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UNIVERSITI TEKNOLOGI PETRONAS

IMPROVED GAS MIGRATION CONTROL IN OIL WELL CEMENT

by

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IMPROVED GAS MIGRATION CONTROL IN OIL WELL CEMENT

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GHULAM ABBAS

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DECLARATION OF THESIS

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hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UTP or other institutions.

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DEDICATION

I dedicate this thesis to my father Rahib Qumbrani, brothers Muhammad Akhtar, Muhammad Abbas and sister in law Ali Gul for support during my studies and encouragement for this great achievement.

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ABSTRACT

Polymers are being used as multifunctional additives that control fluid loss, free water and prevent gas migration through cement slurry. Most of the polymers degrade at 80 °C and lose their viscosity with increasing temperature. The reduction in the viscosity of polymers leads to fluid loss, free water separation and decreases hydrostatic pressure which causes gas migration. In this research, Hydroxypropylmethylcellulose (HPMC) has been studied and used in cement slurry to prevent gas migration. The objectives of this study were to determine the viscosity of HPMC solutions, API properties and gas migration of HPMC based cement slurries at 90 °C. The viscosity of 1.5 and 2 wt% HPMC solutions was determined at different temperatures of 30 to 100 °C with respect to shear rates. The cement slurries of 16.5 ppg density were prepared using 0.20 to 0.80 gps concentration of HPMC solutions with fluid loss, dispersant, retarder and defoamer additives. The API properties of the cement slurries were determined in terms of rheology, fluid loss, free water, sedimentation, thickening time, compressive strength and gel strength at 90 °C. The experimental results showed that the HPMC solutions increased in viscosity at 90 °C. The addition of 0.30 to 0.50 gps HPMC improved the rheology, decreased the fluid loss to 20 ml and yielded 3 ml of free water. The HPMC polymer also increased the thickening time from 3:23 hours to 6:29 hours and compressive strength up to 4100 psi. The transition time of the cement slurries was less than 45 minutes that met the API criteria for gas migration prevention. It was observed that the pore pressure of the HPMC based cement slurries was less than 20 psi for 6 to 8 hours injection of gas. It was concluded that the HPMC polymer acts as a viscosifying agent at 90 °C. It was improved API properties and prevents gas migration through cement slurries at 90 °C.

ABSTRAK

Polimer digunakan sebagai bahan tambahan untuk pelbagai fungsi seperti mengawal kehilangan cecair, air bebas dan mencegah penghijrahan gas di dalam buburan simen. Kebanyakan polimer mengalami degradasi pada 80 ^{0}C dan pengurangan kelikatan dengan suhu meningkat. Pengurangan kelikatan polimer mengakibatkan kehilangan cecair, pemisahan air bebas dan mengurangkan tekanan hidrostatik yang menyebabkan penghijrahan gas. Dalam kajian ini, Hydroxypropylmethylcellulose (HPMC) telah dikaji dan digunakan dalam buburan simen untuk mengelakkan penghijrahan gas. Objektif kajian ini adalah untuk menentukan kelikatan HPMC, kriteria API dan penghijrahan gas melalui buburan simen berasaskan HPMC pada suhu 90 ^{0}C. Kelikatan 1.5 dan 2% berat HPMC telah ditentukan pada suhu yang berbeza daripada 30 hingga 100 ^{0}C berdasarkan kadar ricih. Buburan simen dengan kepadatan 16.5 ppg telah disediakan dengan menggunakan kepekatan HPMC 0.20 hingga 0.80 gps beserta bahan tambahan kehilangan cecair, dispersant, pelengah dan defoamer. Kriteria API buburan simen ditentukan dari segi reologi, kehilangan cecair dan air bebas, pemendapan, masa penebalan, kekuatan mampatan dan kekuatan gel pada 90 ^{0}C. Keputusan eksperimen menunjukkan bahawa kelikatan HPMC meningkat pada 90 ^{0}C. Penambahan 0.30 hingga 0.50 gps HPMC mempertingkatkan reologi, mengurangkan kehilangan cecair hingga 20 ml dan menghasilkan 3 ml air bebas. Masa penebalan polimer HPMC juga meningkat dari jam 3:23 hingga jam 6:29 dan kekuatan mampatan sehingga 4100 psi. Masa peralihan buburan simen adalah kurang daripada 45 minit yang memenuhi kriteria API untuk mencegah penghijrahan gas. Di samping itu, turut dapat diperhatikan bahawa tekanan liang pada buburan simen beasaskan HPMC adalah kurang daripada 20 psi selama 6 hingga 8 jam suntikan gas. Oleh itu, kesimpulan dapat dibuat bahawa polimer HPMC bertindak sebagai agen kelikatan pada 90 ^{0}C. Polimer HPMC juga mempertingkatkan kriteria API dan menghalang penghijrahan gas melalui buburan simen pada 90 ^{0}C.

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T	Time in minutes
wt%	Weight percent
V_t	Volume of filtrate

CHAPTER 1

INTRODUCTION

This chapter provides the background of study, problem statement, proposed solution, objectives, research design, scope and significance of this research.

1.1 Background of Study

Well cementing is the process of placing the cement slurry in annular space between the formation and casing in order to provide zonal isolation to the wellbore. The aim of cementing operation is to prevent water or gas migration from high permeable zone to low permeable zone in the well or to the surface. Improper zonal isolation and weak hydraulic seal between the cement and formation or casing and the cement is the main cause of seepage. In this situation, the well may never run at its full producing potential [1]. Therefore, the American Petroleum Institute (API) introduced standard properties, including the rheology of cement slurry, fluid loss, sedimentation, free water, thickening time, compressive strength, hydration and gel strength development that must be optimized to achieve zonal isolation within the cement slurry.

Several types of chemical admixtures, such as accelerators, retarder, fluid loss additives, defoamers, anti gas migration agents and extenders have been used to optimize the properties of the cement slurry. The performance of additives is influenced by chemical and physical properties of the Portland cement that is not applicable for oil well cementing. In order to compete high pressure and temperature bottom hole conditions, special classes of cement called Oil Well Cement (OWC) specified by API have been used in the designing of cement slurries [2]. There are eight different classes of OWC that are specified by depth, formation characteristics and temperature ranges. The usage of OWC with different types of additives in order to improve slurry properties for zonal isolation at high temperature is still unexplored. On the other hand, the interaction of OWC with several additives is incompatible and that affects the properties of cement slurry. This result in poor cement slurry performance and the completely failure of cementing system [3]. The use of several additives increases the operational cost of cementing. To minimize the incompatibility issue and operational cost, it would be ideal to reduce the number of additives in cement slurry by using multifunctional additives.

Currently, various polymers have been used in the petroleum industry for different operations. The most engaged polymers are Hydroxyethylcellulose (HEC), Hydrolyzed Polyacrylmide (HPAM), Carboxymethyl Hydroxyethylcellulose (CMHEC), Guar, Hydroxypropylguar (HPG), Starch, Xanthan, Welan gum, Scleroglucan and Diutan [4]. These polymers are widely used as multifunctional additives in cement slurries. They reduce the quantity of chemical admixtures and improve the cement slurry performance. These biopolymers are being used as viscosifying agents in cement slurries, reduce the permeability, control the fluid loss and free water separation. The biopolymers also maintain the hydrostatic pressure above the formation pressure that ultimately prevents the gas migration through the cement slurry. The usage of biopolymers overcomes the problem of incompatibility and operational cost [5].

The polymers being used in oil well cementing are degradable at high temperature. The viscosity of the polymers decreased with increasing temperature. The low viscosity of the polymer at high temperatures leads to fluid loss, free water separation and sedimentation that becomes the cause of hydrostatic pressure reduction. Therefore, the cement slurries with polymers do not prevent the gas migration through cement slurry. At present, the industry is exploring deep and high temperature wells. In order to reduce the cost, various polymers have been used in cement slurry as multifunctional and gas migration control additives. However, these polymers have a limitation in regards to temperatures. Such limitations are: HPAM polymers are unstable above 70 ^{0}C [6], Starch and Carboxymethylcellulose (CMC) degrade significantly above 90 ^{0}C [7], Xanthan gum drops in viscosity above 85 ^{0}C [8]. The most widely used Hydroxyethylcellulose (HEC) is stable below 60 ^{0}C in pH region of 6-8 [9]. Yet, there is no widely acceptable polymer that can be used in oil well cementing operation for the prevention of gas migration above 80 °C. Responding to this discussion, it is necessary to source for such polymers that can increase viscosity with the increase in temperature. The polymer can work as multifunctional additive and prevents the gas migration though cement slurry.

1.2 Problem Statement

Biopolymers are well known viscosifying agents for cement slurries. At high temperature, biopolymers do not work efficiently to control gas migration during cementing. The biopolymers degrade at high temperature and decrease the viscosity with increasing temperature. The biopolymers loses their viscosity at elevated temperature and do not control the fluid loss or gas migration through cement slurry above 80 °C [10,11]. The decrease in viscosity of biopolymers leads to hydration and fluid loss through the cement slurry. The hydration decreases the water volume in the slurry and effectively reduce the cement pore pressure [12]. At the same time, hydraulic cement starts to develop gel strength where it cannot transmit full hydrostatic pressure. The loss of fluid and decrease in cement pore pressure during this period lowers the hydrostatic pressure to less than formation pressure [13]. As the result gas starts to migrate through cement slurry due to this hydrostatic pressure reduction.

1.3 Proposed Solution to Research

This thesis presents Hydroxypropylmethylcellulose (HPMC) as an additive in oil well cementing. The HPMC polymer is the derivative of cellulose ether which consists of an anhydroglucose unit with hydroxyl group on second number carbon chain. HPMC is water soluble polymer and behave as a viscosifying agent in aqueous solution [14]. The HPMC solution starts to change into gel at gelation temperature. Then, the viscosity of solution will increase with temperature [15]. The increase in viscosity at gelation temperature is unique property of HPMC that is not found in other cellulose type polymer. The increasing viscosity at gelation temperature makes it stable at high temperature. HPMC polymer is found stable at high temperature. Therefore, it is expected that it will also act as multifunctional additive in cement slurry and prevent the gas migration through the cement slurry. To achieve these goals, the following objectives were designed with their methodology.

1.4 Objectives

This research is based on the implementation of HPMC polymer as a multifunctional additive in cement slurry. The polymer will improve oil well cement slurry properties and prevent gas migration through cement slurry at 90 °C. Thus the objectives of this thesis are as listed below.

1. To determine the viscosity of HPMC solutions at various temperatures (30 to 100 °C) with respect to shear rate (1 to 1000 s^{-1}).
2. To analyze the effect of HPMC polymer on API properties (rheology, fluid loss, free water, sedimentation, thickening time, compressive strength, transition time) of cement slurries at 90 °C.
3. To determine the gas migration through HPMC based cement slurries at 90 °C.

1.5 Research Design

The laboratory experiments are crucial to understand the cementing process. Therefore, a number of extensive experiments were designed on HPMC based cement slurry to control the gas migration at 90 °C. This process was divided into three phases:

In phase 1 literature review was carried out to choose the materials i.e., HPMC polymer, additives and API cement. It also includes the preparation of materials, such as preparation of HPMC solutions and cement slurries using different concentrations of HPMC with other additives.

Phase 2 was dealing with the experiments of HPMC solution and cement slurries. In this phase, the effect of temperature, concentration and shear rate on the viscosity of HPMC solution were evaluated. The API properties of cement slurry were determined in terms of rheology, fluid loss, free water, sedimentation, thickening time, compressive strength and static gel strength for measuring transition time.

Phase 3 presented the gas migration test using Cement Hydration Analyzer (CHA). The gas migration though cement slurries was determined using different concentration of HPMC polymer with additives at 90 ^{0}C.

1.6 Scope of Research

In this study, two solutions of HPMC polymer were tested. The concentrations 1.5 wt% and 2 wt% of HPMC solution were prepared using different amounts of polymer in distilled water. The shear viscosity of HPMC solutions was determined at 30 ^{0}C to 100 ^{0}C with respect to shear rates (1 to 1000 s^{-1}). The effect of temperature was achieved at two temperatures 80 ^{0}C and 90 ^{0}C. After the viscosity measurement, 16.5 pounds per gallon (ppg) density of cement slurries were prepared. In which, the concentration of HPMC solutions with other additives was added from 0.20 to 0.80 gallon per sack (gps). The rheology of cement slurries was determined at ambient temperature and 90 ^{0}C at different shear rates of 3 to 600 rpm. Then, the fluid loss, free water, thickening time, compressive strength, transition time and gas migration tests were determined at 90 ^{0}C using 16.5 ppg density of cement slurries.

1.7 Significance of Research

This research provides a comprehensive study of HPMC polymer effect on properties of oil well cement slurries subjected to high temperature. This study has succeeded in ascertaining the HPMC polymer as a viscosifying agent that has increasing the viscosity in hostile environment of wellbore. Therefore, HPMC polymer can be used as a multifunctional additive for improving oil well cement slurry properties and a gas blocking agent to controlling gas migration. The experimental results of this research open a new window for petroleum industry by preventing gas migration at 90 ^{o}C. On the other hand, the polymers employed currently are not applicable for prevention of gas migration above 80 ^{0}C without addition of other chemicals.

1.8 Thesis Organization

The thesis has been divided into five chapters, including this introductory chapter. Chapter Two covers the literature review of gas migration, causes and different chemical admixtures for the prevention of gas migration. Chapter Three provides the extensive methodology used in this research. Chapter Four consist of the results and discussion of the experiments, while Chapter Five presents the conclusions and recommendations for future study.

CHAPTER 2

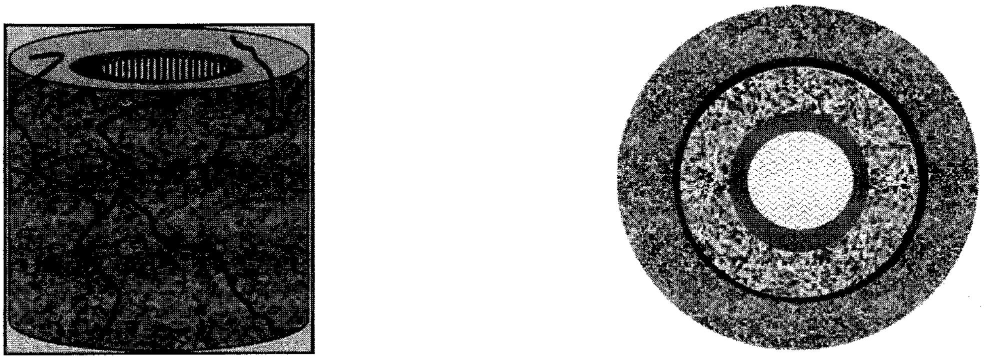
THEORY AND LITERATURE REVIEW

This chapter provides the causes of gas migration, mechanism of gas migration and literature of previously used methods for prevention of gas migration. It also provides the theory of ether cellulose and their derivatives, chemistry of HPMC polymer, properties and applications of HPMC polymer.

2.1 Gas Migration through Oil Well Cementing

Oil and gas wells have been cased to optimize the flow rate and to produce hydrocarbons economically and safely till the end of the well life. It is possible to produce the well without casing (open hole completion). However, for high temperature and high pressure deep wells, it is essential to use casing in the wellbore. In order to protect the casing string from corrosion, to support the axial load of casing and to support the wellbore, it is very necessary to seal the annulus between casing and formation through cement.

During cementing operation, gas migration through high permeable zone to low permeable zone or to the surface has been considered as a severe problem for petroleum industry. Gas migration through cement column can occur during primary process and secondary process of cementing [16]. In primary process, gas migration can occur during or immediately after placement of cement slurry. In secondary process, gas migration can occur anywhere at the time of well production. The change in physical behaviour of cement slurry due to various factors is the cause of primary gas migration. This process leads to a leakage in the early stage of cement placement. The secondary gas migration occurs due to some mechanical or operational failure in cementing job. The hydraulic and mechanical stress creates cracks in the cement sheath that become the cause of gas migration as shown in Figure 2.1.



Side view of Cement sheath cracks Top view of debonding

Figure 2.1: Cement sheath cracking and debonding from formation [1].

In this research more attention has been given to the primary gas migration during placement and at initial stage of cementing, while the secondary gas migration has been shown less attention.

2.2 Causes of Primary Gas Migration

The gas migration through cement column was first recognized in gas storage well in the mid 1960's. It was thought that this problem happened due to the interaction between the casing and cement formation [17]. Gas migration through cement column mostly occurred in deep well completion across the gas intervals where it increased the pressure in the annulus of production and intermediate casing. In the early stages 1960 to 1970, parameter such as density of fluid, mud removal through annulus, setting of cement up the annulus in the wellbore, cement hydration, cement/casing/formation bonding, and bridging were recognized as the primary causes of gas migration through the cement column.

2.2.1 Density Alteration of Cement Slurry

During cementing operation the density of the fluid is altered due to the variation in wellbore temperature and the presence of gas bubbles that were trapped in the wellbore before cementing. These gas bubbles are considered as the potential gas kick. The presence of these gas bubbles becomes the cause of density reduction of fluid. Therefore, the variation in the density of mud, preflushes, spacers and cement slurries are due to gas bubbles reducing the hydrostatic pressure of cement column [19, 20]. If this hydrostatic pressure of cement column decrease than the formation gas pressure, gas kick could be occur and further reduction of hydrostatic pressure becomes the cause of gas migration.

2.2.2 Poor Mud Removal

Improper and poor mud removal creates channels during the cementing operation. The mud channels are created due to the improper displacement of the drilling fluid by cement slurry. These mud channels are formed at the narrow side of the annulus and especially in that area where shear stress becomes very low. After displacement of the cement slurry, these mud channels remain under pressure. This increases the permeability inside the cement slurry. The increase in permeability generates a continuous channel to the surface. The gas migration occurs from the formation to another low permeable zone or to the surface [20].

2.2.3 Setting of Cement Slurry

The early setting of cement slurry inside the wellbore also contributes to increase the possibility of gas migration through the cement slurry. The gas migration due to early setting becomes more risky in deep wells because of high temperature [17]. Before the cementing operation the circulation of fluid decreases the geothermal rock temperature near the wellbore. At that time, circulating fluid increases the geothermal rock temperature at some points up the annulus [23]. Under this high temperature environment, the cement slurry sets faster at these high temperature points inside the annulus. Early setting of cement slurry in annulus restricts the transmission of hydrostatic pressure to the lower gas zones. The reduction of hydrostatic pressure promotes the gas migration through cement column.

2.2.4 Cement Hydration

Cement hydration is a severe cause of gas migration through cement slurry. The hydrostatic pressure of cement column decreases due to cement hydration. The volume of the cement slurry is reduced during cement hydration reaction [23]. An exothermic reaction between cement and water is involved during cement hydration process [24]. The initial hydration reaction of cement particles with water leads to 0.1 to 0.5 wt% volume reduction of cement slurry. The hydration process consumes mixed water and forms the crystalline pore structure which further reduces the volume up to 3% of cement [20]. This volume reduction due to the hydration process cannot be compensated. As a result, the pore pressure of cement slurry is dropped. This decreases the hydrostatic pressure of cement column above formation. Thus, the gas starts its migration due to the reduction of hydrostatic pressure during the cement hydration process.

2.2.5 Bridging of Cement Particles

During cementing operation, the bridging of particles restricts the transmission of hydrostatic pressure. It mostly forms in narrow annulus with long thickening time and occurs during the static condition of slurry [25]. The bridging of cement occurs due to cement filtrate cake in front of permeable zone. The buildup of bridge in annulus depends upon the thickness of cement filtrate cake. The particles of cement make bridge as thickness of cement cake increase at one place inside the wellbore [17]. Bridging in cement slurry occurs at permeable formation in wellbore and cement slurry has not transmitted the hydrostatic pressure below it. Gas migration may occur through the cement slurry if there is any gas zone below the bridging.

2.2.6 Fluid Loss through Cement slurry

Fluid loss is recognized as most causative factor for gas migration through cement slurry. Fluid loss through cement slurry happens due to the deposition of mud filter cake against the formation under static condition. When the slurry comes in contact with permeable zone, it loses fluid (filtrate) to the zone and deposits a filtrate cake at that point [17].

During displacement, the cement slurry passes through permeable zone and will lose more fluid to the zone. This creates a thick filter cake of cement solids. The growth of cement filter cake increase the undesired density of cement slurry that require more circulation pump pressure to displace the cement slurry [26]. This increase in circulation pressure may become the cause of formation breakthrough especially in that condition when hydrostatic pressure is already close to the break down formation pressure. This continues filtrate loss into permeable zone decreases the volume of slurry into the annulus. It also increases the solid to water ratio and builds the bridge near the high permeable zone [25]. The loss of fluid in permeable zone becomes the cause of cement pore pressure and hydrostatic pressure reduction that allows the gas to migrate into the wellbore.

2.2.7 Free Water Separation

The effect of excessive free water separation from cement slurry was observed and recognized as a severe problem for gas migration in 1979 [27]. The separation of free water creates cracks and voids due to the differential settling and accommodation of water at casing/cement or formation/cement interface. These voids or cracks form a channel through the cement column. The free water moves upward along the high side of the annulus through the channel and collects at hte formation/cement interface. As result, the separation of free water allows the gas migration to take place [20]. In 1979, Webster and Eikerts performed an experiment to observe the effect of free water inside the slurry. They constructed nine foot long model with gas entry source and inclined up to 70 degree angle to observe the effect as like in deviated hole. It was observed that the water was separated through the slurry and collected at the top of the model. Therefore on the basis of experimental result it was concluded that the separation and flow of water create channel that may becomes the cause of gas migration [27].

The separation of water/ fluid from the homogeneous slurry reduces the strength of slurry. Therefore, the designed cement slurry is incompetent to hold the partials inside the cement slurries [10]. That causes the heavier particles to settle rapidly down to the bottom. This uniformity of particles inside the slurry is known as settling, or particle segregation or sedimentation. The free water phenomenon is not only a behaviour of chemical stability inside the cement but also a result of gravitational segregation due to the hydrostatic pressure by cement [28].

2.2.8 Gelation of Cement Slurry

The gelation of cement slurry is very important parameter with regards to the lowering of hydrostatic pressure. In 1970, Carter and Slagle pointed out that the hydrostatic pressure of cement column may be decreased due to the thixotrophy and gelation of slurry. But they did not explain the phenomenon of hydrostatic pressure reduction [17]. As early as 1973, Carter et al., attempted to analyze the effect of thixotrophy and gelation on hydrostatic pressure. In 1982 Cooke et al., through laboratory and field experiments, validated the reduction of hydrostatic pressure during slurry displacement and cement hydration process [29]. Again in 1982, Sabins et al., gave the presumption about the gel strength development, effect of gelation on hydrostatic pressure and concept of the transition time of cement slurry [12].

The time interval of gelation state depends upon the temperature, pressure, composition of cement and water- cement ratio. After the placement of cement slurry into the wellbore, cement slurry begins to change from liquid to solid state. The period during which cement slurry changes from hydraulic fluid to highly viscous fluid (like gel) and viewing some solid characteristics is called transition time [30]. The development of gel restricts the hydrostatic pressure transmission through the slurry. During the transition state, cement slurry will begin to support itself and reduce the slurry's ability to transmit hydrostatic pressure. After placement of cement slurry, it develops enough gel strength to restrict the conveyance of full hydrostatic pressure. At that time the transition time starts. The transition time ends at the time when the slurry develops enough gel strength to control percolation of gas through cement [31].

2.3 Mechanism of Gas Migration

During and after placement of cement slurry inside the annulus, the cement slurry acts as a liquid and fully transmits the hydrostatic pressure to the formation. A small amount of fluid is lost through the cement to the surrounding formation. This fluid loss can be compensated by an equal decrease in the slurry volume and a downward movement of cement slurry [32]. At the same time wellbore temperature, formation pressure and heterogeneous characteristics of formation changes the properties of cement slurry. During this period, the denser and heavy particles of cement will settle down and cement hydration reaction will begin. Further losses of fluid from the slurry, grows the cement filter cake to the permeable formation that decreases cement column pressure against the formation.

Initially, during the pumping of slurry, the flow of slurry erodes the cement cake and after short transient time, it stops growing. But after complete displacement, the filter cake grows freely and rapidly [25]. The reduction of slurry pressure was depending upon the fluid loss rate and compressibility of slurry. Due to the hydration reaction, the particles of cement start to develop gel strength. The initiation of gel strength prevents the further settling and sedimentation of cement particles. Further fluid of loss into permeable formation through gelled slurry will reduce the volume of cement slurry that cannot be compensated by the movement of slurry. At the same time the cement slurry enters in the transition state. The gel strength range 100 to 500 Ib/ 100 ft^2 is called transition period of cement slurry [31]. The hydrostatic pressure is continuously dropped at transition stage as shown in Figure 2.3. Therefore, the hydrostatic pressure of cement slurry will decrease to lower than the formation pressure. The complete schematic for understanding the gas migration mechanism is shown in Figure 2.2.

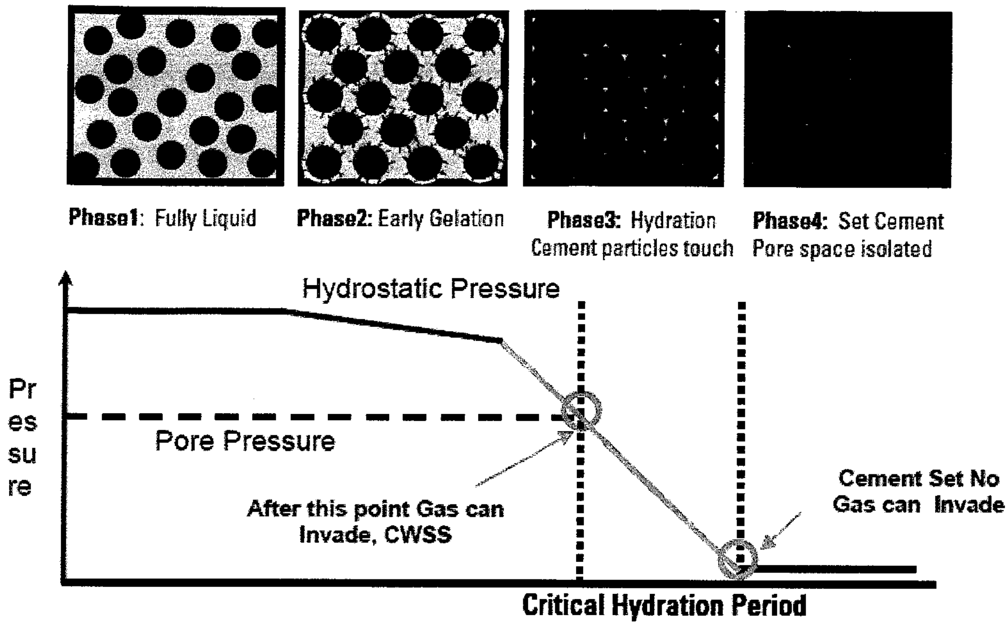


Figure 2.2: Different phases of cement setting and gas migration mechanism [33].

