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Evaluation of robust epoxy resin sealants for wellbore integrity applications

Mohammed Mousa M Alkhamis

Department: Geosciences and Geological and Petroleum Engineering

EVALUATION OF ROBUST EPOXY RESIN SEALANTS FOR WELLBORE

INTEGRITY APPLICATIONS

by

MOHAMMED MOUSA M ALKHAMIS

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Approved by:

Abdulmohsin Imqam, Advisor
Shari Dunn-Norman
Kelly Liu
Taghi Sherizadeh
Mahmoud Elsharafi

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ABSTRACT

During the drilling and completion phases of oil and gas wells, cement is placed in the wellbore as a barrier between the casing and the formation. All casing strings must be cemented to protect and support the casing, and to isolate production zones. The primary cement must prevent the wellbore fluids from migrating in an annular flow path so as to allow the wells to be utilized without any control problems. The primary cement may fail to deliver full zonal isolation due to several reasons such as insufficient mud removal before the cementing, casing expansion, and contraction, high fluid losses, cement free fluids, inadequate hydrostatic pressure, high-pressure tests and temperature variations across the cement causing micro-annuli and cracks that may allow fluids to migrate. In addition, if the cement is placed in zones where corrosive fluids are presented, chemical degradation could compromise the cement integrity. If any of these failures occurred during the life of the well, remedial job must be performed to restore the well integrity. Failing to restore the cement integrity may lead to unwanted severe consequences to the environment, the equipment, and personnel. This work presents the results of evaluating several epoxy resin sealants that have the potential to replace the conventional Portland cement used in remedial jobs. This study includes the rheological behavior, curing kinetics, injectivity, plugging performance against water and CO_2, chemical resistance, and the mechanical properties of epoxy resin sealants. This work compares the results of epoxy resin sealants to that of the conventional Portland cement. The findings obtained from this work can be utilized in optimizing the cement remedial operations.

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IN THE NAME OF ALLAH, THE MOST BENEFICENT, THE MOST MERCIFUL.

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1. INTRODUCTION

1.1. STATEMENT AND SIGNIFICANCE OF THE PROBLEM

Gas, oil, and water are natural resources that can be found in subterranean formations. Recovering these valuable resources, usually require drilling a wellbore into the pay zone formation. During the drilling and completion phases of a wellbore, a casing (steel pipe) is run in the wellbore to provide an annulus for cementing. Next, primary cement is placed in the annulus as a barrier between the casing and the formation. The cement main functions are to protect and support the casing, and to isolate production zones. The cement must prevent the wellbore fluids from migrating in an annular flow path to allow the well to be utilized without any control problems. The main objective of cementing the annulus is to provide zonal isolation of the formations that have been penetrated by the wellbore. The cement must restrict any fluid communication during the life of the well among these various formations and the surface. If the primary cement failed to deliver full zonal isolation at any period of the life of the well for any reason, a remedial job must be performed to restore the integrity of the cement.

Despite the numerous amount of research and the numerous field operations that have been conducted throughout the world, cement failures are still occurring within the life of wells from the drilling phase to the abandonment of the well (Santos, 2015). During the life of the well, the primary cement may fail to deliver full zonal isolation due to several reasons such as insufficient mud removal prior to cementing, casing expansions, and casing contractions. These conditions may cause micro annuli either between the cement and the casing or between the cement and the formation. Other failures such as channels may occur because of high fluid losses, cement free fluids, and inadequate hydrostatic pressure. Besides, high-pressure tests and temperature variations across the cement may cause cracks in the cement sheath. Also, if the cement is placed in zones where corrosive fluids are presented, chemical degradation could compromise the cement integrity. If any of these failures occurred during the life of the well, a remedial job must be performed to restore the well integrity. Failing to restore the cement integrity may lead to unwanted severe consequences to the environment, the equipment, and personnel.

The potential leakage of fluids may compromise the efficiency of hydrocarbon recovery and carbon storage. Carbon storage is part of the Carbon Capture and Storage (CCS) program, which is a program that has been developed to reduce the amount of Carbon Dioxide (CO_2) emissions in the atmosphere by capturing, transporting, and securely injecting CO_2 in depleted oil and gas reservoirs or in deep saline aquifer formations (CCS Association, 2019). CO_2 is one of the greenhouse gases that became a serious issue due to its impact on the environment. CO_2, which makes up around 81% of the greenhouse gases emitted in the United States according to the U.S. Environmental Protection Agency (EPA, 2016), can raise the global temperatures, reduce water supplies, and alter the growing season for food crops. For these reasons, it is essential to ensure that the CO_2 injection wells are well isolated to keep the CO_2 underground and prevent any gas migration. Gas migration, when occur, is reported through pressure buildup, referred to as “sustained casing pressure (SCP),” and can be a significant safety hazard. The Mineral Management Service of the United States reported in 2003 that SCP affects more than 8,000 wells in the Gulf of Mexico (Rusch, 2004). The well integrity of these wells must be restored to put them back safely into production or permanently plug them.

The Norwegian standard defines well integrity as "application of technical, operational, and organizational solutions, to reduce risk of uncontrolled release of formation fluids throughout the life cycle of a well" (Norsok D-010, 2013). Norsok D-010 specifies that: "there shall be two well barriers available during all well activities and operations, including suspended or abandoned wells, where a pressure differential exists that may cause uncontrolled outflow from the borehole/well to the external environment". For these reasons, primary cement designs must be optimized in such a way that accomplish short and long-term zonal isolation. In addition, existing wells with zonal isolation issues must be remediated by either squeeze cementing or any other sealant materials. Squeeze cementing is a process in which a cement slurry is placed in the voids behind the casing. This process is one of the most common methods that have been implemented in the field to repair the cement (Shryock and Slagle 1968). As an alternative, other sealant materials may be injected into the voids such as epoxy resin sealants. Epoxy resin sealants, which are mixtures of resin and hardeners, were effectively used in the past few years. However, this type of materials as wellbore integrity sealants are relatively new and require an intensive and comprehensive laboratory work to prove their effectiveness. To the author's best knowledge, there are many epoxy resin sealants available in the market, but the temperature activated epoxy resin sealant is the only sealant that has been used in the petroleum field. Temperature activated epoxy resin sealant requires a certain temperature level to cure and develop enough strength to plug cements' gaps. In this study, the temperature-activated epoxy resin sealant was investigated at different temperatures to quantify its curing time, rheological behavior, injectivity, plugging performance, chemical resistance, and mechanical properties. In addition, two more types of epoxy resins were evaluated in this research as potential cement sealants. This work evaluated three types of epoxy resin sealants and compared their results to that of the conventional cement.

1.2. EXPECTED IMPACTS AND CONTRIBUTION

Results obtained from this work will promote the use of epoxy resin sealants in the restoration of wellbore integrity. In addition, the results gathered herein will help in understanding the curing mechanism and the performance of epoxy resin sealants. The variety of epoxy resin sealants prepared and tested in this work can be used to optimize the selection of the sealant and will help in answering which epoxy can be used for which application depending on the location of the cement failure and the environment in that location. The following points are expected from this work:

- Rheological characterization of three epoxy resin sealants prepared using different base resins and several hardeners. This characterization will help in selecting the appropriate sealant depending on the factors that affect the operation such as the viscosity of the sealant as a function of temperature and shear rate, the curing time with respect to temperature, and the elastic and viscous moduli. The sealants investigated herein are for low, moderate, and high temperature applications or in other words for primary cement failures at the surface and/or at intermediate or production sections of the well.

- The plugging performance of the epoxy sealants against water and CO_2 in both cement cores and steel tubes will be assessed and compared to other sealants, which will provide insights into the applicability and the ability of these sealants to be alternative to the well-known Portland cement.
- Evaluation of the mechanical properties of the epoxy resin sealants including compressive and tensile strength. This evaluation will be compared to the cement results.
- The durability of the epoxy sealants compared to conventional cement in harsh conditions will answer the big question of how safe to use a relatively expensive material in the long term. This is also important from the environmental point of view.
- The injectivity of the epoxy resin sealants (solids-free) is compared to semi-solids sealants, and solids materials. In this study, the effect of the size and shape of the voids on the injectivity of the sealants is characterized. In addition, to the effect of the viscosity of the fluid and the effect of the injection flow rate on the injectivity, which will help in optimizing the selection of the proper sealant based on the application.

1.3. OBJECTIVES

The primary objective of this research is to provide an intensive and comprehensive laboratory study of several wellbore sealant materials in particular epoxy resin to solve wellbore integrity problems. The following objectives will be established from this research:

- To provide a better understanding about the appropriate epoxy resin sealant that should be applied for different environments including high temperature curing epoxy for deep well applications, low temperature curing epoxy for surface remedial operation, and moderate temperature epoxy for the applications that require moderate temperatures like a cement failure remediation that could occur behind an intermediate casing. This is important to eliminate the concern of premature curing which may result in a failure in restoring the integrity of a well.
- To examine the ability of the sealants to shut off water and CO_2 migrations. This includes determining the limits of the sealants in terms of blocking pressure.
- To investigate the injectivity of several materials and analyze the main factors that affect the injectivity of these materials, which is a key property that has not received enough attention in the literature on wellbore integrity. In this study, the primary factors affecting the injectivity will be studied individually to identify which were major and which were minor. These factors include the type of fluid, void size into which the remedial fluid is injected, viscosity of the fluid, flow rate of the injection, heterogeneity of the void, and effect of the injection on the properties of the injected fluid.

The results collected from this work will provide comprehensive knowledge and insights into epoxy resin sealants and their performance in restoring zonal isolation. The results will also highlight the vital role of temperature on the performance of this type of sealant compared to conventional Portland cement. This research covers the lack of laboratory works that investigate the performance of epoxy resin sealants in the oil and gas industry and compare their performance to that of cement and other polymer sealants.

1.4. SCOPE OF WORK

To achieve the objectives of this study, this work was divided into three main tasks as shown in Figure 1.1. Task 1 was conducting literature review and collect information related to cement failures. Task 2 was evaluating the injectivity of several types of cements' sealants including solids-free sealants (epoxy sealants and polymer solutions), semi-solids sealant (particle gel), and solids sealant (cement) in different void space features. Task 3 was an evaluation of three types of epoxy resin sealants. The evaluation included rheological characterization, plugging performance, chemical resistance, and mechanical properties.

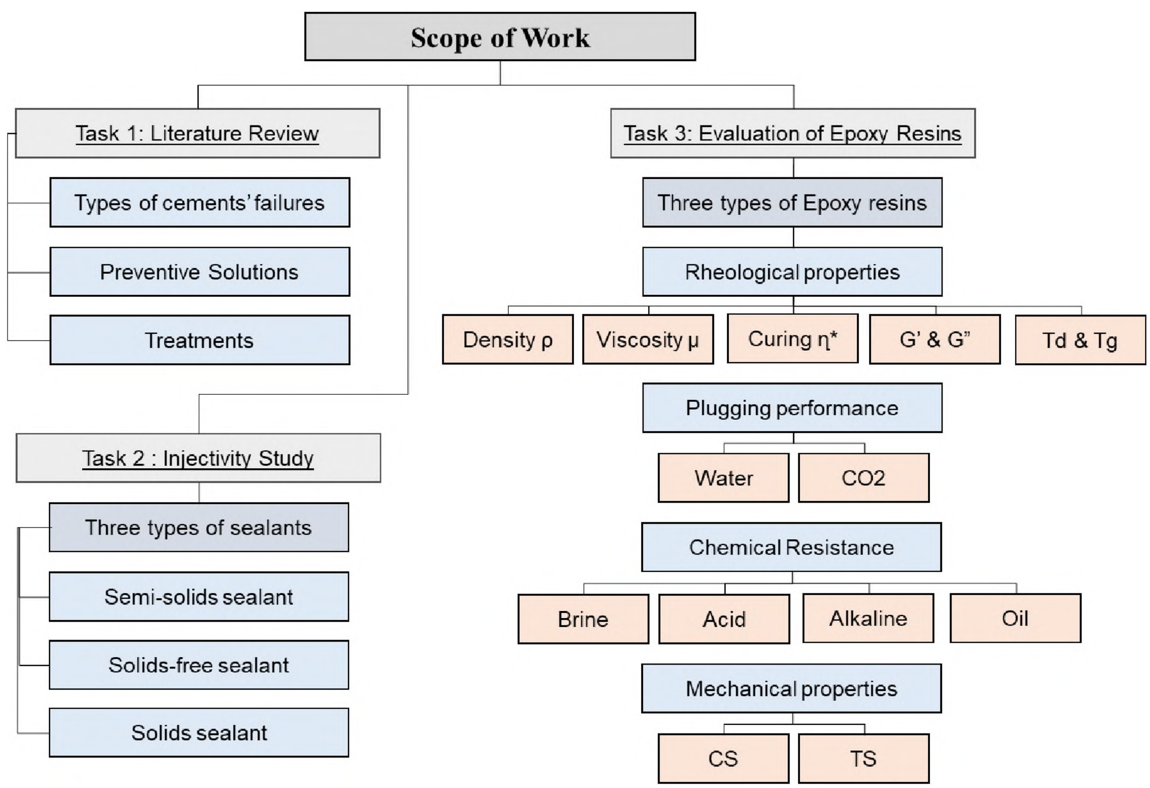

Figure 1.1. Research scope of work.

PAPER

I. A SIMPLE CLASSIFICATION OF WELLBORE INTEGRITY PROBLEMS RELATED TO FLUIDS MIGRATION

ABSTRACT

This work classifies in a simple form the wellbore integrity problems that cause fluids migration in oil and gas wells. This paper updates the categorization provided by [1], which classifies the wellbore integrity problems based on the time of manifestation. [1-3], and other researchers categorized the problems related to wellbore integrity similarly. These categorizations are found in multiple publications and are representative of most cements' failures that may lead to fluids migration. However, most of these categorizations are too broad and do not clearly state and describe the wellbore failures. In addition, categorizing the failures based on the time of their appearance may not be enough to realize, recognize, and understand how connected the failures are. Well-integrity failures are continuously occurring and represent major issues in the oil and gas wells. Therefore, a new and simple classification that accounts for the potential locations of each failure, the causes of the failures, and methods to stop the failures is necessary. This paper aims to classify the failures that may occur in the wellbore into four main failures: (1) micro-annuli that forms between the cement and its surroundings, (2) channels that creates pathways through the body of the cement, (3) fractures/ cracks in the cement sheath, and (4) cement chemical and mechanical degradation. Definition of these problems, causes of these problems, and solutions for those failures are summarized

in this paper to clarify, understand, and provide a guideline to prevent and avoid such problems. Along with the classification, general considerations for the cement-testing methodologies against these failures are listed. This work also provides the oil and gas industry with some of the technologies that have been used to avoid oil cements failures.

1. INTRODUCTION

Gas communication, gas leakage, gas seepage, gas and fluids migration, and multiple extra names are synonyms of a repetitive issue that occur in the oil and gas wells. This problem occurs due to failing in achieving full zonal isolation and results in costly maintenance operations and threats to the environment and surrounding communities. Gas migration or fluids migration in general can be a flow between different formations and zones, and fluids flow from one formation to the surface. Flow to the surface if it transpires it would be as short as within minutes or few hours of completing the well. On the other hand, flow between different formations and zones may not be spotted for weeks, months, or even years. Despite the numerous amount of laboratory research and field jobs that have been applied around the world, these flows are still turning out within the life's cycle of the wells starting from the drilling of the wells until the plug and abandonment of the wells [1]. One of the common methods in reporting gas migration is through a pressure buildup called sustained casing pressure (SCP). Studies have shown that around 60 % of the offshore wells in the Gulf of Mexico (GOM) have reported sustained casing pressure [4]. The well integrity can be defined as "application of technical, operational, and organizational solutions, to reduce risk of uncontrolled release of formation fluids throughout the life cycle of a well" [5]. Norsok D-010 specifies that: "there shall be two well barriers available during all well activities and operations, including suspended or abandoned wells, where a pressure differential exists that may cause uncontrolled outflow from the borehole/well to the external environment". For these reasons, optimizing the cement slurry designs is essential to guarantee zonal isolation [6].

During the drilling and completion phases of a well, a casing (steel pipe) is run in the wellbore to provide an annulus for cementing. The casing depth is determined using pressure safety drilling window, which contain a range of pressure from pore pressure to fracture pressure [7]. Next, a cement slurry is mixed at the surface and pumped through the casing to the annulus. A schematic diagram of a wellbore indicating the role of the cement as a barrier between the casing and the formation is shown in Figure 1. All casing strings must be cemented for two main objectives: (1) to protect the casing and support it and (2) to fully isolate production zones. For the well to be used without control problems, the cement must prevent the wellbore fluids from migrating in an annular flow path. Fluid communication should be restricted at any period of the life of the well among the various formations and the surface, no matter which fluids these formations are saturated with, water, oil, or gas [8-9]. The cement sealing off the annulus may be exposed to different condition that may compromise its integrity. Conditions such as pressure and temperature regimes, high mechanical loads from pressure testing and production operations. To achieve good cement job that provide full zonal isolation and that is able to withstand those conditions many considerations must be considered. All the variables related to cementing jobs must be analyzed, from the drilling to abandonment.

To achieve this and as a start, the cement failures must be classified according to their locations in the wellbore and the reasons behind them.

Figure 1. A schematic diagram of a well.

Today, studies focus on preventing these cement problems by modifying the formulation of the cement slurry, but the main drawback is that most of the studies deal with each failure individually, which leads to many inaccurate assumptions. That is why this study has been conducted.

The simple classification showed in Figure 2 classifies the potential cement failures into four categories: (1) micro-annuli that form at the cements' interfaces, (2) channels that are created through the cement body, (3) fractures/ cracks that develop in the cement, and (4) cement chemical and mechanical degradation. The simple categorization is constructed based on the location at which the failure may occur in the wellbore.

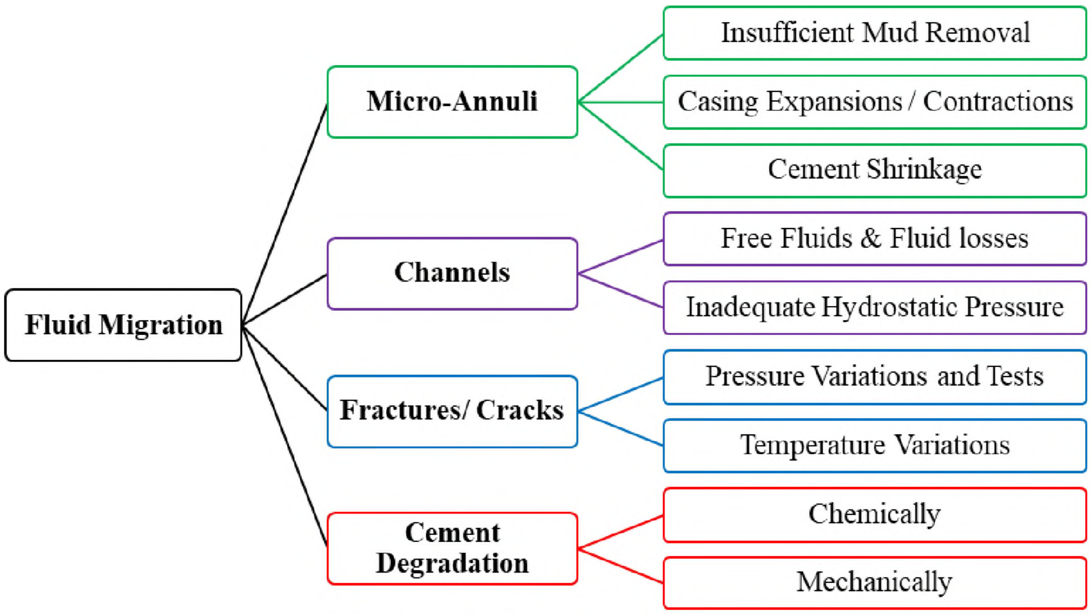

Figure 2. A simple classification of potential pathways for fluids to migrate.

2. MICRO-ANNULI AT THE CEMENTS' INTERFACES

The micro-annuli at the cements' interfaces should be understood and the causes of these gaps that potentially allow fluid migration should be studied to be avoided [3]. These gaps may allow pathways for formation fluids to migrate from high to low pressure formations, yielding to a dangerous pressure buildup [10-13].

The micro-annuli are mainly formed because of the debonding between the cement and its surroundings, as shown in Figure 3. Therefore, it is essential to design a cement slurry that is able to adhere with its surroundings. Failure to achieve and maintain such adhesion could lead to fluid communication between the different formations and between one formation and the surface [14].

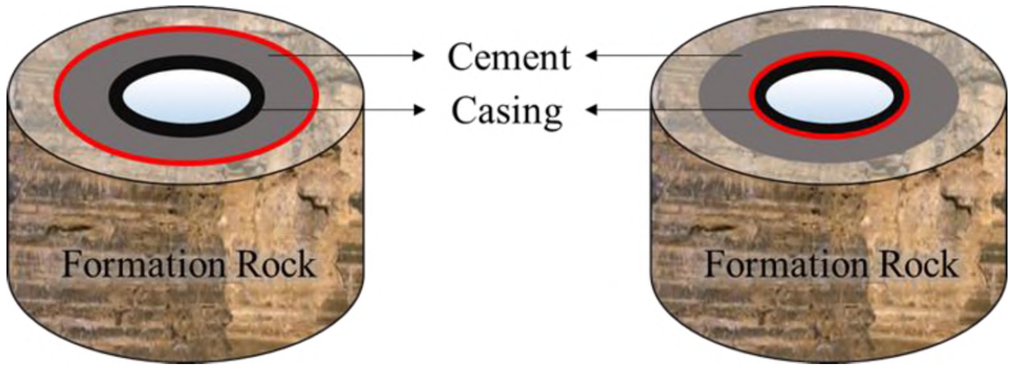

Figure 3. An illustration of the locations where micro-annuli may form in the wellbore.

The causes of the debonding between the cement and its surroundings are (1) insufficient mud removal resulting from drilling operation, (2) casing expansions and contractions resulting from production and/or completion operations, and (3) cement shrinkage, which is a phenomenon that cement go through during the hydration and setting processes. Table 1 lists the causes of micro-annuli and the time of occurrence for each of the causes.

Table 1. Causes of micro-annuli formation and their time of appearance.

The Cause	Time of Occurrence	Reference
Insufficient mud removal	During the drilling and cementing of the well	[15]
Casing expansions and contractions	At any period of the life's cycle of the well	[11]
Cement shrinkage	During the cementing process and/or after a long time of operation	[16]

2.1. INSUFFICIENT MUD REMOVAL

Eliminating the remaining mud from the well, especially the mud cake formed on the formation is a key step for attaining complete seal for the wellbore. Insufficient mud removal could compromise the zonal isolation. The mud cake formed on the formation is desirable for drilling operation to reduce fluid losses. However, it is undesirable for the cementing as the mud cakes are mainly made of polymers that degrade with time. The degraded mud cakes leave behind paths for fluids to migrate. In 1940, [17] revealed the effects of several parameters on the mud removal. Parameters like drilling fluid conditions, pipe centralization and movement during cementing, pump rate, and density differences between fluids, besides the effects of pre-flushes. This area of research resumed in the 1960s as [18] showed the benefits of pumping a highly dispersed slurry that is low in density and exhibit low fluid loss on the mud removal. During the same period, [19] presented their work, which showed the effects of surfaces types on the adhesion properties between cement and casings. Their conclusions included that good adhesion is achievable when coating the casing with sand, while the least adhesion was by having an oily casing. Another practice that must be considered is the centralization of the casings and was the topic of discussion in the 1970s, especially in deviated wells [6]. By late 70s, [20] estimated the mud mobility factor (MMF), which measures in percent the removed drilling fluid. By the 1980s, the industry finally started to combine all of the above parameters during the process of removing the mud. Table 2 summarizes the most important parameters regarding mud removal. It is vital when cementing a well to consider the importance of centralizing the casing to avoid settling related issues, the geometry of the hole to pump sufficient amount of cement, and the type of casing used.

Also, circulating the well and conditioning the drilling fluids to break the mud into thinner fluid, pre-flushing the well either mechanically or chemically, and the use of spacers must be considered side by side with the correct formulations of the fluids used.

Table 2. Notes for effective mud removal.

	Consideration	Notes	Reference
1	Casings Centralization	To prevent particles settling and to distribute the cement evenly	[6, 17]
2	Borehole geometry	To calculate the volumes accurately to ensure sufficient cements' height	[6, 20]
3	Casing selection	The type of casing, the running speed that may breakdown the formation, exacerbating the situation with loss circulation zones	[6, 19]
4	Circulating the well and mud conditioning	To thin the mud and break the gel particles left on the walls of the well	[6]
5	Pre-flushes	Mechanically by pumping the fluids to a turbulent flow at a specific rate: • Limited by well security. • Restricted by the wide side of the annulus. Chemically by flushing fluids in a laminar flow: • Using chemicals to pre-flush. • Protects the formation and provides stability.	[6, 21-23]
6	Spacers	Essential Spacers' Properties: • Compatibility with mud and cement. • Rheological properties and spacer weight. • Ability to alter the wettability of the casings and the formations. • Contact time and flow rates.	[6, 21-27]

2.2. CASING EXPANSIONS AND CONTRACTIONS

The expansion and contraction of the casings are other causes of micro-annuli. Casing expansion/contraction are outcomes of internal pressure or thermal stresses exerted to the casing [28]. Increasing the internal pressure or the temperature in the wellbore may expands the casing (ballooning effect), the cement then will counteract the forces from the expansion, which will induce stresses in the cement [29]. Consequently, if these stresses exceed the cement strength, failures will occur, and fluids migration pathways may be created. A decrease in the density of the drilling fluids or reduction in the temperature contracts the casing, resulting a gap between the cement and the casing [3, 8, and 11]. The consequences of casing expansions/ contractions should be mitigated through enhancing the shear strength and the tensile bonding strength of the cement design [14] as these consequences may compromise the cement integrity, resulting in permanent cement damages.

2.3. CEMENT SHRINKAGE

About 120 years ago, Le Chatelier, 1900 reported that Portland cement can shrink up to a 4.6% reduction in volume after setting. This was an important discovery. To measure the cement shrinkage, Le Chatelier used a simple setup that was a flask, named later the flask method [16]. In this method, the cement is poured in a flask and covered with water. The flask has a tube connected to it and the difference in the water volume in the tube is described as volume shrinkage of cement. Since that discovery and up until today, cement shrinkage has become an interesting topic. Many researchers around the world have been conducting laboratory experiments to scientifically explain the phenomenon of cement shrinkage. Cement shrinkage can be divided into two general shrinkages:

The first one: “Chemical shrinkage” this type of shrinkage is from the reaction between the water and the mineral compositions of the cement. There is a decrease in the absolute volume of the cement as the products from the exothermic reaction are lower in volume than that of the reactants. Such a reduction in volume may lead to micro-annuli formed, especially during early ages of the hydration process of the cement [30-31]. One of the theories indicates that the great factor influencing this type of shrinkage is the generation of calcium-silicate crystals. Throughout the hardening process of the cement, water adsorbs onto and is absorbs into these crystals, which reduces the total volume of the cement paste [32].

The second one: “Bulk shrinkage,” this type of shrinkage is associated to the cements’ pore structure as the cement loses its fluidity during the hardening and a capillary network is formed. The water contained in this network is consumed over time (dried); therefore, the capillary tension increases in the pores. In consequence, the pores collapse to release the tension, which originate a reduction in the external volume [16]. The pore contraction has the biggest effect on shrinkage [31 and 33].

How Does Cement Shrinkage Allow Fluids to Enter the Cement? The cement transformation from liquid state to solid state may allow fluids entry. Upon adding the water to the cement, the hydration starts, and the cement develops what is called static gel strength (SGS). When the cement slurry develops enough SGS, the transition time start. This transition time prevents full transmission of hydrostatic pressure. The transition time finishes as soon as the slurry advances enough solid properties to stop percolation of gas through the cement [34]. The shrinkage is of little importance prior to the start of transition period, as the movement of fluids will compensate the shrinkage in earlier stages. However, fluids may enter the cement column if the hydrostatic pressure drops under the pore pressure of the formation during the transition time. The hydrostatic pressure fall off is mainly caused by the first type of shrinkage, so it is essential to minimize it [31].

Cement shrinkage may let fluids to enter the cement, allowing fluid communication to take place. Measuring the cement shrinkage precisely is vital to enable the elimination of its effects. Multiple experimental methodologies were developed to quantify cement shrinkage. One of the very first methods is the flask method mentioned earlier in this paper is. In spite of the fact that it is still applied, the amount of slurry utilized in this test may affect the overall results negatively [16]. During the last decade, countless attempts were made to construct a method and a procedure to estimate the shrinkage accurately, for example [30-32, 35-36]. These works made an effort to modify the traditional methods specified by the American Petroleum Institute (API) namely, ring-mold, membrane method, and cylindrical-sleeve, to obtain results that are more reliable. Table 3 lists some of the most important considerations to achieve optimal measuring methodology.

