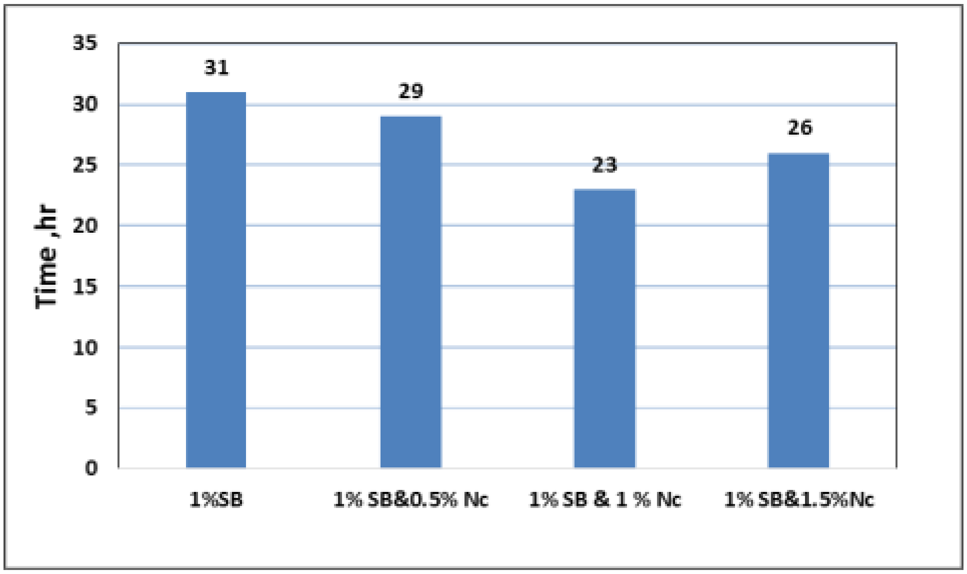

Figure 4-62 Transient time for the strength development from 50 to 500 psi for 1% untreated SB and Nc

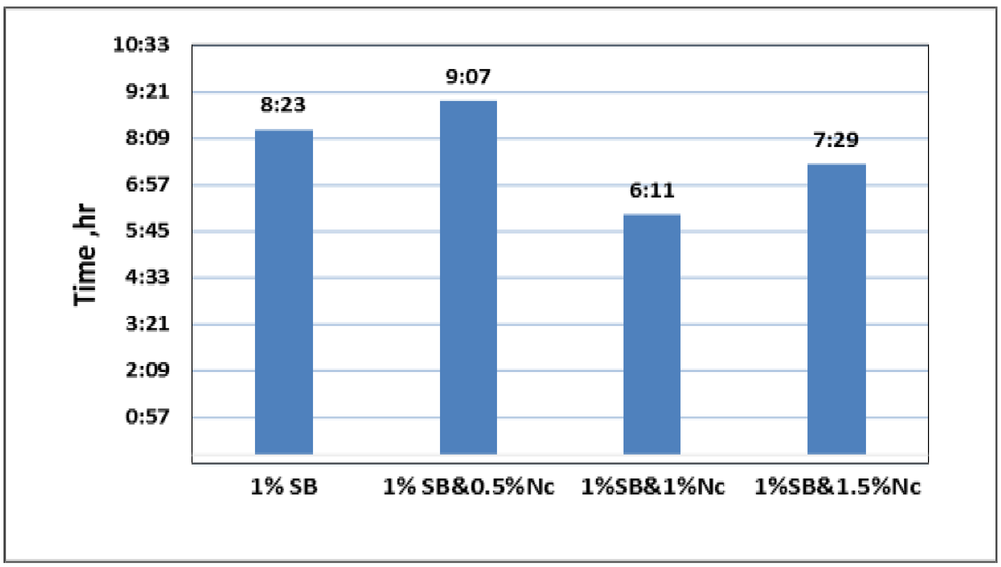

Figure 4-63 Time to achieve 2000 psi for 1% untreated SB and Nc

In short, the addition of Nano clay with 0.5, 1, 1.5% to the cement mix containing 1% untreated SB resulted in enhancement in the early strength development. In the case of the final strength obtained, we notice that the addition of 0.5% Nano clay did not have a significant effect on the final strength, and it was around 6,300 psi. After that, when 1% Nano clay is added, enhancement in strength was observed, and the final strength was around 6700 psi was reported. The increase in the amount of Nano clay added, resulted in a reduction in the compressive strength, for example, 1.5% Nano clay gave compressive strength of 6371 psi. **Figure 4-64** shows the final compressive strength 1% SB admixed with 0.5, 1, and 1.5% Nano clay. Hence, the cement system containing 1% untreated SB gave the highest strength compared with all systems.

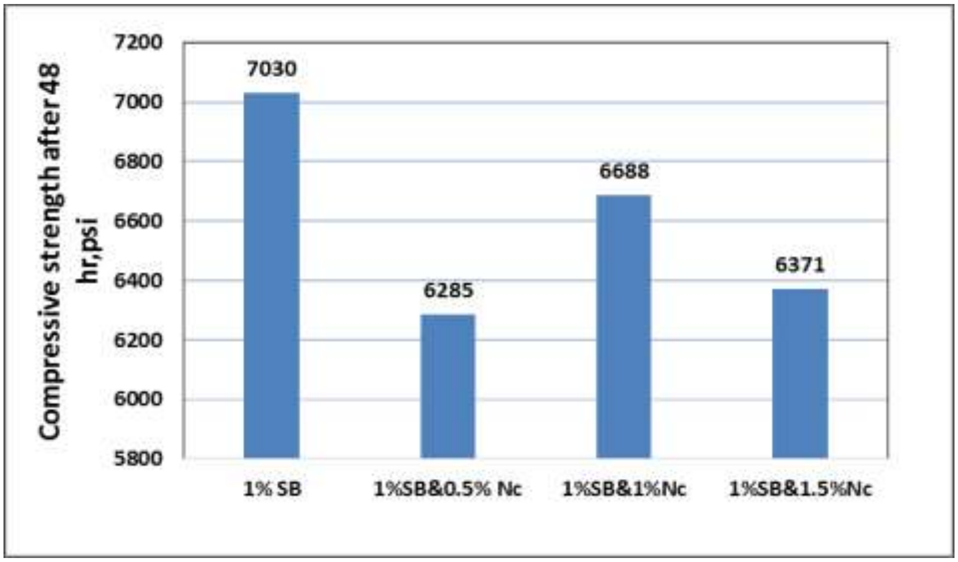

Figure 4-64 Final compressive strength after 48 hours

4.3.8 Effect of 1% SB with Nano Clay on Porosity and Permeability of the Cement

Permeability is an important property, and 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 it. After the cement cubes cured for 24 hours in the curing machine, cement plugs are drilled out of them. Porosity and permeability tests are conducted using automated porosimter /permeameter under a confining pressure of 500 psi. **Table 4-18** Porosity and permeability of 1% untreated SB with 0.5. 1, and 1.5 % Nano clay after 24 hours curing.

Table 4-18 Porosity and permeability of 1% untreated SB and 0.5, 1, and 1.5% Nc after 24 hours

Properties	Base mix	1% SB	1%SB&0.5%Nc	1%SB&1%Nc	1%SB &1.5%Nc
Porosity %	31	30.795	30.007	26.048	28.457
Permeability (md)	0.0041	0.003	0.0074	0.0025	0.0327

It was obvious that the addition of Nano clay to the cement mix containing 1% untreated SB to the cement mix resulted in a reduction the both porosity and permeability as explained in **Figure 4-65**, and **Figure 4-66**. The optimum cement mix design according to the porosity and permeability results was 1% untreated SB admixed with 1% Nano clay, which caused a significant reduction in porosity and permeability which were 26%, and 0.0025 md lower compared with optimum 1% untreated SB of around 30.7% and 0.003 md respectively.

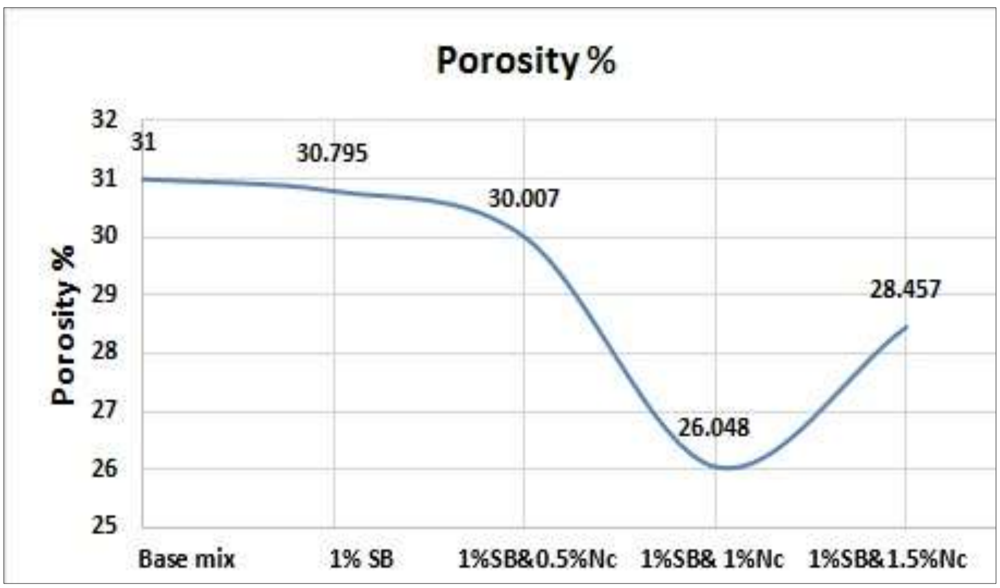

Figure 4-65 Porosity of cement 1% untreated SB and 0.5, 1, and 1.5% Nc after 24 hours curing

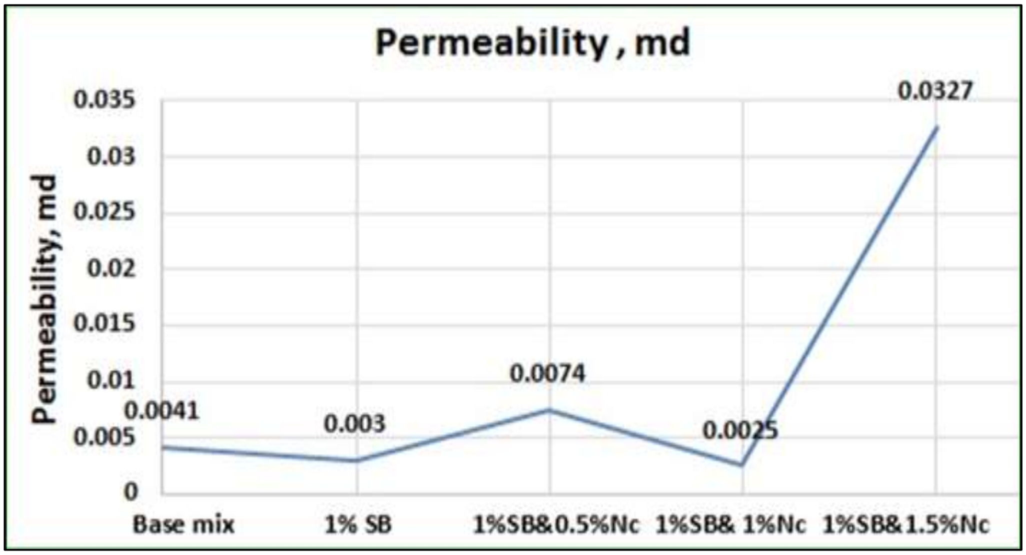

Figure 4-66 Permeability of 1% untreated SB and 0.5, 1, and 1.5% Nc after 24 hours curing

4.3.9 Microstructural Analysis for 1% SB with Nano Clay Admixed with Cement

The cement composition is analysed by exposing the cement to structural tests such as SEM and XRD. XRD and SEM results for cement containing 1% untreated Saudi bentonite is explained thoroughly in the above suction.

In the case of adding Nano clay to the optimum cement mix containing 1% untreated Saudi bentonite is explained as follows:

First, the addition of 0.5% Nano clay to the optimum mix containing 1% untreated Saudi bentonite.

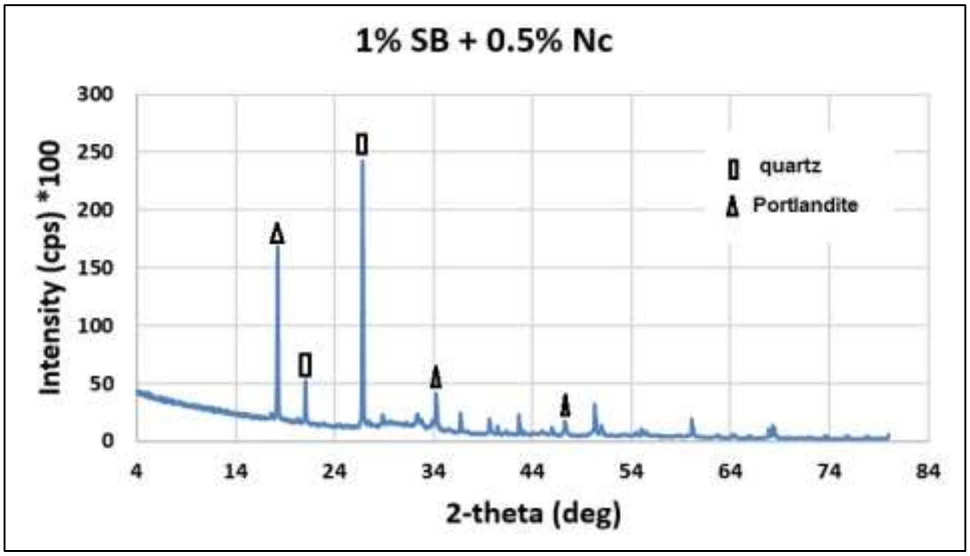

Figure 4-67 XRD hydration products of 1% untreated SB and 0.5% Nc cured at HPHT for 24 hours

When 0.5% Nc added to the cement mix containing 1% untreated SB with, the amount of the produced quartz crystals is considerably high, and could be related to the hydration reaction between both Nano clay and the untreated Saudi bentonite, where huge amount of silica found in the mix as showed clearly in XRD hydration products (see **Figure 4-67**). Here we also detected Portlandite in the final hydration products which will later transform to calcium silicate hydrate. The quartz crystals are merged and combined with each other produced a perfect network structure in the final resulted cement paste as shown in the SEM image (**Figure 4-68)**. However, this compressive strength was lower compared with that using pure 1% untreated Saudi bentonite, which showed that the effect of quartz crystals is not noticeable as evidence of higher compressive strength. **Figure 4-69** illustrates the SEM element weight analysis for 1% untreated SB and 0.5% Nc cured at HPHT for 24 hrs. It was obvious that the final cement product contains a considerable weight percentage of silica, and calcium, which demonstrations the formation of high percentages of CSH in the final harden cement.

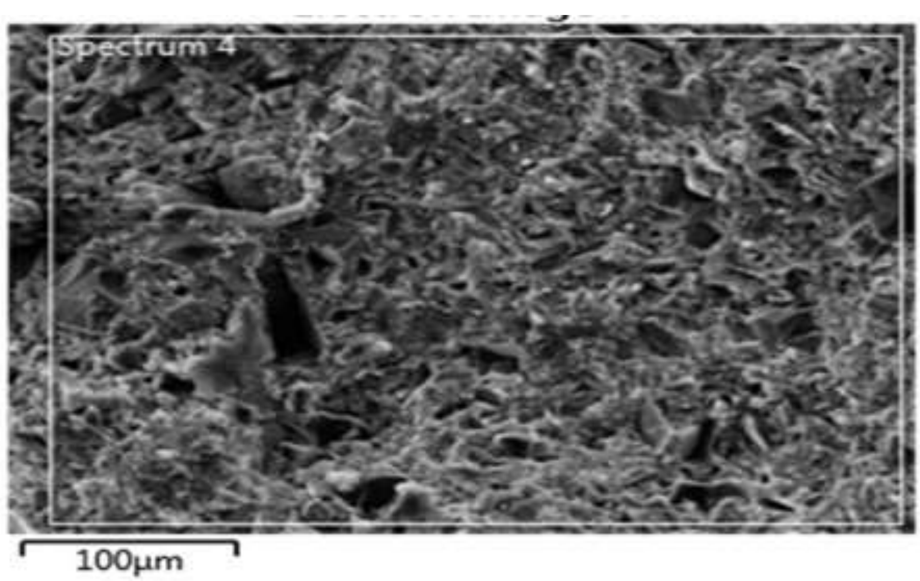

Figure 4-68 Hydration products (SEM) with 1% untreated SB and 0.5% Nc at HPHT for 24 hours

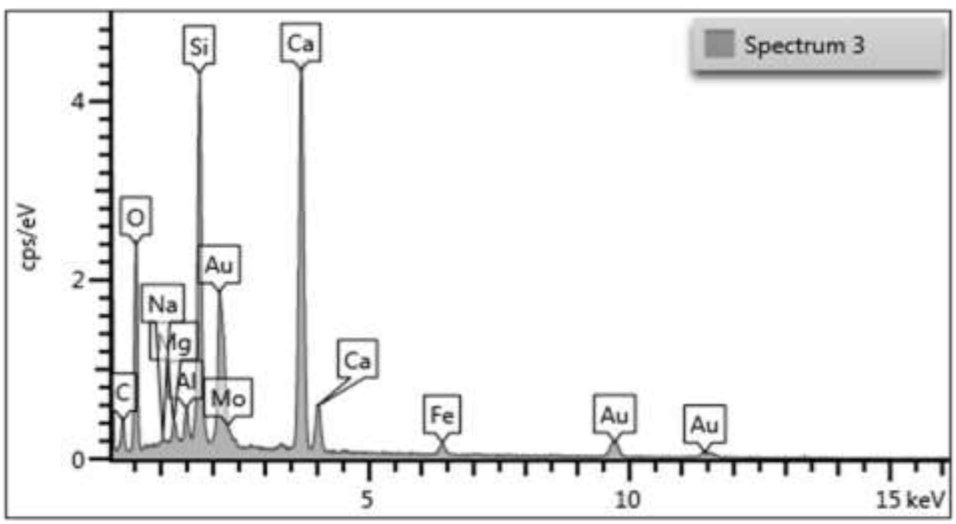

Figure 4-69 SEM photograph of hydration products of 1% untreated SB and 0.5%Nc at HPHT for 24 hours

As we mention above, the addition of 0.5% Nano clay to the cement mix with 1% untreated Saudi bentonite to the cement mix resulted in more polymerization as detected in the final produced cement sheath. In fact, if we compare the SEM images of a size of 5 microns for the three cement systems, cement base mix, 1% untreated SB, and 1% untreated SB admixed with 0.5% Nc, it was observed that the cement base mix showed more vugs and less polymerization compared with the cement system containing 1% as shown in **Figure 4-70** . On the other hand, cement mix with Nano clay showed more polymerization and more filling of the holes and the vugs compared with 1% untreated SB cement mix.