In the above given Figure 2.2, different phases of cement slurry are shown. In phase-1, the cement slurry acts as liquid and fully transmits the hydrostatic pressure. After completion of the displacement phase the cement slurry enters the transition state when it gains the gel strength of 100 Ib/ 100 ft^2. The second phase of early gelation starts and the hydrostatic pressure begins to decrease. So in this phase the hydrostatic pressure becomes very low and the slurry enters into third stage of hydration. At this phase the settling and temperature affects the slurry properties that increases free water, sedimentation and fluid loss. The loss of fluid and volume reduction further decreases the hydrostatic pressure. At one point the hydrostatic pressure becomes equal to formation pore pressure and gas starts to migrate. In this phase, the hydrostatic pressure reaches the minimum value and continues gas migration creates permanent channels in the cement column. In last phase, the cement becomes solid and hydrostatic pressure becomes constant at minimum pressure. At this phase, further gas cannot occupy by channels but the channels that have already been created at the hydration phase become the cause of permanent gas migration for the whole life of well.

2.4 Gas Migration Prevention Methods

Various methods have been proposed and implemented for prevention of gas migration during cementing operation over the years. The different types of cements, additives and polymers have been used for prevention of gas migration. Some of the most important ones are given below in detail.

2.4.1 Compressible Cement

The compressive cement system for controlling gas migration was introduced in 1979 and in 1980, good field results were reported [34]. Compressible cements were used to maintain the cement pore pressure above the formation pressure which would control gas migration from permeable zone into cement slurry. Foamed cement and In-situ gas generators are the two types of compressible cements that were used for controlling the gas migration. In the case of foamed cement the used gas continuously expanded and that decreased the volume of the cement slurry [35]. Thus, expansion of foamed gas compensates for the reduction in the volume of slurry during the transition state and maintains the hydrostatic pressure of slurry higher than the formation pressure [36].

The In-situ gas generator conditioned the pore pressure of the cement and it was maintained by a chemical reaction that generated gas in wellbore. Mostly, nitrogen or hydrogen is generated for maintaining the hydrostatic pressure of cement slurry during transition state. On the other hand, the compressible cement system is more expressive than conventional cement system and requires more engineering design. The generation of gas during this phenomenon becomes the cause of potential hazards and effects the environment [37].

2.4.2 Expensive Cement

The creativity of micro channels or minuscule cracks in the cement sheath has been considered as a most important factor for gas migration. The use of expensive cement in oil well cementing has been reported as a successful way to control gas migration in this condition [38]. The expansion inside the cement has been generated through gas generation and crystal growth techniques. In 1979, Griffin et al., introduced the expanding cement that would provide zone isolation [39].

The working mechanism of expanding cement depends upon the growth and nucleation of some specific minerals within a set cement matrix. The addition of minerals such as anhydrous calcium sulphoaluminate, calcium sulphate and lime in Portland cement has been used to prepare expanding cement [39]. The expansion capacity of this type of cement is more than 10 times that of the expanding cement that contains Portland cement with salt. Initially the expansion of cement using expanding cement was usually less than 1%. However, in the early stages of 2000, the expensive cements achieved the bulk volumetric expansion of up to 3% [40]. The expansion cement can be used to seal only micro channels between the cement sheath and casing or formation but it is very difficult to use the expansion cement for sealing large channels. Furthermore, it is also very necessary to ensure that expanding cement does not disturb or fracture the cement.

2.4.3 Impermeable Cement

The mobility of fluid inside the pore space of cement is also the cause of gas migration. Impermeable cement has been used to immobilize the cement filtrate within the pore space of cement slurry and thus prevent the gas migration through cement slurry [37]. The method to reduce the mobility of fluid within pore space is to increase the filtrate viscosity by using polymeric materials. Increasing the viscosity at tested temperature immobilizes the fluids that control the fluid loss and gas migration. One other method to reduce the mobility of the fluid is to bridge the throats of pore spaces using swell able bridging particles. The bridging materials control the filtrate inside pore space of cement and prevent the entry of gas through permeable zone [41]. The system of impermeable cement is composed of bridging agent and viscosifying polymers that have been applied to a wide range of temperatures.

2.4.4 High Gel Strength and Thixotropic Cement

Thixotropic cement has been used in some special cases where conventional cement slurries were unsuccessful. Thixotropic cement is thinner than conventional cement at the time of initial pumping and develops high gel strength after displacement. The materials i.e., Bentonite, certain sulphate salts or polymers, are used in Portland cement to prepare thixotropic and high gel strength cement [33].

The research on the transition time and gel strength of cement slurry has indicated that the thixotropic behaviour and high gel strength of cement slurry may help to prevent the flow of gas through cement slurry [12]. The high gel strength cement creates large gas bubbles and it has been considered that gas migration may be prevented due to these large bubbles. However, the bubbles that have smaller sizes than the pore spaces also become the cause of gas migration and the gel strength is no longer appropriate factor. In 1987, Sykes and Logan proved that high gel strength tends to lead to high fluid loss that further becomes the cause of dehydration and bridging [42]. It was proved that high gel strength cement is also one of the most important causes of the reduction of differential pressure.

2.4.5 Right Angle Set Cement

Right angle set (RAS) cement is one of the most important techniques to prevent gas migration through cement column. RAS cement has low almost negligible gelation tendency and due to hydration kinetics these cements set rapidly. The RAS cement is prepared by mixing the Portland cement with calcium sulphate hemihydrates (plaster) [43]. During cementing operation, the plaster sets rapidly and maintains the hydrostatic pressure in front of the permeable zone while the cement sets after some hours. RAS slurries have very low consistency during placement. Then, the viscosity of slurry increases rapidly and reaches 100 Bearden unit of consistency (Bc) in very short time [44]. This sharp increase in viscosity decreases the transition time and cement slurry obtains the gel strength of 500 Pa in very short time. RAS cement is considered good technique for prevention of migration but it has some limitation. RAS is applicable for low temperature ranges. The behaviour of slurry depends upon the properties of plaster; if the supply of plaster is inconsistent it will change the setting time of the slurry [43].

2.4.6 Quick Setting Cement

In 1985, D.J.Sepos introduced a new quick setting cement system for controlling gas migration by reducing the transition time of cement slurry. Quick setting cement has an advantage other cement systems in terms of short transition time, high and early strength development and adjustable thickening time from minutes to several hours. The quick setting cement has been used for shallow depths and low temperature wells. The effective temperature range for the quick setting cement was up to 38 °C [45].

2.4.7 Surfactant

In 1975 Marrast et al., patented the use of surfactant as a foaming agent in cement slurries for prevention of fluid migration through the permeable zone [46]. During displacement of the surfactant bearing cement slurry, the surfactant solution filtrates immediately and fills the pores of the wellbore. The migrated gas reacts with surfactant and creates a foam barrier that reduces the effective permeability of cement. Furthermore, the reaction of surfactant with gas creates a resistance to gas flow during the displacement and setting of the cement [20]. In 1993 K.M Cowan et al., performed a laboratory experiment using surface active agents in cement slurry and successfully implemented it in the cement operation for the prevention of gas migration [47].

2.4.8 Latex Additive

In 1984 Parcevaux et al., reported, the latex as a gas migration control agent through the cement slurry [48]. Later on, the application of latex in well cementing extended to a wide range of well conditions and was used in field applications in cementing operations [48]. The addition of latex in Portland cement improved the properties of cement slurry. Latex has low viscosity, shrinkage, pumping and setting time. It also has excellent fluid loss control and sealing properties [50]. When latex contained cement slurry is squeezed into permeable zone, the latex particles fill the pore space of formation which makes a bridge and forms a filtrate cake.

The effect of temperature and pressure has coalesces the latex particles and forms an impermeable protective sheath of latex in front of formation and cement. As the gas begins to migrate from the permeable zone, first the gas comes in contact with impermeable latex barrier. It prevents the further process of gas migration to the annulus of wellbore. The latex additives also work as multifunctional additive in the cement slurry and also have other beneficial properties. The small particles of latex act as surfactants, lubricants, and also have improved rheological properties in cement slurry. The low shrinkage and bonding action of latex also improves the shear bond strength and zone isolation [51].

2.4.9 Fluid Loss Additives

The loss of fluid during displacement of the cement slurry is most important factor of gas migration of fluid through the permeable zone to the wellbore. As following the steps of previous investigators, in 1975 Christian et al., emphasized that fluid loss should be controlled for prevention of gas migration. This is because the dehydration of cement slurry occurs due to fluid loss and it may be the cause of gas migration [52]. In 1976, Garcia and Clark proved from the experimental result, that the major cause of fluid migration is fluid loss which decreases the hydrostatic pressure of the cement column [53].

In 1977, Cook and Cunningham recommended maximizing the fluid loss control agent in the cement slurry during the cementing of high formation pressure and the permeable zone [54]. In order to prevent fluid loss, lot of chemicals and polymers have been used in cement slurries as Fluid Loss Additives (FLA). Fluid loss occurs during the dynamic condition and static conditions of slurry. For controlling gas migration through cement slurries, fluid loss should be low and at the minimum, approximately 50 ml/30 min [27]. It is very necessary to use fluid loss control additives in cement slurry to prevent fluid loss and gas migration. Fluid loss control is very important factor for cementing operations; therefore, lot of chemicals and polymers have been used in cement slurries to decreases the amount of fluid loss.

2.5 Polymers in Oil Well Cementing

The main function of the polymer is to increase the viscosity and decrease the permeability. Different types of polymers are being used in cementing operation to increase the viscosity of slurries. Polymers have been used in the cement slurry to control the settling, fluid loss and free water separation, to reduce porosity and permeability as well as prevent fluid migration. In cement slurry, the polymers are absorbed in the hydrating cement particles that make a polymeric film in front of the cement particles. This absorption of the polymer in cement slurry reduces permeability [55]. The polymers are being used as viscosifying agent that increase the cohesive force between the particles of the cement and prevent the sedimentation of heavier particles of the cement.

Polymers such as Polyacrylamide [56], Polyvinyl alcohol (PVA), Polyethylene imine (PEI) [57], Polysaccharides group and Crosslinking polymer [58] have been widely used in cement slurries. In 1991 Allen et al., patented the Welan gum to be used as a fluid loss control additive. It showed uniform density in the cement slurry that prevented the settling of particles [59]. In 1993, K.K Ganguli reported that several biopolymers would control the free water completely and prevent the formation of channels in the cement column.

The use of biopolymers also has suspending characteristics that can prevent the settling and sedimentation in the cement slurry [10]. Synthetic polymers have also been used in cement slurries. These synthetic polymers have been used in slurry to control the fluid loss in lightweight slurry, weighted slurry and heavy weight slurry. A.F.Strange concluded that the use of synthetic polymer in cement slurry improves the displacement mechanism, minimize the bridging of cement particles, provides early casing protection and prevents gas migration [60].

2.5.1 Polysaccharides in Oil Well Cementing

Polysaccharides are being used to improve the API properties of cement slurry. The Polysaccharides and their derivatives include Hydroxyethylcellulose (HEC), carboxymethyl hydroxyethylcellulose (CMHEC), Guar, Hydroxypropylguar (HPG), Starch, Xanthan, Welan gum, Scleroglucan, Diutan and Carrageenan polymers. They are being used as viscosifying agents and multifunctional additives in cement slurries [4]. The increase of viscosity and particle suspension ability are the most common properties of polymers.

The polysaccharides are used to prevent fluid loss, free water and gas migration through the cement slurry. The polymers that are being used in the oil industry undergo shear thinning or thermal thinning problems. The other polymers are susceptible to thermal, chemical and mechanical degradation resulting in the loss of viscosity [61]. The polysaccharides HEC, HPG and Guar are random-coil type polymers and are widely used in oil well cementing. These types of polymers undergo thermal thinning problems at high temperatures. The viscosity of these polymers sharply reduces with respect to temperature and then the polymer loses the desired properties of cement slurry [62]. An excess concentration of polymers can be used to increase the viscosity at high temperatures but it will also increase the viscosity of slurry at surface condition [63]. The viscosification of cement slurry at surface condition requires high pump pressure. This becomes the cause of formation fractures in the case when the fracture pressure becomes equal to the pore pressure.

Recently polysaccharides were modified with carbonates and other chemicals to increase the viscosity of polymer with increase in the temperature [4]. The use of chemicals with polymers increases the cost of the operation. It may also affect the properties of the cement and other additives. Therefore, it is necessary to use such type of polymers that increase viscosity with the increasing temperature without addition of any chemicals.

2.6 Cellulose Ethers and Their Derivatives

Cellulose ethers are cellulose type water soluble polymers. The cellulose is natural and most abundant. For more than 50 years, cellulose polymers have played an important role in different fields of applications. The cellulose ether and their derivatives are used as film formers, thickeners, binders, suspension aids and water retention agents. The cellulose ether also functions as lubricants, permeability reducers, surfactants and emulsifiers [64]. In addition, the solution of cellulose ether is stable at high temperature and has thermal gel. The thermal gel is unique property of cellulose ether in all types of polymers and it plays a key role in high temperature applications.

Cellulose ether is available in two basic types, i.e., Methylcellulose and Hydroxypropylmethylcellulose (HPMC). In this research, HPMC polymer has been used. Therefore, the HPMC polymer has been defined briefly.

2.6.1 Chemistry of HPMC Polymer

Cellulose ether is manufactured by heating the cellulose fibre with caustic solution and treating it with methyl chloride. This reaction yields the methyl ether of cellulose. The reacted product is purified and ground to a fine and uniform powder. HPMC is a natural carbohydrate and it contains the repeating structure of anhydroglucose unit as shown in Figure 2.3.

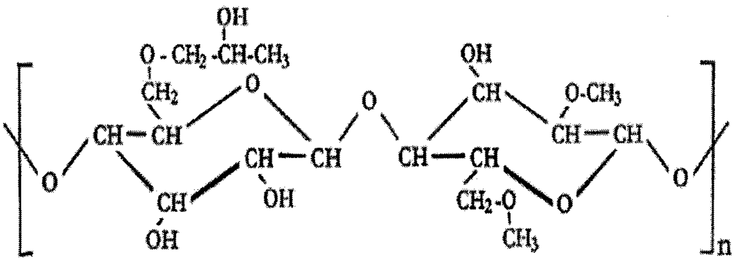


Figure 2.3: HPMC Chemical structure with degree of substitution of 1.5 [65].

In the preparation of HPMC, propylene oxide is used in addition to methyl chloride to obtain the substitution of hydroxypropyl on the anhydroglucose unit. The substitution –OCH2CH (OH) CH3, contains secondary hydroxyl on the second number carbon chain and is also considered to form propylene glycol ether of cellulose. This product contains varying ratios of hydroxypropyl and methyl substitution. This varying ratio affects the organic solubility and the thermal gelation temperature of HPMC solution.

The amount of substitute group on cellulose ether is characterized by degree of substitution (DS) and molar degree of substitution (MS). The number of OH groups of an anhydroglucose unit that are etherified is called the degree of substitution [66]. The molar degree of substitution is the average numbers of substituent groups per anhydroglucose unit. The etherification in HPMC polymer occurs at free OH group from the hydoxypropylation and develops side chains. Therefore, the molar degree of substitution of hydroxypropyl has values of greater than 3 [65]. The DS and MS are very important characteristics of cellulose ether for solubility in water. Li et al., showed that the sample of HPMC polymer had DS range of 1.4 to 2.0. This was easily soluble in water and it showed a thermal reversibility solution gel at different temperature ranges. The increasing value of DS affected the solubility of HPMC in water. Furthermore, it was observed that as the DS value increased the solubility in the water was decreased [67].

2.6.2 Properties of HPMC Polymer

The thermal gelation of HPMC is unique and important property. The solution of HPMC polymer is converted into gel at certain temperature. The gel of HPMC is completely reversible and gains the original shape of the solution upon cooling [68]. As the temperature of the solution increases, the dehydration process starts and the viscosity of solution decreases. At a specific temperature, when the gelation point is reached, sufficient dehydration of the polymer occurs to cause the polymer to polymer interaction. then the solution begins to gel [69]. Upon cooling the polymer solution, the gel begins to reverse and the viscosity of solution decreases rapidly. At the end, the viscosity of the cooling solution begins to merge with the original. Furthermore, it increases the viscosity with increasing temperature [14].

The HPMC polymer has the property to increase its viscosity above gelation temperature. Like other polymers, the HPMC polymer decreases its viscosity with an increasing of the temperature. After this, when the temperature reaches the gelation temperature, the viscosity of solution starts to increase [15]. At gelation temperature, the HPMC polymer starts to create a gel network that is based on the hydrophobic interaction of the cellulose ether's methyl group as shown in Figure 2.4.

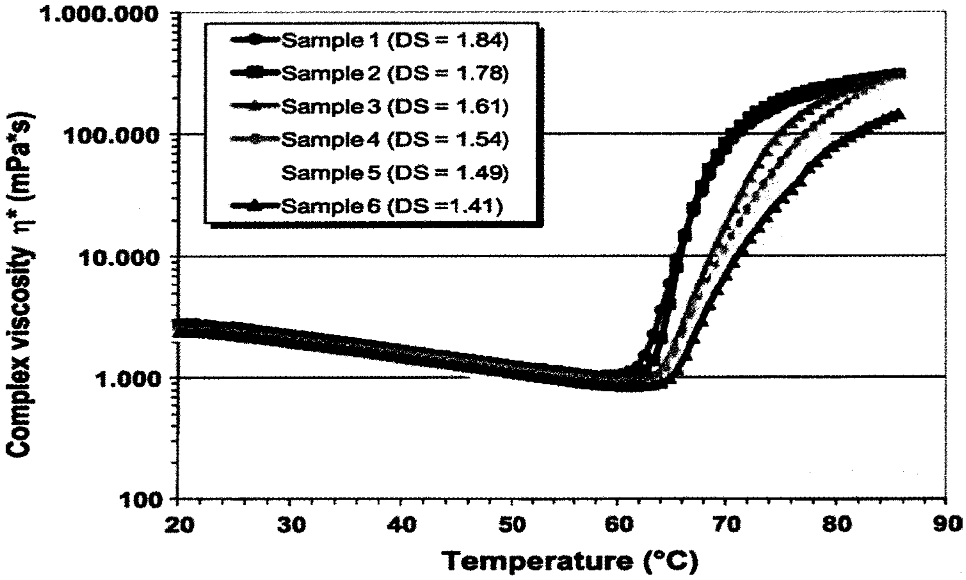


Figure 2.4: Viscosity versus temperature of HPMC solution [67].

The gelation temperature of HPMC polymer is depends upon the concentration and DS of the polymer. The high value of DS of the polymer showed a sharp change in viscosity. Moreover, for the sample of low DS, the viscosity increase was slightly shifted to higher temperature.

2.6.3 Applications of HPMC Polymer

HPMC polymer has a great demand due to its water solubility, gelation property and availability in a wide range of molecular weights [64]. HPMC polymer has wide range of applications in hydrocarbons research and production, preparation of food, ceramic processing, construction materials and biomedical.

HPMC polymer has been used in motor cement since the last decade. In cement, the HPMC polymer has been used to increase the bond strength between the steel and cement [70], decrease the thermal expansion [71], and increase the tensile strength, ductility and compressive module [72]. The HPMC polymer increases the setting time and retards the cement hydration [73]. In mortar cement, the HPMC polymer improves the fresh mixture properties. The modified cement with HPMC polymer has shown higher water retention than unmodified cement [74]. The water retention of HPMC in cement is due to the thickening property and enhanced viscosity of the polymer. The hydrophilic part of HPMC fixes the molecules of water, which prevents the evaporation and absorption into the porous media [75]. As HPMC acts as a viscosifying agent it prevents the free water separation, decreases the particle segregation and improves the cement homogeneity [76].

Recently, the HPMC polymer was used in laboratory, where HPMC polymer was used as a thermoreversible gel for conformance control. Through the laboratory analysis it was concluded that HPMC polymer has greater long term thermal stability and lower viscosity at ambient temperature than other used polymers. Through the experiment of sandpack flow, it was also concluded that it was an excellent permeability reducing agent [77].

CHAPTER 3

METHODOLOGY

This chapter consists of three sections, material description, preparation of materials and experiments. The materials section describes the selection of additive, cement and polymer. The second section presents the preparation method of HPMC solution and cement slurry. It also describes the preheating procedure of the slurry. The experiments are based on the viscosity measurement of HPMC solutions, rheology measurement, fluid loss, free water, thickening time, compressive strength, static gel strength and gas migration measurement through cement slurry. The flow of research worked is given in Figure 3.1.

3.1 Materials

In this section the materials such as API class cement, additives and polymer are discussed. The preparations of HPMC solutions and cement slurries for experiments are also explained in detail.

3.1.1 Polymer

The cellulose ether has various derivatives such as, Hydroxyethylcellulose (HEC). Hydroxypropylmethylcellulose (HPMC), Methylhydroxyethylcellulose (MHEC), Methylcellulose (MC) and Carboxymethylcellulose (CMC). These derivatives are distinguished by their chemical structure and chemical reaction. In this research Hydroxypropylmethylcellulose (HPMC) was used for the prevention of gas migration in through cement slurry. HPMC polymer was available in various types, which have

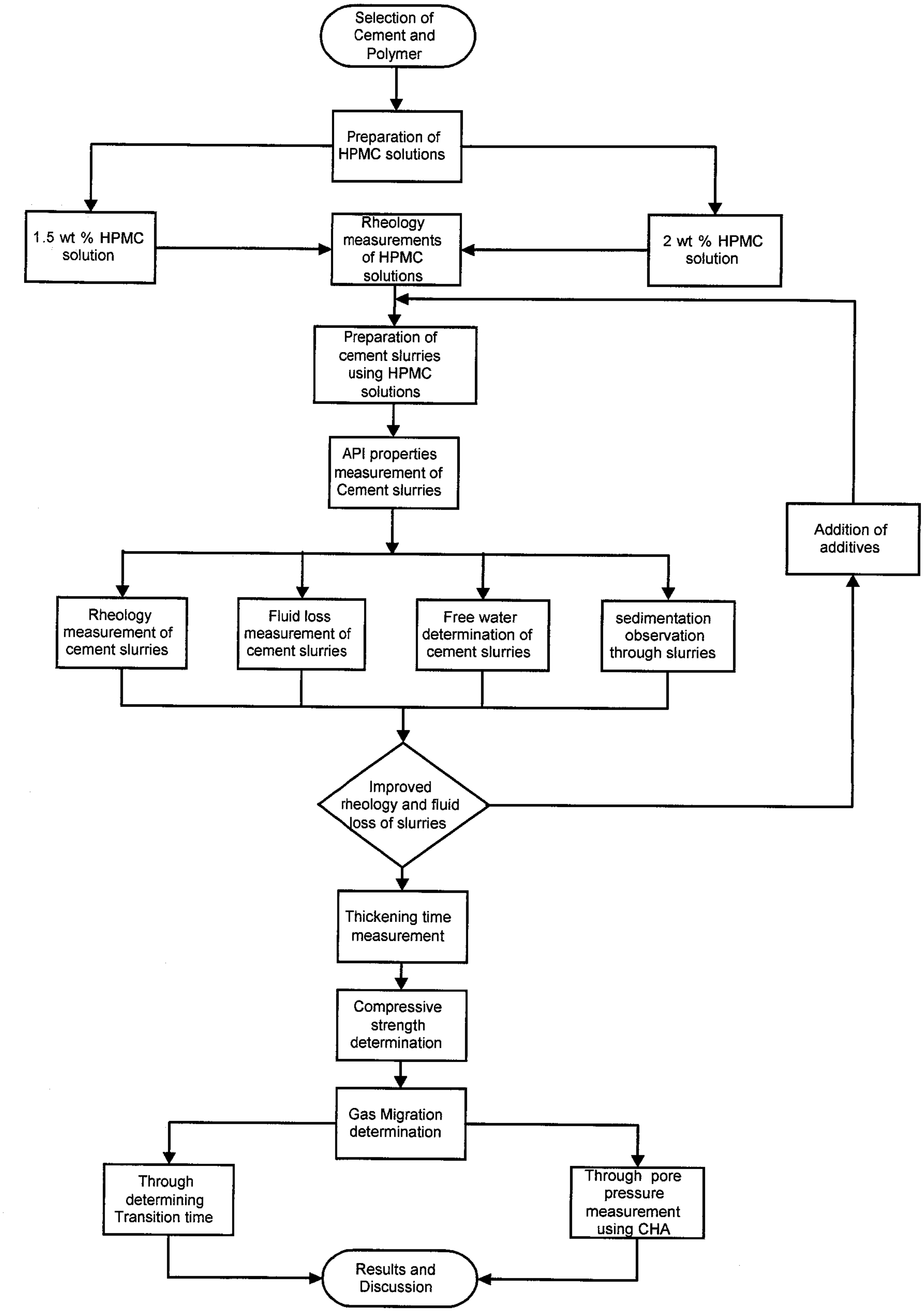


Figure 3.1: Flow Chart of methodology

different molecular weights, viscosities and particle sizes. The HPMC polymer was available in different ranges of typical properties. The viscosity range of HPMC polymer was 80 to 180000 cP. The product of HPMC polymers are known by their viscosities. On the base of literature and viscosity range, product PM-400 of HPMC polymer was selected for performing experiments. The HPMC polymer was provided by the Yillong Chemical Group Limited, China. HPMC polymer is non-ionic and water soluble polymer. The physical properties of PM-400 HPMC are given below in Table 3.1

Table 3.1: Physical properties of HPMC polymer.

