

# Foamed Cement Formulation for Iranian Casing and

## **Liner Cement Job**

## By:

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### A THESIS SUBMITTED TO THE DEPARTMENT OF PETROLEUM ENGINEERING IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE IN PETROLEUM ENGINEERING

October 2018







This Thesis Is Supported and Sponsored by National Iranian Drilling Company (NIDC)

#### October 2018

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A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Science (M.Sc.)

In Petroleum Engineering

Petroleum University of Technology, Ahwaz

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**DEDICATION** 

This thesis is Dedicated to my beloved parents

for their love, endless support, encouragement, and

sacrifices.

#### ABSTRACT

Cementing is one of the most important and fundamental phases of any oil and gas wells drilling operations. In addition, by increasing the oil and gas production, the reservoir becomes depleted. Thus, for production rate enhancement, new wells are drilled. Cementing jobs at low pressure or fractured layers must be conducted by light or ultra-lightweight slurries. Therefore, the formation damage that is caused by slurries, reduces essentially. One of the latest technologies for ultra-lightweight cementing jobs is the foamed cement formulation. In this thesis, the new foamed cement formulation and designing for Iranian liner and casing cement jobs are investigated. Foamed cements are prepared at high shear rates by sodium lauryl ether sulphate (SLES) as a foaming agent. The optimum concentration for SLES and optimum mixing procedure are evaluated and other additives for a particular property are added to the slurry. Different tests such as foam stability, density measurement, rheology, free water, thickening time, compressive strength, fluid loss, porosity, permeability, microscopic photography and CT scan image processing are conducted for each of cement samples. The results of these experiments for FBJ (bestformulated foamed cement in this study) are compared to other common lightweight cements that are used in Iranian fields, for instance, south Azadegan oil field. The FBJ foamed slurry consists different components, for best foamed cement properties and formulation, that are 778g net class G cement, 350g distilled water, 0.78g (0.1% BWOC) SLES as a foaming agent, 0.78g (0.1% BWOC) JR120 as a retarder and 0.78g (0.1% BWOC) boric acid. The results illustrate that the FBJ has one of the lowest densities in the Iranian cementing industry with enough compressive strength. The

density of FBJ is 75pcf with more than 1300psi compressive strength. Its thickening time is near 3 hours and its rheology becomes better in wellbore condition and this is a result of bubbles ball-bearing effect. Due to the foamy structure of FBJ, this slurry has no free water and fluid loss. The porous media of FBJ's set-cement are not interconnected and this is proved by porosity, permeability, microscopic photography and CT scan tests. Also, CT scan analyses show that FBJ's foamy structure is saved in the set-cement in high pressure- high temperature HPHT condition.

#### ACKNOWLEDGMENT

First and foremost, I would like to thank God Almighty for giving me the strength, knowledge, ability and opportunity to undertake this research study and to persevere and complete it satisfactorily. Without his blessings, this achievement would not have been possible.

I would like to express my deep gratitude to Professor Dr. Seyed Reza Shadizadeh and Dr. Jamshid Moghadasi, my research supervisors, for their patient guidance, enthusiastic encouragement and useful critiques of this research work.

I take pride in acknowledging the insightful guidance of Eng. Hadi Tabandejou, my industrial advisor, NIDC's engineering and well cement planning department manager, for sparing his valuable time whenever I approached him and showing me the way ahead.

I am particularly grateful for the assistance given by NIDC's engineering and well cement planning department experts, Eng. Saeed Owrak, Eng. Hamed Ranjbar and Eng. Mohammad Khodadadian.

I thank Eng. Maryam Behnia, NIDC's cement, and drilling acid laboratories quality control administrator, and Dr. Alireza Shakibafard, the manager of TABA Medical Imaging Center, for their help during this project conduction.

I would also like to extend my thanks to the technicians of the cement laboratory of NIDC and Petroleum University of Technology's petroleum, cement and material laboratories for their help in doing the experimental research of this thesis.

I would like to acknowledge the people who mean the world to me, my parents and my brothers. I don't imagine a life without their love and blessings. Thank you, dad and mom, for showing faith in me and giving me the liberty to choose what I desired. I consider myself the luckiest in the world to have such a supportive family, standing behind me with their love and support.

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### NOMENCLATURES

А	Cement Plug Cross Section Area (cm <sup>2</sup> )	
AC	Atmospheric Consistometer	
ACI	American Concrete Institute	
API	American Petroleum Institute	
API RP	American Petroleum Institute Recommended Practice	
ASTM	American Section of the International Association	
Bc	Bearden units of Consistency	
BHCT	Bottom-hole Circulating Temperature	
BHSP	Bottom-hole Static Pressure	
BHST Bottom-hole Static Temperature		
BWOC By Weight of Cement		
C1, C2, C3 Lightweight Cement Composition 1, Composition 2, Composition		
CDA	DA Set-cement Compressive Strength to Slurry Density Ratio (psi/pc	
CMHEC	Carboxymethyl Hydroxyethyl Cellulose	
CMT	Cement	
cP	Centipoise	
CS	Compressive strength	
CT Scan	Computed Tomography Scan	
D	Darcy	
DC	Direct Current Voltage Consistency	
DW	Distilled Water	
FA	Foam Agent	
FBJ The Current Thesis Formulated Foamed Cement with Foam Ag Boric Acid +JR120		

FL	Fluid Loss	
FLA	Fluid Loss Additives	
FLC	Fluid Loss Control	
ft	Feet	
G	Net Class G Cement	
g, gr	Grams	
gal	Gallon	
HEC	Hydroxyethly Cellulose	
HPHT	High Pressure High Temperature	
hr	Hour	
HSR	High Sulphate Resistance	
HT	High Temperature	
in	Inches	
ISO	International Organization for Standardization	
Κ	Permeability (mD, D)	
L	Liters	
lb <sub>m</sub>	Pound Mass	
$lb_{\rm f}$	Pound Force	
LCM	Lost Circulating Materials	
min	Minutes	
mD	Milli Darcy	
mg, mgr	Milli Gram	
mL	Milli Liter	
MPa	Mega Pascal	
MPD	Managed Pressure Drilling	
MSR	Moderate Sulphate Resistance	

NIDC	National Iranian Drilling Company		
0	Ordinary Cements		
OF	The Foamed Cement with Optimum Concentration Foam Agent		
OPC	Ordinary Portland Cement		
pcf	Pounds per Cubic Feet		
PNS	Polynapthalene Sulphonate		
ppg	Pounds per Gallon		
psi	Pounds per Square inch		
PUT Petroleum University of Technology			
PV	Plastic Viscosity (cP)		
PW	Piping or City Water		
RPM	Round per Minute		
Q <sub>foam</sub>	Foam Quality		
R.T	Room Temperature		
s <sup>-1</sup>	Inverse Second		
SBM	Synthetic-Based Mud		
Sec, s	Seconds		
SG	Specific Gravity		
SLES	Sodium Lauryl Ether Sulphate		
SL. WT.	Slurry Weight (pcf)		
Spec API	Specification for a Test by American Petroleum Institute		
Т	Temperature		
TD	Total Depth		
TT	Thickening Time (min)		
TVD	True Vertical Depth		
UCA	Ultrasonic Cement Analyzer		

V	Volume (cc or cm <sup>3</sup> )
VG meter	Viscosity-Gel meter
Sx	Sacks
WOC	Waiting on Cement
wt	Weight (gr, mgr)
YP	Yield Point (lb <sub>f</sub> /100ft <sup>2</sup> )
2D	Two Dimensional
3D	Three Dimensional
°C	Centigrade Degree
$\Delta P$	Differential Pressure (psi)
°F	Fahrenheit Degree
γ	Gamma, Shear Rate (s <sup>-1</sup> )
μ	Mu, Viscosity (cP)
φ	Phi, Porosity (%)
ρ	Rho, Density (pcf, ppg, etc.)
τ	Tau, Shear Stress (psi)

#### **CHAPTER ONE**

#### **INTRODUCTION**

#### **1.1. Fundamental Descriptions**

When an oil or gas well is drilled, pipe (casing or liners) is put into the ground to protect the well by keeping formation debris from falling into it and even against well collapse. The casing also provides a barrier against formation pressures and formation fluid flows, isolates freshwater aquifers, and provides smooth, solid surfaces for further work in the well. The casing is typically made of steel or corrosion-resistant metal alloys to provide for high strength. In some cases, fiberglass or composites are used to make a casing that is highly resistant to corrosion. A guide shoe (a rounded, steel cover or cap) is placed at the end of the casing in the hole to protect the casing and guide it during installation into the well. The casings and liners are bonded to the formation by drilling cement [1].

The cement is used as a sealing material while drilling an oil and gas well. Pumping cement behind the casing provides a barrier to the fluids flow from or into the formation and bonds the casing or liner to the formation. This is called a primary cement job. Cement is also pumped for other reasons like sealing off perforated casing or plugging zones. The annulus, the space that remains between the casing and the formation, is then filled with cement by pumping the cement slurry through the casing and back up the annulus. A wiper plug ("top" plug) is used behind the cement to push the cement up the annulus. A wiper plug ("bottom" plug) is always a good idea to run ahead of the cement. A displacement fluid (such as water) is pumped behind the plug to push it and the cement down the casing [2].

There are many reasons for pumping cement slurry while drilling operations, the most important functions of primary cementing are:

- Cement supports the casing, so the cement should completely surround the casing; this is where centralizers on the casing help. If the casing is centered in the hole, a cement sheath should completely envelop the casing;
- Cement seals off formations to prevent fluids from one formation migrating up or down the hole and polluting the fluids in another formation (also known as zonal isolation). For example, cement can protect a freshwater formation (that perhaps a nearby town is using as its drinking water supply) from saltwater contamination;
- Cement helps prevent blowouts by setting rapidly;
- Cement protects the casing from the corrosive effects that formation fluids (as salt water) may have on it;
- Cement protects the casing from shock loads when drilling deep;
- Cement seals off formation areas that might allow fluids to leak off (lost circulation or thief zones);
- Cement protects the environment by controlling the flow of fluids;
- Cement can be used to plug an old well (abandonment) or to plug a depleted zone [3].

A successful primary cementing job is crucial for the well integrity to allow continuing the drilling operations. Successful primary cementing operations result in a cement sheath to bond and support casing and provide zonal isolation. Good zonal isolation helps prevent the loss of production, control inter-zonal flow and/or flow to the surface, reduce water production and improve confinement of stimulation treatments. In other cases, cement has to be pumped into the well and forced to flow into the formation; this process is called secondary or squeezing cementing job. Without adequate zonal isolation, wells are unlikely to reach their full producing potential. The ultimate goal of cementing is to provide well integrity through zonal isolation a durable hydraulic seal in the wellbore that allows selective fluid production from subsurface formations and prevents leaks into other formations or to the surface. As production companies pursue reserves in high-cost, high-risk environments, well cementing and sustained wellbore integrity are increasingly critical to the success of the reservoir exploitation cycle. Well cementing draws on several disciplines, including chemistry, mechanical engineering, fluid mechanics, mathematics, and geology. Figure 1.1 illustrates the simple schematic of the cemented well.



Figure 1.1. The simple schematic of a cemented well [1]

Many wells are drilled in areas where weak zones cannot support a cement column of normal density in excess of 15 ppg (112 pcf). These situations require the use of low-density cement systems that reduce the hydrostatic pressure of the fluid column during cement placement. Lightweight additives such as bentonite and gilsonite are used to reduce the weight of the slurry to as low as 12 ppg. Another method to reduce the density of cement is to mix cement slurries with foaming agents and gas (air or nitrogen). With foamed cements, densities as low as 8-10 ppg can be achieved while maintaining good strength properties in the cured cement. Fluids in wellbores exert hydrostatic pressure on points downhole. The hydrostatic pressure is dependent upon the fluid column height and the density of the fluid. Formations which are very weak or which contain holes or pressure from vugs are able to support the hydrostatic from only very light fluids. Some of these formations will not even support a column of water. Conventional cements mixed with water must always have a density in excess of 8.33 ppg and most of the time is more than 15 ppg (for conventional cements); thus, frequently it is not possible to place such slurries in a wellbore. Foamed cement has been developed to allow cementing in such cases. Foamed cement can be a solution for cementing problems related to formations which are fractured (or which, have a low fracture gradient), highly permeable, vuggy or cavernous. Applications include primary cementing, remedial cementing, lost-circulation solutions and annular thermal insulation [3].

#### 1.2. Objective

The aim of this thesis is to formulate and design new foam cement for Iranian casing and liner cement job for the first time in Iran oil industry. By increasing the oil and gas production rates and the end of the most of Iranian reservoirs life, the pressure of reservoirs is decreased. Furthermore, close to 87% of Iranian reservoirs are fractured. The most common problems during a cement job in drilled wells in depleted and fractured reservoirs are slurry lost and formation fracturing. Thus, in these reservoirs, lightweight cement must be used. Lightweight cement has a column of slurry with the low hydrostatic pressure that causes the least damages in the depleted and fractured reservoirs. Unfortunately, in Iran, most of the lightweight cements have the low compressive strength and some of them, which have enough CS, could not reduce the density very much and they have expensive additives. The reason for this limitation in density reduction is that all of the common additives are solid. This new work for lightweight cement designing as a foam cement in Iran oil well-cementing industry eliminates most of the conventional cements disadvantages. This innovation in foam cement formulation and mixing procedure makes an economical and optimum cement job operation.

This research is supported and sponsored by National Iranian Drilling Company (NIDC) and most of the thesis's experimental jobs are done in NIDC's Cement Laboratory. Several experiments were conducted such as Surface and Under Temperature Rheology and Density, Thickening Time, Free Water, Compressive Strength, Fluid Loss, Permeability and Porosity tests. Two different photographing and image analyzing are used for better cement plugs investigation. One of them is CT Scanning and another is Microscopic Photographing. Most of these tests are done under the High Pressure and Temperature (HPHT) condition. Also, some tests and experiments for foam stability and its behavior are conducted.

#### **1.3.** Thesis Outline

This thesis includes five chapters. In chapter one an introduction to this thesis is introduced. In chapter two, the background of cement and cementing, different cement jobs and operations, cement types and categories of additives, foam structure and applications, foam cement history and its benefits are explained. A literature review of previous works about foam cementing, foam cement formulations and different lightweight cement designing and additives is at the end of the second chapter. In the next chapter, a list of experimental instruments, procedures for conducting experimental tests and materials which were used, are presented. In the fourth chapter, the results of experimental tests for different foam cement designing and a vast investigation on the optimum formulation are presented and interpreted. Finally, in chapter five, the conclusions that were obtained from this research are discussed and recommendations for future works are presented.

#### **CHAPTER TWO**

#### **BACKGROUND AND LITERATURE REVIEW**

#### **2.1. Introduction**

In this chapter, the background of cement and its classes, different cement job and cementing technology, foam structure and properties, foam agents, foamed cement technology and etc. are explained. Moreover, the history and benefits of foam cementing in the petroleum industry and well cementing are discussed. At the end of this chapter, the projects and works of other engineers and researchers that were conducted already are presented. Particularly, the works on literature and projects focused on the foamed cement are described.

#### 2.2. Background

#### 2.2.1. Cement

Since Persian, Greece and Roman empires at 800 BCS, cement was used in their building. But the structure of those cement and their using technology are so different than today's cement and cementing job. Until today, this technology has changed a lot during the centuries all around the world. The basis of today's cement is Portland cement. Portland cement, the most common type of cement in general use around the world as a basic ingredient of concrete, mortar, stucco and non-specialty grout, was developed in England in the mid19th century, and usually originates from

limestone. James Frost produced what he called "British cement" in a similar manner around the same time but did not obtain a patent until 1822. In 1824, Joseph Aspdin patented a similar material, which he called Portland cement because the render made from it was in color similar to the prestigious Portland stone which was quarried on the Isle of Portland, Dorset, England. However, Aspdin's cement was nothing like modern Portland cement but was the first step in its development, called a proto-Portland cement. Joseph Aspdin's son William Aspdin had left his fathers company and in his cement manufacturing apparently accidentally produced calcium silicates in the 1840s, a middle step in the development of Portland cement. William Aspdin's innovation was counterintuitive for manufacturers of "artificial cement", because they required more lime in the mix (a problem for his father), a much higher kiln temperature (and therefore more fuel), and the resulting clinker was very hard and rapidly wore down the millstones, which were the only available grinding technology of the time. Manufacturing costs were therefore considerably higher, but the product set reasonably slowly and developed strength quickly, thus opening up a market for use in concrete. However, cementing technology has grown in tow branches of engineering: Civil Engineering and Petroleum Engineering. Each of American Concrete Institute (ACI), American Society for Testing Materials (ASTM) and American Petroleum Institute (API) developed standards for different cement and cementing job operations [4]. In this thesis, API standards are more focused than others.

A typical plugging material is classified as one that, when combined with a proper amount of water, without the need to use any additives, results in a slurry with cementitious properties. This can be a single ingredient or several combined, but the combination must always be the same, also when additives are used. The one, by far, most frequently used is Portland cement as described before. In 1952 the American Petroleum Institute (API) established a classification system for oilfield cements

because the conditions in which cements are used in the oil-industry differ significantly to those at ambient conditions in construction. Their most important property, therefore, is the one, which defines them as hydraulic cements, viz. not to build up compressive strength as a result of a drying process, but as a result of hydration, a reaction between the cement compounds and water. There are eight classes, named A to H, arranged by the depth and according to temperatures they are used at. All of the APIcements are Portland cement-based and consist principally of the same four ingredients, which are tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetra calcium aluminoferrite, only varying in fineness and mixing proportions. The variation results in different water requirement, hardening behavior and sealing performance. In some classes, cements can be categorized due to their sulphate resistance, which depends on the tricalcium aluminate content, and named: ordinary (O), moderate sulphate resistance (MSR) and high sulphate resistance (HSR). Class E, F, and G were developed for the use in deeper wells. They are known as "retarded cements". By reducing the faster-hydrating portions and using larger particles in these cements, the setting and hardening time are increased and longer pumping times allowed. Cements of these classes were manufactured first, so since that time cement additives have improved a lot and as a consequence, such common "retarded cements" are hardly used today. For special cases, there is a number of additives that are blended to the cement. Most frequently used today, are cements of classes G and H which are essentially identical but vary in coarseness. Class G cements are significantly finer than class H. This proves by different water requirements, going along with the different surfaces. The basis of foam cement designing in this research is class G cement [5]. Every company has its own preferences how or with which additives to adjust the cement properties to special circumstances. Additives will be discussed later. Table 2.1 details the API Cement Classification and their features.

Cement Classes	Properties
А	Intended for use from the surface to 6000-ft (1830-m) depth, when special properties are not required. Available only in ordinary type (similar to ASTM C150 Type I) (O type)
В	Intended for use from the surface to 6000-ft (1830-m) depth, when conditions require moderate to high sulfate resistance. Available in both moderate (similar to ASTM C150, Type II) and high sulfate resistant types. (HSR type)
С	Intended for use from the surface to 6000-ft (1830-m) depth, when conditions require high early strength. Available in ordinary and moderate (similar to ASTM C150, Type III) and high sulfate-resistant types. (MSR & HSR types)
D	Intended for use from 6000- to 10000-ft depth (1830- to 3050-m) depth, under conditions of moderately high temperatures and pressures. Available in both moderate and high sulfate resistant types. (MSR & HSR types)
Е	Intended for use from 10000 to 14000-ft (3050- 4270 m) depth, under conditions of high temperatures and pressures. Available in both moderate and high sulfate-resistant types. (MSR & HSR types)
F	Intended for use from 10000-to 16000-ft (3050-4880 m) depth, under conditions of extremely high temperatures and pressures. Available in both moderate and high sulfate resistant types. (MSR & HSR types)
G and H	Intended for use as a basic cement from the surface to 8000-ft (2400-m) depth as manufactured or can be used with accelerators and retarders to cover a wide range of well depths and temperatures. No additions other than calcium sulfate or water, or both, shall be interground or blended with the clinker during manufacture of Class G cement. Available in moderate and high sulfate-resistant types. (MSR & HSR types)
# 2.2.1.1. Chemistry of Cement

Portland cement is the most used cement in oil wells. It is also called; ordinary Portland cement (OPC) because of its manufacturing in the rotary kiln when ingredients are mixed in molten form at proper proportions. Some other types of cement are used in the wells when the condition of the well does not allow OPC. In OPC, compressive strength develops due to its hydration in which water and other compounds react with each other [2].

#### 2.2.1.2. Manufacturing of Portland Cement

This section highlights the information about cement manufacturing. Clinkers are the main ingredient in Portland cement manufacturing process. It is the material that is produced by a cement factory. It comprises calcium silicates, calcium aluminates, and calcium aluminoferrite. Gypsum, a form of calcium sulfate, is also added at the end to make the final product. To produce clinkers for Portland cement, calcareous and argillaceous materials are necessary. The calcareous material contains lime and can be obtained from limestones, shell deposits, corals, precipitated CaCO3 and industrial processes. Argillaceous materials are usually yielded from shales, clays, marls, mudstones, volcanic ashes, fly ashes, and blast furnace slag. These sources are necessary for silica, alumina and iron oxide production which is a vital part of OPC [4]. Table 2.2 shows the mineralogical composition of Portland cement.

Cement manufacturing can be summarized in five steps [3]:

# **2.2.1.2.1. Preparation of raw materials**

Raw materials are converted into finely powdered state and are blended efficiently. A stable chemical composition is essential for kiln feed. This process can is done in either dry or wet conditions. Both grinding and blending are done with the dry material in dry process whereas water-based slurry is used in the wet process.

Oxide Composition	Cement Notation	Common Name	Concentration (WT %)		
3CaO.SiO <sub>2</sub>	$C_3S$	Alite	55-65		
2CaO.SiO <sub>2</sub>	$C_2S$	Belite	15-25		
3CaO.Al2O <sub>3</sub>	C <sub>3</sub> A	Aluminate	8-14		
4CaO.Al <sub>2</sub> O <sub>3</sub> .Fe <sub>2</sub> O <sub>3</sub>	C <sub>4</sub> AF	Ferrite Phase	8-12		

Table 2.2. Mineralogical composition of Portland cement [6]

#### 2.2.1.2.2. Heat treatment

It is performed in the rotary kiln when the raw materials have already been passed through preheater. Solid material in the kiln slides down as the kiln is made inclined and rotates slowly at 1 to 4 RPM. Due to burning in the kiln, a large amount of greenhouse gasses gets produced which can be reduced by using alternates to fossil fuels or by improving kiln's fuel efficiency. Different reactions take place in a kiln which eventually results in clinkers production. Figure 2.1 presents the process.

### 2.2.1.2.3. Cooling

Early and long-term compressive strength of cement depends upon how the clinkers are cooled after heat treatment. Slow cooling rate result in cement which less hydraulically active having high early strength but weak in the long term. Too fast cooling gives opposite results. The optimum cooling process initially lowers the temperature to 1250 °C and then rapidly cools it at a rate of 18 to 20 °C/min.

# 2.2.1.2.4. Grinding

Clinkers are then grinded with gypsum (a form of calcium sulfate) which is used to increase the setting time of the cement. Lack or absence of gypsum results in a phenomenon of flash setting which occurs due to the formation of hydrocalumite. Too much hemi hydrated calcium sulfate results in the gypsum precipitation which further leads to the early setting of cement called false setting. Tubular mills are used to ground the clinkers which make use of hard steel balls.