The direction of shrinkage is another important parameter to be considered. For example, disking may occur due to axial shrinkage. If the cement exhibits axial shrinkage and cannot slide, disking occurs as illustrated in Figure 4. To avoid such cement failure the direction of shrinkage must be monitored during the testing of cement shrinkage.

Table 3. A list of the most important considerations for measuring cement shrinkage.

	Consideration	Note	References
1	Data Recording	Monitoring the first 24 Hrs. of the hydration process	[16, 30, and 32]
2	Accuracy of Instrument	The changes in shrinkage might be very small	[30]
3	Curing Conditions (P & T)	High pressure/temperature increase shrinkage	[16, 30-31]
4	Conditions (P & T) Must Remain Constant	Depressurizing has huge impact	[35]
5	Permeability of Formation	Whether cement will be exposed to water	[16]
6	Confining Environment in which the test is being conducted	To determine the direction of shrinkage	[16, 35]
7	Shear History of Slurry	Shearing the slurry reduces the shrinkage	[16]

Figure 4. Cement disking caused by axial shrinkage.

In addition, the properties of the cement slurry formulation must be perfected, beginning with selecting the proper water to cement ratio. Excessively minimizing the water content in the cement may compromise the compressive strength of the cement [32] although maximizing it reduces shrinkage. Also, the shrinkage when it occurs affects the bonding of the cement with its surrounding [39]. Additives can be introduced to the cement slurry to overcome shrinkage. Additives like bentoniter, sodium silicate, magnesium oxide (MgO), calcium oxide (CaO), and silica powder. Those additives can help reducing the effect of cement shrinkage and enhancing the bonding strength between the cement and the steel casing [37]. Table 4 lists additional considerations when designing a cement with low shrinkage.

Table 4. Considerations when designing a cement with low shrinkage.

	Consideration	**Note**	**References**
1	Free water	To reduce shrinkage free water must be minimized	[32]
2	Water to cement ratio	Increasing the water amount increases shrinkage	[16, 32]
3	Slurry Yield	The higher the yield the lower the shrinkage	[32]
4	Amount of Additives Must Be Optimized	Excessive expansion could yield to gas leakage	[35, 38]
5	Shortening Transition Time	This helps avoiding gas migration	[16, 31]
8	Thermal Behavior of the Cement	The thermal behavior has an effect on the shrinkage	[3]

3. THE CREATION OF CHANNELS

The channels that can be created through the cement sheath represent a dangerous issue, mostly in presence of hydrogen sulfide gas (H_2S) [40], the channel can be only in a small part of the cement sheath or extended to the whole section. However, the micro-annuli discussed above can provide for the channels a pathway allowing fluids migration.

Fluids may channel the cement mainly due to two reasons: (1) the cement design has high fluids loss or free fluids content and (2) improper hydrostatic pressure (the density of the slurry is not enough to exert pressure that keeps the formation fluids in the formations. Channels are created when the hydrostatic pressure exerted by the cement falls below the pore pressure of the formation. This may happen in the initial hydration process of the cement. Figure 5. Shows the locations of the channels in the wellbore.

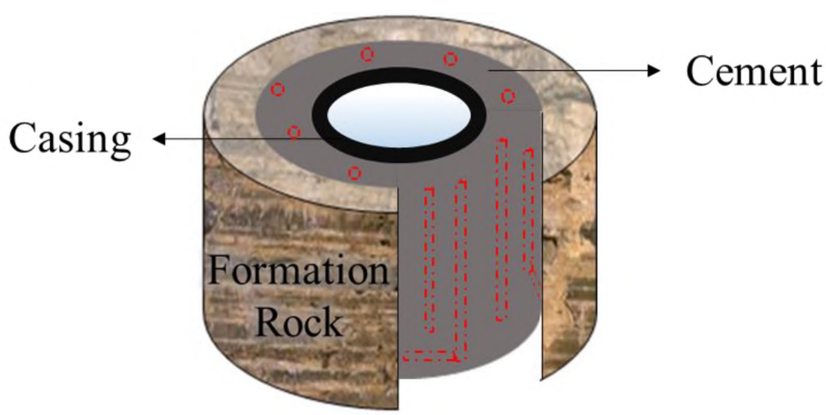

Figure 5. Channels created in a cemented annulus.

3.1. FREE FLUIDS AND FLUID LOSSES

During the placement process of the cement in the annulus of the wellbore and the setting time, the cement may lose some of its fluids to the porous formations. If the fluid loss is high, invasion of fluids to the liquid cement may take place. This fluids influx may establish irreversible pathways in the cement allowing the fluids to migrate between formations and, possibly, even to the surface [24]. The fluids lost from the cement may lead to a higher density slurry (possibly higher than the fracture gradient of the formation) that may break the formation. In addition, in this process, the cement particles may plug the formations' pores, constructing a cement filter cake. The thickness of the filter cake may increase as the displacement continues, which would lead to an increase in the required circulating pressure. Extreme cake thickness may plug the annulus, disabling additional displacement [41-42]. In addition, a cement slurry with fluids less than the planned might set prior to the end of the cementing, which would cause the column of cement to reach an insufficient height [43].

The hydration process of the cement is the main parameter that controls fluids migration. During the transition time of the cement, fluids can channel the cement. Ideally, cement slurry that sets immediately after placement will eliminate the channels problem. Nonetheless, such a slurry using Portland cement is impossible. Thus, shortening the transition time is the only way to mitigate this problem. Lowering the cement fluid loss may help also but the fluid loss additives tie up the water required for the hydration and release it in a slow manner through the hydration process. Yet, some of the water required for hydration may be driven out, creating micro-capillaries in the cement, another path for fluids to migrate. In addition, this may affect other properties of the cement, generating additional issues [41] such as the dilution of cement particles, which would negatively affect the compressive strength of the cement [32]. On the other hand, optimizing the required amount and correct type of fluid loss additives will maintain the fluidity of the slurry and will reduce the risk of premature dehydration [44]. The importance of minimizing free fluids, which are fluids that set apart from cement slurry when left at static conditions, was proved by [45]. This work proved lowering the fluid loss must be accompanied with low free fluids and preferably no free fluids.

3.2. INADEQUATE HYDROSTATIC PRESSURE

Inadequate hydrostatic pressure exerted by the cement column during its initial hydration may also create irreversible pathways for fluids through the cement sheath [1]. In the wellbore, two pressures must be kept: the formation (pore) pressure and the parting (fracture) pressure. The cement should exert a pressure higher than that of the pore pressure and lower than that of the fracture pressure [40]. Failing to do so increases the likelihood of having channels or damaging the formation. Fluids can only invade the cement slurry if the formation pressure exceeds the hydrostatic pressure. The hydrostatic pressure can be controlled by controlling the density of the cement. Controlling the density is by the use of additives. Several additives may be added to the cement slurry to increase or reduce the density such as barite and hematite for increasing the density and bentonite and glass spheres to reduce it. There is also the water-extended cement systems, which have relatively low density but also lower compressive strength, longer thickening time, and higher permeability compared to normal cement systems. One more approach to reduce the density is the injection of nitrogen gas with stabilizers to the slurry known as foam cement. This technique provides strong, low density, and low permeability cement system [46] but on the other hand a higher cost cement especially when it comes to the required equipment.

4. THE INITIATION OF CRACKS/FRACTURES

The initiation of fractures in the cement is considered a long-term issue. In several cases, cementing jobs were conducted effectively, resulting a good sealant, but eventually a pressure buildup was noticed [12]. Cement cracks may be the reason of pressure build up. Moreover, the cement deteriorates with time as result of post-cementing jobs like integrity tests, production, and stimulation [47]. Thermodynamic cycles also contribute in the initiation of this type of features [11]. Also, over pressurizing the casing or applying high thermal loads can destroy the cement [3, 48]. Cracks are also results of chemical degradation when some chemicals invade the cement and expand originating cracks inside the cement [49].

4.1. CASING PRESSURE VARIATIONS

At the end of the cementing job, the well is subjected to pressure tests to determine the integrity of the casing. High testing pressures can cause radial cracks in the cement [29, 48, 50-51]. These tests also develop shearing forces at the outer part of the casing, and in consequence, cracking might occur in the inner part of the cement [8]. In addition, conducting these tests before the cement fully cured rises the probability of forming micro-annuli [48]. Pressure variations in the casing appears continuously due to mud replacement (change in density), pressurizing tests (integrity and leak-off), stimulation, perforation, and/or remediation operations. When the pressure variation is an increase in pressure (positive pressure), this will lead to radial or shear cracks. Radial, if the cements' stiffness is larger than the stiffness of the formation and shear when the cement has larger flexibility [51]. For the case of negative pressure, micro-annuli may form. Figure 6 differentiates between the two damages.

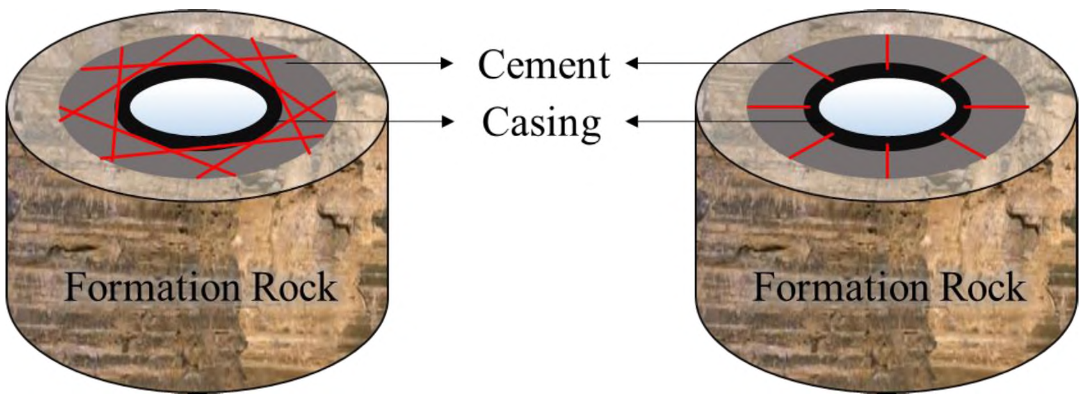

Figure 6. The left side shows the effect of shear damaging and the right side shows the radial cracks.

4.2. TEMPERATURE VARIATIONS

The effects of temperature changes are similar to those of the pressure changes. For the high temperature operations, the cement would be subjected to high temperature variations. The casing expands as result of the temperature increase, which may leave the cement in a large deviatory state of stress. Cement may crack from excessive temperature changes. The cracks may occur due to tension failure [51] or compressive failure [52]. The cracks may start at the cement-casing interface and propagates to the cement-formation interface. The crucial effects of these cracks are present when the casing contracts back to its original state (relaxes), causing the cracks to open and leading to annular flow [53]. It is important not to expose the cement sheath with a large temperature increase as the casing will expand more than the cement, which will induce compressional stresses in the cement (radial cracks) and similarly in temperature decrease the casing will contract more than the cement inducing tensile stresses in the cement (debonding). Reduce the sharp increase and decrease in temperature when possible. Table 5 summarizes the main notes for cement mechanical properties design.

Table 5. Mechanical properties notes.

	Consideration	**Note**	**Reference**
1	Cement Stiffness	Has a great impact on the mechanical properties	[51]
2	Formation Stiffness	Has a great effect on the mechanical properties	[8, 51]
3	Cement Thermal Properties	To count for changes in the cement volume	[29, 52]
4	Expected Loads on The Cement	To avoid overloading	[52]
5	The Confining Effect	Confining pressure on the cement supports it	[52]
6	Cement Flexibility	To avoid the consequences of casing expansion	[29, 51, and 54]
7	Cement Pore Pressure	High pore pressure may increase the chances of micro-annuli	[51]

5. CEMENT DEGRADATION

Cement like any other material may degrade with time. The degradation can be as result of chemical interactions or mechanical causes. For the petroleum industry, durability of the cement used is an important parameter. Durable cement or durability of cement may be defined as a cement that preserves its mechanical properties and low hydraulic conductivity (porosity and permeability) with time [55]. Durability of oil well cement may be compromised by one of two degradations (1) chemically and (2) mechanically.

Chemically: The oil well cement is in contact with several kinds of fluids and gases during the lifetime of the well and even after abandonment. This contact may degrade the cement. Thus, the durability of cement must be examined in the presence of different corrosive fluids that can be found in oil and gas reservoirs, such as hydrogen sulfide, carbon dioxide, and brines.

Mechanically: The oil well cement is also under compressive and tensile loadings during the lifetime of the well and after abandonment. These loadings may exceed the cement strength, leading to unwanted consequences compromising zonal isolation.

In the hydration process, Portland cement produces calcium-silicate-hydrates (C-S-H) and calcium hydroxide (CH). The C-S-H is responsible for increasing the strength, while CH does not contribute as much. Generally, lowering the permeability of the cement and reducing its calcium hydroxide content help increasing the chemical resistance [49]. Chemicals can penetrate the cement and expand causing internal cracks or dissolve into the cement and destabilize the C-S-H crystals. Laboratory experiments for testing the durability of the cement must simulate the downhole thermodynamic conditions (pressure, temperature, fluids, etc.) to better understand the cement degradation and prevent it. For some cases, an alternative to Portland cement is essential especially for permanently plugging [55].

6. CEMENT SOLUTIONS

Along with the good cementing practices described in this paper, the cement properties can be modified in a way that prevent the wellbore integrity failures from occurring. In this section of this work, some of the cementing systems that have the potential to overcome the mentioned failures are briefly listed and reviewed.

6.1. FOAMED CEMENTS

Foam cement is a cement slurry that is mixed with foaming agent (nitrogen or air) along with an adequate amount of stabilizers. This type of cement was proposed back in the 1980s [56]. This type of cements is mainly used for low fracture gradient formations where cements with low density are required. Foamed cements are low in density and behave like thixotropic fluids, meaning the cement slurry remains fluid when sheared but develop high viscosity when the shearing stops [57]. This behavior reduces the transition time of the cement and hence reduces the probability of fluids migrations. This cement develops relatively high compressive strength and high elasticity [58].

6.2. ELASTIC (FLEXIBLE) CEMENTS

Flexible or elastic cements are cements that are mixed with elastomers (rubber) and fibers. Adding such additives to the cement slurry increases the tensile strength of the set cement and lowers the Young's modulus [58] thereby enhance the mechanical properties of the cement. Enhancing these properties reduces the possibility of having cracks and fractures within the cement sheath as results of casing expansion and contraction.

6.3. DENSITY CONTROLLED CEMENTS

One of the main concerns when cementing the oil and gas wells is the density of the cement. Reducing the density of the cement requires increasing the amount of water in the slurry known as water-extended cements, which might compromise other properties like the strength of the set cement.

Other methods to reduce the cements' density is adding other extenders such as sodium and potassium silicates. These extenders also have limitations when it comes to the strength of the cement and the thickening time of the cement. This led to the use of glass beads (microspheres that have the ability to lower the density without or with little effect on the strength).

On the other hand, increasing the cement density can be achieved by adding weighting agents such as barite and hematite. Generally, controlling the cement density helps avoiding the channeling problems.

6.4. GEOPOLYMER AND POLYMERIC CEMENTS

Polymeric cements or cement s that contains polymers are cement systems with superior properties. Enhancing Portland cement by adding thermosetting polymers such as epoxy resin or latex like styrene butadiene latex enhances the mechanical properties of the cement like tensile strength, elasticity, and more importantly the bonding properties. These types of cements have better resistance against chemicals and contaminations. Geopolymer cement is another new product that may have the potential to overcome the shrinkage problems. Geopolymer cement is a mixture of fly ash, which is a by-product with an activator. The activator is a mixture of sodium silicate and sodium hydroxide (NaOH). The strength of this type of cement depends on the molarity of the NaOH and the amount of sodium silicate added.

7. DETECTION TECHNOLOGIES AND TREATMENTS FOR WELLBORE FAILURES

So far, this work discussed the cement failures that may occur during the life span of oil and gas wells, the causes of those failures, and some of the preventive solutions to avoid those failures. However, those failures have occurred in many wells around the globe and the integrity of the wellbores are prone to more failures in the future. For this reason, it is vital to discuss the failures' detection techniques and the available solutions and treatments for wellbore integrity failures along with some of the parameters that affect the selection criteria of the treatments.

7.1. DETECTION TECHNIQUES

After the placement of the cement in the annular between the casing and the rock formations, there are several tests and loggings conducted to confirm zonal isolation. Some of these are conducted during the life of the well and some after well abandonment. Typically, an annular pressure test, also known as "casing pressure test" is run. Any casing below the conductor should be tested to 500 psi or 0.22 psi/ft, whichever is greater [59]. The test is run for about 12 hours. If there are more than 10% reduction in pressure within the first 30 minutes or there are any indication of leakage, the casing should be recemented or repaired to ensure zonal isolation and no significant leak in the subsurface system. This is not the only method as there many other accepted methods such as annular pressure monitoring and radioactive tracer survey. There are also tests to examine the state of the wells after some time have passed such as fiber optic temperature surveys, noise-log, and the cementation logs such as cement bond logs, ultrasonic imager tool (USIT), and isolation scanner [60]. To detect the annulus content and whether micro annuli have formed cement bond logging are being used to measure the bond between the casing and the cement. The measurements are conducted by using acoustic sonic and ultrasonic tools and displayed in millivolt units, decibel attenuation, or both. An increase in the decibel attenuation or decrease in the millivolts are indications of better-quality bonding of the cement and its surroundings [61]. For the channels and cracks that might develop within the cement sheath, isolation scanner is used for detection. Isolation scanner evaluate the cement job and the casing conditions to confirm the effectiveness of the annular barrier for zonal isolation. This technique distinguishes the low-density solids from liquids to point out whether channels have been created in the cement sheath or not.

Another detection technique is evaluating the state of the casing in the wellbore using caliper survey and wall thickness survey to identify whether the casing in a good condition or corrosion, deformation, and weight loss is occurring [60].

7.2. TREATMENTS FOR WELLBORE FAILURES

In the past years, the oil and gas industry have worked hard to develop several materials to be used for treating cement failures. Starting by the use of conventional oilfield cement to the use of nanotechnologies. Generally, the treatment for all of the categories is selected based on many parameters including but not limited to the aperture size, the equipment available on site, the accessibility to the failure zones, etc. As a rule of thumb, if the size is more than 400 micrometer, conventional oil field cement is injected to the affected zones to repair the primary cement. If the size is less than 400 micrometers but higher than 120 micrometer, micro-fine cement and ultra-fine cement are the solutions as those two types of cement have smaller particle size compared to the conventional cement. If the size is less than 120 micrometer, advanced technologies such as polymer resin are being used to mitigate the problem. Polymer resins are thermoset materials that are solids-free and can penetrate very tight gaps [62].

For all the classes of failures mentioned in this work, polymer resins (epoxy resins) seems to be the most effective solution. It is a new technology for the oil and gas industry but it has been applied successfully in oil and gas wells lately and the reason why we it is believed that this type of material is one of the best is its ability to develop high compressive and tensile strength reducing the possibility of having cracks and fractures in the cement, it ability to penetrate small gaps that other sealing materials cannot access, its ability to develop bonding with cement, formations, and steel filling the micro annuli around the cement, and its stability under high temperature conditions.

8. CONCLUSIONS

This study provides a guideline to better understand wellbore integrity problems and work in avoiding them. Publications from around the world were reviewed, experimental techniques were evaluated, and data from laboratory experiments were analyzed to summarize wellbore integrity failures. This study classified the wellbore integrity problems in a simple way to help prevent those failures. The main conclusions obtained from this work are as follow:

- Today, the petroleum industry focuses on the short-term properties, which is good for the first days after the cement placement. However, the long-term properties are as important as the short-term and tests should be conducted for the durability of the cement.
- The initial state of stress in the cement sheath must be studied to estimate the limitation of the cement downhole.
- To better evaluate the state of stress in the cement after setting, the hydration process of the cement and time effect should be analyzed.
- It can be concluded from this review that the sealant material for oil and gas wells must follow these criteria:
    - Has low environmental impact with desired density.
    - Low fluid losses and very little to zero free fluids.

- High mechanical strength and experience low shrinkage.
- Short transition time and chemical resistance.
- Adhesive properties with steel and different types of formations.

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II. SEALANT INJECTIVITY THROUGH VOID SPACE CONDUITS TO ASSESS REMEDIATION OF WELL CEMENT FAILURE

ABSTRACT

The primary cement of oil and gas wells is prone to fail under downhole conditions. Thus, a remedial operation must be conducted to restore the wellbore integrity and provide zonal isolation. Many types of materials are currently used and/or have the potential to be employed in wellbore integrity applications, including, but not limited to, conventional Portland cement, microfine and ultrafine cement, thermoset materials, and thermoplastic materials. In this study, several types of materials were selected for evaluation: (1) conventional Portland cement, which is the most widely used in remedial operations in the petroleum industry, (2) polymer resin, which is one of the most recent technologies being applied successfully in the field, (3) polymer solutions, and (4) polymer gel, which is a semisolid material that has shown potential in conformance control applications. This work addresses injectivity and the parameters that affect the injectivity of these materials, which to the authors' best knowledge have not been addressed comprehensively in the literature. The results of this study demonstrate the effects of several factors on the injectivity of the sealants: void size, viscosity of the sealant, injection flow rate, and heterogeneity of the void. The results also promote the use of solids-free sealants, such as epoxy resin, in wellbore remedial operations because epoxy resin behaved like Newtonian fluid and can therefore be injected into very small voids with a minimum pressure requirement.

1. INTRODUCTION

During the life of oil and gas wells, the wellbore cement is subjected to numerous types of failures, with many causes, as addressed by Alkhamis and Imqam (2021). Failures include, but are not limited to, the formation of micro-annuli between the well cement and its surroundings, cracks and fractures within the cement sheath, and channels that may develop during the hydration process of the cement. The failures may occur due to insufficient mud removal before the cementing operation, improper hydrostatic pressure delivered by the cement slurry during the primary cementing operation, casing expansion and contraction, and/or post-cementing causes, such as high-pressure tests and high-temperature variations during production (Thiercelin et al., 1998; Alkhamis and Imqam, 2018). Whether these failures occurred during drilling and completion, production, or even after abandonment, a remedial cementing job or “secondary cementing” operation is performed to restore the wellbore cement integrity. The well integrity is known as “application of technical, operational, and organizational solutions, to reduce risk of uncontrolled release of formation fluids throughout the life cycle of a well” (Norsok D-010, 2013). For this reason, the wellbore cement integrity must be maintained through the life of the well and after abandonment. Prior to any remedial operation, an injectivity test, using solids-free fluids, is conducted to determine some of the parameters required for secondary cementing as this operation requires a careful analysis. This analysis includes estimating the significance of the problem, evaluating the associated risk factors, selecting the proper sealing material, choosing the placement technique, and assessing the economic costs.

Many materials can be used to seal the fluids' pathways, which are created in the wellbores when failures occur, including conventional Portland cement, microfine cement, ultrafine cement, polymer gels, and polymer resins. The advantages and disadvantages of each of these sealing materials is discussed below.

For decades, Portland cement has been employed as the first choice for remedial jobs (Shryock and Slagle, 1968). In a process known as squeeze cementing, the cement slurry is forced into an opening in the casing to fill the voids behind the casing. The cement will then hydrate inside the voids, plugging any pathways for fluid migration. Although, this might sound like an easy process, it is associated with many complications. For example, the process may require more than one squeeze to plug the voids (Sanabria et al., 2016), or it might need a different squeezing technique, such as hesitation squeeze. In addition, this operation is limited by the void size (Jones et al., 2014), as the cement's solid particles may form a bridge in narrow clearances (Davis, 2017). Microfine and ultrafine cement are also limited in penetrating voids less than 300 microns in width (Wasnik et al., 2005). Another limitation is the vulnerability of the cements' thickening time during which it can be affected by contamination (Dahlem et al., 2017). Also, this operation requires casing perforation in general. As alternatives, polymer gels and polymer resins may be used in cement remediation to overcome the limitations of conventional cement.

Polymer gels are mixtures of polymers and crosslinkers that can be combined and optimized to transform from liquids at the surface to semisolids in place. The polymerization may be activated by pressure, temperature, salinity, or acidity. Polymer gels can penetrate small voids, but the 3-D structure of the gel may break down at high temperatures, losing its ability to trap fluids such as water in conformance control applications. Other limitations of polymer gels are their lack of mechanical strength (Wasnik et al., 2005) and bonding properties (Abdulfarraj and Imqam, 2019).

On the other hand, polymer resins, which are mixtures of base resins and hardeners (i.e., “curing agents”), have superior properties, as reported repeatedly in the literature. Polymer resins are defined as “free flowing polymer solutions that can be irreversibly set to hard, rigid solids” (Morris et al., 2012). The exceptional properties of polymer resins vary and include pre-curing (e.g., their tunable rheological behavior) (Alsaihati et al., 2017); surface wetting and adhesion abilities (Brooks et al., 1974; Shaughnessy et al., 1978); the ability to penetrate narrow voids (Todd et al., 2018); tunable density (which can be used for areas with a narrow fracture gradient) (Sanabria et al., 2016); and resistance to contamination (Perez et al., 2017). In addition, polymer resins possess excellent post-curing properties, such as high mechanical strength (Ali et al., 2016; Elyas et al., 2018), high resistance to significant strain (Khanna et al., 2018), and good bonding properties (Genedy et al., 2017). As a bonus, the polymerization reaction of polymer resins forms no by-product during hardening (Muecke, 1974), resulting in very little to no shrinkage. These properties have been also reported by Alkhamis et., (2019).

Selecting the proper sealant depends on the field job itself, meaning there are no general guidelines or rules to be followed in every case. However, there are some materials properties that can help to increase the possibility of a successful remedial operation. This work focuses on estimating the injectivity of several materials and analyzing the main factors that affect the injectivity of these materials, which is a key property that has not received enough attention in the literature on wellbore integrity. In this study, the primary factors affecting the injectivity were studied individually to identify which were major and which were minor. These factors included the type of fluid, void size into which the remedial fluid was injected, viscosity of the fluid, flow rate of the injection, heterogeneity of the void, and effect of the injection on the properties of the injected fluid.

2. EXPERIMENTAL METHODOLOGY

2.1. MATERIALS

2.1.1. Cement. The cement used in this study was prepared by mixing API class H cement obtained from Halliburton company with distilled water. The grain size ranged from 10 to 150 microns. The water/cement ratio was 0.38, as stipulated in API specification 10A (API, 2010). The mixing was conducted in accordance with the mixing procedure of API RP 10B-2 (API, 2013), in which water was added first to a two-speed, bottom-drive laboratory blender, after which dry cement was added gradually to the blender while mixing at low speed for approximately 15 s. Then, the speed of the blender was increased to high speed for around 35 s.

2.1.2. Preformed Particle Gel (PPG). LiquiBlockTM 40K, a cross-linked polyacrylic acid/polyacrylamide copolymer particles gel obtained from Emerging Technologies was mixed with 1% NaCl brine solution. The dry gel particles were added to the brine solution during mixing with a magnetic stirrer. The solution was then left overnight to ensure full swelling. Next, the swollen particles were sieved and applied in this study as a semisolid material. The dry particle sizes ranged from 420 to 841 microns (20-40 mesh).

2.1.3. Hydrolyzed Polyacrylamide Polymer (HPAM). A commercially available 20% hydrolyzed polyacrylamide polymer was mixed with distilled water using a magnetic stirrer. The powdered polymer was added slowly to the water while mixing, and the mixing was continued for around 24 hours to obtain a homogenous solution. Three concentrations were used, 0.1% (1000 ppm), 0.3% (3000 ppm), and 0.6% (6000 ppm), with the polymer solutions being solids-free materials.

2.1.4. Epoxy Resin. Epoxy resin was prepared by mixing an epoxy resin with an aromatic hardener at room temperature. The base resin was a diglycidyl ether of bisphenol A (DGEBA) obtained from Miller-Stephenson Chemical Company diluted with cyclohexanedimethanol diglycidyl ether (CHDGE), obtained from the same company. The diluent amount added to the resin was 100% by resin weight. The selection of this amount was based on a previous study conducted by Alkhamis and Imqam (2019). The aromatic hardener was diethyltoluenediamine (DETDA), obtained from Albemarle Corporation. The diluted DGEBA was accurately weighed into a glass beaker with the appropriate amount of DETDA (52% by weight of the diluted resin). The sample was stirred thoroughly at which point the curing agent was completely dissolved and a clear homogeneous mixture was obtained.

2.2. RHEOLOGICAL MEASUREMENTS

For the rheological measurements, three types of instruments were used. A dynamic shear rheometer (DSR) with parallel plates system supplied by Anton Paar measured the viscosity of the epoxy resin and the storage modulus (G’) of the PPG. A rotational viscometer (model 800) supplied by OFI Testing Equipment, Inc. (OFITE), characterized the viscosity behavior of the cement. A rheometer supplied by Brookfield Ametek, model DV3T assessed the viscosity of the polymer solutions.