Parameters	**Values**
Appearance	white fine powder
Methoxy content (%)	22-26
Hydroxypropyloxy Content (%)	5-11
Viscosity (cP)	400-500
PH Value	4.5-8.5

3.1.2 API Cement

The API class cements have been used in oil well cementing. There are eight different types of API class cements. All of the types of cement are differentiated by contents of chemicals and their percentages in the cement. Generally, all types of cements have been used in cement slurries. The selection of cement type from API classes depends upon temperature, density, formation characteristics etc. In this research, the class G oil well cement was selected for the preparation of cement slurry. The class G cement is High Sulphate Resistant (HSR) with the specific gravity of 3.20. It was supplied by Baker Hughes, Malaysia. The chemical composition of class G cement is given in Table 3.2.

Table 3.2: Chemical composition of Class G cement.

Chemical Composition	**Wt.%**
C_3S (tricalcium silicate)	62.5
C_2S (dicalcium silicate)	9.3
C_3A (tricalcium aluminate)	2
C_4AF (tertacalcium aluminoferrite)	14.5
$S0_3$ (Sulphur trioxide)	2.61
MgO (Magnesium oxide)	2.3
K_2O (Potassium oxide)	0.32
Na_2O (Sodium oxide)	0.46

3.1.3 Cement Additives

Cement additives are commonly used to improve the API properties of slurry. There were different types of additives. In this research, Dispersant (CD-33L), Defoamer (FP-9LS), Fluid Loss additive (FL66-L) and Retarder (R-21LS) were used for the experiments. All of the additives were in liquid form and measured in gps. The additives were provided by Baker Hughes, Malaysia. The specific gravity of additives is given below in Table 3.3.

Table 3.3: Specific gravity of cement additives.

Additives	**Specific Gravity**
Dispersant (CD-33L)	1.18
Defoamer (FP-9LS)	0.90
Fluid Loss additive (FL66-L)	1.06
Retarder (R-21LS)	1.08

3.1.4 Preparation of Materials

This section presents the preparation of HPMC solutions and cement slurries. The sample of HPMC polymer was in powder form. The wet solution of HPMC polymer was prepared in order to determine the rheology of HPMC polymer. Next, the solutions of HPMC polymers were used in cement slurry to achieve the desired objectives of this research.

3.1.5 Preparation of HPMC Solution

Two different concentrations of HPMC solutions were prepared. The solutions of 1.5 wt% and 2 wt% HPMC polymer were prepared using HPMC polymer with distilled water. The quantity 1.5 gm of HPMC and 98.5 ml of distilled water was used for preparing the 1.5 wt% HPMC solution. The second HPMC solution of 2 wt% was prepared using 2 gm of HPMC polymer and 98 ml of distilled water. The total volume of HPMC solutions was 100 ml. The specific gravity of HPMC solutions was 1.02.

3.1.5.1 Test Procedure

1. The measured quantity of distilled water was poured in baker and was kept on the hot magnetic plate.
2. The magnetic stirrer was placed inside the baker. The rotation of the stirrer was 4 to 8 rotation per minute (rpm) and was adjusted through the hot magnetic plate. The rotation of stirrer generated the vortex of water.
3. The measured quantity of HPMC polymer was sprinkled on the shoulder of vortex. The addition of the polymer was slow to avoid the lumping of powder.
4. The rotation of magnetic stirrer was continued for 3 to 4 hours at the temperature of 40 °C in order to completely dissolve the polymer in water.

3.1.6 Preparation of Cement Slurry

The equipment high-shear blender type mixer (Constant Speed Mixture) as shown in Figure 3.2 was used to prepare the cement slurries. The quantity of 600 ml of the cement slurry was prepared for each slurry. The materials were mixed in the blender which consisted of bottom driven blades. The procedure for preparation of cement slurry according to API RP 10B-2 is given below in steps.

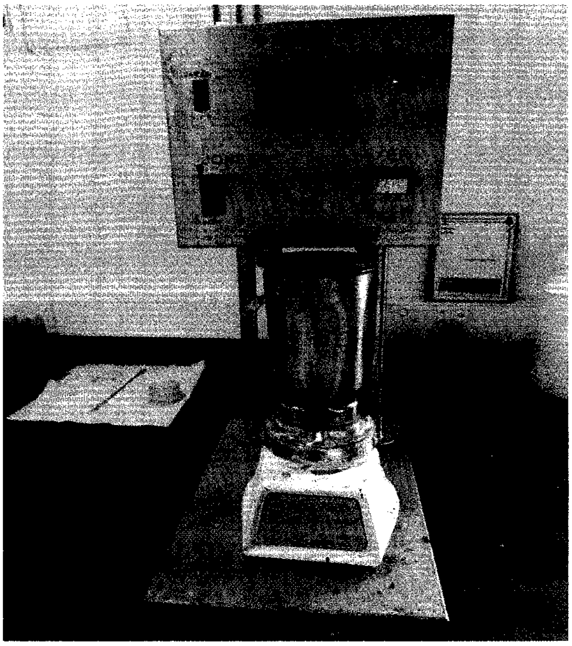


Figure 3.2: Constant speed mixture

3.1.6.1 Test Procedure

1. Prior to mixing, the cement, additives and distilled water were weighted in grams.
2. The weighted water and liquid additives were poured into the blender.
3. The mixer was switched ON and the rotation was maintained at 4000 ± 200 rpm to thoroughly disperse the liquid additive before the addition of the dry materials.

4. The cement was added in the blender within 15 seconds. Then, the cover was placed on the blender and mixing continued at 12000 ± 500 rpm for 35 seconds.

3.1.7 Preheating of Cement Slurry

The preheating of cement slurry is very important for performing the rheology, fluid loss and free fluid test. The rheology and free fluid test were performed at elevated temperature. Therefore, it was necessary to preheat the slurry at 90 °C. The equipment Atmospheric Consistometer was used for stirring and conditioning the cement slurry. The main operating parts of the Atmospheric Consistomer are rotating cylinder, slurry container, stationary paddle assembly and liquid bath. The Atmospheric Consistomer is shown in Figure 3.3.

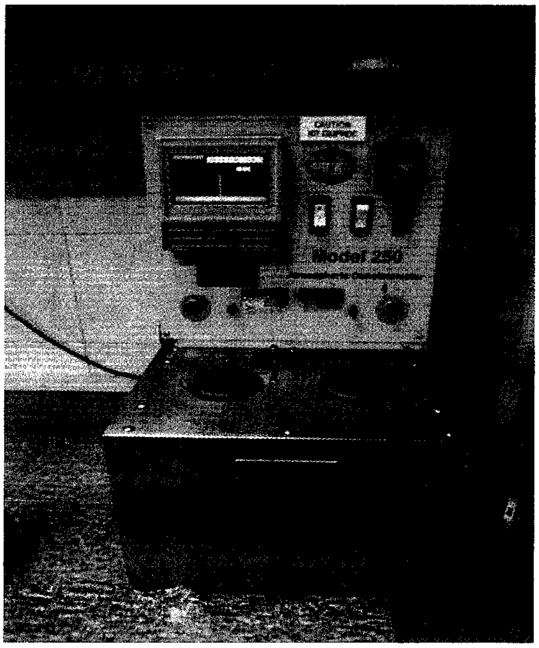


Figure 3.3: Atmospheric consistometer

3.1.7.1 Test Procedure

1. Prior to starting the preheating of cement slurry, the level of the fluid was checked in the bath. The level of the fluid was covered at the top level of cylinder.

2. The temperature of the liquid bath was adjusted through the use of up and down arrows on the front screen.
3. The heater was switched ON and the paddle was placed in the cylinder.
4. The rotational speed of motor was set at 150 rpm using hand tachometer.
5. The cement slurry was prepared and poured into the cylinder up to the level marked inside the cylinder.
6. The lid was placed on the top of test cylinder over the end of the paddle. It was rotated clockwise and moved downward until the pins engaged in the torque shaft.
7. The complete assembly of test cylinder was placed into the bath and it was locked using the pins with the slot on the rotor.
8. The motor was switched ON and the time for preheating was set for 20 minutes.

3.2 Experiments

This section provides the test objectives, procedure and equipment for each experiment in detail.

3.2.1 Viscosity Measurement of HPMC Solutions

This section describes the procedure for viscosity measurement of HPMC solutions. The viscosity of 1.5 and 2 wt% HPMC solutions was determined at different temperature ranges with respect to shear rates.

3.2.1.1 Viscometer

The viscosity of HPMC polymer was measured by using High Temperature and High Pressure viscometer (HTHP) of OFITE (Model 1100) as shown in Figure 3.4. HPHT viscometer could determine the viscosity at the maximum temperature to 260 ^{0}C and pressure of 2500 psi. The viscometer contains couette coaxial cylinder with combination of bob and rotor. HPHT viscometer has different combinations of sets of rotor and bob. R1B1 set is usually used and the required volume for this set is 42 ml. The sample contained cup was attached with the rotor and it moved around the bob. When the shear rate is applied, the fluid in the annulus between rotating cup and bob generates the torque. It converts in the shear stress and calculates the shear viscosity through automatic data acquisition system of ORCADA TM software.

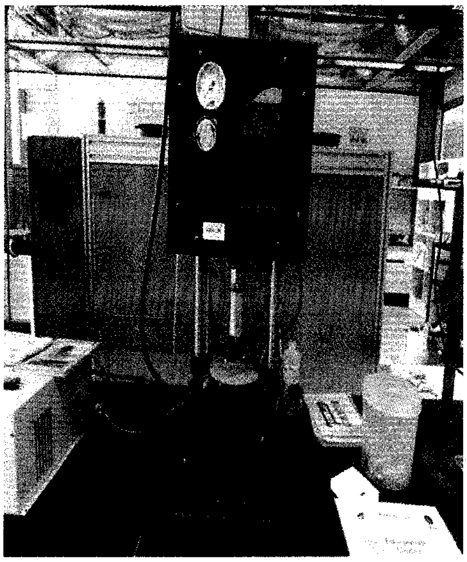


Figure 3.4: Viscometer model OFITE 1100

3.2.1.2 Test Procedure

1. Prior to performing the viscosity measurement, the viscometer was calibrated using standard fluid of 200 cP.
2. The thermocouple was passed through the bob. The thermocouple was kept straight in order to obtain the accurate data of temperature.
3. The bob was installed in the rotor. Then, the sample was poured into the cup.
4. Accordingly, the rotating cup was installed in the viscometer.
5. The desired temperature was adjusted through ORCADA software. The test run was defined in term of geometry shear rate and temperature. The shear rate varied from 1 to 1000 s^{-1}.
6. The test run was executed as the temperature reached desired value.
7. Once the test was completed, the data was imported to MS Excel.

3.2.1.3 Measurement Plan of Viscosity

The viscosity of HPMC solutions (1.5 and 2 wt%) was determined at different temperatures. The minimum shear rate was 1 s^{-1} and the maximum shear rate was 1000 s^{-1}. The first run was conducted on HPMC polymer of 1.5 wt% at 30 ^{0}C. The second test run was of 1.5 wt% concentration at 40 ^{0}C. The third was at 70 ^{0}C. This process was performed continuously until 100 ^{0}C with increments of 10 ^{0}C. In the same way, tests were also conducted using the 2 wt% concentration of HPMC polymer at 30 to 100 ^{0}C.

3.2.2 Density Measurement of Cement Slurries

The density of the designed cement slurries was determined using Pressurized Mud Balance according to API RP 10B-2 [78]. The density of cement slurries was determined in ppg. The slurry cup, sliding weight, fulcrum and pressurizing plunger were main parts of pressurized mud balance. The equipment used for density measurement is shown in Figure 3.5.

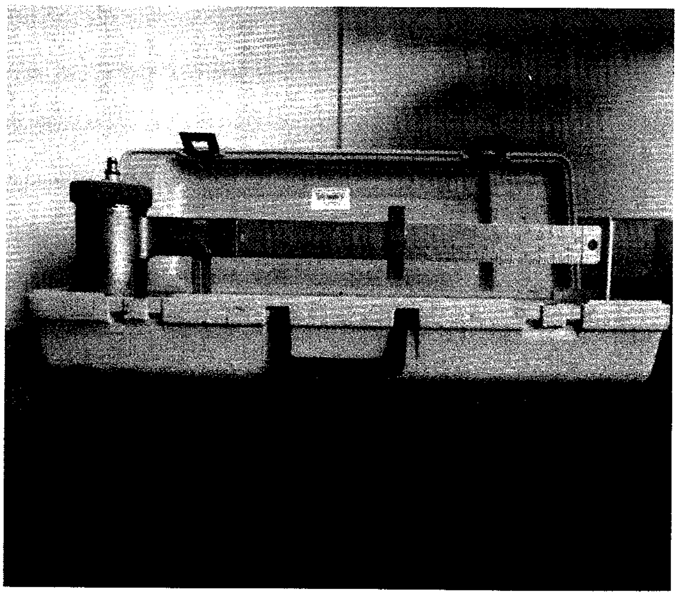


Figure 3.5: Pressurized mud balance

3.2.2.1 Test Procedure

1. The cement slurry was poured into cup. In next, the pressure cup was screwed on.
2. The pressurizing plunger (same as syringe) was filled with cement slurry and attached to the cup.
3. The pressure was applied in order to collapse the air bubbles in the slurry.
4. Next, the device was placed on the fulcrum and the weight was slide on the scaled rod of mud balance.
5. When both sides of the mud balance became balanced, the value of weight was noted down on scaled rod. The value of weight in ppg was the density of cement slurry.

3.2.3 Rheology Measurement of Cement Slurries

The rheology of designed cement slurries was determined at ambient temperature and 90 °C. The rheology of cement slurries at ambient temperature was determined directly after the preparation of slurries. The cement slurries were preheated using Atmospheric Consistometer for rheology at 90 °C. The procedure to preheat the slurry was described previously in section 3.1.7.1.

3.2.3.1 Rotational Viscometer

Rotational viscometer is standard equipment used to determine the rheology of cement slurries. Fann Rotational Viscometer (Model 35) was used to determine the rheology of cement slurries. The rotational viscometer is designed with rotating cylinder (couette type). The rotational viscometer contains R1 rotor sleeve, B1 bob, F1 tension spring and steel slurry cup. The volume 350 ml of slurry is used to determine the rheology of slurries. The viscometer consists of six dials of shear rates. The rheology of slurries was determined in terms of shear stress by giving different shear rates. The rotation viscometer is shown in Figure 3.6.

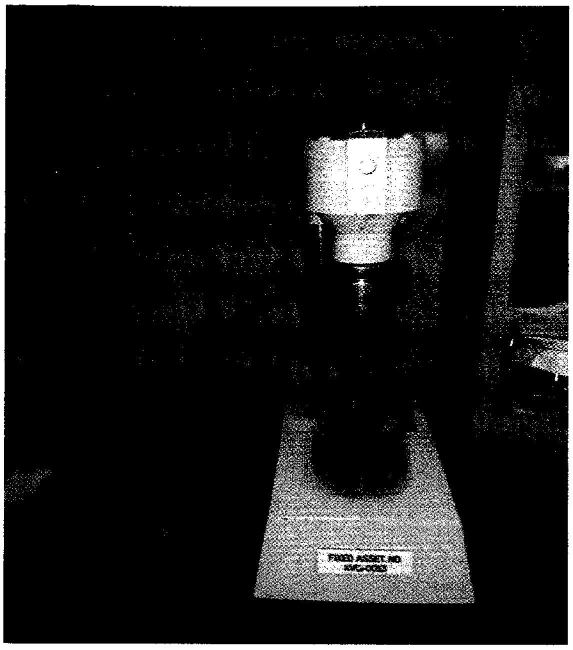


Figure 3.6: Fann 35 rotational viscometer

3.2.3.2 Test Procedure

1. The cement slurry was poured into stainless steel sample cup.
2. The slurry cup was placed on plate of viscometer and raised up until the slurry level reached the inscribed line on the sleeve of rotor.
3. The switch of viscometer was set at low speed. The motor was turned ON and the gear shift knob was moved to 300 rpm. The rheology (shear stress) was noted on the top dial.
4. The switch was changed to high speed. The gear knob was turned to 200 rpm for taking the next reading.
5. In the same way, the speed was changed alternatively high to low and the knob was turned to 100, 6, 3 and 600 rpm respectively.

3.2.4 Gel Strength Measurement

The gel strength is one of most important part of rheology. The gel strength of slurries was determined after the rheology measurement. It was measured using same

Rotational Viscometer. The operational procedure of gel strength measurement is given below.

3.2.4.1 Test Procedure

1. The cement slurry was poured into sample cup and fixed using same procedure described above for rheology test in section 3.3.3.2.
2. The cement slurry was stirred at 600 rpm high speed using the knob and the motor was turned in OFF position.
3. For 10 second or 10 minute gel strength, the motor was kept in OFF position for 10 seconds or 10 minutes.
4. The position of knob was set at low 3rpm and the motor was turned ON.
5. The reading was taken at the maximum deflection of the dial before the gel broke.

3.2.5 Fluid Loss Measurement

Fluid loss measurement is important for oil well cement slurry especially for prevention of gas migration. The test was designed to investigate the impact of HPMC polymer for controlling fluid loss through cement slurries. The cement slurries were prepared using various concentrations of HPMC solutions. The fluid loss through cement slurries was determined using High Temperature High Pressure (HTHP) Filter Press at 90 °C.

3.2.5.1 HTHP Filter Press

The OFITE HTHP Filter Press was used to determine static fluid loss through slurry according to API RP10B-2. The equipment consists of pressure regulators, pressure controlled source, high pressure test cell, heating jacket and test cell. The maximum temperature and pressure range of this equipment is 204 °C and 1500 psi respectively. The volume required for fluid loss measurement is 175 ml which was capacity of test cell of HPHT filter press. The fluid loss measured across the filtration medium of 325 mesh screen which is backed up by 60 mesh screen for 30 minutes of time interval. The equipment HTHP Filter Press for determination of fluid loss is shown in Figure 3.7.

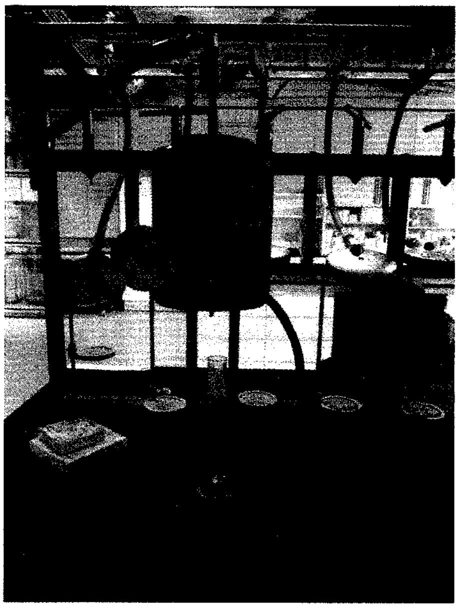


Figure 3.7: OFITE HTHP filter press

3.2.5.2 Test Procedure

1. The O rings, pressure gauges, control valve of nitrogen cylinder and relief valve were checked for safety and it was insured that all parts were in good condition.
2. The Power supply switch was turned ON and the dial of temperature was adjusted in order to increase the temperature of the heating jacket.
3. The thermocouple was inserted in the heating jacket for checking the temperature.
4. The test cell was filled with preheated slurry to proper level of volume. The screw of cell was tightened and both of the pressure relief values were closed using Allen wrench.
5. The test cell was placed in the cabinet of heating jacket. It was ensured that filtrate side of cell was downside.

6. The test cell was rotated until it was placed properly in the jacket. The thermocouple was removed from jacket and inserted into the top of test cell for checking the temperature of slurry inside the cell.
7. The nitrogen line was connected to top of cell. The pressure was adjusted to 1000 psi using pressure regulator.
8. The valve of test cell was opened from the top in order to pressurize the cell.
9. The graduated cylinder was placed under the bottom valve of cell. It was opened slowly till one and half turn and immediately the timer was started.
10. The reading of fluid loss was taken after five minute interval until 30 minutes in terms of millilitres.
11. After 30 minutes, the filtrate loss was noted and it was the final fluid loss.
12. Lastly, the bottom valve and nitrogen pressure valve were closed and the heater was turned off.
13. In some tests the slurry blew out before 30 minutes. In this condition, the experiment was stopped and the volume of filtrate in the graduated cylinder and total time was noted at the time of blow out.
14. The fluid loss in blow out condition was determined using given formula below.

$$\text{API Fluid Loss} = 2\,V_t \frac{5.477}{\sqrt{t}} \qquad (3.1)$$

where

V_t Volume of filtrate in ml

t Time in minutes

3.2.6 Free Water Measurement

The free fluid through cement slurries was determined using graduated cylinder according to API criteria. The aim of determining free water was to observe the effect of HPMC polymer on cement slurries for controlling free water. The fee water measurement graduated cylinder is shown in Figure 3.8.

Figure 3.8: Free water measurement graduated cylinder

3.2.6.1 Test Procedure

1. The volume of 250 ml of preheated cement slurry was poured in 250 ml graduated cylinder and was covered using aluminium film for protection and prevention of evaporation.
2. The graduated cylinder was kept at the inclination of 45 degree according to the API criteria and the slurry filled graduated cylinder was left for 2 hours.
3. After 2 hours, the separated free fluid from the top of slurry was removed through syringe or pipette and was measured in millilitres. The measured quantity of fluid was the total volume of free water.

3.2.7 Thickening Time Measurement

The thickening time test of cement slurries is designed to determine the time in which slurry remains in liquid and pumpable state. The thickening time of slurries measured in term of consistency over time. The consistency is measured in Bearden units (Bc). The maximum consistency of cement slurries is 100 Bc. At this consistency the cement slurry becomes hard and cannot be pump. The time required to reach the maximum consistency of 100 Bc is thickening time. The aim of this test was to determine the effect of HPMC polymer on the thickening time.

3.2.7.1 HPHT Consistometer

The Model 40-600 Pressurized HPHT Consistometer of CTE was used to determine the thickening time of cement slurries. Pressurized Consistometer determine the thickening time at maximum temperature of 204 °C and 25000 psi pressure. The Consistometer consists of cylindrical slurry cup assembly, stationary paddle assembly, pressure vessel, oil chamber and hydraulic system. The heat is supplied by internal tubular heater which is controlled by automatic temperature control system program. The pressure setting is controlled by the pressure release valve. The consistency of cement slurry is indicated in the electronic digital panel meter and is also recorded on the chart as DC voltage generates from the potentiometer. The consistency is generated on the graph by the data acquisition system. The equipment HPHT Consistometer is shown in Figure 3.9.

Figure 3.9: HPHT consistometer

3.2.7.2 Test Procedure

1. Prior to starting the experiment, it was confirmed that all front panel valves were close.
2. The parts of cylindrical slurry cup were greased and assembled.
3. The cement slurry was poured into the slurry cup and the open side of cup was tightened and closed.
4. The power switch and motor were turned ON and the slurry cup was placed in the pressure vessel where it was be in rotation in the vessel.
5. The bail was attached to the top of potentiometer and it was placed the pot mesh of potentiometer in the pressure vessel above slurry cup.
6. The slurry cup and pot mesh were properly engaged. It was observed that the digital meter was increasing the consistency. It confirmed that the pot mesh was properly set on the slurry cup.
7. The lid was placed on the pressure vessel and closed. The thermocouple was inserted in pressure vessel.
8. The air supply valve was opened in order to transfer the oil in the pressure vessels. The oil was escaping from the thermocouple. Therefore, the valve of thermocouple was tightened.
9. The program (temperature and socking time) was set using digital monitor screen. Lastly, the heater was turned ON and the test was started using touch screen.
10. The desired pressure was adjusted frequently until the maximum consistency was achieved. At the end of test, the air supply valve was turned to the closed position and the remaining valves were opened.

3.2.8 Compressive Strength Measurement

Compressive strength is a mechanical property of cement slurry. There are two ways of determining compressive strength. One is destructive, the compressive strength of a hard specimen (cement sample). The second is non-destructive, the compressive strength of cement slurry. In this research, the non-destructive method was used to determine the compressive strength of cement slurry under bottom hole conditions.

The compressive strength of cement slurry is determined ultrasonically. It determines the time interval of velocity change and ultrasonic energy change under wellbore conditions of temperature and pressure.

3.2.8.1 Static Gel Strength Analyzer

A breakthrough instrument, Static Gel Strength Analyzer (SGSA) Model 5265 of Chandler Engineering was used to determine compressive strength and gel strength (transition time). This equipment determines compressive strength and gel strength simultaneously under curing conditions. The working principle of SGGA is based on the measuring of change in energy level as ultrasonic signals pass through the cement slurry under given temperature and pressure. The instrument SGSA collects the ultrasonic signals that passed through the slurry. Next, it performs the post processing of the data that determine the compressive strength versus time and static gel strength in terms of transition time versus time plot. SGSA has operational limit of 204 °C temperature and 20000 psi pressure. The equipment consists of test cell, pressure valves, cell body, transducer and thermocouple. The volume of 200 ml was used in test cell for experiments. SGSA has data acquisition software that converts the results in real time graphs. The equipment SGSA is shown in Figure 3.10.