Moreover, it was observed that the addition of 0.5% Nano clay caused some cracks as well as some vugs in the final produced cement sheath as illustrated in the SEM image comparison. These cracks observed in the SEM image might be the reason for the reduction in the final compressive strength compared with the pure mix with untreated Saudi bentonite.

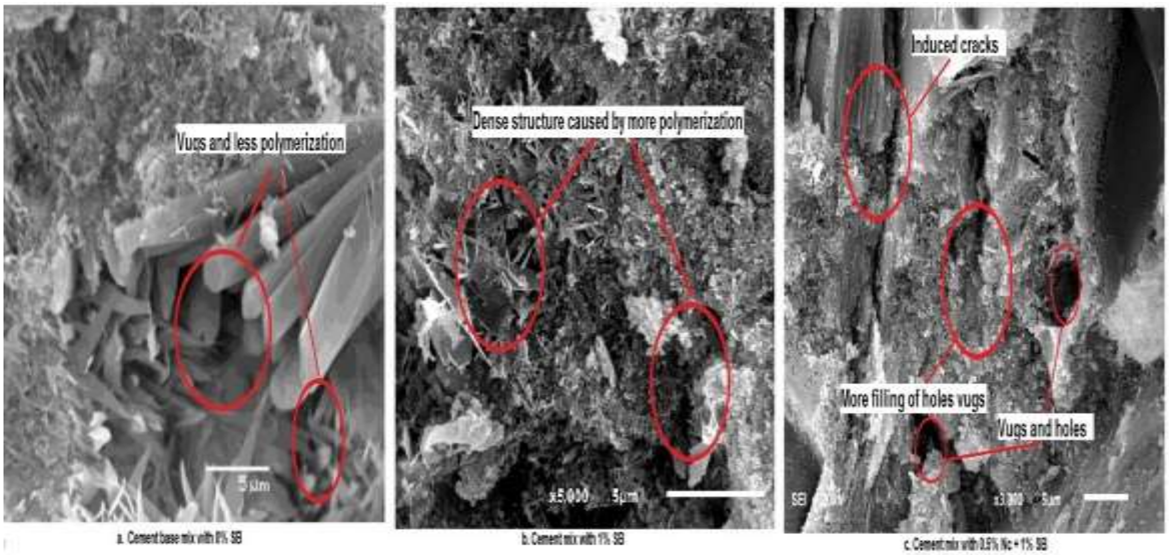

Figure 4-70 SEM 5 micron image comparing cement base mix, 1% untreated SB, and 1% untreated SB with 0.5% Nc

Here the same trend was observed as in the 1% untreated Saudi bentonite, where an ideal network structure as well as strength enhancement was achieved with 1% Nano clay addition to the optimum 1% untreated Saudi bentonite.

The higher the compressive strength, the higher percentages of CSH formed in the hydrated product. Since CSH is a good type of crystal, it weaves and forms an ideal and well-proportioned network structure in the hardened cement as shown in **Figure 4-71**. Cement slurry with Nano clay and untreated Saudi bentonite is resulted in big quantities of this favourable crystal due to the availability of silica in the mix. So, the addition of 1% untreated SB and 1% Nc produce a dense structure, and enhances the compressive strength as appeared clearly in the SEM image (see **Figure 4-72**). **Figure 4-73** shows an SEM element analysis for 1% untreated SB and 1% Nc 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 huge amounts of CSH in the final harden cement.

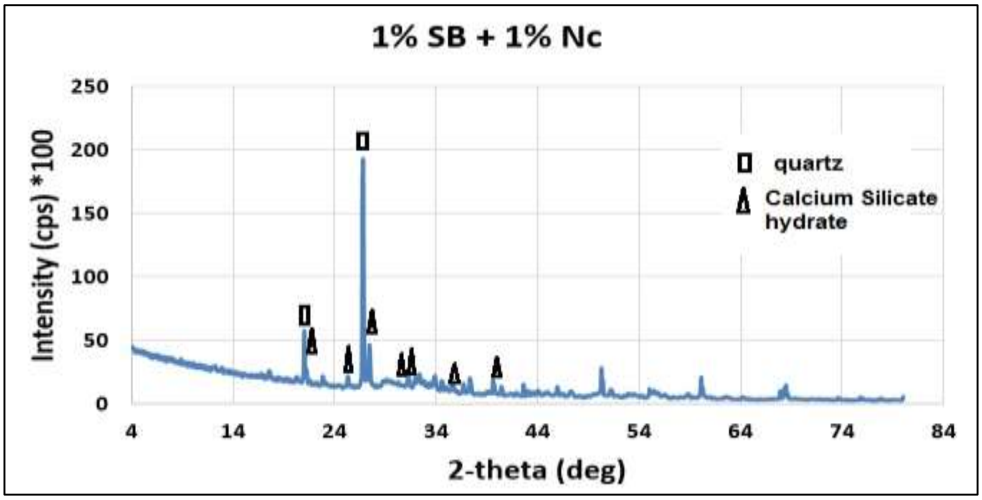

Figure 4-71 XRD hydration products of 1% untreated SB and 1%Nc at HPHT for 24 hours

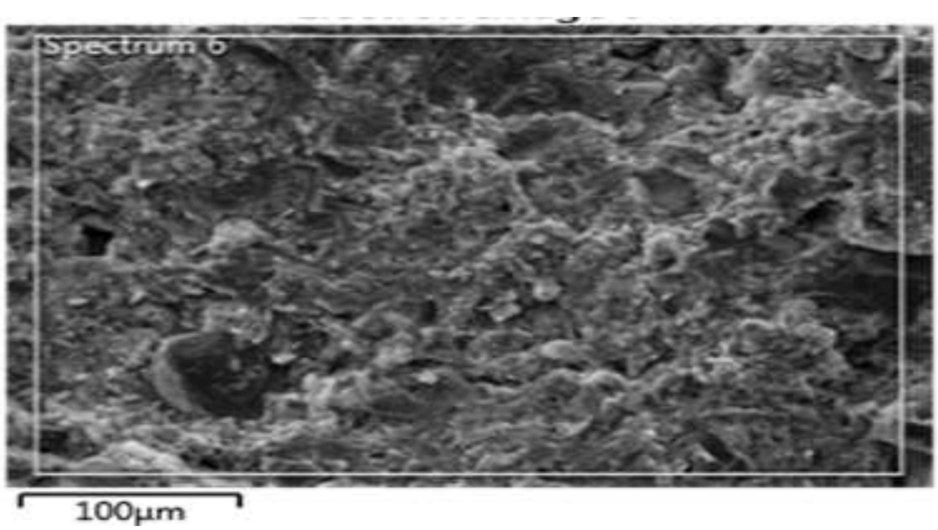

Figure 4-72 Hydration products (SEM) with 1% untreated SB and 1% Nc at HPHT for 24 hours

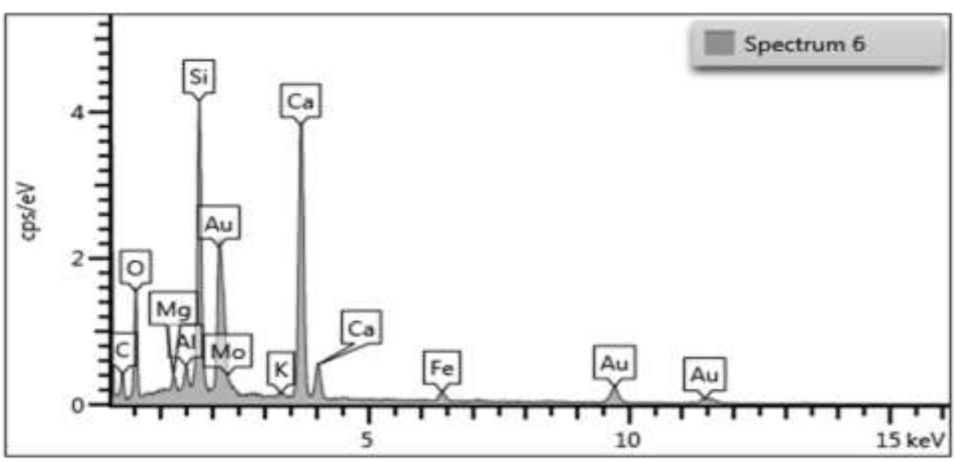

Figure 4-73 SEM photograph of hydration products of 1% untreated SB with 1% Nc at HPHT for 24 hours

It was clear that the addition of 1% Nano clay to the cement mix containing 1% untreated Saudi bentonite caused more polymerization as detected in the final produced cement sheath compared with all mixes with both Saudi bentonite as well as Nano clay. In fact, if we compare the SEM images of a size of 5 microns for the three cement systems, cement base mix, 1% untreated SB, and 1% untreated SB admixed with 1% Nc, it was observed that the cement base mix showed more vugs and less polymerization compared with the cement system containing 1% untreated SB as shown in **Figure 4-74** . On the other hand, cement mix with Nano clay showed significant improvement in polymerization and more filling of the holes and the vugs compared all cement mixes. Moreover, it was observed that the addition of 1% Nano clay caused minor vugs in the final produced cement sheath as illustrated in the SEM image comparison, which might be the reason for the slight reduction in the final compressive strength when compared with that of the pure mix with 1% untreated Saudi bentonite. In short, 1% Nc showed the best filling behaviour of pores in the cement paste, and produced a more tight and dense structure, with a slight reduction in the final strength.

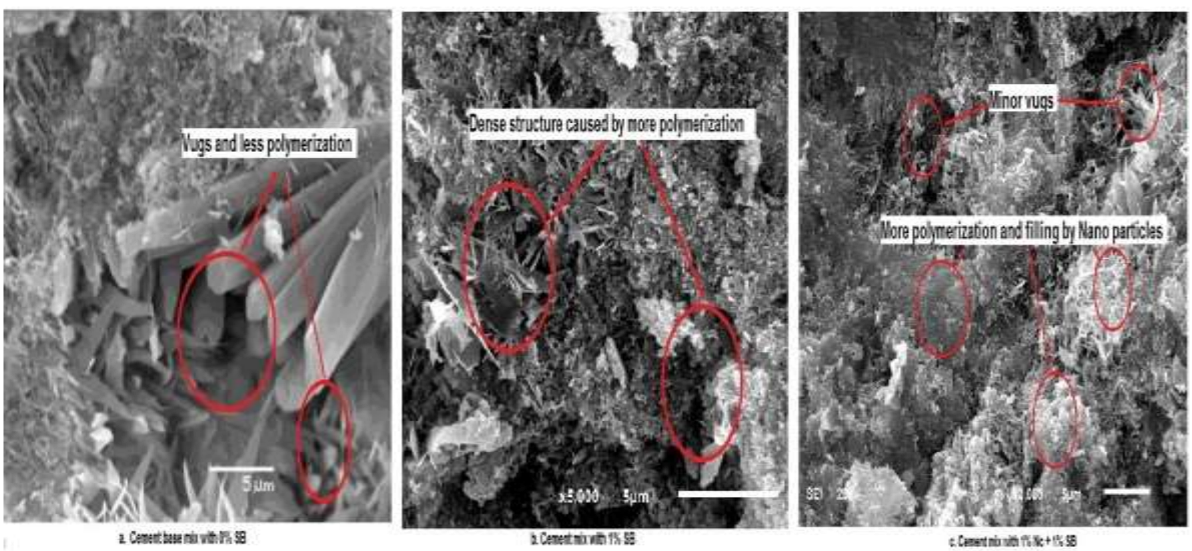

Figure 4-74 SEM 5 micron image comparing cement base mix, 1% untreated SB, and 1% untreated SB with 1% Nc

When 1% untreated SB mixed with 1.5% Nc with the cement slurry, the same cement behaviour was observed as described above as in the case of adding 0.5% Nano clay. As a matter fact, adding 1% untreated SB mixed with 1.5% Nc produce quartz associated with an increase in the pozzolanic reaction. It was also clear that calcium silica hydrate crystals were also formed in the cement past, which plays an important role in the accelerating compressive strength development. Also Portlandite was also detected in the final cement hydration products which will later transform into calcium silicate hydrate products. Unlike, the behaviour observed in 1% also SB with 1% Nc, the compressive strength was reduced with increasing the percentages of the added Nano clay. The reason for this fall in the compressive strength might be due to the reduction in the amount quartz crystals formed in the final harden cement paste as shown clearly in the XRD results as well as Portlandite which detected in the final cement paste (see **Figure 4-75**). **Figure 4-76** represents the SEM results of 1% untreated SB mixed with 1.5% Nc cured at HPHT for 24 hours. From the SEM picture it was clear that the hardened cement had gaps and voids all over the structure. **Figure 4-77** shows the SEM element weight analysis for 1% untreated SB mixed with 1.5% Nc cured at HPHT for 24 hours. It was obvious that the final cement product contains higher weight percentages of silica and calcium, which demonstrations the formation of CSH in the final harden cement.

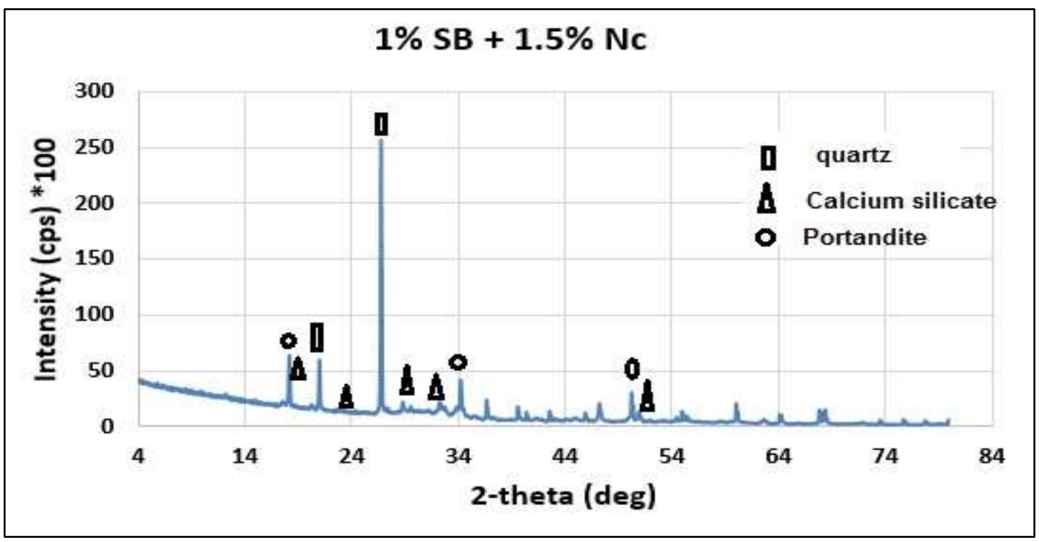

Figure 4-75 XRD hydration products with 1% untreated SB and 1.5%Nc at HPHT for 24 hours

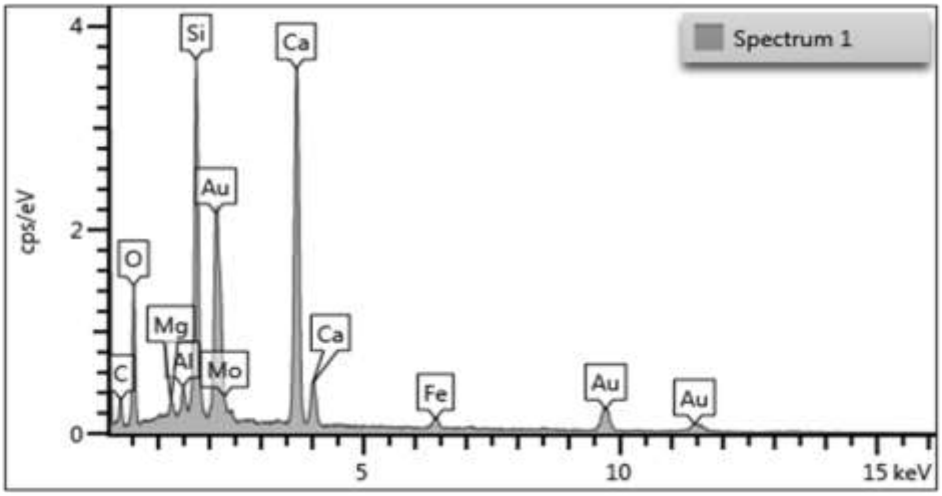

Figure 4-76 SEM photograph of hydration products with 1% untreated SB and 1.5%Nc at HPHT for 24 hours