To measure the viscosity of the epoxy resin, samples ranging from 0.5 to 1.0 ml of epoxy resin were placed on the lower plate of the instrument, and the upper plate was lowered to maintain a gap of 0.5 to 1.0 mm. The readings were taken in both ascending and descending order in a range from 0.1 1/s to 1000 1/s. To measure the storage modulus of the PPG, a sample of the swollen gel underwent in a similar procedure to the one undergone by the epoxy, but in this test, an oscillatory motion was applied at a frequency of 1 Hz to estimate the strength of the PPG.

For the cement’s viscosity measurements, the cement slurry was mixed and preconditioned at room temperature for 20 minutes. Then, the slurry was poured into the viscometer cup. The dial readings were taken in both ascending and descending order, with the highest speed being 300 rpm so not to disturb the slurry. The slurry viscosity readings were recorded as the average of the two dial readings at each speed.

For the HPAM viscosities, the solutions were prepared and samples of 8 ml were poured into the cup of the rheometer; readings were taken in both ascending and descending order. The rotational speeds were in the range of 0.1 to 250 rpm, with a waiting time of two minutes between each speed. The viscosity values presented in this work are the average values of the readings at each speed. The torque percentages were also recorded at each speed, and any torque value lower than 10% was removed as recommended by the rheometer’s manufacturer.

2.3. INJECTIVITY MEASUREMENTS

Prior to any remedial operation, an injectivity test is performed to set the pressures and flow rates at which remedial fluids can be pumped into leakages zones. This test helps in determining the key parameters for the treatment as well as the major limitations of the operation.

In this study, the experimental setup (Figure 1), consisted of a syringe pump, an accumulator, two pressure transducers, and stainless-steel tubes with various inner diameters (i.e., 0.876, 1.753, and 4.572 mm). This setup was prepared to establish the injectivity of several materials that have been employed or have the potential to be used for wellbore integrity applications. The materials included conventional API cement, solids-free polymer solutions, epoxy resin, and semisolid particle gels (PPG).

First, the accumulator was filled with the tested material and the injection began at a low flow rate of 1 ml/min. As it was, increased to 2, 4, and 8 ml/min, the injection pressure and the halfway pressure were recorded by pressure transducers. The fluids were collected from the outlet to be observed visually and tested using the rheological measurements mentioned above. Then, the injectivity of the fluids was calculated using Equation 1.

$$Injectivity = \frac{Injection\ Flow\ Rate}{Injection\ Pressure} \tag{1}$$

where the injection flow rate is expressed in ml/min, the injection pressure is expressed in psi, and the injectivity is expressed in ml/psi*min.

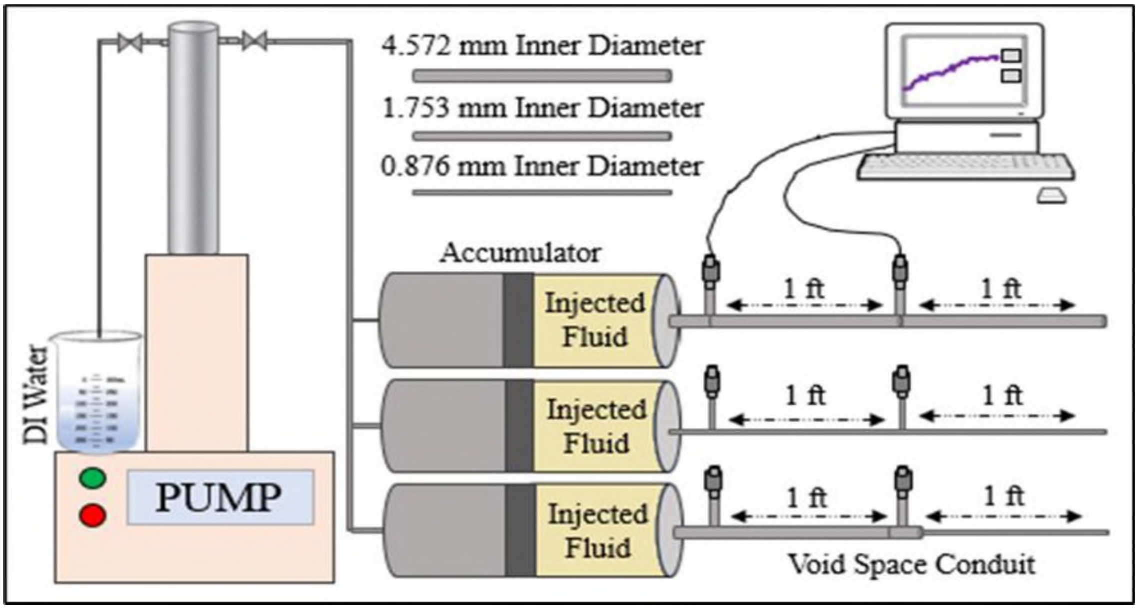

Figure 1. Illustration of the injectivity setup.

3. RESULTS AND ANALYSIS

3.1. RHEOLOGICAL RESULTS

Multiple parameters affect the injectivity of any material, with one of the most significant being its viscosity. In this study, the viscosity of the cement was measured using a rotational viscometer. Figure 2a shows the viscosity behavior of API cement class H. The cement exhibited a behavior similar to that of Bingham plastic model, which requires a yield stress to initiate flow.

On the other hand, the epoxy resin behaved like a Newtonian fluid (Figure 2b), where no stress or only a very small stress was required to initiate flow, and the viscosity was independent of the shear rate. The viscosity of the epoxy resin was found to be around 400 cp.

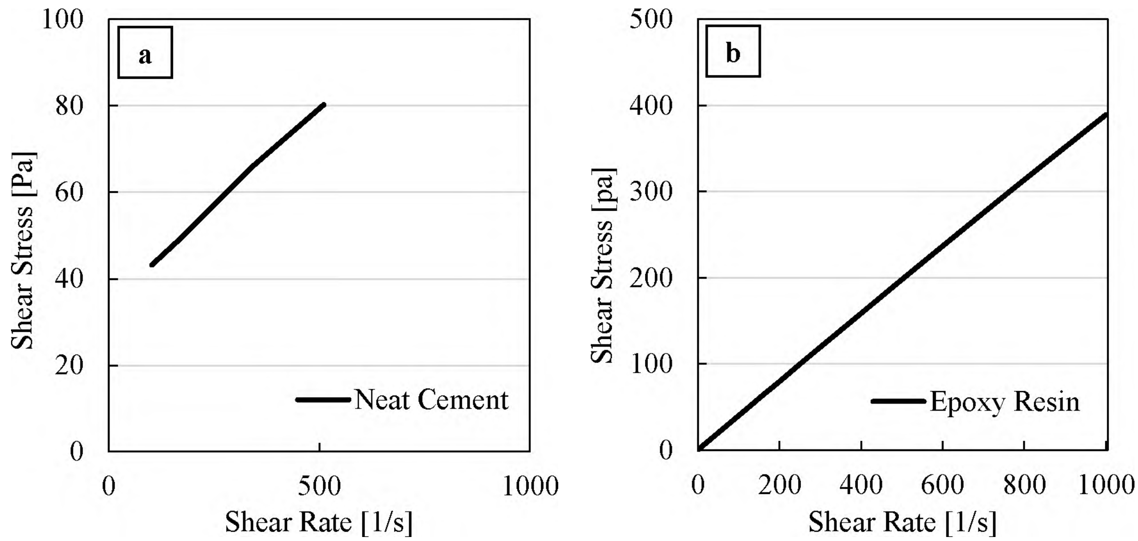

Figure 2. Viscosity results of (a) class H cement and (b) epoxy resin.

For the PPG, since the material is semisolid, the rheology characterization makes more sense in terms of the storage modulus, which represents the strength of the material. The strength of PPG can be controlled by adjusting the salinity of the brine in which the gel particles are swollen (Imqam et al., 2017). However, the swelling capacity will also be affected. In general, the higher the salinity, the higher the strength and the lower the swelling ratio. In this study, the gel particles were swollen in 1wt% NaCl, resulting in a G' of 850 Pa, estimated using the DSR.

For the HPAM solutions tested in this study, a rotational rheometer with a cup was used. HPAM is a solid-free solution that is widely used in enhanced oil recovery applications. For wellbore integrity it can mixed with an initiator to create a 3-D network to plug cement gaps. In this work, three concentrations of HPAM were studied; 0.1% (1000 ppm), represented in this paper as low viscous solution (LV); 0.3% (3000 ppm), represented in this paper as moderately viscous (MV); and 0.6% (6000 ppm), represented in this paper as highly viscous (HV).

Figure 3 illustrates the rheological behavior of the HPAM solutions. The HPAM solutions experienced shear thinning behavior in which the viscosity decreased by increasing the shear rate. Different concentrations were selected to study the effect of the viscosity of the material on its injectivity.

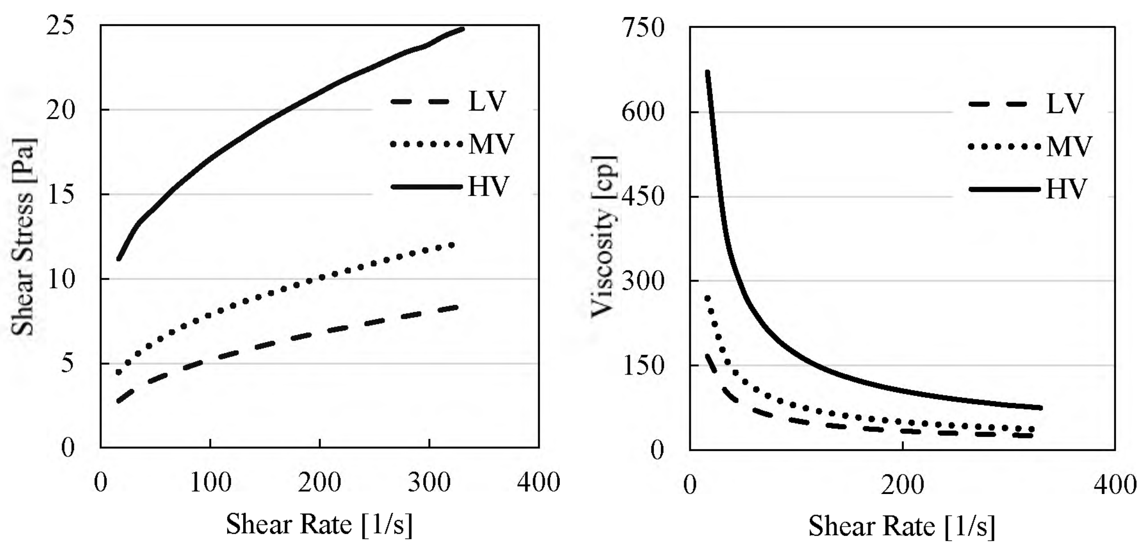

Figure 3. Viscosity results for HPAM solutions.

3.2. SEALANT INJECTIVITY RESULTS

The injectivity of the materials tested was measured using the setup in Figure 1. Several experiments were conducted to better identify and understand the most important factors that affect cement remedial operations and the selection of the proper sealant. These factors include the type of fluid injected, void size into which the remedial fluid is injected, viscosity of the fluid, flow rate of the injection, heterogeneity of the void, and effect of the injection on the properties of the injected fluid.

3.2.1. Effect of the Void Size on Injectivity. Three void sizes were used, consisting of two-foot tubes with inner diameters of 0.8763 mm, 1.753 mm, and 4.572 mm. The fluids' injection pressure and halfway pressure were monitored and recorded. The injectivity of each fluid at various conditions was calculated based on the flow rate used and reaching stable pressure. Figure 4a illustrates the injection pressure of the 0.1% (1000 ppm) polymer solutions (i.e., LV polymer solution), showing that after approximately 10 minutes, the solutions reached stable pressure. The pressure increased as the void size decreased. The pressure was around 0.07 psi when the 4.572 mm void was used and increased to around 2.85 psi when the 0.8763 mm void was used. Both experiments were run at a flow rate of 1 ml/min. This huge increase in pressure reduced the injectivity by approximately 96.5% (from 10 to 0.348 ml/psi*min). There were no changes in the appearance of the solutions before and after the injection, which correlates to the viscosity measurements that will be presented later in this paper.

Figure 4b shows the injection pressure of PPG vs. time. The void size had a more significant impact on the injectivity of the PPG, increasing the injection pressure from around 2 to 80 psi using a flow rate of 1 ml/min for both tests. In addition, permanent deformation of the gel particles was observed at the outlet of the 1.753 mm void but not in the 4.572 mm void.

The permanent deformation of the PPG and the great increase in the injection pressure when the 1.753 mm void was used eliminated the need to use smaller sized void.

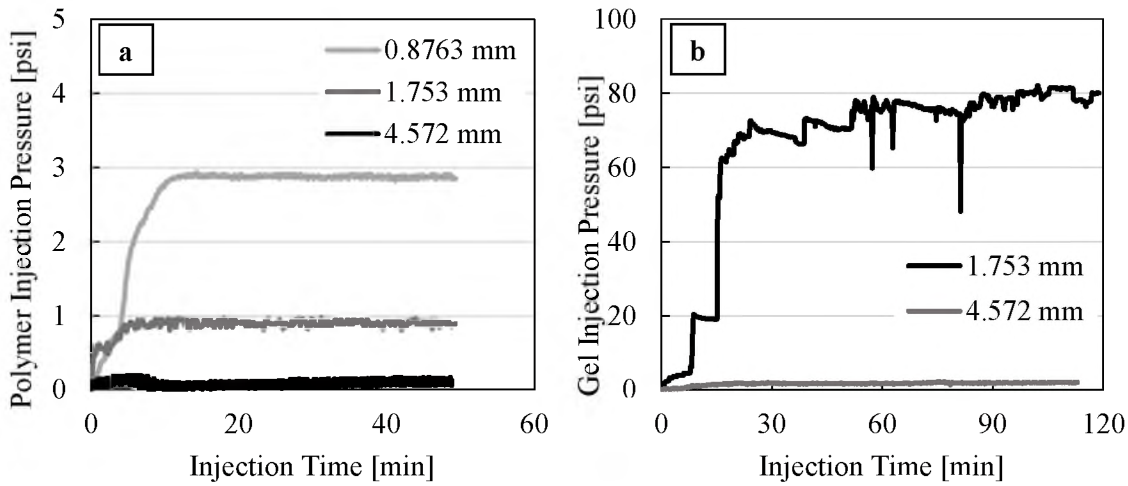

Figure 4. Effect of the void size on the injectivity of (a) HPAM, and (b) PPG.

Conventional Portland cement is the most widely used material for oil and gas wells repairs. However, Portland cement has limitations, and primarily the size of the gaps that the cement can penetrate, as evident in Figure 5a. With a void size of 4.572 mm, the cement injection pressure was slightly higher than that with LV polymer solution and relatively smaller than with the PPG. In this experiment, the cement showed high injectivity and passed through the void easily. However, when the void size was reduced to 1.753 mm, the cement injection pressure increased drastically to more than 500 psi. The associated reduction in the injectivity of the cement was near 99.78%. This effect of the void size on the cement injectivity has led the industry to shift or consider shifting to alternative sealants for wellbore remediation operations. In addition, the industry has sought other solutions, such as altering the methods of injecting the remedial sealants. Figure 5b demonstrates how using the same void size (1.753 mm) but switching the injection mode from running squeeze to hesitation squeeze helped raise the chances of successful cement placement. Lastly, in Figure 5b, the effect of the constant injection pressure can be seen in the far right section of the graph, which shows that the cement was not able to pass through the first foot to the point where the second pressure recorder was located; however, switching the method of injection facilitated the process, as shown in P_2 at the 18th minute. Also, the constant injection pressure supported the continual increase in P_2, which indicate that the cement was flowing inside the void. At the effluent of the void of these experiments, there were two major observations worth mentioning. First, when using the larger void size (4.572 mm), the effluent manifested at first as drops of water, followed by cement slurry, then again as a few drops of water, followed again by cement slurry, indicating that the water might have separated from the cement slurry during the injection. This separation can greatly impact the outcome of the remedial operation. Second, during the 1.753 mm test, there were only a few drops of water, but the cement was able to plug the void. These changes in the cement fluidity can be overcome using additives, such as fluid loss additives. In this study, only neat cement was injected to reduce the complexity of the tests.

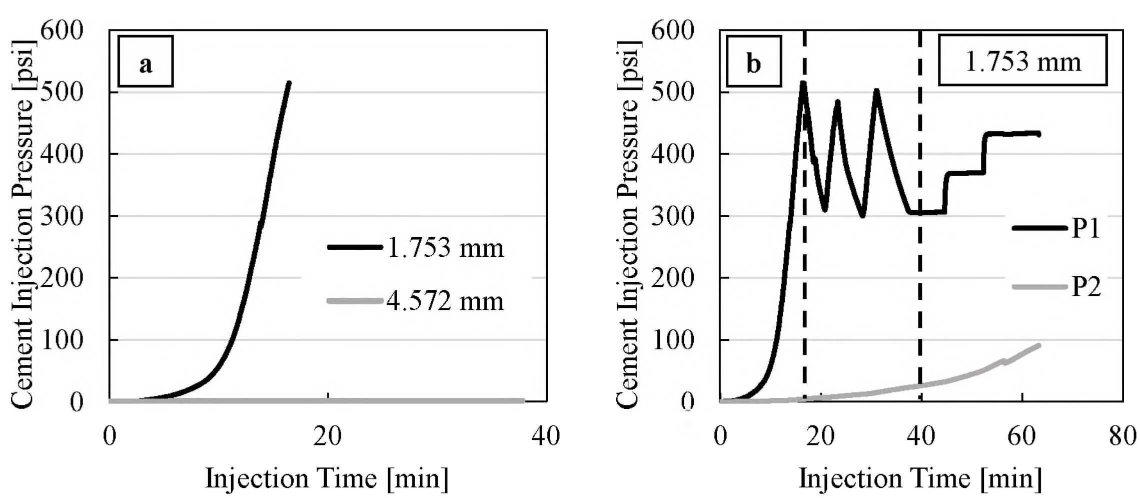

Figure 5. Effect of the void size on the injectivity of cement.

On the other hand, the epoxy showed similar behavior to the HPAM, in terms of injectivity with respect to the void size. Figures 6a shows the low injection pressure required to force the epoxy into the 1.753, and 4.572 mm voids. The injectivities of the epoxy at a flow rate of 1 ml/min were 0.27 and 10.0 ml/psi*min. However, the injectivity reduced to approximately 0.025 ml/psi*min when 0.8763 mm void was used (see Figure 6b), which is due to the high viscosity of the epoxy. This viscosity can be altered using diluents, reactive materials that can reduce the viscosity with minimum effects on mechanical properties, as reported in the literature.

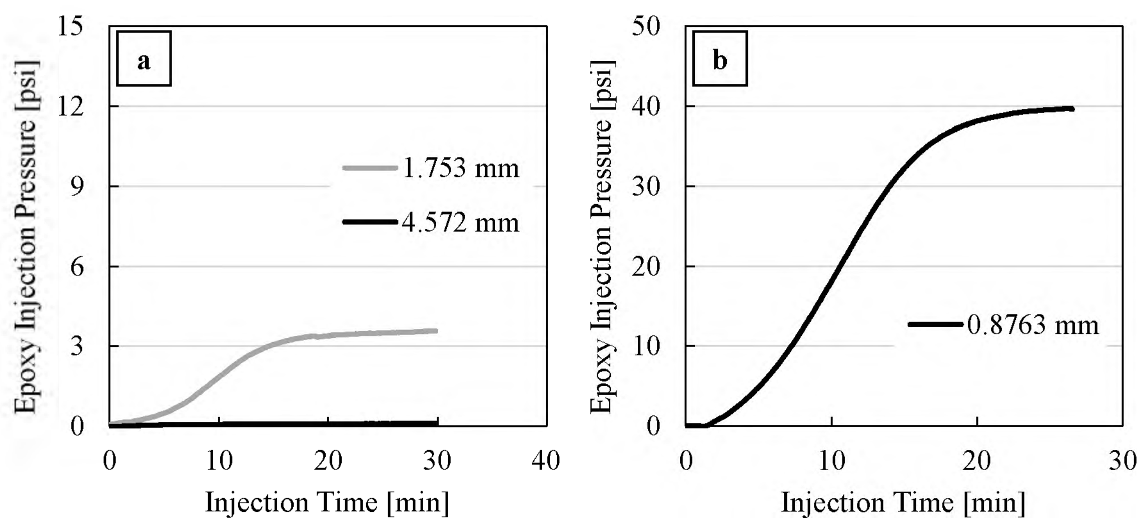

Figure 6. Effect of the void size on the injectivity of epoxy (a) 1.753, and 4.572 mm voids, and (b) 0.8763 mm void.

3.2.2. Effect of the Viscosity of the Fluid on the Injectivity. To understand whether the viscosity of the injection fluid had a major impact on the injectivity of the materials in the remedial jobs, three polymer solutions were injected into the same void size at the same flow rate (2 ml/min). The polymer solutions were HPAM with low viscosity (LV), HPAM with moderate viscosity (MV), and HPAM with high viscosity (HV). HPAM was selected because the rheological results of HPAM showed shear thinning behavior. The three HPAM solutions were injected into the smallest void size used in this study (0.8763 mm). Figure 7a shows the polymer injection pressure. Similar to the previous tests, the injection continued until a stable pressure was reached. In this case, the stabilized pressures for the LV, MV, and HV polymer solutions were around 3.9, 5.3, and 10.72 psi, respectively. These injection pressures were compared to that of water and the viscosity of the injection fluid played a major role in the injectivity of the material (see Figure 7b), which shows the relationship between the injection pressure and the flow rate. Figure 7b was generated using the same polymer solutions but different flow rates (i.e., 1, 2, 4, and 8 ml/min). This relationship allowed for studying the combined effect of the flow rates and the viscosity on the injectivity of the materials. The relationship between the injection pressure and the flow rate for water is linear, unlike the relationship for the polymer solutions.

Results similar to these should be considered in the field when sealants are applied. It is better for a successful sealant placement to employ a Newtonian fluid where the pressure at each flow rate can be predicted effectively. Additionally, it is beneficial that only a low or very small yield stress is required to initiate flow.

It is noteworthy that the injectivity of the Newtonian fluid (water herein) did not change by changing the flow rate. This will be discussed further in the results of the epoxy resin injection.

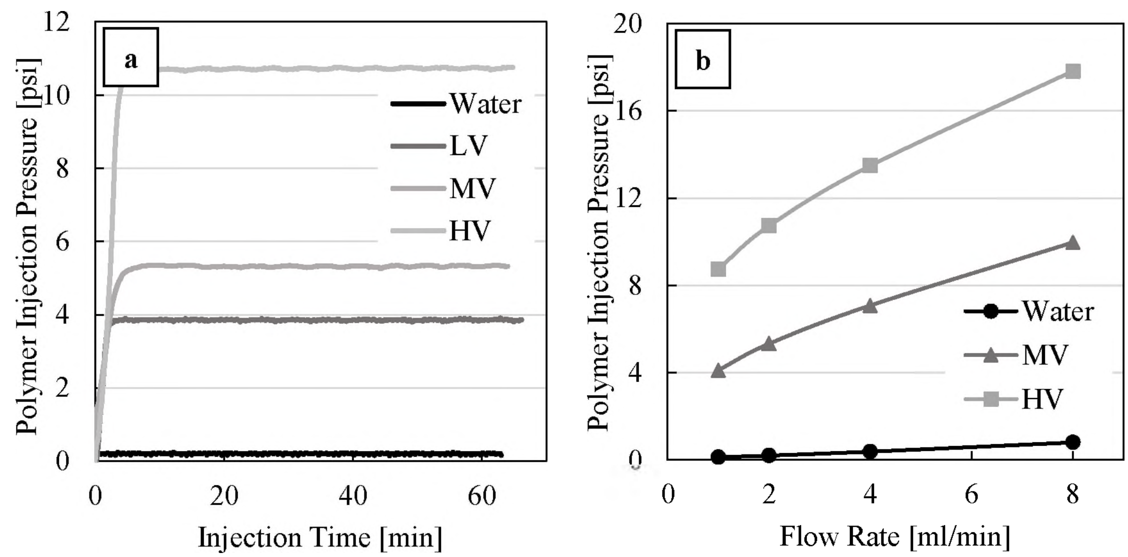

Figure 7. Effect of the viscosity on the injectivity of the polymer solutions.

3.2.3. The Effect of Flow Rate on Injectivity. For each material studied herein, flow rate experiments were conducted using multiple void sizes to study the effect of flow rate on the injectivity of the sealants. First, HPAM solutions were injected into the void. Figure 8a shows the results of injecting the moderate viscosity HPAM solution into a 0.8763 mm void. As can be seen, increasing the flow rate resulted in an increase in the injection pressure. The calculated injectivity associated with these measurements also showed an increase in the injectivity from 0.114 ml/psi*min at a flow rate of 1 ml/min to 0.803 ml/psi*min at a flow rate of 8 ml/min. Similar results were obtained when the low viscosity and high viscosity polymer solutions were injected. The results of changing the void size were also consistent with this behavior. However, the relationship between the stabilized injection pressure and the flow rate were not linear (Figure 8b), the opposite of the results obtained when water was injected.

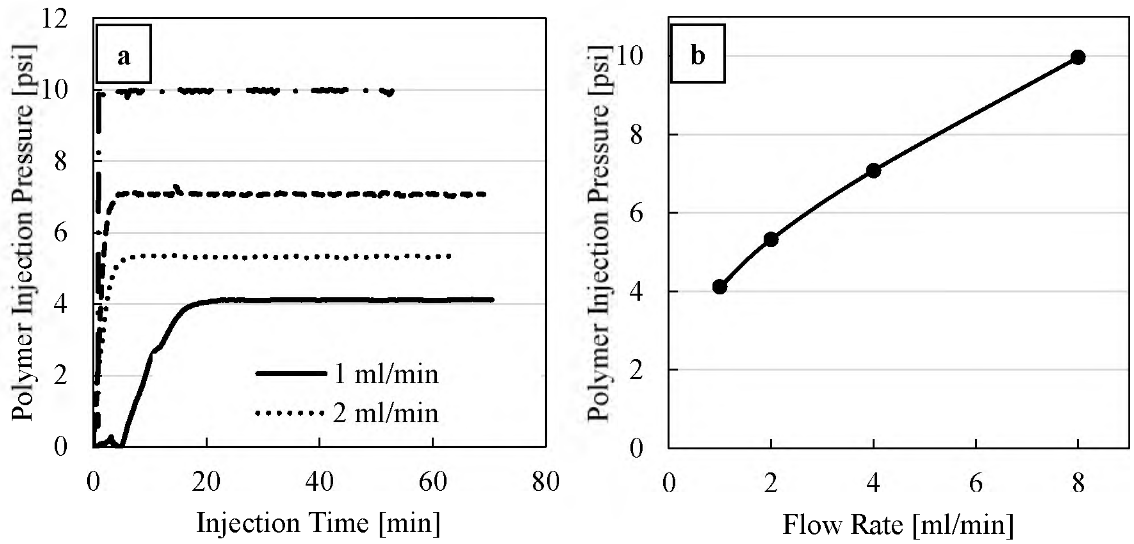

Figure 8. Effect of the injection flow rate on the injectivity of the HPAM solution.

The PPG showed similar results to those of the HPAM solutions, as shown in Figure 9a but with the PPG, it was even more difficult to define a clear relationship between the injection pressure and the flow rates, as shown in Figure 9b. These results further complicate the remedial operation. The behavior of the PPG is to the elasticity of this semisolid material. The gel particles deformed inside the void space, resulting in a high fluctuation in the pressure readings. The results presented here are for a void of 4.572 mm, the largest size used in this study. For the smaller size (1.753 mm), the results were even more complicated, and the gel particles left the void with permanent deformations. This might be solved using semisolid gel particles that can reassociate inside the void, creating an impermeable network capable of permanently plugging the cement features.