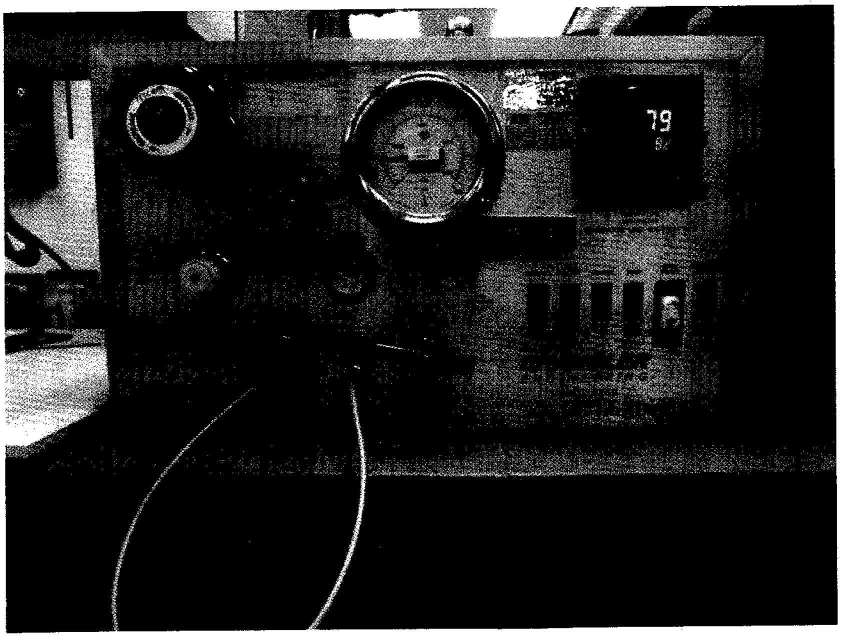


Figure 3.10: Static gel strength analyzer (SGSA)

3.2.8.2 Test Procedure

1. The sealing components and both O rings of the lid of test cell were checked. The test cell and threads of both lids were greased with silicone grease.
2. The bottom plug lid was placed in the vice. In the next step, the transducers were installed and the bottom plug lid was screwed with cylinder test cell end.
3. It was confirmed that market T arrow was at the top side and then it was tightened by hand.
4. The cement slurry was prepared according to API RP 10B-2.
5. The fill gage was kept in the test cell. The cement slurry was poured in and it was filled up to the middle of the first mark on the test cell.
6. The water was injected on the top of cement slurry till the second mark of fill gage and the gage was taken out from test cell.
7. The top lid was placed on the test cell and it was tightened using cell wrench.
8. The test cell was placed in the heater assembly of SGSA. Next, the bottom transducer was passed from the heater assembly and was fixed in the front panel of equipment.
9. The U shaped pressure pipe was attached with the top lid and top of analyzer.
10. The top transducer cable was connected to the BNC connector of the autoclave. The thermocouple was installed in the high pressure pot of top lid.
11. The water supply valve was turned ON until the water came out from the thermocouple vent hole. Then, the thermocouple connection was tightened.
12. The pump switch was turned ON and the pressure was adjusted to approximately 100 to 200 psi using pressure adjust valve. The desired pressure was adjusted using large pressure regulator.
13. The temperature and ramp time were set using the program temperature controller.
14. The heater switch was turned ON from the front panel SGSA and the temperature program was activated.
15. The test was ready to determine the compressive strength, transition time and gel strength.

3.2.9 Static Gel Strength Measurement

Gel strength measurement is critical to determine the gas migration through cement slurry. It evaluates the tendency of slurries to allow inflow of fluid. For gas tight conditions, the slurry should gain the gel strength of 500 Ib/100 ft^2 in less than 30 minutes according to API criteria. As the gel strength of slurry increases 100 to 500 Ib/100 ft^2, the hydrostatic pressure decreases and the slurry loses the strength to prevent gas migration. After 500 Ib/100 ft^2, the cement slurry starts to convert to the solid state and it obtains enough hydrostatic pressure to control migration. Therefore it was necessary to determine the transition time (gel strength 100 to 500 Ib/100 ft^2) to evaluate the gas migration through slurry. The equipment SGSA was used to the determination of the transition time of cement slurry.

3.2.10 Gas Migration Through Cement Slurry Test

The effect of HPMC polymer for preventing gas migration has been determined in this section. The equipment Cement Hydration Analyzer (CHA) has been used to determine the gas migration through cement slurries.

3.2.10.1 Cement Hydration Analyzer

The model 7200 CHA of Chandler Engineering as shown in Figure 3.11 was used to determine the gas migration. The CHA has a closed system in which N_2 is injected from the bottom at any time during its hydration. The ability of gas migration is determined by whether or not the nitrogen gas injection pressure passes through the cement slurry. The gas migration is predicated by measuring the pore pressure. The gas will migrate through the cement column if the pore pressure increase continuously or becomes equal to the injection pressure of nitrogen gas. If the slurry is gas tight the pore pressure will continue to drop during hydration and possibly reach the vacuum. The CHA consist of disposable cylinder, transducers, gas supply valve, air supply valve, pressure gauges, regulators and gas injection H_2O purge valve. The total volume of 417 cc was used for experiment of gas migration in CHA. The maximum operating temperature of CHA is 163 °C. The maximum operation pressure of gas inlet is 3000 psi, water inlet 160 psi, air supply maximum 160 psi.

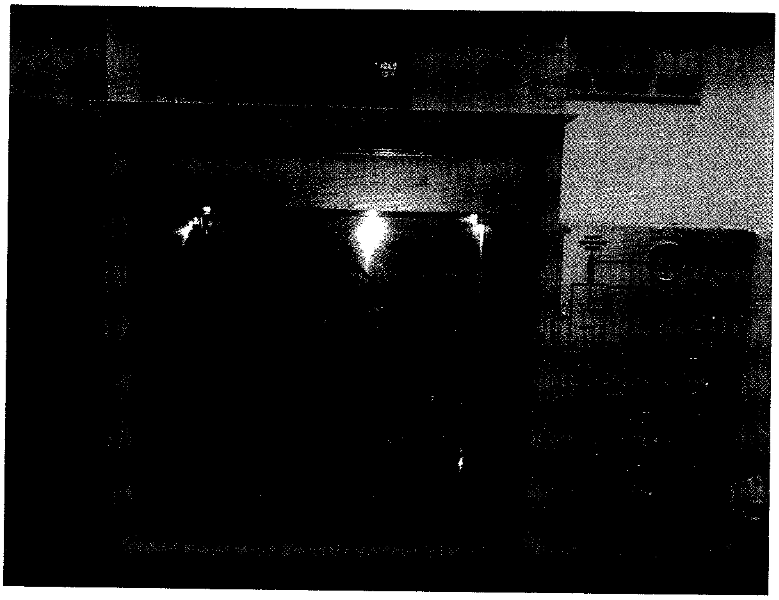


Figure 3.11: Cement hydration analyzer (CHA)

3.2.10.2 Theory of Operation

This section describes the operating mechanism of CHA. The disposable cylinder has been filled with slurry and placed in oven. The temperature of cement increased with increasing oven temperature. The increase in the cement temperature is adiabatic condition because the temperature of cement will increase continuously until hydration is complete. Then it becomes constant. Since, the cement slurry inside the cylinder is pressurized by opening the H_2O confining pressure at 350 psi on the opposite side of diaphragm. With the opening of H_2O confining pressure the cement starts to shrink and decreases the pressure. The shrinkage of cement slurry compensate by the movement of diaphragm that adjusts the pressure and kept the slurry in pressurized condition. For the experiment of gas migration using CHA, the gas injection pressure should be less than H_2O confining pressure. The gas injection pressure has been set at 150 psi and pore pressure set at 350 psi. With the opening of confining valve the pore pressure starts to decrease continuously. After some time, when the pore pressure has reached pre gas injection pressure (150psi), the H_2O confining pressure valve has been closed and the gas injection valve became open.

The CHA control software automatically operates both valves and the gas is allowed to enter the cell. After the injection of the gas, if the pore pressure increases or remains same as the gas injection set value, the gas migrates through the cement. On the other hand, if the pore pressure decreases continuously and becomes zero or constant at the low pre pressure that means the gas has not migrated.

3.2.10.3 Test Procedure

1. The CHA vessel was disassembled through the unit. The diaphragm/stem assembly was cleaned and reassembled.
2. The lower end cap, O ring and diaphragm assembly were submerged in the water container and all the parts were reassembled underwater.
3. The retainer was placed on the top of the diaphragm. It was clamped and the diaphragm was sealed.
4. The assembly was removed from the water. Then the water was injected using syringe in order to fill the entire course throughout the ports.
5. The "O" ring was greased and installed in the grooves of the retainer.
6. The complete assembly of lower end cap, diaphragm and retainer was placed into the stand inside the oven unit of CHA.
7. Another "O" ring was grease and installed in the groove of the upper end cap.
8. The cement temperature RTD was covered with TFE tubing and installed on the upper end cap through the tee and was tightened.
9. The metal wafer (for protection), RTD, and upper end cap were placed in the oven but it was not tightened.
10. A temporary connection was made between the water purge tube and the gas injection tubing inside the oven for checking the pressure and leakages.
11. The temporary circuit was pressurized with water by closing the water confining and gas injection valve
12. The water backflow, flush out air from the vessel stem and gas injection tube were checked out through opening the gas injection water purge valve to drain position

13. The gas injection water purge valve was closed when the water started to come out. The 3- way valve was also closed and the temporary connection was disassembled
14. The gas injection tubing was connected to the gas injection fitting and it was sealed by tightening the tube fitting.
15. The oven door was closed and the oven was preheated to the temperature 90 °C.
16. The cement slurry was prepared according to API RP10B-2 and the slurry was poured into the tube of the vessel.
17. The upper end cap with O ring and RTD with wafer assembly were placed on top of sample tube.
18. The tubes were connected to the water Fill / Purge port and pore pressure port. It was sealed by completely tightening the tubes.
19. The oven door was closed and the temperature was changed according to the desired test temperature.
20. The 3 way valve was opened slowly, which allowed the water to fill the top of the vessel above the cement and water.
21. In the next step, the water purge valve was opened and it filled the water above the cement in the chamber. The water purge and 3 way valve was closed when all the air came out from the cell.
22. The water confining valve was opened and the confining pressure was adjusted to the desired test pressure.
23. The pore pressure and the gas injection pressure were set and the test was run for 24 hour time period.

CHAPTER 4

RESULTS AND DISCUSSION

In this chapter, the rheological properties of HPMC solutions are discussed under the effect of concentration and temperature. The API properties of HPMC based cement slurries are also discussed. Further it is followed by gas migration test using HPMC solution in slurries and analyzes the effect of polymer for controlling gas migration.

4.1 Rheological Measurements

In this section, the flow viscosities of HPMC solutions have been measured according to the plan as discussed in section 3.2.1.3. The effects of concentration and temperature have been determined and are discussed below.

4.1.1 Effect of HPMC Concentration on Viscosity

The effect of concentration on viscosities of HPMC solutions (1.5 wt% and 2 wt%) was determined at different shear rates. The temperature of viscosity measurement was 30 °C. The maximum shear rate for viscosity measurement was 1000 s^{-1} and the minimum shear rate was 1 s^{-1}. The graphical representation of HPMC solution with increasing concentration is shown in Figure 4.1. It was observed that the viscosities of two different HPMC solutions were decreases with respect to shear rate (1 to 1000 s^{-1}).

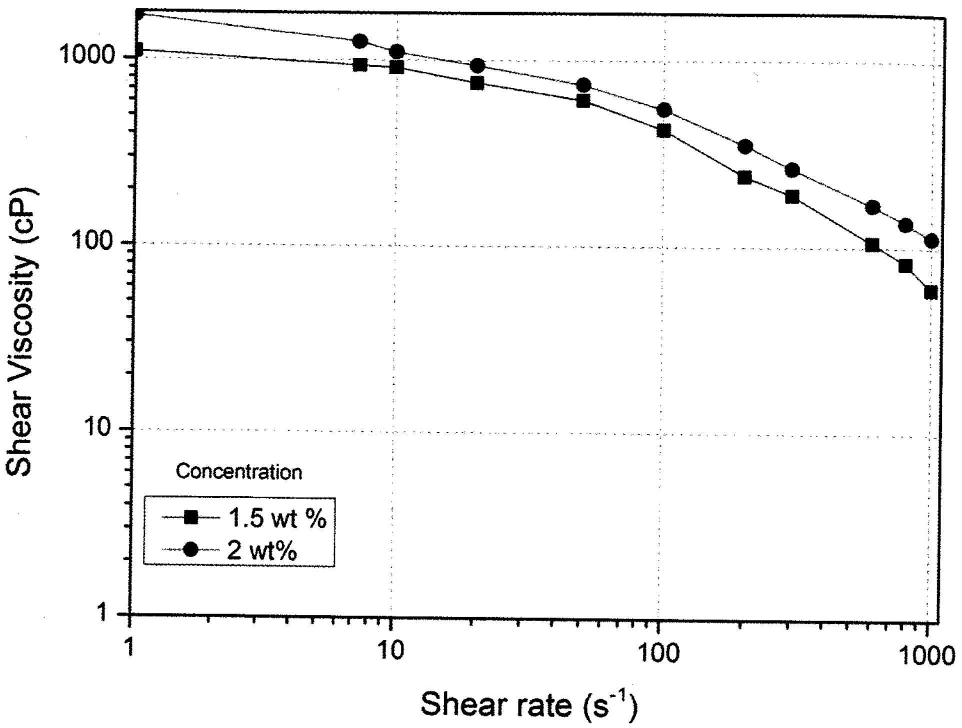


Figure 4.1: Shear viscosities versus shear rates of 1.5 and 2 wt% concentration of HPMC solution at 30 °C.

As the concentration of HPMC solution was increased from 1.5 to 2 wt%, the viscosity of solution was also increased from 1100 to 1723.7 centipoises (cP) at shear rate 1 s^{-1}. The Figure 4.1 shows that the increment of 0.5 wt% of HPMC solution increased the viscosity of solution by an average of 623 cP. This phenomenon of increasing the viscosity of HPMC solution depends upon the molecules of polymer. The increment in viscosity is attributed to the forces of attraction between the polymer molecules. The particles of polymer come close to each other and it increases the bond strength between the molecules. Therefore, the increase in concentration in term of wt% increases the viscosity of polymer.

The solutions of HPMC polymer showed non-Newtonian flow in viscometer experiment. It also showed that shear rate was inversely proportional to the shear viscosity of HPMC solution. It was also noticed that both solutions of HPMC polymer exhibited pseudo plastic behaviour. The consistency of flow behaviour start from 1s^{-1} shear rate and showed non linear graph.

4.1.2 Effect of Temperature on Viscosity of HPMC Solutions

The temperature is considered as a most important and critical parameter for reducing polymer viscosity. The viscosity of HPMC solutions was determined at different temperatures (30 to 100 °C) with respect to shear rate. The viscosity profile of 1.5 wt% HPMC solution at different temperatures is shown in Figure 4.2.

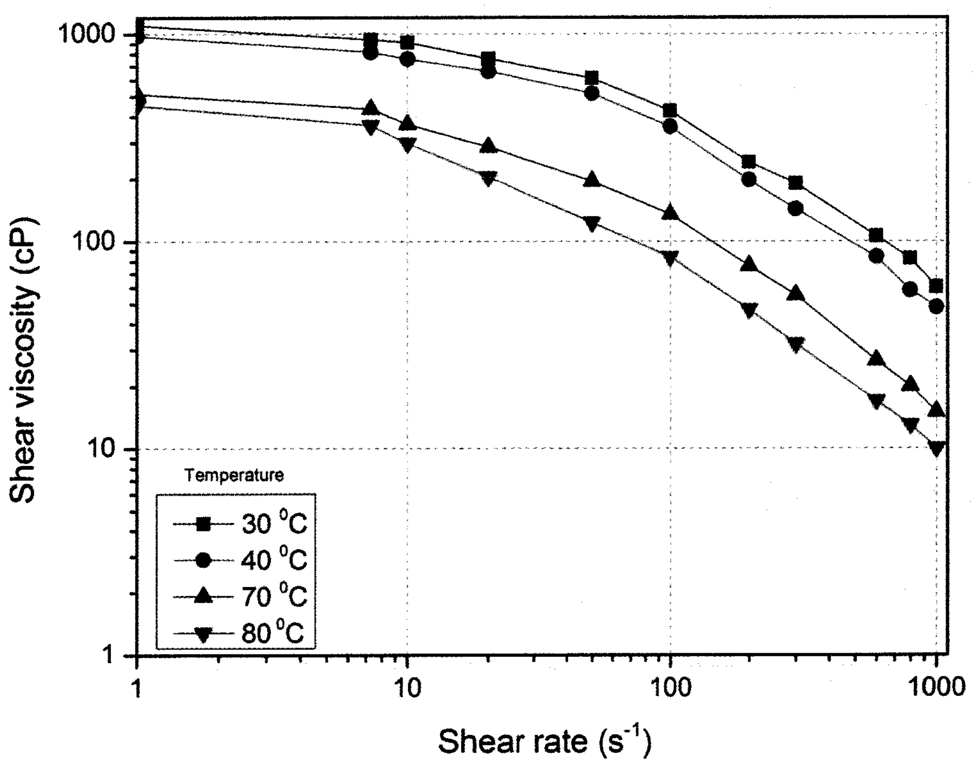


Figure 4.2: Viscosity of 1.5 wt% HPMC solution at different temperatures (30, 40, 70 and 80 °C) with respect to shear rate (1 to 1000 s^{-1}).

Figure 4.2 shows the viscosity of HPMC solution from 30 to 80 °C. The effect of temperature was inversely proportional to the viscosity of HPMC solution. The viscosity of HPMC solution is decreased with increase in the temperature (30 to 80 °C). In the same way, the viscosity of 2 wt% HPMC solution was also determined at different shear rates as shown in Figure 4.3.

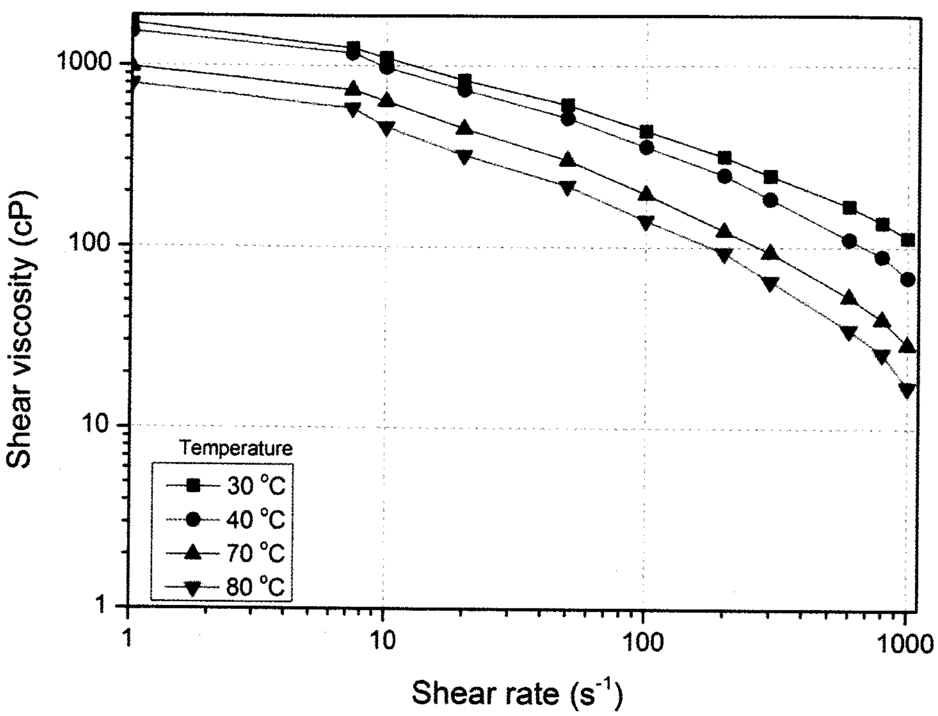


Figure 4.3: Viscosity of 2 wt% HPMC solution at different temperatures (30, 40, 70 and 80 °C) with respect to shear rate (1 to 1000 s^{-1}).

Correspondingly, Figures 4.2 and 4.3 showed that the viscosity of both solutions deceased with increase in temperature. As like other polymer, the HPMC polymer has thermal thinning behaviour by increasing temperature. In previous section, the shear thinning occurred by the change of longer ether chains into smaller ether chain with shear rates. Now, the shear thinning at elevated temperature was imposed by thermal fluctuation on the body of ether molecules. Under this condition, the temperature became another factor leading to the decrease in viscosity of HPMC solutions.

As the temperature of 1.5 wt% HPMC solution increases from 30 to 80 °C, the viscosity of solution decreased from 1100 to 451cP at 1 s^{-1} and 60 to 10 cP at 1000 s^{-1} shear rates. The average decrease in viscosity at 1 s^{-1} shear rate was 41% and at 1000 s^{-1} shear rate it was 16% at 80 °C. In the same way, the viscosity of 2 wt% solution decreased from 1723 to 793 cP at 1 s^{-1} and 113 to 17 cP at 1000 s^{-1} shear rates with the average of 46% and 15%. On the other hand it was observed that when the temperature was increased from 80 to 90 °C the viscosity of both solutions increased. The shear viscosity of 1.5 wt% solutions was increased from 451 to 590 cP at low shear rate and 10 to 21cP at high shear as shown in Figure 4.4.

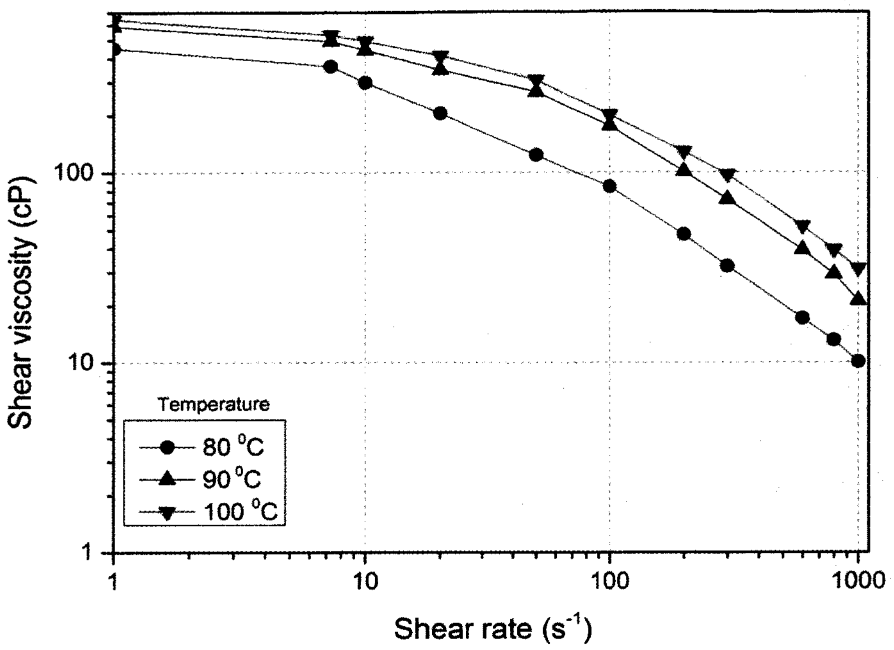


Figure 4.4: Viscosity of 1.5 wt% HPMC solution at 80, 90 and 100 °C temperature.

The viscosity of 2 wt% of HPMC solution was determined and is shown in Figure 4.5. Here, the viscosity of 2 wt% HPMC solution increased from 793 to 1160 cP at 1 s^{-1} shear rate and 17 to 37 cP at 1000 s^{-1} shear rate.

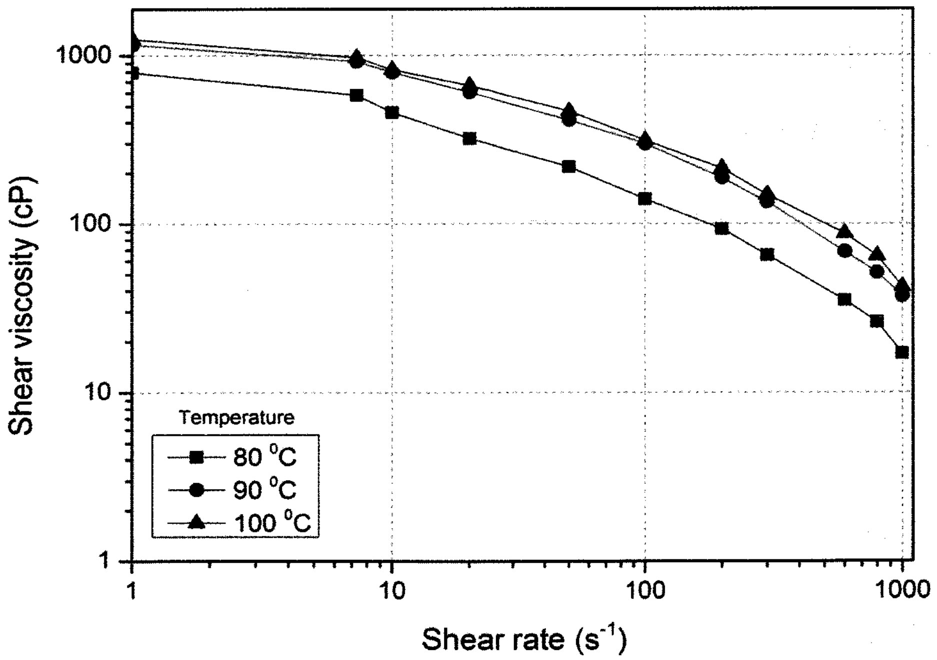


Figure 4.5: Viscosity of 2 wt% HPMC solution at 80, 90 and 100 °C temperature.

This was the property of HPMC polymer that above gelation temperature it will increase the gel strength and viscosity. The increase in viscosity of HPMC solutions was due to the hydrophobic interaction between the molecules of methoxyl group. With increasing temperature, the HPMC solutions lose their water of hydration and that becomes the cause of viscosity reduction. But at gelation temperature, sufficient dehydration of polymer occurs causing the polymer to polymer association and the solution begins to increase the viscosity. The amplified viscosity at gelation temperature is unique property of HPMC polymer and has not been observed in other cellulose type polymers. Further, it was also observed that at 100 °C the viscosity of both HPMC solutions was greater than the viscosity at 90 °C. This result shows that after gelation temperature, the viscosity of polymer will increase continuously as the temperature increases.