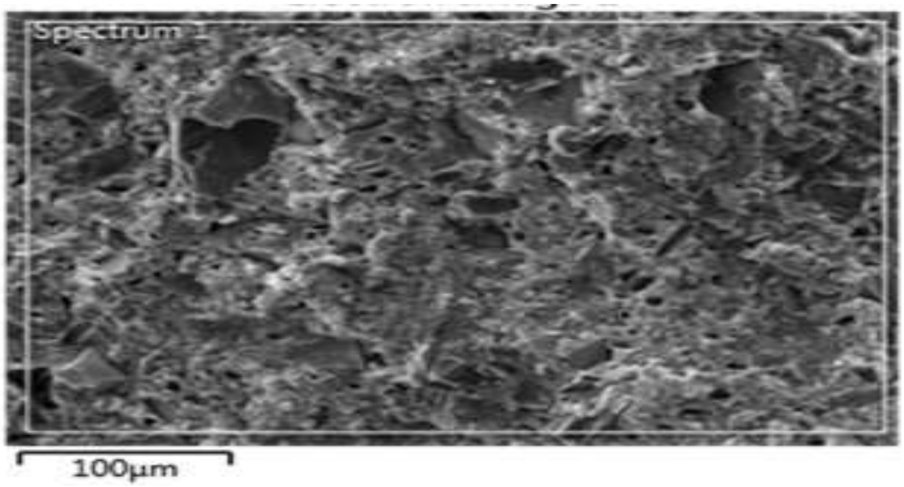

Figure 4-77 Hydration products (SEM) with 1% untreated SB and 1.5%Nc at HPHT for 24 hours

As we mention above, the addition of 1.5% Nano clay to the cement mix containing 1% untreated Saudi bentonite to the cement mix resulted in a considerable polymerization as detected in the final produced cement sheath. In fact, if we compare the SEM images with a size of 5 microns for the three cement systems, cement base mix, 1% untreated SB, and 1% untreated SB admixed with 1.5% Nc, it was observed that the cement base mix showed more vugs and less polymerization compared with the cement system containing 1% untreated SB as shown in **Figure 4-78** . On the other hand, cement mix with Nano clay showed more polymerization, more filling of vugs and holes compared with 1% untreated SB cement mix. Moreover, it was observed that the addition of 1.5% Nano clay caused some cracks as well as some vugs in the final produced cement sheath as illustrated in the

SEM image below, which might be considered as the reason for the reduction in the final compressive strength compared with the pure mix with 1% untreated Saudi bentonite. Another reason might be related to the incompatibility of the cement final products which caused more vugs and cracks in the final cement paste.

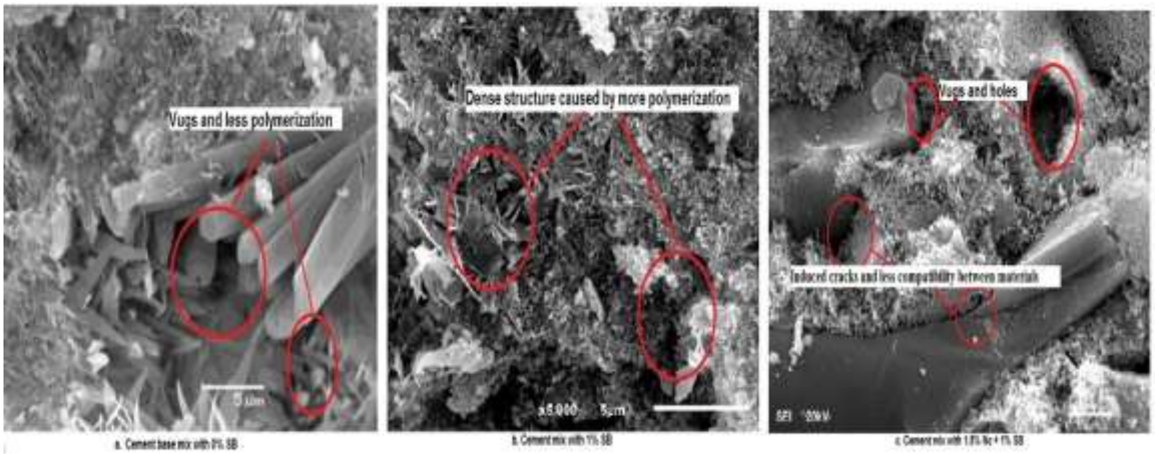

Figure 4-78 SEM 5 micron image comparing cement base mix, 1% untreated SB, and 1% untreated SB with 1.5% Nc

4.4 Performance of 1.9% Treated SB with 1.9% Commercial Bentonite on Cement Properties (101 PCF)

Saudi bentonite when used untreated showed poor results, where the swelling properties as well as the dispersion of the bentonite particles were not good as the commercial bentonite, and it showed settling at the bottom of the beaker. Treated (upgraded) Saudi bentonite developed by (Musaab, 2014) showed good results were no settling was observed associated with good dispersion throughout the whole mix. In addition to that, the treated Saudi bentonite showed almost the same results exerted by commercial imported bentonite when used in the case of drilling mud for drilling applications.

Here upgraded Saudi bentonite was tested in the case of using it as an extender in oil well cementing application, and then compare the results with that obtained from 1.9 % bwoc commercial bentonite in order to make the decision whether or not use it as an alternative to the commercial bentonite in oil well cementing applications.

At the start, 1.9 % bwoc commercial bentonite was used to produce a cement slurry with a density of (101 PCF) 13.5 Ib/gal. After that, this low density cement was subjected to cement tests such as thickening time, free water rheology, etc. and its effect on the cement properties was reported. Properties obtained from this was considered as the baseline in our comparison study.

Upgraded (Treated) Saudi Bentonite

Bentonite grades are usually occurring as a relatively thin bed formed from altered volcanic ash very close to the surface (Al-Homadhi, 2007). Samples are first collected from Khulays area, and then dried, grinded up to a particle size of less than 75 microns and prepared for the tests. After that, samples were subjected to XRD, SEM, EDX, and then treatment process.

From the XRD results, the commercial bentonite and raw (untreated) local Saudi bentonite showed a very good agreement in the XRD spectrum peaks, which proves that Khulays clay is bentonite. The main difference between the two results is specified in two peaks in the local bentonite d = 3.34, and d = 7.096 (see **Figure 4-79**).

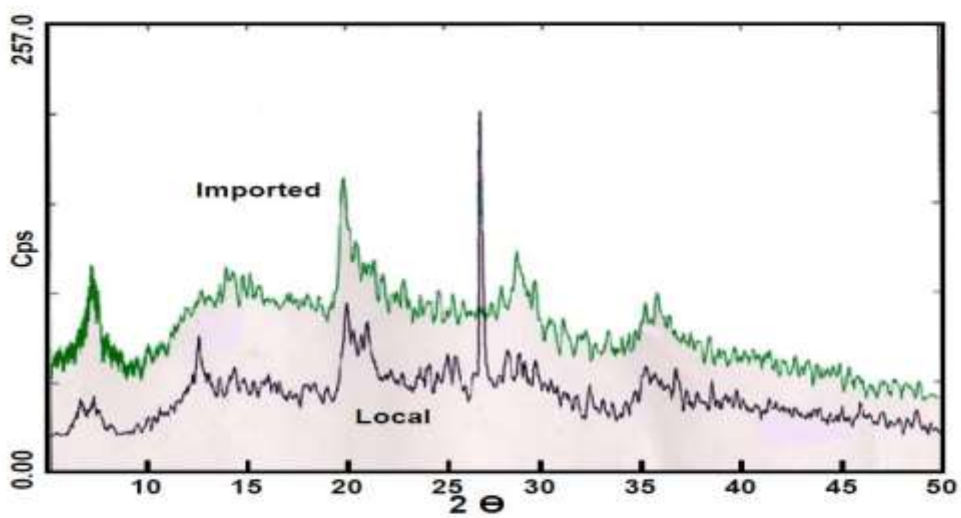

Figure 4-79 XRD results for local Saudi and commercial bentonite (Al-Homadhi, 2007).

The first peak proves the presence of high percentages of Quartz in the local Saudi bentonite. The second peak shows small concentration of Kolinite in the structure. A comparison between SEM image results for both untreated local Saudi bentonite, and commercial bentonite is explained in **Figure 4-80**. From the SEM images, it is obvious that this clay is bentonite as shown in **Figure 3-2**.

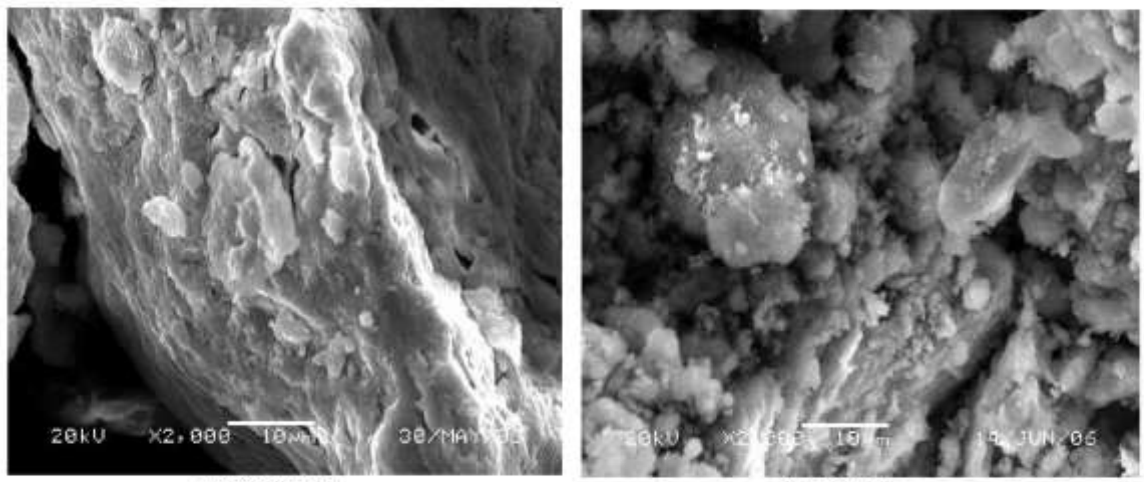

Figure 4-80 SEM images for commercial, and local Saudi bentonite (Al-Homadhi, 2007).

EDX analysis tests were conducted on both raw Saudi local bentonite, and commercial bentonite (see **Figure 4-81**). From the results we notice that there were impurities in the raw bentonite such as iron, and low sodium to calcium ratio when compared with commercial bentonite.

Treatment Process

Soda ash treatment is applied to change the local Saudi bentonite from the calcium base bentonite into the bentonite Sodium base. There were two phases of treatment conducted on the local bentonite, (1) purifying from impurities such as iron, etc., and (2) then this treatment is followed by a thermal treatment process (Mosaab, 2014).

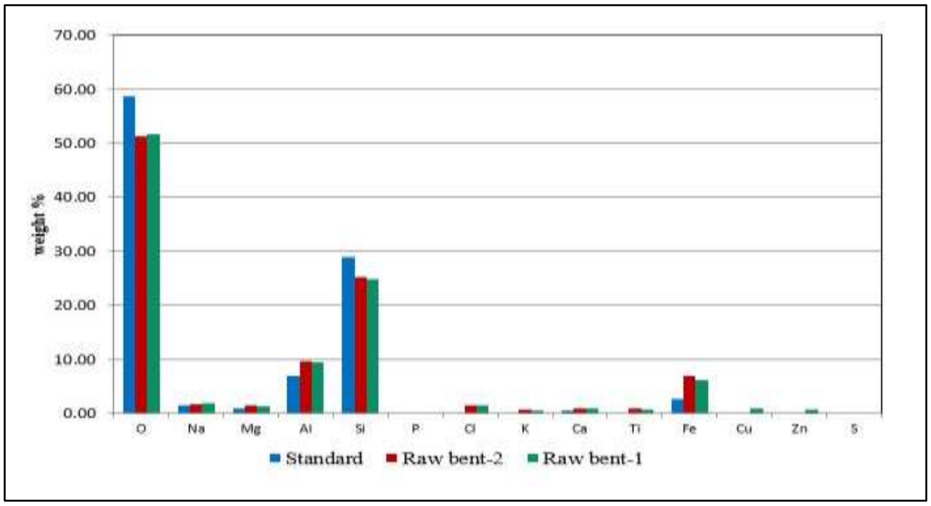

Figure 4-81 EDX analysis for raw and commercial bentonite (Musaab, 2014)

It was clear that after the first phase treatment, the amount of iron in the local treated decrease to a value close to that exposed by the commercial bentonite as shown in **Figure 4-82**. In addition, a slight improvement was observed in the case of Na/Ca ratio as shown clearly in **Figure 4-83**.

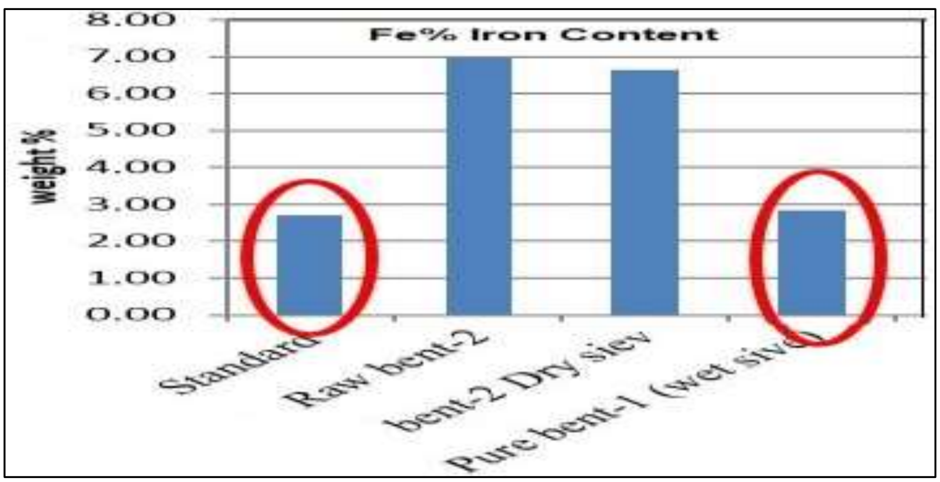

Figure 4-82 Iron content for raw local, treated, and commercial bentonite (Musaab, 2014)

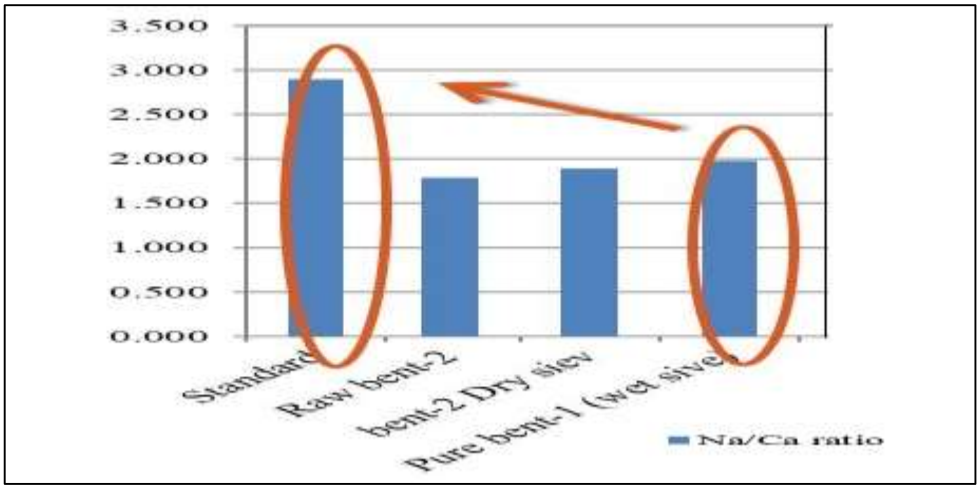

Figure 4-83 Na/Ca ratio for raw, treated, and commercial bentonite (Musaab, 2014)

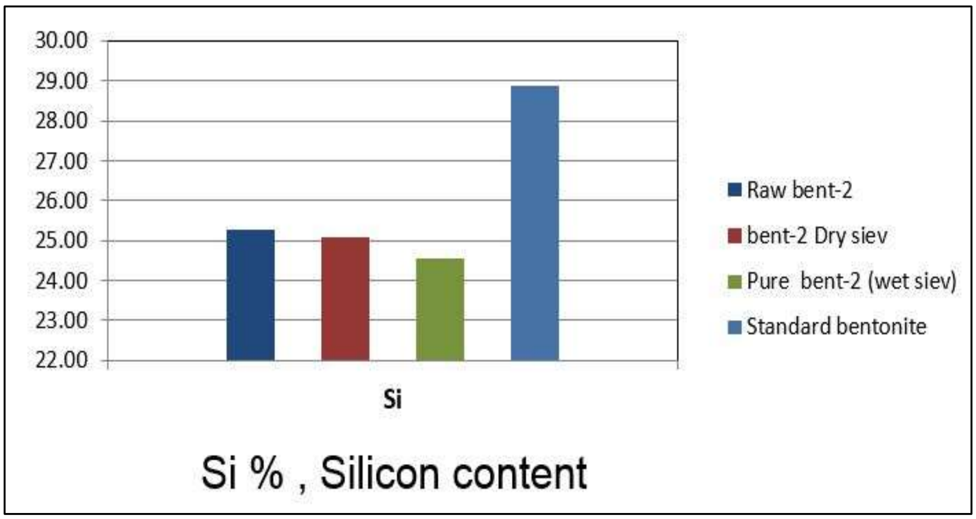

Figure 4-84 Silicon content for raw, treated, and commercial bentonite (Musaab, 2014)

Cement System Design

Table 4-19 explains the cement slurry design for the particular well with the addition of treated (upgraded) Saudi bentonite.