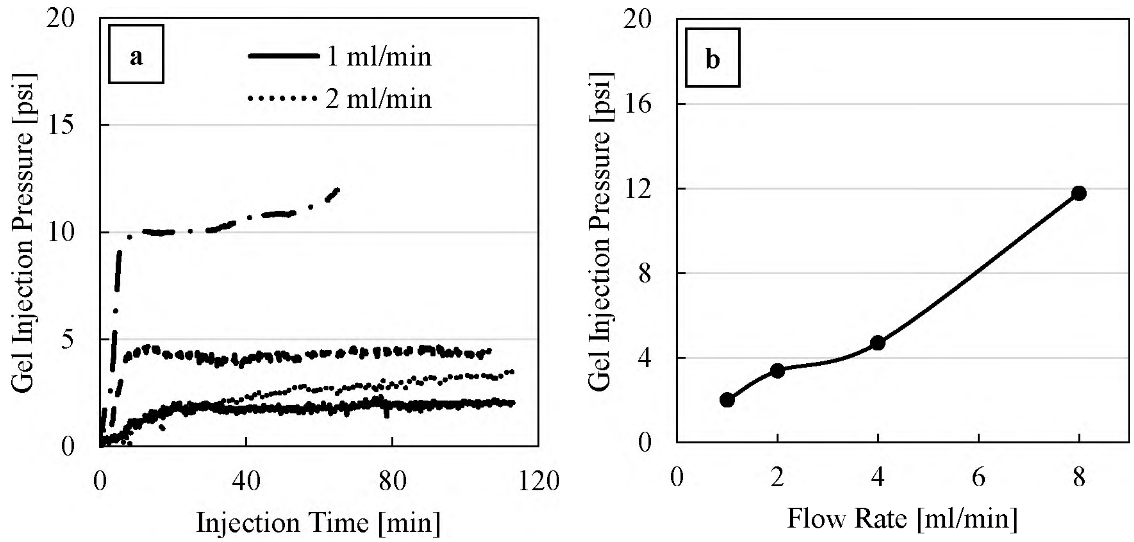

Figure 9. Effect of the injection flow rate on the injectivity of the PPG.

Epoxy, which is one of the most recent technologies in wellbore integrity, behaved differently. When the rheological properties of the epoxy resin were tested, it showed Newtonian behavior, which is the preferred behavior for this application in terms of the yield stress required to initiate movement. Figure 10a shows the results of injecting epoxy resin into a void size of 1.753 mm, where increasing the flow rate of the injection resulted in an increase in the injection pressure. However, in this case the relationship between the injection pressure and the flow rate was linear, as shown in Figure 10b.This is significant and advantageous because in this case, the injectivity was independent, and the injection pressure could be predicted precisely prior to any remedial job, leading to a placement with less risk of fracturing the cement and its surroundings, which would exacerbate the situation.

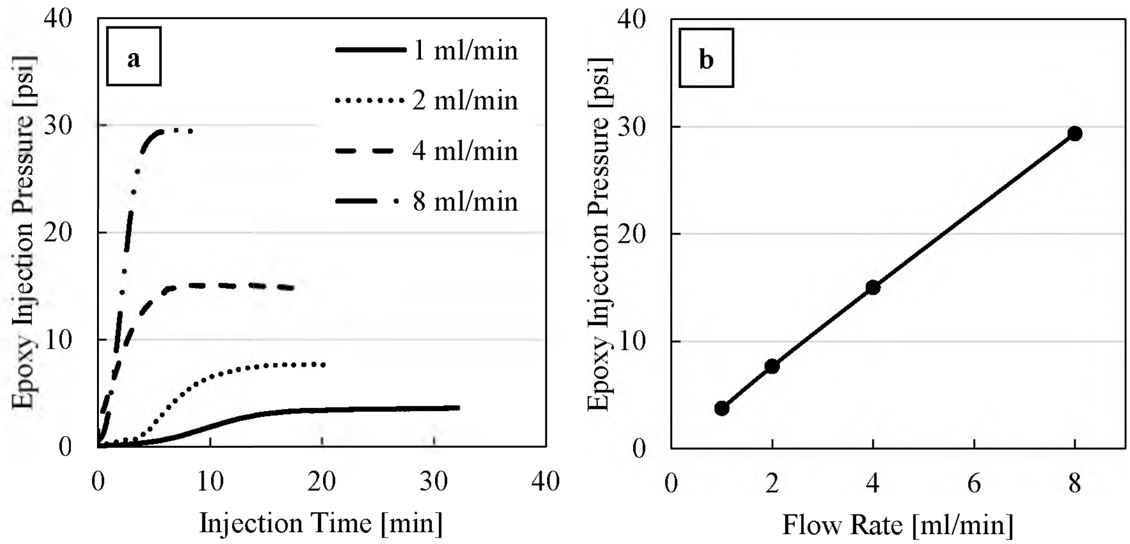

Figure 10. Effect of the injection flow rate on the injectivity of the epoxy resin.

3.2.4. Effect of Heterogeneity on the Injectivity. The effect of the heterogeneity of the voids on the injectivity of the sealants in Figures 11a and b, which show the injection pressure of the PPG and HPAM solutions, respectively. These figures show the pressure reading at various locations in the void. The solid-free material (HPAM) experienced no effect as a result of the heterogeneity, while the pressure reading for PPG rose and decreased and also reached higher values than those obtained using a uniform void of the same size. This suggests that using a solids-free sealant reduces the risk of false readings during the placement of the sealant.

Figure 12a shows that epoxy sealants experiences minimum effect when injected into heterogenous void space. The pressure readings recorded at both the inlet and halfway of the void were close to each other and the material was smoothly flowing in the void. Figure 12b illustrates the effect of increasing the flow rate in a heterogenous void. Again, the epoxy flowed smoothly, and the pressure readings were close. The injectivity was slightly higher than that of the 1.753 mm void but lower than the 4.572 mm void. Additionally, the flow rate had very little effect on the injectivity.

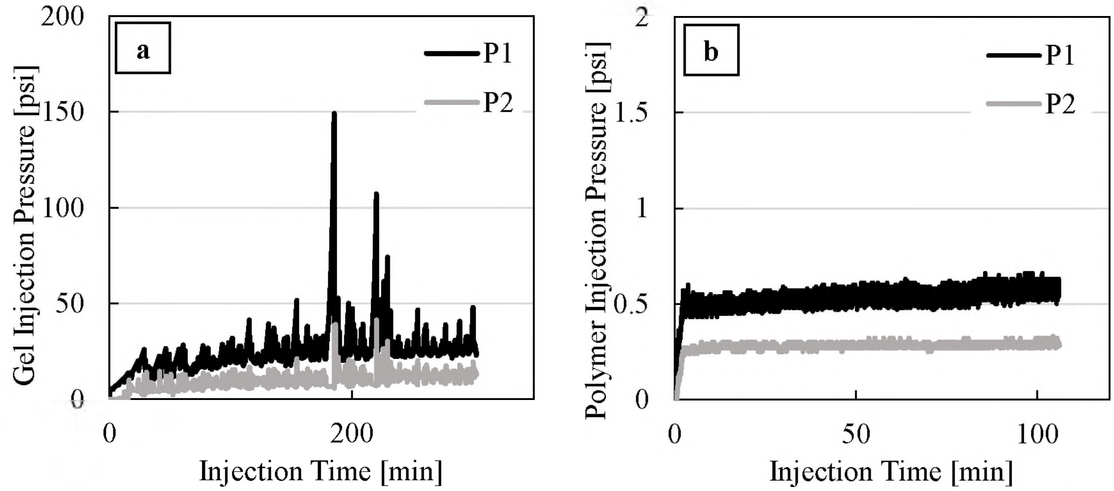

Figure 11. Effect of the heterogeneity on the injectivity of the (a) PPG, and (b) HPAM solutions.

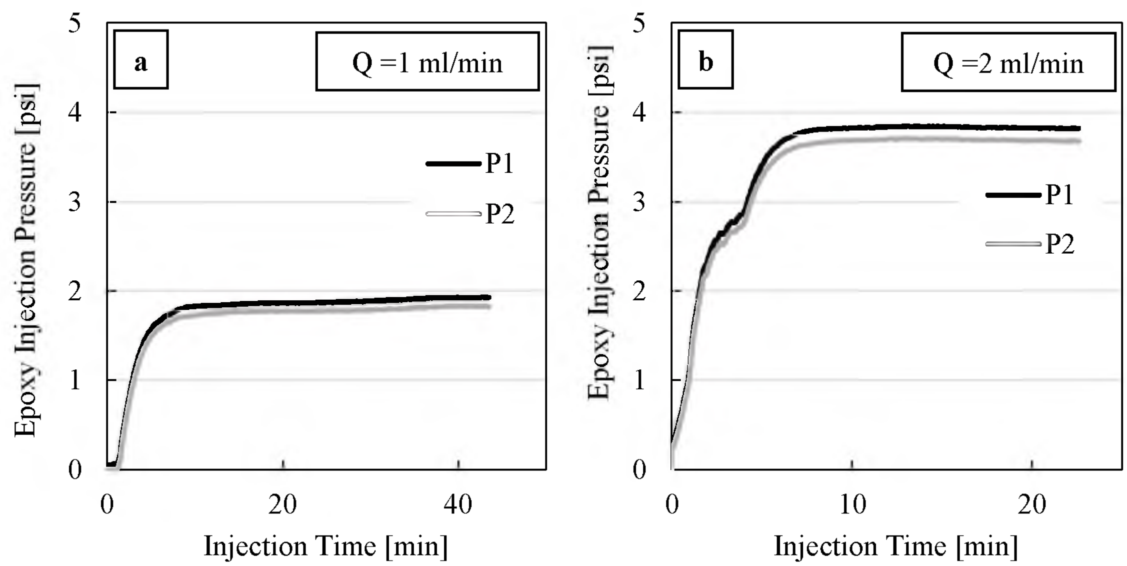

Figure 12. Effect of the heterogeneity on the injectivity of the epoxy (a) 1 ml/min, and (b) 2 ml/min.

Table 1 summarizes the results of the various sealant injectivities obtained at different flow rates, void sizes, and viscosity. Only Newtonian fluids showed the same injectivity when the flow rates varied.

Table 1. Effect of flow rate, void size, heterogeneity, and viscosity on the injectivity.

Flow Rate [ml/min]	Void size, 1/16" (0.8763 mm)					Void size, 1/8" (1.753 mm)				Void size, 1/4" (4.572 mm)				Heterogeneous Voids		
	Water	LV (HPAM)	MV (HPAM)	HV (HPAM)	Epoxy Resin	Solids-free (HPAM)	Epoxy Resin	Semisolid (PPG)	Cement	Solids-free (HPAM)	Epoxy Resin	Semisolid (PPG)	Cement	Solids-free (HPAM)	Epoxy Resin	Semisolid (PPG)
1	7.710	0.348	0.243	0.114	0.025	1.163	0.270	0.0117	0.002	10	10.000	0.4975	0.909	1.786	0.519	0.0354
2	10.005	0.519	0.376	0.187	-	1.852	0.262	0.0231	-	10.526	11.050	0.5917	-	2.899	0.522	0.0514
4	10.509	-	0.565	0.297	-	3.077	0.267	0.0414	-	19.048	11.799	0.8949	-	3.809	0.536	-
8	10.005	-	0.803	0.449	-	4.444	0.272	0.0602	-	30.769	12.780	0.6797	-	5.298	0.543	-

3.3. RHEOLOGY ANALYSIS AFTER SEALANT INJECTION

Earlier, it was stated that the cement had undergone phase separation during and after the injection into the voids, which can affect the efficiency of the cement inside the cement gaps. In addition, we mentioned that a deformation effect occurred when PPG was injected into the 1.753 mm voids, which might negatively affect the remedial operation. Conversely, the HPAM solutions and epoxy resin maintained their rheological behavior after injection. For the HPAM, the results of the rheology were almost identical before and after the injection, including injection at different flow rates. Figure 13a shows the rheology results of the HPAM solutions before the injection, while Figure 13b displays the results directly after the injection. Figure 13 only presents the results of injecting the HPAM solutions into the smallest void (0.8763 mm). Similar results were obtained for the other flow rates (Figures 14a and b) and for the epoxy resin system. These results are good indication and can wrap up the results of this study and a conclusion can be drawn that solids-free sealants such as epoxy resin might be the most effective solution for wellbore integrity applications.

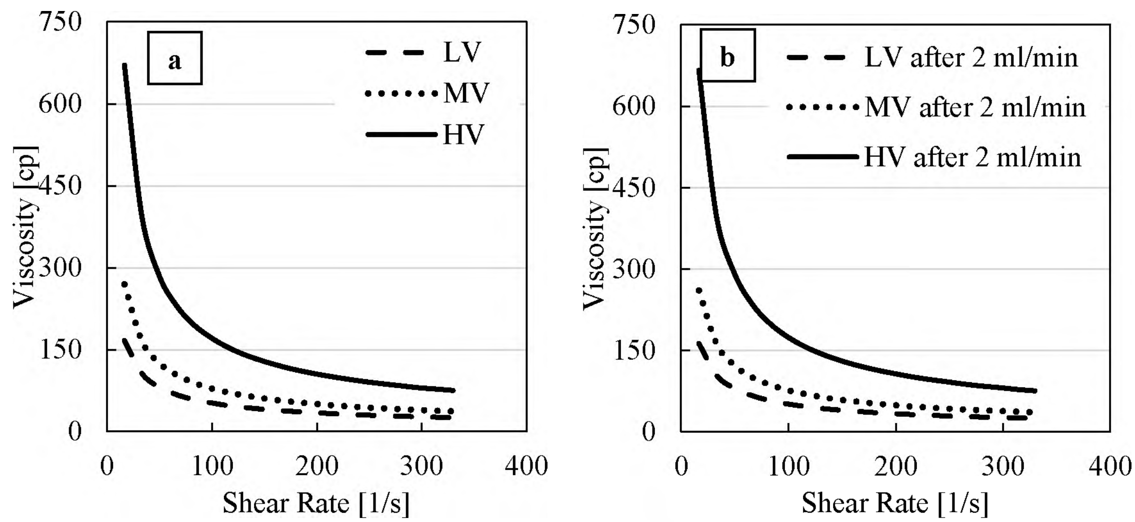

Figure 13. Effect of the injection on the rheology of the HPAM solutions (2 ml/min).

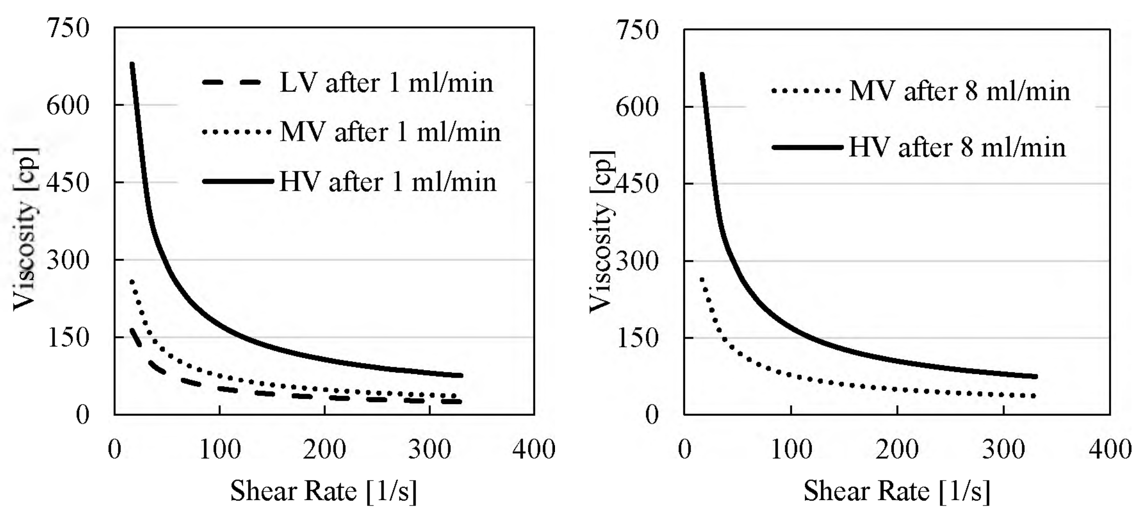

Figure 14. Effect of the injection on the rheology of the HPAM solutions (8 ml/min).

4. CONCLUSIONS

This work presented the injectivity of several materials that can be used in the wellbore remediation of oil and gas wells. The effects of several factors were studied in detail, allowing several conclusions to be drawn. The main conclusions are summarized below:

- The void size, viscosity of the sealants, injection flow rates, and heterogeneity of the voids played major roles in determining the injectivity of the sealants. Having a sealant with Newtonian behavior was beneficial in eliminating the effect of the flow rate.
- Solids-free sealants exhibited the most potential to successfully remediate wellbores in terms of the injectivity of the material.
- Solids-free sealant demonstrated high injectivity and low degradation after injection.
- The epoxy resin showed Newtonian behavior, and the injectivity showed the effect that Newtonian materials have on the injectivity.
- The cement presented a huge limitation in terms of its ability to penetrate small voids.
- The PPG showed good injectivity, but unless this injectivity is correlated with the ability to develop enough strength to hold reservoir fluids in place, this injectivity is not useful.

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III. SOLIDS-FREE EPOXY SEALANT MATERIALS' INJECTIVITY THROUGH CHANNELS FOR REMEDIAL JOB OPERATIONS

ABSTRACT

Remedial cementing job is an operation conducted in the petroleum industry to restore the primary cementing integrity of oil and gas wells. The remedial job is needed when the integrity of the primary cement is compromised, and the wellbore is prone to fluids migration. This work studies the behavior of several sealants that can be used in a remedial job including solids-free material, semi-solids material, and Portland cement. The focus of this study is on testing the rheological behavior, the injectivity, the effect of the size of cement's voids on the injectivity of different sealants, and the strength of the sealants. Laboratory experiments were conducted to evaluate the performance of several sealants in different cement's voids sizes. The results of this work demonstrate that solids-free materials such as epoxy resin has the highest injectivity among the tested sealants even in very small gaps. The epoxy resin develops higher compressive strength than that of the conventional Portland cement. This work points out the importance of selecting the appropriate type of sealant on the effectiveness of the remedial job.

1. INTRODUCTION

During the drilling and completion phases of oil and gas wells, cement is placed in the wellbore as a barrier between the casing and the formation. All casing strings must be cemented to protect and support the casing, and to isolate production zones. The operation of cementing the wellbore casings is called primary cementing. The primary cement must prevent the wellbore fluids from migrating in an annular flow path so as to allow the wells to be utilized without any control problems. The main objective of cementing the annulus is to provide zonal isolation. No fluid communication should happen during the life of the well among the formations and the surface, no matter which fluids these formations are saturated with water, oil, and gas (Thiercelin et al., 1998; Alkhamis and Imqam, 2018; Ahdaya and Imqam, 2019).

The primary cement may fail to deliver full zonal isolation due to several reasons such as insufficient mud removal before the cementing, casing expansion, and contraction. These conditions may cause micro annuli either between the cement and the casing or between the cement and the formation. Other failures such as channels may occur because of high fluid losses, cement free fluids, and inadequate hydrostatic pressure. In addition, high-pressure tests and temperature variations across the cement may cause cracks in the cement sheath. Also, if the cement is placed in zones where corrosive fluids are presented, chemical degradation could compromise the cement integrity. If any of these failures occurred during the life of the well, remedial job must be performed to restore the well integrity. Failing to restore the cement integrity may lead to unwanted severe consequences to the environment, the equipment, and personnel. The well integrity is defined as “application of technical, operational, and organizational solutions, to reduce risk of uncontrolled release of formation fluids throughout the life cycle of a well” (Norsok, 2013). For this, the integrity of the cement must be maintained through the life of the well and after abandonment.

Remedial cementing job, also known as secondary cementing, is an operation conducted to restore the primary cementing integrity of a well. This operation requires as much attention as primary cementing if not more. Many considerations must be taken into account to guarantee success. All parameters related to the remedial job must be analyzed carefully, starting with estimating the significance of the problem, evaluating the associated risk factors, conducting injectivity test, selecting the proper sealant, picking out the placement technique, and assessing economic costs. Generally, there are two main pumping techniques used in remedial squeeze operations, (1) running squeeze, in which the zone of interest is isolated and the cement is squeezed continuously into the zone, (2) hesitation squeeze, where cement slurry is pumped intermittently with short shutdown periods (Goodwin, 1984; London et al., 2013).

For remedial operations, squeeze cementing is usually the method of repair (Shryock and Slagle 1968). Squeeze cementing is the process of forcing a cement slurry into a hole in the casing and the voids behind the casing. A properly designed cement slurry will cure inside the voids without fluid losses to the matrix of the formation. However, this method may require multiple squeezes to achieve shut-off (Sanabria et al., 2016). This method is also limited by the leakage size (Jones et al., 2014) as the cement contains solid particles that may starts bridging in narrow clearances (Davis, 2017). Even micro cement can be limited in penetrating gaps of less than 300 microns in width (Wasnik et al., 2005). Another limitation related to micro cement is the cements' thickening time that get affected by contamination (Dahlem et al., 2017). Generally, squeezing cement require casing perforations. One of the alternative techniques to squeeze cementing is the use of cross-linked polymers and polymer resins, which can easily penetrate small gaps.

A combination of polymer and cross-linker can be optimized on the surface to transform from liquid form to semi-solids at downhole temperature. Although, polymer gel can penetrate micro pores and channels, the 3D network structure of the gel breakdown at high temperatures and lose its ability to trap fluids such as water in water shut off applications. The other limitation of cross-linked polymers is their lack of mechanical strength (Wasnik et al., 2005). In addition, to the weak bonding between these polymers and their surroundings (Abdulfarraj and Imqam, 2019).

Polymer resin systems are mixtures of base resin and curing agents. Polymer resin systems can be defined as "free flowing polymer solutions that can be irreversibly set to hard, rigid solids." (Morris et al., 2012). Many researchers mentioned the superior properties of polymer resin systems as sealant such as (Alsaihati et al., 2017), who mentioned their good and tunable rheological behavior. Todd et al., 2018 discussed how solids-free material could penetrate small gaps. The good wetting and adhesive properties of these sealants for mineral surfaces were studied by (Brooks et al., 1974 and Shaughnessy et al., 1978). The flexibility in density which is good for areas of narrow fracture gradient was mentioned by (Sanabria et al., 2016), and the resistance to contamination was studied by (Perez et al., 2017). In addition, to these liquid properties, solid cured sealant provides high mechanical strength (Ali et al., 2016 and Elyas et al., 2018), resists significant strain (Khanna et al., 2018), develops good bonding properties (Genedy et al., 2017). In addition, the polymerization reaction of polymer resin systems forms no by-product during (Muecke, 1974). Alkhamis et al., 2019 evaluated in details an epoxy resin system and the results were promising. For these reasons, this type of sealant is being used for cement remedial jobs.

This work, which is an extension of (Alkhamis et al., 2019), presents the results of testing several sealants including cement, semi-solids, and solids-free polymer to point out some factors related to remedial jobs. The study includes the rheological behavior, the injectivity, the effect of the size of cement's voids on the injectivity of the sealants, and the strength of the sealants. The findings obtained from this work can be utilized in optimizing the cement remedial operations.

2. EXPERIMENTAL DESCRIPTION

2.1. EXPERIMENTAL MATERIALS

2.1.1. API Class-H Cement. The cement systems used in this study were prepared using American Petroleum Institute (API) Class-H cement and distilled water. Using gas Pycnometer, the specific gravity of the cement was measured to be 3.18. The chemical composition of Class-H cement was obtained using X-ray fluorescence spectroscopy (XRF). Table 1 lists the chemical composition of class-H cement.

Table 1. The chemical composition of class-H cement.

Comp.	CaO	SiO_2	Fe_2O_3	Al_2O_3	SO_3	MgO	K_2O	SrO	TiO_2	Other
Wt %	65.72	20.36	6.19	3.17	2.26	1.32	0.43	0.21	0.16	0.18

2.1.2. Epoxy Resin. A mixture of Bisphenol A diglycidyl ether resin (BADGE), which is an undiluted difunctional resin and cyclohexane dimethanol diglycidyl ether

(CHDGE), which is a reactive diluent was used as the base resin material in this study. The base resin was cross-linked (cured) by an aromatic curing agent known as diethyltoluenediamine (DETDA). Figure 1 shows the chemical structure of the epoxy resin components.

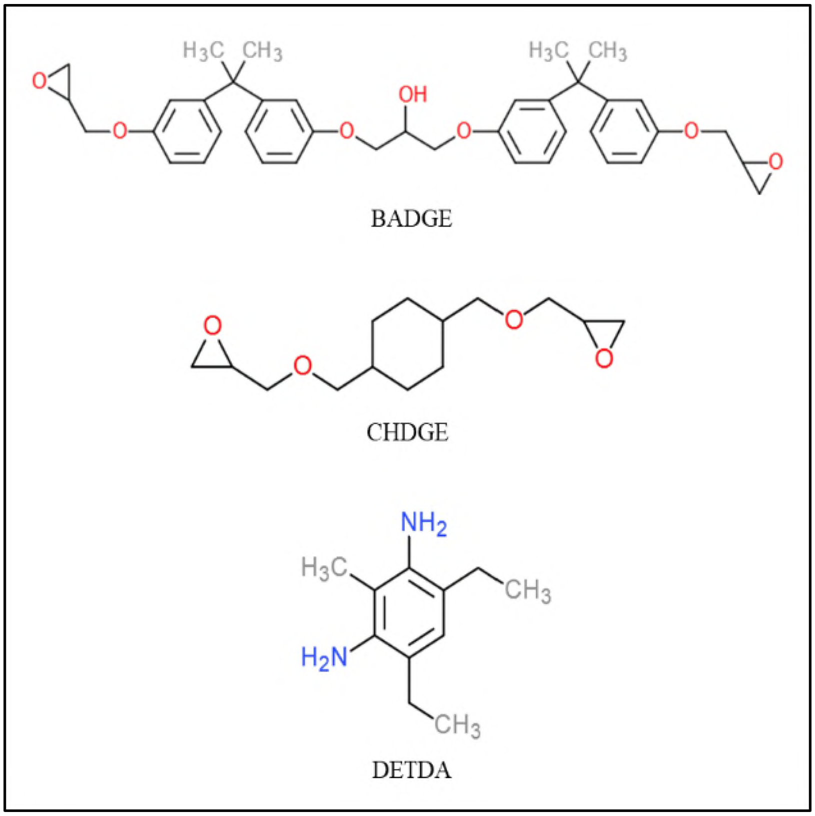
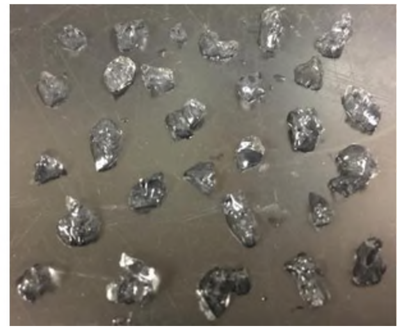


Figure 1. The chemical structure of the epoxy resin components.

2.1.3. Preformed Particle Gel. A commercial superabsorbent polymer was used as semi-solids material in this study. The preformed particle gel (PPG) is a cross-linked polyacrylic acid/polyacrylamide copolymer. The dry particles size are around 400-800 microns as shown in Figure 2. The PPG samples were swollen in a brine solution, which consists of distilled water and 1.0% sodium chloride (NaCL).

a) Dry PPG



b) Swollen PPG

Figure 2. The PPG in its dry condition and after swollen (Imqam et al., 2017).

2.1.4. Hydrolyzed Polyacrylamide Polymer. The hydrolyzed polyacrylamide polymer (HPAM) used in this work was 20% hydrolyzed. It is a commercially available polymer. It was provided as a white granular powder. The HPAM was mixed with distilled water and used in the injectivity experiments to mimic solids-free material.

2.1.5. Cement Paste Preparation. All API cement slurries were mixed following API RP 10B-2 (2013) procedure at room temperature in a two-speed bottom-drive laboratory blender. Dry cement particles were added to the blender at a uniform rate while mixing at low speed for around 15 seconds. Then, the blender was covered while the mixing continued for 35 seconds at high speed. All cement systems had a water/cement ratio (WCR) of 0.38 in accordance to API specification 10A (API, 2010).

2.1.6. Epoxy Resin Preparation. To prepare the epoxy resin mixture, a specific amount of the diluted resin were weighted and mixed at room temperature by hand and/or using a magnetic stirrer until a homogenous fluid was obtained. Then, a calculated amount of curing agent was added to the blend and mixed at low shear rate until the mixture was clear and homogenous. For the high temperature experiments, the mixture was heated until the desired temperature while stirring using heated magnetic stirrer.

2.1.7. Preformed Particle Gel Preparation. To prepare the preformed particle gel (PPG) system, the dry particles of PPG were added to 1% brine solution and left for 48 hours to ensure that the particles are fully swollen. Then, the particles were placed in a sieve to separate the particles from the excessive brine solution.

2.1.8. Hydrolyzed Polyacrylamide Preparation. To prepare the hydrolyzed polyacrylamide polymer (HPAM), a specific amount of the polymer was mixed with a specific volume of distilled water at room temperature using a magnetic stirrer at low shear rate for 24 hours.

3. EXPERIMENTAL METHODOLOGY

This part of the study provides the description and procedure of each experiment conducted in this work. The experiments include the sealants' rheological behavior measurements, the isothermal curing measurements, the injectivity measurements, and the strength measurement.