The literature showed that the polymers such as Xanthan gum, Guar gum, Hydrolyzed Polyacrylamides and other polysaccharides exhibited thermal thinning properties, loses their viscosity above 70 °C and degrades at high temperature. Besides this, the HPMC polymer also has the shear thinning behaviour until 80 °C. However, it increases viscosity at 90 °C. This illustrates that the HPMC solution has good thermal thickening property and it enhances the viscosity at 90 °C. Therefore, the HPMC polymer has more potential than other polymer to act as viscosifying agent at high temperature.

4.2 API Properties of Cement Slurries

The API properties were determined to analyze the effect of HPMC polymer on cement slurries. The rheology, fluid loss, free water, thickening time, compressive strength and static gel strength tests of HPMC based cement slurries have been discussed in this section.

4.2.1 Formulation of Cement Slurries

The cement slurries were prepared according to API criteria. The density of designed cement slurries were confirmed through the density measurement experiment as discussed in session 3.2.2. The experimental result shows that the density of cement slurries was 16.5 ppg. The cement slurries were composed of class G cement, HPMC solution, FLA, dispersant, retarder and defoamer. The formulation of all cement slurries depended upon the concentration of each material. The formulation of cement slurries is given below in Table 4.1 and 4.2.

Table 4.1: Formulation of 1.5 wt% HPMC based cement slurries.

Slurry	**Cement BWOC**	**Defoamer gps**	**Dispersant gps**	**Retarder gps**	**FLA gps**	**HPMC gps**
1	100	0.02	-	-	-	-
2	100	0.02	-	-	-	0.20
3	100	0.02	-	-	-	0.30
4	100	0.02	-	-	-	0.40
5	100	0.02	-	-	-	0.55
6	100	0.02	-	-	-	0.70
7	100	0.02	-	-	-	0.80
8	100	0.02	0.02	0.03	0.50	0.20
9	100	0.02	0.03	0.03	0.40	0.30
10	100	0.02	0.04	0.03	0.30	0.50
11	100	0.02	0.05	0.03	0.40	0.30
12	100	0.02	0.05	0.03	0.40	-
13	100	0.02	0.05	0.03	0.40	0.40
14	100	0.02	0.05	0.03	0.40	0.50

Table 4.2: Formulation of 2 wt% HPMC based cement slurries.

Slurry	**Cement BWOC**	**Defoamer gps**	**Dispersant gps**	**Retarder gps**	**FLA gps**	**HPMC gps**
15	100	0.02	-	-	-	0.20
16	100	0.02	-	-	-	0.30
17	100	0.02	-	-	-	0.40
18	100	0.02	-	-	-	0.50
19	100	0.02	-	-	-	0.65
20	100	0.02	-	-	-	0.80
21	100	0.02	0.02	0.03	0.50	0.20
22	100	0.02	0.03	0.03	0.40	0.30
23	100	0.02	0.04	0.03	0.30	0.50
24	100	0.02	0.05	0.03	0.40	0.30
25	100	0.02	0.05	0.03	0.40	-
26	100	0.02	0.05	0.03	0.40	0.40
27	100	0.02	0.05	0.03	0.40	0.50

4.2.2 Rheology of Cement Slurries

The rheology of cement slurry is a significant property for the displacement of cement slurry at surface condition. In cementing operations, the cement slurry undergoes three different stages. In first stage the slurry behaves like liquid and can flow. The second stage is when cement slurry starts to solidify and behaves as semi solid and the third stage is when cement becomes solid. The rheology determination is related to first stage where the slurry behaves as a liquid. To make sure that the cement slurry can be pumped into the wellbore safely; the rheology of cement slurry should be low. The rheology of cement slurries was determined in terms of shear stresses (fann θ readings) for different shear rates (rpm). The rheology of 1.5 HPMC solutions based cement slurries was determined and is shown in Table 4.3.

Table 4.3: Rheology of 1.5 wt% HPMC based cement slurries.

Slurry	**Temperature (°C)**	**Rheology (Shear stress)**						**10 sec Gel Strength Ib/100 ft^2**	**10 min Gel Strength Ib/100 ft^2**
		600 rpm	**300 rpm**	**200 rpm**	**100 rpm**	**6 rpm**	**3 rpm**		
1	ambient	249	163	121	98	51	37	39	59
	90	190	110	74	52	25	15	19	36
2	ambient	273	221	151	114	58	49	51	73
	90	249	194	137	90	36	27	27	43
3	ambient	+300	240	180	120	67	62	63	80
	90	274	225	158	100	50	30	32	49
4	ambient	+300	270	212	125	94	68	71	86
	90	291	243	176	109	71	38	39	58
5	ambient	+300	295	225	150	104	79	79	96
	90	+300	257	198	127	80	47	48	66
6	ambient	+300	+300	249	177	129	91	92	109
	90	+300	279	217	150	100	64	66	83
7	ambient	+300	+300	271	196	148	98	100	123
	90	+300	+300	241	170	126	76	78	96
8	ambient	+300	+300	230	150	121	73	74	88
	90	285	250	196	114	84	47	52	65
9	ambient	241	189	137	86	40	31	33	39
	90	226	171	119	70	25	15	18	37
10	ambient	285	223	165	111	76	41	42	62
	90	263	201	160	104	67	34	36	51
11	ambient	148	103	76	50	29	20	22	37
	90	137	89	61	35	16	08	10	24
12	ambient	127	100	64	48	27	19	20	35
	90	113	86	47	31	12	06	08	23
13	ambient	162	118	82	60	34	26	29	43
	90	149	101	68	45	23	15	18	34
14	ambient	178	128	94	74	50	38	40	51
	90	164	115	79	59	34	22	24	39

The rheology of cement slurries was also determined by changing the concentration of HPMC solution. The rheology of 2 wt% HPMC solution based cement slurries is shown in Table 4.4.

Table 4.4: Rheology of 2 wt% HPMC based cement slurries.

Slurry	**Temperature (^{o}C)**	**Rheology (Shear stress)**						**10 Sec Gel Strength Ib/100 ft^2**	**10 min Gel Strength Ib/100 ft^2**
		600 rpm	**300 rpm**	**200 rpm**	**100 rpm**	**6 rpm**	**3 rpm**		
15	ambient	281	229	164	121	66	57	61	79
	90	257	206	147	97	42	32	34	47
16	ambient	+300	249	186	132	74	68	74	92
	90	281	233	164	107	57	37	40	62
17	ambient	+300	276	223	154	100	74	82	106
	90	298	248	182	115	76	43	46	76
18	ambient	+300	290	235	116	109	79	87	114
	90	+300	257	198	127	81	47	51	85
19	ambient	+300	+300	253	172	123	86	91	122
	90	+300	270	210	140	95	58	62	97
20	ambient	+300	+300	278	201	156	104	110	135
	90	+300	+300	247	176	131	83	86	110
21	ambient	+300	+300	265	179	142	93	90	115
	90	298	265	216	135	118	50	53	100
22	ambient	265	250	232	150	124	80	81	102
	90	245	225	196	132	98	65	67	82
23	ambient	290	262	242	165	145	85	87	98
	90	272	213	187	143	110	70	70	87
24	ambient	153	108	81	56	34	23	24	38
	90	142	93	64	39	19	10	12	27
25	ambient	127	100	64	48	27	19	20	35
	90	113	86	47	31	12	06	08	23
26	ambient	169	125	88	67	42	31	33	47
	90	157	107	73	51	26	18	20	34
27	ambient	183	134	98	79	54	41	43	56
	90	169	118	81	63	38	25	26	39

The rheology of cement slurries determined at ambient temperature to evaluate the effect of HPMC solution at surface condition and at 90 ^{o}C for elevated temperature (wellbore condition). It was observed that the rheology of cement slurries increases by addition of HPMC polymer. The rheology of cement slurries 2, 7 consisted 1.5 wt% HPMC solutions and slurries 15 to 20 of 2 wt% solution showed higher rheology than slurry-1 without HPMC as shown in Table 4.3 and Table 4.4. The effect of HPMC solutions on rheology of cement slurries can be analyzed from Figure 4.6. The given figure shows the rheological behaviour of 1.5 wt% HPMC based cement slurries at ambient temperature and 90 °C. The trend of rheology at different concentration of HPMC solutions was observed only at 200 rpm. The rheology of cement slurries was increased with increasing concentration of HPMC solution in gps. The slurry-2 contains 0.20 gps HPMC solution showed low rheology than slurry-3 to slurry-7 at ambient temperature and 90 °C as shown in Figure 4.6. The cement slurry-7 contains maximum concentration of 0.80 gps HPMC showed maximum rheology in cement slurries.

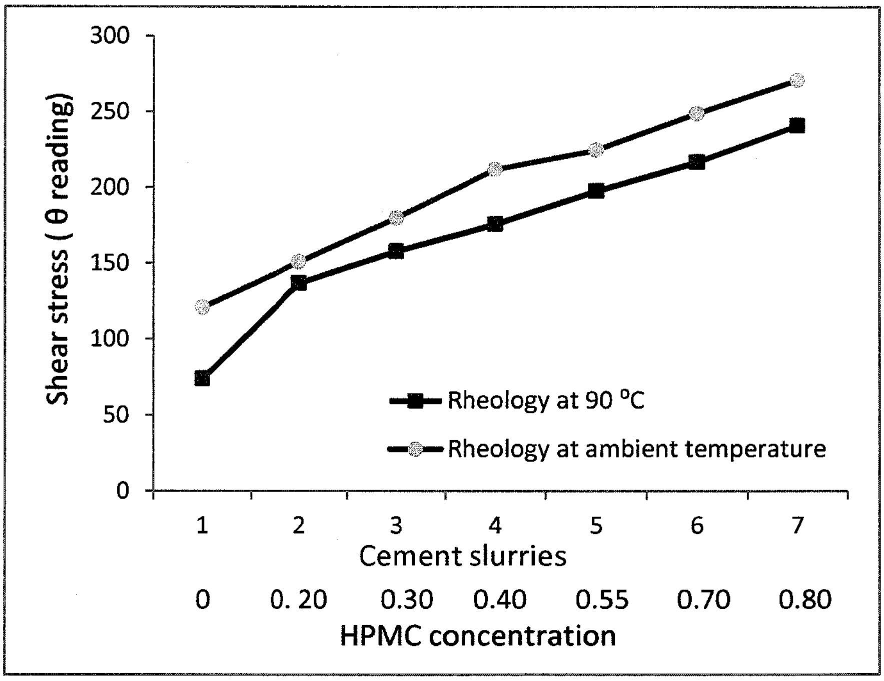


Figure 4.6: Rheological behaviour of 1.5 wt% HPMC based slurries at ambient temperature and 90 °C (200 rpm).

The rheology of 2 wt% HPMC based cement slurries was also determined at ambient temperature and 90 °C. It was observed that the rheology of cement slurries was increased with concentration of HPMC solution. Figure 4.7 demonstrates the effect of 2 wt% HPMC solution on the rheology of cement slurries.

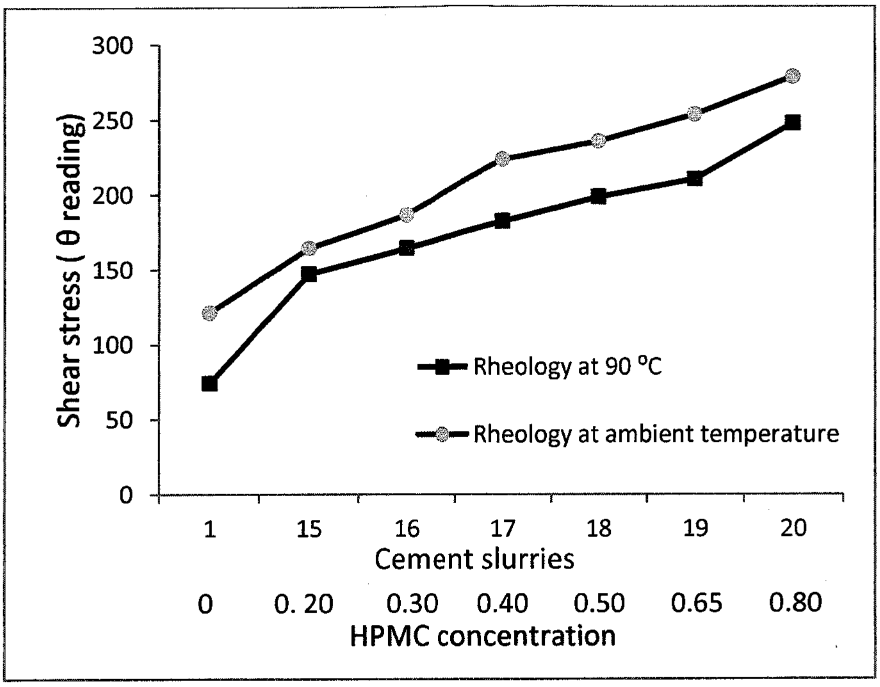


Figure 4.7: Rheological behaviour of 2 wt% HPMC based slurries at ambient temperature and 90 °C (200 rpm)

The change in concentration 1.5 wt% to 2 wt% of HPMC solution increased the rheology of slurries. The slurries 15 to 20 showed high rheology at both temperatures than slurries 2 to 7. The rheology of cement slurry was affected by the viscosity of polymer. HPMC polymer was viscosifying agent. The addition of HPMC solution in cement slurry increased the viscosity of slurry. The high viscosity of cement slurry affected the pressure drop during the displacement of cement slurry at surface condition. The high viscosity of HPMC will increase the pressure drop of cement slurry and it will require high pump pressure for displacement of cement slurry. The high rheology of cement slurry can also become the cause of formation fracture during displacement.

The gel strength at 10 seconds and 10 minutes is also an important parameter of rheology. The gel strength of cement slurries was determined at 10 seconds and 10 minutes at ambient temperature and 90 °C. The experiment of gel strength measurement showed that as the concentration of HPMC solution (1.5 wt%) changed from 0.20 to 0.80 gps, the gel strength of 10 seconds increased from 51 to 100 Ib/100 ft^2 and 73 to 123 Ib/100 ft^2 for 10 minutes at ambient temperature as shown in Figure 4.8.

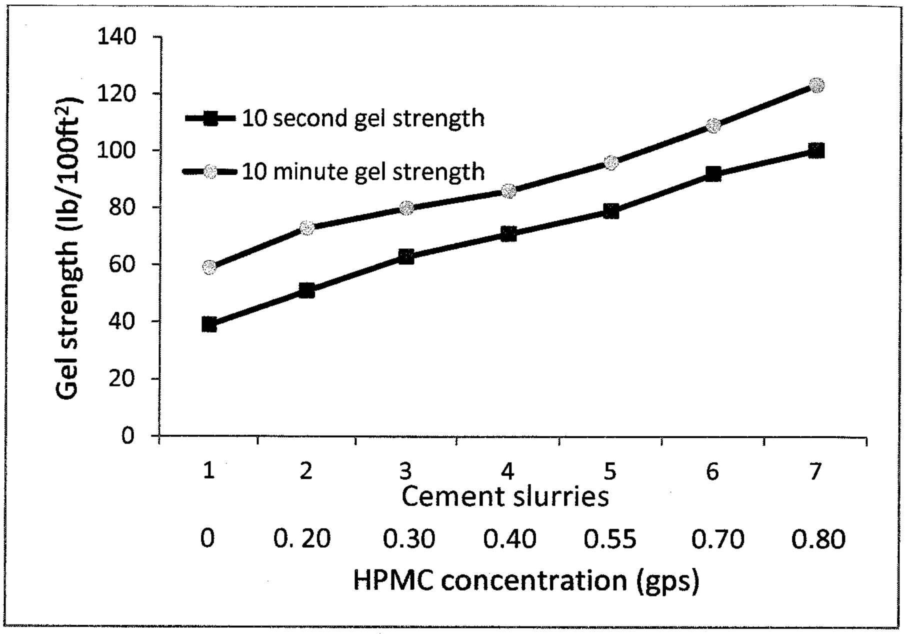


Figure 4.8: 10 seconds and 10 minutes gel strength of 1.5 HPMC based cement slurries at ambient temperature.

To investigate the effect of increasing HPMC concentration in term of wt%, the gel strength of 2 wt% based cement slurries were determined. The gel strength of 2 wt% slurries increased from 61 to 110 Ib/100 ft^2 for 10 seconds and from 79 to 135 Ib/100 ft^2 for 10 minutes at ambient temperature as shown in Figure 4.9.

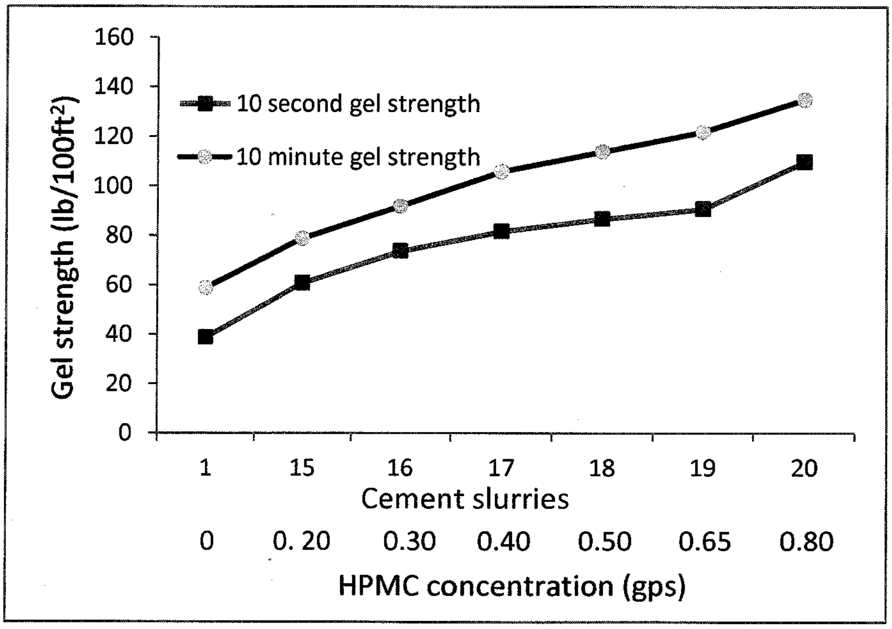


Figure 4.9: 10 seconds and 10 minutes gel strength of 2 wt% HPMC based cement slurries at ambient temperature.

The gel strength of cement slurries was increased with the change of solution and change in concentration in cement slurries. The high gel strength of cement slurries was due to the high viscosity of HPMC solutions at ambient temperature. Further, the gel strength of cement slurries was also determined at elevated temperature 90 °C. The gel strength of 1.5 wt% HPMC solutions is shown in Figure 4.10.

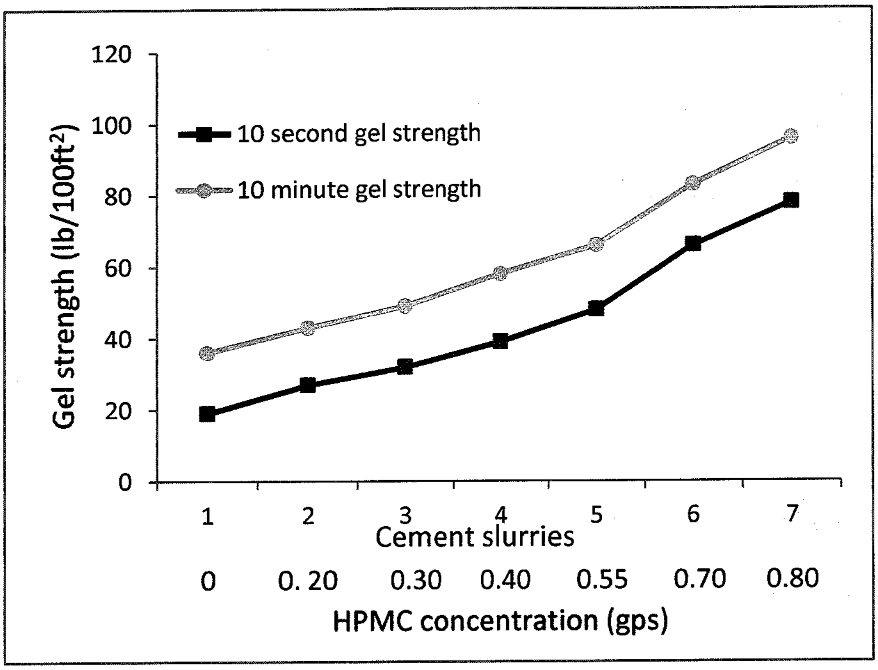


Figure 4.10: 10 seconds and 10 minutes gel strength of 1.5 wt% HPMC based cement slurries at 90 °C.

The gel strength of slurry-1 was 19 Ib/100 ft ^{2} and 36 Ib/100 ft ^{2} at 10 seconds and 10 minutes respectively. The slurry-1 was without HPMC solution and it showed low gel strength. The slurry-2 contained 0.20 gps HPMC showed 27 Ib/100 ft ^{2} gel strength at 10 seconds and 43 Ib/100 ft ^{2} at 10 minutes. The gel strength of cement slurries was increased with the change in concentration. The slurry-7 contains maximum concentration 0.80 gps of HPMC solution showed 78 Ib/100 ft ^{2} and 96 Ib/100 ft ^{2} at 10 seconds and 10 minutes respectively. In the same way, the gel strength of 2 wt% HPMC solutions was also determined at 90 °C and shown in given Figure 4.11.

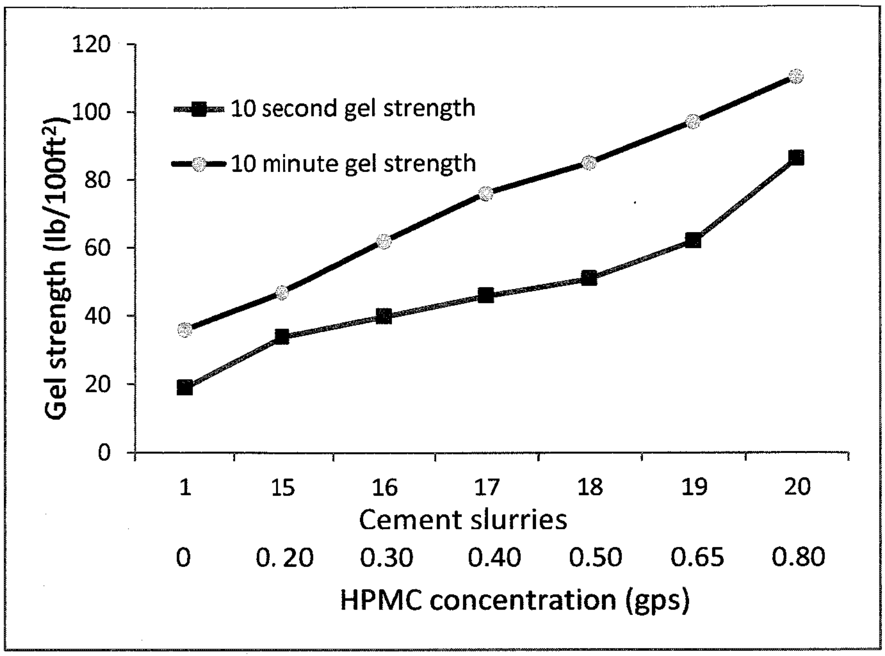


Figure 4.11: 10 seconds and 10 minutes gel strength of 2 wt% HPMC based cement slurries at 90 °C.

The same effect of increasing gel strength was observed in 2 wt% based cement slurries 15 to 20. Initially, at low concentration of 0.20 gps of HPMC solution, the gel strength of cement slurries was low 34 Ib/100 ft ^{2} at 10 seconds and 47 Ib/100 ft ^{2} at 10 minutes. Then the gel strength of cement slurries increased with concentration and gel strength 87 Ib/100 ft ^{2} at 10 seconds and 110 Ib/100 ft ^{2} at 10 minutes of slurry-20 was obtained using 0.80 gps HPMC solution.

The increasing concentration of HPMC polymer in term of weight% also affects the rheology of slurry. It was observed that as the amount of polymer increased 1.5 to 2 wt%, the rheology of cement slurry increased. The high rheology of 2 wt% HPMC based cement slurries was due to the high molecular weight and higher viscosity of solution. Therefore, 2 wt% HPMC contained cement slurries showed high rheology than 1.5 wt% HPMC solutions.

It was also observed that the gel strength of HPMC based slurries 2 to 7 and slurries 15 to 20 were higher than cement slurry without HPMC. The gel strength of slurries should be low for prevention of gas migration. After displacement, the slurry starts to solidify. During this phase the particles of polymer and cement comes close to each other. The attraction of particles to each other increases the gel strength of slurry. If the slurry composition has high gel strength, it will stop the transmission of hydrostatic pressure on formation and lead the migration of gas.

It is concluded that the increasing concentration of HPMC increases the rheology in terms of dial reading valves (shear stress) and gel strength. For an optimized cement slurry and gas migration control the rheological properties of cement slurries should be low. Therefore, it was necessary to decrease the rheology of slurries using additives. The dispersant (CD33L) was used in the remaining slurries for improving the rheology. The minor quantity of dispersant 0.02 to 0.05 gps was used in cement slurries as shown in Table 4.1. The other additives were also used in the slurry and the effect of remaining additives will be discussed in the next section of fluid loss and thickening time. The additive dispersant was being used in cement slurry to disperse the particles of the cement slurry that decrease the gel strength and rheology. The concentration 0.05 gps of dispersant with 0.30 gps of HPMC solution decreased the gel strength up to 22/37 Ib/100 ft ^{2} of 1.5 wt% HPMC based slurry-11 at ambient temperature. For 2 wt% HPMC based cement slurry-24, it decreased the gel strength up to 24/38 Ib/100 ft ^{2} for 10 seconds and 10 minutes respectively at ambient temperature.