Table 4-19 Cement slurry design with bentonite

Properties	Values
Slurry Density (Approx.), PCF	101
Water Cement Ratio	1.00
Slurry Yield	-
Thickening Time	4 - 5 hours
Class G cement powder + 35% silica flour + 1.9% bwoc treated Saudi bentonite + 0.5% Fluid loss control agent (H22A) + 0.15% Retarder (HR-25) + 0.8% Retarder (HR-5) + 0.25gm Defoamer	

At the start of our cement test, 1.9% commercial bentonite cement system will be prepared and tested for cement properties (as a reference line). After that, 1.9% treated Saudi cement system will be prepared and test first for the cement free water.

Hydration of Treated (Upgraded) Saudi Bentonite

The bentonite used in these experiments is first hydrated in water before using it with the cement. The procedure of bentonite hydration is explained as follows:

1. Water and bentonite powder are first weighted and prepared.
2. Water is poured in the blender with the bentonite and then mixed at low speed of 4000 RPM for 20 minutes.
3. The blender is removed, covered, and left for overnight.
4. Finally, the amount of water needed is added and the testing procedure is done as explained in the experimental program.

4.4.1 Effect of Treated SB on the Cement Slurry Free Water

Water 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.

From **Table 4-20**, it is obvious that the addition of 1.9% bwoc of treated Saudi bentonite resulted in higher amounts of free water accumulated at the top of the cement compared with that obtained from that commercial bentonite (see **Figure 4-85**).

Table 4-20 Free water results of commercial, and treated Saudi bentonite

Free water (2 hours aging) ml/250ml	Bentonite		
	1.9% Standard	1.9% Treated Saudi	3.8 % Treated Saudi
Sample 1	1.8	3.6	1.9
Sample 2	1.9	3.2	2.1
Sample 3	2.2	3.15	-
Average	**1.96**	**3.32**	**2**

So, to solve the problem of the slight increase in the free water, we tried to increase the amount of treated Saudi bentonite added. When 3.8 % treated Saudi bentonite used, the amount of free water collected at the top was almost the similar to that obtained from commercial bentonite (of around 2 ml). Also, when 1.9 % of treated Saudi bentonite used, gelling was not good, and some settling was observed through the gradual cylinder. On the other hand, the settling behaviour was almost vanished when 3.8% of treated Saudi bentonite used in the cement system. Furthermore, the upgraded Saudi bentonite did not cause any distribution in the particle suspension property within the final produced cement.

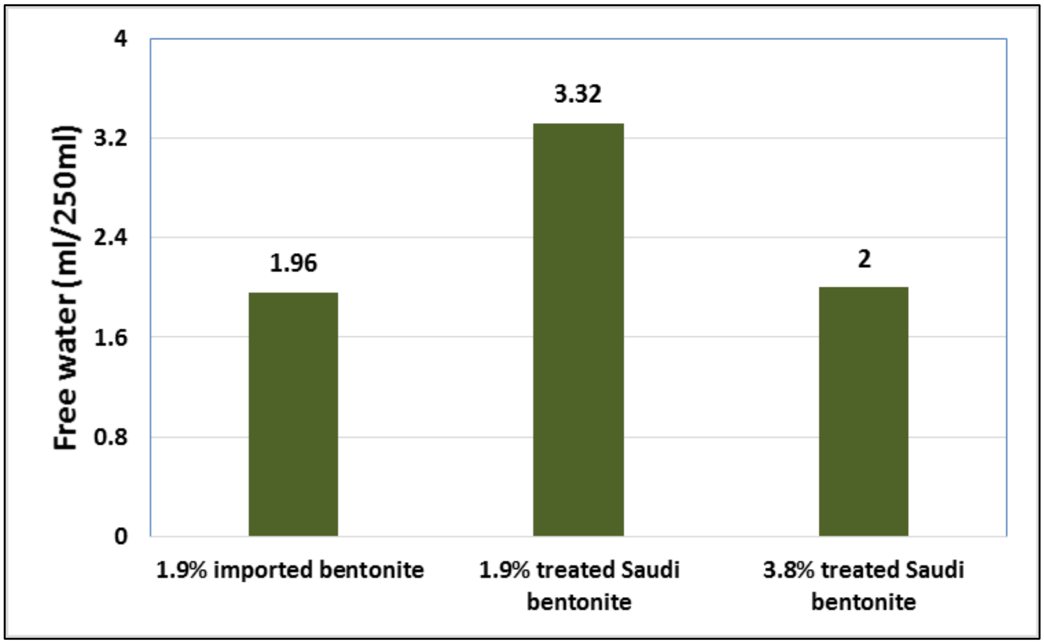

Figure 4-85 Free water results of commercial and treated Saudi bentonite

4.4.2 Effect of Treated Saudi Bentonite on Cement Rheology

Table 4-21 shows the fann readings for 1.9% commercial as well as 1.9, and 3.8% treated Saudi bentonite. When 1.9% bwoc treated Saudi bentonite added to the cement mix, the rheology reading was too low compared with that obtained from commercial bentonite. On the other hand, when the treated Saudi bentonite percentage used doubled into 3.8% bwoc, we observed improvement in rheology reading as shown in the green line in **Figure 4-86**.

Table 4-21 Fann readings for both commercial and upgraded SB

RPM	Bentonite		
	1.9% commercial	1.9% Treated Saudi	3.8% Treated Saudi
3	7.25	3.65	5.5
6	8.75	4.5	6.5
100	11.5	6	8
200	16.75	10.5	12
300	23	13	14.5
600	32	21.5	25.5

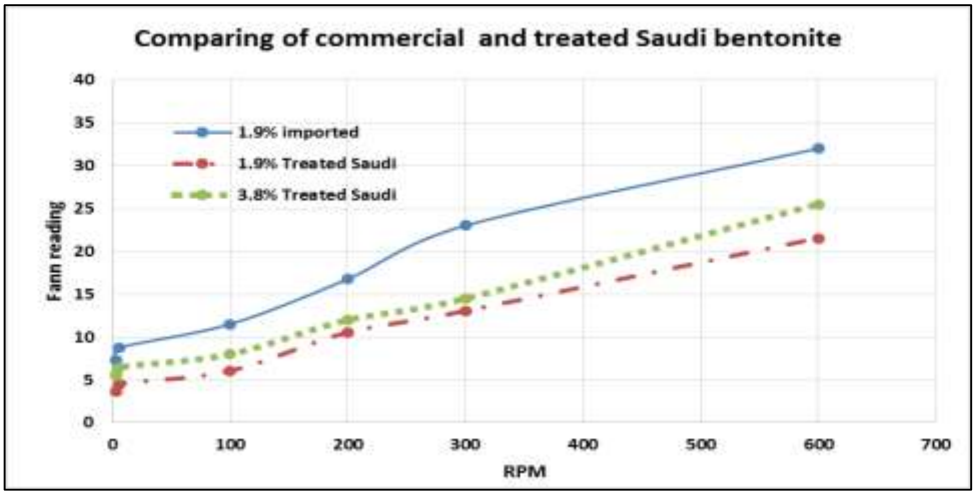

Figure 4-86 Fann readings for commercial and treated SB

It is obvious that doubling the quantity of the treated Saudi bentonite from 1.9 to 3.8% bwoc the resulted in enhancement in the rheological cement properties like plastic viscosity and also yield point to a value closer to that exerted by 1.9% commercial bentonite as shown in **Table 4-22**.

Table 4-22 Plastic viscosity, and yield point results of commercial and treated SB

Property	Bentonite		
	1.9% commercial	1.9% Treated Saudi	3.8% Treated Saudi
PV (cps)	12	9	10
YP (lb_f /100ft^2)	9	4	6

Figure 4-87 shows the plastic viscosity trend of commercial and treated Saudi bentonite. It was clear that 1.9 % treated Saudi bentonite added to the cement mix gave a plastic viscosity from 9 cp which is considered low compared with 12 cp obtained from the commercial bentonite. Raising the percentage to 3.8% caused an improvement in the plastic viscosity of around 10 cp. Unlike this improvement observed in plastic viscosity, the yield point trend has been slightly improved (see **Figure 4-88**). The addition of 1.9 % treated Saudi bentonite resulted in 4 Ib/100 ft^2 yield point which was too small compared with that commercial bentonite of around 9 Ib/100 ft^2. When the percentage of treated bentonite doubled into 3.8%, an enhancement was observed in the value of the yield point which increased to 6 Ib/100 ft^2 which indicated enhancement. Yield point has some side effects on cement properties, as a result, completion engineers take this parameter into consideration when designing the optimum cement slurry for the proposed job.

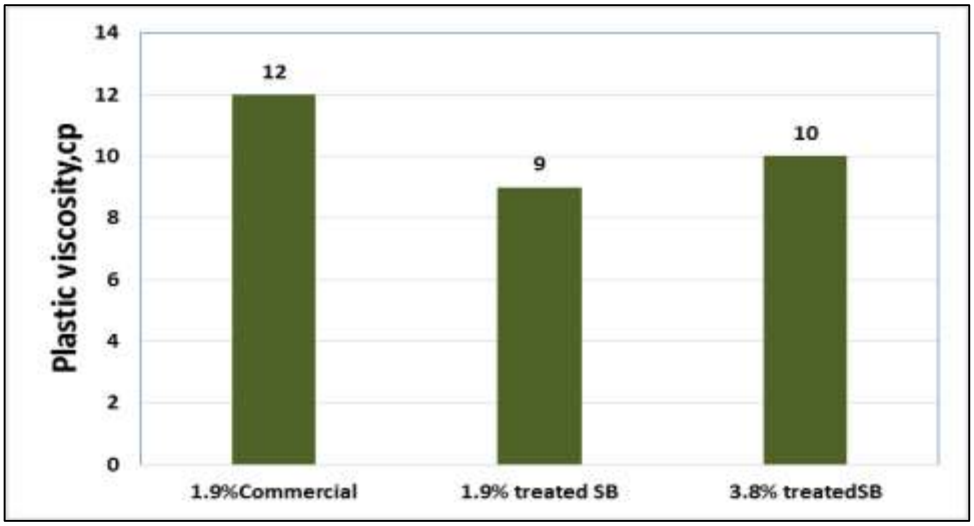

Figure 4-87 Plastic viscosity results of commercial and treated SB

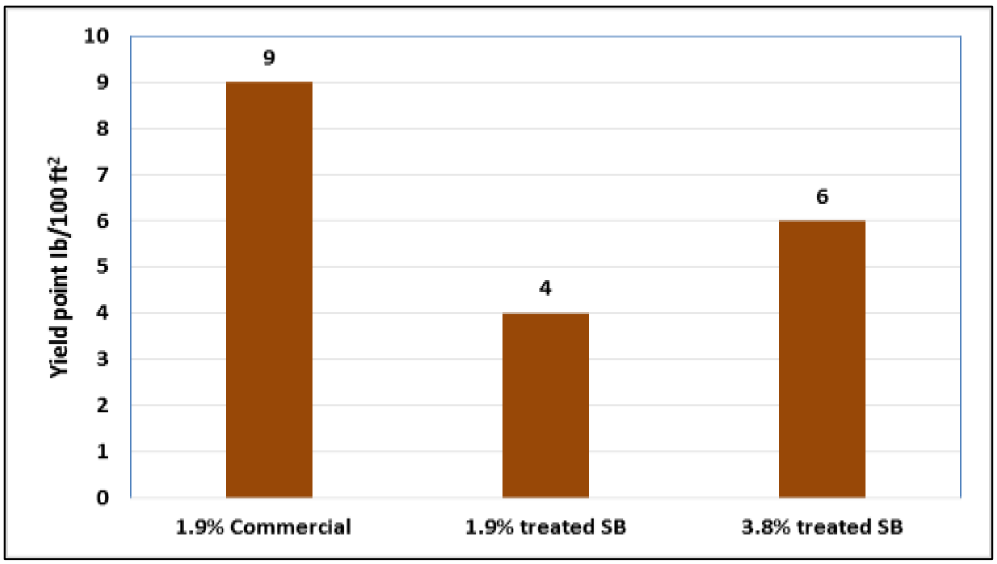

Figure 4-88 Yield point results of commercial and treated SB

4.4.3 Effect of treated Saudi Bentonite on Cement Gel Strength

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. **Table 4-23** illustrates the gel strength of commercial as well as treated Saudi bentonite conducted using fann Viscometer. From the table, we noticed that 10-sec, and 10-min gel strength obtained from 1.9% bwoc treated Saudi bentonite were low compared with that obtained from commercial bentonite. However, when the amount of the treated Saudi bentonite doubled to 3.8%, an improvement was observed in the results, where the gel strength results for 10-sec gel was increased from 4.5 up to 8.5 $Ib_f/100\ ft^2$, the same as the commercial bentonite as shown in **Figure 4-89**.

The same trend is observed when 1.9% treated Saudi bentonite cement slurry is subjected to 10-min gel strength test. Before doubling the added percentage 10-min gel was around 21 $Ib_f/100\ ft^2$, and then it was improved and gave a value almost the same 37 $Ib_f/100\ ft^2$ which is higher compare with commercial bentonite. This proves that the treated Saudi bentonite gives excellent results in the case of 10-min gel strength better than the commercial bentonite.

Table 4-23 Gel strength results of commercial and treated SB

Property	Bentonite		
	1.9% commercial	1.9% Treated Saudi	3.8 % Treated Saudi
Gel 10-sec	8	4.5	8.5
Gel 10-min	32	21	37

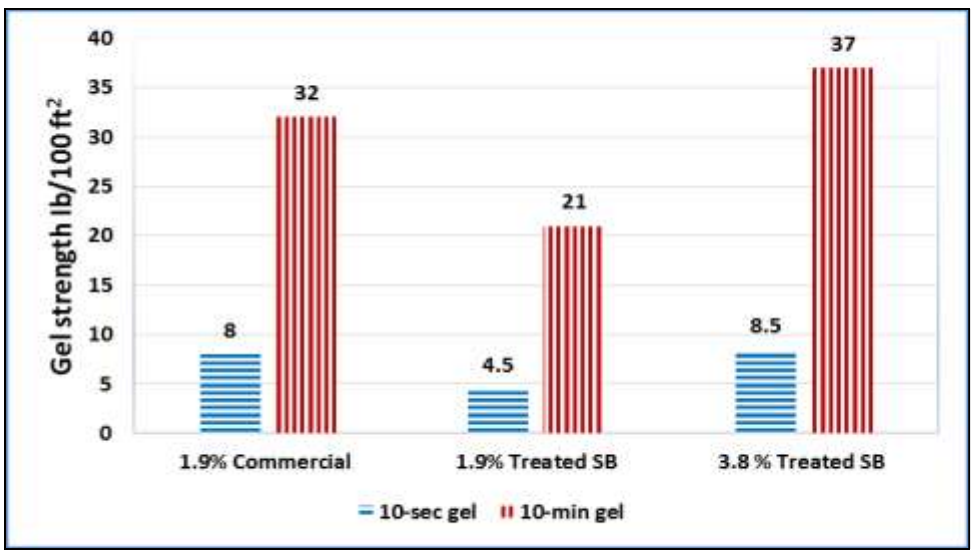

Figure 4-89 Gel strength results of commercial and treated SB

4.4.4 Effect of Treated SB on Cement Slurry Density

Well control is one of the most important issues that engineers should carefully consider during drilling and cementing. A pressurized mud balance is normally used to measure the cement density in field as well as in the laboratory. Here in this case, a cement system design was provided from Saudi Aramco, where bentonite is added at 1.9% bwoc to produce a low density used in cementing operations. The cement system design gave a density of a fixed value of 13.5 Ib/gal (101 PCF).

4.4.5 Effect of Treated SB on the Cement Slurry Thickening Time

Thickening time cement test gives us an indication about the time period the cement remains pumpable under certain conditions. At the start of the test, 1.9% commercial and 3.8% treated Saudi bentonite cement systems have a consistencies of (22, and 19) Bc respectively. When the conditions of the test are applied, this value reduced and then remain stable for a certain time until the 100 Bc value reached, which is an indicator that the cement is now unpumpable. It was clear that the addition of commercial or treated Saudi bentonite to the cement resulted in increasing in the thickening period time as shown in **Figure 4-90**, **Figure 4-91**, **Figure 4-92**, and **Figure 4-93**.