3.1. RHEOLOGICAL MEASUREMENTS

An advanced Anton Paar Rheometer, which is a dynamic shear Rheometer (DSR) with parallel plates system, was used to measure the viscosity of the HPAM and the epoxy resin systems. Samples of 0.5 to 1.0 ml of the materials were placed on the lower plate of the instrument and the upper plate was lowered to a gap of 0.5 to 1.0 mm. The reading were taken in both ascending and descending order in a range of 0.1 1/s to 1000 1/s. The Rheometer was also used to measure the storage moduli (G') of the PPG, which represent the strength of the material. G' was measured at a frequency of 1 Hz.

The viscosity of the cement slurries was measured using an Ofite viscometer (model 800). For these measurements, the slurries were preconditioned before obtaining the rheological readings at room temperature and atmospheric pressure. The dial readings were taken in ascending order, and then in descending order with highest speed of 300 rpm. Higher speeds were not used to avoid disturbing the cement slurries. The ratio of the two readings at each speed was used to help qualify some of the slurry's properties (API RP 10B-2 2013). The slurries rheological measurements were recorded as the average of the two dial readings at each speed.

3.2. ISOTHERMAL CURING MEASUREMENTS

These measurements are executed to estimate the gelling time of the epoxy resin to define its workability. This information is essential to be known to protect the downhole equipment and to ensure a safe and successful placement of the sealant inside the cement voids. For these measurements, sinusoidal oscillatory tests using the dynamic shear Rheometer (DSR) were performed at an angular frequency of 10 rad/s and the complex viscosity increase with time was monitored while the preheated epoxy resin sample was curing at high temperature. In these tests, disposal parallel plates of 25 mm in diameter were used as the tests were run until the epoxy resin reached a complex viscosity of 9,000,000 centipoise. The plates were discarded after each experiment.

3.3. INJECTIVITY MEASUREMENTS

The injectivity test is a well-known test in the oil and gas industry. This test is performed to establish the rate and pressure at which fluids can be pumped into the treatment target without fracturing the formation. This test is conducted in the field prior to any remedial job to help determine the key parameters of the treatment and the operating limitations. In this work, an experimental setup consists of a syringe pump, an accumulator, two pressure transducers, and stainless-steel tubes with different inner diameters were used as shown in Figure 3. First, the accumulator was filled with the tested sealant, the injection started at different flow rates (1, 2, 4, and 8 ml/min), the injection pressure and the halfway pressure were monitored and recorded by the pressure transducers. The effect of flow rates on the injectivity of each type of the sealants, the effect of the size of the voids on the injectivity of the sealants, and the effect of the heterogeneity of the voids on the sealants were studied along with some effects of the size of the voids on the properties of the tested sealants.

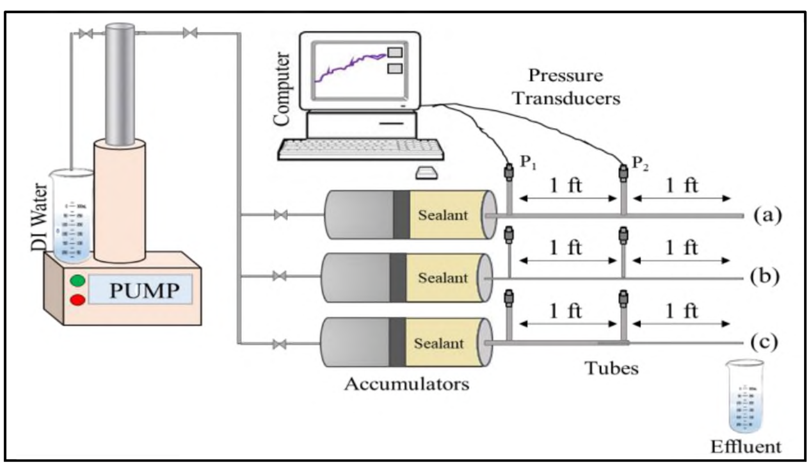


Figure 3. The Injectivity setup: (a) represent 4.572 mm inner diameter tube, (b) represent 1.753 mm inner diameter tube, and (c) represent a combination of both tubes.

3.4. COMPRESSIVE STRENGTH MEASUREMENTS

For the compressive strength measurements of the epoxy resin, the epoxy resin was preheated to 80°C prior to pouring it into 2” × 2” × 2” cubic molds. The molds then were placed in an oven at 80°C for 24 hours. Then, the cured cubes were carefully removed from the molds. The specimen height and width were measured using a caliper, and the minimum surface area was calculated. A hydraulic press was used to measure the force required to crush the samples. The compressive strength was reported in [psi]. For the compressive strength measurements of the cement, the same procedure was followed except for the curing condition as the cement was cured in a water bath at room temperature.

4. RESULTS

The results of each experiment are presented and analyzed in this section according to their importance in the application of the sealants in the remedial operation.

4.1. RHEOLOGICAL AND ISOTHERMAL CURING RESULTS

First, the viscosity of the solids-free material (diluted epoxy resin) was measured using the Rheometer. Figure 4a presents the shear stress vs shear rate chart. The diluted epoxy resin showed Newtonian rheological behavior with no or very low yield stress. The viscosity was found to be around 400 cp at room temperature with no effect of changing the shear rate on the viscosity. The amount of the diluent was around 50% by weight of the total mixture. Different concentrations of diluent were also studied and their results are presented in a different study that focuses on the epoxy resin as a sealant and the effect of the diluent on the viscosity of the resin, the results can be found in (Alkhamis et al., 2019). Then, using the Ofite viscometer the rheological behavior of the neat class-H cement slurry was obtained, and the cement behaved like Bingham plastic model. This behavior as shown in Figure 4b requires some yield stress, which is the minimum stress required to initiate flow. In addition, the storage moduli (G') of the PPG, which represent the strength of the material was found to be 850 pa.

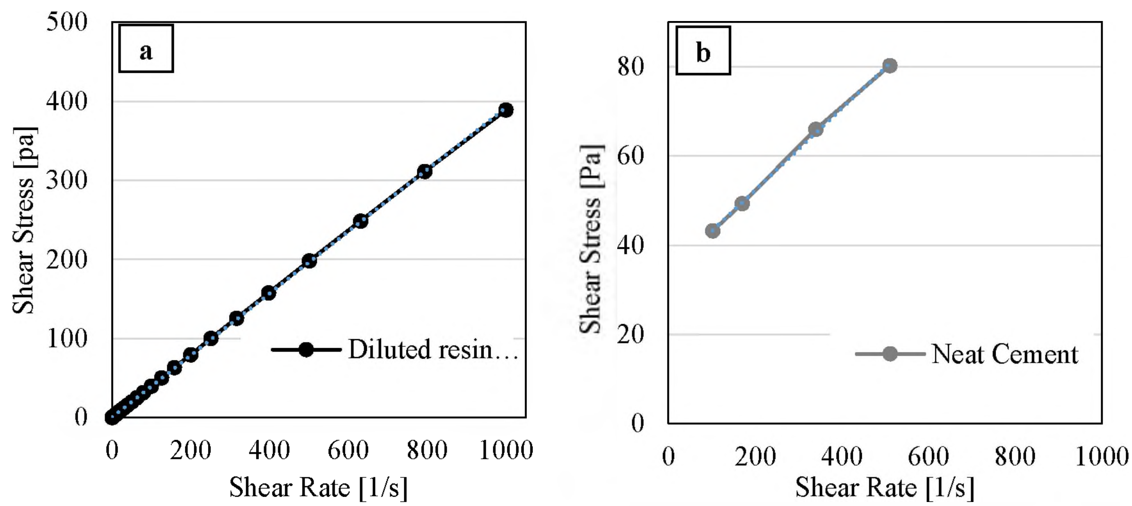


Figure 4. The viscosity results for the diluted resin and the cement.

Since the solids-free material studied here was epoxy resin, which require elevated temperature to be activated, the viscosity of the epoxy at higher temperatures was evaluated. The temperature presented here is 80 ˚C different temperatures are presented in (Alkhamis et al., 2019). Figure 5a shows the effect of the higher temperature on the viscosity of the epoxy resin. The viscosity was found to be around 23 cp and the behavior is Newtonian like. Figure 5b shows the complex viscosity vs time, which represent the isothermal curing result of the epoxy resin at 80 °C. The epoxy resin's complex viscosity was increasing slightly for around 6 hours from 23 cp to around 400 cp, which could be where the gelling time started. Then, the liquid material started to transform to solid after around 8 hours. After 10 hours, there was a rapid increase in the complex viscosity reaching around 24,000 cp. When the system cured for around 14 hours, the complex viscosity was around 9,000,000 cp. After obtaining this result, HPAM with similar viscosity (around 23 cp) was selected to be used in the injectivity measurements. HPAM exhibited shear-thinning behavior but it was used in this study anyway because the results that will be obtained using HPAM will underestimate the injectivity of solids-free sealant especially at low flow rates. It was essential to use different material than epoxy resin in the injectivity measurements because this type of experiments require high amount of sealant and because the epoxy require the addition of heat source to the setup used.

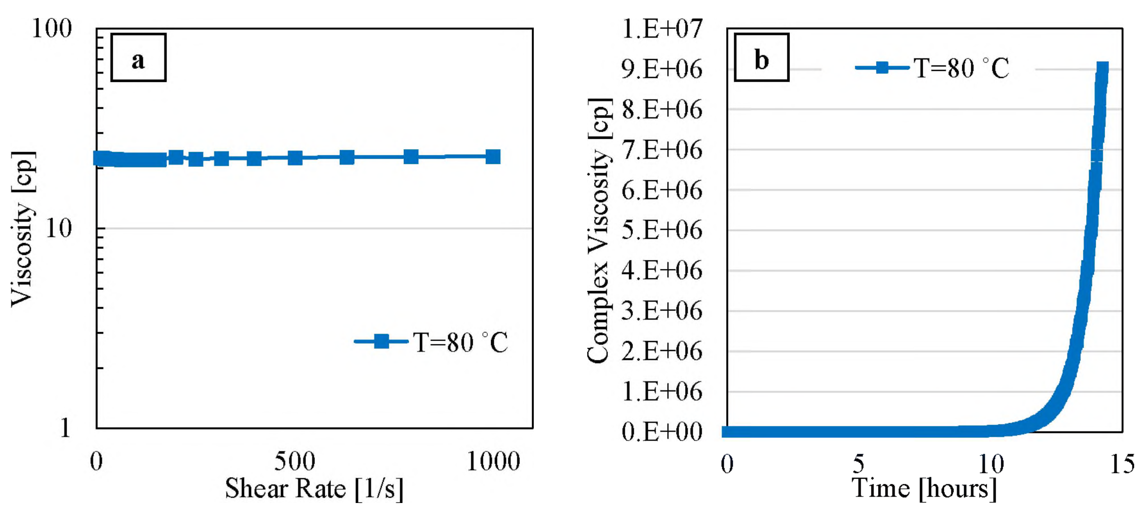


Figure 5. The effect of temperature on the viscosity of the epoxy resin and the curing process.

4.2. INJECTIVITY RESULTS

Before any remedial job, injectivity test may be conducted by injecting a solids-free material through the annulus of the wellbore from the surface or through perforations in the casing to determine the injectivity factor. This practice's objective is to increase the success rate of the remedial job (Alsaihati et al., 2017). Solids-free systems are needed in the applications where the injectivity is very low and the injectivity factor is very high. For example, in an application where the injectivity factor is between 2000 and 4000 psi*min/bbl, micro fine cement can be used while in an application where the injectivity factor is greater than 6000 psi*min/bbl, cement is not a good choice (Cowan, 2007). The injectivity factor is inversely proportional to the injectivity.

In this study, the injectivity of a solids-free material (HPAM, which has similar viscosity as the epoxy resin), semi-solids material (PPG), and solids material (conventional Portland cement) were obtained. Using the setup shown in Figure 3, different sealants were injected at four flow rates (1, 2, 4, and 8 ml/min) into the designed channel models a, b, and c. The injectivity of the sealants at the different flow rates were calculated using the following equation:

$$Injectivity = \frac{Injection\ flow\ rate}{Injection\ pressure} \tag{1}$$

where, the injection flow rate is expressed in [ml/min]; the injection pressure is expressed in [psi]; and thus, the injectivity is expressed in [ml/min*psi].

4.2.1. Solids-Free Materials Injectivity Results. Figure 6 illustrate the injection pressure results for the solids-free material in void size of 1.753 mm and 4.572 mm inner diameters. The prepared solids-free solution was poured in the accumulator and the injection started using the syringe pump at low flow rate (1 ml/min). Then, the flow rate was increased from 1 to 2 to 4 to 8 ml/min. The injection continued until stable pressure was achieved after each injection rate. As shown in Figure 6a the injection of the solids-free material at 1 ml/min stopped after injecting around 60 ml of the sealant and the pressure monitoring showed stable pressure of around 0.86 psi. This pressure and flow rate were used in the injectivity equation and the injectivity was around 1.163 ml/min*psi. The same protocol was followed but with increasing the flow rate from 1 to 2 to 4 to 8 ml/min, respectively. Increasing the flow rate from 1 to 8 ml/min increased the stable pressure to around 1.78 psi, which is a low pressure considering the size of the void and the high flow rate. This result indicate that the solids-free materials have high injectivity even in very small gaps. The injectivity increased by increasing the flow rate. Similarly, Figure 6b shows the stable injection pressures of the four injections but into the larger 4.752 mm. The stable injection pressures for the flow rates were lower than 0.4 psi. The solids-free material was able to penetrate both tubes very easy and with no deformation.

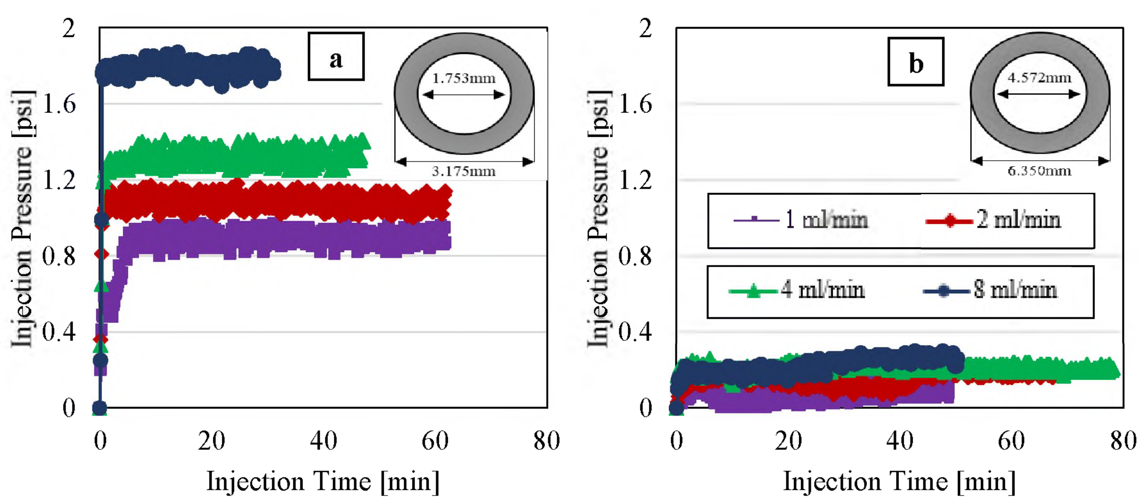


Figure 6. The injection pressure of solids-free sealant.

4.2.2. Semi-Solids Materials Injectivity Results. Figure 7 illustrate the injection pressure results for the semi-solids material into channel size of 1.753 mm and 4.572 mm inner diameters. To measure the injectivity of semi-solids material, the prepared PPG particles were packed in the accumulator and the injection started using the syringe pump at low flow rate (1 ml/min). Then, the flow rate was increased from 1 to 2 to 4 to 8 ml/min. The injection stopped when the injection pressure reached stable pressure.

The injectivity of the semi-solids material at 1 ml/min in the small tube was around 0.0117 ml/min*psi, which is a lower injectivity when compared to the solids-free material at the same flow rate and void size. The same protocol was followed in injecting the semi-solids material but with increasing the flow rate from 1 to 2 to 4 to 8 ml/min, respectively. Figure 7a shows the stable injection pressures of the four injections. Increasing the flow rate from 1 ml/min to 8 ml/min increased the stable pressure by around 62%. The injection pressure of the semi-solids material at a high flow rate (8 ml/min) in the small tube was around 138 psi, which is a high pressure when compared to the solids-free material. This result indicate that the semi-solids materials have good injectivity, but the properties of the PPG changed as the particles clearly deformed. The deformation of the particles can be easily visualized.

In a similar way, the PPG was injected in model (a) and the stable injection pressures for the four flow rates are shown in Figure 7b. The semi-solids material was able to penetrate the larger tube with pressures lower than 12 psi. Interestingly, the semi-solids material in this case did not deform when collected from the outlet of the setup. This indicates the importance and the effect of the size pf the voids on the properties of semi-solids materials when used as possible sealants for wellbore integrity applications.

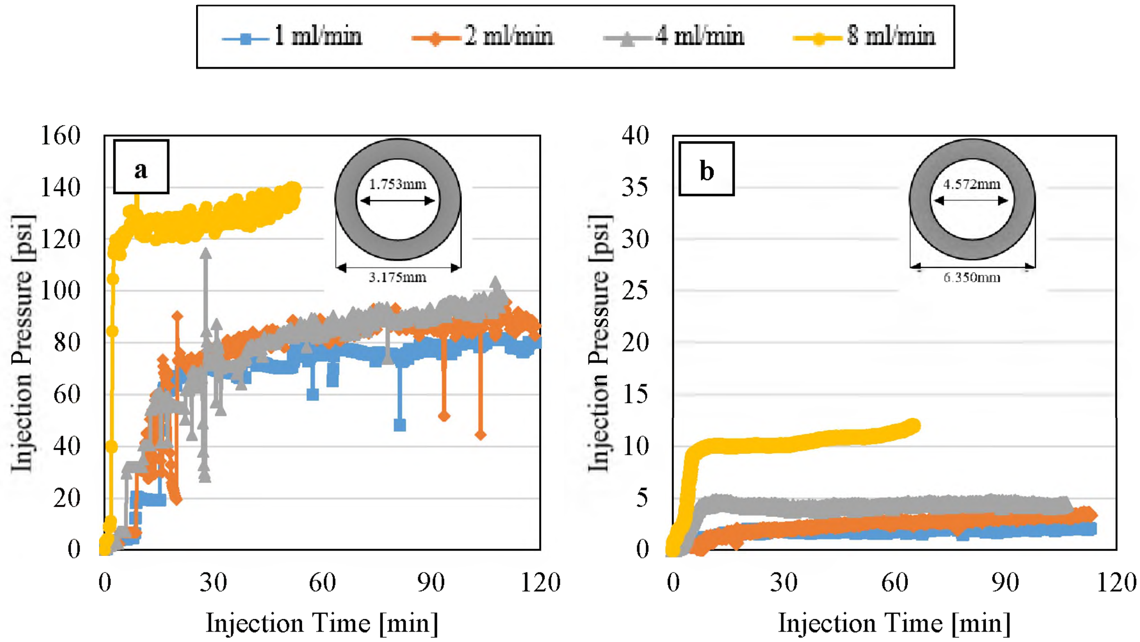


Figure 7. The injection pressure of semi-solids sealant.

4.2.3. Solids Materials Injectivity Results. For the cement injectivity measurements, the cement systems were mixed and preconditioned in accordance to API specifications and then were poured in the accumulator. Figure 8 illustrate the injection results of the cement.

Figure 8a shows the injection pressure of the cement slurry at constant flow rate of 1 ml/min. The stable pressure was around 1.1 psi, which is higher than that of the solids-free material but relatively lower than the semi-solids material. The injectivity was calculated to be 0.909 ml/min*psi. The cement penetrated the large channel (4.752 mm) easily and showed high injectivity. However, when the smaller channel was used the injectivity reduced by 99.78 % and the injection pressure increased to around 514 psi at the same flow rate (1 ml/min) as shown in Figure 8b. This experiment was not stopped after this reading as the injection continued but after switching to hesitation squeeze mothed.

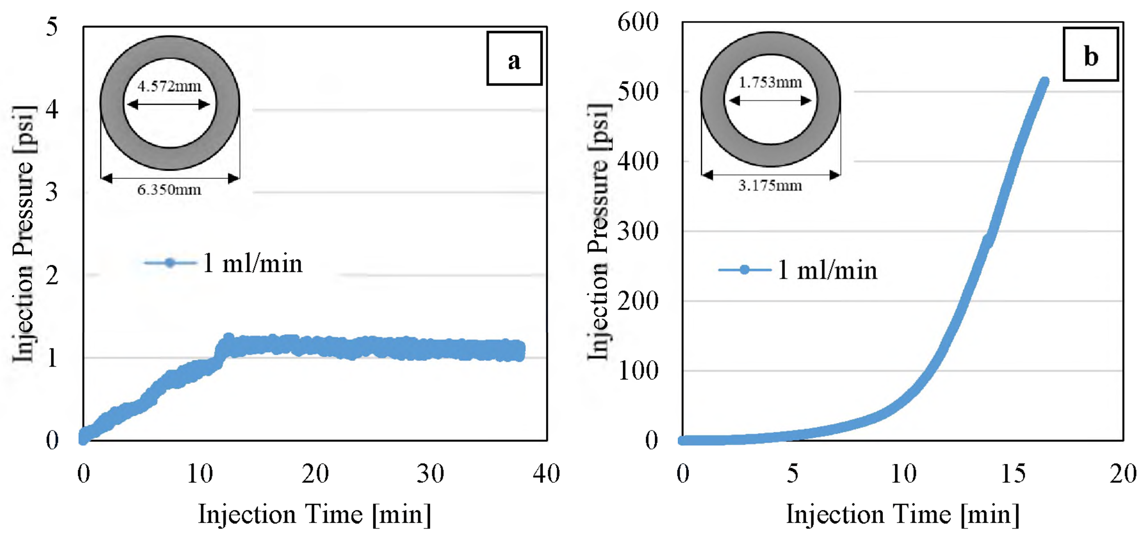


Figure 8. The injection pressure of cement as a sealant.

Referring to the injectivity result of the cement in the large channel, it is important to point out that the cement injection pressure started to fluctuate after around 40 minutes of injection as shown in Figure 9. The pressure seems to have separated the water from the cement as drops of water started to appear in the effluent between times to time.

This can be overcome using additives like fluid loss additives in the cement, which create more complicity to the remedial operation. The cement system used in this study was neat cement with no additives.

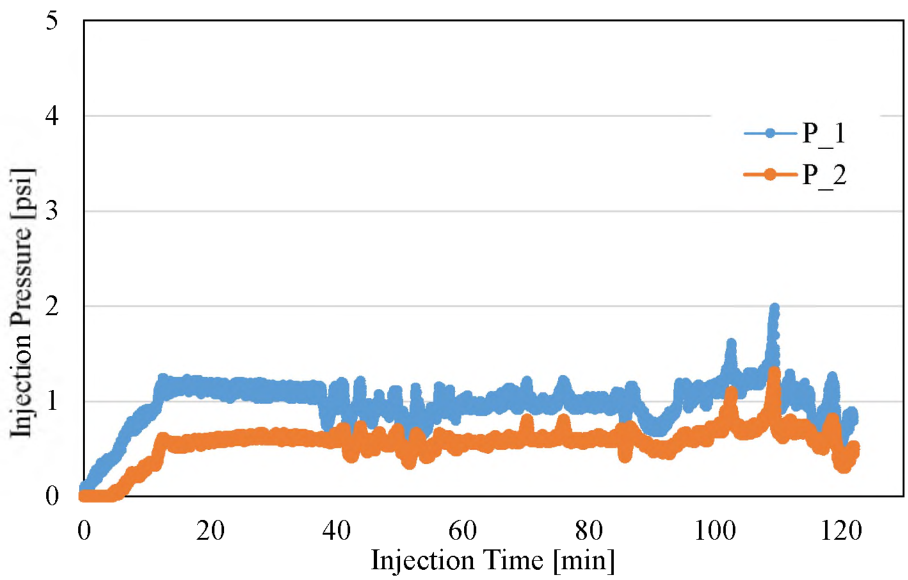


Figure 9. The injection pressure of cement as a sealant in model (a).

The very low injectivity of the cement in the small channel was related to following running squeeze method, where continuous pumping is used to force the cement into the squeeze interval (phase 1 in Figure 10). Using this method the injection pressure increased to around 514 psi and the reading of the halfway pressure transducer (P_2) was around zero. After reaching the high injection pressure in this experiment, it was interesting to switch to hesitation squeeze method in which the pumping sequence is started and stopped repeatedly. Using this method, the cement was forced in the small channel efficiently as shown in Figure 10 (phase 2), where P_2 increased to around 25 psi. Then, the injection continued with constant pressures (phase 3 in Figure 10). Using this method P_2 kept increasing until it reached around 100 psi indicating that the cement has penetrated the small channel. However, the effluent during this experiment was only few drops of water and no cement was produced. This part of the study will be expanded in the future to better understand the injectivity of cement. The placement method of the cement will be also studied.

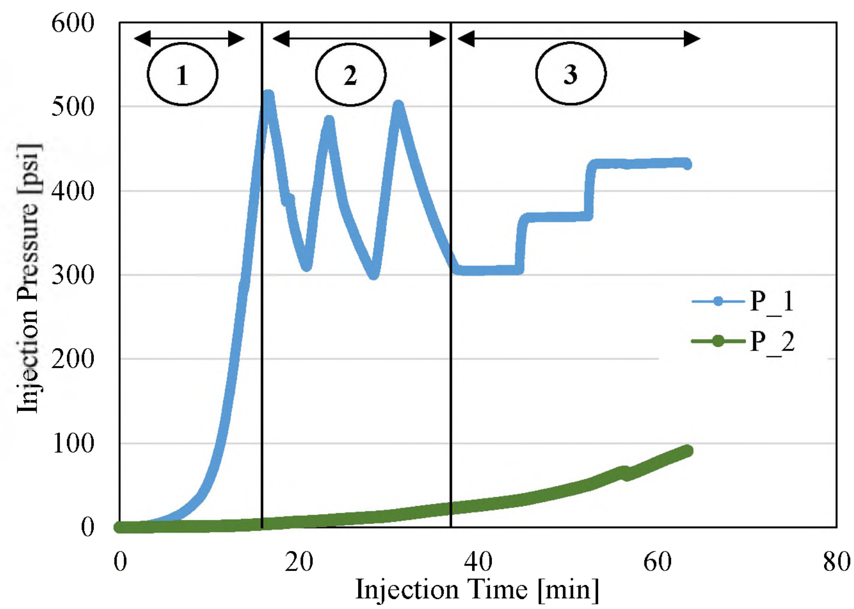


Figure 10. The injection pressure of cement as a sealant in small tube size.

4.2.4. Sealants Injection in Heterogeneous Features Results. For solids-free sealant, the injection pressure was not affected significantly when the heterogeneity was introduced. As shown in Figure 11a, the injection pressure of the sealant was lower than that of the small channel and higher than that of the large channel. This result indicates the ability of the solids-free material to penetrate the cement's channels at low pressures even in the presence of different channel. This important for the remedial operations to ensure successful placement of the sealant and full zonal isolation. The effect of smaller clearances must be studied in the future. Figure 11b presents the effect of heterogeneity on the injection pressure of semi-solids material. The results here indicate more significance of heterogeneity on the injection pressure of semi-solids material when compared to solids-free material. The pressure fluctuated and reaching stabilized pressure was harder. It can be noticed that the particles of the semi-solid material were packed in the large channel and the pressure increased before they entered the smaller channel. The particles were also deformed as a result of penetrating the small channel.

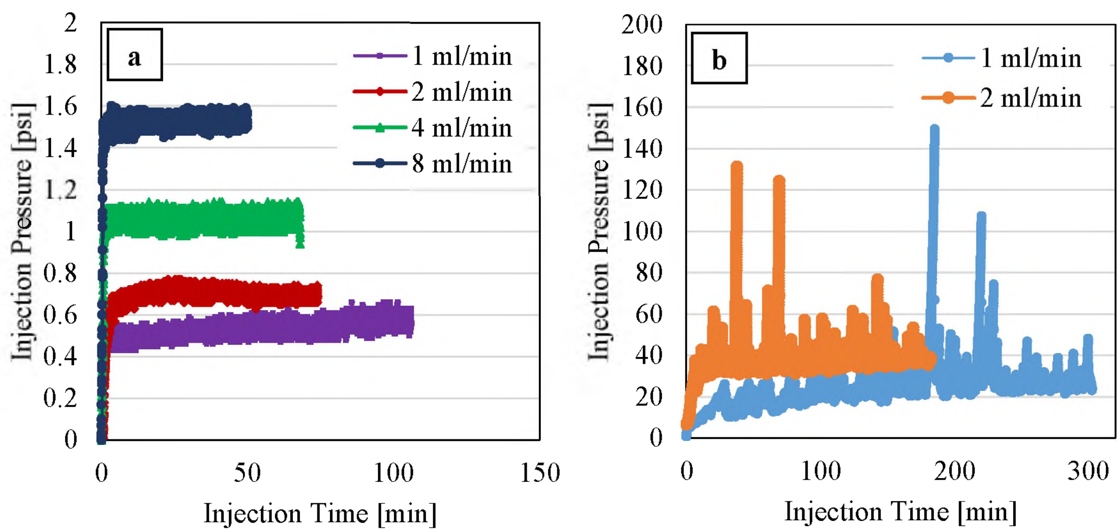


Figure 11. The injection pressure of solids-free sealant (a) and semi-solids sealant (b) in a heterogeneous model.