Cross-Section View of Kiln	<b>Nodulization Process</b>	Clinkering Reactions
To 1,292°F [700°C]: Raw materials are free-flowing powder.	Particles are solid and do not react with each other.	Water is lost. Dehydrated clay recrystallizes. Clay particle Limestone particle
1,292–1,652°F (700–900°C): Powder is still free-flowing.	Particles are still solid.	As calcination continues, free lime increases. Reactive silica combines with CaO to begin forming C <sub>2</sub> S. Calcination maintains feed temperature at 1,562° F [850° C].
2,102–2,192°F [1,150–1,200°C]: Particles start to become "sticky."	Reactions begin between solid particles.	When calcination is complete, temperature increases rapidly. Small belite crystals form from the combination of silicates and CaO.
2,192–2,462°F [1,200–1,350°C]: As particles start to agglomerate, the liquid holds them together. The rotation of the kiln initiates coalescing of agglomerates and layering of particles.	The capillary forces of the liquid keep particles together.	Above 2,282°F [1,250°C], Round belite crystals a liquid phase is formed. Liquid allows reaction between belite and free Ca0 to form alite. Angular alite crystals
2,462–2,642°F [1,350–1,450°C]: Agglomeration and layering of particles continue.	Nodules will form with sufficient liquid. Insufficient liquid will result in dusty clinker.	Belite crystals decrease in amount but increase in size. Alite increases in size and amount.
Cooling	Clinker nodules remain unchanged.	Upon cooling, the C <sub>3</sub> A and C <sub>4</sub> AF crystallize in the liquid phase. Lamellar structure appears in belite crystals.

Figure 2.1. Production of Clinkers (Raw Feed to Finished Product) [3]

# 2.2.1.2.5. Storage

Quality of Portland cement can be maintained if stored in the dry environment. Moisture may induce certain properties which result in low strength after setting. Therefore, the humidity should be monitored and kept as low as possible in the warehouse.

## 2.2.2. Cementing Job Technology

There are two different main cement job process, Primary and Remedial cementing job. Each of them has some subsets. In this section, these processes will be introduced briefly [7]:

## 2.2.2.1. Primary Cementing Job

The main objective of primary cementing is placing cement slurry behind the casing. This can be performed in different methods: single stage, multi-stage, and inner string cementing. In a single stage, the cement is pumped into the casing, down to the shoe then up into the annulus. In some cases, when the casing string is long or the formation cannot support the hydrostatic pressure of a column of cement, a multi-stage can be applied. The procedure is cementing one part of the annulus, and then the second part is cemented by pumping the cement through a multi-stage tool from the casing to the annulus up to the surface or pre-determined depth.

#### 2.2.2.1.1. Single Stage Cementing Job

It is the most common cementing operation in the drilling process. After running the casing with all its accessories: shoe, float collar, and centralizers, and spacing out the casing string from the bottom, the cementing head is set at the top of the string. It has to be sure about the top and bottom plugs which are very important for a successful job. The casing is circulated at least for one string volume to clean it and to cool the bottom of the wellbore. The spacer is pumped and then the bottom plug (wiper plug) is dropped. The cement is pumped after dropping the bottom plug, and then the cement is followed by the top plug. When the bottom plug reaches its seat at the float collar, its diaphragm is ruptured (Pressure increase at the surface is an indication of plug landing) and the cement flows through the plug down to the casing shoe, up into the annulus. When the top plug reaches the float collar, it lands on the bottom plug and stops the displacement of the cement by the drilling fluid. The pumping rates should be slowed when the plugs reach the float collar. The casing has to be pressure tested when the top plug lands on the float collar. The pressure has to be bled off slowly to check the valves functioning in the float collar and the casing shoe. If there is any backflow, the volume has to be pumped again, increase the pressure and keep it till the cement hardens.

The photo of a cementing head can be seen in Figure 2.2. The simple procedure of single stage cementing job is presented in Figure 2.3.



Figure 2.2. Cementing Head [3]

## 2.2.2.1.2. Multi-Stage Cementing Job

This procedure of cementing is used when the pumping rate is long, high pump pressures or the hydrostatic pressure can exceed the fracture pressure of some troublesome formations. The operation is split into two stages, that is shown in Figure 2.4.



Figure 2.3. Single Stage Cementing Job [1]



Figure 2.4. Multi-Stage Cementing Job [1]

# 2.2.2.1.3. Inner String Cementing

Conventional cementing techniques are not suitable for large diameter casing due to many reasons:

- Large displacement volumes;
- Long pumping time;
- Plugs can stick in the casing due to their large dimensions.

Inner string cementing is performed by using the stinger which is run by the drill pipe to the casing shoe and stubbed in it. Once the stringer is engaged, drilling fluids are circulated to ensure that there is no leak which can allow fluids to flow from the drill pipe into the casing. The cement slurry is then pumped through the drill pipe, down to the casing shoe up to the surface through the annulus. The job is finished when getting cement on the surface into the cellar and then the stinger is disconnected and pulled above the shoe. Circulation through the drill pipe is necessary for such a situation to flush the drill pipe from any cement. In this type of cementing operations, no cement plugs are used. Figure below illustrates the inner string cementing job process.



Figure 2.5. Inner String Cementing Job [1]

# 2.2.2.2. Remedial (Secondary) Cementing Job

Secondary or remedial cementing consists of two broad categories: squeeze cementing and plug cementing.

# A) Squeeze Cementing Jobs

This type of jobs is based on forcing the cement slurry through the perforated casing into the annulus or the formation. Squeezing process is performed as a remedial operation and it is applied to:

- Maximizing oil production by sealing off gas and water production zones;
- Cementing parts of the annulus in case of primary cementing failure;
- Sealing off lost circulation;
- Preventing undesirable fluids migration into producing zones
- Repairing casing by forcing cement at leaking points.

The squeezing jobs are performed in two ways: high-pressure squeeze and low-pressure squeeze.

# **B)** Cement Plugs

Cement plugs are placed into the casing or the open hole for many purposes:

- Abandoning depleted zones;
- Sealing off lost circulation zones;
- Sidetracking or directional drilling;
- Abandoning the entire well.

There are two techniques to place cement plugs: dump bailer and balanced plug.

#### **2.2.3. Cement Properties**

This section summarizes the major properties of the cement slurry. Some of these properties affect the selection of cement type and for a specific cementing job.

#### 2.2.3.1. Density

Should be the same as mud to minimize the risk or blowouts or lost circulation. Density is a basic parameter which, affects the other parameters such as rheology, interface length, thickening time and etc. This parameter is very important and critical in lightweight cement designing like foam cement [5].

### 2.2.3.2. Rheology

To correctly design a primary cement job, the rheological properties of the cement slurry, spacer, and chemical wash must be known. These properties are required to:

- Determine fluid-flow properties;
- Predict friction-pressure drops;
- Identify slurry mixability and stability problems.

For cementing applications, the fluid rheology is calculated from the rheological parameters measured in the laboratory normally with a rotational viscometer (e.g., Fann 35 or Chan 35 viscometer). Depending on the fluid behavior, the rheological properties are expressed in terms of the Newtonian, Power Law, Bingham Plastic or Herschel Bulkley fluid model. Due to equipment and practical constraints, the accurate measurement of rheological parameters is usually limited to a maximum test temperature of 194° (90°C) [5].

#### 2.2.3.3. Free Water and Settling

When a slurry is static for a period of time prior to the set, water may separate from the slurry (free water), migrate upward, and accumulate either in pockets or at the top of the column. This separation indicates slurry instability which can create problems with thickening time, strength and other properties, and result in incomplete zonal isolation, particularly in a highly deviated wellbore. The free-water test is designed to measure the separation tendency in the laboratory, using a 250 mL graduated cylinder. The free water should be zero for liner cementing, deviated well cementing and the prevention of gas migration. For vertical wells with no gas-migration problems, the Client usually specifies a maximum allowable percentage (e.g., 1.0 %).

Another possible outcome of a slurry under static conditions for a period of time prior to the set is the suspended solids may tend to separate from the slurry and settle toward the bottom of the cement column. Such sedimentation indicates slurry instability and can produce a change in slurry density, leading to annular invasion and possible loss of well control. During rheology testing and calculations, settling tendencies are indicated by sediment left in test equipment and a negative yield-point value. Sedimentation must be especially prevented in highly deviated and horizontal wells, and wells where gas migration may be a problem. The larger and coarser particles in an unstable slurry settle at a faster rate than smaller particles. The finer particles at the top of the slurry column can remain in suspension a long time and thus the slurry may not exhibit true free water, but the slurry is unstable [5].

# 2.2.3.4. Thickening Time

Thickening-time tests are designed to determine the length of time during which a cement slurry remains in a pumpable, fluid state under simulated wellbore conditions of temperature and pressure. The wellbore conditions during primary cement jobs and squeeze cement jobs have appreciable differences, requiring individual discussions on the thickening time testing for each job type. Thickening time tests for plug jobs should be performed using the test method for running squeeze jobs [5].

## 2.2.3.5. Compressive Strength

Compressive strength measurements are designed to provide some indication of the ability of a set cement to provide zonal isolation and to protect and support the pipe. These measurements are also used as criteria for deciding when cement can be drilled out or perforated and the suitability of whip-stock plugs. The crush and UCA test methods are used to determine the compressive strength. For the crush test method, the compressive strength is calculated from the uniaxial force required to crush cubes that were cured under specific temperature and pressure conditions. The UCA test method continuously measures the sonic travel time of ultrasonic energy through a cement sample as it cures under simulated wellbore conditions of temperature and pressure. This sonic travel time is well correlated to compressive strength. The compressive-strength values obtained using either the crush test or the UCA are indicative of the cement integrity under uniaxial loading (no lateral constraint). In the wellbore, the cement is subject to complex triaxial loading, and the failure stresses may be substantially different from those observed in the standard compressivestrength test. Furthermore, the compressive-strength measurement provides no guide to the shear strength of the casing/cement or the casing/formation bond.

From experience, the industry has established minimum compressivestrength requirements for different cement applications which normally result in successful cementations. For primary cement jobs, testing is performed at temperatures and pressures simulating the bottom-hole conditions and the top-of-cement conditions for long cement columns. For

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plug jobs, the compressive strength at the top of the plug is the primary interest. For cement squeezes, the cement system is tested under conditions simulating the squeeze depth [5].

### 2.2.3.6. Fluid Loss

Slurries for primary cement jobs require fluid-loss control to:

- maintain cement slurry properties during slurry placement;
- prevent slurry dehydration which may cause bridging and excessive pump pressures, especially in narrow annuli across permeable formations;
- reduce filtrate loss to permeable formations which can accelerate the loss of hydrostatic head and permit well fluids to flow and invade the cement;
- minimize formation damage by cement filtrate.

For squeeze cementing, the desired cement placement and filter-cake growth (and job success) are achieved through correct fluid-loss control. Fluid-loss tests are static tests which provide a measurement of slurry dehydration. For a primary cement job, these tests do not represent the fluid loss during placement (termed dynamic fluid loss) [5].

#### **2.2.3.7.** Foam Stability

Even though this is not the main slurry test, however, it is very useful in foam cement designing. The foam stability of a foamed slurry must be tested to ensure that the gas will not break out of the slurry. If the gas was to coalesce, then the increasing bubble size would cause it to rise to the surface or form gas pockets in the cement [8].

#### 2.2.3.8. Permeability

The permeability of the cement sheath is a vital parameter with regard to zonal isolation. This is especially true with low-permeability producing zones that are separated by a thin barrier. According to the literature and the WELL CEMENTING textbook states that well cements exhibiting waterpermeability less than 0.1 mD will prevent interzonal communication. A water permeability of 0.1 mD approximately equals a gas permeability of 1.0 mD. Many gases producing formations have a gas permeability of less than 1.0 mD; therefore, this maximum permeability limit should be interpreted as too high. Most set cements have permeabilities much lower than most producing formations. At temperatures, less than 200°F (93°C), the permeability of cement decreases with age and temperature. After a 7-day curing period, the permeability is usually too low to measure [9].

## 2.2.3.9. Other Tests

In sometimes for better analyzing of hardened cement, the porosity of foamed cement plugs is measured. Microscopic photographing and CT scanning can help for best-hardened cement structural designing.

### 2.2.4. Cement Additives

In well cementing, Portland cement systems are designed for temperatures ranges from below freezing in permafrost zones to 662°F (350°C) in thermal recovery and geothermal wells. They also encounter pressures ranging from ambient to 30000 psi (200 MPa) in deep wells. Accommodation of such variations in conditions was only possible through the development of cement additives. Typical chemical additives for oil and gas well-cementing operations include; accelerator, retarders, extenders,

fluid loss and loss circulation additives, dispersants, and many more are used for specific regions and properties.

### 2.2.4.1. Accelerators

Accelerator is a chemical additive used to speed up the normal rate of reaction between cement and water which shortens the thickening time of the cement, increase the early strength of cement, and saves expensive rig time. Cement slurries used in shallow wells where temperatures are low requires accelerators to shorten the time for Waiting-on-Cement (WOC) before drilling operation can be resumed. In deeper wells, the higher temperatures promote the setting process, and accelerators may not be necessary. Calcium Chloride and Sodium Chloride are the most commonly used. However, Calcium Chloride (CaCl<sub>2</sub>) is undoubtedly the most efficient and economical accelerator. Other types of accelerators include sodium metasilicate, potassium chloride, and gypsum.

Calcium Chloride is generally added in concentrations of 2% - 4% by weight of cement (BWOC), but over 6% BWOC its performance becomes unpredictable and premature setting may occur. Calcium Chloride is effective at temperatures between 40°F and 120°F (4°C and 49°C) in concentrations of 2% to 4% by weight of cement (BWOC), or equivalent liquid concentrations of 40 gal/100 Sx (2%). These results are summarized in Table 2.3. Salt (NaCl) is a slight accelerator at low concentrations and becomes a retarder at high concentrations. Figure 2.6 shows the effect of NaCl on the thickening time and compressive strength better. Maximum acceleration occurs at a concentration of about 5-15 % by weight of mixing water for cements containing no bentonite. At concentrations above 15%, the effectiveness of sodium chloride as an accelerator is reduced. Saturated sodium chloride solutions tend to act as a retarder rather than an accelerator. Seawater is extensively used offshore as it has 25 g/L NaCl but the concentration of magnesium of about 1.5 g/L must be considered. This thickening time obtained with seawater usually is adequate for cement placement where bottom-hole temperatures do not exceed 160°F [3].

CaCl <sub>2</sub> (BWOC%)	91°F	103°F	113°F			
	Thickening Time					
0	4:00	3:30	2:23			
2	1:17	1:11	1:01			
4	1:15	1:10	0:59			

Table 2.3. Calcium Chloride Thickening Time on Portland cement



Figure 2.6. Effect of sodium chloride on thickening time and compressive strength development [3]

## 2.2.4.2. Retarders

Retarders are chemical additives used to decrease the speed of cement hydration. The cements commonly used in well applications do not have a sufficiently long fluid life (thickening time) for use at Bottom Hole Circulating Temperatures (BHCTs) above 100°F (38°C) is explained. the retardation process is not completely understood but it is known that retarders bind to calcium ions and are able to inhibit the growth of ettringite crystals. Besides extending the pumping time of cements, most retarders affect the viscosity to some degree. Retarders do not decrease the ultimate compressive strength of cement but do slow the rate of strength development. Retarding effects of a retarder depends upon a number of factors including a dosage of the additives, curing conditions among others. Therefore, BHCT should be carefully predicted so that the correct retarder concentration is used to avoid flash setting or very long set up time due to the over-retarded cement slurry. The most common retarders are natural lignosulfonates and sugars derivatives explained the cellulose derivatives chemical nature of the retarder to be used is dependent on the cement phase (silicate or aluminate). Lignosulfonates and hydroxyl carboxylic acids are retarders that are believed to perform well for oil well cements with low C<sub>3</sub>A contents. Lignosulfonates are normally used at circulating temperatures up to 200°F. The commonly used hydroxyl carboxylic acids are normally derived from naturally occurring sugars. Sugar compounds are normally used at circulating temperatures from 200°F to 300°F.

A lignosulfonate is a metallic sulfonate salt derived from the lignin recovered from processing wood waste. The resultant aqueous solution of lignosulfonic-acid, simple sugars, starches, and natural gums are known as lignin liquor. The composition of the liquor is dependent on the wood source and the reaction conditions. Performance of the liquor as a retarder is dependent upon the proportioning of these compounds, their molecular weights, and the degree of sulfonation. Its chemical structure is illustrated in Figure 2.7. As a result of processing, three grades of lignosulfonate are available for the retardation of cement slurries. Each grade is available as calcium/sodium or sodium salts. The most common retarder among the three types may be calcium lignosulfonate. Its effectiveness is limited to temperatures above 200°F. Concentrations of 0.1% BWOC - 1.0% BWOC are used in most slurry applications to give both predictable thickening times and compressive strengths [3]. The typical effect of a lignosulfonate on class G cement is presented in Figure 2.8.



Figure 2.7. Basic lignosulfonate chemical structure [3]



Figure 2.8. The typical effect of a lignosulfonate on class G cement [3]

Hydroxyethyl cellulose (HEC) and carboxymethyl hydroxyethyl cellulose (CMHEC) are the two cellulose polymers used in well-cementing applications. Traditionally, the only cellulose that is considered as a retarder is CMHEC. This is large because it is functional as a retarder up to approximately 230°F (110°C) BHCT at the same concentrations as calcium lignosulfonate, but it also provides good fluid-loss control. The hydroxyl carboxylic acids are well known for their antioxidant and sequestering properties that benefit cement-slurry performance as a retarder.

### 2.2.4.3. Extenders

Many formations will not support long cement columns of highdensity slurries, these slurries weights need to be reduced to protect formations that have low fracture gradient or for economic purposes. To reduce the weight of cement slurries, extenders are used. Extenders are also known as water adsorbing or lightweight inert materials. A reduction of slurry density reduces the hydrostatic pressure during cementing of weak and fragile formations or depleted reservoirs. They also reduce the amount of cement needed for cementing operation and because they are less expensive than cement, they bring considerable savings. Extenders work by allowing the addition of more water to the slurry to lighten the mixture and to keep the solids from separating. These additives change the thickening times, compressive strengths and water loss. In reducing slurry density, the ultimate compressive strength is reduced and the thickening time is increased. For example, extenders such as bentonite, foamed cement, and microspheres decrease the cement slurry density and final compressive strength at 100°F (38°C) after a 24-hour curing period.

Three types of extender are normally used: water extender, gas, and low-density aggregate. The challenge of water extender is that the slurry will become too thin and the cement will settle and have free water. The disadvantage of adding the extra water is that the strength of the set cement is lessened by the dilution. If low density and higher strength are required, the density of the slurry can be reduced with gas extender. Stable foam cement will have discrete air bubbles that lower the density of the slurry but do not dilute the strength as much as water. The density of the cement slurry is reduced when significant quantities of such extenders are present. It can be obtained from volcanic ash, diatomaceous earth and fly ash. Bentonite is by far the most common type of additive used to lower cement slurry density. Bentonite concentrations of 2 to 16% BWOC have been used in cement design. Bentonite effect on the CS is presented in Figure 2.9. It is able to hold water which is 16 times its volume and it therefore also ensures no free water evolves during cement set up. Also, the higher water content lowers the resistance to sulphate attack and increases the permeability of the set cement. Table 2.4 compares different extenders effects range and their benefits [3].



Figure 2.9. Effect of bentonite on compressive strength [3]

Table 2.4. Summary of Extenders [3]

Extender	Range of Slurry Slurry Densities Obtainable (Ibm/gal)	Performance Features and Other Benefits		
	<mark>6 11 1</mark> 6			
Bentonite	11.515	Assists fluid-loss control		
Fly ashes	13.1 <mark>□</mark> 14.1	Resist corrosive fluids		
Sodium silicates	11.114.5	Available in solid or liquid form; effective at low concentrations; ideal when mixing slurry with seawater		
Microspheres	8.5 15	Good compressive strength, low permeability, thermal stability, and insulating properties		
Foamed cement	615	Good compressive strength and low permeability		

# 2.2.4.4. Heavy Weight Agents

The main purpose of heavyweight additives is to restrain high formation pressures. The main requirements for heavyweight agent (weighting agents) are that they have a specific gravity greater than the cement, consistent particle size distribution and low water requirement. Chemically weighting agents are inert in the cement slurry and do not interfere with logging tools. The most obvious way of increasing cement density is to reduce the amount of water in the cement slurry. However, slurries with densities greater than 17.5 ppg would be too thick to mix and pump without weighting agents. This would, therefore, require dispersants to maintain pumpability. The most common weighting agents are Ilmenite, Hematite, and Barite. Other weighting agents are a heavy particulate material such as salt, Ottawa sand or titanium oxide.

Hematite (Fe<sub>2</sub>O<sub>3</sub>), Iron Oxide, is the most commonly used heavy weighting additive. The specific gravity of hematite ranges from 4.9 to 5.3, depending on purity. It can be used to overcome many shortcomings of barite. The high specific gravity of hematite can be used to raise slurry densities to 22 ppg. Ilmenite (FeO.TiO<sub>2</sub>), Iron Titanium Oxide, is not as commonly used as hematite, although it has some advantages over hematite. The specific gravity ranges from 4.5 to 5. Although ilmenite has a slightly lower specific gravity than hematite, it requires no additional water and provides about the same slurry density increase as hematite at comparable concentrations. Like hematite, ilmenite has little effect on thickening time or compressive strength. Barite (BaSO<sub>4</sub>), Barium Sulphate, is not normally used in cementing as a weighting agent because of its high surface area and high-water demand. The specific gravity ranges from approximately 4.0 to 4.5. Barite can be used to attain slurry densities of up to 18 ppg. It also causes a reduction in strength and pumpability [3]. Densification of cement slurries with various weighting agents is shown in the next page Figure.



Figure 2.10. Densification of cement slurries with various weighting agents [3]

# 2.2.4.5. Fluid Loss Additives (FLA)

Fluid loss additive is also known as permeability plugging additive. Fluid loss additives are commonly employed in field cementing operations reduce the rate at which water from cement is forced into permeable formations when a positive differential pressure exits into the permeable formation. That is, it prevents dehydration of cement slurry. As the liquid phase of the cement passes into the formation, the filter cake is formed on the formation face. Fluid loss additives function primarily by promoting the deposition of a low permeability filter cake, thereby limiting the rate of filtrate loss to permeable strata. Fluid loss additives are normally polymers such as cellulose, polyvinyl alcohol, polyalkanolamines, polymers of polyacrylamides, and liquid latex such as styrene butadiene latex. Most fluid loss additives increase the slurry viscosity, although some retard it to some degree. According to cellulose derivatives are the most common fluid loss additives and normal concentration vary from 0.3 to 3.0% by weight of the cement. The most common organic FLA are Organic Polymers (Cellulose), Carboxymethyl Hydroxyethyl Cellulose (CMHEC) and Hydroxyethyl Cellulose (HEC) [3]. HEC effect on the fluid loss rate of class A cement is presented in the Figure below.