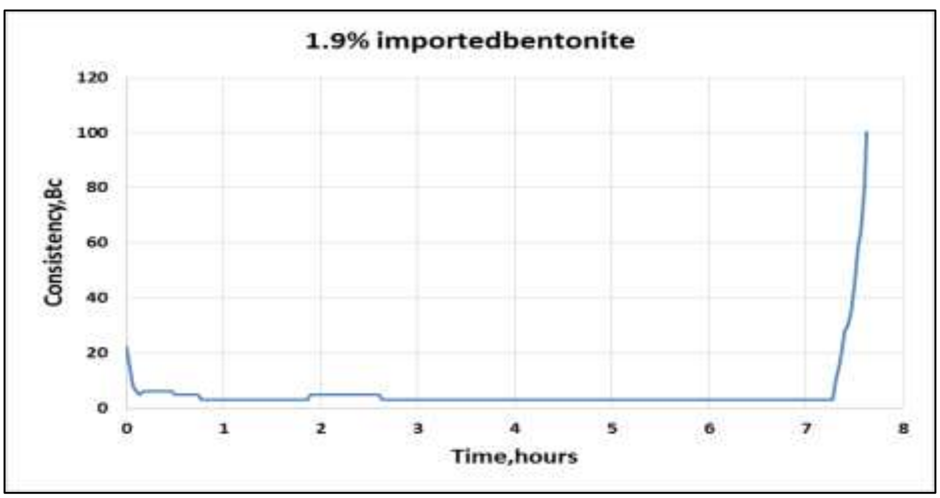

Figure 4-90 Thickening time of commercial bentonite

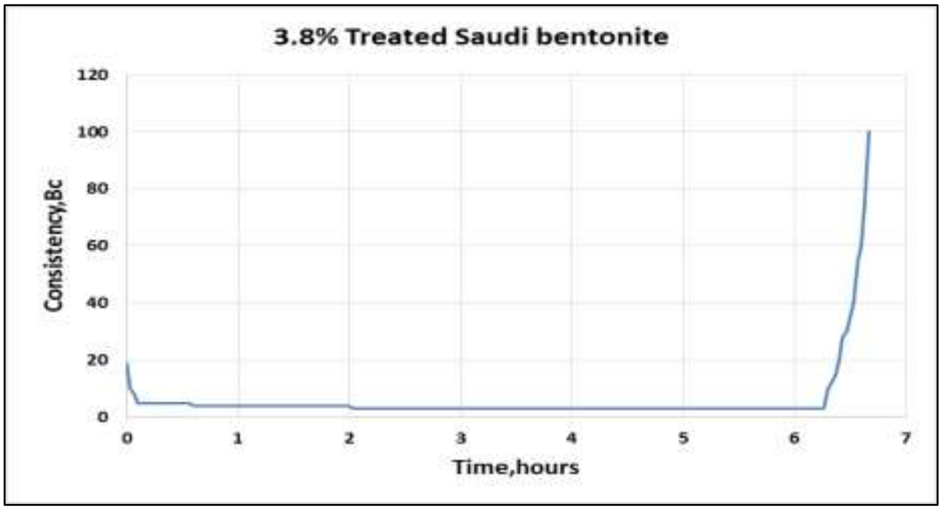

Figure 4-91 Thickening time of treated Saudi bentonite

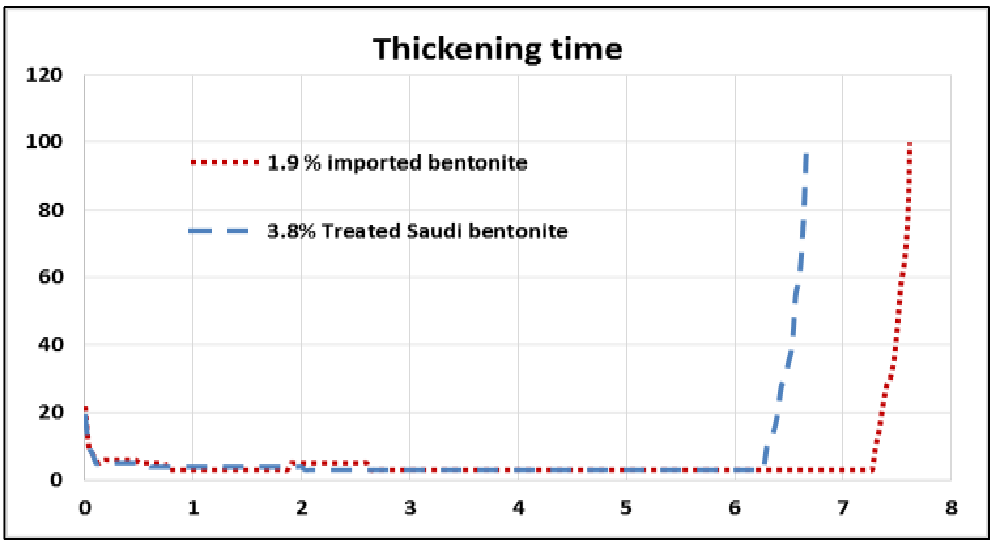

Figure 4-92 Thickening time of commercial and treated Saudi bentonite

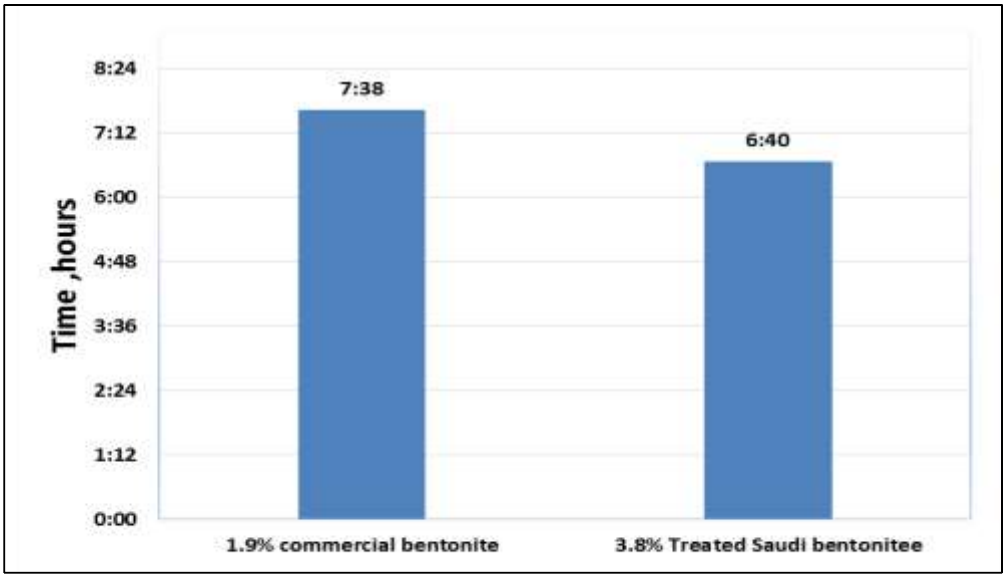

Figure 4-93 Cement thickening time of commercial and treated SB

At the start of the cement test, commercial bentonite as well as treated Saudi bentonite has a higher consistency of 22, 19 Bc respectively as showed in **Figure 4-94**. After that, when the test conditions are applied, this consistency values reduced and then remain constant until the cement slurry reaches the 100 Bc and become thick and unpumpable. It was observed that it takes longer time to reach 40 Bc for all cement slurries. **Figure 4-95** shows the time to reach 40, 70, 100 Bc consistencies. All the cement slurries take a long time to reach a consistency of 40 Bc, leaving only 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.

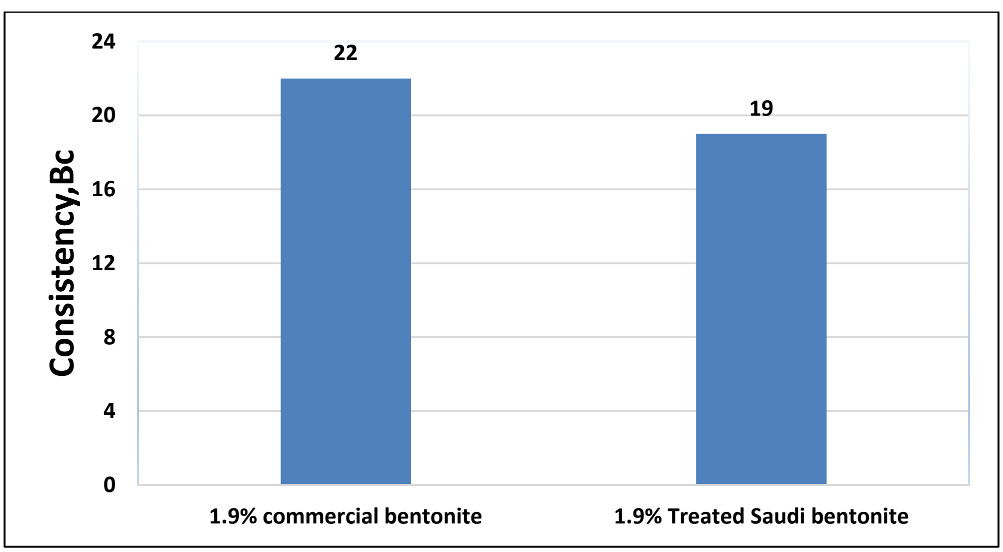

Figure 4-94 Consistency at the beginning of thickening time cement test

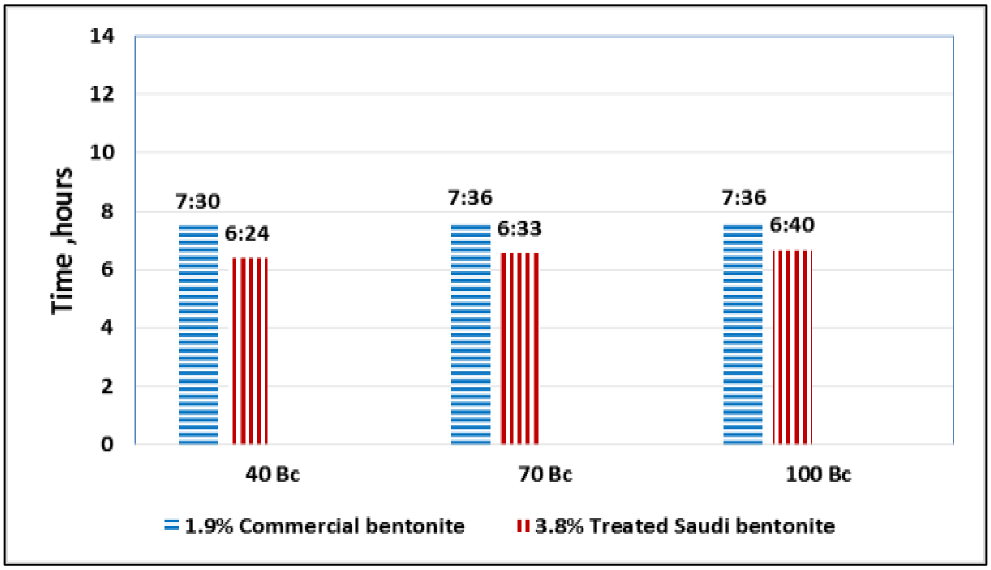

Figure 4-95 Time to reach 40, 70, 100 Bc consistencies

4.4.6 Effect of treated SB on Cement Compressive Strength by Crushing Method

Compressive strength is an important issue that drilling engineers carefully consider before resuming any drilling operation. In fact, cement integrity and long-term bearing ability are determined by the compressive strength properties. **Table 4-24** shows the results of the compressive strength of 1.9% commercial and 3.8% treated Saudi bentonite after 24 hours. The final compressive strength for both systems were almost similar of around 1350 which proves 3.8% treated Saudi bentonite perform in the same manner that commercial bentonite do as shown in **Figure 4-96**.

Table 4-24 Compressive strength of commercial and treated Saudi bentonite after 24 hours

Sample	1.9% commercial bentonite	3.8% Upgraded Saudi bentonite
1	1334	1378
2	1449	1334
Average	**1342**	**1356**

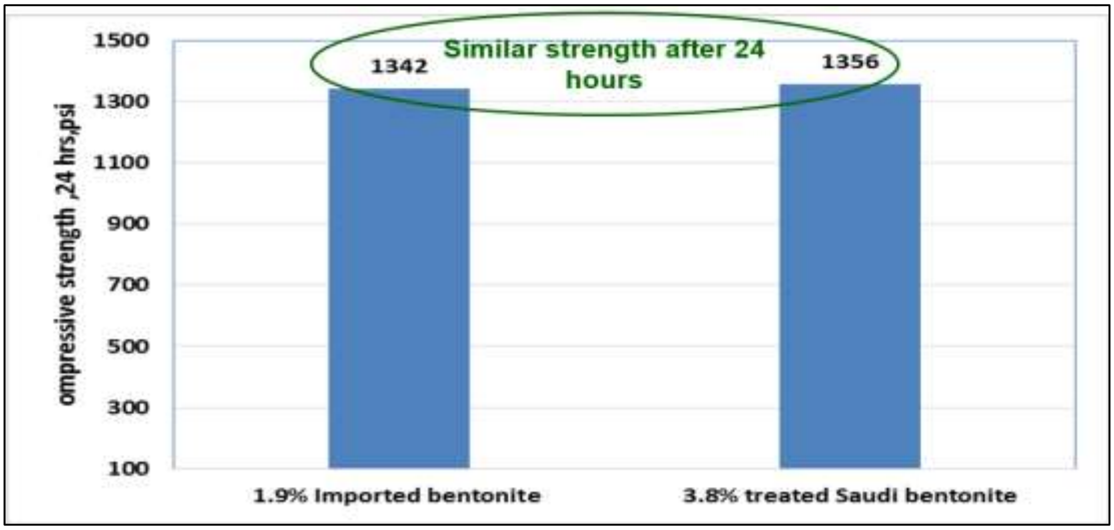

Figure 4-96 Compressive strength of commercial and treated Saudi bentonite after 24 hours

4.4.7 Effect of Treated SB on Compressive Strength by Sonic Method

UCA cement test was also implemented on the cement mix containing commercial as well as treated Saudi bentonite. **Figure 4-97**, **Figure 4-98,** and **Figure 4-99** show the results compressive strength obtained from UCA for 1.9% commercial bentonite, 1.9, and 3.8% treated Saudi bentonite admixed with cement.

Figure 4-97 Strength development of cement with 1.9% commercial bentonite by UCA for 48 hours

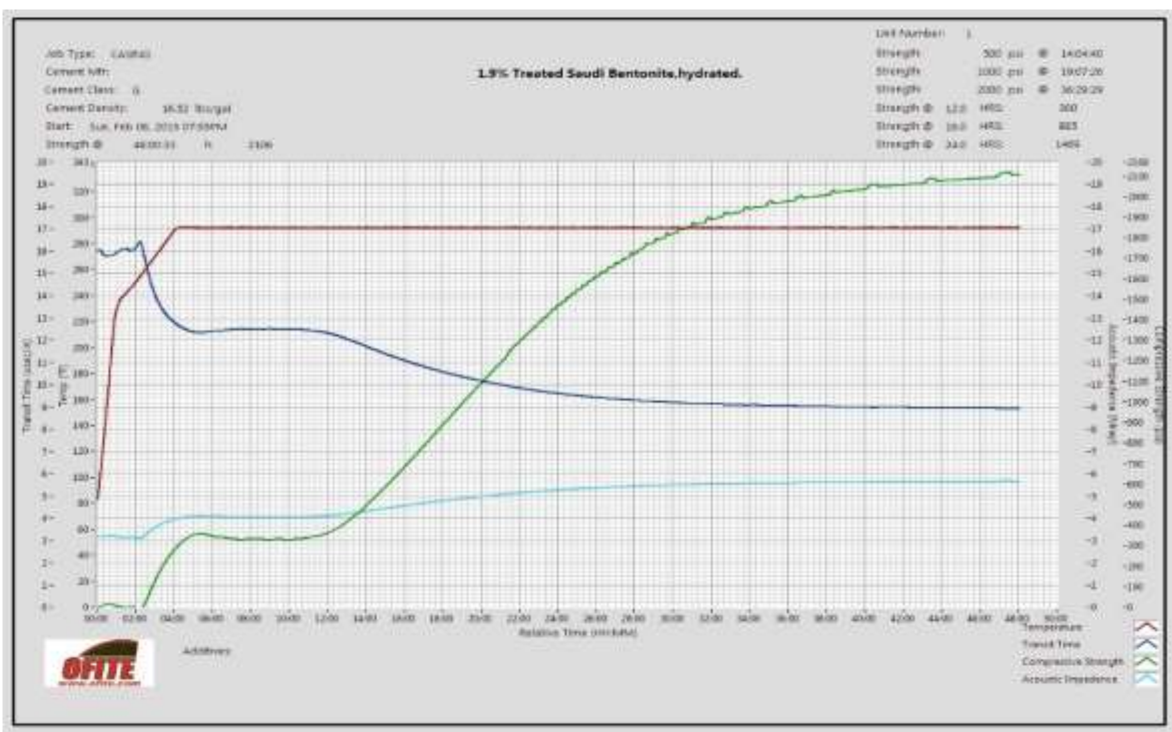

Figure 4-98 Strength development of cement with 1.9% treated SB by UCA for 48 hours