4.2.5. Sealant Materials Injectivity in Different Features. Table 2 summarizes the injectivity results of the sealants. It can be seen clearly that the injectivity results of the solids-free sealant were higher than that of the semi-solids material (PPG) and the solids material (conventional Portland cement).

For the small channel size (1.753 mm), the solids-free material penetrated the tube without any deformation or changes in the properties and the results of the injectivity ranged between 1.163 ml/min*psi to 4.444 ml/min*psi at different flow rates. The injectivity of the solids-free sealant increased when the flow rate was increased. On the other hand, the semi-solids material showed lower injectivity results in the small channel size and the polymer particles deformed upon flowing through the tube. This result indicates that the semi-solids material can penetrate the small channels but the properties of the material may change and affect the plugging performance. The trend of the injectivity with respect to the flow rate was similar to that of the solids-free material. For the solids material, the cement faced difficulties to penetrate the small channel size and required higher pressures and addition to the use of different injection method.

For the larger channel size (4.572 mm), the solids-free material and the semi-solids material were able to penetrate the tube easily and showed higher injectivity values. Once again, the solids-free material had the highest injectivity of the three sealants. It is important to point out that the semi-solids material in this case was able to penetrate the tube with no deformation and the injectivity results for the different flow rates were between 0.4975 ml/min*psi and 0.8949 ml/min*psi. The cement was also able to penetrate the large channel void and it noticed the importance of the fluid loss additives in the cement slurry to avoid the water separation and loss of properties. Generally, as expected the injectivity of the sealants increased when the size of the feature was increased.

For the heterogeneous model, there was no significant effect of heterogeneity on the flow of solids-free material but there was some effect on the semi-solids material as reaching stable pressure was difficult and the results of the injection pressure fluctuated. Based on these findings, solids-free material could be the best choice for cement remedial operations.

Table 2. Summary of the estimated injectivity of different sealants in [ml/min*psi].

Flow Rate [ml/min]	Channel size (1.753 mm)			Channel size (4.572 mm)			Heterogeneous Channel	
	Solids-free Sealant	Semi-solids Sealant	Cement Sealant	Solids-free Sealant	Semi-solids Sealant	Cement Sealant	Solids-free Sealant	Semi-solids Sealant
1	1.163	0.0117	0.002	10	0.4975	0.909	1.786	0.0354
2	1.852	0.0231	-	10.526	0.5917	-	2.899	0.0514
4	3.077	0.0414	-	19.048	0.8949	-	3.809	-
8	4.444	0.0602	-	30.769	0.6797	-	5.298	-

4.3. COMPRESSIVE STRENGTH RESULTS

The compressive strength of the cement sheath was found to be around 720 psi after 24 hours curing and around 3,816 psi after 72 hours. On the other hand, the epoxy resin compressive strength was found to be higher than 8,500 psi after 24 hours. The epoxy resin samples did not fail at high loads and show high ductility when compared to Portland cement. These results prove that epoxy resin have higher strength than Portland cement and can be used more effectively. The PPG as a material has no compressive strength.

5. CONCLUSIONS

By studying the three types of sealants that can be used in cement remedial jobs, several findings were obtained. These findings are based on the results and the analysis of the rheological measurements, the injectivity results, and the compressive strength result. The main conclusions are summarized below:

- The epoxy resin exhibited Newtonian behavior with very low or no yield stress on contrast of Portland cement, which exhibited Bingham-plastic flow.
- The injectivity of the solids-free material is higher than that of the semi-solids and solid cement especially in small channel voids.
- The solids-free material was able to penetrate the small channel voids at very low pressures.
- Unlike solids-free sealant, both solid cement and semi-solids sealant deformed when they flow through small size tube indicating that the size of the cement voids play a major role on the performance of these sealants.
- The curing time of the epoxy resin can be controlled, and the compressive strength of the epoxy resin is higher than that of the cement.

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IV. LABORATORY STUDY USING POLYMER RESIN SYSTEMS TO REMEDIATE WELLBORES: RHEOLOGICAL CHARACTERIZATIONS, CHEMICAL RESISTANCE, PLUGGING PERFORMANCE, AND MECHANICAL PROPERTIES

ABSTRACT

Carbon capture and storage (CCS) in oil and gas reservoirs always requires maintaining full control of fluids within a well so as to prevent unintended fluid migration, which may harm the environment. The well integrity must be maintained to implement such projects successfully. The cement in oil and gas wells, when compromised, may provide pathways for fluids to migrate. Cement is one of the most common sealants employed to seal such pathways. Recently, alternatives to conventional Portland cement were developed to overcome cement limitations. One of those alternatives is thermosetting sealant.

This study is intended to evaluate three types of thermosetting materials, epoxy resins. The study includes examining the rheological behavior of the sealants under different temperatures, the curing kinetics of the sealants, the effect of temperature on the curing time, the thermal degradation and glass transition temperatures, the injectivity of the sealants in small gaps, the chemical resistance of the sealants in presence of corrosive fluids, the plugging capability, and the mechanical properties. The findings of this work prove the ability of the epoxy resins to penetrate and plug cements' pathways, providing zonal isolation.

1. INTRODUCTION

To employ oil and gas reservoirs for carbon capture and storage projects, the well integrity of the oil and gas wells must be maintained as these wells have been identified to be the most vulnerable to provide pathways for CO2 to escape (Todorovic et al., 2016). The well integrity must also be maintained during any period of the life of oil and gas wells from drilling the well through production and even after plug and abandonment. Conventional Portland cement have been used for primary, secondary, and plugging and abandonment cementing. The cement is placed in the annulus as a barrier between a steel pipe casing and the various formations. The cement must be able to protect and support the casing, and to isolate production zones, preventing wellbores' fluids from migrating in an annular flow path so as to allow the well to be used without any control problems (Alkhamis and Imqam, 2018). However, during the life of the well, the cement is prone to deterioration and the cement may fail to deliver full zonal isolation. Cements' failures may occur due to chemical causes or due to mechanical reasons (Jimenez et al., 2016; Alkhamis and Imqam, 2021). In both cases, the outcomes can severe and can affect the environment and the human life. In order to overcome the consequences of the cement failures, researchers all over the world have been working in developing alternative materials that can be applied safely to remediate cements' leakages and restore the integrity of oil and gas wells whether to continue extracting oil and gas, plugging the wells for good, or to use the wells for CO2 storage projects.

As an alternative to Portland cement, epoxy resins can be used as sealants materials. Epoxy resins are thermosetting polymers, which contain in their unset phase (prior curing) one or more epoxide groups. Epoxide group is one or more three-membered rings, known also as oxirane, and/or epoxy. The molecular weight of epoxy resins varies greatly. They exist in the forms of solids and liquids with wide range of viscosities. Chemically, the epoxide groups in the resin may react with many types of curing agents that contain hydroxyl, carboxyl, amine, amine group. The result of the reaction is a hard 3D cross-linked network. Some other types of epoxy resins may be cross-linked by themselves through catalytic homopolymerization. Some systems will cure at ambient temperature, but many require heat to cure (T=150-200 ˚C) (Marfo et al., 2015). Other additives or fillers and diluents may be added to modify the properties of the final product.

Todorovic et al., 2016 tested the ability of a commercially available epoxy resin sealant to plug artificial fractures that were created in cement cores. The sealant sealed the fractures completely in one case, where the permeability of a fracture (created between steel plate and cement) was reduced from 47 Darcy to almost zero and reduced the permeability in a second sample (cement to cement fracture) from 1717 Darcy to 41 Darcy. Alsaihati et al., 2017 evaluated the rheological behavior, the mechanical properties, and the thickening time of an epoxy resin sealant and compared the obtained results to that of a polyester based sealant. The results showed the ability of the epoxy resin to develop high compressive strength in short time and the tensile strength ranged from 100 to 2000 psi. In addition to the low viscosity of the epoxy resin sealant at ambient temperature (26.7 ˚C). The system was applied in gas wells in Saudi Arabia and was able to seal leakages. In 2018 Khanna et al., presented a field case where a leakage in cement was detected. Epoxy resin system was injected into a channel of approximately

0.3875 inches. Due to the elastic nature, high penetration capacity, and the fluid immiscibility of the epoxy resin system, a rig-less remedial operation was conducted successfully. Singh et al., 2019 demonstrated the ability of epoxy resin to repair casing leak. In this work, the type, concentrations of resin and the curing agent, in addition to, the temperature of reaction and glass transition temperature were discussed. This study proved how extremely easy to execute a remedial operation using epoxy resin. Several other superior properties of epoxy resin sealants were discussed in several publications such as the surface wetting and adhesion abilities (Brooks et al., 1974; Shaughnessy et al., 1978); the ability to penetrate narrow voids (Todd, L et al., 2018); tunable density (which can be used for areas with a narrow fracture gradient) (Sanabria et al., 2016); and resistance to contamination (Vicente et al., 2017). In addition, the high mechanical strength (Ali, A et al., 2016; Elyas et al., 2018), stability at high temperature (Bertram et al., 2018), and good bonding properties (Moneeb et al., 2017). As a bonus, the polymerization reaction of polymer resins forms no by-product during hardening (Muecke, 1974), resulting in very little to no shrinkage. These properties have been also reported by Alkhamis et., (2019).

In this work, three epoxy resin systems were selected for evaluation. One that can be cured at room temperature without the need of elevated temperatures (Epoxy resin A). A second one that cures at room temperature but require longer time to cure (Epoxy resin B) because such sealant can be used where moderate temperatures are present. The moderate temperatures will accelerate the chemical reaction of the sealant, reducing the curing time. The third sealant (Epoxy resin C) is an epoxy resin that was applied in the field successfully. This sealant requires elevated temperature to cure and this work is an attempt to understand the effect of varying the temperature on the curing of this sealant. This work characterizes three epoxy resin systems based on rheological properties, chemical resistance, plugging performance, and mechanical properties.

2. EXPERIMENTAL METHODOLOGY

2.1. MATERIALS

2.1.1. Epoxy Resin A. The mixture of epoxy resin A was prepared by mixing at room temperature epoxy Novolac (Phenol, polymer with formaldehyde, glycidyl ether, and o-Cresyl glycidyl ether) with an aliphatic hardener (a blend of benzyl alcohol, 1,2-cyclohexanediamine, diethylenetriamine, and Bisphenol A). Both the resin and the hardener were obtained from Euclid Chemicals Company. The resin was accurately weighed in a glass beaker and then the hardener was added (50% by weight of resin). The sample was stirred thoroughly at which point a clear homogeneous mixture obtained.

2.1.2. Epoxy Resin B. The mixture of epoxy resin B was prepared by mixing at room temperature a 1:1 stoichiometric mixture of the base resin with an aliphatic hardener. The base resin is diglycidyl ether of bisphenol A (DGEBA) obtained from Miller-Stephenson Chemical Company, Inc. diluted with cyclohexanedimethanol diglycidyl ether (CHDGE) obtained from Miller-Stephenson Chemical Company, Inc. The diluent amount added to the resin was 100% by weight of resin. The selection of this amount was based on previous study conducted by (Alkhamis and Imqam, 2020). The aliphatic hardener was polyetheramine (PEA) obtained from Huntsman corporation. The diluted DGEBA was accurately weighed into a glass beaker with the appropriate amount of PEA (35% by weight of diluted resin). The sample was stirred thoroughly at which point the curing agent was completely dissolved and a clear homogeneous mixture obtained.

2.1.3. Epoxy Resin C. The mixture of epoxy resin C was prepared by mixing at room temperature a 1:1 stoichiometric mixture of the base resin with an aromatic hardener. The base resin was the same base resin of epoxy resin B. The aromatic hardener was diethyltoluenediamine (DETDA) obtained from Albemarle chemical company. The diluted DGEBA was accurately weighed into a glass beaker with the appropriate amount of DETDA (52% by weight of diluted resin). The sample was stirred thoroughly at which point the curing agent was completely dissolved and a clear homogeneous mixture obtained.

2.1.4. Class-H Cement. The cement used was prepared by mixing API class H cement obtained from Halliburton company with distilled water. The water/cement ratio was 0.38, as stipulated in API specification 10A (API, 2010). The mixing was conducted in accordance with the mixing procedure of API RP 10B-2 (API, 2013), in which water was added first to a two-speed, bottom-drive laboratory blender, after which dry cement was added gradually to the blender while mixing at low speed for approximately 15 s. Then, the speed of the blender was increased to high speed for around 35 s.

2.2. DENSITY MEASUREMENTS

Since the epoxy resin sealants studied in this paper are intended for wellbore remedial operations, it was important to control their density. In general, a density that is higher than the density of water is desired. In this work, the densities of the mixed epoxy resin sealants were measured using simple weighting method, where a specific volume of each epoxy resin was placed on a high precision balance to measure the mass. Then, the density of each epoxy resin was calculated by dividing the mass of the epoxy by its volume. The density was recorded in [gm/ml] and converted to [lbm/gal].

2.3. SHEAR VISCOSITY MEASUREMENTS

The viscosities of the epoxy resin sealants were measured using a dynamic shear Rheometer (DSR) (model: MCR 302) with parallel plates system supplied by Anton Paar. For these measurements, samples of about 0.5-1.0 ml of each epoxy resin sealant were placed on a disposal plate of 25 mm in diameter on the lower part of the instrument. Then, the upper plate was lowered to a gap between 0.5 to 1.0 mm. The readings were taken in both ascending and descending order in a range of 0.1 1/s to 1000 1/s. The aim of these measurements was to evaluate the effect of shear rate on the viscosities of the epoxy resin sealants. Testing the sealants under different shear rates mimics the placement of the sealants in the wellbore.

2.4. ISOTHERMAL CURING MEASUREMENTS

The isothermal curing experiments of the epoxy resin sealants were conducted to identify the gelation time of the epoxy sealants and the effect of temperature on the gelation time. Defining the workability of the sealants in terms of gelation is essential in the proposed application of this work to protect downhole equipment and to ensure a safe and successful placement of the sealants in the wellbore without premature curing. Sinusoidal oscillatory tests using the same DSR were conducted at an angular frequency of 10 rad/s (1.5915 Hz) and the complex viscosity, storage modulus, and loss modulus change with time were monitored. The samples were placed following the same procedure mentioned in section 2.3. The isothermal curing of the epoxy resin samples was measured at different temperatures ranges between room temperature to 120 ˚C.

2.5. CALORIMETRY MEASUREMENTS

The calorimetric properties of the three epoxy resins evaluated in this study were studied by using a differential scanning calorimeter (DSC), model SDT Q600 V20.9 Build 20. For this measurement, epoxy resin samples of approximately 5-10 mg were used in alumina pans. The measurements were performed under nitrogen atmosphere in temperature ramp mode from room temperature up to 600 ˚C. The temperature rate was set to 5 ˚C/min. The goals of these measurements were to evaluate the heat reaction of the epoxy resins as a function of curing temperature and time. In addition, to evaluate the thermal degradation temperature of the epoxy resins along with the glass transition temperature (T_g). Five experiments were conducted using the three epoxy resins. The first three were using epoxy resin A. The samples were cured at three different temperatures room temperature, 50, and 80 ˚C. The other two were using epoxy resin B and Epoxy resin C cured at 50 and 80 ˚C, respectively.

2.6. CHEMICAL RESISTANCE MEASUREMENTS

Following the injection of cement or other materials in the well. The materials are subjected to corrosive fluids downhole. The downhole conditions vary from low to high temperatures/pressures and the presence of corrosive fluids that can compromise the sealants. To test the ability of epoxy resins to withstand corrosive fluids, specimens of epoxy resins and cement were immersed in water, 50% sulfuric acid, 98% sulfuric acid, 10% sodium hydroxide (NaOH) solution, 50% NaOH solution, mineral oil, 10% sodium chloride (NaCl) solution, and 36% NaCl solution at room temperature. In addition, samples were placed under pressure and temperature of supercritical CO2. The weight change was recorded after 3 days, 28 days, and 3 months. The results of the performance of the epoxy resin sealants were compared to that of cement.

2.7. INJECTIVITY AND PLUGGING PERFORMANCE MEASUREMENTS

Prior to any remedial operation, an injectivity test is performed to set the pressures and flow rates at which remedial fluids can be pumped into leakages zones. This test helps in determining the key parameters for the treatment as well as the major limitations of the operation. The injectivity of the epoxy resin sealants was estimated by injecting the sealants into stainless-steel tubes with various inner diameters (i.e., 0.876, 1.753, and 4.572 mm) at constant flow rate and recording the injection pressures. Then the injectivity was calculated by dividing the flow rate by the injection pressure.

The plugging performance of the epoxy resin sealants was evaluated using the experimental setup in Figure 1. The experimental setup, consisted of a syringe pump, an accumulator, two pressure transducers, a hand pump, and a core holder. The syringe pump was used to inject the water/CO2 in the accumulator and then to the core holder, where the cement cores were placed. The hand pump was used to apply confining pressure around the cores forcing the water/CO2 that was injected by the syringe pump to the cement channel and the pressure transducers were used to record the pressure drop across the cement cores. The confining pressures around the cores were maintained at pressure of 2600 psi during the water experiments and at pressure of 1000 psi during the CO_2 experiments. A second syringe pump and a back-pressure regulator were used in the CO_2 experiments to control the flow of the CO_2 at the outlet.

The permeabilities of the cement channels prior to the treatments were estimated by calculating the pressure loss across the cement core using Hagen-Poiseuille equation (Equation 1), which is a physical law that obtains the pressure drop across uniform cylindrical pipes assuming incompressible and Newtonian fluid that flows in laminar flow. The calculated pressure drop was then substituted in Darcy Law (Equation 2), leading to Equation 3 that was used to estimate the permeability of the channels.

$$\Delta P = \frac{8 * \mu * L * Q}{\pi * r^4} \tag{1}$$

where ΔP is the pressure loss expressed in (pa), μ is the dynamic viscosity expressed in (pa.s), L is the core length expressed in (m), Q is the volumetric flow rate expressed in (m^3/seconds), and r is the channel radius expressed in (m).

$$\Delta P = \frac{Q * \mu * L}{k * A} \tag{2}$$

where, k is the permeability expressed in (mD), A is the cross-sectional area of the channel expressed in (cm^2), Q is the volumetric flow rate expressed in (cm^3/seconds), μ is the dynamic viscosity expressed in (cp), L is the core length expressed in (cm), and ΔP is the pressure loss expressed in (atm).

$$k = 2.0428 * 10^{10} * d^2 \tag{3}$$

where, k is the permeability expressed in (mD) and d is the channel's diameter expressed in (inches).

The permeabilities of the channels after the treatment were calculated using Darcy Law (Equation 2) and the pressure drops in this case were measured using the transducer placed at the inlet of the core holder of Figure 1. The permeabilities were calculated using water. Each permeability result presented in this study is the average value of three experiments.

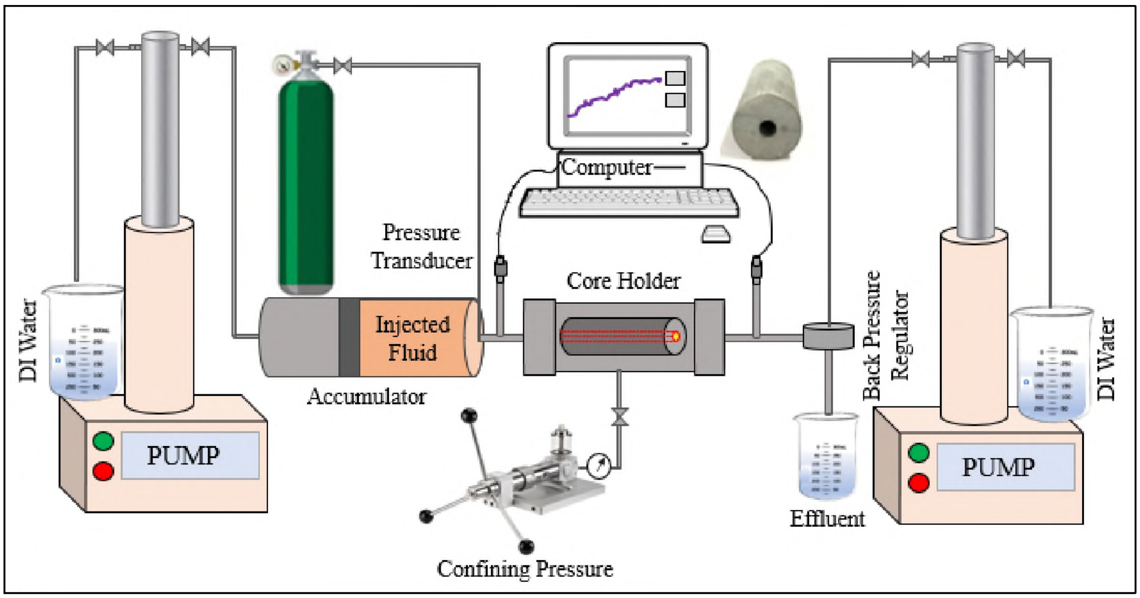

Figure 1. Illustration of the plugging performance setup.

2.8. MECHANICAL MEASUREMENTS

2.8.1. Compressive Strength Measurement. Determining the compressive strength of the sealants is essential to ensure the ability of the sealants to withstand downhole conditions. Compressive strength test simply measures the force needed to crush a sample of any material. For this test, the sealants were mixed and poured in 2” × 2” × 2” cubic molds and/or 2” x 4” cylindrical molds. The molds then were placed in a curing bath at the desired temperature for 24 hours. Then, the cured samples were carefully removed from the molds. The specimens' dimensions were measured using a caliper and the minimum surface area was calculated. Any specimen of the cubic samples with height less than 48 mm (1.9-inch) was discarded as recommended by (API RP 10B-2 2013). Finally, the specimens were placed in a hydraulic press where force was applied until failure. Axial/lateral laser sensors were used to detect axial/lateral deformation of the samples. The compressive strength results were calculated as force over area. At least three samples for each sealant were measured.

2.8.2. Tensile Strength Measurements. Tensile strength is another parameter that must be measured to ensure the wellbore long-term integrity. When the cement tensile strength exceeded, a radial crack may form along the cement sheath axis, which would create a high conductivity path for the fluids to migrate (Iremonger et al., 2015). Herein, the tensile strength was measured using an indirect method that is known as the Brazilian test. The technique used in this test is applying diametric compression force to induce tensile stresses across the diameter of the sealant cylindrical sample until failure occur. For this test, 2" × 4" cylindrical molds were used. The molds were placed in a curing bath at the desired temperature for 24 hours. Then, the samples were removed from the molds and sawed into three samples of 1" in thickness. The samples diameter and thickness were measured and documented. Then, the samples were placed in a hydraulic press machine where force was applied until failure. It is important to check if the failure crack is parallel to the load direction, otherwise the result is not reliable (Iremonger et al., 2015).

3. RESULTS AND DISCUSSION

3.1. DENSITY RESULTS

After mixing the sealants, samples of 0.2 to 0.3 ml were weighted in order to obtain the densities of the sealants. The density of each of the three sealants was higher than that of the water. The densities of the Epoxy Resins A, B, and C were 1.2, 1.13, and 1.05 gm/ml, respectively. These densities can be easily modified, when required, by adding materials with high specific gravity known as “weighting agents”. In this paper, the density was measured for the pure sealants without any additives. Table 1 lists the results of the density measurements. The values listed in the table are the average values of three measurements were conducted.

Table 1. The density of the epoxy resin systems.

Sealant	Volume [ml]	Mass [gm]	Density [gm/ml]	Density [lbm/gal]
Epoxy Resin A	0.3	0.360	1.20	10.0145
Epoxy Resin B	0.5	0.566	1.13	9.44699
Epoxy Resin C	0.2	0.210	1.05	8.76267

3.2. RHEOLOGICAL RESULTS AND ANALYSIS

3.2.1. Effect of Shearing and Temperature on Viscosity. It was essential to measure the viscosity of sealants prior to injecting them into the cement gaps. The viscosity of Epoxy resin A, which was intended for application of low temperatures was measured at three temperatures, 24, 30, and 40 ˚C as shown in Figure 2. The viscosity of Epoxy resin A at room temperature ranged between 1000 to 1100 cp with a small effect of shear rate. At this temperature the material exhibited a shear thinning behavior as can be seen in Figure 2a. Increasing the temperature from 24 to 40 ˚C reduced the viscosity of the epoxy from around 1100 to approximately 700 cp.

Figure 2b shows the shear stress vs. shear rate for Epoxy resin A and one important parameter that affect the placement of sealants rose, which is the yield stress. The sealant shows very low yield stress that is required to initiate flow. The measurements of the viscosity wee only conducted at small and low range of temperature since the material react faster under higher temperatures, which will be explained later in this work.

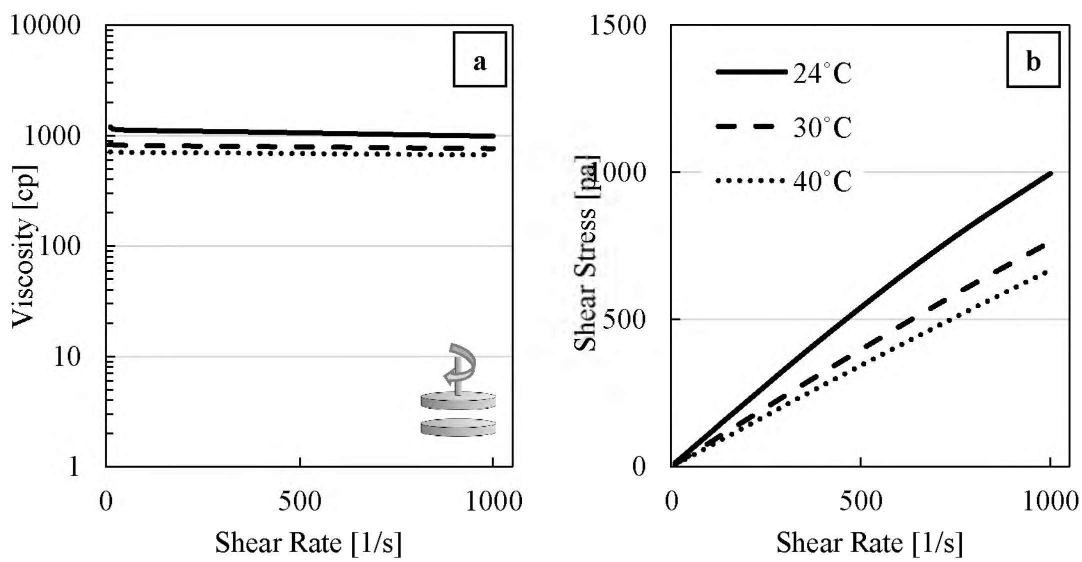

Figure 2. (a) Viscosity results of Epoxy Resin A as a function of shear rate. (b) The rheological behavior of Epoxy Resin A.

The viscosity of Epoxy resin B was measured at wider range of temperatures from 24 to 80 °C and the effect of temperature on the viscosity was higher than Epoxy resin A (see Figure 3a). First, the viscosity of Epoxy resin B at room temperature is lower than that of Epoxy resin A. This is favorable for the application of Epoxy resin B as it was intended for deeper targets in the well, where moderate temperatures are expected. The low viscosity of Epoxy resin B is a result of two parameters implemented in this sealant: (1) the use of reactive diluent in the base resin, which was mixed to reduce the viscosity of the resin and (2) the low viscosity characteristic that the curing agent offers. The viscosity of Epoxy resin B at room temperature was around 118 cp and decreased to around 30 cp when the temperature increased to 80 °C. The material exhibited Newtonian like behavior, where no stress or only a very small stress was required to initiate flow, and the viscosity was independent of the shear rate (see Figure 3b).