. The high rheology and high gel strength was observed as the concentration of HPMC increased from 0.20 to 0.80 gps. But at 90 °C the rheology and gel strength of slurries were lower than the rheology and gel strength at ambient temperature. This was due to the reduction of viscosity with increasing temperature. It was also observed that the rheology of cement slurries was almost same at both temperatures. In normal condition the rheology of slurries decreased with increasing temperature. Due to this reason the rheology at both temperatures showed big difference. But for HPMC polymer at 90 °C it acted as a viscosifying agent and increased the viscosity. Therefore at 90 °C the viscosity of cement slurries remained high and big difference in rheology was not observed at both temperatures. Besides of this, the other polymers that are being used in cement slurries lose the viscosity at high temperature and huge differences in rheology was observed at surface and at elevated temperature. In order to decrease this problem the industries use other additives and chemicals in slurry to maintain the rheology of slurry for high temperatures.

4.2.3 Effect of HPMC Solution on Fluid Loss

The control of fluid loss through cement slurry is very important for prevention of gas migration. The loss of fluid at elevated temperature is one of the major causes of gas migration. The fluid loss was determined using different concentrations of HPMC solution. The test was carried out according to the procedure presented in section 3.2.5.2. Slurry-1 without HPMC polymer showed high fluid loss of 740 ml as shown in Table 4.5.

Table 4.5: API properties of 1.5 wt% HPMC based cement slurries (Free water, fluid loss and sedimentation).

Slurry	**Temperature (°C)**	**Free water (ml)**	**Fluid loss (ml/30 minutes)**	**Sedimentation Comments**
1	90	21	740	Heavy
2	90	17	596	Heavy
3	90	12	510	Medium
4	90	08	438	Medium
5	90	05	306	None
6	90	00	240	None
7	90	00	168	None
8	90	03	38	None
9	90	1.5	28	None
10	90	00	43	None
11	90	02	28	None
12	90	03	39	None
13	90	0.5	25	None
14	90	0.5	19	None

In this test the fluid was lost before 30 minutes and gas was collected in the cylinder. The loss of fluid before 30 minutes is called blow out. Therefore, the total fluid loss of this slurry was calculated using equation 3.1. In next cement slurries, 1.5

and 2 wt% HPMC solutions were used as given in Table 4.1 and 4.2. The fluid loss results of HPMC based cement slurries is shown in Table 4.5. In slurry-2, the concentration 0.20 gps of HPMC was used. The fluid loss of slurry-2 was 596 ml for 1.5 wt% HPMC. As the concentration of HPMC solution was increased from 0.20 to 0.80 gps as shown in Table 4.1. The fluid loss dropped from 596 ml/30min for 0.20 gps to 168 at 0.80 gps (1.5 wt% solution) HPMC as shown in Figure 4.12. The cement slurry-7 contained maximum concentration of 0.80 gps of HPMC solution and showed 168 ml fluid loss in 30 minutes.

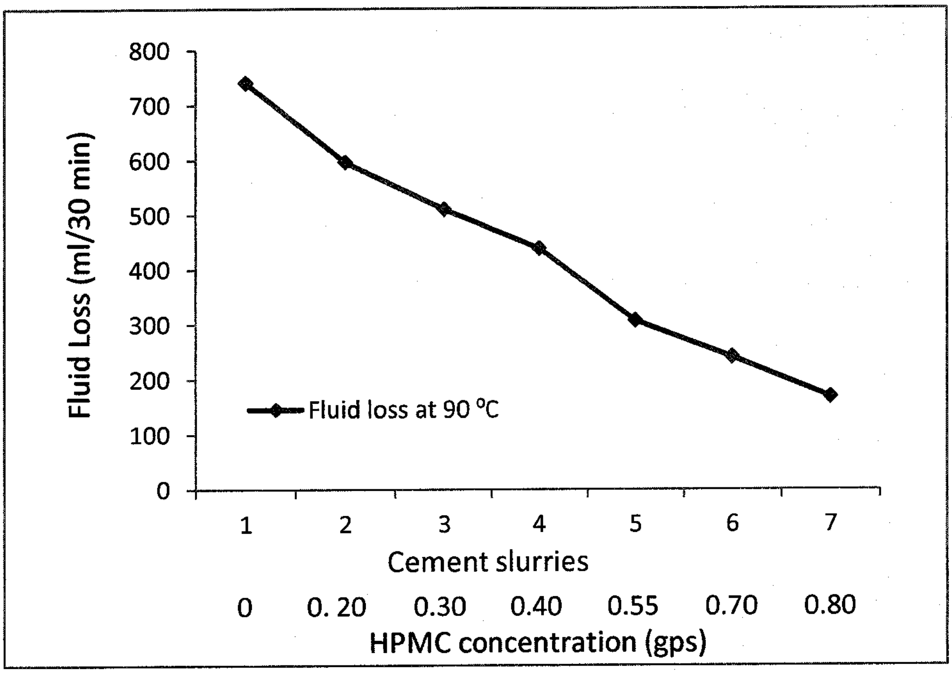


Figure 4.12: Fluid loss of slurries 1 to 7 at 90 °C

Further, the fluid loss of 2 wt% HPMC solution was also determined. To analyze the effect of 2 wt% HPMC solution, the fluid loss of cement slurries 15 to 27 are shown in Table 4.6. The cement slurry-15 showed 548 ml fluid loss in 30 minutes. Further by increasing concentration of 0.30 gps the fluid loss of slurry-16 was 484 ml. The fluid loss of slurry-17 was obtained 402 using 0.40 gps concentration of HPMC solution. Again by increasing concentration up to 0.80 gps the fluid loss was decreased to 140 ml of slurry-20. The fluid loss through slurry 1 to 7 and slurry 15 to 20 determined using equation 3.1. The trend of fluid loss decreases with increasing 2 wt% HPMC concentrations is shown in Figure 4.13.

Table 4.6: API properties of 2 wt% HPMC based cement slurries (Free water, fluid loss and sedimentation).

Slurry	**Temperature (°C)**	**Free water (ml)**	**Fluid loss (ml/30 minutes)**	**Sedimentation Comments**
15	90	15	548	Heavy
16	90	11	484	Medium
17	90	07	402	Medium
18	90	04	288	None
19	90	00	196	None
20	90	00	140	None
21	90	02	25	None
22	90	01	27	None
23	90	00	39	None
24	90	00	24	None
25	90	03	39	None
26	90	00	22	None
27	90	00	15	None

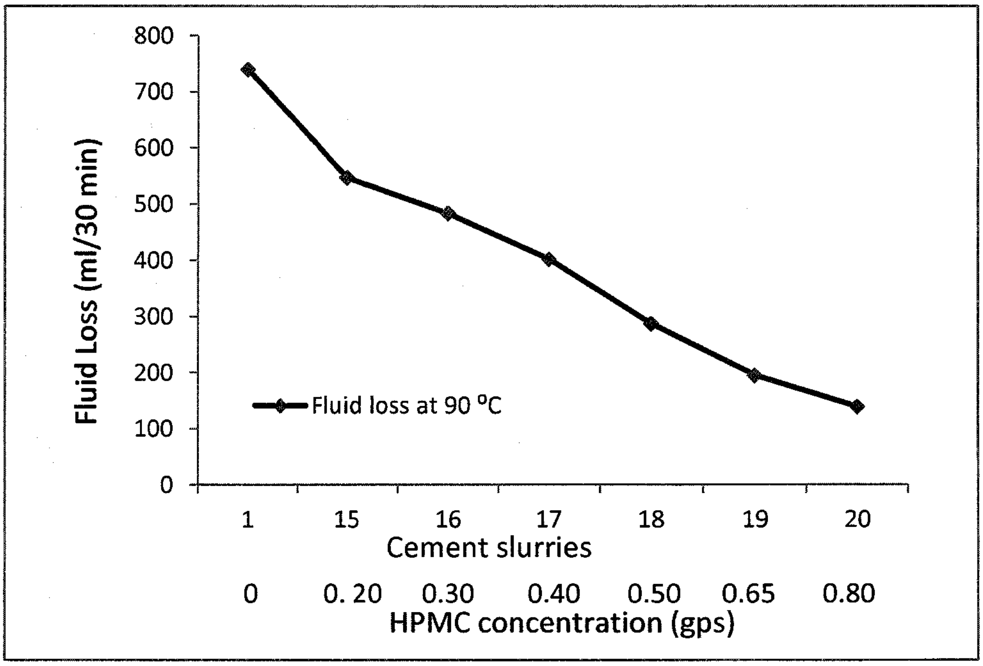


Figure 4.13: Fluid loss of Fluid loss of slurry 1 and 15 to 20 at 90 °C.

The experimental results show that HPMC polymer was decreases the fluid loss. In cement slurry, the particles of HPMC polymer was attached to the surface of cement pores spaces. At the same time, the reaction with water increases the slurry viscosity that plugs the interstitial pore spaces of cement filter cake. In this way, the HPMC reduces the filter cake permeability that becomes the cause of fluid loss reduction through cement slurry. It was also observed that the fluid loss can be reduced by increasing concentration of HPMC. On the other hand, the increasing concentration of HPMC increases the rheology and gel strength of cement slurry. High rheology of cement slurry creates problems during displacement and becomes the cause of formation fracture.

According to API criteria the fluid loss should be less than 50 ml/30 minutes for prevention of gas migration. Therefore, it was necessary to use FLA with HPMC polymer that would improve the rheology and decrease the fluid loss. The concentration 0.30 to 0.50 gps of FLA (FL-66L) with HPMC polymer, Retarder (R-21LS) and Dispersant (CD-33L) was used in remaining cement slurries. The concentration of 0.30 to 0.50 gps of FLA decreased the fluid loss through cement slurry. It was observed that using FLA of 0.30 gps with 0.50 gps of HPMC and other additives obtained the fluid loss less than 50 ml/30 min for both solutions of HPMC polymer as shown in Table. 4.5 and Table 4.6.

Further the impact of HPMC for controlling fluid loss was analyzed through slurry 11, slurry 12 of 1.5 wt% HPMC solution, slurry 24 and slurry 25 of 2 wt% HPMC solutions. Slurries 11 and 24 consisting of 0.40 gps FLA, 0.30 gps HPMC with other additives showed 28 and 24 ml/30min fluid loss respectively at 90 °C. Slurries 12 and 25 which contained 0.40 gps FLA with only dispersant and retarder showed 39ml/30 fluid loss. It was observed that HPMC polymer had positive impact on cement slurry that improved the property of FLA and decreased the fluid loss through cement slurry. Moreover, further fluid loss was decreased by using 0.40 and 0.50 gps HPMC polymer with same concentration of FLA. The dispersant is not only used for improving rheology but it also decreased the fluid loss and improved the properties of cement slurry. The aim of determining the fluid loss of 1.5wt% and 2 wt% was to investigate the effect of different concentrations of HPMC polymer. It was observed that the fluid loss of 1.5 wt% solution was lower than the 2 wt%

solution of HPMC. This showed that increasing concentration of solution in term of wt% decreases the fluid loss through slurry.

4.2.4 Effect of HPMC on Free Water Separation

Free water is an independent API property of cement slurry and it plays an important role for gas migration. The free water is unreacted water that separates from cement slurry under static condition. The free water of HPMC based cement slurries was tested and the effect of HPMC for controlling free water was theoretically analyzed. The performance of HPMC polymer for controlling free water was compared through cement slurry-1 and cement slurries 2 to 7 as shown in Table 4.5. Slurry-1 without polymer showed 21 ml of free water within the time duration of 2 hours. The free water of slurry-2, which contained 0.20 gps of HPMC (1.5 wt%) solution was 17 ml. It showed that the addition of HPMC decreased the amount of free water. The free water separation through cement slurries became less by increasing the concentration of HPMC polymer as observed in cement slurry-3 to slurry-7 and slurry-15 to slurry-20 as shown in Table 4.5 and 4.6. The HPMC polymer completely dissolved in water and increased the viscosity of cement slurry at low concentrations. The absorption quality of HPMC polymer was more than pure cement. Therefore, the free water that was separated from cement slurry absorbed by HPMC polymer and enhanced the viscosity.

It was observed that HPMC polymer completely prevented the free water at high concentration 0.70 gps for both solutions. The free water of the remaining cement slurries 8 to 13 was less than 05 ml, because the FLA was used in these slurries. FLA's improved the properties of HPMC polymer for controlling free water separation. However, dispersants had a negative impact on cement slurry and promoted free water separation [79]. The effect of dispersant was observed in cement slurries 8 to 14 and slurries 21 to 27 which contained dispersant and FLA. The addition of 0.05 gps dispersant showed 0.5ml free water separation as showed in slurry 13 and 14. The dispersant was used in cement slurry in order to reduce the rheology and decrease the viscosity. Therefore, as the concentration of dispersant increased the viscosity of slurry decrease. The decreased viscosity of slurry had an effect on the free water through cement slurry. However, the free water of these slurries was less than 5 ml in presence of dispersant.

4.2.5 Sedimentation Observation of Cement Slurries

The settling and sedimentation of cement particles contribute to the reduction of hydrostatic pressure. The sedimentation of cement particles alters the density of cement. After displacement of cement slurry the heavy particles settle down and this increases the density of slurry. While the density of remaining slurry becomes low, this reduces the hydrostatic pressure of cement column above formation pressure. The reduction of slurry density becomes the cause of gas migration.

In this study the sedimentation through slurries was analyzed through visual observation in graduated cylinder. It was observed that the HPMC prevents the sedimentation of particles as shown in Table.4.5 and 4.6. Slurry-1 without HPMC polymer showed heavy sedimentation of particles. However, the use of HPMC polymer changes the heavy sedimentation into medium sedimentation. Slurry-3 and slurry-4 containing 0.30 and 0.40 gps HPMC showed medium sedimentation. The increasing concentration of HPMC decreased the sedimentation of particles and no sedimentation was visualised in cement slurries 5 to 7. The HPMC polymer was viscosifying agent and it increased the viscosity at 90 ^{o}C temperature. As the HPMC polymer used in cement slurry it increased the viscosity of slurry. HPMC polymer increased the cohesive force between the particles and restricted the sedimentation of particles. The HPMC polymer became more effective for settling prevention when FLA was used in slurry as shown in Table.4.5 and 4.6.

4.2.6 Effect of HPMC on Thickening Time of Cement Slurries

The aim of thickening time test was to evaluate the effect of HPMC polymer on the pumpability of cement slurry. The thickening time of cement slurries was determined by changing the concentration of additives. The additive retarder has been used in cement slurry in order to increase the thickening time and pumpability of cement slurry. In this research 0.03 gps concentration of retarder was also used. The small quantity of retarder was used according to API equipment safety criteria and HSE policy of cementing lab. The thickening time of slurry-12 was determined using HPHT Consistometer. The slurry-12 consisted of different additives. The slurry-12 was composed of 0.02 gps of defoamer, 0.05 gps dispersant, 0.03 gps retarder and 0.40 gps FLA as given in Table 4.1 section 4.2.1. The thickening time of slurry-12 is shown in Figure 4.14.

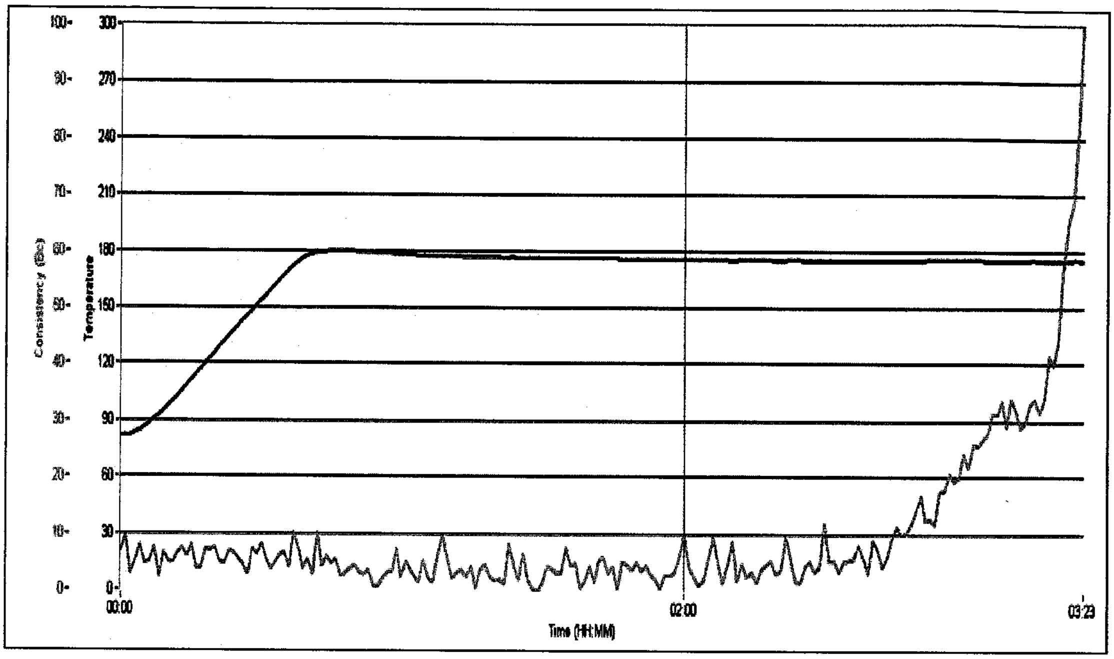


Figure 4.14: Thickening time of slurry-12 (without HPMC Solution).

It was observed that the consistency of slurry was low. The slurry-12 remained in liquid state for 3 hour and it could flow. But after this the consistency of slurry starts to increase. Figure 4.14 showed that the slurry gained the maximum consistency of 100 Bc in 3 hour and 23 minutes. Therefore, the thickening time of slurry-12 was 3:23 hours.

Next, the thickening time of slurry-11 was determined. In this cement slurry HPMC solution was used. The cement slurry-11 was prepared using 0.30 gps of 1.5 wt% HPMC solution with 0.02 gps defoamer, 0.05 dispersant, 0.03 retarder and 0.40 gps FLA. The thickening time profile of slurry-11 is shown in Figure 4.15.

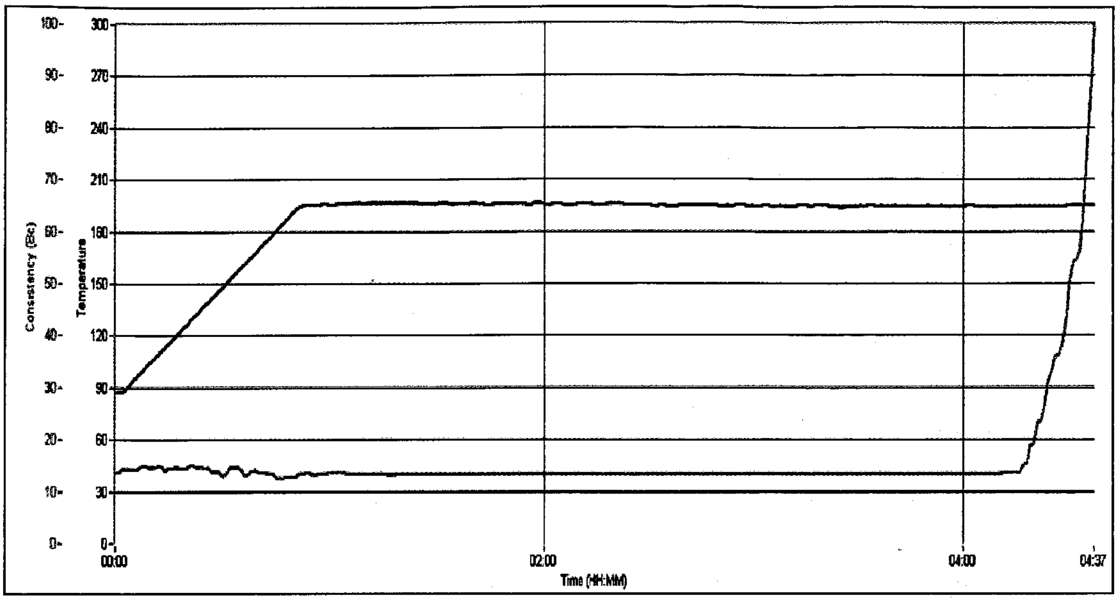


Figure 4.15: Thickening Time of Slurry-11 (0.30 gps of 1.5 wt% HPMC solution).

The thickening time of cement slurry-11 was increased from 3:23 to 4:37 hours by the addition of 0.30 gps of HPMC polymer. In the same way, the thickening time of slurry-13 was determined using 0.40 concentration of HPMC polymer as shown in Figure 4.16.

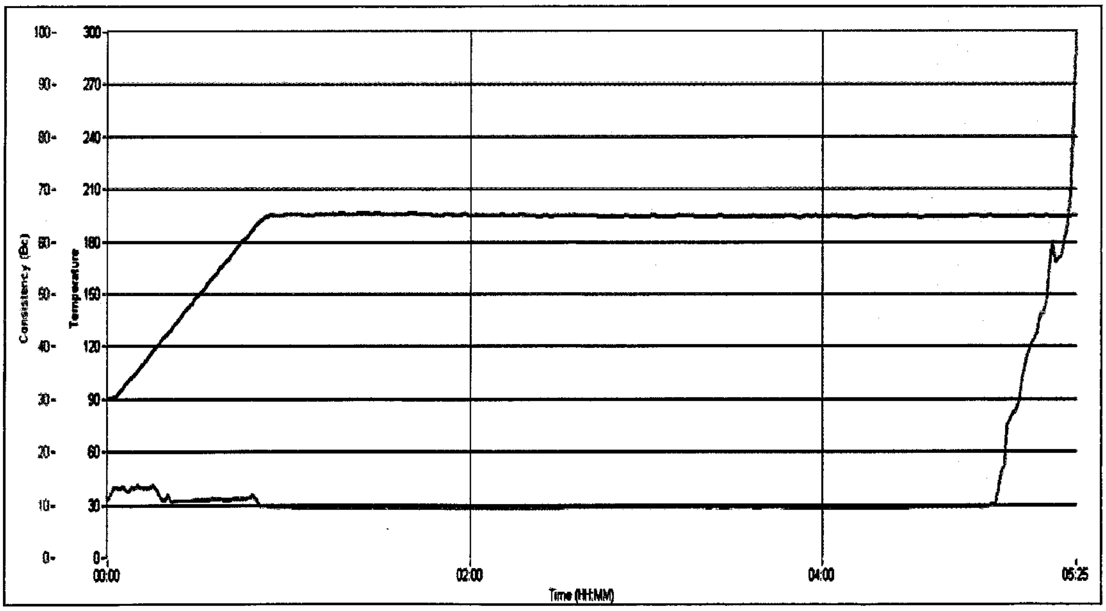


Figure 4.16: Thickening time of slurry-13 (0.40 gps of 1.5 wt% HPMC solution).

It was observed that as the concentration of HPMC solution increased from 0.30 to 0.40 gps the thickening time of cement slurry-13 was increased. The total thickening time of slurry-13 was 5:25 hours. Further the thickening time of cement slurry-14 was determined as shown in Figure 4.17.

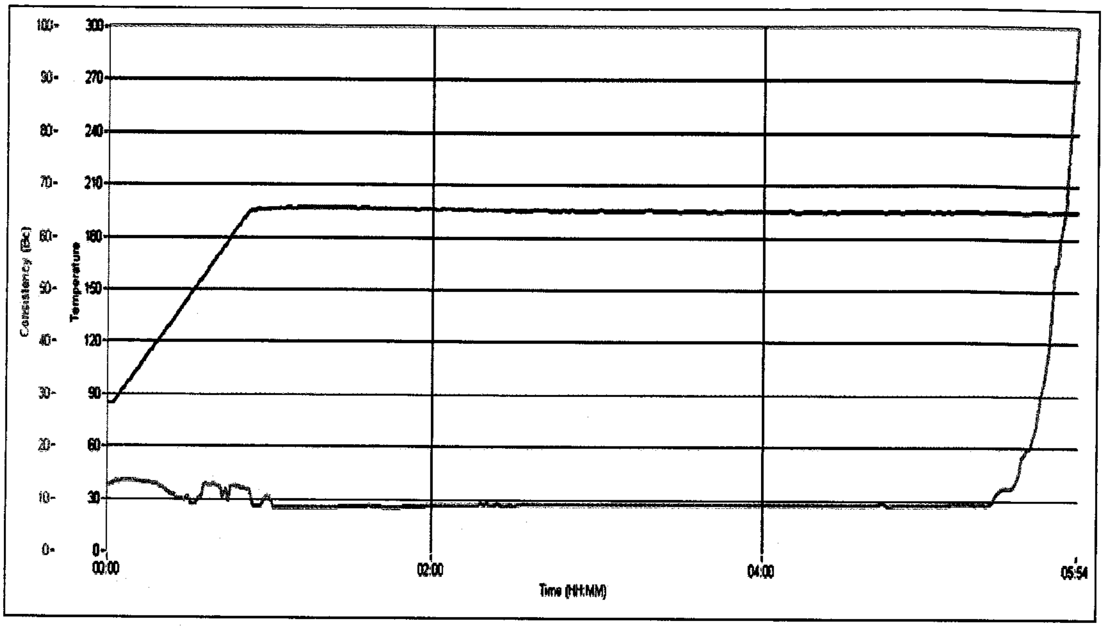


Figure 4.17: Thickening time of slurry-14 (0.50 gps of 1.5 wt% HPMC solution).

From the results it was observed that with increasing the concentration of HPMC polymer, the thickening time of cement slurry was increased. In the end, 5:54 hours of thickening time was obtained using 0.50 gps concentration of 1.5 wt% HPMC solution in cement slurry-14. The results of above test confirmed that HPMC polymer is a suitable retarder. The molecular structure of HPMC polymer played an important role for increasing the thickening time of cement slurry. This was due to the basic structure of HPMC that belongs to the cellulose family and is a member of hydroxyl group in forming long chain polymer. The property of long chain polymers is that it slows down the setting reaction and the slurry remains in liquid state for long time. The thickening time of cement slurries was also determined using 2 wt% concentration of HPMC polymer. The thickening time of slurry-24 consisted of 0.30 gps concentration of 2 wt% HPMC as shown in Figure 4.18.