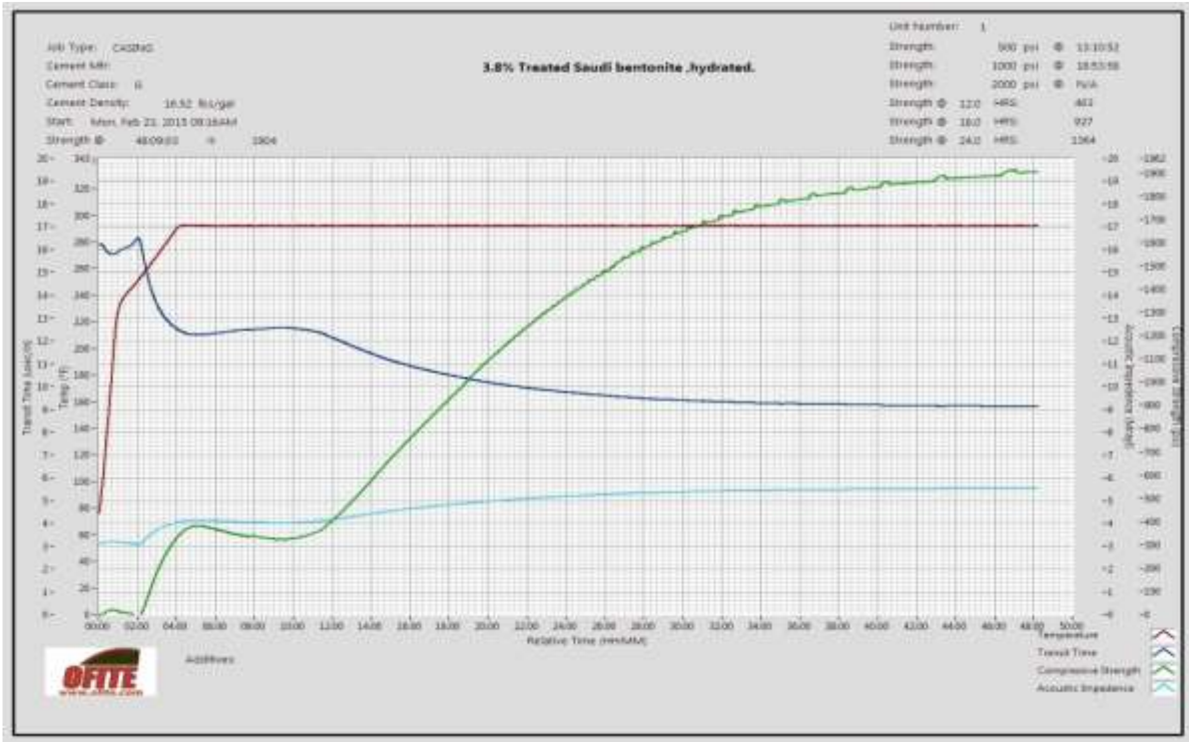

Figure 4-99 Strength development of cement with 3.8% treated SB by UCA for 48 hours

Table 4-25 shows strength development of commercial as well as treated Saudi cement systems at different times. When 1.9% treated Saudi bentonite was tested using UCA, the final compressive strength was higher than that obtained from the commercial bentonite cement system. The reason of this higher strength might be because of settling behaviour associated with these percentages as explained in the table. On the other hand, when 3.8% treated Saudi bentonite used, from above test of the free water as well as the rheology we observed that the settling property vanished, and the final compressive strength was almost the same around 1800 psi similar to that of commercial bentonite. **Table 4-26** shows the time the cement systems takes to achieve a strength of 50, 500, 1000, and 1500 psi.

Table 4-25 Compressive strength of the commercial and treated Saudi bentonite at various times

Time, hours	Compressive strength, Psi		
	1.9 % commercial bentonite	1.9 % Treated SB	3.8 % Treated SB
12:00	276	360	403
18:00	792	885	927
24:00	1287	1469	1364
48:00	1794	2100	1904

Table 4-26 Time to achieve a strength of 50, 500, 1000, and 1500 psi

Compressive Strength (psi)	Time to reach 50,500,1000, and 1500 psi strength		
	1.9 % Commercial bentonite	1.9 % Treated SB	3.8 % Treated SB
50	2:48	2:40	2:26
500	14:53	14:05	13:11
1000	20:10	19:07	18:54
1500	27:50	24:26	26:31

It was obvious that treated Saudi bentonite has an accelerating effect, since it showed early strength development compared with the cement system containing 1.9% commercial bentonite (see **Figure 4-100**, and **Figure 4-101**). It is obvious that the addition of 3.8% treated Saudi bentonite resulted in higher compressive strength of around 1900 psi, which is higher by 100 psi compared with commercial bentonite that produced 1800 psi, when systems cured for 48 hours as showed in **Figure 4-102**.

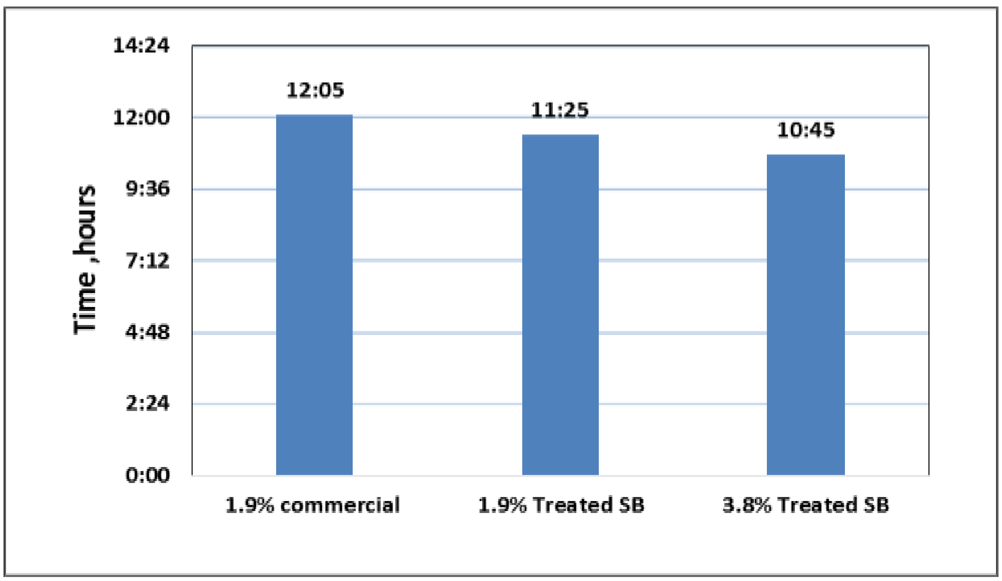

Figure 4-100 Transient time of the strength development between 50, and 500 psi

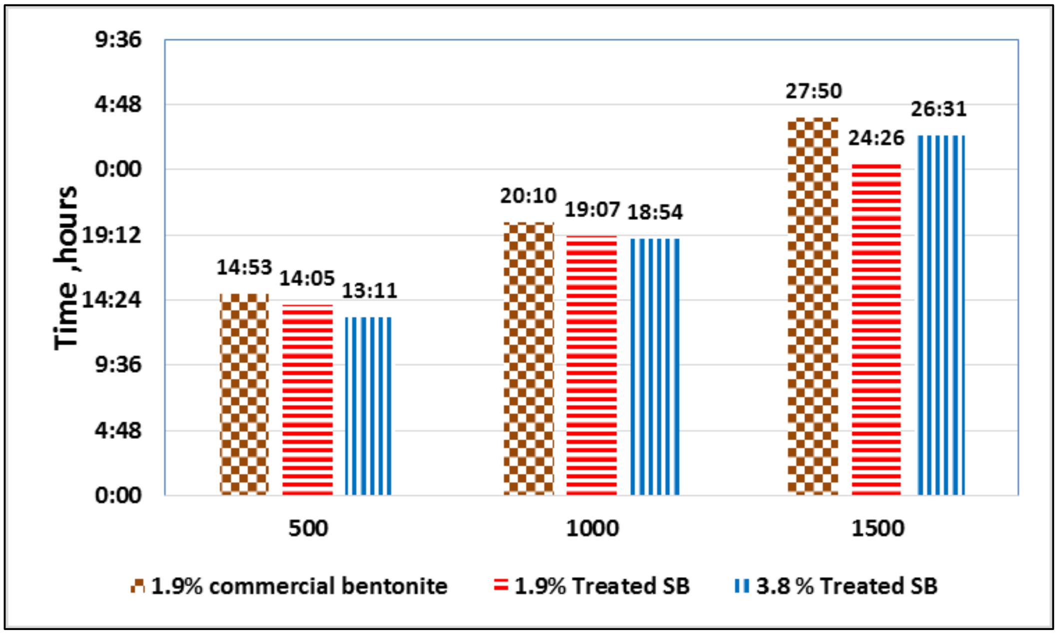

Figure 4-101 Time to reach 500, 1000, and 1500 psi strength

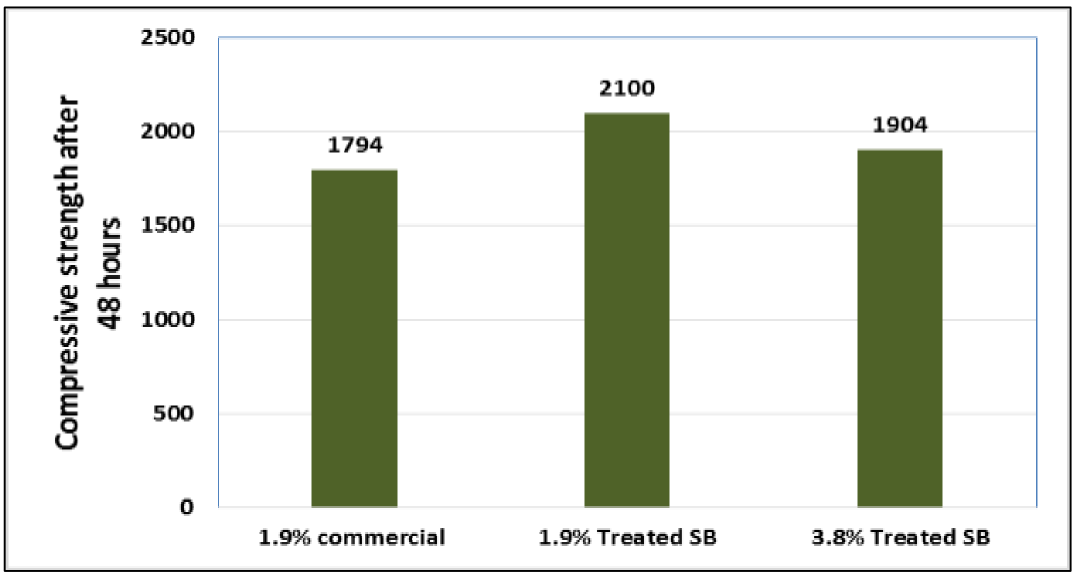

Figure 4-102 Final compressive strength of commercial and treated SB after 48 hours

4.4.8 Effect of Treated SB on Porosity and Permeability

Permeability is an important property, and 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 it.

In these experiments, after the cement cubes cured for 24, 48 hours in the curing machine, cement plugs are drilled out of them. Porosity and permeability cement tests are conducted using automated porosimter/permeameter under a confining pressure of 500 psi. **Table 4-27**, and **Table 4-28** represent porosity and permeability cement results of treated Saudi and commercial bentonite after 24, 48 hours curing.

Table 4-27 Porosity of cement with 1.9% commercial, and 1.9, 3.8% treated SB after 24 hours curing

Porosity %	1.9% Commercial bentonite	1.9% Treated SB	3.8% Treated SB
24 hours	51.45	-	47.55
48 hours	52.35	49.73	50.93

Table 4-28 Permeability of cement with 1.9% commercial, and 1.9, 3.8% treated SB after 24 hours curing

Permeability (md)	1.9% Commercial bentonite	1.9% Treated SB	3.8% Treated SB
24 hours	1.522	-	1.7481
48 hours	0.2244	0.0196	0.0359

From **Figure 4-103** we observed that when 3.8% treated Saudi bentonite added to cement mix, the produced cement had almost the same porosity compared with that obtained from 1.9% commercial bentonite of around 51%. In fact, the addition of treated Saudi bentonite caused a slight enhancement in the porosity of the produced cement sheath. Consequently, this improvement gives the treated Saudi bentonite some credit to be used in oil well cementing industry as a replacement of the commercial bentonite.

This trend was also observed in the case of cement permeability. As we can see in **Figure 4-104**, addition of treated Saudi bentonite resulted in a significant enhancement in the permeability of the cement sheath. In short, in the case of porosity and permeability, using 3.8% treated Saudi bentonite bwoc gave better results compared with commercial bentonite in low density cements which make it more favourable in using in the oil field cement industry.

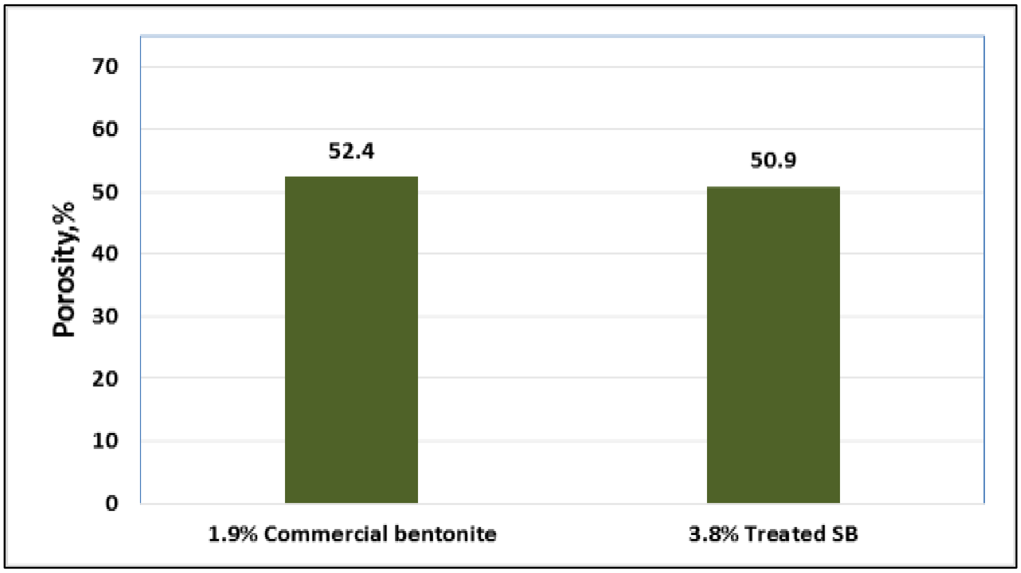

Figure 4-103 Porosity of cement with 1.9% commercial, and 3.8% treated SB after 48 hours

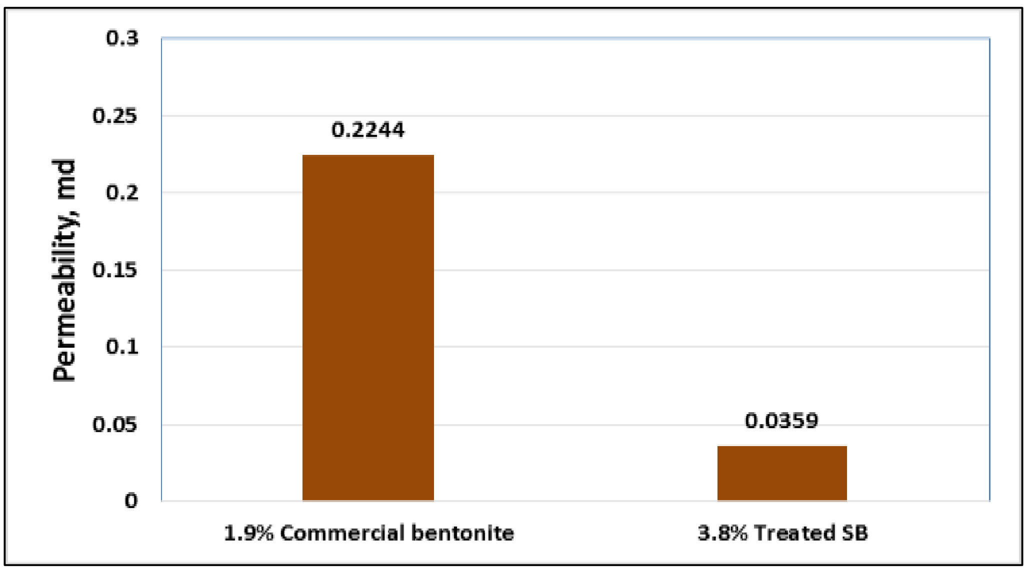

Figure 4-104 Permeability of cement with 1.9% commercial, and 3.8% treated SB after 48 hours

4.4.9 Microstructural Analysis Treated SB on the Cement

XRD and SEM tests are conducted on the cement systems containing 1.9 % commercial bentonite and 3.8 % treated Saudi bentonite and the results are described in details as follows:

4.4.9.1 Cement System with 1.9% Commercial Bentonite

It is well known that higher values of compressive strength can be obtained when higher percentages of CSH are appeared in the hydrated product. **Figure 4-105** shows the XRD hydration products with 1.9 % commercial bentonite at HPHT for 24 hours. The final hydrated products detected in the XRD were quartz (silicate), calcium silicate hydrate as well as Portlandite which later will transform to CSH. The high dense structure obtained due to the availability of quartz (silicate) which is a good kind of crystal, where it could interweave and join each other to build a perfect and well-network structure in the hardened cement (see **Figure 4-106**).