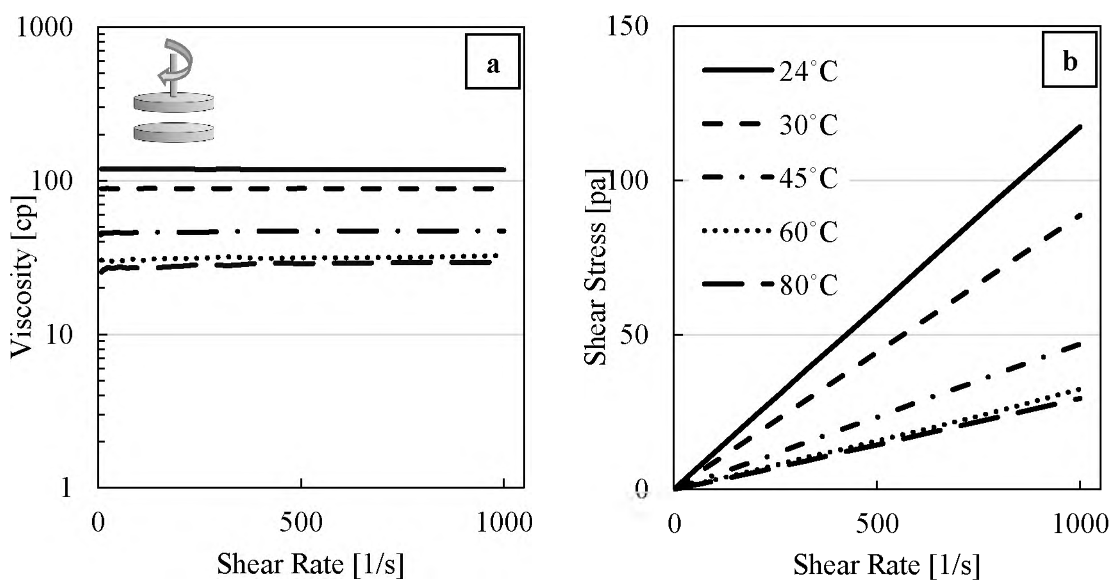

Figure 3. (a) Viscosity results of Epoxy Resin B as a function of shear rate. (b) The rheological behavior of Epoxy Resin B.

On the other hand, Epoxy resin C, which was intended for application of high temperature experienced the greatest impact of temperature on viscosity. Increasing the temperature from room temperature to 80 °C reduced the viscosity by approximately 95% (from 466 to 23 cp), almost 22% higher reduction in viscosity than that of Epoxy resin B. Figure 4a shows the viscosity of Epoxy resin C vs. shear rate. Similar to Epoxy resin B, this sealant exhibited Newtonian like behavior (Figure 4b). The low viscosities of Epoxy resin B and C is desirable property as those materials were intended for deep applications (cement gaps at deeper locations in the well) and the low viscosities will help injecting those sealants into cement gaps with lower pressures requirements.

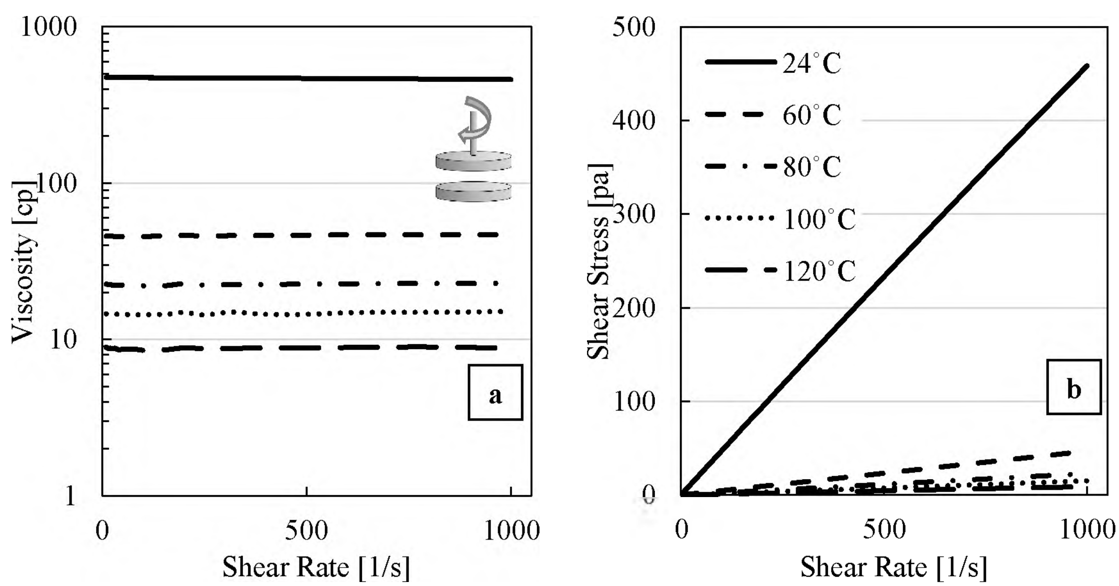

Figure 4. (a) Viscosity results of Epoxy Resin C as a function of shear rate. (b) The rheological behavior of Epoxy Resin C.

3.2.2. Effect of Temperature on Curing Kinetics. The effect of temperature on the curing of the three epoxy resins was conducted to identify the gelation time.

Identifying the gelation time of the sealants is essential to protect downhole equipment from premature curing and to ensure a safe and successful placement of the sealants in the wellbore. Placing the sealants in the cement gaps successfully is not enough to guarantee full zonal isolation, it is important to confirm that the epoxy resin is cured in place to provide zonal isolation before proceeding with other operations.

First, Epoxy resin A was mixed and placed on the plate of the rheometer at room temperature and the test started. The curing time of the epoxy at room temperature is shown in Figure 5. At early stages of the curing G' and G'' show low values, where G'' was higher than G', showing that the system is in the viscous region. As time proceeded, specifically, after 11 hours the system reached the gelation time at which G' crossed over G'' and the system entered the elastic region. This point is called gel point. Increasing the temperature of the curing showed that the temperature has an inverse relation to the gelling point as the gelling point of the Epoxy resin A at 30 ˚C was approximately 8 hours.

Similar trend can be seen on Figure 5 for the temperatures 45 and 60 ˚C. However, at those two temperatures the curing time is too short and pre-maturing problems can occur in the field. Figure 6 shows complex viscosity increasing with time during the curing process. It can be seen the effect of temperature on the complex viscosity. This property can help understanding the state of the sealant with respect to time. From these results it can be concluded that the epoxy resin require shorter sitting time than the cement and thus shorter remedial job time.

It is also useful to consider the wait on the operation time. Shortening the waiting time can save a great amount of money.

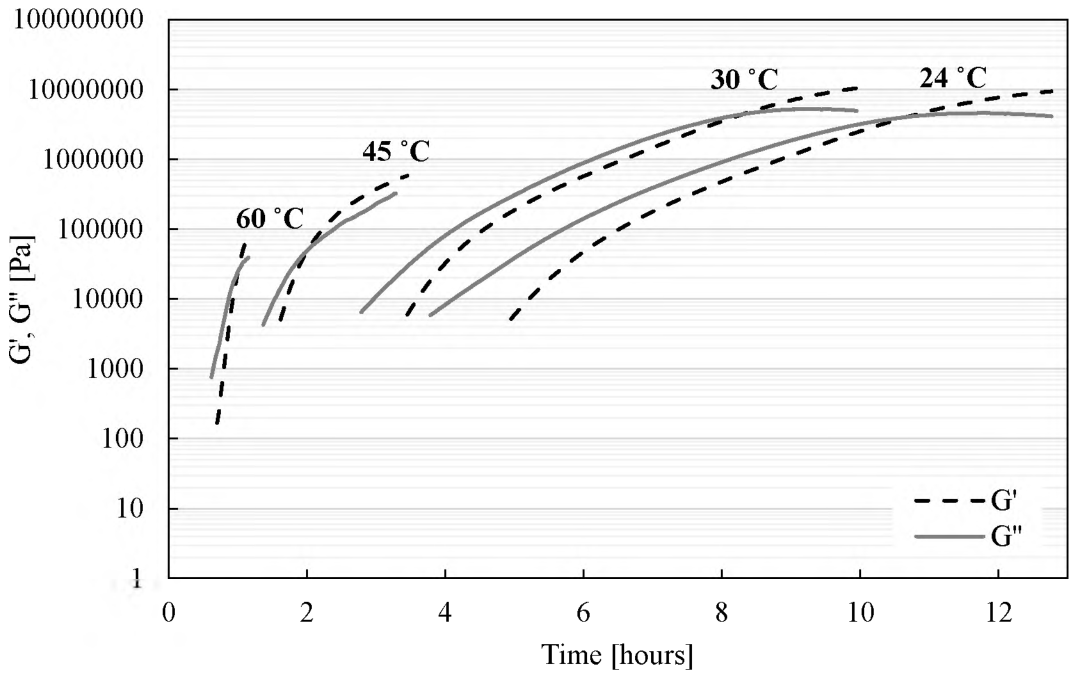

Figure 5. Effect of the temperature on curing of Epoxy Resin A.

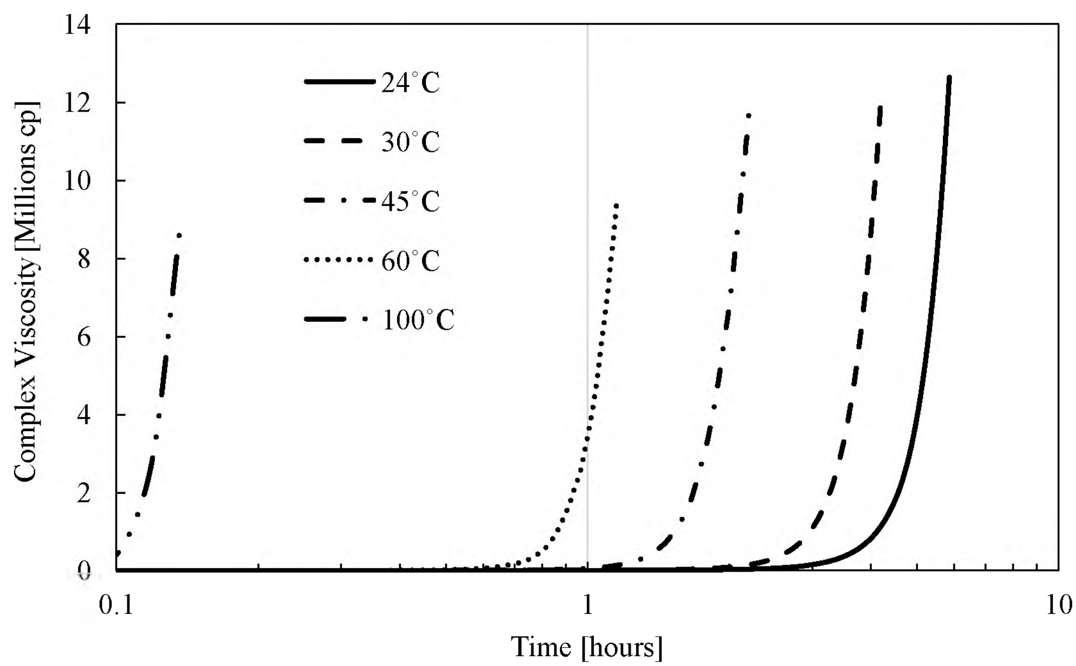

Figure 6. Complex viscosity results of Epoxy Resin A.

Second, the curing kinetics of Epoxy resin B were determined employing the same procedures of followed in determining the curing kinetics of Epoxy resin A. The curing experiments were conducted at room temperature then increased to up to 100 ˚C. Epoxy resin B showed the same behavior of Epoxy resin A as can be seen on Figures 7, 8, and 9. At room temperature, the time to reach the gel point was around 65 hours which is a very long time for rig workover. The long time of curing because of the use of aliphatic amine hardener. For this reason, Epoxy resin B was selected for moderate temperature applications in which the temperature will speed up the curing process as shown in Figure 8. In addition, it can be observed that at temperatures as high as 60 ˚C, the curing time is short so, this suggest that this material can be used for temperatures between room to 50 ˚C.

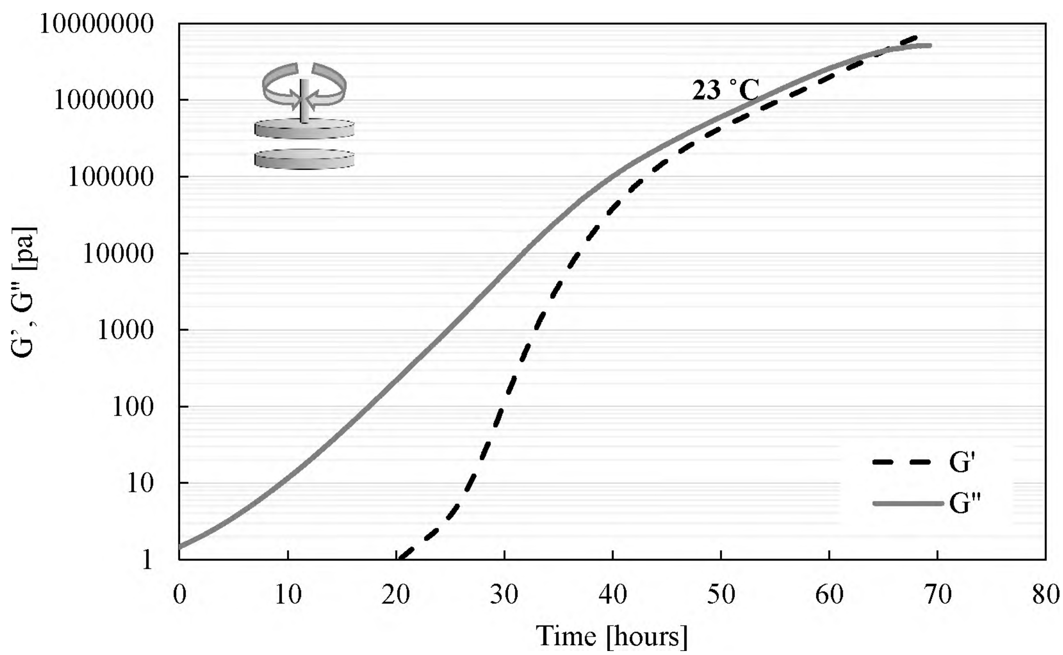

Figure 7. Curing results of Epoxy Resin B at room temperature.

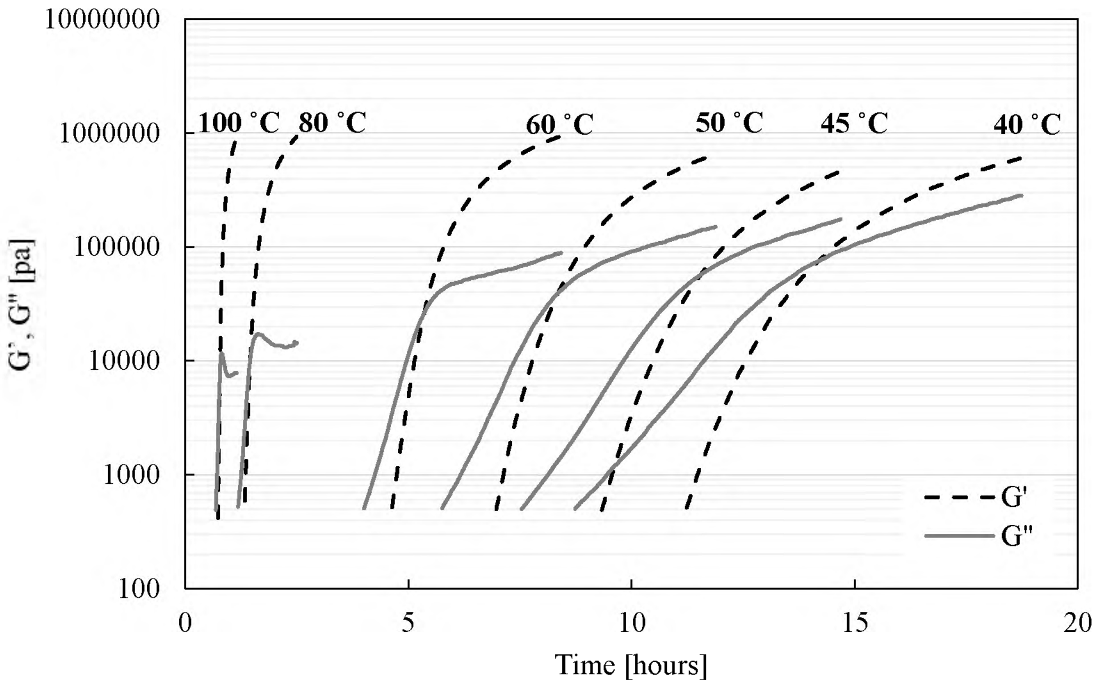

Figure 8. Effect of the temperature on curing of Epoxy Resin B.

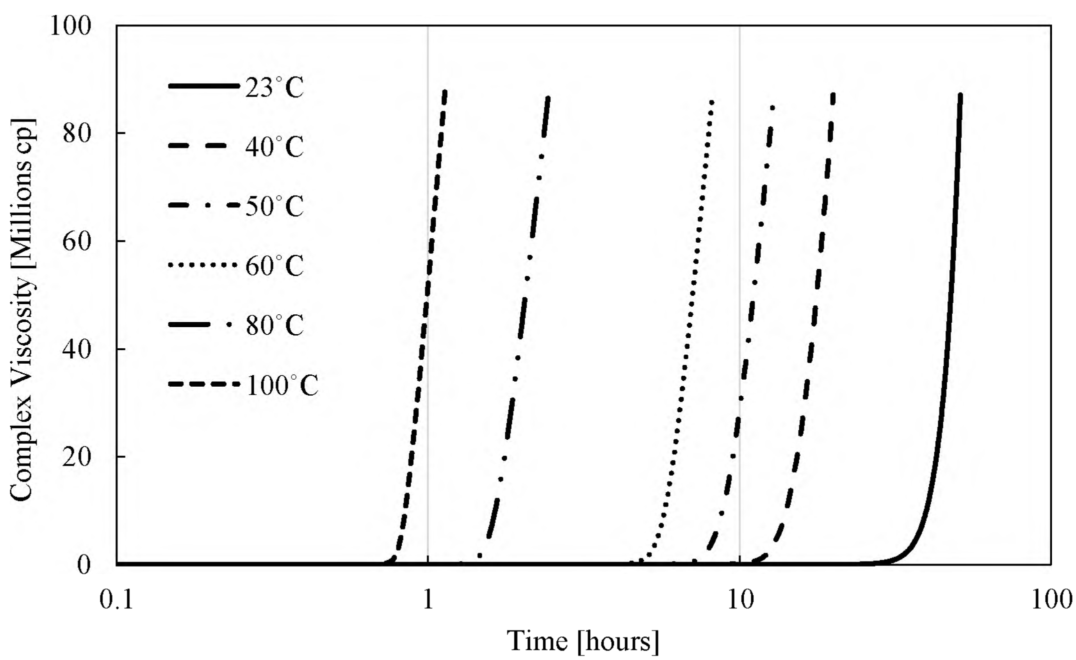

Figure 9. Complex viscosity results of Epoxy Resin B.

The last set of experiments were conducted using Epoxy resin C in which aromatic amine hardener was employed. This type of hardener requires elevated temperature to cure. This sealant was used successfully in the field as addressed by (Alsaihati et al., 2017; Elyas et al., 2018; and Singh et al., 2019) but curing time of this sealant needed to be studied and especially the effect of temperature on the curing. Once again, the temperature reduced the curing time of the epoxy resin (see Figures 10 and 11). At 80 ˚C, the time required for the sealant to reach the gel point was approximately 18 hours while at 100 ˚C was 7 hours.

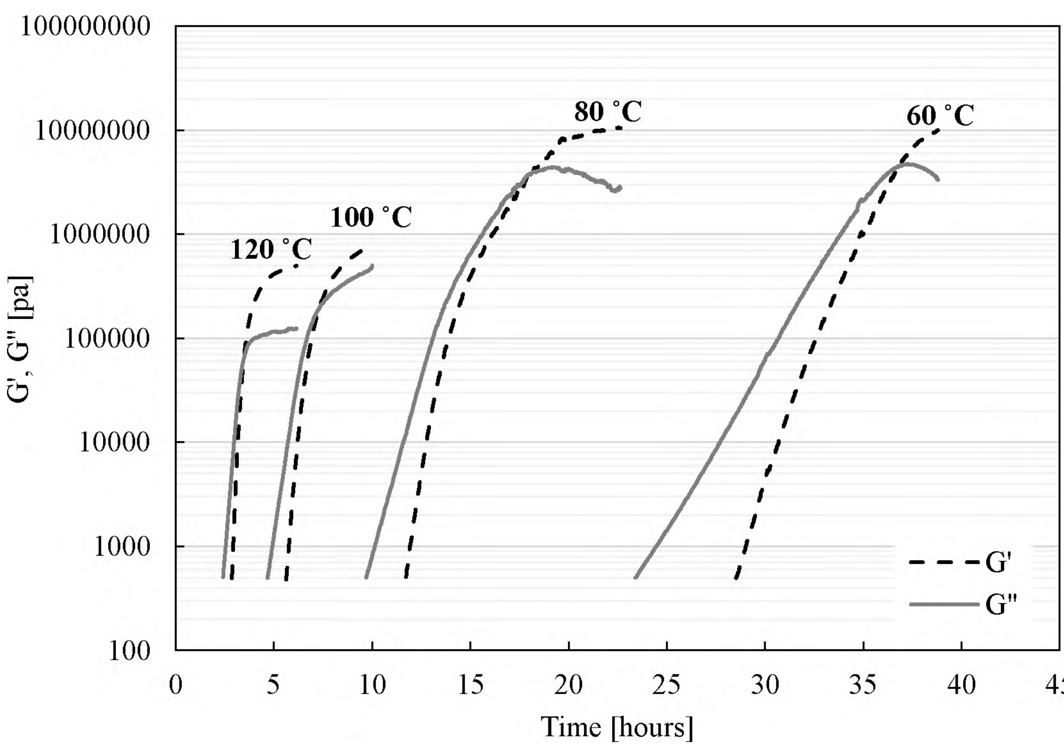

Figure 10. Effect of the temperature on curing of Epoxy Resin C.

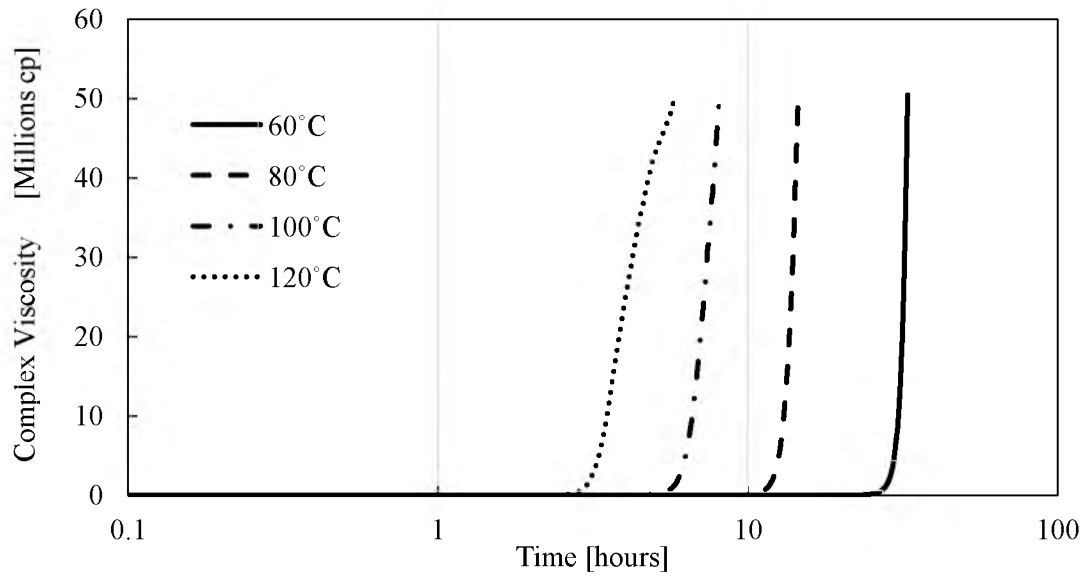

Figure 11. Complex viscosity results of Epoxy Resin C.

These results are accurate for the time required for the sealants to form 3D network that is able to stop well's leakage but the pumping time and the thickening of the material during the pumping process are different and require the use of consistometer where the workability and the consistency of the sealants are monitored while the material is under continuous shearing. The measurements here mimic the curing of the sealants after the placement inside the cement gaps and how much time is required for sealants to develop enough strength to withhold downhole conditions.

3.2.3. Sensitivity of Curing due to Temperature Change. It is known that a pre-flush is usually conducted prior to any remedial operation. This pre-flush is conducted to estimate the pressure and temperature of targeted zones. The pre-flush is important when cement is used but it is more important when thermosetting materials are employed. Figures 12a and b demonstrate this importance. A change of temperature as low as 5 °C affected the curing time by around 20%. The temperature estimation during the pre-flush must more accurate and the change in the temperature after the pre-flush but before the sealant placement must also be considered to ensure successful remedial job.

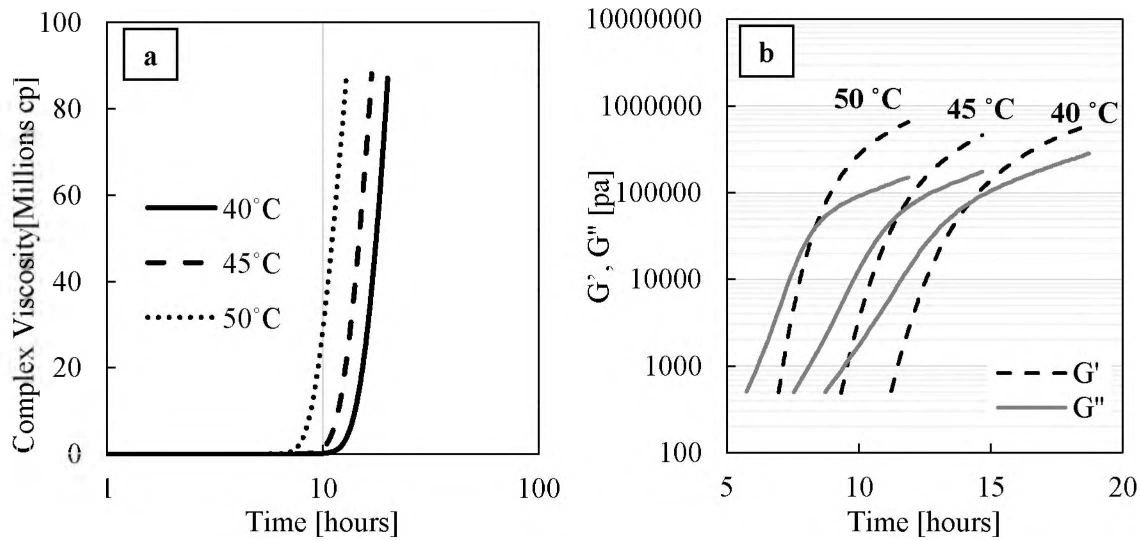

Figure 12. Effect of the small temperature change on curing of Epoxy Resin B.

3.3. CALORIMETRY RESULTS AND ANALYSIS

The calorimetry measurements were conducted to identify the thermal degradation temperatures of the epoxy resin sealants. In all the cases, first stage mass loss is related to the loss of surface moisture, inherent moisture and other volatiles present in the samples. As some of the chemicals are not 100% pure but either diluted resin and/or hardener. It means they contain some solvent, which will also contribute to this first stage mass loss. Figures 13, 14, and 15 show mass loss. For Epoxy resin A, the mass loss was in three stages. The first was from room temperature to 210 °C, where up to 15.65% of the mass was lost. The second stage can be attributed to the breakdown of the methylene linkages presented in most Novolac resins. Around 220 °C and up, transformation of ether bridges to methylene bridges occurs with simultaneous loss of formaldehyde. At this stage, thermal crosslinking occurs in most of Novolac resins. Figure 13 shows that up to 46.73% of mass was lost in the range of 215 °C to 350 °C. For the third stage of mass loss, 350 °C and above, the degradation showed is associated with the aromatic ring structure present. Beyond this mass loss, whatever organic residue is remaining will attribute to the remaining mass at the end. For the glass transition temperature of Epoxy resin A, multiple transitions can be seen and that can be attributed to the glass transitions of the individual component and not a specific T_g of a crosslinked polymer. T_g of this crosslinked polymer system should be between 160 to 180 °C. Both the mass loss and T_g are at higher temperatures than the proposed application for this sealant. These results indicate that Epoxy resin A can be used for low temperature applications safely.