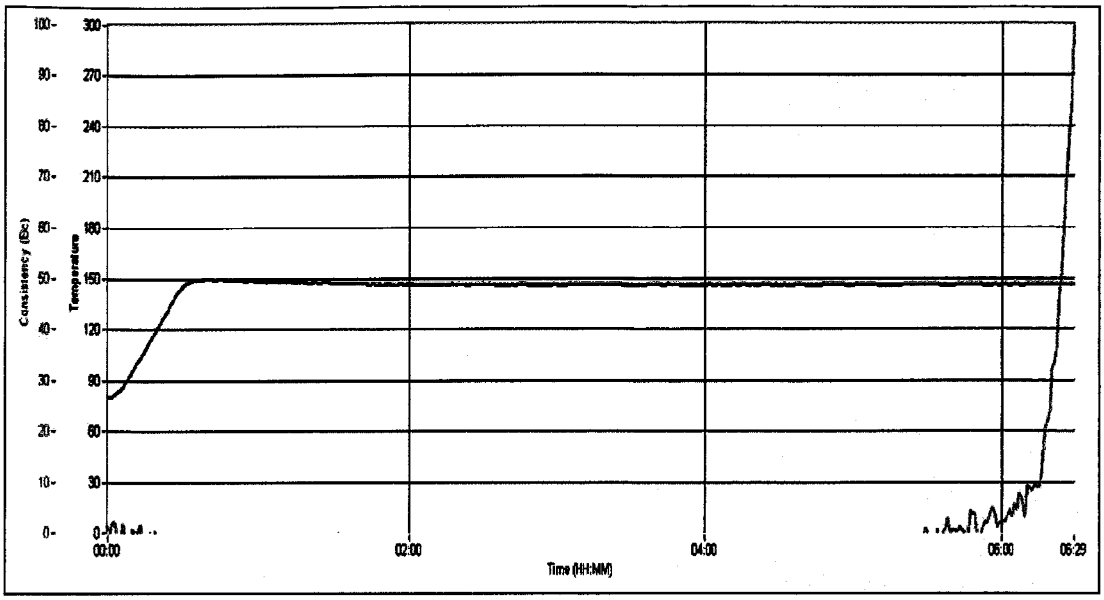


Figure 4.18: Thickening time of slurry-24 (0.30 gps of 2 wt% HPMC solution).

It was observed that slurry-24 showed low consistency at initial stages than other slurries. Initially it showed 3 Bc consistency for the duration of 5 hours and 50 minutes. Next the consistency was increased rapidly and gained the maximum consistency in 6:29 hours. It was the thickening time of slurry-24. In Figure 4.18, the consistency line was disappeared at some points. This was due to the low consistency of slurry at initial stage. Next the thickening time was also determined by changing the concentration from 0.30 to 0.40 gps of 2 wt% HPMC solution. The thickening time of slurry-26 is shown in Figure 4.19.

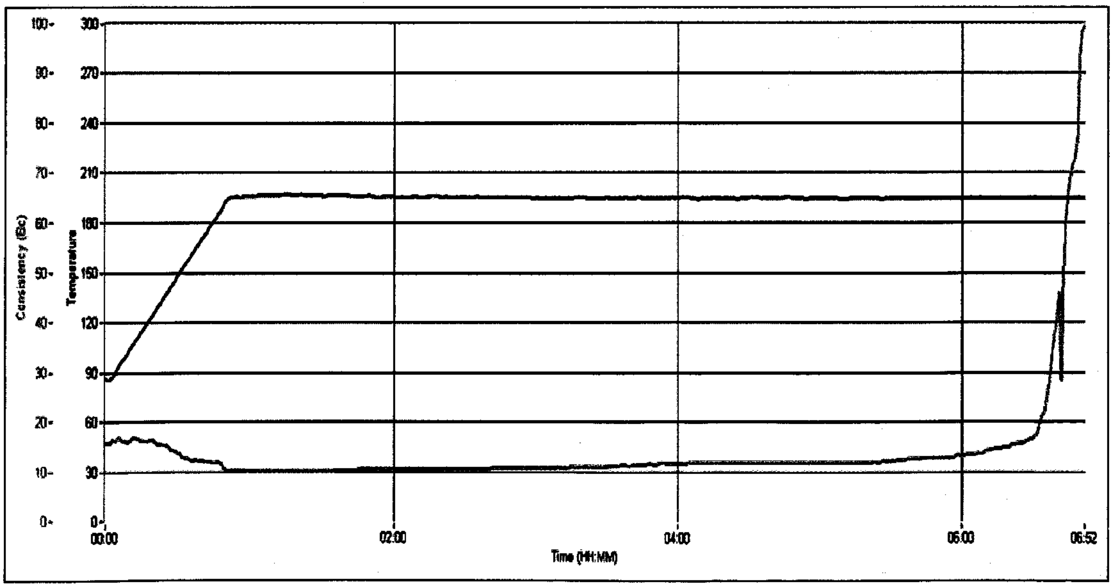


Figure 4.19: Thickening time of slurry-26 (0.40 gps of 2 wt% HPMC solution).

It was observed that as concentration of HPMC solution was increased, the thickening time of cement slurry also increased. The effect of HPMC concentration was analyzed through slurry-11 and slurry-24. Both slurries contained 0.30 gps concentration of HPMC but they both had different concentrations of HPMC polymer in terms of wt%. The thickening time of slurry-11 was 4:37 hours and slurry-24 was 6:29 hours. Because slurry-11 consisted 1.5 wt% HPMC polymer and it had lower gel strength and viscosity than slurry-24 which contained 2 wt% HPMC polymers.

The results of thickening time test showed that HPMC polymer had positive impact on the cement slurry for increasing thickening time. The slurries that contained HPMC polymer showed higher thickening time than slurry-12. It was observed that the HPMC based cement slurries has low consistency from the start. The consistency of cement slurries was less than 15 Bc. The low consistency showed that slurry was in the liquid phase and easy to pump. The continuous low consistency showed that cement slurries remained in liquid state for a long time and the slurry would transmit full hydrostatic pressure. It was also observed that the HPMC based cement slurries showed right angle set cement behaviour. This was because the slurries consisted of low consistency that continued for long several hours before rapidly rising to 70 Bc. This kind of behaviour of HPMC polymer was due to the kinetic reaction that allowed the slurry to maintain hydrostatic pressure for long time on the permeable zone. In this way, the HPMC polymer developed a low permeability matrix that would prevent the gas migration. Through using HPMC polymer, the cement slurry remained pumpable for more than the required time allowing it to be placed efficiently in the annular space.

4.2.7 Compressive Strength of Cement Slurries

The compressive strength of cement slurries was determined to evaluate the effect of HPMC on compressive strength. The compressive strength data is very important for cement slurry to evaluate the performance after displacement. The industry specification states that 500 psi compressive strength is required. The 500 psi compressive strength is considered enough resiliency for the shock load for drilling operation and to hold the pipe in the hole [30]. The time duration to determine the compressive strength depends upon depth and temperature. The shallow depth wells require the strength in 8 to 12 hours and deep wells require it in 16 to 24 hours.

The compressive strength of cement slurries was determined according to deep well requirements. The compressive strength of cement slurries was determined using SGSA. It was observed from the results that the compressive strength of slurry-12 without HPMC polymer was 2430 psi in 18 hours as shown in Figure: 4.20.

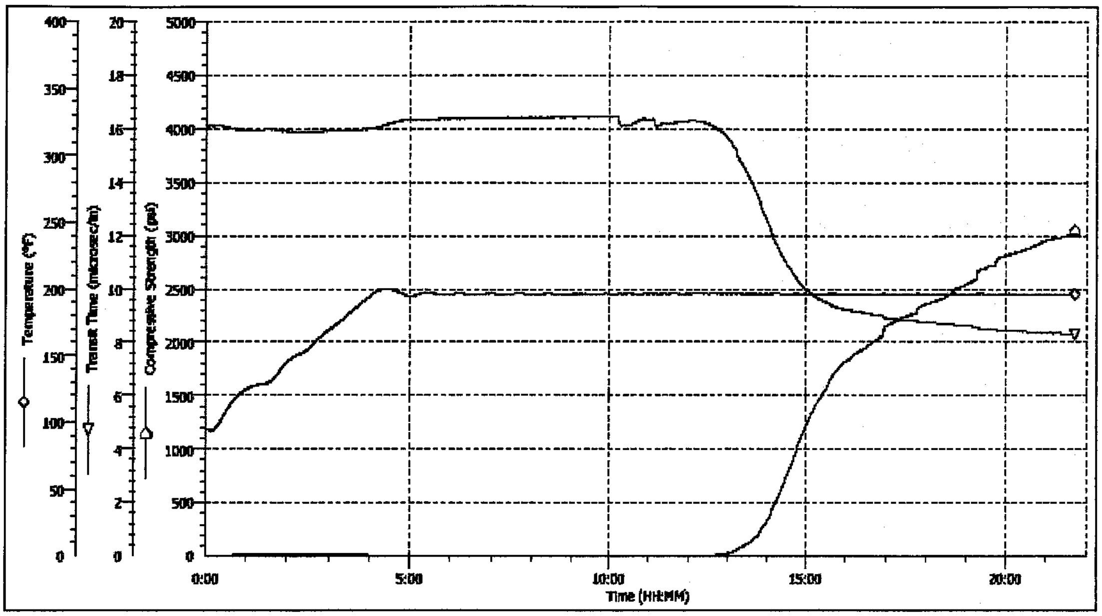


Figure 4.20: Compressive strength of slurry-12 (without HPMC polymer).

Slurry-11 that was prepared using 30 gps concentration of 1.5 wt% HPMC showed the compressive strength of 4100 psi in 18 hours as shown in Figure 4.21. The compressive strength of the cement slurry was increased to 59% using 0.30 gps HPMC solution. This increase in compressive strength of cement slurry was due to the 3-D structure of HPMC long chain that creates chemical bond with free Ca^{++} cations. Besides this, the compressive strength of cement slurry was also increased due to the water retention property of HPMC polymer. This property of HPMC polymer decreased the permeability of cement which became the cause of enhanced compressive strength.

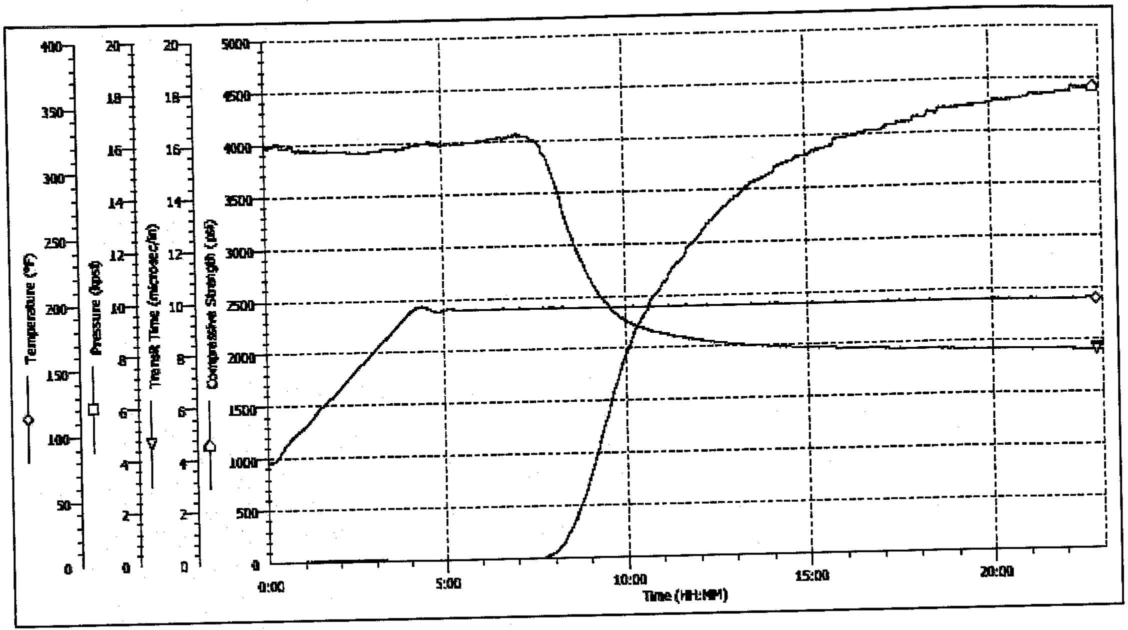


Figure 4.21: Compressive strength of slurry-11 (0.30 gps of 1.5wt% HPMC solution).

The compressive strength of slurry-13 using 40 gps HPMC obtained 3700 psi as shown in Figure-4.22. A high percentage of HPMC polymer decreased the compressive strength of cement slurry. Here, the HPMC polymer was acting as a retarder. The increased concentration of polymer increased the retarding (thickening) time of cement slurry and the slurry remained in liquid form for a long duration of time. Therefore, the increased concentration of HPMC decreased the compressive strength [79].

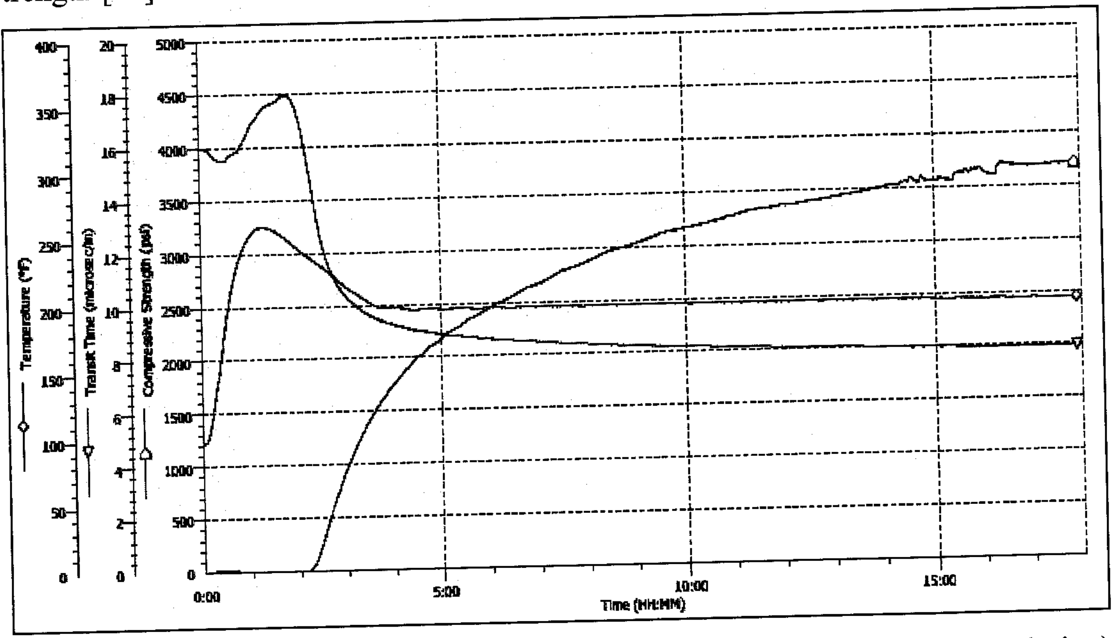


Figure 4.22: Compressive strength of slurry-13 (0.40 gps of 1.5 wt% HPMC solution).

The compressive strength of cement slurries was also determined using 2 wt% HPMC solution. It was observed that by using high percentage of HPMC polymer the compressive strength was decreased as shown in Figure 4.23.

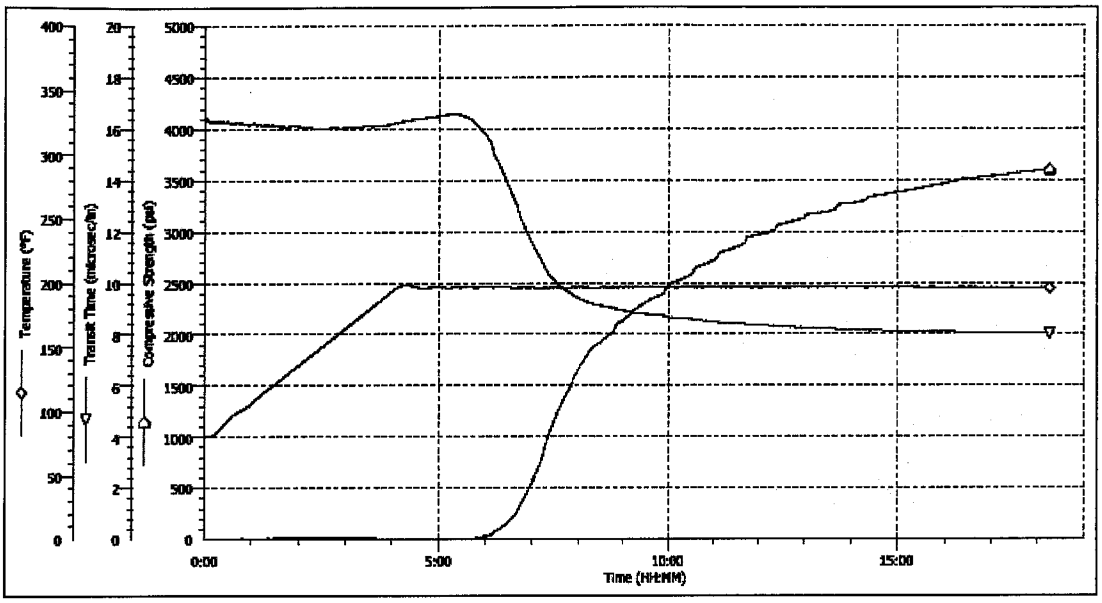


Figure 4.23: Compressive strength of slurry-24 (0.30 gps of 2 wt% HPMC solution).

From the comparison of slurry-11 and 24 which contained 30 gps of 1.5 and 2 wt% HPMC solution respectively, the compressive strength of cement slurries were decreased 4100 psi to 3600 psi. Further, the compressive strength became 2700 psi of slurry-27 using high concentration 50 gps of 2 wt% HPMC solution as shown in Figure 4.24.

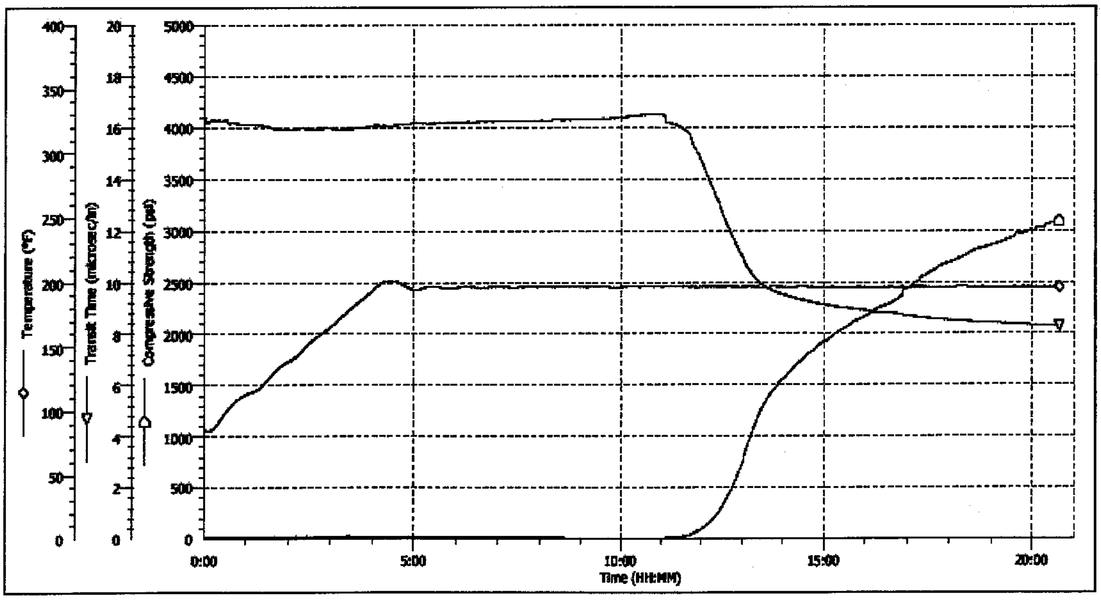


Figure 4.24: Compressive strength of slurry-27 (0.5 gps of 2 wt% HPMC solution).

The decrease in strength was due to the retarding and gelation property of HPMC polymer. Moreover, the compressive strength of cement slurry was also affected by the increased concentration of dispersant in cement slurry. The variation in compressive strength also depends upon the water cement ratio, the quantity of additive and polymer. The experimental results illustrate that small concentration of HPMC polymer can increase the compressive strength but high concentration becomes the cause of strength reduction. Moreover, using HPMC polymer the compressive strength of slurries is greater than 2000 psi in 18 hours. That is enough and suitable for cementing operation.

4.3 Gas Migration Analysis through Cement Slurries

This section is based on the measurement of transition time and pore pressure of cement slurries. The transition time of cement slurry was determined through static gel strength measurement method. The static gel strength is API property of cement slurry. But the measurement of transition time during the development of gel strength gives the idea of gas migration through cement slurry. Therefore, the gas migration through HPMC based cement slurries was determined using two different methods and the results are discussed given below in detail.

4.3.1 Transition Time Measurement of Cement Slurries

The gel strength development is very important property of cement slurry. The gel strength starts to develop immediately after static condition of cement slurry. The development of gel strength is very important in aspect of determining the potential of gas flow. For the determination of gas migration, the transition time of cement slurries has been determined. During the transition period of cement slurry the hydrostatic pressure of cement slurry gradually decreases. That becomes the cause of gas migration. Therefore, the transition time of cement slurry should be low to prevent the reduction of hydrostatic pressure [80]. In first experiment the transition time of slurry-12 was determined. It was prepared without HPMC polymer. The static gel strength of slurry-12 is shown in Figure 4:25 given below.

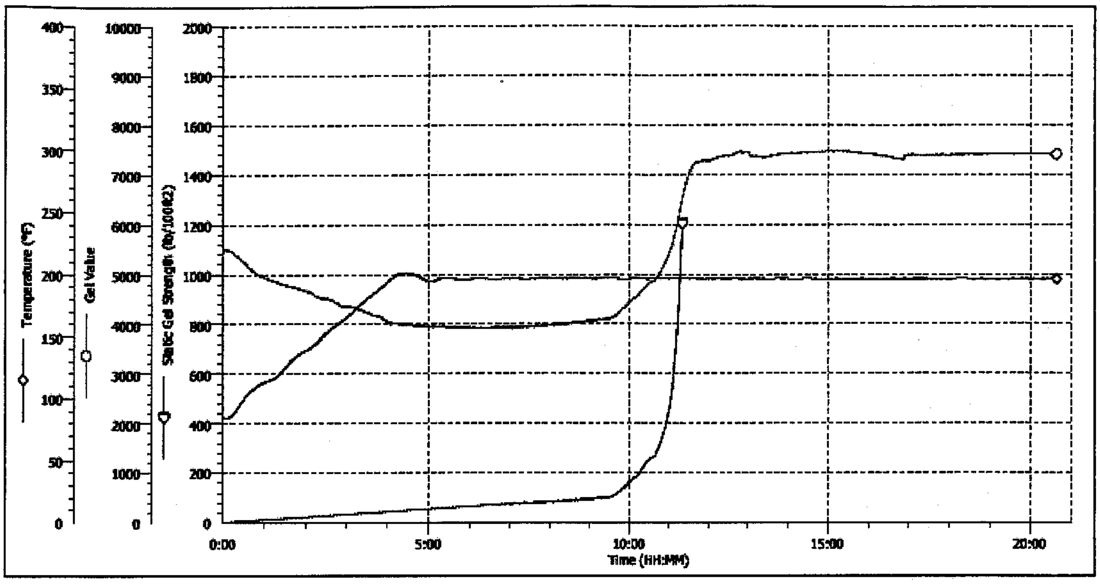


Figure 4.25: Transition time of slurry-12 (without HPMC polymer).

The transition time of slurry-12 was 110 minutes because it developed gel strength from 100 lb/100ft^2 to 500 lb/100ft^2 in 1 hour and 50 minutes. For the gas tight cement slurry that would be applicable for gas migration prevention the transition time should be below 45 minutes according to API criteria. Therefore in order to determine the impact of HPMC polymer on cement slurry the static gel strength of other slurries was determined using different concentration of polymer. The transition time of slurry-8 which contained 0.20 gps concentration of 1.5 wt% HPMC solution with FLA, dispersant, retarder and deformer was determined. The static gel strength of slurry-8 is shown in Figure 4.26 given below.

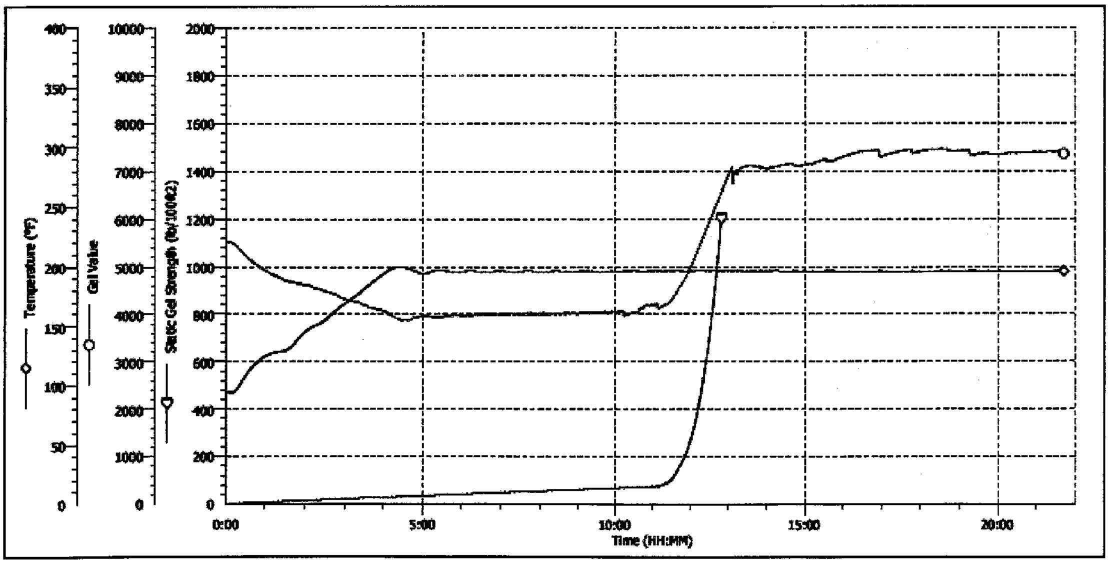


Figure 4.26: Transition time of slurry- 8 (20 gps of 1.5 wt% HPMC solution).