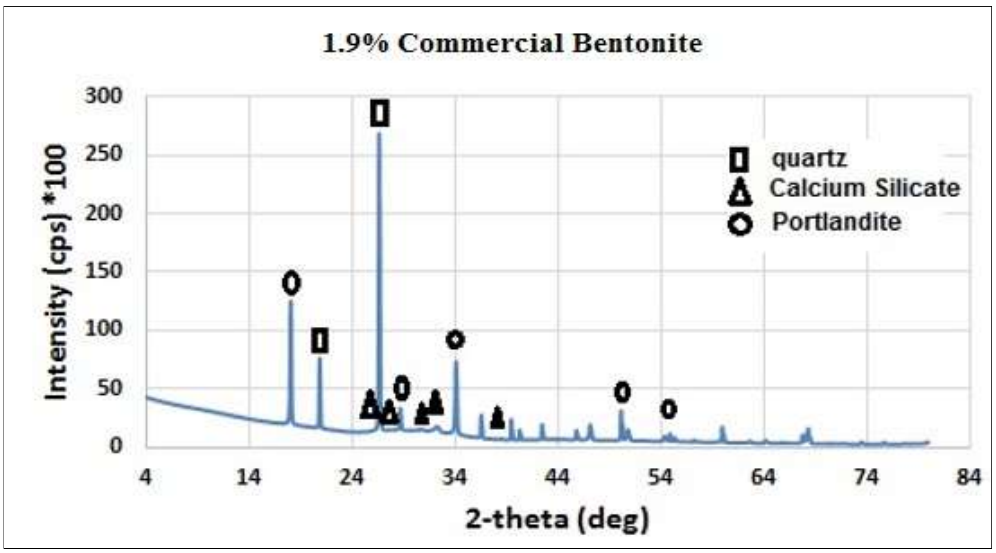

Figure 4-105 XRD hydration products with 1.9% commercial bentonite at HPHT for 24 hours

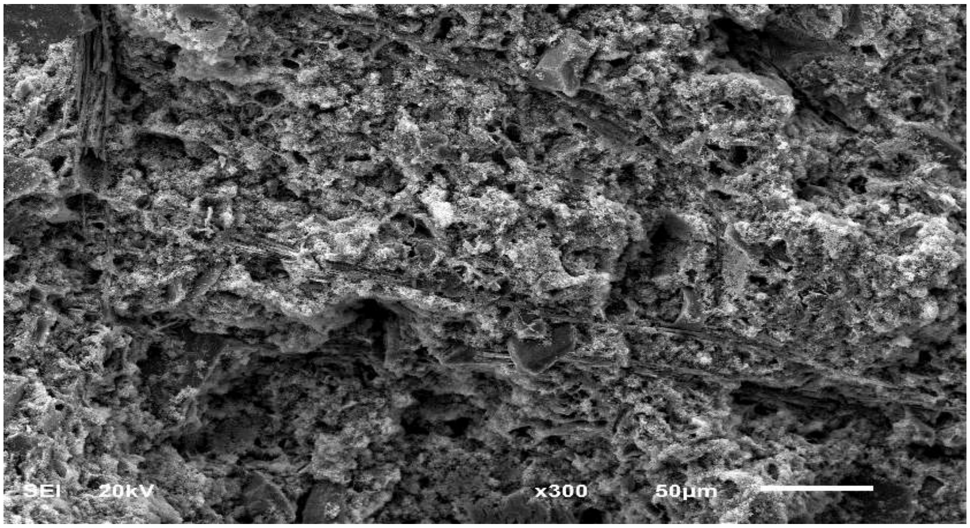

Figure 4-106 SEM photograph of hydration products with 1.9% commercial bentonite at HPHT for 24 hours

In fact addition of 1.9% commercial bentonite resulted in dense structure, and cause improvement in the produced compressive strength. **Figure 4-107** shows SEM element analysis for 1.9% commercial bentonite cured at HPHT for 24 hours. From the EDX results, it was clear that the final cement product contains a considerable weight percentages of calcium silica hydrate which confirm the formation of CSH in the final harden cement.

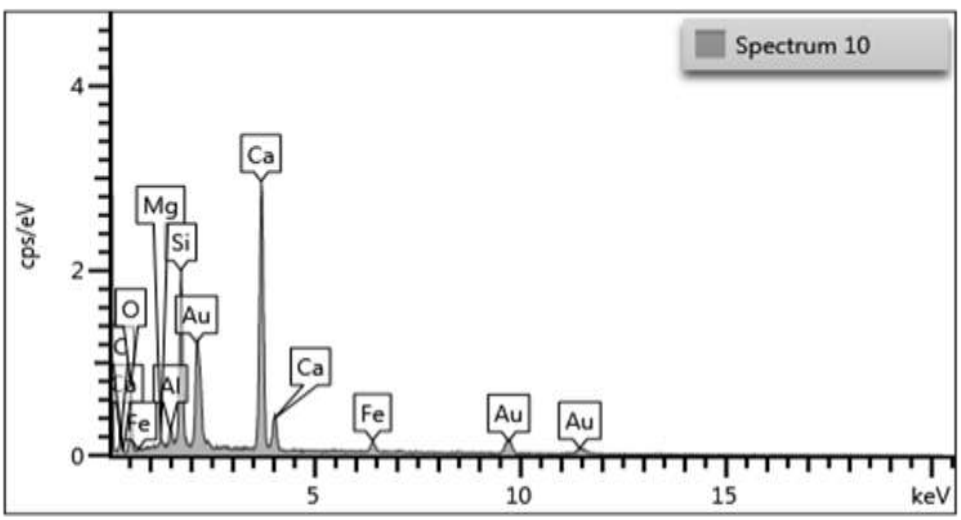

Figure 4-107 Hydration products (SEM) with 1.9% commercial bentonite at HPHT for 24 hours

4.4.9.2 Cement System with 3.8% Treated Saudi Bentonite

The same trend was observed with the addition of 3.8% treated Saudi bentonite. It was clear that treated Saudi bentonite caused more polymerization in the final cement paste. It also gave higher compressive strength as a result of higher percentages of CSH, which are detected in the hydrated product. **Figure 4-108** shows the XRD hydration products with 3.8% treated Saudi bentonite at HPHT for 24 hours. The final hydrated products detected in the XRD were quartz (silicate), calcium silicate hydrate as well as Portlandite which later will transform to CSH. It is well known that CSH is a good kind of crystal, where it could interweave and join each other to build an ideal and well-proportioned network structure in the hardened cement as shown in the SEM image (see **Figure 4-109**). Consequently, the addition of 3.8% treated Saudi bentonite to the cement mix resulted in dense structure, and cause enhancement in the final strength as proved using the sonic and the crushing method.

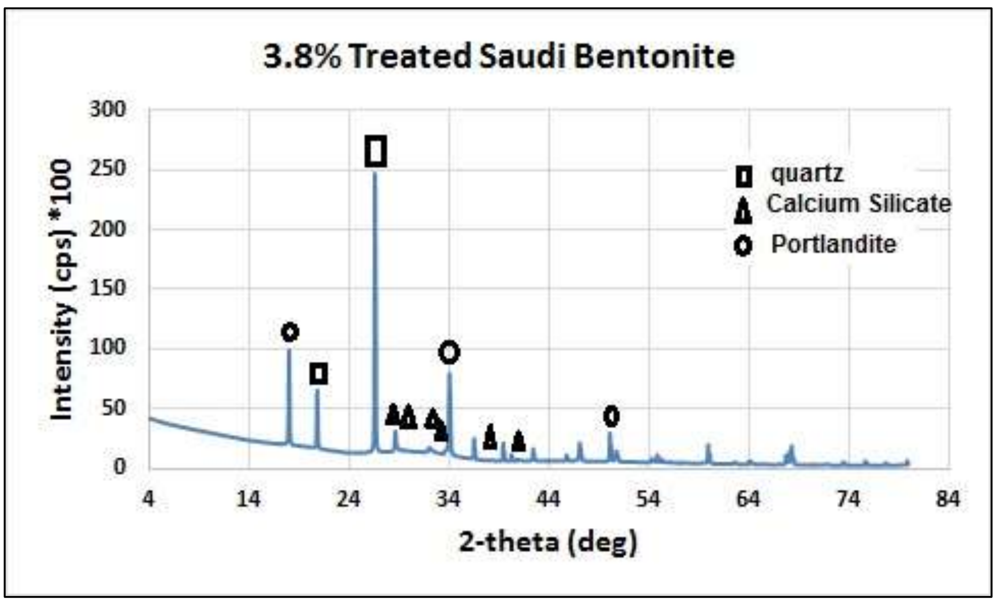

Figure 4-108 XRD hydration products with 3.8% treated SB at HPHT for 24 hours

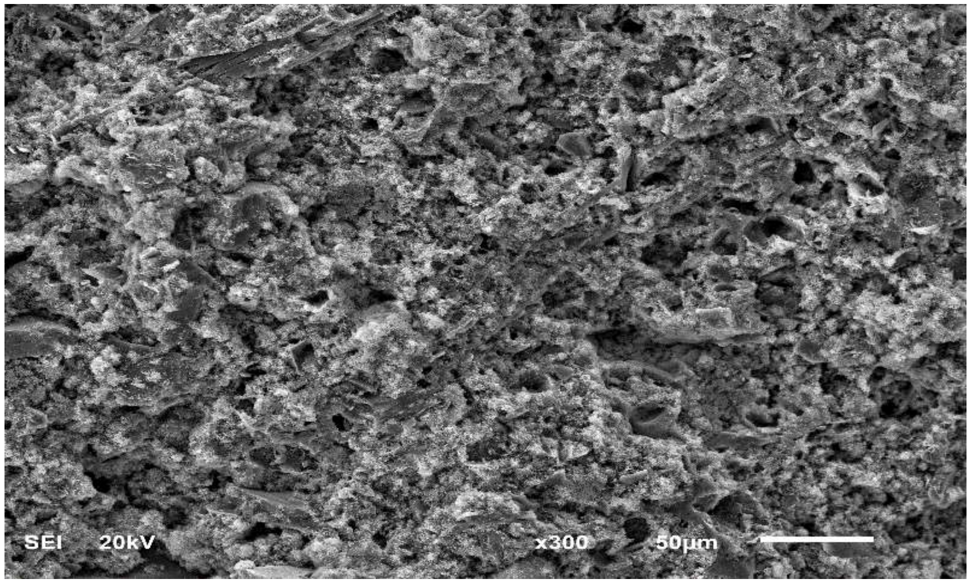

Figure 4-109 SEM photograph of hydration products with 3.8% treated SB at HPHT for 24 hours

It was obvious that adding 3.8% treated Saudi bentonite to the cement mix produced a dense structure, and caused enhancement in the final value of the reported compressive strength. **Figure 4-110** shows SEM element analysis for 3.8% of the treated Saudi bentonite cured at HPHT for 24 hours. From the EDX results, it was observed that the final cement product contains higher weight percentages of calcium silica hydrate which confirm the formation of CSH in the final harden cement.

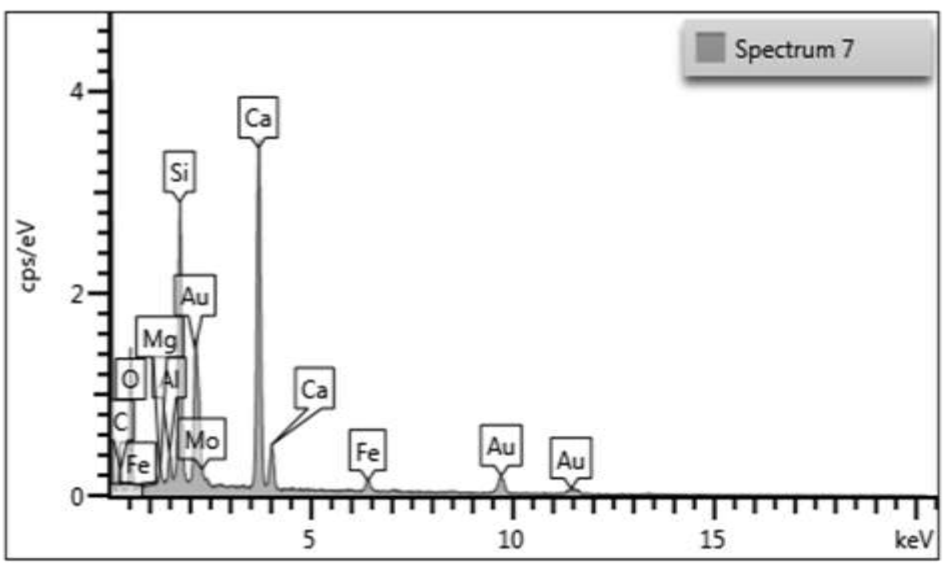

Figure 4-110 Hydration products (SEM) with 3.8% treated SB at HPHT for 24 hours

4.4.9.3 Comparing SEM Results for Commercial and 3.8% Treated Saudi Bentonite

As we mention above, that the addition of 3.8% treated Saudi bentonite to the cement mix resulted in more polymerization as detected in the final produced cement sheath. In fact, if we compare the SEM images of 5 micron size for both cement systems, 1.9% commercial bentonite and 3.8% treated Saudi cement system, we observed that 1.9% commercial bentonite cement mix exposed more vugs and showed less polymerization compared with the cement system containing 3.8% treated Saudi bentonite as shown in **Figure 4-111**.

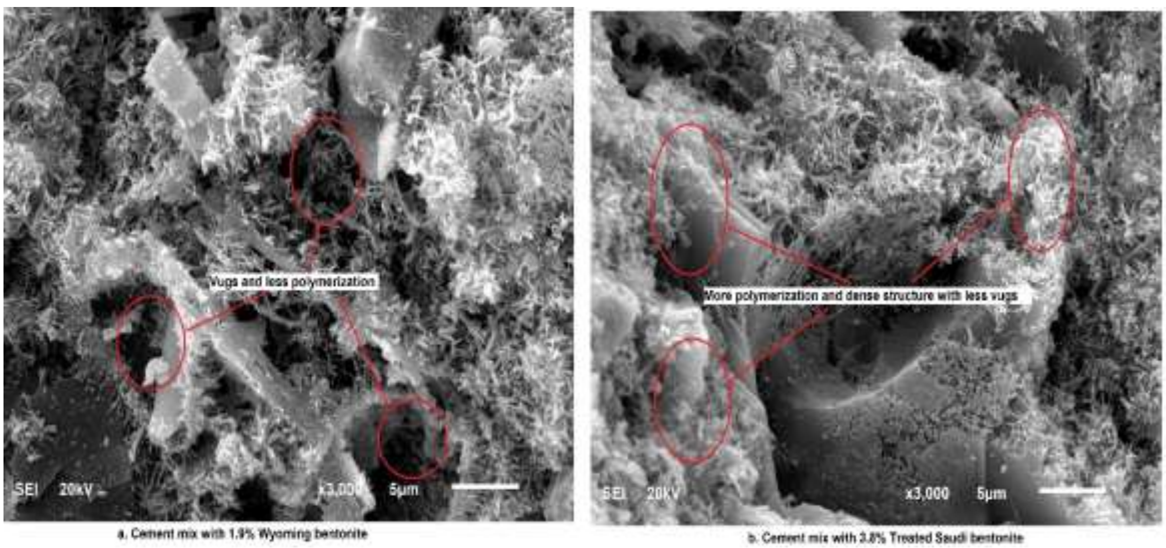

Figure 4-111 SEM 5 micron images comparing 1.9% commercial and 3.8 % treated SB

CHAPTER 5

Conclusions and Recommendations

5.1 Conclusions

In this work we examined the effect of adding three materials on the cement properties under HPHT conditions. The effect of adding untreated Saudi bentonite, untreated Saudi bentonite admixed with Nano clay, and the treated (upgraded) Saudi bentonite on the cement properties under HPHT conditions has been investigated and reported. The conclusions obtained from this work are summarized as follows:

1. Experimental findings of cement system with untreated Saudi bentonite on the cement slurry properties:
    a. It works as cement retarder, since it slows the hydration reaction, which is advantageous in the case of cementing deep wells.
    b. Zero free water was observed in all cement systems after aging for two hours, where untreated Saudi bentonite blocks the capillaries and stops the water flow.
    c. It produced almost the same cement density, and resulted in an enhancement in the plastic viscosity and yield point of the produced cement slurry. This enhancement in cement properties makes it good viscosifier, and helps in efficient mud displacement.
    d. It lowered the amount of the fluid loss, which gives more control the water cement ratio as well as cement density.
    e. A cement system containing untreated Saudi bentonite caused a slight reduction in porosity associated with a considerable decrease in permeability.
    f. The optimum percentage used was 1% untreated Saudi bentonite, and it had the following advantages:
        - 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 8 hours and 23 minutes, which is lower than the interval period (10 hours and 52 minutes) obtained by using the cement base mix. This behavior is advantageous in reducing wait on cement time.