Figure 2.11. Typical fluid-loss performance of HEC in normal-density [3]

#### **2.2.4.6.** Lost Circulation Additives

Lost circulation additive is also known as macro plugging materials. Lost-circulation additives are used to plug zones that tend to draw in the fluid because they are unconsolidated or weak. Large particulates can be placed in the cement slurry to prevent fracturing or to bridge existing fractures. These particles should have a broad particle size distribution, should not accelerate or retard excessively, should have sufficient strength to keep a fracture bridged, and should be inexpensive and non-toxic. Organic Lost Circulation Materials (LCM), traditionally utilized in drilling fluid formulations, should not be used in the cement slurry. Although they achieve the objective of sealing the permeable zones, after the well has been completed, the organic material is carbonized, leaving high porosity within the loss zones, thus providing a flow path for possibly corrosive formation fluids. The most common materials are ground coal, ground gilsonite, and ground walnut hull. Gilsonite is a naturally occurring, solid carbonaceous material that is classified as an asphaltite. It is a relatively pure hydrocarbon without significant amounts of mineral impurities [3].

### 2.2.4.7. Dispersants

Dispersants also are known as friction reducing additives are added to improve upon the flow properties of the cement slurry. In particular, they are used to offset overly-high viscosity and some slurries tendency to gel. Dispersants assist in providing fluid loss control for high-density slurries. They also help establish turbulent flow at low pumping rate when needed. Dispersant allows the water content of the cement to be lowered without making it difficult to pump. At right concentration, dispersants improve cement homogeneity and lower its permeability. However, an overdose of dispersant can produce phase separation in the cement slurry that results in cement particles settling out of solution and the development of free fluid. The most common dispersant is the sodium salt of Polynapthalene Sulphonate (PNS). It is available as a calcium and/or sodium salt, and can be obtained in both solid and liquid form. PNS can also allow higher solidsto-water ratio slurries to be designed with improved properties. PNS materials are polymeric with molecular weights ranging between about 3000 and 20000 [3].

#### 2.2.4.8. Expansion Additives

Expansion additives cause the exterior dimensions of set cement to grow slowly when the cement is in the presence of down-hole fluids. This minor growth of the exterior dimensions of the slurry causes the cement to bond better to pipe and formation. The most common additives for this use are based on calcium sulphoaluminate and calcium oxide. CaO and MgO are two of the most effective additives to create excellent expanding cement [10].

# 2.2.5. Foam and Its Structure

A foam is a colloidal dispersion in which a gas is dispersed in a continuous liquid phase. The dispersed phase is sometimes referred to as the internal (disperse) phase and the continuous phase as the external phase. Despite the fact that the bubbles in persistent foams are polyhedral and not spherical, it is nevertheless conventional to refer to the "diameters" of gas bubbles in foams as if they were spherical. In practical occurrences of foams, the bubble sizes usually exceed the classical shape limit given above, as may the thin liquid film thicknesses. In fact, foam bubbles usually have diameters greater than 10 mm and may be larger than 1000 mm. Foam stability is not necessarily a function of drop size, although there may be an optimum size for an individual foam type. It is common but almost always inappropriate to characterize a foam in terms of a given bubble size since there is inevitably a size distribution. This is usually represented by a histogram of sizes, or, if there are sufficient data, a distribution function [11].

A two-dimensional slice of a general foam system is depicted in Figure 2.12. The general foam structure is contained by the bulk liquid on the bottom and on the upper side by a second bulk phase, in this case, gas. In a persistent foam the bubbles are not spherical but have become transformed into foam cells, polyhedra separated by almost flat liquid films. Such foams are referred to as dry foams or Polyederschaum. The polyhedra are almost, but not quite, regular dodecahedra. Within the magnified region of Figure 2.12, the various parts of the foam structure are clarified. The gas phase is separated from the thin liquid film, by a two-dimensional interface. In reality is not a sharp dividing surface between the gas and liquid properties. Dictated by mathematical convenience, the physical behavior of this interfacial region is approximated by a two-dimensional surface phase (the Gibbs surface). For the purposes of this thesis, a lamella is defined as the region which encompasses the thin film, the two interfaces on either side of the thin film, and part of the junction to other lamellae [11].



Figure 2.12. Illustration of a generalized foam system showing aspects of a foam lamella [11]

The arrangement of films coming together at equal angles of 120° (called the Steiner angle) results from the equalization of the surface tension vectors, or contracting forces, along with the liquid films. The bubbles in a foam arrange themselves into polyhedral such that, along with the border of a lamella, three lamellae always come together at angles of 120°, the border where they meet is termed a Plateau border. In three dimensions, four lamellae meet at a point at the tetrahedral angle, approximately 109°. Observations of dynamic foams show that whenever more films happen to come together, a rearrangement immediately takes place to restore junctions of only three films at Plateau borders in two dimensions, four lamellae in three dimensions. The three-dimensional foam bubbles are spherical in wet foams (i.e., those with gas volume fractions of up to  $\varphi = 0.74$ , the maximum volume fraction possible for an internal phase made up of uniform, incompressible spheres), but the foam bubbles start to distort in drier foams, in the range 0.74  $< \phi < 0.83$  (approximately). In still drier foams the foam cells take on a variety of polyhedral shapes ( $\varphi > 0.83$ , approximately). There are many polyhedrons that can exist in foams and froths. One of the

model shapes that may be the most stable, in terms of minimizing surface free energy, is the Kelvin tetrakaidecahedron, which has eight non-planar hexagon faces and six planar quadrilateral faces. It should be remembered, however, that most foams contain a distribution of shapes (and sizes) [12].

#### **2.2.6. Foams Application**

Foams are commonplace in foods, shaving cream, fire-fighting foam, mineral flotation, and detergents. Thus, in view of the fact that the concentration of bubbles greatly affects the properties of foams; the production, dispersion, and maintenance of colloidal gas bubbles are crucial to foams and related materials. Often, natural and synthetic soaps and surfactants are used to make fluid foams containing colloidal gas bubbles. These agents reduce the interfacial tension and, perhaps, the viscosity at the gas/liquid interface, making the foam stable. Also, some soluble proteins that denature upon adsorption or with agitation of the liquid phase can stabilize foams by forming insoluble, rigid layers at the gas/liquid interface. Foams may also be applied or encountered at all stages in the petroleum recovery and processing industry (oil well drilling, well cementing, reservoir injection, oil well production, and process-plant foams). A class of enhanced oil recovery process involves injecting a gas in the form of a foam. Suitable foams can be formulated for injection with air/nitrogen, natural gas, carbon dioxide, or steam. Micro-foams (also termed colloidal gas aphrons) comprise a dispersion of aggregates of very small foam bubbles in aqueous solution. They can be created by dispersing gas into surfactant solution under conditions of very high shear. The concept is that, under the right conditions of turbulent wave break-up, one can create a dispersion of very small gas bubbles, each surrounded by a bi-molecular film of stabilizing surfactant molecules. Under ambient conditions, the bubble diameters are typically in the range 50-300 mm. There is some evidence that such micro-foams tend to be more stable than comparable

foams that do not contain the bi-molecular film structure [13]. Other foam occurrences are pinpointed in the Table 2.5.

Field	Foam			
Environment and meteorology	Polluted river foams, bubbles of air in oceans and rivers			
Foods	Soda heads, whipped cream, meringue, ice cream			
Geology, agriculture, and soil science	Foam fumigant, insecticide and herbicide blankets, bubbles of volatiles in magma			
Manufacturing and materials science	Foam fractionation, pulping brown stock foam, detergent foam, flotation de-inking froth			
Biology and medicine	Vacuoles, insect excretions, contraceptive foam, gastrointestinal foam			
Petroleum production and mineral processing	Refinery foams, oil and bitumen flotation froth, fire-extinguishing foam, explosion suppressant foam, mineral flotation froths			
Home and personal care products	Shampoo suds, shaving cream, contraceptive foam, bubble bath foam, hairstyling mousse			

Table 2.5. Some of the foam occurrences [11,13]

In the next section, foam cement is described. Foamed cementing technology and its benefits are focused on this thesis.

# 2.2.7. Foamed Cement

Foamed cements are coarse dispersions of a base cement slurry, a gas (usually air or nitrogen), a foaming surfactant, and other materials to provide foam stability. The base cement slurry is usually a conventional 15–16 ppg system. The density of the gas is, for all practical purposes, 0 ppg. Therefore, the foam density is adjusted by varying the air or nitrogen concentration. Although foamed cement was first used by the construction industry more than 60 years ago, its first application in well cementing occurred in 1979. Foamed cementing technology has been evolving ever since. Foamed cements are generally less expensive than systems containing glass microspheres or cenospheres. Foamed cement can be mixed at lower densities than conventional microsphere systems and yet maintain acceptable properties. Foamed cement has densities as low as 5 ppg in situations in which compressive strength and permeability were not critical [3].

Virtually any oil well cementing job can be considered a candidate for foamed cementing, including primary and remedial cementing functions onshore and offshore, and in vertical or horizontal wells. Although its design and execution can be more complex than standard jobs, foamed cement has many advantages that can overcome these concerns, because it:

- Is lightweight;
- Provides excellent strength-to-density ratio;
- Is ductile;
- Enhances mud removal;
- Expands;
- Helps prevent gas migration;
- Improves zonal isolation;
- Imparts fluid-loss control;
- Is applicable for squeezing and plugging;
- Insulates;
- Stabilizes at high temperatures;
- Is compatible with non-Portland cements;
- Simplifies admix logistics;

- Enhances volume;
- Has low permeability;
- Is stable to crossflows;
- Forms a synergistic effect with some additives, which enhances the property of the additive [14].

The low density of foamed cements reduces losses to potential producing zones, and increased well productivity may result. More recent applications of foamed cements include controlling shallow flows below the mudline in deep water wells, deterring compaction damage in soft formations, and resisting damage from external stresses placed on the cement sheath. The stability of foamed cement is affected by the foaming agent, the quantity of gas, the chemical and physical composition of the slurry, thermodynamic factors, and the mixing method and conditions. Stable foams exhibit spherical, discrete, disconnected pore structures with a clearly defined cement matrix. Unstable foams have non-spherical and interconnected pores, caused by the rupture and coalescence of gas bubbles. Such unstable foams have a sponge-like structure and develop lower compressive strength, higher permeability, and inferior bonding properties.

Foams are categorized by their *quality* ( $Q_{foam}$ ), or the ratio of the volume occupied by the gas to the total volume of the foam [11]. (expressed as a percentage)

$$Q_{foam} = \frac{V_{gas}}{V_{foam}} \times 100$$
 EQ. 2.1

As the foam quality varies, two structural situations occur. Concentrated foams are mostly gas phase and consist of polyhedral gas cells separated by thin liquid films. Dilute foams consist of nearly spherical bubbles separated by thick liquid films. Foamed cement belongs to the second category, with a quality not exceeding 80% and usually less than 50%. Foamed cement is a three-phase system (gas/liquid/solid), with many phenomena occurring at the interfaces. This system is in constant evolution because of the reorganization of gas bubbles that may grow, shrink, or coalesce, and because of the chemical reactions that occur in the base cement slurry. Foams are difficult to characterize because they are shear history-dependent fluids, and their texture is strongly affected by the mixing procedure. Foamed cements made under largescale field conditions, with high shear rates and high pressure, have been found to be more stable than foamed cements made under laboratory conditions [3].

The most common method to prepare foamed cement at the wellsite is to mix a base cement slurry with all the additives except the surfactants and then mix surfactants in high shear with slurry or inject the surfactants and the gas as the slurry is being pumped downhole.

A stable base slurry is a prerequisite for a stable foamed cement system. The same mechanisms that produce free fluid or solids segregation will contribute to the destabilization of foamed cements. The selection of the base slurry density depends on the required set-foamed-cement properties. Normal-density base slurries will lead to higher compressive strengths. However, such slurries require larger volumes of gas to achieve a given foam density; therefore, the resulting permeabilities will be higher. Conversely, less dense base slurries (prepared using lightweight filler materials) require less gas and will produce foamed cements with lower permeabilities; however, the compressive strengths will be lower. To select suitable foamers and stabilizers for cement, one should consider the following criteria [15]:

- safety and handling considerations;
- compatibility;
- effect on the cement strength and permeability;
- stability;
- efficiency;

• cost.

The duration of foam stability must be longer than the setting time of the base slurry. In addition to the base slurry, foaming agent, stabilizers, and additives, one must use a gas that is inert (air or nitrogen) with respect to the cement properties. Laboratory testing of foamed cement under simulated downhole conditions is difficult. Because of the pressure and temperature dependence (spatially temperature, the effect of temperature on foams properties is more than pressure effect) of the foam volume, curing a foamed cement at high pressure and temperature requires different equipment than that used for conventional slurries [16].

When the density of conventional cement slurries is reduced by adding water or other extenders, the amount of cementitious material is diluted. Because of the large density difference between water and gas, much less gas is required to reduce the density by an equal amount. Using gas results in less dilution and, therefore, less impact on cement properties. Consequently, the physical properties of foamed cements are similar to those of conventional lightweight cements that are 2 to 4 ppg heavier. Introducing a gas to a liquid medium significantly reduces the rate at which the liquid will flow through porous media. Foamed cements, with and without fluid-loss additives, have the lower fluid-loss rates and this is the result of gas phase presenting. No routine rheological measurement is made on foamed cement slurries. Therefore, different rheology measurements are defined such as Atmospheric, High Temperature, High-Temperature High Pressure and etc. Instead of testing foamed systems, a common procedure is to measure the thickening time of the base slurry containing the additives, surfactants, and stabilizers. This method gives a reasonable estimate of the working time for the foamed slurry. Thus, there are not so many differences between conventional and foam cement thickening time procedures [3].

## 2.3. Literature Review

In this section, different research works and projects on foam cement and foam cementing technology are described. These researches are introduced from the past to the present, therefore, researchers and engineers' innovations, formulations and designing on the foam cementing technology are investigated.

McElfresh et al. (1982) introduced some of foam cement applications such as placing strong cement across weak or highly fractured zones without losing circulation; filling vugs or porous thief zones; or simply as an inexpensive light-weight filler slurry. They found that thickening times could be determined directly on the foam cement in existing equipment with only minor modifications; the fluid loss and the rheology could be measured directly with no modifications. They used a nitrogen injection system to prepare their foam cement samples. Pressurized chambers are used for CS measurement. All tests are done under bottom well condition. They mentioned that physical properties of the foam cement are adequate to serve the needs of well cementing and, because of this, foam cementing is becoming a viable and accepted alternative in the cementing area [17].

Harms and Febus (1985) suggested that usually, the use of foamed cement offers a low-density cementitious material that develops adequate compressive strength while avoiding fallback problems that are caused by density. After hardening, foamed cement has reduced density, and it usually provides the advantages of temperature stability and heat insulation properties. In their paper, the properties of foam cements are discussed and more than 60 cementing jobs completed with foam cement are summarized. They reported several Cement Bond Log (CBL) and determined different foam cement properties in various condition [18]. Figure 2.13 shows the effect of foam cement density on its CS at constant 90°F temperature, which achieved by Harms and Febus.



Figure 2.13. 90°F compressive strength development of foam cement [18]

New invention and idea for foam/cement mixer are developed by Gray and Masters (2000). In their patent, they mentioned that a foam of very small, microscopic bubbles of surfactant, each bubble containing a small quantity of water, is mixed with a water/cement mixture having a water to cement ratio of less than 0.33. The small bubbles have a ball bearing effect on the smaller cement, increasing plasticity or flowability and thus reducing the water requirement of the mixture. The foamed cement mixture is laid to form the desired concrete structure, and the bubbles disintegrate or transform to leave voids of similar size uniformly dispersed throughout the concrete structure [19].

Narayanan and Ramamurthy (2000) classified the investigations on the properties of aerated concrete in terms of physical (microstructure, density), chemical, mechanical (compressive and tensile strengths, modulus of elasticity, drying shrinkage) and functional (thermal insulation, moisture transport, durability, fire resistance, and acoustic insulation) characteristics. Their comprehensive investigation is more focused on the foam concretes properties in civil engineering [20].

Kearsley and Wainwright (2001) studied the effects, on the properties of foamed concrete, of replacing large volumes of cement (up to 75% by weight) with both classified and unclassified fly ash. Their paper reports only on the results of the compressive strength of concretes cured under sealed conditions and shows that up to 67% of the cement could be replaced without any significant reductions in strength. There appears to be little difference in the performance of the ungraded and the graded fly ashes used in this investigation. Equations based on effective water/cement (w/c) ratio have been developed to predict the strengths up to 1 year, of foamed concretes made with densities ranging from 1000 to 1500kg/m<sup>3</sup>. The calculated results compare well with the experimental results [21].

Griffith et al. (2004) presented and discussed the engineering analysis to determine the optimum foamed cement sheath properties for integrity during the life of the well at HPHT conditions. Also, their paper compares foamed cement to non-foamed slurries in achieving these objectives. Other important issues discussed are the performance of air or nitrogen at HPHT conditions and the rheological properties of the foams. Key issues addressed are the state and solubility of nitrogen or air under downhole conditions and the integrity of the cement sheath during the life of the well. Thermodynamic solution theory and experimental studies are applied to the former, and finite element analysis is applied to the later. Case examples are presented discussing foamed and conventional operations at near HPHT conditions for some Norwegian, North Sea wells [22]. Cement properties are contrasted for the different foamed and conventional cement slurry properties with respect to:

- Achieving the HPHT objectives, such as placement; efficiency and sheath properties
- Pre-job design to obtain the objectives;
- Job planning and procedures;
- Job execution;
- Logging of foamed cement;

• Post-job evaluation of the cement systems.

They understood and achieved by this research that:

- Gas (Nitrogen or Air) solubility plays a minor role in downhole foamed-cement quality, and is difficult to accurately estimate;
- Over-treating the foamed cement with excessive air and N<sub>2</sub> injection causes a greater error in downhole quality than the equivalent erroneous undertreatment;
- Foamed cement is likely to provide better displacement efficiency at borderline conditions than a conventional system if properly designed;
- Foamed cement, if properly designed, is likely to provide better long-term zonal isolation at extreme conditions than conventional cement systems through improved mechanical properties [22].

Ahmed et al. (2009) investigated the results of an experimental study of foamed cement rheology. Viscosity curves of foamed cements were obtained using a flow-through rotational viscometer. Foamed cements with different foam qualities were generated under different pressures using a foam generator/viscometer apparatus. The foam qualities during the tests ranged from 0% to 30%, and the shear rate varied between 5 s<sup>-1</sup> and 600 s<sup>-1</sup>. Experimental results indicate that: 1) unlike conventional aqueous foams, low-quality cement foams have a lower viscosity than the base fluid; 2) as the cement foam quality (gas volumetric fraction) increases from 10% to 30%, the viscosity also increases; and 3) the viscosity of low-quality cement foam slightly increases after depressurization or expansion [23]. They could analyze their results by a simple VG meter or viscometer, however, they designed a foam generator/viscometer for accurate measurement as a schematic in Figure 2.14. They used the Herschel Bulkley model and results for different foam cement systems are reported in Table 2.6.



Figure 2.14. Schematic diagram of foam generator/viscometer [23]

Table 2.6. Rheology parameters for different tests on various foamed slurries [23]

Parameter	Base fluid	Foam 1	Foam 2	Foam 3a	Foam 4	Foam 5	Foam 6	Foam 7	Foam 8	Foam 3a*	Foam 3b <sup>#</sup>	Foam 3c*
τ <sub>γ</sub> [Pa]	0.00	5.50	3.20	3.40	5.00	2.60	8.30	0.90	2.70	4.79	3.36	4.95
m	0.59	0.66	0.79	0.86	0.69	0.75	0.76	0.60	0.57	0.86	0.89	1.02
k [Pas <sup>m</sup> ]	3.48	1.26	0.49	0.38	1.34	0.93	0.95	2.73	3.81	0.78	0.35	0.18

The Herschel Bulkley model equation is written below:

$$T_y = T_0 + k\gamma^m$$
 EQ. 2.2. Herschel Bulkley fluid model equation [23]

Ramamurthy et al. (2009) studied the properties of foam concrete. They highlighted that based on the review, the following research needs have been identified: 1) developing affordable foaming agent and foam generator, 2) investigation on compatibility between foaming agent and chemical admixtures, use of lightweight coarse aggregate and reinforcement including fibers, 3) durability studies, and 4) factors influencing foam concrete production viz., mixing, transporting and pumping. Most of the investigations on foamed concrete have been confined to the evaluation of
its properties rather than on the foam characteristics, which has bearing on the strength of the foamed material. Foam stability in concrete is one of the important aspects to ensure the fine and uniform texture throughout the whole hardening process. Though for the given density and strength requirement, many proportioning methods and guidelines have been proposed, as such there is no standard mix proportioning method available for foam concrete [24].

Just and Middendorf (2009) revealed that foam concretes are divided into two groups: on the one hand the physically foamed concrete is mixed in fast rotating pug mill mixers by using foaming agents. This concrete cures under atmospheric conditions. On the other hand, the autoclaved aerated concrete is chemically foamed by adding aluminum powder. Afterward it is cured in a saturated steam atmosphere. Their innovation by adding aluminum powder for increasing the CS is used in civil engineering and building structures. These foam concretes are new and innovative building materials with interesting properties: low mass density and high strength. Responsible for these properties are the macro-, meso- and microporosity. Macropores are created by adding aluminium powder in different volumes and with different particle size distributions. However, the microstructure of the cement matrix is affected by meso- and micropores. In addition, the matrix of the hardened cement paste can be optimized by the specific use of chemical additives for concrete. The influence of aluminium powder and chemical additives on the properties of the microstructure of the hardened cement matrices were investigated by using petrographic microscopy as well as scanning electron microscopy and CT scanning [25]. Some of their sample photographing are shown in the Figure 2.15.



Figure 2.15. Foam concrete imaging (SEM and CT Scan) [25]

Crandall et al. (2014) have produced the first high-resolution X-ray computed tomography (CT) three-dimensional images of atmospheric and field generated foamed cement across a range of foam qualities. CT imaging enabled the assessment and quantification of the foamed cement structure, quality, and bubble size distribution in order to provide a better understanding of this cement. Ultimately, this research will provide industry the knowledge to ensure the long-term well integrity and safe operation of wells in which foamed cements are used. Initial results showed that a systematic technique for isolating air voids can give consistent results from the image data, laboratory-generated foamed cements tend to be uniform, and that high-gas fraction foamed cements have large interconnected void spaces. These images analyzing are done because of the manufactured slurry solidifies in the sub-surface environment the distribution of gas voids can affect the resultant strength, permeability, and stability of the wellbore casing [26]. Some of their computed tomography scanning from different foam quality samples are illustrated in Figure 2.16.