The above figure shows that slurry-8 gained the gel strength of 100 lb/100ft^2 in 11 hours. The gel strength 0 to 100 lb/100ft^2 is considered zero gel strength. The slurry remained in liquid state during this phase and transmitted the full hydrostatic pressure [81]. Next the slurry reached 100 lb/100ft^2 to 500 lb/100ft^2 in the time duration of 54 minutes so that was the transition time. According to the API criteria, this cement slurry was also considered unfit for gas migration prevention application. It was observed that the implementation of HPMC polymer reduced the transition time of cement slurry. The HPMC is high gel strength polymer and in cement slurries it increases the gel strength. Therefore, as the HPMC polymer added in cement slurry, it gained 500 lb/100ft^2 in short time. That became the cause of transition time reduction. The transition time of cement slurry can be reduced further by increasing the concentration of HPMC. The static gel strength of 30 gps HPMC contained in cement slurry-11 was determined and shown in Figure 4.27.

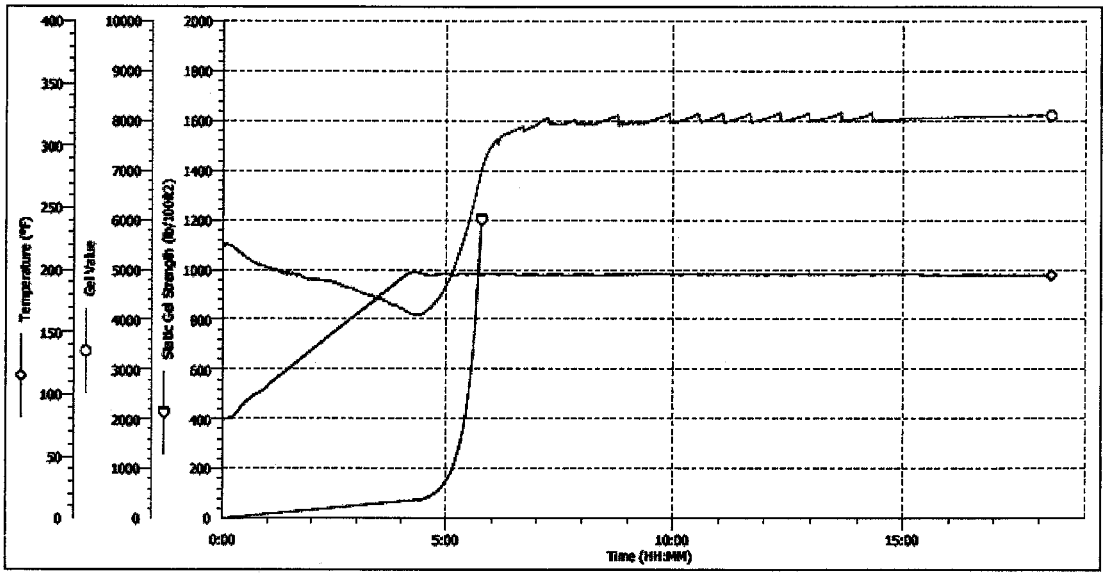


Figure 4.27: Transition time of slurry-11 (30 gps of 1.5 wt% HPMC solution).

In the first stage the cement slurry remained in liquid state and the zero gel strength of slurry was 290 minutes. Then within 39 minutes it obtained the gel strength value of 500 Ib/ 100ft^2. Therefore the transition time of slurry-11 was 39 minutes. This slurry could prevent the influx of wellbore fluid. Further the static gel strength of cement slurry-13 was determined as shown in Figure 4.28. Slurry-13 was prepared using 0.40 gps concentration of 1.5 wt% HPMC solution with other additives.

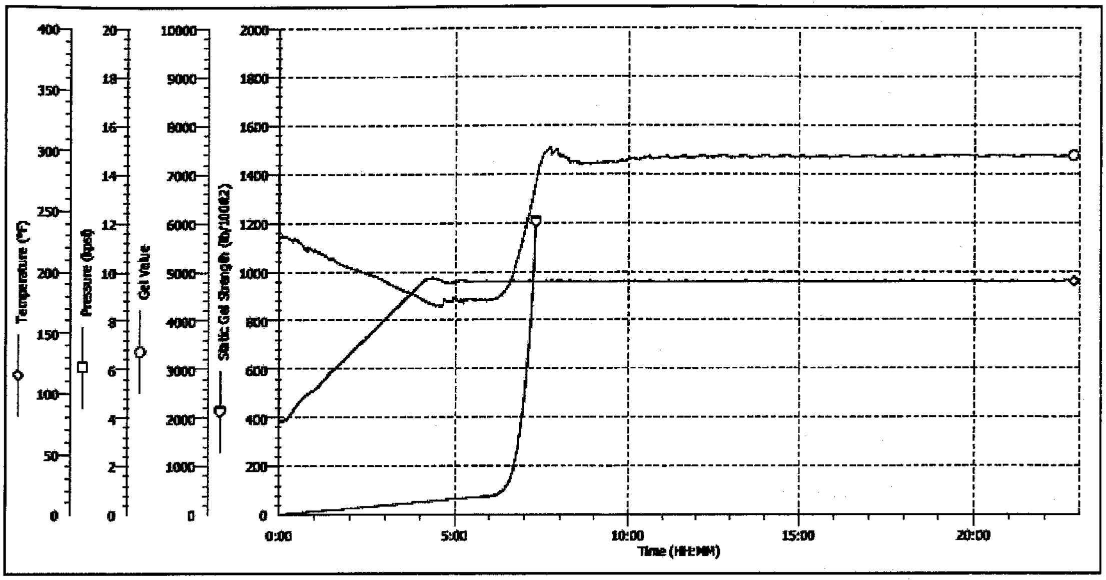


Figure 4.28: Transition time of slurry -13 (40 gps of 1.5 wt% HPMC solution).

The zero gel strength value of slurry-13 was 6:32 hours and it completed the transition time in 7:00 hours. This showed that the transition time period of the slurry was 28 minutes. The graph (Figure 4.28) showed that the rapid gel strength developed after 100 Ib/ 100ft^2. The slurry developed 500 Ib/ 100ft^2 of gel strength in short time than other slurries. That allowed the cement slurry to become self supporting and reduced the hydration period of the setting cement. Further, one test was conducted using 2 wt% HPMC solutions. The static gel strength of slurry-24 was determined using 0.30 gps of 2wt% HPMC solution as shown in Figure. 4.29.

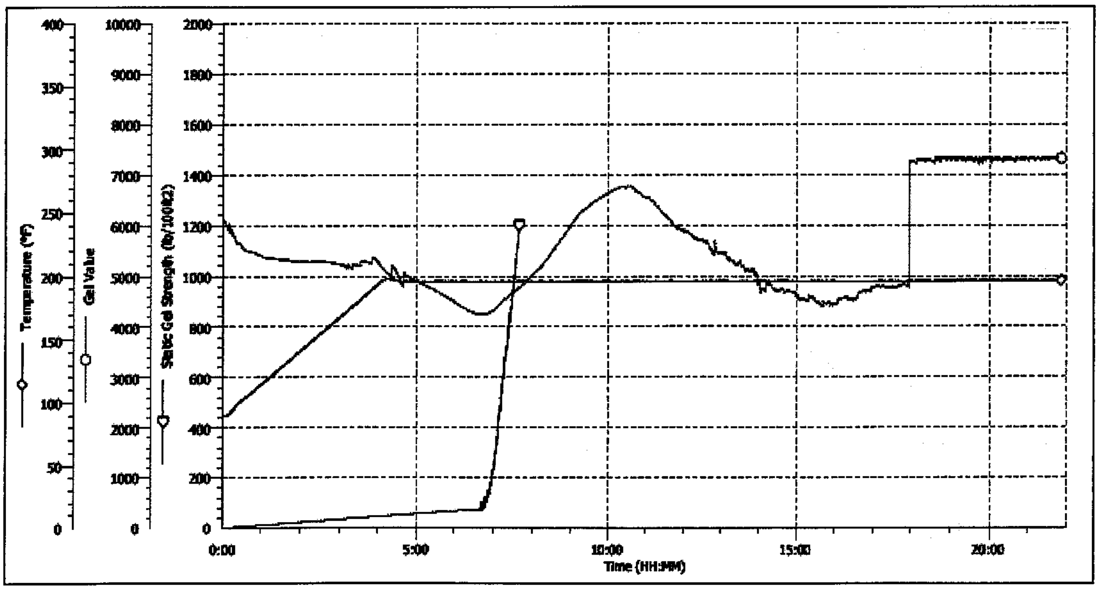


Figure 4.29: Transition time of slurry-24 (30 gps of 2 wt% HPMC solution).

It was observed that with increasing the concentration of 1.5 wt% to 2 wt% the transition time was reduced and cement slurry gained 500 Ib/ 100ft^2 in short time. The transition time of slurry-24 was 24 minutes. The increased concentration of HPMC polymer increased the gel strength of cement slurry. As a result, the slurry developed the desired value of 500 Ib/ 100ft^2 in a short time than the 1.5 wt% based slurries. From this result, it was concluded that slurry 11, 13 and 24 are good candidates for prevention of gas migration. Because the transition time of these slurries was low and they developed 500 Ib/ 100ft^2 in short time. After the completion of transition time the cement slurry became self supporting and the hydrostatic pressure remained higher than formation pressure. Further the conformation of gas migration prevention of these slurries was completed through next experiment using Cement Hydration Analyzer (CHA).

4.3.2 Gas Migration Test of HPMC Based Cement Slurries

The gas migration through cement slurries was determined using CHA. The gas migration of all cement slurries was determined at 194 °F that is equivalent to 90 °C. For the gas migration test the H_2O confining pressure was set at 350 psi and minimum pore pressure at 150 psi. The gas migration of cement slurry-12 without HPMC solution is shown in Figure 4.30.

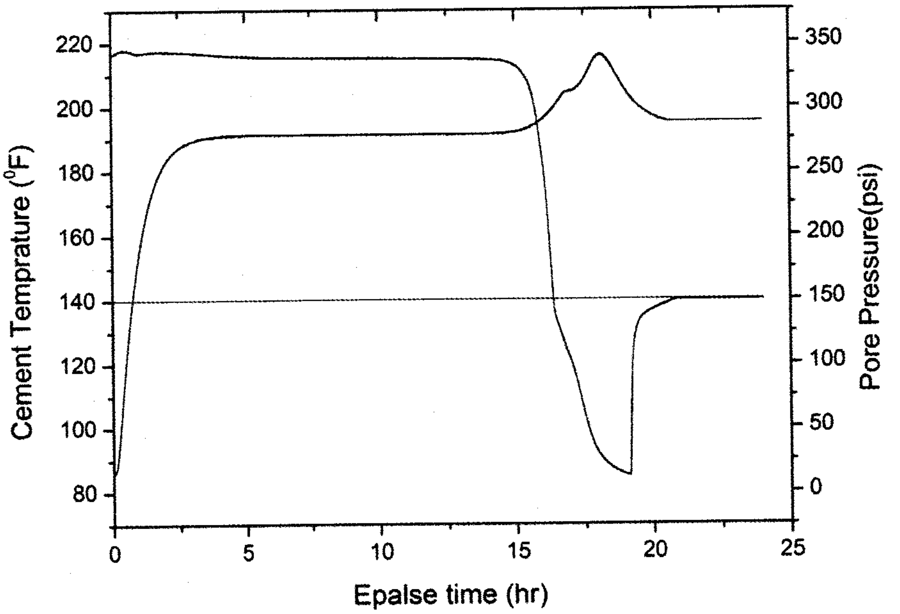


Figure 4.30: Gas migration test of slurry-12 (without HPMC Polymer).

In the initial stage, the H_2O confining valve was open and the gas injection valve was close. The cement temperature of the cement slurry was increased from 90 °F (32.2 °C/ room temperature) with increasing time and the pore pressure was decreased from 342 psi. In 2 hours the cement temperature reached elevated temperature. In the start of experiment the cement slurry was in liquid stage and the pore pressure was decreased slowly. The cement slurry started gel development after 16 hours at 194 °F (90 °C). The sharp decrease in pore pressure was due to the gelation behaviour of the cement slurry. The decrease in pore pressure gave the indication of cement shrinkage. When H_2O confining pressure reached 150 psi minimum pore pressure, the gas injection valve was opened and the confining pressure valve automatically closed. The pore pressure continuously decreased and reached 14 psi within 19 hours. Here, slight increase in temperature was observed. The increase in temperature gave the indication of cement hydration. Then, the pore pressure started to increase and reached 147 psi pressure within 1 hour. The increase in pore pressure showed that the slurry was not gas tight because the pore pressure reached the minimum pore pressure. The gas was migrated from cement slurry-12. Next the gas migration of cement slurry-8, which consisted of 20 gps concentration HPMC with other additives, was determined as shown in Figure: 4.31.

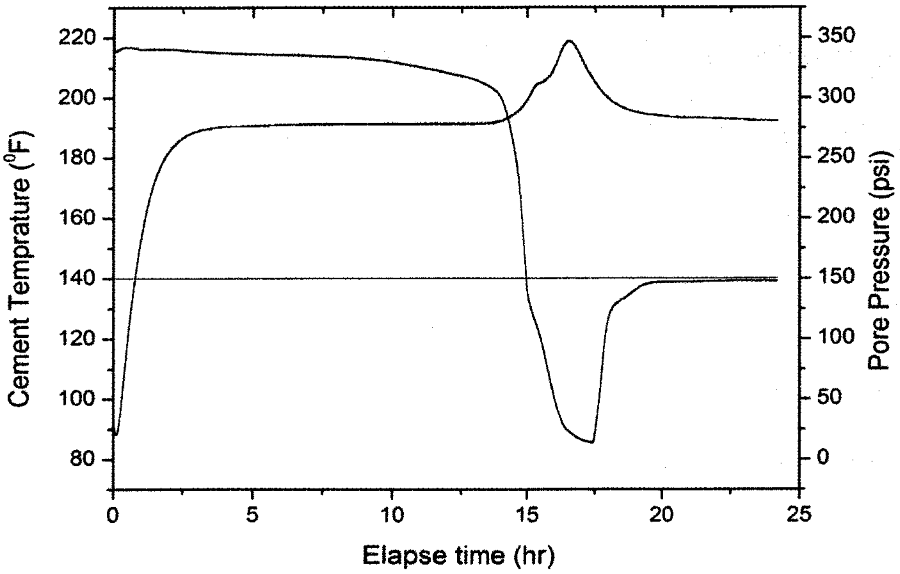


Figure 4.31: Gas migration test of slurry-8 (20gps of 1.5 wt% HPMC solution).

It was observed that slurry-8 was also not gas tight cement slurry because the pore pressure became equal to the gas injection pressure. Like previous slurry-12, initially the pore pressure decreased with time elapsed and the slurry started to develop its gel strength in 15 hour because the sharp reduction in pore pressure was observed. Here, the chemical shrinkage took place after the initiation of gel strength. The shrinkage of cement slurry showed that the slurry reduced the volume and decreased the hydrostatic pressure. As the cement slurry reduced the hydrostatic pressure, it increased the pore pressure from 15 psi to 150 psi. Moreover, it became equal to the constant gas injection pressure that was maintained at 150 psi. The test result indicates that the slurry-8 was not gas tight and gas could migrate through the cement slurry. The gas migration of cement slurry-11 was determined and the result is shown in Figure 4.32 given below.

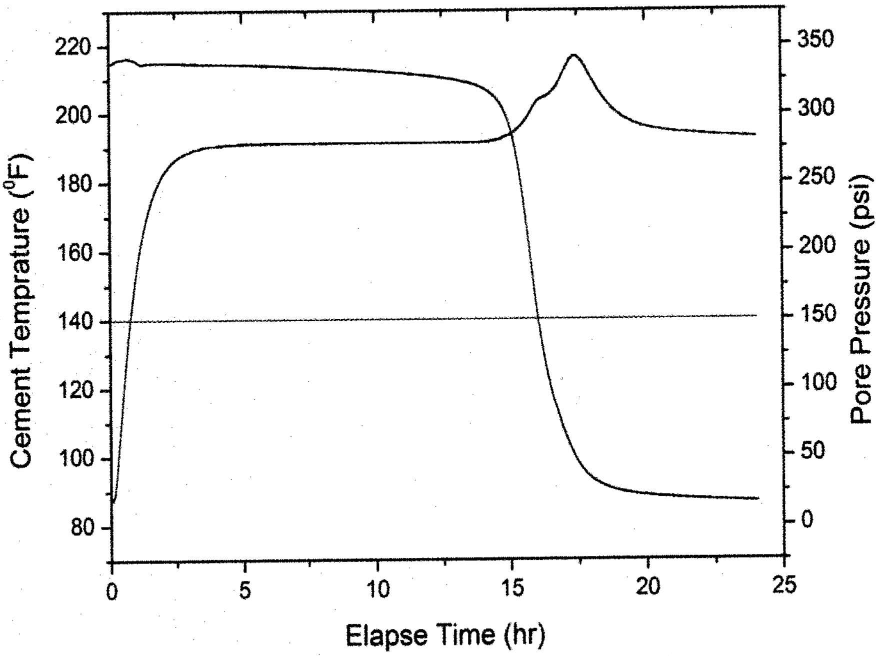


Figure 4.32: Gas migration test of slurry-11 (30gps of1.5 wt% HPMC solution).

It was observed that cement slurry-11 acted as liquid and fully transmitted the hydrostatic pressure. Within 16 hours the cement slurry started to develop gel and the pore pressure line dropped from 310 to 17 psi. Unlike the previous slurry, the pore pressure was not increased after the completion of gelation phase of slurry. The cement hydration process started after 18 hours because of the rise in temperature. The pore pressure of cement slurry decrease and then it became constant. Here, the injected gas did not affect the pore pressure that remained constant and low at 14 to 17 psi for the continued 6 hour gas injection. The constant pore pressure result indicates that cement slurry-11 was gas tight and performed effectively in gas migration control. In the cement slurry, the HPMC solution increased the viscosity of slurry. The HPMC solution decreased the permeability, fluid loss and shrinkage of slurry. Therefore, the hydrostatic pressure remained higher than formation pressure and the pore pressure became constant at 14 to 17 psi.

Further the gas migration of cement slurry-13 was also determined. In composition of cement slurry -13, 40 gps HPMC, 0.50 gps FLA, 0.05 gps dispersant and 0.03 retarder were used. The gas migration result of cement slurry-13 is shown in Figure 4.33.

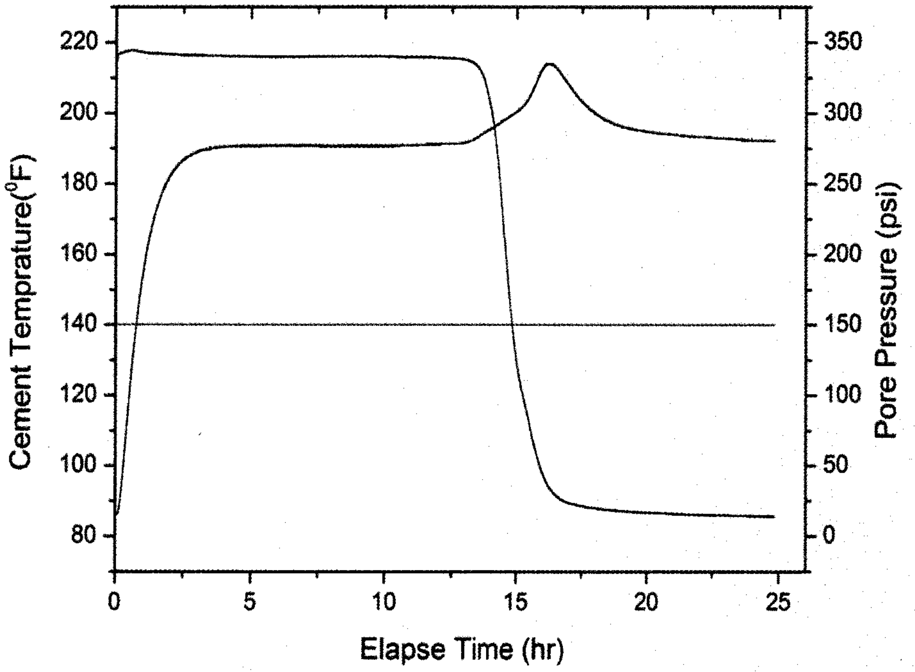


Figure 4.33: Gas migration test of slurry-13 (40gps of 1.5 wt% HPMC solution).

The above Figure 4.33 showed that slurry-13 was also gas tight. The pore pressure was not increased as the cement started to hydrate. As for slurry-11 the pore pressure decreased due to the gel strength development. The cement started to hydrate in 16 hours and the pore pressure became low at 13 psi. It remained constant until the completion of experiment of 24 hours. In last, the gas migration of slurry-24 was determined. This slurry contained 30 gps concentration of 2 wt% HPMC solution. The gas migration test of slurry-24 is shown in Figure-4.34 given below.

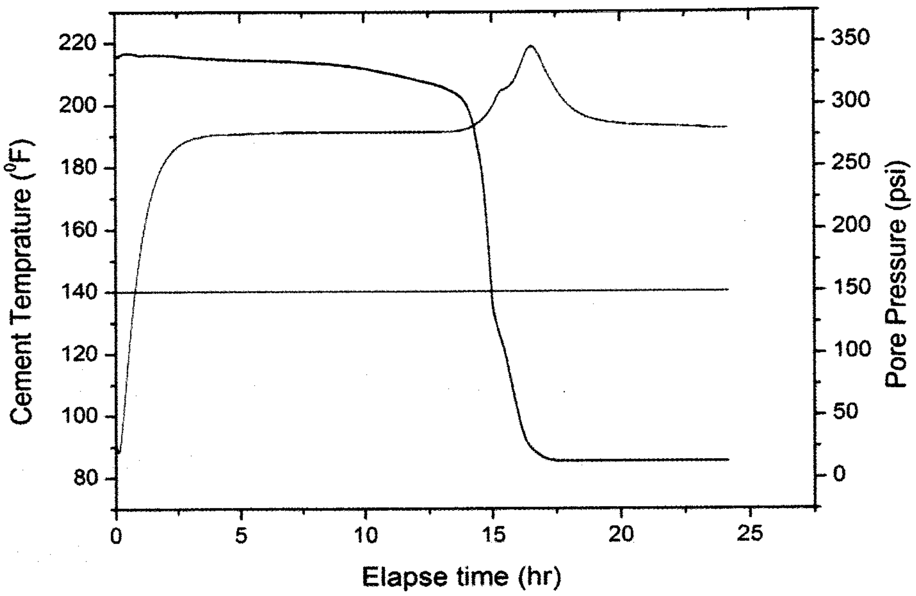


Figure 4.34: Gas migration test of slurry-24 (30gps of 2 wt% HPMC Polymer)

It was observed that slurry-24 prepared using 2 wt% HPMC solutions was also gas tight because after the hydration process of slurry, the pore pressure remained constant for 8 hours. The aim of this slurry test was to observe the effect of increasing concentration of HPMC polymer in term of wt%. Hence, the results of gas migration test concluded that HPMC polymer acts as viscosifying agent and prevents the gas migration through cement slurries at 90 °C.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

This chapter provides the conclusions from the experimental results of this research. Further the recommendations for future works are also presented.

5.1 Conclusions

1. The viscosity of HPMC solution was increased by increasing the concentration of HPMC solution in term of weight percent. The HPMC solutions (1.5 wt% and 2 wt%) showed increased viscosity at 90 °C. The enhanced viscosity of solutions proved that HPMC polymer was stable and acted as viscosifying agent at 90 °C.
2. HPMC solutions compact the influence of temperature for decreasing the rheology of cement slurries at high temperatures. The fluid loss was reduced from 740 to 168 ml/30 of slurry-7 by addition of 0.80 gps of HPMC solution. It showed that HPMC polymer acts as fluid loss control additives in cement slurry.
3. The addition of HPMC solutions in cement slurry completely prevents the free water separation and sedimentation of particles. The enhanced thickening time 5.54 hours of slurry-14 and 6.52 hours of slurry-26 were obtained. It showed that HPMC solution acts as a retarding agent in cement slurry. The HPMC based cement slurries showed high compressive strength. The compressive strength of cement slurries increased from 2430 psi to 4100 psi by the addition of 30 gps of HPMC solution.
4. The low transition time 39 minutes, 28 minutes and 24 minutes of cement slurry- 11, 13 and 24 were obtained. At high temperature, the HPMC polymer increased the gel strength that reduced the transition time of cement slurries.

Hence, the HPMC solution had a positive impact for reducing the transition time of cement slurry.

5. In gas migration test, the pore pressure of HPMC based cement slurries was less than 20 psi and it remained constant for 6 to 8 hours of continues gas injection. Hence, the experimental result showed that the HPMC polymer acts as a viscosifying agent that reduces the permeability of cement slurry and prevent the gas migration through cement slurry.

5.2 Recommendations

1. The viscosity of HPMC solutions has been determined up to 100 °C. Further the rheology of HPMC solution with brine and high salt concentration should be determined. These tests will be beneficial for petroleum industry in EOR and profile modification application.
2. The implementation of HPMC polymer in cement slurries showed good results and prevented gas migration at 90 °C. Therefore, it is suggested that further tests of gas migration and API properties of HPMC based slurries should be conducted at extremely high temperatures (150 °C - 300 °C).
3. Primary gas migration through cement slurry was successfully prevented using the HPMC polymer. However, further tests should be conducted for investigating the secondary gas migration.

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3. Ghulam Abbas, Sonny Irawan, “Experimental Study of Gas Migration Prevention Through Cement Slurry Using Hydroxypropylmethylcellulose,” *14UNCV-167804-MS- SPE/EAGE European Unconventional Resources Conference & Exhibition,* Vienna, Austria, February 25-27, 2014.