- The high compressive strength achieved was around 7000 psi with this percentage tested using sonic method after 48 hours of the curing process.
- This trend was also conformed when material tested using the crushing method, where the highest compressive strength reported was around 6850 psi after 24 hours of the curing process.

g. Further addition of untreated Saudi bentonite affected the cement properties and lowered the final strength.

h. Microstructural analysis showed that untreated Saudi bentonite particles block the capillaries by filling the pores in the cement, so a dense cement structure is achieved.

2. Experimental findings of cement system with 1% untreated Saudi bentonite admixed with Nano clay on the cement slurry properties:

a. It acts a retarder, and resulted in zero free water when aged for two hours.

b. It gave almost the same cement density, and caused significant improvement in the plastic viscosity and yield point which helps in the case of mud removal.

c. It reduced the cement fluid loss.

d. When Nano clay with percentages of 0.5, 1, and 1.5% added to the cement mix containing 1% untreated Saudi bentonite, a slight reduction in the enhancement of the cement properties was observed. For example, the final compressive strength with all Nano clay cement systems was reduced when compared with 1% untreated Saudi bentonite cement system. However, the addition of Nano clay, for instance, 1%, showed more advantages in other cement properties.

e. The optimum percentage used was 1% untreated Saudi bentonite admixed with 1% Nano clay and it had the following advantages:

- Early strength development was achieved with UCA, where the 2000 psi strength was reached after 6 hours and 11 minutes, which is the lowest time period achieved compared with all cement systems.

- It provided the highest compressive strength (6700 psi) of the Nano clay system, which is in fact lower by 4% compared with 1% untreated Saudi bentonite cement system when tested using sonic method after 48 hours of the curing process.
- The same trend was confirmed by the crushing method, where the highest compressive strength reported was around 6525 psi, which is lower by 4.7% from 1% untreated Saudi bentonite cement system after 24 hours of the curing process.
- The lowest porosity and permeability results achieved was around 26%, and 0.0025 md, which were considered low compared with that obtained from 1% untreated Saudi bentonite cement by 30.7% and 0.003 md respectively.

f. Microstructural analysis showed Nano clay particles block the capillaries by filling the pores, and produced a dense cement structure. In short, Nano clay acts as a filler when added to the cement mix.

3. Experimental findings of cement system with treated (upgraded) Saudi bentonite on the cement slurry properties:

   a. Almost Similar results of using 1.9% of commercial bentonite on the cement slurry have been obtained with the double quantity (3.8%) of treated Saudi bentonite besides obtaining good gelling property associated with excellent cement dispersion through the whole mix without any settling of the cement particles. The obtained results give a good indication that treated Saudi bentonite can be used as an alternative to commercial bentonite for low density cement systems in the case of oil well cementing applications.
   b. It acts as a cement retarder, where it slows the hydration reaction.
   c. Free water was almost the same as the 1.9% commercial bentonite.
   d. When 1.9% treated Saudi bentonite used, poor rheology was obtained. On the other hand, 3.8% treated Saudi bentonite gave acceptable rheology, where the plastic viscosity and yield point results were close compared with 1.9% commercial bentonite.

e. A similar strength (around 1350 psi) was obtained for both systems, 3.8% treated, and 1.9% commercial bentonite when tested using the crushing method after 24 hours of the curing process.
f. Early strength development was achieved with 3.8% treated Saudi bentonite compared with 1.9% commercial bentonite tested using sonic method, for example, the 1500 psi strength was reached after 26 hours and 31 minutes, which is lower than the interval period (27 hours and 50 minutes) obtained by using commercial bentonite cement system. This early strength development behavior helps in the case of reducing the wait on cement time (WOC).
g. The final strength was 1904 psi, which is high with 100 psi compared with commercial bentonite cement system (around 1794 psi) after 48 hours of the curing process.
h. Significant reduction in permeability associated with a slight decrease in porosity was achieved with the treated bentonite compared commercial bentonite cement system.
i. Microstructural analysis displayed that treated Saudi bentonite particles block the capillaries, and formed a more dense cement structure when compared with the commercial bentonite cement system.

5.2 Recommendations

- In this work, the effect of untreated Saudi bentonite, and untreated Saudi bentonite admixed with Nano clay has been investigated in the presence of silica products such as silica flour. Thus, removing silica products from the cement design might give better results, and improve the cement system performance.
- Testing the performance of untreated Saudi bentonite, untreated Saudi bentonite admixed with Nano clay, and the treated Saudi bentonite in the case of cementing shallow wells with low pressure and temperature, and also with different water cement ratio.
- Nano clay, untreated Saudi bentonite, Nano silica gave high integrity to cement sheath when tested separately in conditions of both high temperature applications.

The combinations of them should be designed and tested to see their effect on the properties of oil well cement.

- Treated (upgraded) Saudi bentonite showed similar properties compared with that of commercial bentonite. Adding Nano clay to the cement mix with different percentages might result in significant improvement in the properties of the cement sheath as well as the cement performance.
- This research is focused on the experimental results in the presence of untreated Saudi bentonite, upgraded Saudi bentonite, as well as Nano clay. Different kinds of analytical models should be designed and investigated to see their effect on cement properties such as cement rheology and mechanical properties.

Appendix: SEM images for cement system having 0, 1, 2, and 3 % of untreated Saudi bentonite

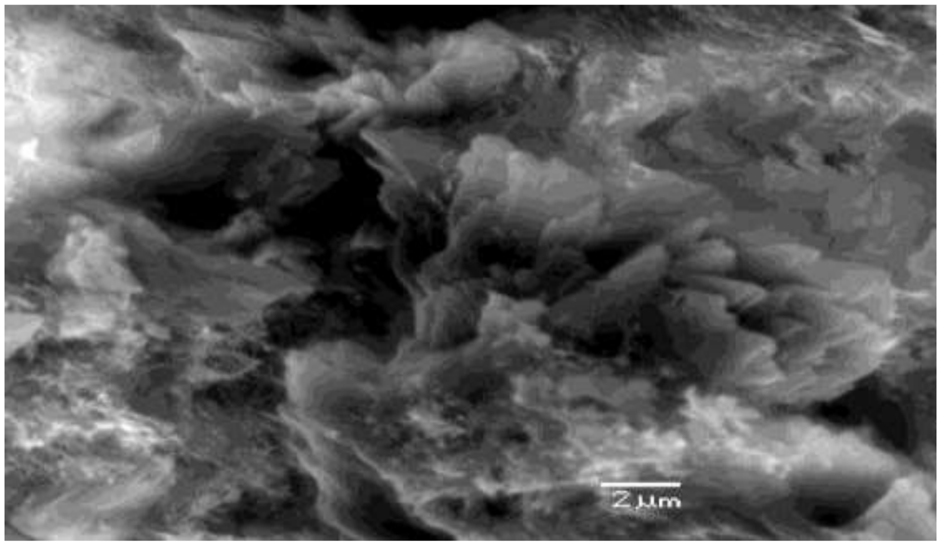

Figure A- 1 SEM of simple G class cement at HPHT for 24 hours

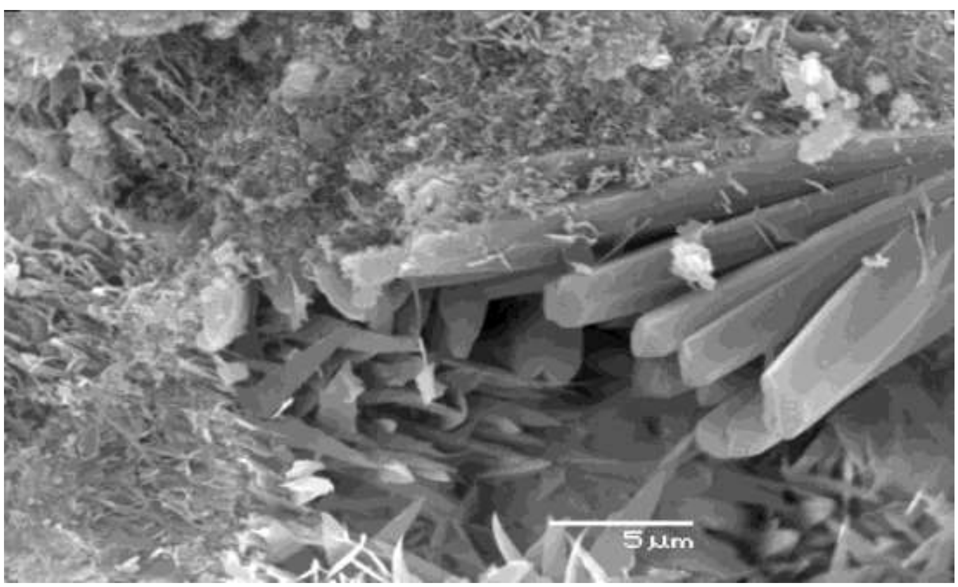

Figure A- 2 SEM of cement having 0% untreated SB at HPHT after 24 hours

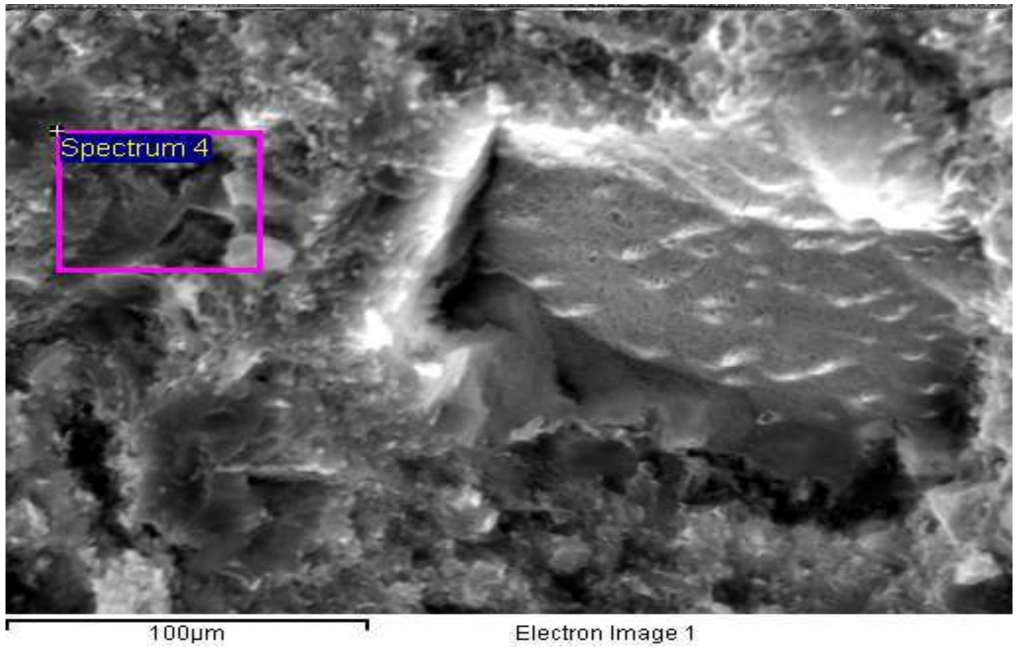

Figure A- 3 SEM of cement having 0% untreated SB at HPHT after 48 hours

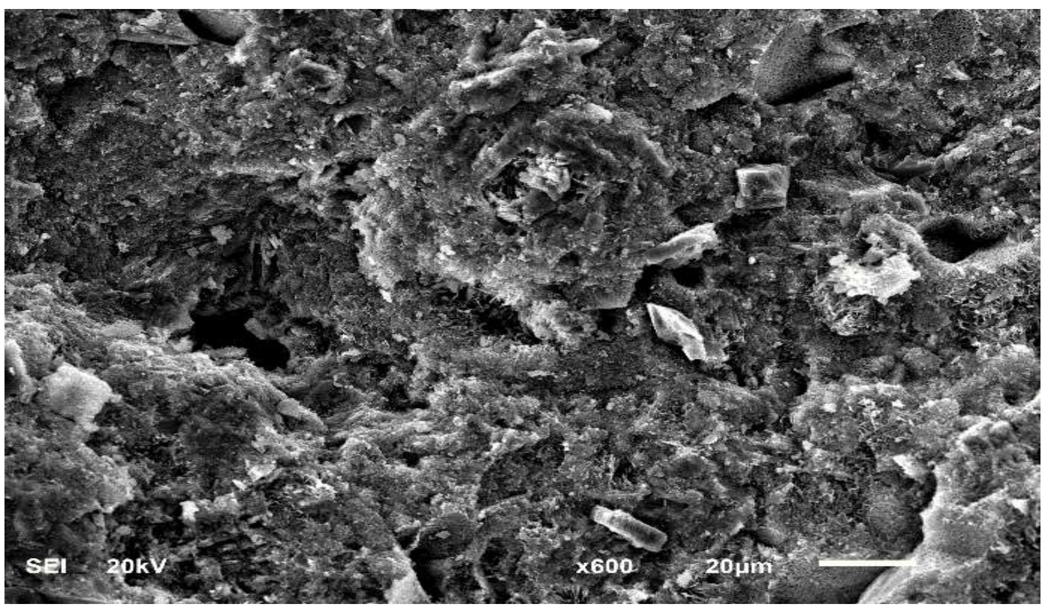

Figure A- 4 SEM of cement having 1% untreated SB at HPHT after 24 hours

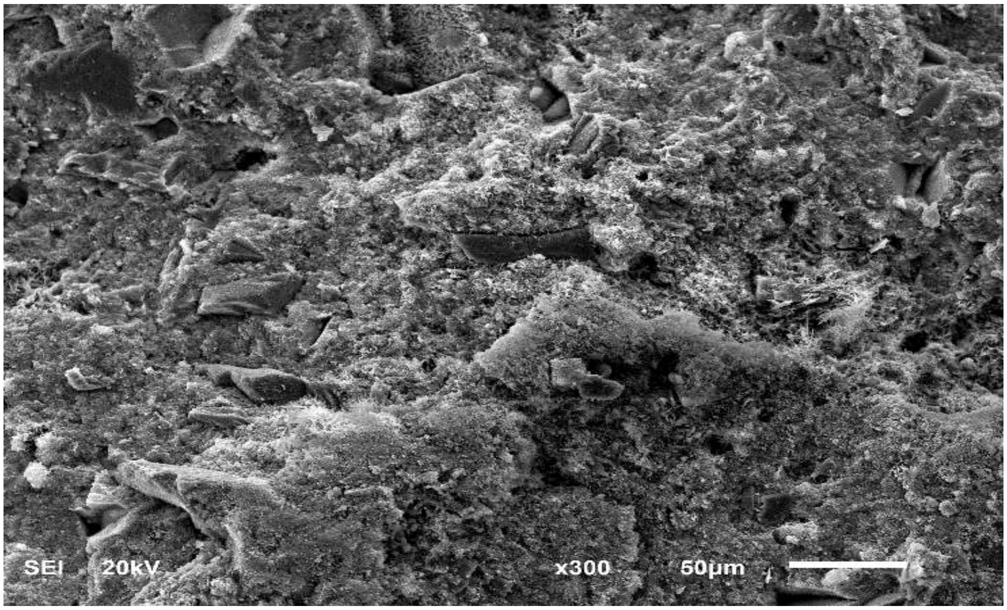

Figure A- 5 SEM of cement having 2% untreated SB at HPHT after 24 hours

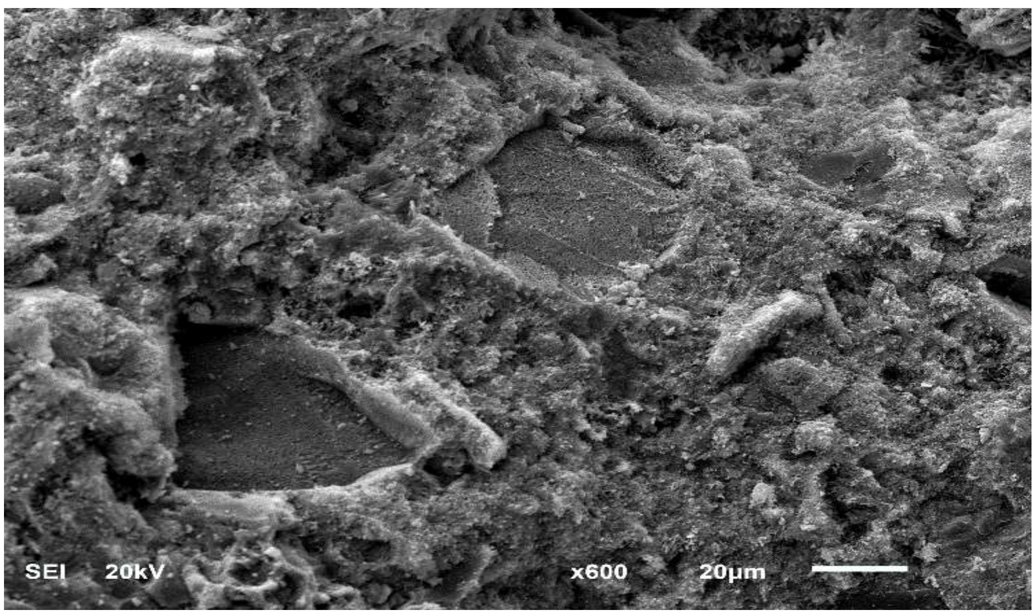

Figure A- 6 SEM of cement having 3% untreated SB at HPHT after 24 hours

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