Figure 2.16. The 2D section of different foam fraction cement by CT Scanning [26]

Kutchko et al. (2014) expressed that the use of foamed cement systems for deep-water applications has been increasing and is often the system of choice for shallow hazard mitigation. They developed a predictive relationship between the mesostructured and physical properties of foamed cements used in offshore applications. Samples of foamed cement have been generated using both atmospheric laboratory and highpressure field preparation methods. Field-generated foamed cement samples were collected under constant pressure (CP) sample cylinders using the same full-scale field equipment used to generate foamed cements in a well. These samples were scanned while inside the CP cylinders using X-ray Computed Tomography with a scan resolution of approximately 35 µm. Results of the laboratory testing indicate a correlation between foam quality, bubble size distribution and physical properties such as strength and permeability. Initial results also highlight key differences in the laboratory and field-generated foamed cements. They are provided a better understanding of the effects that foam cement production, transport downhole, and delivery to the wellbore annulus has on the overall sealing process [27].

They reported the effect of foam quality on the porosity, permeability, compressive strength and Young's modulus for two different foam agents, FCR1 and FCR2. These results can be seen in Figure 2.17. It shows that by increasing the foam quality (FQ) from 10% to 40%, the porosity and permeability are increased and the CS and Young's modulus is decreased [27].

Pang et al. (2016) used X-ray microcomputed tomography (micro-CT) to elucidate relationships between the foaming process, foamed cement microstructure or morphology, and macroscopic performance of the material's mechanical properties. Foamed cement slurries were prepared using a traditional multiblade laboratory blender to investigate the

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influencing factors on foamed cement properties. The influences of shear rate, mixing energy, surfactant concentration, and base cement slurry composition on the properties of set foamed cement were specifically studied [28].



Figure 2.17. The effect of foam quality on the porosity, permeability, compressive strength and Young's modulus for two different foam agents FCR1 and FCR2 [27]

Approximately 25 foamed cement slurries with foam qualities (FQs) ranging from 20% to 80% were produced. The microstructure and macroscopic performance of the foamed cement were quantified using micro-CT analysis and uniaxial compression tests. Test results indicate there is an important mixing energy threshold value that needs to be supplied to produce stable foamed cement. The maximum achievable FQ is determined by the shear rate during the foaming process. Once the minimum qualifications for obtaining stable foamed cement and a target FQ are met, a further increase in mixing energy or shear rate has little effect on the foamed cement microstructure or macroscopic performance. However,

excessive mixing energy supplied at a high shear rate can lead to a higher initial slurry temperature, a less homogenous microstructure, and relatively poor mechanical properties after the foamed cement has set [28].

Kutchko et al. (2016) assessed three sets of field-generated foamed cement at in-situ conditions using surface operations. In this data set, industrial bottled N<sub>2</sub> gas was used to generate the field samples versus the cryogenic nitrogen in previous tests. The advantage in using bottled N<sub>2</sub> was the ability to mix and pump at lower rates while maintaining an accurate nitrogen delivery, thus allowing collection of low-quality foamed cement samples. results of X-ray Computed Tomography scans of the constant pressure sample cylinders which showed that collection processes have a dramatic influence on the structure of the cured foamed cement. Physical properties such as porosity and permeability were also measured and indicate a strong relationship between foam quality and homogeneity of the samples are presented. They ultimately offered the ability to predict the behavior of foamed cements under in situ conditions when compared to laboratory generated foamed cements [29].

Glosser et al. (2016) defined atomization energy as a new parameter which affects the foamed cement generated features in field or laboratory. It is shown that the "atomization energy" imparted by the high-pressure injection of nitrogen gas into the field mixed foamed cement slurry is – by a significant margin – the highest energy process, and has a major impact on the void system in the cement slurry. Quantifying the energy exchanges across the laboratory and field processes provides a basis for understanding relative impacts of these variables on cement structure, and can ultimately lead to the development of practices to improve cement testing and performance [30].

Gieger et al. (2016) presented an assessment of the physical and mechanical properties of the field generated foam cements. These

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properties are measured to determine the performance of cements generated at field conditions, and for comparison with data sets obtained from laboratory-generated cement. These measured properties are also integral data for the development of empirical relationships used to estimate the properties of cements existing in the wellbore. Measurements of mechanical properties include both dynamic and static testing methods. These systematic variations provide insight into how foam cement properties may be altered when exposed to pressure variations within the wellbore. Empirical relationships are developed between parameters such as compressive strength, permeability, porosity, p-wave velocity, and Young's modulus for foam cements using linear and non-linear least-squared regression analysis. Preliminary empirical relationships showed strong associations between compressive strength and each of the various properties of interest. This was a promising development in efforts to predict the properties of in-situ cements. The empirical relationships reported in their study may help to provide, using the few measured properties of placed cements, a method of estimating physical and mechanical properties. Their paper Information is intended to aid cement design and wellbore planning for more reliable wellbores [31].

Ahmed and Hashmath (2017) addressed that the density of foam concrete usually varies from 25 pcf or 400 kg/m<sup>3</sup> to 100 pcf or 1600 kg/m<sup>3</sup>. The density is normally controlled by substituting fully or part of the fine aggregate with foam. They described that, the foam concrete doesn't pass many of strength test like conventional concrete but as it is a lightweight cement it doesn't need to match the standards of conventional cement. They expressed that, this type of cement is basically used for the preparation of oil and gas wells lightweight cementing, hole filling cementing, precast blocks, insulating floor screed, insulating roof screed, filling of hollow blocks, sunken portion filling, precast wall elements or panels, Prefabricated insulation boards etc. Their study is carried out by adding coconut shell fibers and crimped Steel fibers to foam cement and comparing their compressive strength with normal foam concrete. The compressive strength of foam concrete increase with the increase of coconut shell fibers at 7% and gradually decreased at 9%. Addition of steel fibers also increases the weight of the concrete and worked effectively in a low density of concrete [32].

Antunes et al. (2017) investigated the foam cementing case study in Brazil for a ultradeep water well. It is located in water depths ranging from 1500 m to 2100 m and presents hydrocarbon accumulation in sandstone formations comprised of ten units. While conducting infield drilling, depleted zones with severe lost circulation issues were expected. Cementing operations in such conditions can be challenging and require special attention to cementing the drill-string and completely achieving zonal isolation objectives. During preplanning, foamed cement was considered based on its ability to mitigate formation compaction caused by formation depletion, the capability to fill lost-circulation voids compared to nonfoamed slurries, its excellent displacement properties, and good cement bond results, making it an excellent solution for this challenging scenario. The 12<sup>1</sup>/<sub>4</sub> in. production section was drilled to total depth (TD) using managed pressure drilling (MPD) technology and synthetic-based mud (SBM) at a density of 8.7 ppg + surface backpressure (SBP). The goals for the cementing operation were the same as for the other production wells. The cementing challenges were significant and required the use of lightweight slurry to remain within the operational pressure window. Cementing best practices were closely followed, helping ensure a good bond was obtained under these conditions. Finally, the post-job cement bond log showed excellent bonding to the formation and pipe, which meant competent zonal isolation and wellbore were delivered for production [33].

Cunningham et al. (2017) studied the difference between laboratory and field generated foamed cement. In the literature, there is a metaresearch on the different foam cementing projects. laboratory testing is used to estimate the performance characteristics of foamed cement slurries that are designed for well applications. It is well known that significant differences exist between the methods used to generate foamed cement in the laboratory and those used to generate foamed cement for field applications. The implications of these differences on foamed slurry characteristics (e.g., bubble size distribution) and performance properties (e.g., stability, permeability, mechanical properties) has not been well analyzed or well understood. They examined the differences between foamed slurries generated with laboratory equipment and field foamed cementing equipment. Methods for generating and capturing foamed cement samples using field equipment are discussed, as well as analytical methods for evaluating the characteristics of foamed cements [34].

Rommel et al. (2017) used synthetic foam-agent ratio with water of 1:20. The composition of the foam concrete made with mixed cement: sand 1:2.75 with w/c 0.425 with the composition of the foam respectively 0%, 2%, 3% and 4% by weight of cement used. Testing is done to the density, strength and water absorption of foam concrete. Results of testing the density of foam concrete age 28 days obtained the lowest value of 1560.40 kg/m<sup>3</sup> at 4% usage foaming agent. While the strength largest retrieved 13 MPa, as water absorption reached 8.54% in 2% foam agent, better than the concrete without the foaming agent with the composition of the mixture of cement and water in the same. In their study, they focused on the characteristics of foam concrete with the usage of foam agent percent variation and the foamed cementing technology in civil engineering [35].

Dhanunjaya et al. (2018) analyzed the effect of natural fibers on the foamed cements (concretes) properties. They introduced that, some fibers

actually reduce the strength of cement. The amount of fibers added to a cement mix is expressed as a percentage of the total volume of the composite (cement and fibers), termed "volume fraction" ( $V_f$ ).  $V_f$  typically ranges from 0.1 to 3%. The aspect ratio (I/d) is calculated by dividing fiber length (I) by its diameter (d). Fibers with a non-circular cross-section use an equivalent diameter for the calculation of aspect ratio. If the fiber's modulus of elasticity is higher than the matrix (cement or mortar binder), they help to carry the load by increasing the tensile strength of the material. Increasing the aspect ratio of the fiber usually segments the flexural strength and toughness of the matrix. However, fibers that are too long tend to "ball" in the mix and create workability problems. Sisal fiber effect on CS are studied and they found that the strength of the foam concrete without fiber is 0.6 MPa and with fiber is 0.65 MPa, and this shows the increase in strength by adding fiber [36].

The summary of literature review, which is explained, is presented in Table 2.7 briefly.

#### 2.4. Summary of Literature Review

The most of foamed cements in this literature were designed by a particular method for a specific foaming agent. Each of these techniques and formulations is monopolized by a few companies such as Haliburton and Schlumberger. Therefore, the details of foamed cements composition were unknown. In the current research, the new technique for foamed cement preparation procedure (on the base of high shear rate method in API RP 10B-2/4) is developed and a new formulation for foamed cements is introduced. This formulation has a simple procedure for preparation and all of its components are not so expensive. Moreover, all of the current designed foamed cement additives are accessible in Iran and its preparation and experimental tests do not need very complex apparatuses. The summary of literature review is listed in Table 2.7.

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Table 2.7. Summary of literatu	re review
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Researcher	Year	Title	Description
P. M. McElfresh and V. C. Go Boncan	1982	Applications of foam cement	In this discussion is the description of an improved atomizer for generating foam cement. From yard tests utilizing field equipment, it was found that this device improved the foam stability by means of better disbursement of the nitrogen in the cement slurry.
W. Harms and J. Febus	1985	Cementing of fragile-formation wells with foamed cement slurries	The properties of foam cements are discussed and more than 60 cementing jobs completed with foam cement are summarized.
P. T. Gray and D. R. Masters	2000	Foam and foam/cement mixture	Design the foamed Cement mixture, which is laid to form a desired concrete structure, and the bubbles disintegrate or transform to leave voids of similar size uniformity dispersed throughout the concrete structure.
Narayanan and Ramamurthy	2000	Structure and properties of aerated concrete: a review	They classified the investigations on the properties of aerated concrete in terms of physical, chemical, mechanical and functional.
Kearsley and Wainwright	2001	The effect of high fly ash content on the compressive strength of foamed concrete	They studied the effects, on the properties of foamed concrete, of replacing large volumes of cement (up to 75% by weight) with both classified and unclassified fly ash.
J. E. Griffith et al.	2004	Foam cement engineering and implementation of cement sheath integrity at high temperature and high pressure	Presents and discusses the engineering analysis to determine the optimum foamed cement sheath properties for integrity during the life of the well at HPHT conditions.
R. Ahmed et al.	2009	Rheology of foamed cement	Presents results of an experimental study of foamed cement rheology. Viscosity curves of foamed cements were obtained using a flow through rotational viscometer.

K. Ramamurthy et al. Just and Middendorf	2009 2009	A classification of studies on properties of foam concrete Microstructure of high-strength foam concrete	The focus of this paper is to classify literature on foam concrete in terms of constituent materials (foaming agent, cement), mix proportioning, methods, hardened properties of foam concrete. They revealed that foam concretes are divided into two groups: on the one hand the physically foamed concrete is mixed in fast rotating pug mill mixers by using foaming agents.
Crandall et al.	2014	Foamed cement analysis with computed tomography	They have produced the first high-resolution Xray computed tomography (CT) three-dimensional images of atmospheric and field generated foamed cement across a range of foam qualities.
B. Kutchko et al.	2014	Assessment of foamed cement used in deep offshore wells	Research is being conducted to develop a predictive relationship between the mesostructure and physical properties of foamed cements used in offshore applications. Samples of foamed cement have been generated using both atmospheric laboratory and high-pressure field preparation methods.
X. Pang et al.	2016	Influencing factors of the microstructure and macroscopic performance of foamed cement	This study uses X-ray microcomputed tomography (micro-CT) to elucidate relationships between the foaming process, foamed cement microstructure or morphology, and macroscopic performance of the material's mechanical properties.
B. Kutchko et al.	2016	A look at processes impacting foamed cements	Industrial bottled N <sub>2</sub> gas was used to generate the field samples versus the cryogenic nitrogen in tests. Physical properties such as porosity and permeability, were also measured and indicate a strong relationship between foam quality and homogeneity of the samples.
Glosser et al.	2016	Relationship between operational variables, fundamental physics and foamed cement properties in lab and field generated foamed cement slurries	They defined atomization energy as a new parameter which affects the foamed cement generated features in field or laboratory.

		Physical and	
Gieger et al.	2016	mechanical properties of field- generated foam cements: implications for cement property prediction and modeling	They presented an assessment of the physical and mechanical properties of the field generated foam cements.
Ahmed and Hashmath	2017	Experimental investigation of strength characteristics of foam concrete	Their study is carried out by adding coconut shell fibers and crimped Steel fibers to foam cement and comparing their compressive strength with normal foam concrete.
T. Antunes et al.	2017	First application of foamed cement for production operation in ultra- deep-water well: Case history	Presents a case history where foamed cement was used for a production casing in an ultra-deep-water well in Brazil. The 12 1/4-in. production section was drilled to total depth (TD) using managed pressure drilling (MPD) technology and synthetic-based mud (SBM) at a density of 8.7 lbm/gal + surface backpressure (SBP).
E. Cunningham et al. 2017		Defining the difference between laboratory and field- generated foamed cement	Examine the differences between foamed slurries generated with laboratory equipment and field foamed cementing equipment. Methods for generating and capturing foamed cement samples using field equipment are discussed.
E. Rommel et al.	2017	Characteristics of foam concrete with usage of foam agent which varies (review on density, strength, and water absorption)	The composition of the foam concrete made with mixed cement: w/c 0.425 with the composition of the foam respectively 0%, 2%, 3% and 4% by weight of cement used. Testing is done to the density, strength and water absorption of foam concrete.
Dhanunjaya et al.	2018	An experimental study on the durability properties of foam concrete with addition of natural fibers	They analyzed the effect of natural fibers on the foamed cements (concretes) properties.

#### **CHAPTER THREE**

# MATERIALS, EXPERIMENTAL EQUIPMENTS AND PROCEDURES

### **3.1. Introduction**

Experimental tests for any research and its development to the industry job is necessary. Hence, before the oil and gas wells cementing jobs are started, laboratory tests must be done to reduce the risks of this fundamental parts of drilling operations. Obviously, for the first foam cement formulation innovation in the Iranian oil industry, these tests were done in extreme accurate condition. All of the experimental jobs were conducted under NIDC's oil wells cementing engineers and technician's supervision. API RP 10B-2 to 4 explains the operational procedures for preparing cement slurries for the well in the laboratory. Cementing Engineering Manual and Cementing Material Manual was used for better cement designing in this research. Also, ISO 10426-4, (Methods for atmospheric foamed cement slurry preparation and testing) was very useful for better test evaluations.

This chapter summarizes the new foam cement slurry materials properties and measurement techniques by different apparatuses. In the first section of this chapter materials are introduced and in the second section, the experimental instruments and experiments procedures are described. Different tests to reach the best foam cement properties are done and the target is to formulate a foam cement (as a new lightweight cement

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designing and technology) with low and acceptable CS to density ratio, lowest permeability and free water as possible, good rheology and fluid loss behavior with enough thickening time.

# 3.2. Materials

For best foam cement formulation, different materials are used as that explained in the following sections:

# **3.2.1.** Cement

Class G cement is used as the basic slurry design. The properties of each API cement classes are investigated in chapter 2. Figure 3.1 shows the image of common class G cement and its compounds percentages and properties are presented in Table 3.1. Delijan cement factory product was used in this project. Class G cement is the basic cement in Iranian cementing operations and that is adaptable to different additives and new designing.



Figure 3.1. Image of class G cement

Table 3.1. Class G cement properties [1]

API	Compounds (%)			Fineness CS (8hr)		CS (8hr) 140°F	ТТ	Free water	
Class	C <sub>3</sub> S	C <sub>2</sub> S	C <sub>3</sub> A	C <sub>4</sub> AF	(cm²/gr)	(psi)	(psi)	(min)	(%)
G	50	30	5	12	1400- 1700	300	1500	85- 120	5-9

#### 3.2.2. Water

In this study distilled water (DW) is used for best accuracy. The reason for DW usage is that piping (city) water (PW) has different soluble ions, which affect the slurry properties and react with cement additives. Moreover, at different time and conditions, the PW properties may change and however, the DW properties always are constant. As a result, all cement tests are precise and repeatable and this is caused by DW constant features.

#### 3.2.3. Retarders

Three different lignosulfonates are used in this work to reach the best foam cement formulation. Each of them tolerates a specific range of temperature. These materials which used in NIDC's cement laboratory have commercial names. Two of them are OR5 and OR12, that are calciumlignosulfonate and the third one is sodium-lignosulfonate which name is JR120. The basic structure of lignosulfonates is showed before, in chapter 2, Figure 2.7. Table 3.2 illustrates the temperature ranges of the effective retarding behavior of these materials and also, their chemical structure, properties, and their chemical formula. Figure 3.2 shows NIDC's available retarding additives. Note that details of the exact retarding behavior of these additives are not available and this causes the difference in their efficiency in different ranges of temperature. Table 3.2 reports the mono structure and formula of lignosulfonates as polymers.



Figure 3.2. Different retarders that are used in this thesis, from left to right: OR5, OR12 and JR120

Retarder	OR5	OR12	JR120	
Туре	Calcium- Lignosulfonate	Calcium- Lignosulfonate + Organic Acid	Sodium - Lignosulfonate + Organic Acid	
Effective Temperature Range (°F)	210>T	210 <t<400< th=""><th>200<t<800< th=""></t<800<></th></t<400<>	200 <t<800< th=""></t<800<>	
Chemical Formula	C <sub>20</sub> H <sub>24</sub> C	$C_{20}H_{24}Na_2O_{10}S_2$		
Molecular Weight	528	534.502		
Color	Dark Brown	Light Brown	White to Yellow	
Specific Gravity	1.0 1.5		1.1	
Chemical Structure				

Table 3.2. Different NIDC's retarders properties

# 3.2.4. Boric Acid

Boric acid, also called hydrogen borate is a weak, monobasic Lewis acid of boron, which is often used as an antiseptic, insecticide, flame retardant, neutron absorber, retarder aide or precursor to other chemical compounds. In this foam cement formulation, for compatibility improvement between foam agent and retarders, boric acid was added. Its photographic illustrates in the Figure below. Other boric acid properties are listed in Table 3.3. Boric acid is soluble in water and alcohols. For instance, at near 210°F its solubility is 27.53 mgr/100mL in water [37].



Figure 3.3. Photographic of Boric Acid

Table 3.3. Boric Acid Properties [38]

Chemical Formula	Molecular Weight	Appearance	рН	Specific Gravity	Formal Charge	Chemical Structure
H <sub>3</sub> BO <sub>3</sub>	61.831	White crystalline solid	5.1	1.437	0	H <sup>-0</sup> H <sup>-0</sup> H <sup>-0</sup>

#### 3.2.5. Foam Agent

Sodium laureth sulfate (SLES), an accepted contraction of sodium lauryl ether sulfate (SLES), is an anionic detergent and surfactant found in many personal care products (soaps, shampoos, toothpaste etc.). SLES is an inexpensive and very effective foaming agent (FA). Its chemical formula is  $CH_3(CH_2)_{11}(OCH_2CH_2)_n OSO_3Na$ . Sometimes the number represented by *n* is specified in the name, for example, laureth-2 sulfate. The product is heterogeneous in the number of ethoxyl groups, where *n* is the mean. It is common for commercial products for n=3. This FA is known as an anionic surfactant and the reason for this surfactant type usage is that they are very effective in aqueous solvents. They are more powerful in foaming ability than non-ionic or cationic surfactants. The base of each slurry is water and this was caused by choosing SLES as an anionic surfactant with high foaming ability [39]. SLES's image and its chemical structure are illustrated in Figure 3.4. Another reason for SLES usage is that anionic surfactants are not affected by hard water and its ions, thus it is a very good choice in cementing operations.



Figure 3.4. Photographic and chemical structure of foam agent, sodium lauryl ether sulfate (SLES)

Some of the SLES physical properties are listed in the Table below. These properties of this foaming agent are achieved specifically for this study by experiments.

Material	Physical Condition	Color	Specific Gravity	рН
SLES	Liquid	Colorless	$1.10 \pm 0.03$	$8.8 \pm 0.5$

Table 3.4. Physical prop	perties of SLES
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#### **3.2.6.** Other Materials

Some materials and additives which used for common lightweight cements designing are listed in the Table 3.5. Lightweight cements are used for better comparison between them and new foam cement properties.

Additives	Description
O-Lightweight	Expander (Ceramics bubbles are filled with $O_2$ and $N_2$ )
JFLC320	Fluid loss controller
O-CFR4	Dispersant (Cement Friction Reducer)
O-MicroBlock	A liquid expander with specific gravity near 1.40
O-GasBlock	Gas migration controller
TA47	Antifoam agent
Micro Silica	Expander with CS extender

Table 3.5. Some common lightweight slurry additives

# **3.3. Sample Preparation**

This section summarizes the foam slurry measurement techniques. In this thesis, different foam cement sample preparation methods are used and the target is achieved. This optimum mixing and designing procedure will be reported in mixer's procedure section. The reason for choosing this method and why this is the optimum procedure will be discussed in chapter four. Basically, API RP 10B-2 and ISO 10426-4 are used for better sampling. In the following sections of this thesis the optimum foam cement, which is formulated and designed in this work by different experiments, will be called FBJ.

#### 3.4. Apparatuses

For best investigation of foam cement samples, different instruments were used and tests were conducted.

#### 3.4.1. Mixer

A two-speed mixer of propeller type is typically used to prepare 600mL of the slurry by making use of 4000RPM and 12000RPM rotation speed. The mixer in manual mode can rotate from 0 to 16000RPM. Mixers have two different blades that both of them are very useful for foam slurries. These blades are shown in Figure 3.6, the longer blades are more effective in foam slurries mixing. Photographic of NIDC's mixer is illustrated in Figure 3.5. The foam slurries were prepared by this mixer [3].



Figure 3.5. Photographic of NIDC's cement laboratory mixer



Figure 3.6. Two different mixer's blades

# 3.4.1.1.Procedure

The optimum and exact design procedure for best foam cement properties are attained and FBJ formulation procedure is obtained as following steps:

- 1. Place the 350gr DW in the mixing container.
- 2. Turn on the mixer at 4000RPM rotation speed.
- 3. Add 0.78gr (0.1 % BWOC) boric acid to water at 4000RPM. Remain to mix for one minute.
- 4. Add 0.78gr (0.1 % BWOC) JR120 to solution at 4000RPM. Remain to mix for one minute.
- 5. 778g class G cement is added to the mixture in 15s. Remain mixing process for 35 seconds at 4000RPM.
- Pour 4.84gr (0.07 gal/Sx) foaming agent (SLES) in 10s at 4000RPM. Remain to mix for 35 seconds.
- 7. Switch the fix 4000RPM mixing mode to variable manual mixing position.
- 8. Increase the rotation speed from 0 to 12000RPM in manual mode in one minute.
- 9. Remain to mix at 12000RPM for 35 seconds.
- 10. Turn off the mixer at 12000RPM. Note that during all of these 10 steps, do not place the cover on the mixing container to allow the air to enter the slurry.

FBJ composition and formulation as this thesis goal is reported below in Table 3.6:

Item	Cement and Additives Name	Concentration (BWOC)	Amount (gr & cc)
1	Class G cement	100%	778
2	Distilled Water	-	350
3	Boric Acid	0.1%	0.78
4	JR120	0.1%	0.78
5	SLES	0.07 gal/Sx	4.84

Table 3.6. Foam cement composition (FBJ formulation)

# 3.4.2. Balance

Accurate mass measurement is the prerequisite for any cement designing. Digital mass balances that were used in this project for cement and additives weighting are shown in Figure 3.7.



Figure 3.7. Different NIDC's digital balances

# 3.4.3. Mud Balance

This is an accurate, self-contained measuring device used to determine the density of drilling fluids and cement slurries. It has a range of 7 to 24ppg or specific gravity of 0.84 to 2.88. The mud balance consists of a constant volume sample cup and lid connected to a balance arm that has graduated scales. A rider is moved along the balance arm to indicate the scale readings. There is a knife edge attached to the arm near the balance cup, and a bubble level built into the knife edge to level the arm. A fulcrum is mounted on a base stand if used, or in the plastic carrying case, if it is used. Figure 3.8 shows the NIDC's cement laboratory mud balance [40].



Figure 3.8. Image of NIDC's cement laboratory mud balance

# 3.4.3.1. Procedure

- 1. The balance cup should be clean and dry before it is filled with a slurry sample.
- 2. Foam cement samples should not be deaerated, unlike other slurries.
- 3. Place the base stand or the carrying case on a surface that is approximately level.

- 4. Fill the balance cup with the sample to be tested. Put the lid onto the balance cup by pushing it downward with a slow rotating motion until it is firmly seated. Make sure that some of the test samples is forced out through the vent hole.
- 5. Clean any sample from the outside of the balance cup and lid.
- 6. Fit the knife edge of the balance arm into the fulcrum and balance the assembly by moving the rider along the arm. The mud balance is horizontal when the level bubble fluctuates an equal distance to either side of the center line.
- Take the reading from the side of the rider nearest the balance cup. The measurement reading should be reported to the nearest 0.1ppg or 0.5pcf.
- 8. Empty the sample from the cup. Clean and dry the entire assembly as soon as possible.

This is clear that this measurement procedure can be run for under downhole temperature samples for measuring the HT sample density.

#### **3.4.4.** Viscometer (VG Meter)

Viscometers or VG meters (Viscosity-Gel meter) are designed to measure the rheological properties of test fluids by measuring shear stress at specific shear rates. Viscometers are versatile instruments that have been used in research laboratories, field and mobile labs, and onsite QC testing. Each viscometer can be equipped with a variety of bobs, rotors, and springs. This provides the user with a wide measurement range in addition to providing different gap sizes depending upon the fluid being tested. The measurement fluid is contained within the annular space or shear gap between the rotor and bob. The rotor is rotated at known velocities (shear rates) and the viscous drag exerted by the test fluid creates torque on the bob. This torque is transmitted to a precision torsion spring, and its deflection is measured and related to shear stress. The equations used to calculate the fluid viscosity or sometimes by using different software such as CemCADE for slurries, viscosity can be calculated [41]. The image and structure of the VG meter are illustrated in Figure 3.9.



Figure 3.9. Structure and photographic of NIDC's viscometer

# 3.4.4.1. Procedure

- 1. Fill the sample cup with the fluid to be tested up to the 350mL scribed line.
- 2. Place the sample cup on the sample cup table and rotate the cup until the three feet on the cup are engaged in the holes.
- 3. Raise the sample cup and cup table until the fluid level meets the scribed line on the rotor. Tighten the locking nut on the sample cup table.
- 4. Operate the motor at one of the preset 12 (1, 2, 3, 6, 10, 20, 30, 60, 100, 200, 300, 600RPM) speeds. Turn the rotary dial to the desired speed. Initially, running at high speed may be beneficial to quickly fill the annular space between the rotor and bob. This is especially beneficial for high viscosity fluids.

- 5. Observe the reading from the dial in the instrument by viewing through the illuminated lens. The pointer will indicate the dial reading. Allow the reading to stabilize before recording the result. Record the observation as a dial reading at the selected RPM (shear rate).
- 6. A schedule of increasing motor speeds (shear rates) is recommended to study "unsheared" fluid viscosity. Alternately, a schedule of decreasing motor speeds is used to study sheared viscosity behavior. Consult the industry standard for a recommended schedule of test speeds.
- 7. Clean the instrument thoroughly upon completion.
- 8. Remove the rotor by holding the top portion of the rotor shaft and unscrewing the rotor nut clockwise. Avoid hitting the bob as the rotor is removed since it may damage the bob shaft. The machined surfaces of the rotor that fit into the rotor shaft must be kept clean and without scratches to preserve the accurate alignment of the rotor.
- 9. Remove the bob by turning counterclockwise until the rotation is impeded by the mechanical stop. Twist the bob while gently pulling downward. Always clean the bob and remove any debris from the tapered hole. It is very important to keep the mounting surface in the bob very clean for proper mechanical alignment of the bob.
- 10. To replace the bob, push gently upward and twist counterclockwise to lock the bob to the bob shaft [42].

In this thesis, CemCADE software with NIDC license is used for Herschel Bulkley model of foam cement slurries. Some of the equations that can be used in rheology experiment and calculations are as below [5]:

Apparent Viscosity (cP) = 
$$AV = \theta_{600RPM}/2$$
 EQ. 3.1

$$\begin{aligned} Plastic Viscosity (cP) &= PV = \theta_{600RPM} - \theta_{300RPM} \text{ or} \\ PV (cP) (Proposed for cement calculations) \\ &= 1.5(\theta_{300RPM} - \theta_{100RPM}) \end{aligned} \qquad \text{EQ. 3.2} \end{aligned}$$

$$\begin{aligned} \text{Yield Point} & \left(\frac{lb}{100ft^2}\right)(\text{approximate value}) = \text{YP} \\ &= \theta_{300RPM} - \text{PV} \quad \text{or} \\ \\ \text{Yield Point} & \left(\frac{lb}{100ft^2}\right)(\text{Proposed for cement calculations}) \\ &= \theta_{300RPM} - 1.5(\theta_{300RPM} - \theta_{100RPM}) \end{aligned}$$

Yield Point 
$$\left(\frac{lb}{100ft^2}\right)$$
 (exact value) = YP  
= 1.065 × ( $\theta_{300RPM} - PV$ )

Also, in VG meter the following equations are established for shear stress and shear rate calculation:

Shear Rate 
$$(1/sec) = \dot{\gamma} = 1.703 \times RPM$$
 EQ. 3.5

Shear Stress 
$$\left(\frac{lb}{100ft^2}\right) = \tau = 1.0678 \times \theta$$
 EQ. 3.6

# 3.4.5. Atmospheric Consistometer (AC)

Atmospheric Consistometer is used for various tests of oil well cements as detailed in the API Spec 10 A/B. The apparatus is used in conjunction with tests for:

- Determination of Water Content of Slurry;
- Determination of Fluid Loss;
- Determination of Rheological Properties of Cement Slurries;

Thus, rheological properties of foam slurry in hole-bottom temperature condition is gathered by moving the sample from atmospheric consistometer cell to VG meter container after 30 minutes which is heated.

Atmospheric Consistometer consists of a stainless-steel water bath that houses two slurry containers. The slurry containers are rotated by engaging the pins of the lid with the slots on the rotator. The rotators are fitted with timing sprockets driven by the motor, which is factory set at 150 rpm. The belt also drives an impeller that agitates the water bath. AC indicates consistency and temperature on a panel-mounted strip chart recorder. Apparatus also includes audible alarms that sound at preset consistency values. The alarm set points are individually adjustable on the recorder. Slurry consistency is expressed in Bearden units of consistency, Bc, where 100 Bc is equivalent to the spring deflection observed with 2080 grams-centimeter of torque (400grams weight) using the weight-loaded calibrating device. The measuring of thickening time, as determined in a high-temperature high-pressure (HPHT) consistometer or atmospheric consistometer, is related to the torque being placed on a stationary paddle within the rotating slurry cup. As the cement thickens, the torque increases on the spring-loaded potentiometer connected to the paddle shaft. This torque is recorded as a Direct Current (DC) voltage across a resistor on top of the potentiometer. The actual viscosity of the cement can then be derived from a linear plot of DC volts versus viscosity in Bearden Units of Consistency (BC) [41]. Figure 3.10 presents the atmospheric consistometer and its structure which is used in this project and NIDC's cement laboratory. Also, its slurry container is shown in Figure 3.10.

#### 3.4.5.1.Procedure

- 1. Remove the slurry container.
- 2. Prepare sample and fill the slurry container.
- 3. Attach the container lid to the slurry container.

4. Install the slurry container in the instrument. Be sure the bottom roll pin on the container lid fits into the brass rotator sleeve slots.



5. Turn the container lid until the torque bar fits into the anchor stop.

Figure 3.10. Photographic of atmospheric consistometer and its structure and cell

- 6. Verify the Mic plug is connected to the appropriate socket located on the front panel.
- 7. Turn the master switch on.
- Use the temperature controller to set the desired temperature or heating profile. At this project, the circulating temperature is assumed 170°F.
- 9. Turn on the motor switch. Turn on the heater switch.
- 10. An alarm will sound upon completion of the test (the alarm is factory set to 100 Bc and will independently sound for each cylinder).
- 11. After completion of the test, the alarm will sound. Turn off the motor switch.
- 12. Remove the slurry container. If only one slurry container is removed, the motor switch can be turned on to finish the other test if necessary.
- 13. Before draining the water, turn off the heater and power switches [43].

#### 3.4.6. High-Pressure High Temperature (HPHT) Consistometer

Thickening time or the time cement slurry remains able to be pumped into the well, is the most critical properties in designing cement. A short thickening time is desired while maintaining the special properties of the cement's design. The thickening time of cement can be measured in a laboratory by testing a sample of the cement slurry in a pressurized Consistometer. The elapsed time between an initial application of pressure and temperature on the slurry sample and the development of 100 Bearden units of consistency (Bc) is the thickening time for the sample at a particular specification test schedule. The Pressurized Consistometer incorporates a rotating, cylindrical Slurry Cup equipped with a stationary paddle assembly enclosed in a pressure chamber designed for a working pressure of 275MPa (40000psi) at a maximum temperature of 600°F (315°C). An air-operated hydraulic pump generates pressure to the cylinder assembly. Heat is supplied to the chamber by a 5000-watt internal tubular heater controlled by the automatic temperature control system program. Thermocouples are provided for determining the temperatures of the oil bath and cement slurry. The programmable temperature controller will automatically control the rate of temperature rise of the slurry (i.e., temperature gradient). When the slurry reaches the desired maximum temperature, the controller will hold the slurry temperature at that level. The slurry container is rotated at a constant speed of  $150 \pm 15$  RPM by a Magnetic Drive. The viscosity (i.e., consistency) of the cement slurry is indicated by a meter and is recorded on a chart as a DC voltage obtained from a potentiometer installed within the pressure cylinder. The potentiometer contains a standardized torsion spring, which resists the rotating force of the paddle. Rotational force is proportional to consistency of the cement slurry [44]. Two different HPHT consistometers that are used

in NIDC's cement laboratory at this thesis are illustrated in Figure 3.11. Also, one foamed cement that was thickened is showed in this Figure.



Figure 3.11. Two different HPHT consistometers and a thickened foam cement in NIDC's cement laboratory

# 3.4.6.1. Procedure

- 1. Turn the Power switch ON.
- Check to be certain that the slurry cup and pot mech are properly engaged. Turn the Motor switch to ON. No rubbing noise should be heard.
- 3. Slide the thermocouple through the test cell plug into the slurry cup paddle shaft.
- 4. Next, fill the test cell with oil.
- 5. To apply the initial pressure to the test cell without pressure control, turn the Pump Switch to the MANUAL position. When the pressure reaches the desired level (4000psi for this project) turn the Pump Switch to the OFF position.
- 6. Turn the Heater Switch to the ON position, the Pump Switch to the AUTO position and start the timer and set the temperature controller on 170°F at near one hour at this project. (i.e., the temperature reaches to 170°F in one hour, automatically) After the final

temperature is reached for the schedule being run, the controller will continue on a programmed soak until the schedule is completed and DC volt reaches to 7 and alarm will be heard. A buzzer will sound, signaling the slurry has reached the required consistency. Now the consistency, temperature, pressure versus time for better analyzing of TT is available [45].

#### **3.4.7.** Free Water and Settling Tests Apparatuses

Free water is defined as water not required for cement hydration. When the cement slurry flow stops, free water separates on the top of the cement column. A cement slurry is basically made up of dry cement mixed with water in a correct ratio. For foam cements, free water and settling tests are very critical and this is because of their low density. Free water and settling tests have the same and simple apparatuses. As is showed in Figure 3.12, one 250mL-graduated cylinder, an aluminium foil cover and a pipette for measuring the free water volume are necessary for these tests [46].



Figure 3.12. Free Water test

#### **3.4.7.1.** The procedure for Free Water Test

Different methods are used for slurry free water measurement. Such as API, Dowell, Amoco, BP and Chevron methods. The most common methods in the industry experiments are API and Dowell. In this project, the Dowell method is recommended. The two types of tests are (1) simple free-water and density-gradient test and (2) 45°-angle free-water test. These methods procedures are as follow:

- 1. After slurry preparation, condition the slurry for 20 min in an atmospheric consistometer at the bottom-hole circulating temperature (BHCT). Here the BHCT was assumed 170°F.
- 2. Pour the slurry into a 250-mL graduated cylinder.
- 3. Seal the graduated cylinder to prevent evaporation and place on a vibration-free surface for two hours.
- 4. Measure the volume of free water [3].

The procedure for the  $45^{\circ}$  angle free water test is the same as the above procedure, except that the graduated cylinder is inclined at  $45^{\circ}$  for the two-hour period. For sedimentation, these are more severe conditions.

#### **3.4.7.2. Procedure for Settling Test**

A Go/No-Go or Dowell Settling Test as outlined in the following procedure:

- 1. Prepare the cement slurry.
- 2. Start a thickening-time test.
- 3. When the specified temperature and pressure have been reached, record the slurry consistency (Bc) and turn off the consistometer drive.
- 4. After 10 minutes have elapsed with the fluid at static conditions, restart the consistometer drive and record the maximum consistency reading at the instant of startup.

5. A "Go" is a maximum consistency less than 70 Bc. A "No Go" is a maximum consistency greater than 70Bc. Any spacer or cement slurry with a "No Go" result is not to be recommended or used. Another way is that pour the slurry into the graduated cylinder and investigate the settling visually as detailed in Figure 3.13 [45].



Figure 3.13. Visual settling test in the graduated cylinder [1]

Three different slurry settling conditions are shown above. Sedimentation is the most sever settling and after that segregation is not an ideal settling condition. Ideal slurry settling feature is that slurry after two hours for a free water test, has the state as same as the situation before running the test (i.e., without segregation and sedimentation).

#### **3.4.8.** Ultrasonic Cement Analyzer (UCA)

Ultrasonic Cement Analyzer (UCA) is an instrument that measures the compressive strength of API cement under high temperature and highpressure conditions. The instrument measures the velocity of sound through the sample then relates this value to the compressive strength using proprietary algorithms. The data is presented graphically as well as being stored in a Microsoft Access database file (MDB). The image of NIDC's cement laboratory UCA and its front panel is detailed in Figure 3.14.



Figure 3.14. Photographic of UCA and its front panel

# 3.4.8.1. Procedure

- 1. Pour approximately 200cc of cement slurry into the greased test cell.
- 2. Continue to pour cement in test cell until the level is 1/4 inch (6mm) below the circular lip in the cylinder. Use the Slurry Level Gauge to obtain the proper fill level.
- 3. Use a small amount of water to continue filling the cell up to the water fill line indicated on the slurry level gauge.
- 4. Screw the top plug into the top of the cylinder. The test cell is ready to be installed in the heating jacket.
- 5. Align the pressure port in the top plug with the high-pressure filter on top of the autoclave assembly. Attach the high-pressure filter and U-tube connection to the top port on the cell.
- 6. Install the thermocouple in the other high-pressure port in the top plug.
- 7. The test cell and autoclave are now ready to begin a compressive strength test.
- Set the pressure at 4000psi and bottom-hole static temperature (BHST) at near 210°F. Adjust the connections between UCA and computer and start the test. The computer draws the graph between CS, Transit time, Temperature versus time. Generally, UCA tests are run for 24hr or 48hr [44, 47].

#### **3.4.9.** Curing Chamber

The Pressure Curing Chamber is used for curing tensile or compression specimens of oil well cements at elevated temperatures and at pressures above atmospheric, simulating conditions in the well. The specimen slurries are poured into molds, and the molds are lowered into the pressure curing cylinder. The cylinder plug is installed, the thermocouple is inserted into the cylinder head, and the cylinder is filled with water to expel air. Heat, regulated by an Automatic Temperature Program System, and pressure are then applied to the cylinder in accordance with applicable schedules. Maximum pressure and temperature are maintained until shortly before the end of the curing time specified. The temperature is then reduced, the pressure is regulated to atmospheric, and the test specimens are removed for testing [45]. The photographic of NIDC's curing chamber and its molds are presented in the Figure 3.15.



Figure 3.15. NIDC's cement laboratory curing chamber and its testing molds
# 3.4.9.1. Crush Test Procedure by Curing Chamber and Hydraulic Press (Hydraulic Jack)

- 1. Pour the cement slurry into the prepared molds.
- 2. Place the cover plate on top of the molds.
- Place the molds in a pressurized curing chamber (to near 4000psi for this thesis experiment) which is heated to the desired test initiation temperature. Assume BHST near 210°F for this research.
- 4. Once the BHST is reached, maintain it for the majority of the curing period. The curing period is the elapsed time from the start of the pressure and temperature application to testing the specimen for strength. Typical curing periods are 8, 12 and 24 hours.
- 5. At 45 min prior to the end of the curing period, remove the molds and prepare the samples for testing.
- 6. Measure the cross-sectional areas of the sides of the sample cube that are to contact the load-bearing plates of the hydraulic press. The most cubic samples have the 2 (in)\*2 (in)\*2 (in) dimensions. Therefore, each cross section is 4in<sup>2</sup>.
- 7. Using the hydraulic press, crush the sample cube and record the force required to break it.
- 8. Report the compressive strength as the force required to break the cube divided by the smallest cross-sectional area in contact with the plates of the hydraulic press. Average the compressive strengths of all acceptable sample cubes made from the same slurry and tested at the same time. For this instrument, the CS is calculated from equation 3.6. This is clear that the F is the force which is applied to the sample by a hydraulic press. Photographic of a hydraulic press (jack) is presented below [45].

$$CS (psi) = \frac{F (lb_f)}{4 (in^2)}$$
EQ. 3.7



Figure 3.16. NIDC's cement laboratory hydraulic press (hydraulic jack)

# 3.4.10. High-Pressure High-Temperature (HPHT) Filter Press

Fluid loss values for different slurries can be compared only if standard fluid-loss testing procedures are followed. A high-temperature, high-pressure fluid loss cell or a stirred fluid loss cell is used to perform a fluid loss test. Thermocouples must be mounted inside the cell's wall or immersed in the slurry to measure the jacket or slurry temperature, respectively. Pressure gauges with  $\pm 50$  psi accuracy enable the applied differential pressure (by compressed nitrogen) to be determined. A graduated cylinder is required to contain and measure the expected volume of filtrate collected during the test [40]. Figure 3.17 illustrates the NIDC's cement laboratory HPHT filter press cell (left) and a stirred fluid loss cell (right). These thesis experiments are run by HPHT filter press cell.



Figure 3.17. HPHT filter press cell (left) and a stirred fluid loss cell (right).

# **3.4.10.1. Procedure**

- 1. The slurry is mixed according to section 3.4.1.1 procedure.
- 2. The slurry conditioning should start at a temperature appropriate for the well conditions.  $(T = 170^{\circ}F)$
- 3. An atmospheric consistometer is used for slurry conditioning.
- 4. The conditioned slurry is then poured into the fluid loss cell.
- 5. The fluid loss cell, preheated to the test temperature.
- 6. A differential pressure of  $1000 \pm 50$  psi is applied to the fluid loss cell and the bottom valve of the cell is opened to start the test.
- 7. The slurry temperature is maintained at the specified temperature for the duration of the test. The filtrate is collected in the graduated cylinder. The total filtrate volume is recorded to  $\pm 1$  mL at 30 sec. and 1, 2, 5, 7.5,10,15,25 and 30 minutes.
- 8. If the fluid-loss test went the full 30 minutes (no nitrogen blowout), then the "API Fluid Loss" is calculated as follows:

API Fluid Loss = Filtrate Volume 
$$(mL) \times 2$$
 EQ. 3.8

If nitrogen blew through before the full 30 minutes, then the "Calculated API Fluid Loss" is determined as follows. where  $Q_t$  is the volume of filtrate (mL) collected at the time t (min) of the blowout.

Calculated API Fluid Loss = 
$$2 \times Q_t \times \frac{5.477}{\sqrt{t}}$$
 EQ. 3.9

The values for API Fluid Loss and Calculated API Fluid Loss are reported in units of mL/30 min [3].

#### 3.4.11. Cement Permeameter

As zonal isolation heavily relies on the permeability of cement in the wellbore, it is vital to measure the permeability of set cement in the laboratory so that necessary changes can be made prior to the execution of cement operation. Cement Permeameter provides a convenient laboratory method for checking the permeability of a cement sample under well conditions. The cement sample remains water saturated during the aging period and its permeability is measured with water [42].

The required equipment to perform a permeability test is 1) Permeameter, 2) Pressure medium, 3) molded sample holder (including brass or stainless-steel mold) or cored sample holder, 4) measuring or recording devices for gas and water flow rates. The Petroleum University of Technology's (PUT's) cement laboratory's cement permeameter is showed in Figure 3.18. Permeability tests for this project are done by this equipment.



Figure 3.18. Photographic and structure of PUT's cement permeameter

## 3.4.11.1. Procedure

- 1. Prepare the cement slurry and Pour into a clean, ungreased cement permeameter mold which has been placed on a flat plate and sealed around the outside with a thin film of grease. Figure 3.19 shows permeameter molds that filled with the FBJ foam cement sample.
- 2. Cure cement under the desired test conditions.
- 3. Remove sample from the curing chamber or bath and cool underwater to room temperature. The photographic of the cooling procedure under distilled water is illustrated in Figure 3.19 (right image).
- 4. Saturate the sample completely with distilled water.
- 5. Seal the mold in the sample holder assembly with the side marked top facing up. Place one O-ring on the top and one on the bottom of the mold to seal it in the sample holder. Do not tighten the cover of the sample holder yet.
- 6. With the aspirator bottle 12 to 24 in higher than valves (as shown in Figure 3.18, open valves slightly to allow water to flow past the

cement mold as the holder cap is screwed into place. Water will flow out of the cement holder.



Figure 3.19. Cement permeameter molds and samples cooling procedure in the distilled water bath (rightest picture)

- 7. Open the valve on the aspirator bottle to allow flow to begin. Water will flow through the bottom of the sample holder.
- 8. Place the pipette with the desired range into the flexible mount on the top of the mold holder.
- 9. Flow water through the sample for a minimum of 15 minutes or until about 1 mL has been forced through the sample into the measuring tube.
- 10. Measure the flow rate at least twice during the 15-minute interval. To accomplish this, record the level of water in the pipette. Immediately after the level is noted, start a timer. After a sufficient period of time has elapsed (generally about 5 minutes), stop the timer and record the final level in the pipette. The flow rate should be calculated in mL/sec as follows:

Flow Rate 
$$\binom{mL}{s}$$
  
=  $\frac{Initial \ level \ in \ pipette \ (mL) - Final \ level \ in \ pipette \ (mL)}{Time \ (s)}$  EQ. 3.10

- 11. The pressure should be recorded each time a flow rate measurement is made. The pressure on the gauge will be the differential pressure for the sample. In this project, the 100psi-pressure for differential pressure is assumed.
- 12. The liquid permeability of the cement is calculated by Darcy's law as follow:

$$K(mD) = 14700 \frac{Q(mL) \times \mu(cp) \times L(cm)}{A(cm^2) \times \Delta P(psi)}$$
 EQ. 3.11

In this equation, the cross-sectional area is  $(A=\pi r^2)$  and the differential pressure is  $(\Delta P = P_i - P_o)$ . Where  $P_i$  and  $P_o$  are inlet and outlet pressure respectively. Also, r is the cement plugs radius [3].

### **3.4.12.** Ovens and Water Bath

Conditioning of different samples is one step of each cement experiments. Water baths are used for best slurry conditioning in different tests such as CS measurement in atmospheric pressure and low temperatures or slurry conditioning after cure chamber for crush test and etc. In Figure 3.20 two different water baths which were used in this research are presented.

Before porosity tests will be started, cement plugs which are in cement permeameter molds should be exacted. As the foam cement is bonded to the molds wall, these filled molds are heated in the oven at a temperature near 130°C (265°F). More expansion of mold than foam cement helps better cement plugs extraction from molds. Two different ovens that were used in this research is illustrated in Figure 3.21.



Figure 3.20. Two different water baths (PUT and NIDC's cement laboratories)



Figure 3.21. Two different ovens (PUT's laboratories)

# **3.4.13. Digital Caliper (Vernier Caliper)**

The Vernier, dial, and digital calipers give a direct reading of the distance measured with high accuracy and precision. They are functionally identical, with different ways of reading the result. These calipers comprise a calibrated scale with a fixed jaw, and another jaw, with a pointer, that slides along the scale. The distance between the jaws is then read. The digital Vernier caliper that is showed in Figure 3.22, is used for accurate measurement for cement plugs dimensions. These dimensions are the input data of the porosity meter apparatus.



Figure 3.22. Digital Vernier Caliper

#### 3.4.14. Porosimeter

The Helium gas expansion porosimeter enables the determination of a sample's grain and pore volume via an isothermal helium expansion and the application of Boyle's law and Charles' law. Subsequently, porosity and grain density can be calculated. The grain density and porosity is measured on cleaned and dried plugs by a helium porosimeter. The Helium-porosimeter measures the grain volume Vg of the sample. The empty sample container of the porosimeter has a volume VS before the sample is inserted. A reference volume Vr is filled by helium and the pressure Pr is recorded. The gas is expanded from the reference volume to the sample container and the resulting pressure PX is recorded [48]. The grain volume is determined by applying Boyles law:  $P_1V_1=P_2V_2$  or:

$$P_r \times V_r = P(V_r + V_s - V_g)$$
EQ. 3.12

For helium porosimetry, effective porosity is determined by first placing the specimen into a steel chamber (known volume) of the Boyle's law double-celled helium porosimeter. Helium is then allowed to isothermally expand into the chamber from a reservoir of known volume and pressure until equilibrium pressure is reached. The photo of the helium porosimeter that foamed cement plugs porosity measurement are done by that, is presented in Figure 3.23.



Figure 3.23. Image of PUT's petroleum laboratory helium porosimeter

## 3.4.14.1. Procedure

- 1. Put the foamed cement plug in the core chamber.
- Enter the sample dimensions and mass to the porosimeter's digital panel. Dimensions and masses by caliper and balance are prepared respectively.
- 3. Start the test and open the inlet valve.
- 4. Close the inlet valve and put the sample valve in the Expand position.
- 5. Waite until apparatus parameters measurement.
- 6. Close the sample valve.
- 7. Now the porosity, bulk volume, pore volume, and the grain density are available on the monitor [48].

### **3.4.15.** Industrial Microscope

Visual analyses are always one of the inseparable parts of each research. Therefore, for accurate pore size and structure investigation, the microscopic photography from the surface of different cement samples is exploited. The industrial microscope, that is used in this work, has four lenses with different maximization as 5X, 10X, 20X and 100X. By experience and best picture resolution, the 5X lens is used for this thesis research. The image of an industrial microscope that used in this project is demonstrated in Figure 3.24.



Figure 3.24. Photographic of PUT's material laboratory industrial microscope

## **3.4.16.** Computed Tomography Scan (CT Scan)

A CT scan, also known as computed tomography scan, makes use of computer-processed combinations of many X-ray measurements taken from different angles to produce cross-sectional (tomographic) images (virtual slices) of specific areas of a scanned object, allowing the user to see inside the object without cutting. Digital geometry processing is used to further generate a three-dimensional volume of the inside of the object from a large series of two-dimensional radiographic images taken around a single axis of rotation. CT scan is widely used in medicine, although it has very much benefits for industrial researches [49].

In this project, for best foam cement samples analyzing, computed tomography scanning was evaluated. Pore distribution and each sample interior pore structures and connections are gained by this important test. CT scan tests were approached the surprising results. This test is very necessary for new foam cement designing (because of its porous structure) and it is a clear and conclusive evidence for best formulation. This test is done in TABA Medical Imaging Center, Shiraz, Iran, with a new and professional computed tomography scanner as illustrated below.



Figure 3.25. The photo of a computed tomography scanner (CT Scan)

#### **3.5. HPHT Condition of Different Tests**

In different tests and experiments that are conducted in the HPHT condition, the 4000psi pressure was assumed as a moderate pressure for Iranian fields. For temperature, two different bottom-hole static temperature (BHST) and bottom-hole circulating temperature (BHCT) was assumed 210°F and 170°F respectively. These magnitudes were considered for a moderate true vertical depth and temperature gradient in Iranian oil and gas fields. The equations 3.13 and 3.14 are illustrating that how are the BHST and BHCT calculated [3].

BHST (°F) = Temp Gradient 
$$\left(\frac{{}^{\circ}F}{100ft}\right)$$
  
 $\times \frac{TVD_{total}(ft)}{100} + 80$ 
EQ. 3.13

BHCT (°F)  
= 
$$80 + \frac{(0.006061 \times (BHST (°F) - 80)) - 10.0915}{1.0 - (TVD_{total}(ft) \times 0.000015052)}$$
 EQ. 3.14

The above equations are used generally for true vertical depth of more than 10000ft. This is clear that by combining both equations the BHCT calculation can be solved just by temperature gradient and TVD parameters.

#### **CHAPTER FOUR**

#### **RESULTS AND DISCUSSION**

#### 4.1. Introduction

In this chapter, the results that achieved from different experimental tests in this work are mentioned and discussed. The results of basic tests such as mixing, rheology, thickening time, free water, compressibility strength, and fluid loss were gathered in NIDC's cement laboratory. Also, the results of foam stability tests are prepared in NIDC's laboratory. The results of foamed cement permeability and porosity were gained in PUT's laboratories. Finally, the results of the CT scan experiment were obtained in TABA medical imaging center laboratory. All of comprehensive results are presented in this chapter.

Tests are conducted in the wellbore conditions (i.e., wellbore pressure and temperature). For best comparison between this new foamed cement invention and other common lightweight cements, the result of different tests for each slurry is reported. Moreover, the basic 118pcf cement tests result at the specific bottom-hole condition (as same as the foamed cement tests condition) are presented in this chapter. Also, new idea for foam cement application in the wellbore cementing industry is investigated. This new lightweight cement is a perfect and ideal cement for Iranian depleted or high fractured or low-pressure-gradient formations and reservoirs such as South Azadegan field.

#### 4.2. Optimum Foam Concentration

The basic slurry for this study at the standard 118pcf class G cement was assumed[45]. Different tests are conducted on the basic and new formulated foamed cement at the same wellbore condition. The 118pcf slurry for Delijan class G cement (43.96 Batch Number) was designed as the following concentration. This is called the net class G cement concentration.

Item	Cement and Additives Name	Concentration (BWOC)	Amount (gr & cc)	Slurry Volume (cc)
1	Class G cement (SG = 3.14)	100%	778	600
2	Distilled Water	-	350	

Table 4.1. Net and basic 118pcf class G slurry cement

The calculations that run with CemCADE software, required to input the data as SLES, cement and DW specific gravity (SG). By calculating for constant 118pcf slurry, different amount of SLES with different concentration is measured. Note that because of fundamentally behavior of SLES as a surfactant and foam agent, the density of slurry was decreased after the mixing process. The measured amount for cement, DW and SLES are reported in Table 4.2.

All of these slurries are prepared in the same manner for certain and accurate bubble distribution in the slurry. The mixing method is the same as that described in section 3.4.1.1 with the difference that is no additive except SLES here. After each mixing, the density and volume of slurries were evaluated and the results are presented in Table 4.3. Also, foamed cement stability analyses are conducted for each slurry. The foam stability analyzing procedure is that the volume reduction of slurries after 2 hours in

the 1-Liter graduated cylinder is evaluated [11]. Stability results are reported in Table 4.3.

Components	Class G Cement	Distilled Water	SLES		
Number	(gr)	(gr)	gr	gal/Sx	
1	779	353	2.08	0.03	
2	779	351	3.6	0.047	
3	779	350	4.03	0.055	
4	778	350	4.84	0.07	
5	778	349	6.32	0.085	
6	778	348	7.8	0.1	

Table 4.2. The foamed cement samples with different foam agent concentration

In the Table 4.3,  $V_1$  is 600mL as basic slurry volume,  $V_2$  is the slurry volume after sample preparation and  $V_3$  is the slurry volume after 2hr for stability measurement. In addition,  $\rho_2$  is the slurry density in pcf after mixing and  $\rho_1$ -  $\rho_2$  shows the density reduction from the basic 118pcf slurry.

Sample Number	<b>V</b> <sub>2</sub> ( <b>cc</b> )	<b>V</b> <sub>3</sub> (cc)	V <sub>2</sub> - V <sub>1</sub> (cc)	$\mathbf{V}_2 - \mathbf{V}_3$ (cc)	ρ <sub>2</sub> (pcf)	ρ <sub>1</sub> - ρ <sub>2</sub> (pcf)
1	690	675	90	15	97	21
2	695	675	95	20	95	23
3	705	690	105	15	92	26
4	735	725	135	10	90	28
5	650	635	50	15	98	20
6	620	610	20	10	100	18

Table 4.3. The foamed cement samples density and volume

From the Tables 4.2-3 and Figures 4.1 and 4.2, this is clear that sample 4 has the best stability and maximum volume enhancement and the minimum density. Therefore, the sample No.4 concentrations and component amounts are the optimum quantities. The optimum foam agent concentration is 0.07 gal/Sx which is 4.84g. This amount of SLES is the key to this research work. Obviously, the Figures 4.1 and 4.2 show the optimum point for slurry density and volume after mixing.



Figure 4.1. Foamed cement density in different SLES concentration



Figure 4.2. Foamed cement volume in different SLES concentration

#### 4.3. Optimum Mixing Procedure

Foamed slurries preparation has different methods by variety innovations. Totally, there are two basic techniques for foamed cement preparation:

- 1. Gas (air or nitrogen) injection with foam generator into the slurry;
- High shear rate mixing, gas (air) adsorption by foaming agents into the slurry and mixing the gas with a liquid.

Each researcher for their own project suggests a new foaming process and, in this thesis, a new optimum foamed cement preparation method is developed. As described in chapter three, the high shear rate method with a particular mixing operation is used for this project and the question is that how this method was achieved?

Three different mixing techniques for optimum method approaching were conducted [42]. These tests are done on the optimum SLES concentration and number 4 slurry components amounts.

Method1: Variable Rotation Speed (The method as described in the last section)

- Place 350gr DW in the mixing container and turn on the mixer at 4000RPM rotation speed.
- Add 778gr class G cement in 15s. Remain mixing at 4000RPM for 35s.
- Pour 4.84gr SLES into the mixture in 10s at 4000RPM. Remain to mix for 35s at 4000RPM.
- Switch mixing mode from fix 4000RPM to variable manual mode.
- Increase the rotation speed from 0 to 12000RPM in one minute.
- Remain to mix at 12000RPM for 35s.

• Turn off the mixer at 12000RPM.

Method2: Rotation Speed Changing Immediately

- Place 350gr DW in the mixing container and turn on the mixer at 4000RPM rotation speed.
- Add 778gr class G cement in 15s. Remain mixing at 4000RPM for 35s.
- Pour 4.84gr SLES into the mixture in 10s at 4000RPM. Remain to mix for 35s at 4000RPM.
- Switch mixing mode from fix 4000RPM to fix 12000RPM mode.
- Remain to mix at 12000RPM for 35s.
- Turn off the mixer at 12000RPM.

Method3: Constant 4000RPM Rotation Speed

- Place 350gr DW in the mixing container and turn on the mixer at 4000RPM rotation speed.
- Add 778gr class G cement in 15s. Remain mixing at 4000RPM for 35s.
- Pour 4.84gr SLES into the mixture in 10s at 4000RPM. Remain to mix for 35s at 4000RPM.
- Turn off the mixer at 4000RPM.

After each method, the secondary volume and density are evaluated. From these results that are presented in Table 4.4, the best and optimum mixing procedure is reached. As shown in Figures 4.3 and 4.4, the optimum foam cement preparation method with SLES foaming agent and current cement composition is Method 1.

Thus, all of the other tests and compositions are approached on the base of optimum foam cement composition and optimum foam cement mixing process (i.e., SLES concentration is 0.07gal/Sx which weight is 4.84gr by variable rotation speed "Method 1" slurry preparation method).

Method	<b>V</b> <sub>2</sub> ( <b>cc</b> )	$V_2 - V_1 (cc)$	$\rho_2$ (pcf)	$\rho_1$ - $\rho_2$ (pcf)
Method 1	735	135	90	28
Method 2	700	100	93	25
Method 3	675	75	98	20

Table 4.4. The effect of different mixing method on foamed cement volume and density



Figure 4.3. Foamed slurry preparation method effect on its volume



Figure 4.4. Foamed slurry preparation method effect on its density

#### 4.4. Foam Tests

Some of the foam properties were gathered in this work by different rheology and stability tests. Foam properties and behavior at different shear rates are necessary for better-foamed cement designing and analyzing. SLES as a foaming agent in this work was used and different tests are conducted on that.

#### 4.4.1. Foam Stability

Variety tests are introduced by different researchers for analyzing the foam stability such as the lifetime of single bubbles, dynamic tests with continuous gas injection, static tests with a column of foam (or foam with water) in a graduated cylinder or pipette. In this research, the static test was studied [50].

A constant 150cc volume of SLES was poured into the mixing container. For the first time, the foaming agent (FA) was mixed at 4000RPM rotation speed and the next time at 12000RPM. This test was conducted for better investigation of shear rate effect on the foaming process. The result as is prepared in Table 4.5, cleared that higher shear rate causes more air entrance and as a result the more FA expansion. The gas fraction of each sample is calculated by the following equation, Also, this is known as foam quality.

$$Foam Gas Fraction = \frac{Secondary Volume - Primary Volume}{Secondary Volume} \times 100 \qquad EQ. 4.1$$

Rotation Speed (RPM)	Primary Volume (cc)	Secondary Volume (cc)	Gas (Air) Fraction (%)	Foam Density (gr/cm³)	ρ <sub>foam</sub> / ρFA
4000	150	590	74.57	0.275	0.25
12000	150	800	81.25	0.205	0.186

Table 4.5. Foam properties in two 4000 and 12000RPM rotation speeds

In the Table 4.5, the foam density (after mixing) to foam agent density (SLES before mixing) ratio as the density reduction ratio is presented.

Foam stability was conducted in a static condition. SLES was mixed at 12000RPM for one minute, after that, the generated foam was poured into the 1-Liter graduated cylinder as Figure 4.5 and wait for 2 hours. Before and after 2 hours of static condition, the foam volume is reported. The volume reduction ( $V_r$ ) illustrates the foam quality as the following rules of thumb [11]:

- $V_r$  less than 10% of primary volume  $\rightarrow$  Perfect foam stability
- $V_r$  between 10 to 20% of primary volume  $\rightarrow$  Good foam stability
- $V_r$  between 20 to 25% of primary volume  $\rightarrow$  Moderate foam stability
- $V_r$  more than 25% of primary volume  $\rightarrow$  Weak foam stability



Figure 4.5. Schematic of stability test in a graduated cylinder and an image of SLES's foam structure [11]

Table 4.6 and Figure 4.6 present the foam volume during a 2-hours' time period and it is clear that the foam stability is perfect.

Elapsed Time (min)	0	30	60	90	105	120			
Residual Foam Volume (cc)	800	793	780	762	744	723			
Foom Stability	$800 - 723 = 77 \rightarrow (77/800) \times 100 = 9.625\%$								
Foam Stability	9.62	5% < 10	$0\% \rightarrow Pr$	efect Fo	am Stab	ility			

Table 4.6. SLES foam stability



Figure 4.6. SLES foam volume versus time for foam stability measurement

# 4.4.2. Foam Rheology

Rheology of SLES foam generated by 12000RPM-high-shear-rate was investigated. Also, YP and PV of this foam were measured as in Table 4.7.

Rotation Speed (RPM)	600	300	200	100	60	30	20	10	6	3	2	1
Dial Reading	190	134	105	75	58	42	35	24	19	14	11	6
PV (cP)	88.5											
<b>YP</b> (lb <sub>f</sub> /100ft <sup>2</sup> )						45.	5					

Table 4.7. Rheology parameters of SLES's foam



Figure 4.7. SLES's foam rheology which measured by VG meter

The Figure 4.7 shows the rheology of SLES's foam that is generated at 12000RPM. Its behavior and stability show well properties of a foam that is perfect for the foamed cement formulation and designing as current research. Foam rheology, stability, optimum concentration and mixing procedure measurements are showed obviously that these foam properties are very appropriate for this research.

#### 4.5. Other Cements Composition

In this work for better-foamed cement properties analyzing, a comparison between FBJ and other lightweight cements and net class G cement was developed. The composition of net class G cement is described in section 4.2 and for lightweight cements are as follow. All of these lightweight cements were designed for industrial application at wellbore cement job in South Azadegan field by NIDC's cementing engineers.

#### **4.5.1.** Composition 1 (C1) ( $\rho$ = 90pcf)

This cement is known as one of the most lightweight cements that NIDC uses in its projects in low-pressure layers cementing. The composition of this slurry is reported in Table 4.8. This is known as bentonitic cement.

Item	Cement and Additives Name	Concentration (BWOC)	Amount (gr & cc)		
1	Class G cement	100%	327		
2	P-Water	-	427		
3	Micro Silica	18%	59		
4	OR12	0.03%	0.1		
5	OMicro Block	1 gal/Sx	33		
6	OGas Block	0.7 gal/Sx	18		
7	TA47	0.015 gal/Sx	0.62		

Table 4.4.8. The composition of lightweight cement 1 (C1)

# 4.5.2. Composition 2 (C2) ( $\rho$ = 95 pcf)

Composition 2 was conducted for layers which higher CS is required for their cement.

Item	Cement and Additives Name	Concentration (BWOC)	Amount (gr & cc)	
1	Class G cement	100%	408	
2	P-Water	-	427	
3	Micro Silica	12%	49	
4	OR5	0.25%	1.02	
5	OCFR4	0.05%	0.2	
6	OMicro Block	0.55 gal/Sx	22	
7	OGas Block	0.15 gal/Sx	5	
8	TA47	0.02 gal/Sx	0.62	

Table 4.9. The composition of lightweight cement 2 (C2)

#### 4.5.3. Composition 3 (C3) ( $\rho$ = 88 pcf)

The composition 3 is known as Olightweight cement and is one of the most expensive lightweight cements with high enough CS.

Item	Cement and Additives Name	ent and AdditivesConcentrationName(BWOC)	
1	Class G cement	100%	418
2	P-Water	-	265
3	Olightweight	32%	134
4	JFLC320	0.5%	2.09
5	OCFR4	0.15%	0.63
6	OMicro Block	0.2 gal/Sx	8
7	OGas Block	0.5 gal/Sx	17
8	TA47	0.02 gal/Sx	0.63

Table 4.10. The composition of lightweight cement 3 (C3)

## 4.6. Tests on The Optimum Concentration of SLES

The meaning of the optimum point is the foamed slurry with an optimum concentration of SLES (0.07 gal/Sx). In this section, the comparison between foam cement and net class G cement is presented. This optimum concentration will be called OF in this thesis.

#### 4.6.1. Rheology Tests

Rheology test of optimum concentration of SLES in cement, that is gained in section 4.2, and its comparison with net class G cement rheology illustrates the surprising results. These tests are approached in the room and bottom-hole circulating temperature condition. BHCT is assumed 170°F. The results for both OF and class G rheology are prepared in Table 4.11.

Slurry Type	C	<b>)F</b>	Ne	t G
Temperature Rotation Speed (RPM)	Room Temp.	BHCT = 170 °F	Room Temp.	BHCT = 170 °F
300	90	92	81	125
200	80	86	74	112
100	65	73	62	95
60	58	66	54	82
30	50	55	48	70
6	20	11	24	30
3	14	9	11	25
PV (cP)	37.5	28.5	28.5	45
YP (lb <sub>f</sub> /100ft <sup>2</sup> )	52.5	63.5	52.5	80

Table 4.11. Dial reading, PV and YP of OF and net class G cements in room temperature and BHCT

Figures in 4.8-9 show the rheology of OF and net class G samples under room and bottom-hole conditions. From these Figures and PV and YP magnitudes in the Table above, can be concluded that the foam cement sample (OF)'s rheology contrariwise the common slurries such as net class G, will become better in wellbore condition and BHCT. This is because of bubbles expansion and ball-bearing effect of these little bubbles in the slurry. The bubbles reduce the friction of the slurry and in high temperature, they will be expanded and their effects efficiency will be increased. The effect of temperature in bubbles expansion is more than the pressure in their constriction. Thus, the effect of temperature on foamed cements behavior is more than pressure. The OF's PV was decreased and this is causing the better rheology of foamed slurry. The water of net class G slurry (such as other common slurries) evaporates in high temperatures and this is causing the higher friction and higher plastic viscosity and worse rheology. The expansion of bubbles reduces the density of OF from 90pcf to 85pcf, therefore the hydraulic pressure of slurry in the wellbore will be decreased.



Figure 4.8. Room condition rheology of OF and net class G samples



Figure 4.9. The 170°F-temperature rheology of OF and net class G samples

# 4.6.2. Free Water Test

Because of OF's foamy structure and bubbles distribution in the bulk of slurry, the bubbles surface tension helps the water trapping. Thus, the most of foam cements such as OF, have 0cc free water (i.e., they don't have free water). However, the net class G slurry has 6cc, near 10% of slurry volume, free water [42].

#### **4.6.3.** Compressive Strength Tests

the foamed cements CS is lower than net cements CS and this is caused by the porous structure of foamed cement. Figures 4.11 and 4.12 illustrate the CS graph of OF and net class G cements respectively, which are drawn by the UCA system. All of UCA tests were conducted at 4000psi bottom-hole static pressure (BHSP). UCA was run for OF for two BHST, 170°F and 210°F and their graphs are presented in Figures 4.10 and 4.11. By these tests, the effect of temperature on the foamed cements became obvious. The CS of OF at 170°F is 1311psi and by increasing the temperature to 210°F, CS is increased to 1371psi. Therefore, the effect of temperature rising on foamed cements CS increasing is not so much. Net class G cement has 3506psi as its CS at 210°F BHST. This is clear that why net cements are used in tail cement jobs. Pay attention that the density of net class G is 118+ pcf at 210°F and OF is 85pcf at the same temperature and for the reason that density effects directly on the CS, this parameter comparison of these two slurries is not so scientific. Thus, for a better comparison on the CS parameter, all of lightweight cements (C1, C2 and C3) and OF will be compared in the future sections of current study.

#### **4.6.4.** Thickening Time Tests

TT is measured at 170°F BHCT and 4000psi pressure. These tests were conducted for both net class G and OF slurries. The results of these experiments are prepared in Figures 4.13 and 4.14. In addition, these Figures show that the SLES as a foaming agent in the OF composition behaved as an accelerator. It reduced the thickening time of net class G slurry from 88 minutes to 60 minutes for OF slurry. This is clear that 60min for TT of each slurry is not operational for industrial applications.



Figure 4.10. Compressive strength graph of OF slurry sample at 170°F and 4000psi



Figure 4.11. Compressive strength graph of OF slurry sample at 210°F and 4000psi



Figure 4.12. Compressive strength graph of net class G slurry sample at 210°F and 4000psi



Figure 4.13. Thickening Time graph of OF slurry sample at 170°F and 4000psi



Figure 4.14. Thickening Time graph of net class G slurry sample at 170°F and 4000psi

Thickening time graphs (Figures 4.13 and 4.14) illustrated that another foamed cement slurry formulation should be investigated and designed. One-hour TT is a short time for any well cementing job, therefore a retarder must be added to the slurry. Different retarding additives have various effects and the most commons of them are OR5, OR12, and JR120 which were used for best formulation approach.

#### 4.7. Retarders Effects on The OF Foamed Cement

Cement designing is a complex trend to reach the best slurry and setcement properties. In this way, by changing one property, other properties are altered. Thickening time increasing needs some percent retarders and as a result, other properties might be changed. In this section, the trend of best slurry TT and properties approaching are discussed.

The procedure of OF slurry preparation with a retarder is like as something that was described in section 3.4.1.1 and by this difference that here the boric acid is not added to the mixture. Also, instead of 0.1% BWOC JR120, other retarders (OF+OR5, OF+OR12, OF+JR120) were added to the slurry. The concentration or amount of retarder for each foamed cement design was assumed 0.1% BWOC (0.78gr).

As this is illustrated in Table 4.12, all of the retarders were compatible with the OF composition in the surface condition, however, some reactions occurred in high temperature (170°F). The reaction was activated by temperature increasing and that caused to the rheology test was not completed. For better analyzing, the surface rheology and under temperature rheology experiments were conducted and the results are presented in the Table 4.12. All of the retarders as sodium/calcium lignosulfonates are incompatible with SLES as a foaming agent in high temperature and this is cleared that some reactions are activated by heating (temperature increasing) between these materials.

Slurry + Retarder	0	F	OF+	OR5	OF+	OR12	OF+J	R120
Temperature Rotation Speed (RPM)	R.T	170 (°F)	R.T	170 (°F)	R.T	170 (°F)	R.T	170 (°F)
300	90	92	135	error	125	error	25	error
200	80	86	112	error	110	error	20	error
100	65	73	95	error	96	error	12	error
60	58	66	86	error	89	error	10	error
30	50	55	78	error	80	error	7	error
6	20	11	26	error	30	error	5	error
3	14	9	15	error	17	error	4	error
PV (cP)	37.5	28.5	60	error	43.5	error	19.5	error
YP (lb <sub>f</sub> /100ft <sup>2</sup> )	52.5	63.5	75	error	81.5	error	5.5	error

Table 4.12. The effect of different retarders on the OF rheology parameters (dial reading, PV and YP) in room and wellbore conditions (170°F)

By these results in Table 4.12, can be concluded that the reaction between different retarders and sodium lauryl ether sulphate is an endothermal reaction and this was caused that in wellbore condition the DC of the slurry after near 20 minutes immediately increased from 1.6 to 7. Thus, the test must be stopped before the 30min standard time for the rheology test. Slurry became thicker and its thickening time was less than 30min as shown in Figure 4.15, OF+OR5 slurry's TT is about 21min. Retarders not only did not act as a retarder but also acted as an accelerator in OF slurry composition.


Figure 4.15. Thickening Time graph of OF slurry sample with OR5 retarder at 170°F and 4000psi

Table 4.12 and Figures 4.16, 4.17 illustrate that the retarders except JR120 increase both PV and YP and the rheology become worse. This is because of OR5 and OR12 chemical formulas that their calcium ions displace the sodium ions of SLES in the structure of slurry and this movement and displacement will become more severe in higher temperatures. Nevertheless, JR120 has sodium ions as SLES and effects very well as a retarder in the room temperature in OF slurry.





Figure 4.16. Retarders effects on the slurry plastic viscosity at surface condition

Figure 4.17. Retarders effects on the slurry yield point at surface condition

Figure 4.18 pinpoints the rheology of OF+Retarders slurries. This is noticeable that in the surface condition, OR5 and OR12 cause the rheology to become worse and JR120 makes it better. However, under wellbore condition, because of the short thickening time of these samples, their rheology parameters are not available.



Figure 4.18. The rheology of OF with different retarders in surface condition

By temperature raising to the wellbore condition, the effects of JR120 was altered to an accelerator in this composition. Its organic acid made a reaction with SLES (as an alkaline material) as a result that a reaction between SLES and JR120 makes the slurry thicker and eliminates the JR120 retarding effect. The JR120 effects alternation occurred near 145°F temperature. Thus, the DC of slurry in the atmospheric consistometer reached 7 (Thickening time's DC) before the temperature becomes 170°F in 30 min.

As described before, because of foamy structures of foamed slurry samples, none of the OF+Retarder samples don't have settling and free water and the magnitude of free water for these slurries is zero. Permeability and other tests that were conducted for these samples to approach the best foam cement formulation analyzing will be discussed in the future sections.

### **4.8.** Final Formulation (FBJ Design)

After the failure in the slurry designing for its acceptable thickening time, an idea was constructed. This innovation is the boric acid addition to the slurry. Boric acid helps the retarder to operate as its function. By adding 0.1% BWOC (0.78g) as a procedure that is described in section 3.4.1.1, SLES makes a reaction with boric acid and JR120 acts normally.

As explained before, the JR120 acts very well in the room condition but by temperature increasing to the wellbore condition, the organic acid of JR120 made a reaction with SLES. Now, the boric acid reacts with SLES as an alkaline solution in high temperatures and JR120 easily distributes cement components and retards the thickening time of slurry. In this mechanism, boric acid helps retarder effects in the slurry. This composition of cement will be called FBJ in this study. Boric acid in the mixing procedure increased the foaming process efficiency and enhanced the secondary slurry volume and as a result that reduced the final density of foamed slurry by increasing the foam quality of cement. The density and volume of FBJ and different OF+Retarders samples are presented in Table 4.13. The FBJ has the lowest density with maximum slurry volume and the most stable foamed cement between different foamed cement samples in the current project. V<sub>1</sub> and  $\rho_1$  are volume and density of slurry respectively before mixing. V<sub>3</sub> is the slurry volume after 2hr for foamed cement stability measurement. V<sub>2</sub> and  $\rho_2$  are volume and density of foamed cement after the mixing procedure. V<sub>1</sub> and  $\rho_1$  are 600cc and 118pcf for the basic class G slurry. The foam quality of foamed cements is calculated by equation 4.2:

Foamed Cements Quality (%)

$$= \frac{Foamed \ slurry \ volume - Basic \ slurry \ volume}{Foamed \ cement \ volume} \times 100$$
 EQ. 4.2

Table 4.13 and Figures 4.19 to 4.22 are the results which illustrate that FBJ has the primary qualifications as a new foamed cement.

Slurry Samples	V2 (cc)	V3 (cc)	V <sub>2</sub> -V <sub>3</sub> (cc)	V <sub>2</sub> -V <sub>1</sub> (cc)	ρ2 (pcf)	ρ1- ρ2 (pcf)	Foamed Cement Quality (%)
OF+OR5	735	720	15	135	85	33	18.36
OF+OR12	740	725	15	140	86	32	18.92
OF+JR120	870	860	10	270	86	32	31.03
FBJ (OF+JR120+Boric Acid)	925	920	5	325	75	43	35.13

Table 4.13. Volume and density of different foamed slurry samples



Figure 4.19. The Slurry volume of different foam cement samples after mixing (V<sub>2</sub>) and after the 2hr static condition in the graduated cylinder (V<sub>3</sub>)

The photographic of different samples stability test process are presented in Figure 4.20. Figures 4.21 and 4.22 show the density and foamed cement quality respectively for different foamed slurry samples.



Figure 4.20. The image of foamed cement samples stability tests, the samples from left to right: OF+OR5, OF+OR12, OF+JR120, FBJ



Figure 4.21. The density of different foamed cement samples



Figure 4.22. Foam cement quality for different foamed cement samples

### 4.8.1. Rheology Test

The rheology test was conducted for FBJ foamed cement and its results are presented in Table 4.14. From this Table and Figure 4.23, can be concluded that for FBJ foamed Cement the rheology parameters are better than OF and net class G cements parameter. This is because of more amount of bubbles are in the structure of slurry and their distribution is homogeneous and as a result, its movement behavior will become better by more ball-bearing-effect [13]. Unlike other OF+Retarders foamed cement samples, FBJ effects at the wellbore condition as a perfect foamed cement with acceptable rheology features which caused by boric acid being in its slurry composition.

Table 4.14. FBJ foamed cement rheology parameters in surface and high-temperature condition

Rotation Speed (RPM)	300	200	100	60		30	6	3
Dial Reading (R.T)	70	57	44	37		33	20	12
Dial Reading (170°F)	65	53	42	36		33	17	8
PV (cP)	39 (R.T)				1	34.5	(170°F	')
YP (lb <sub>f</sub> /100ft <sup>2</sup> )	31 (R.T)					30.5	(170°F	')



Figure 4.23. The rheology comparison between FBJ, OF and net class G slurries in surface and wellbore condition

As shown in Figure 4.24 the plastic viscosity of foamed cements is decreased by temperature increasing and inversely, the conventional slurries will become thicker, their friction will be increased and as a result, the PV will be increased. However, for most slurries, either common slurry or foamed cement, raising the temperature causes the yield point increasing. FBJ has a unique feature between other foamed cement in the current thesis and this is that its yield point such as its plastic viscosity is decreased by increasing the temperature. These phenomena are because of FBJ's high foamed cement quality and high foams stability in the wellbore condition. The OF+Retarders samples in high temperature were reached to their thickening time and for this reason, the YP and PV of these cements are not available in wellbore condition as reports in the Figure 4.24.



Figure 4.24. PV and YP of different slurries in room temperature and 170°F

#### 4.8.2. Free Water and Settling of FBJ

The free water of FBJ such as other foamed cements in this work is zero. The foamy structure of this cement, the surface tension of air bubbles and water trapping in the cement structure (before hydration) are some reasons for free water to become zero. In addition, after two hours static condition for slurry in the graduated cylinder, no settling was observed. The results picture is prepared in Figure 4.25.



Figure 4.25. The photographic of free water and settling tests results on the FBJ slurry

### 4.8.3. The Thickening Time of FBJ

As described before, in the FBJ composition by making the reaction between SLES and boric acid, the JR120 can affects freely without occupying by foam agent (SLES). This was anticipated that JR120 retards the thickening time. The base of expectations the TT of foamed slurry increased from 20min (for OF+JR120) or 60min (for OF) to 175min for FBJ sample. Near 3 hours TT is very ideal for any operation in the cementing job. The graph of FBJ thickening time at the 170°F BHCT and 4000psi pressure is presented in the Figure 4.26. However, the thickening time of other lightweight slurries which are investigated in section 4.5 is more than FBJ as a foamed cement. TT of compositions 1,2 and 3 are 354, 401 and 217 minutes respectively. The TT graphs of these lightweight slurries are shown in Figures 4.27 to 4.29. Table 4.15 lists the thickening time of different samples for better comparison.

Composition	Cement Type	Thickening Time (min)
FBJ	Foamed Cement	175
C1	Conventional Lightweight Cement	354
C2	Conventional Lightweight Cement	401
C3	Conventional Lightweight Cement	217

Table 4.15. Different slurry samples thickening time

# 4.8.4. The Compressive Strength of FBJ

Generally, the retarders don't affect the cements compressive strength [51]. In this composition, JR120 and the retarder aide (boric acid) didn't affect the CS so much as expectations.



Figure 4.26. The thickening time graph for FBJ foam cement at 170°F and 4000psi



Figure 4.27. The thickening time graph of C1 lightweight cement at 170°F and 4000psi



Figure 4.28. The thickening time graph of C2 lightweight cement at 170°F and 4000psi



Figure 4.29. The thickening time graph of C3 lightweight cement at 170°F and 4000psi

The compressive strength of FBJ which is measured at 210°F BHST and 4000psi BHSP is 1317psi. The graph of FBJ's CS is presented in Figure 4.30. For better comparison, the CS graphs of other lightweight cements are presented in Figures 4.30 to 4.32. One quantity is defined in this thesis as CDA ratio, which equation is 4.3:

$$CDA\left(\frac{psi}{pcf}\right) = \frac{Compressive Strength of Set Cement (psi)}{Density of Slurry (pcf)}$$
 EQ. 4.3

This is clear that if the amount of CDA ratio is greater, the cement has a better property which by lower density tolerates the higher compressive strength and pressure. The magnitude of CDA for different lightweight cements compared to FBJ as shown in Table 4.16.

Sample	Density (pcf)	Compressive Strength (psi)	CDA Ratio (psi/pcf)	Cement Type
FBJ	75	1317	17.56	Foamed cement
C1	90	318	3.54	Conventional cement
C2	95	987.5	10.39	Conventional cement
C3	88	2103	23.90	Conventional cement

Table 4.16. The CDA ratio for different lightweight cements in comparison to FBJ

The CS of FBJ, C1, and C3 samples was measured by UCA and for C2 the Crush test was conducted. The quantity of CDA ratio for a foamed cement as 17.56 psi/pcf is acceptable definitely. The reason is that the foamed cements contain gas in addition to solids and liquids in their structure. Thus, set foamed cements have porous media and as a result, their CS should be lower than some common lightweight cements. Note that the lightweight cements which have greater CS than FBJ, such as C3, is one of the most expensive slurries compositions in the cementing jobs. The price of lightweight cements will be investigated in the last section of this chapter.



Figure 4.30. The Compressive Strength graph of FBJ at 210°F and 4000psi



Figure 4.31. The Compressive Strength graph of composition 1 (C1) at 210°F and 4000psi



Figure 4.32. The Compressive Strength graph of composition 3 (C3) at 210°F and 4000psi

## 4.8.5. The Fluid Loss of FBJ

Foamed cements due to their structure and composition, don't have a fluid loss. Their foamy structure helps the slurry loss reduction to the formations and reservoirs. Although, some tests were conducted on FBJ. The nitrogen was transferred from slurry to the graduated cylinder and no filtrate accumulated in the cylinder. Thus, the test condition was "No-Control". Usually, the fluid loss test is not conducted for foam cement by foamed cement quality greater than 10%. In higher qualities the particular apparatuses are necessary. The Figure 4.33 illustrates the FBJ cement cake after the fluid loss test. The filtrate of FBJ was recorded as 0cc/30min.



Figure 4.33. FBJ cement's cake after fluid loss test

### 4.9. Permeability and Porosity Tests

Set foamed cements have a porous media. One of the properties of a good cement is complete zonal isolation. The cement must prevent the fluids flow to the wellbore. For conventional cements the permeability test is not conducted usually. However, the permeability test for foamed cements is very critical and necessary. The foamed cement should have the porous structure with the lowest permeability as possible. In addition, the porous structure of a foamed cement must be saved in the wellbore condition (high-pressure high temperature (HPHT)). Different permeability tests for different foamed cement samples in this literature were managed and their results are investigated.

The calculated permeabilities are analyzed with porosity magnitude for each sample synchronously. By a combination of these two tests results, some important data were gained. The results of permeability tests are reported in Table 4.17. The differential pressure at this test was fixed at 100psi. The viscosity of distilled water at 24°C is 0.9107cP. Also, Figure 4.34 shows the water permeability of different foamed cements in comparison to net class G cement.



Figure 4.34. Water permeability of net class G cement and different foamed cement samples

Sample	Q (mL/sec)	K (mD)	Sample's Picture
Net class G	0	0	0
OF	0.08/361.1	0.0113	
OF+OR5	0.005/312.79	0.00085	
OF+OR12	0.09/349.13	0.0143	
OF+JR120	0.07/183.84	0.0202	
FBJ	0.06/298.27	0.0109	

Table 4.17. The permeability of net class G and other foamed cements samples

By comparison of the foamed cements samples can be concluded that the FBJ sample has the lowest permeability after OF+OR5 sample. This is caused by the better compound in the cement structure. The boric acid made a reaction with SLES and JR120 affected freely and cement structure prepared strongly. In the other foamed cements samples, the reaction between JR120 and SLES disturbs the strong and impermeable structure of set-cement and this is due to that retarder don't distribute the cement particles very well in the mixture at these conditions.

The porosity results of different samples are presented in the Table 4.18 and Figure 4.35. Base on participation and foamed slurry quality results, the FBJ is the most porous cement between all this thesis foamed cements. As described in last paragraph, FBJ has one of the least permeabilities. Therefore, one surprising result is achieved and this is that the porous media of FBJ as a foamed cement is not interconnected. These isolate pores in this foam cement make a structure such a pumice rock. Thus, FBJ has the minimal necessity of an ideal foamed cement and that is a porous media with a minimum permeability as possible. In the Table below the V<sub>b</sub> and V<sub>p</sub> are bulk and pores volume respectively in the cement plugs.

Sample	Dimensions (cm)		$V_{\rm h}$ (cm <sup>3</sup> )	$V_{\rm p}$ (cm <sup>3</sup> )	Porosity,
	Diameter Length			P	φ (%)
Net class G	2.850	2.470	15.75	0.96	6.09
OF	2.922	2.560	17.17	9.17	53.41
OF+OR5	2.856	2.550	16.33	3.48	21.31
OF+OR12	2.801	2.551	15.71	8.92	56.77
OF+JR120	2.804	2.452	15.14	10.87	71.79
FBJ	2.819	2.515	15.69	11.46	73.04

Table 4.18. The porosity of net class G and other foamed cements samples

For better investigation of foamed cements porous media and ensuring the porosity and permeability calculations, the microscopic photography and CT Scan analyzing were conducted. The results of these tests are reported in the next section.



Figure 4.35. The porosity of Different foamed cement samples in comparison to net class G cement

## 4.10. CT Scan and Microscopic Photography Analyzing

Different samples of foamed cements and net class G cement are prepared. Different cement and foam tests were done on these samples. These samples prepared in surface or HPHT conditions. The Figure 4.36 shows the some of them.



Figure 4.36. Some of the cement samples which prepared in surface or HPHT (210°F and 4000psi) conditions

For best analyzing of each sample and their pore structures, the microscopic photography was used for samples surface investigations and for the interior analyzing of each sample the computed tomography scanning was conducted. Figures 4.37 to 4.42 present the microscopic photography of different foamed cement samples. These images are some proofs for bubble size and their connections to each other. The magnification of these Figures is in the scale of 1mm to 0.5cm length.



Figure 4.37. The image of OF foamed cement sample and its microscopic photography



Figure 4.38. The image of net class G cement sample and its microscopic photography



Figure 4.39. The image of OF+OR5 foamed cement sample and its microscopic photography



Figure 4.40. The image of OF+OR12 foamed cement sample and its microscopic photography



Figure 4.41. The image of OF+JR120 foamed cement sample and its microscopic photography



Figure 4.42. The image of FBJ foamed cement sample and its microscopic photography

As shown in Figures 4.37 to 4.42 the bubbles of OF, FBJ and OF+OR5 samples are isolated. The size of these samples bubble is small and distributed homogeneity in the cement. Moreover, other cement

samples have large interconnected pores that cannot isolate a specific region of a formation. Therefore, other samples such as OF+OR12 and OF+JR120 are not appropriate for cementing jobs in a drilling operation.

Computed tomography scan (CT Scan) is used for best investigation on the foamy structure of foamed cement samples. By this technique, the pore distribution and their connections are cleared. Different CT scan analyses and image processing was conducted. Two-dimensional (2D) and three-dimensional (3D) rendering process were developed for each sample for best results achievement. The DICOM files that are gathered by CT scan test, was inspected by Ginkgo CADx Pro software. The results of this experiment are presented in the following Figures. As shown in figures 4.43 to 4.48 the blue and yellow colors are illustrated cement and black sections pinpoint the pores in the CT Scan test. Each of these pictures is created in the CT scanning with a cross-section width of 0.512 to 0.625 mm. The 3D volume rendering for all of these samples are presented in Table 4.19.

From Figures 4.43 to 4.48 can be answered this question that why the FBJ or OF samples with high porosities have low permeabilities? The pores in these cements are isolated and distributed completely in the foamed cement. By expectations, the net class G cement doesn't have so many pores and voids. However, in other samples, such as OF+JR120 the permeable connections between pores are clearly displayed. In addition, 3D volume rendering illustrates that how much are the foamed cements porous and by this method, the interior porous media for each sample became clear observationally [26].

For best investigation on the foamed cement stability in the HPHT condition, the samples of UCA test are analyzed by computed tomography scanning. The wellbore condition was assumed 210°F and 4000psi. The surprising results were gained by this test. Three different samples were measured by CT scan and their properties are presented in Table 4.20.

Patient: CEMENT Patient ID: 96/01/21-	A	? ? Kg ? m. 20180410 (0008)0030) Modality: CT
		R
Dimensions: 512 x 512 WL: 3979.35/3244.68 Slice: 1 of 1 Volume: 396 Slice thickness: 0.625000 mm. Slice location: - 291.000 Voxel: 0.292969 x 0.292969 x 1 mm.	P	20180410 200125 Made with Ginkgo CADx Pro

Figure 4.43. The CT scan processed image of net class G cement sample

Patient: CEMENT 2 Patient ID: 123456	A	U 20180730 ? Kg ? m. 20180730 (0008(0030) Modaliby CT
L Dimensions: 512x 512 WL: 940.67/1249.15 Slice: 1 of 1 Volume: 90		wouldanity. c i
Slice thickness: 0.5078120232 mm. Slice location: 242.2814636 Voxel: 0.507813x 0.507813x 1 mm.	Р	20180730214351 Made with Ginkgo CADx Pro

Figure 4.44. The CT scan processed image of OF foamed cement sample



Figure 4.45. The CT scan processed image of OF+OR5 foamed cement sample

Patient: CEMENT Patient ID: 96/01/21-	A	? ? Kg ? m. 20180410 {0008 0030} Modality: CT
L Dimensions: 512x 512 WL: 321 60/1235.04 Slice: 1 of 1 Volume: 110 Slice: thisknooc: 0.626000 mm		R
Slice location: -118.500 Voxel: 0,292969 x 0.292969 x 1 mm.	P	20180410 200125 Made with Ginkgo CADx Pro

Figure 4.46. The CT scan processed image of OF+OR12 foamed cement sample

Patient: CEMENT Patient ID: 96/01/21-	A	? ?Kg?m. 20180410 (0008 0030)
L Dimensions: 512x 512		Modality: CT
WL: 813.28/1430.12 Slice:1 of1		
Volume: 306 Slice thickness: 0.625000 mm,		
Slice location:-241.000 Voxel: 0.292969x 0.292969x 1 mm.	P	20180410 200125 Made with Ginkgo CADx Pro

Figure 4.47. The CT scan processed image of OF+JR120 foamed cement sample

Patient CEMENT 2 Patient ID: 1 2 3 4 5 6	Ä	U 20180730 ? Kg?m. 20180730 (0008(0030) Modality: CT
L Dimensions: 512x 512		R
Slice: 1 of 1		
Volume: 925 Slice thickness: 0.5078120232 mm. Slice location: - 279.5683899 Voxel: 0.507813 x 0.507813 x 1 mm.	P	20180730214351 Made with Ginkgo CADx Pro

Figure 4.48. The CT scan processed image of FBJ foamed cement sample

CMT	3D volume Rendering	CMT	3D volume Rendering
Sample	Processed Image	Sample	Processed Image
<b>Net</b> <b>Class G</b> φ = 6.09% K = 0 mD	30- Example         Chean 30- 1000 H (2010)         Chean 30- 2010         Chean 30- 2010         Chean 30- 2010           30- 1000 H (2010)         Chean 30- 2010         Chean 30- 2010         Chean 30- 2010         Chean 30- 2010         Chean 30- 2010           80- 50         Chean 30- 30- 30- 30- 30- 30- 30- 30- 30- 30-	<b>OF</b> + <b>OR12</b> φ = 56.77% K=0.0143 mD	30         AS         CONINT&           54         TADA NEXCAL MARKEN PTTCS           55         Book         France           100         France         France           100
<b>OF</b> φ = 53.41% K=0.0113 mD	ASR CARE CARE CARE CARE CARE CARE CARE CAR	<b>OF</b> + <b>JR120</b> φ = 71.79% K=0.0202 mD	AB LAAA NEDISAL INA KOOSTITU BOG VALUE ARMONTRY BOOV RESN SOOV RESN TABA NEDISAL INA KOOSTITU ALAA NEDISAL INA KOOSTITU AL
<b>OF</b> + <b>OR5</b> φ = 21.31% K=0.00085 mD	25         35         CEMENTS           36         128.80         128.80           36         60.41         128.90           36         20.41         128.90           36         20.41         128.90           36         20.41         128.90           36         20.41         10.01           36         20.41         10.01           37         20.41         10.01           38         20.41         10.01           4         8         9           4         8         9           6         0.05.2         0.015.2           6.21.010.050-1         0.015.2         0.015.2           6.21.010.050-1         0.015.2         0.015.2           6.21.010.050-1         0.015.2         0.015.2	<b>FBJ</b> φ = 73.04% K=0.0109 mD	St.         AS         CAS         CAS <thcas< th="">         CAS         <thcas< th=""> <thcas< th=""> <thcas< th=""></thcas<></thcas<></thcas<></thcas<>

Table 4.19. The processed image of 3D volume rendering by CT scan for different cements

This is obviously showed in Table 4.19 that by the same cement plugs dimensions for net class G, OF and FBJ samples, the weight of each of them is different. Thus, by combining these results and Figures 4.48 to 4.50

can be concluded that the porous media are saved in the HPHT condition. Although, the pores became smaller and as a result, the porosity decreased. Through to small dimensions of pores in these samples, they are pinpointed by small black dots in the yellow cement in the CT scan processed image. Hence, the bubbles of foamed cement were saved in the HPHT condition. Note that the line that is recognizable in the net class G cement sample is caused unfortunately by cracking during plugs transfer from cement laboratory to image processing center.

Cement	Dimensions (cm)		Mass	Sample Photography
Sample	Diameter	Length	(gr)	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~
Net class G	6.78	5.90	311	
OF	6.78	5.90	234	
FBJ	6.78	5.90	196	

Table 4.20. Cement samples properties after HPHT condition (4000 psi and 210°F)

FBJ has the lowest mass in the same dimension to other samples and this a reason for bubbles savage in the set-cement structure. This hypothesis is proven by following CT scan processed images.



Figure 4.49. The CT scan image processed of net class G cement after HPHT conditioning (4000psi and 210°F)


Figure 4.50. The CT scan image processed of FBJ foamed cement after HPHT conditioning (4000psi and 210°F)



Figure 4.51. The CT scan image processed of OF foamed cement after HPHT conditioning (4000psi and 210°F)

#### **CHAPTER FIVE**

## CONCLUSIONS AND RECOMMENDATIONS

The main goal of this project is the new foamed cement designing by experimental methods for casing and liner cementing jobs. The formulation of foamed cements that are used in the drilling industry belongs to some foreign companies exclusively such as Schamberger or Halliburton. In this thesis, some idea and innovations in the experiments and materials were applied and the best formulation and mixing procedure were chosen. Different and various tests were conducted for the best formulated foamed cement (FBJ) and other slurry samples. Rheology, density, foam stability, fluid loss, mixer procedure, thickening time, foamed cement stability and compressive strength experiments were operated at the NIDC cement laboratory. The permeability and porosity tests and microscopic photography were conducted in the PUT cement, petroleum, and material laboratories. The CT scan investigation and image processing were analyzed in the TABA medical imaging center. From this study the following conclusions and recommendations are made.

### **5.1.** Conclusions

1. The 0.07gal/Sx or 4.84gr SLES concentration in the slurry cement is an optimum concentration with maximum volume increasing and density reduction.

- 2. The method 1 (Variable Rotation Speed) has the most efficiency in the mixing procedure for foamed cement preparation in high shear rate.
- 3. The foam that produced in high shear rate from sodium lauryl ether sulphate (SLES) as a foaming agent, has perfect stability and compatibility in the slurries.
- 4. The plastic viscosity and yield point of FBJ (the best and optimum designed foamed cement) are decreased by temperature increasing, whereas, for conventional cements, these parameters are increased in wellbore condition. Generally, the FBJ rheology, contrary to common slurries, becomes better in wellbore condition.
- 5. The free water of FBJ is zero as like as common foamed cements.
- The compressive strength of FBJ to its density (CDA ratio) is one of the highest ratios between common lightweight cements. The CS of FBJ with 75pcf density (as an ultra-lightweight cement), at 210°F BHST and 4000psi BHSP is 1317psi.
- The fluid loss of FBJ due to its foamy structure is recorded 0mL/30min.
- 8. SLES as a foam agent is not compatible with retarders, such as OR5, OR12, and JR120, at high temperatures and makes a reaction with them. The reaction rate is increased by temperature rising. The solution to this problem is the boric acid adding. JR120 affects very well in the FBJ slurry in surface condition and its effectiveness is saved by boric acid adjacent to SLES in high temperatures.
- 9. SLES in the slurry affects as an accelerator. The thickening time of the slurry is decreased more and rapidly by adding the retarder. Finally, SLES, JR120 and boric acid were added to the slurry and its thickening time enhanced to 175min. This is an acceptable operational time for a cementing job. Although, the TT of common lightweight cements is more than 200min and FBJ's TT.

- 10. The FBJ set-cement has one of the highest porosities between different foamed cements. Its porosity is near 73%. However, its permeability is very low. The permeability of FBJ is near 0.011mD and this is the result of that the porous media of this foamed cement is not interconnected. This feature is very necessary and critical for foamed cement designing. The isolate pores and connections are investigated by microscopic photography and CT scan image processing and these tests were proved that the FBJ pore structures are isolate and impermeable.
- 11. The CT scanning of the FBJ sample after HPHT conditioning (the UCA test set-cement samples at 210°F and 4000psi) is cleared that the pores of FBJ were saved and just the size of them became smaller.

## 5.2. Recommendations

- 1. Other methods of foamed cement sample preparation such as gas injection and its efficiency in foam generating should be studied.
- 2. For increasing the FBJ compressive strength the micro silica can be added to the slurry. Pay attention that micro silica usually decreases the thickening time.
- 3. Other retarders effects on this foam slurry composition must be investigated. That might a compatible retarder (without aiding agent) to SLES will be found.

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چکیدہ

سیمان کاری، یکی از مهم ترین و اساسی ترین بخش های یک عملیات حفاری چاه های نفت و گاز محسوب می شود. از طرفی، با افزایش نرخ تولید از مخازن نفت و گاز، این مخازن از لحاظ فشاری تخلیه شده اند. در نتیجه برای افزایش برداشت از مخازن، چاه های جدیدی حفاری می شوند. عملیات سیمان کاری در این لایه های کم فشار و یا لایه های شکافدار، باید با دوغاب های سبک و یا فوق سبک انجام شود. در این شرایط است که آسیب های وارده ناشی از عملیات سیمان کاری، به سازند شدیداً کاهش می یابد. یکی از جدیدترین فناوری های سیمان کاری با سیمان های فوق سبک، طراحی و فرمولاسیون سیمان های فومی است. در این پایان نامه، طراحی و ساخت سیمانی فومی برای عملیات سیمان کاری لوله های جداری و آستری در چاه های نفت و گاز ایران، بررسی شده است. ایـن سیمان فومی با استفادہ از سرعت برشی بالا تھیہ می شود. در این پژوهش سـدیم لوریـل اتـر سـولفات (SLES) به عنوان ماده كفزا انتخاب شده است. ميزان بهينه اين ماده براي تهيه سيمان فومي و طرز تهیه بهینه ساخت این نوع سیمان بررسی شده است. هم چنین افزایه های مختلفی برای ویژگی های خاصی از این سیمان به آن اضافه شده است. تست های مختلفی بر روی نمونه های گوناگون سیمان فومي، تا رسيدن به بهترين طراحي ممكن صورت گرفته است، از جمله: پايداري فوم، محاسبه دانسيته، رئولوژی دوغاب سیمان، حجم آب آزاد، زمان بندش دوغاب سیمان، استحکام تراکمی سیمان سخت شده، میزان هرزروی دوغاب، تخلخل و تراوایی سیمان فومی، عکس برداری میکروسکوپی و سی تی اسکن نمونه های مختلف سیمان فومی سخت شده. نتایج آزمایش ها بـر روی بهتـرین نمونـه سـیمان فومی طراحی شدہ در این پایان نامہ کہ با نام FBJ معرفی می گردد، با نتایج آزمایش های انجام شدہ بر روی سیمان های سبک معمول که در میادین نفت و گاز ایران، نظیر آزادگان جنوبی، مورد استفاده

می باشد، مقایسه شده است. برای بهترین طراحی و فرمولاسیون سیمان فومی (FBJ) با ویژگی های مطلوب، مواد مختلفی مورد استفاده قرار گرفته است. این مواد شامل 778gr سیمان کلاس G، مطلوب، مواد مختلفی مورد استفاده قرار گرفته است. این مواد شامل 778gr (۵٫۷۵ قرار) عار 350gr قرار گرفته است. این مواد شامل 778gr (۵٫۷۶ قرار) با معظر، 350gr (۵٫۷۶ قلار) یا SLES (۵٫۱% قلار) یا ۵٫۳۶ قلار) بوریک اسید می شود. نتایج آزمایش ها نشان می دهند که FBJ دارای کمترین دانسیته با استحکام تراکمی اسید می شود. نتایج آزمایش ها نشان می دهند که FBJ دارای کمترین دانسیته با استحکام تراکمی کافی در صنعت سیمان کاری و حفاری ایران است. دانسیته FBJ معادل ۲۶pcf با بیش از 1300psi استحکام تراکمی استحکام تراکمی است. زمان بندش آن حدود 3 ساعت است و رئولوژی آن در شرایط ته چاهی به دلیل اثر بولبیرینگی حباب های درون دوغاب فومی، بهبود می یابد. بخاطر خاصیت فومی این دوغاب، هم متصل نمی باشد. این ایزوله بودن فضای متخلخل FBJ، با استفاده از تسم مان حرود گران بندش آن حدود 3 ساعت است و رئولوژی آن در شرایط ته چاهی به دلیل اثر بولبیرینگی حباب های درون دوغاب فومی، بهبود می یابد. بخاطر خاصیت فومی این دوغاب، هم متصل نمی باشد. این ایزوله بودن فضای متخلخل FBJ، با استفاده از تست های تخلخل، تراوایی، تصویربرداری میکروسکوپی و سی تی اسکن اثبات شده است. هم چنین، آنالیزها و تصویربرداری های سی تی اسکن بر روی نمونه هایی که در شرایط دما و فشار بالا (HPHT) نگهداری می شدند، نشان

#### مقدمه

وقتی که یک چاه نفت و یا گاز حفاری می شود، لوله هایی به نام لوله جداری و یا آستری به درون چاه رانده می شود تا از ریزش دیواره چاه به درون آن جلوگیری کند. علاوه بر این، لوله های جداری فواید دیگری دارند، که از آن جمله: مانعی برای فشار سازندی هستند، از جریان سیال سازند به درون چاه جلوگیری می کند، مانع آلودگی آب های زیرزمینی می گردد و سطحی صاف و مستحکم از جنس آلیاژ فولاد ایجاد کرده که برای عملیات های بعدی درون چاه مناسب است. لوله های جداری و آستری با سیمان حفاری به دیواره چاه متصل می شوند [1].

سیمان به عنوان یک ماده عایق کننده در چاه های حفاری شده نفت و گاز استفاده می شود. با پمپاژ سیمان به پشت لوله های جداری و آستری، یک عایق برای جریان سیال از و یا به سازند ایجاد کرده و این لوله ها را به سازند متصل می کند. این عملیات را سیمان کاری اولیه می نامند. سیمان کاری برای کاربردهای دیگری نیز به درون چاه پمپ می شود، که در زیرمجموعه عملیات سیمان کاری ثانویه و یا تعمیراتی قرار می گیرد. آنالوس، به عنوان فضای حلقوی مابین دیواره لوله جداری و دیواره چاه شناخته شده که با دوغاب سیمان پر می شود. مکانیزم سیمان کاری در این ناحیه با استفاده از پمپ سیمان از درون لوله جداری به کمک پلاگ های پاک کننده بالایی و پایینی و تجهیزات خاص سیمان کاری حفاری، صورت می گیرد [2].

دلایل مختلفی برای انجام عملیات سیمان کاری حین یک عملیات حفاری وجود دارد. مهمترین دلایل انجام یک عملیات سیمان کاری عبارت اند از:

- سیمان مقداری از وزن لوله جداری را تحمل می کند و این در زمانی اتفاق می افتد
  که لوله کاملاً در مرکز چاه قرار گرفته باشد و سیمان آن را کامل احاطه کند؛
- سیمان سبب عایق سازی سازند شده و مانعی از ورود سیالات لایه ای از سازند به لایه ای دیگر می شود. بدین ترتیب، در نواحی نزدیک به مناطق شهری، با استفاده از این فناوری، آب های زیرزمینی از آلودگی ناشی از سیالات لایه های دیگر سازند، محفوظ می ماند. بدین ترتیب سیمان حفاری، کمکی به محیط زیست نیز می کند؛
- سیمان در صورت قرار گیری به موقع و سریع در جایگاه معین، از فوران چاه جلو گیری
  می کند؛
- سیمان مانعی برای خوردگی لوله های جداری و آستری ناشی از حملات سیالات سازندی نظیر آب نمک، است؛
  - سیمان جلوی نشتی ها و هرزروی های احتمالی را به برخی از نقاط سازند می گیرد؛
- سیمان می تواند چاه های قدیمی را مسدود کند (متروک سازی چاه) و یا اینکه ناحیه مشخصی از سازند و مخزن را که از لحاظ فشاری تخلیه شده است را عایق نماید [3].

بسیاری از چاه ها در مناطقی حفاری می شوند که سست و ضعیف هستند و نمی توانند یک ستون نرمال دوغاب سیمان را با دانسیته بیش از 15ppg (112pcf) تحمل کنند. در این شرایط نیاز است، از یک سیمان سبک که دارای ستون سیال با فشار هیدولیکی پایینی می باشد، استفاده نمود. افزایه های مختلفی نظیر بنتونایت و یا گلیسونایت وجود دارد که سبب کاهش وزن دوغاب سیمان تا حدود 12ppg می شود، ولی استحکام مورد نظر بدست نمی آید. راه دیگری برای کاهش وزن دوغاب سیمان تا سیمان، ترکیب سیمان با یک می اید. راه دیگری برای کاهش وزن دوغاب سیمان تا محدود وی ای گلیسونایت وجود دارد که سبب کاهش وزن دوغاب سیمان تا حدود 12ppg می شود، ولی استحکام مورد نظر بدست نمی آید. راه دیگری برای کاهش وزن دوغاب سیمان تا سیمان، ترکیب سیمان با یک ماده فوم ساز (کف ساز) و گاز (هوا یا نیتروژن) است، که در چنین شرایطی سیمان فومی، با محدوده وزنی حدود ppg 01-8 و با استحکام تراکمی مناسب بدست می آید. در سازندهایی با گرادیان فشاری پایین و یا شکافدار، هرزروی شدید دوغاب سیمان های معمول و یا حتی عدم بازگشت سیمان پمپ شده به سطح بوجود می آید. سیمان های معمولی، به دلیل ترکیب با آب، وزنی بیش از ppg 15 می شرای حدل معمی از ای ایت معرفی شده است. که در بیشتر مواقع بیش از ppg 15 دارند. لذا سیمان فومی برای حدی از معرفی بیش از ppg 15 دارند. لذا سیمان می میمان فومی برای حل پر با میدولی برای می شده به سطح بوجود می آید. سیمان های معمولی، به دلیل ترکیب یا آب، وزنی بیش از gpg 25 در بیشتر مواقع بیش از ppg 15 دارند. لذا سیمان فومی برای حل چنین مشکلاتی معرفی شده است.

#### هدف

هدف این پایان نامه طراحی و فرمولاسیون یک سیمان فومی جدید برای عملیات سیمان کاری لوله های جداری و آستری، برای اولین بار در صنعت نفت و گاز ایران است. با افزایش نـرخ تولیـد از منابع نفت و گاز و رسیدن به اواخر عمر مخازن ایران، فشار ایـن مخازن کـاهش پیـدا کـرده است. از طرفی دیگر، حدود %87 از مخازن ایران، از نوع مخازن شکافدار هستند. بیشتر مشکلات حـین انجـام یک عملیات سیمان کاری در چاه های نواحی کم فشـار و یـا شـکافدار، هـرزروی شـدید سـیمان و یـا شکست سازندی می باشد. پس بایستی از دوغاب های با وزن کم استفاده نمود، که متأسـفانه در ایـران بیشتر سیمان های سبک رایچ، دارای استحکام تراکمـی کمـی هسـتند. سـیمان هـایی هـم کـه دارای استحکام کافی می باشند، دارای وزن بالایی بوده و یا دارای افزایـه هـای بسـیار گـران قیمـت هسـتند. دلیل این محدودیت کاهش وزن در این است که این افزایه ها در سیمان های سبک رایج، عمدتاً جامد وستند، ولی در سیمان های مومی، عامل کاهش دهنده وزن گاز بوده که مـی تـوان دانسـیته آن را در هستند، ولی در سیمان های فومی، عامل کاهش دهنده وزن گاز بوده که مـی تـوان دانسـیته آن را در قیاس با بقیه مواد، صفر در نظر گرفت. این پروژه تحت حمایت صنعتی شرکت ملی حفاری ایران قرار گرفته است. عمده تست های آزمایشگاهی این پایان نامه نظیر تهیه دوغاب سیمان، اندازه گیری دانسیته و رئولوژی آن، زمان بندش، حجم آب آزاد و ته نشینی، استحکام تراکمی و حجم صافاب دوغاب سیمان در آزمایشگاه سیمان شرکت ملی حفاری ایران انجام شده است. تست های دیگری نظیر تراوایی و تخلخل پلاگ های سیمان فومی، تصویربرداری میکروسکوپی و آنالیز سی تی اسکن در آزمایشگاه های دانشگاه صنعت نفت صورت گرفته است. عمده این آزمایش ها در شرایط دما و فشار بالا (HPHT) انجام شده است. هم چنین تست هایی بر روی پایداری و کیفیت فوم ایجاد شده با ماده کف ساز نیز انجام پذیرفته است.

# طرح پایان نامه

این پایان نامه شامل پنج فصل می باشد. در فصل اول، مقدمه ای از پروژه انجام شده، توضیح داده شده است. در بخش دوم، پیش زمینه ای بر سیمان، انواع مختلف عملیات سیمان کاری، کلاس های مختلف سیمان و افزایه های آن، ساختار فوم و کاربردهای آن، تاریخچه ای از فناوری سیمان فومی و فواید آن، شرح داده شده است. همچنین در پایان فصل دوم، بررسی جامعی از کارها و تحقیقات گذشتگان (محققان و مهندسین مختلف از گذشته تا به حال) بر روی طراحی سیمان فومی و فناوری عملیاتی آن، انجام گرفته است. در فصل بعدی، فهرستی از تجهیزات آزمایشگاهی که تست ها با آنها انجام شده، نحوه انجام تست ها و همچنین موادی که در این تحقیق از آن استفاده شده است. پروژه، ارائه شده و بحث و تحلیل مفصلی بر روی این نتایج و بهینه ترین طراحی و فرمولاسیون، انجام گرفته است. در نهایت در فصل پنجم، نتایجی که از این تحقیق بدست آمده بیان شده و پیشنهادهایی برای ادامه این پروژه و کارهای آینده ذکر شده است.