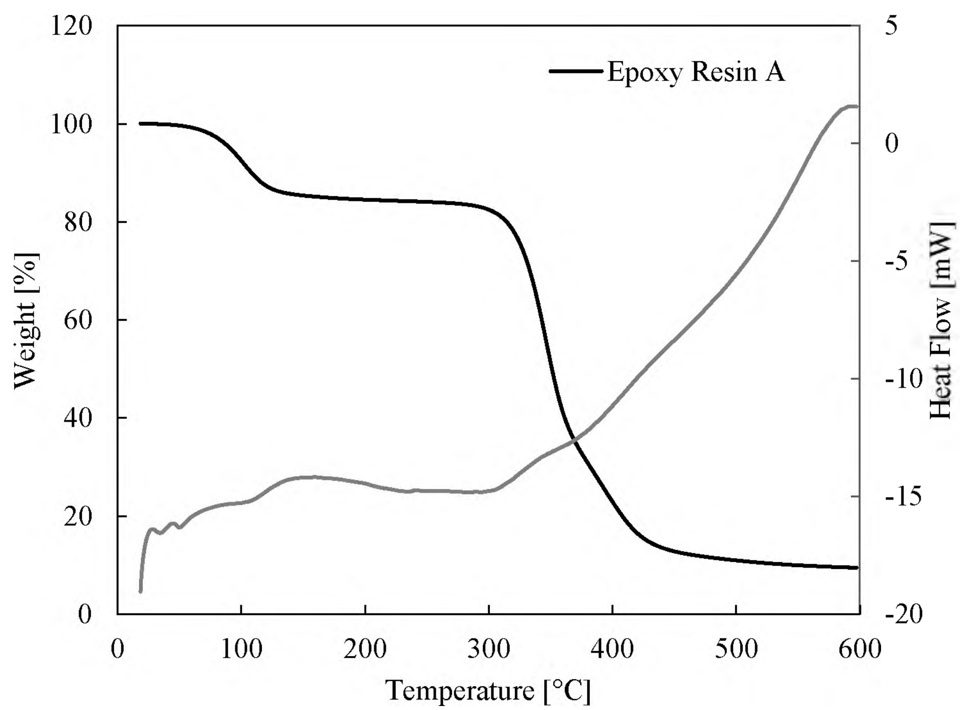

Figure 13. DSC results of Epoxy Resin A cured at room temperature.

For Epoxy resins B and C, it can be observed that there is only one stage of mass loss up to 94.38% for Epoxy resin B and up to 95.56% for Epoxy resin C. This is because these sealants consisted of pure resins and hardeners and even the diluent used was a reactive one, which contributed in getting a fully crosslinked polymer systems without any free or unreactive components.

The Tg of those two sealants were slightly higher than that of Epoxy resin A. It is important to mention that the glass transition temperature and the mass loss occurred at temperatures higher than the proposed application for these materials.

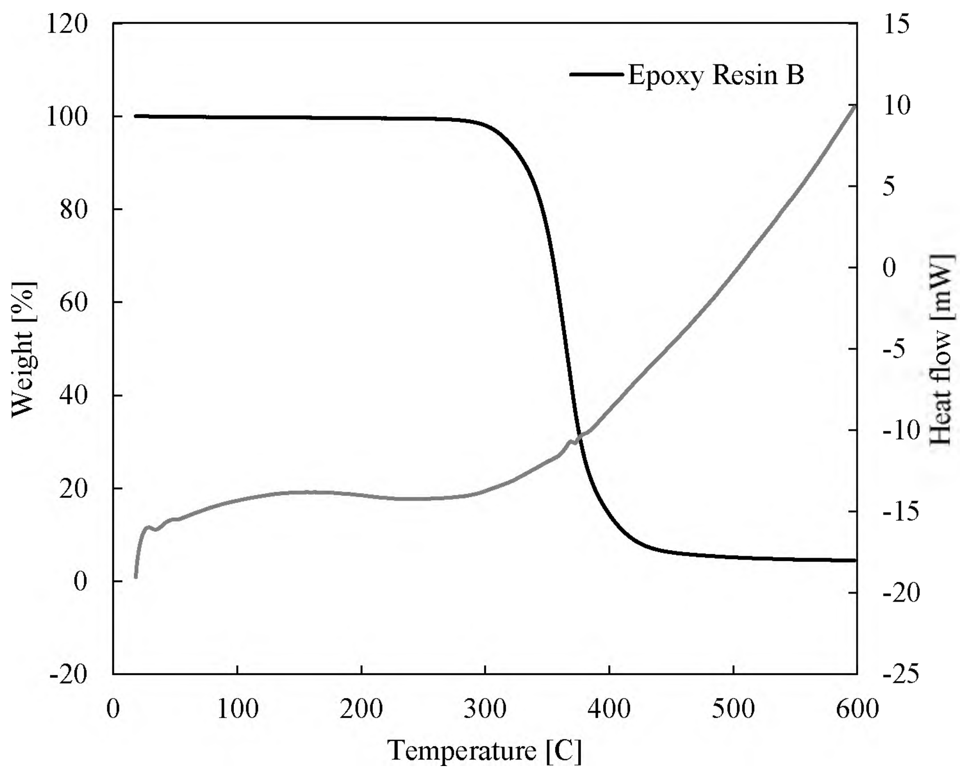

Figure 14. DSC results of Epoxy Resin B cured at 50 °C.

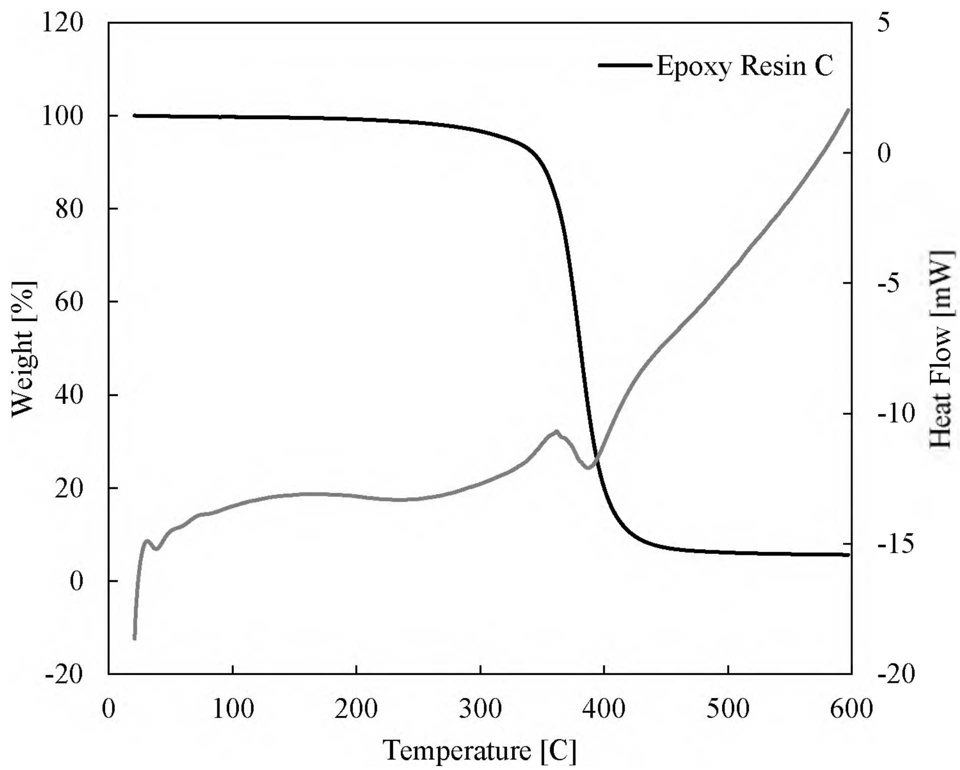

Figure 15. DSC results of Epoxy Resin C cured at 80 °C.

The effect of the curing temperature on the mass loss was also studied using Epoxy resin A as shown in Figure 16. Similar results were obtained for the mass loss in terms of the stages. However, increasing the curing temperature reduced the mass loss slightly in the first and second stages. The reduction was around 1 to 5%. This reduction can happen due to two reasons. The first one is the loss of some volatiles solvents during the curing of the samples before the measurements and the second one can be attributed to the additional induced crosslinking in the system during curing process as a function of temperature.

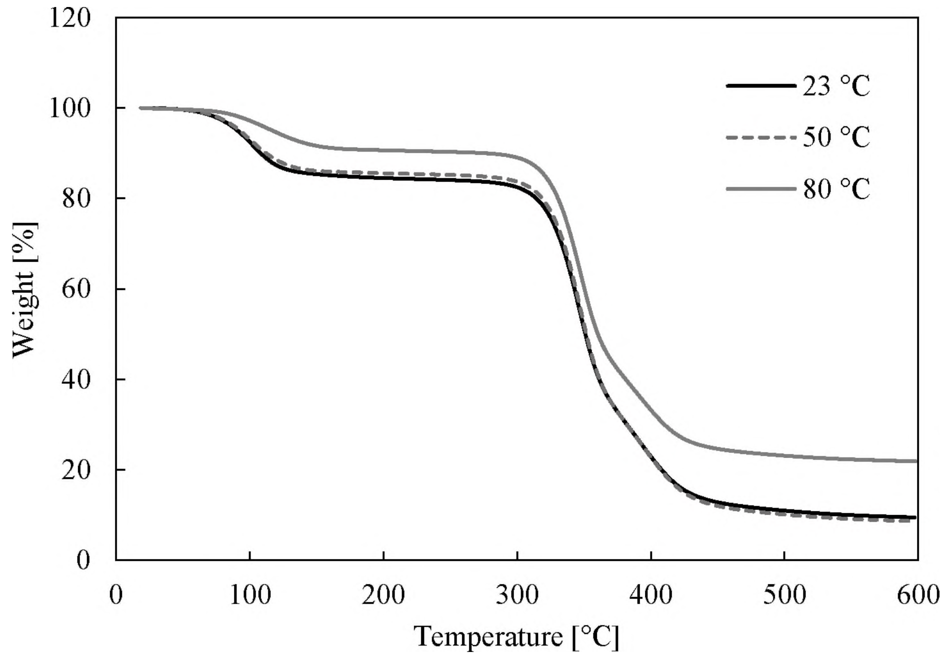

Figure 16. Effect of the temperature change on the mass loss of Epoxy resin A.

3.4. CHEMICAL RESISTANCE RESULTS

The chemical resistance measurements were conducted to evaluate the ability of the epoxy resin sealants to withstand downhole harsh fluids. Two sulfuric acid solutions were prepared for this measurement (98% and 50%). The 50% sulfuric acid was prepared by diluting the 98% sulfuric acid. To prepare 50% NaOH solution a conical flask filled with 500 ml of distilled water was placed in a magnetic stirrer then, 500 grams of NaOH pellets were added gradually. Then, distilled water was added until 1000 ml of 50% NaOH was obtained. The 10% NaOH was prepared in a similar manner. To prepare a 10% NaCl solution, a 100 gram of NaCl was mixed with a liter of distilled water and for the 36% NaCl, 360 grams were mixed with a liter of distilled water. Then, the epoxy resin and cement samples were immersed in the solutions for a total of three months. The weight change was recorded at several periods. Figure 17 shows the samples of Epoxy resin A and the cement immersed in the solutions.

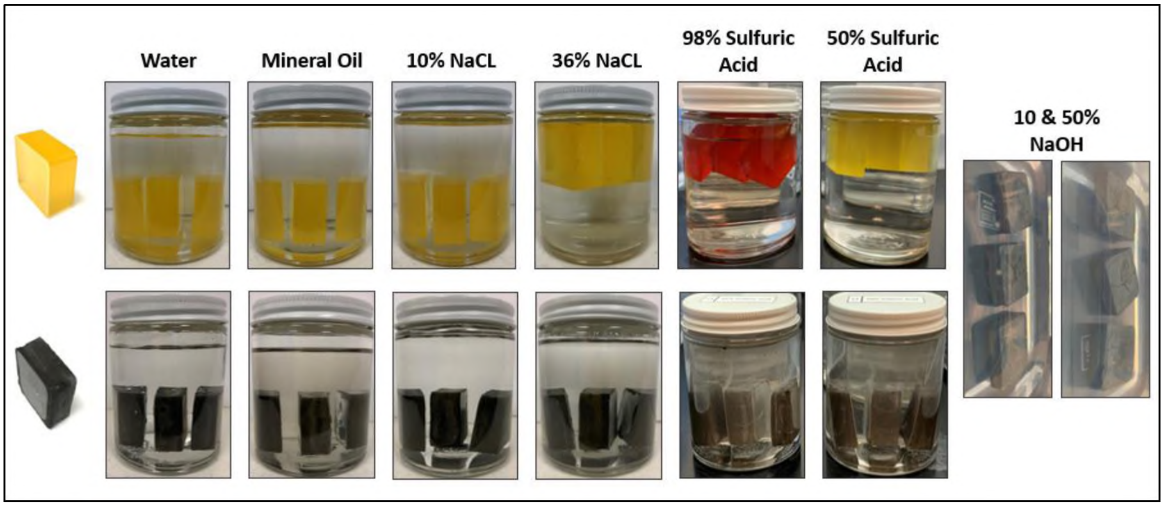

Figure 17. Epoxy resin A and cement samples immersed in the testing fluids.

All the samples experienced weight gain in the water, but the cement had the highest weight gain of around 1.81% after 3 months. All the samples had the same color through the 3 months testing period. Similar behavior was observed in the sodium hydroxide solutions and the sodium chloride solutions but with less weight gain than that of the water, which is due to the difference of the osmotic pressure of water as reported in the literature. All the sealants showed good resistance to alkaline solutions and that was because of the amine hardeners used in the formulations of those epoxy resins. For the mineral oil, the weight change was very small, and it seemed that there is no interaction between the sealants and the mineral oil. On the other hand, the sulfuric acid had major impacts on all the sealants excluding Epoxy resin A. Table 2 lists the results of the cement and Epoxy resin A. The cement in the 50% sulfuric acid lost 0.91% of its weight after 3 days and broke down into small pieces after 28 days. However, the epoxy resin sealants were able to maintain their shape and experienced a weight gain instead. The reason why the cement was showed behavior is that the cement is very alkaline in nature, which makes it susceptible to acid attack. During the hydration process of the cement, the products are calcium silicate hydrate and calcium hydroxide. The acid reacts with the calcium hydroxide resulting in highly soluble calcium salts (calcium sulfate) by-product. Calcium sulfate in turn causes degradation. The dissolution of the calcium hydroxide caused by the acid attack proceeds in two phases. (1) with calcium hydroxide in the cement paste and (2) the acid reaction with the calcium silicate hydrate. As one would expect the second phase will not begin until all calcium hydroxide is consumed. The dissolution of the calcium silicate hydrate, in the most advanced cases of acid attack, can cause severe structural damage to the cement. In the case of the reaction between the cement and the 50% sulfuric acid, low soluble salt was formed, which acted as partial inhibitor, blocked the passages through the cement, which in turn retarded the overall process. This can be seen in Figure 18 (The white powder covering the cement samples). For the epoxy resins tested here, especially B and C, the use of amine in the formulation increased the destruction of the samples in the 98% sulfuric acid. Overall, Epoxy resins cured with amine has good resistance to alkaline solutions and that cured with acid anhydride has good resistance to acid solutions. Figure 18 shows the effects of 50% and 98% sulfuric acid on the Epoxy resin A and the cement from day 1 to 3 months. The samples shown are before and after the immersing in the sulfuric acid. The results of Epoxy resin B and C are presented in Tables 3 and 4, respectively.

Table 2. The weight change of Epoxy resin A and cement as a function of time.

Reagent	% Weight Change as a Function of Time					
	Cement			Epoxy Resin A (LT)		
	3 days	28 days	3 months	3 days	28 days	3 months
Deionized Water	0.34	1.24	1.81	0.23	0.58	1.02
50% Sulfuric Acid	-0.91	D	D	0.22	0.44	0.57
98% Sulfuric Acid	-10.71	-12.66	-16.60	-1.11	-3.62	-3.98
10% NaOH	1.24	1.96	2.70	0.18	0.47	0.84
50% NaOH	0.76	0.67	2.16	0.05	0.10	0.29
10% NaCl	0.66	1.60	2.23	0.22	0.52	0.92
36% NaCl	1.95	2.54	2.84	0.13	0.34	0.60
Mineral Oil	0.37	0.75	0.92	0.02	0.05	0.05
scCO2	NT	0.17	NT	NT	4.07	NT
Notes	Cement Samples were cured for 7 days				D = Destroyed	
	Epoxy samples were cured for 24 hours					

Table 3. The weight change of Epoxy resin B and cement as a function of time.

Reagent	% Weight Change as a Function of Time					
	Cement			Epoxy Resin B (MT)		
	3 days	28 days	3 months	3 days	28 days	3 months
Deionized Water	0.34	1.24	1.81	0.18	0.59	1.00
50% Sulfuric Acid	-0.91	D	D	2.33	8.21	15.34
98% Sulfuric Acid	-10.71	-12.66	-16.60	-18.87	-46.64	D
10% NaOH	1.24	1.96	2.70	0.17	0.46	0.80
50% NaOH	0.76	0.67	2.16	0.05	0.18	0.39
10% NaCl	0.66	1.60	2.23	0.15	0.48	0.84
36% NaCl	1.95	2.54	2.84	0.11	0.32	0.55
Mineral Oil	0.37	0.75	0.92	0.03	0.06	0.07
scCO2	NT	0.17	NT	NT	5.38	NT
Notes	Cement Samples were cured for 7 days				D = Destroyed	
	Epoxy samples were cured for 24 hours					

Table 4. The weight change of Epoxy resin C and cement as a function of time.

Reagent	% Weight Change as a Function of Time					
	Cement			Epoxy Resin C (HT)		
	3 days	28 days	3 months	3 days	28 days	3 months
Deionized Water	0.34	1.24	1.81	0.10	0.32	0.57
50% Sulfuric Acid	-0.91	D	D	8.86	34.17	62.20
98% Sulfuric Acid	-10.71	-12.66	-16.60	-19.68	-38.51	D
10% NaOH	1.24	1.96	2.70	0.09	0.28	0.50
50% NaOH	0.76	0.67	2.16	0.05	0.15	0.33
10% NaCl	0.66	1.60	2.23	0.10	0.31	0.57
36% NaCl	1.95	2.54	2.84	0.07	0.22	0.39
Mineral Oil	0.37	0.75	0.92	0.03	0.07	0.15
scCO2	NT	0.17	NT	NT	2.78	NT
Notes	Cement Samples were cured for 7 days				D = Destroyed	
	Epoxy samples were cured for 24 hours					

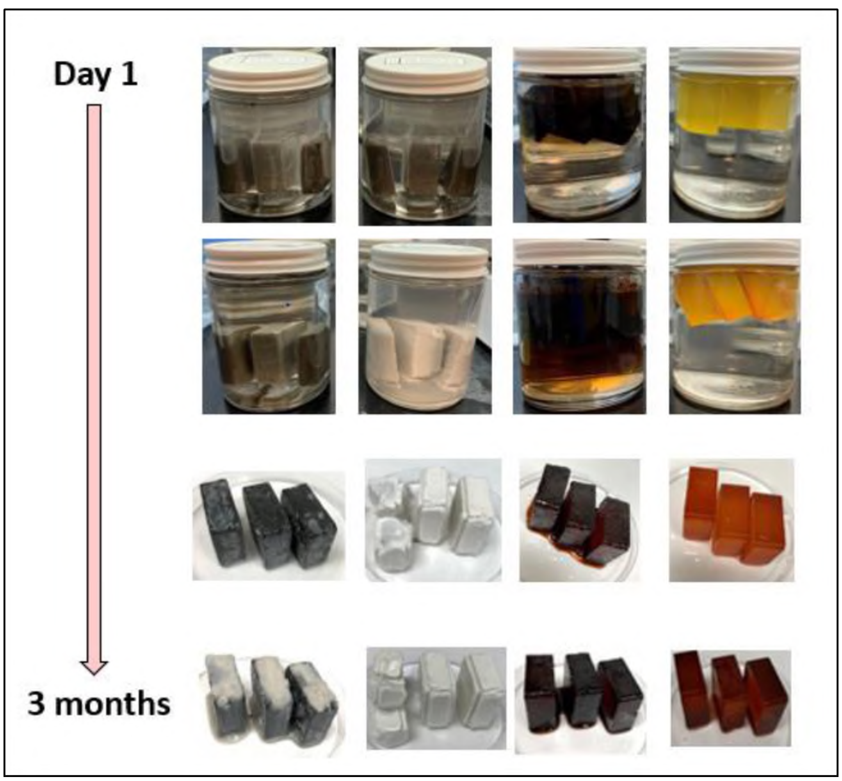

Figure 18. Pictures of the effect of the acid on the sealant compared to the cement.

3.5. INJECTIVITY AND PLUGGING PERFORMANCE RESULTS

Epoxy resin C was injected into three void sizes, consisting of two-foot tubes with inner diameters of 0.8763 mm, 1.753 mm, and 4.572 mm. The sealant injection pressure was monitored and recorded. The injectivity of the sealant was calculated based on the flow rate used and reaching stable pressure. Figures 19 shows the low injection pressure required to force the epoxy into the 1.753, and 4.572 mm voids. The injectivities of the epoxy resin at a flow rate of 1 ml/min were 0.27 and 10.0 ml/psi*min. However, the injectivity reduced to approximately 0.025 ml/psi*min when 0.8763 mm void was used, which is due to the high viscosity of the epoxy. This viscosity can be altered using diluents, reactive materials that can reduce the viscosity with minimum effects on mechanical properties, as reported in the literature.

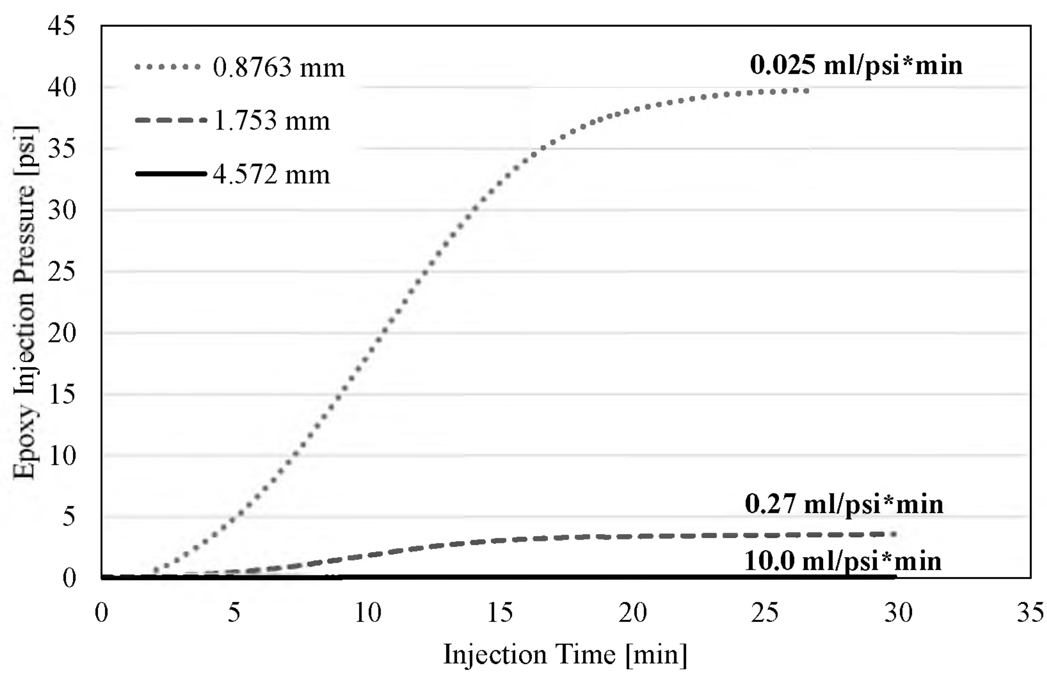

Figure 19. Injectivity of Epoxy resin C (a) 1.753, and 4.572 mm voids, and (b) 0.8763 mm void.

Overall, the capability of the epoxy resin to penetrate these voids with very low-pressure requirement promote such materials for wellbore integrity applications over conventional Portland cement. The estimated injectivity of the epoxy resin here is almost ten times higher than the injectivity of the cement using the same void sizes as addressed by Alkhamis et al., (2020). The same work also demonstrated that the cement particles tend to bridge and require higher pressures and different techniques to penetrate the tubes.

For the plugging performance measurements, the channel length and diameter were measured and the permeability of the channel before the treatment was estimated using Equation 3 (see section 2). Then, the Epoxy resins were placed in the channels and left to cure at the desired temperature. For Epoxy resin A, which was cured at room temperature, the permeability of the channel was around 1196345.4 Darcy. Then, the epoxy was placed in the channel and after 24 hours the core was placed in the core holder shown in Figure 1. After that water was injected at a flow rate of 0.1 ml/min. The pressure started to increase gradually indicating no leakage through the cement core. After approximately 3 hours and 46 minutes and at a pressure of 2140.1 psi, the first drop of water showed up at the outlet of the core holder, but the injection pressure kept increasing. At 2200 psi the injection was switched to constant pressure instead of constant flow rate due to equipment limitation. At the 2200 psi constant pressure the flow rate of the water at the inlet was recorded to be around 0.0113 ml/min. Using Equation 2 (Darcy Equation) the permeability at 2200.1 psi was estimated to be 0.026 mD as shown in Figure 20. Since now we have initiated a new crack in the cement core, it was interesting to measure the reduction in permeability at different pressures so, the injection started at 500 psi constant pressure and there was no water at the outlet of the core indicating that the crack is too small that the 500 pai is not enough to force the water into the crack. Then, the pressure was increased to 1000, 1500, and 2000 psi. The corresponding permeabilities were 0.025, 0.0185, and 0.017 mD, respectively (see Figure 20). For the CO2 experiments, cores with similar artificial channel were used. The epoxy was first placed in the cores and left to cure for 24 hours. Then, the CO_2 was injected. At the beginning the pressure at the inlet was increasing while the pressure at the outlet was zero. When the pressure reached around 650 psi, the pressure at the outlet started to increase indicating that the gas initiated a flow path in the core.

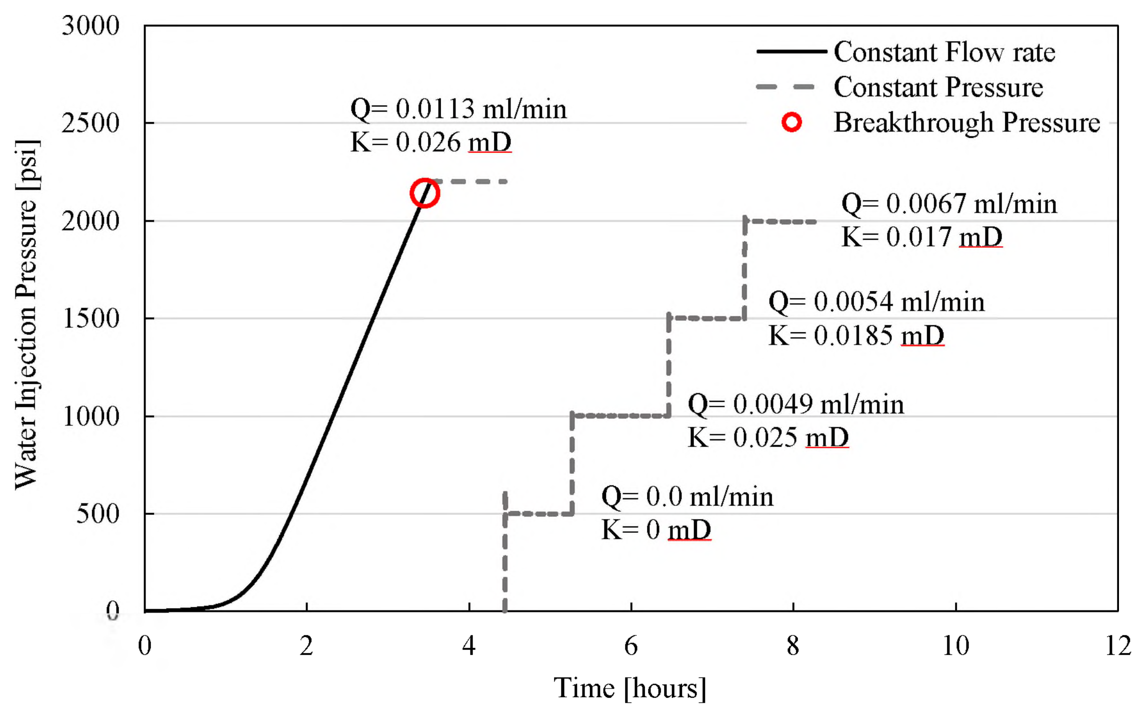

Figure 20. Plugging performance of Epoxy resin A against water.

For Epoxy resin B, the injection started at 0.1 ml/min constant flow rate until the water breakthrough, which was at 2055.5 psi. In this case, there was no reason to switch to constant pressure injection as the pressure drop after the water breakthrough was high as the pressure dropped from 2055.1 psi to around 50.8 psi. The permeability after treatment was estimated to be in the range of 9.6 to 11.3 mD at flow rates ranges between 0.1 ml/min to 1.0 ml/min as described in Figure 21. These results indicate that Epoxy resin B had the lowest permeability reduction of the three epoxy resins tested. For the CO_2 experiments, similarly Epoxy resin B had lowest breakthrough pressure of approximately 400 psi.

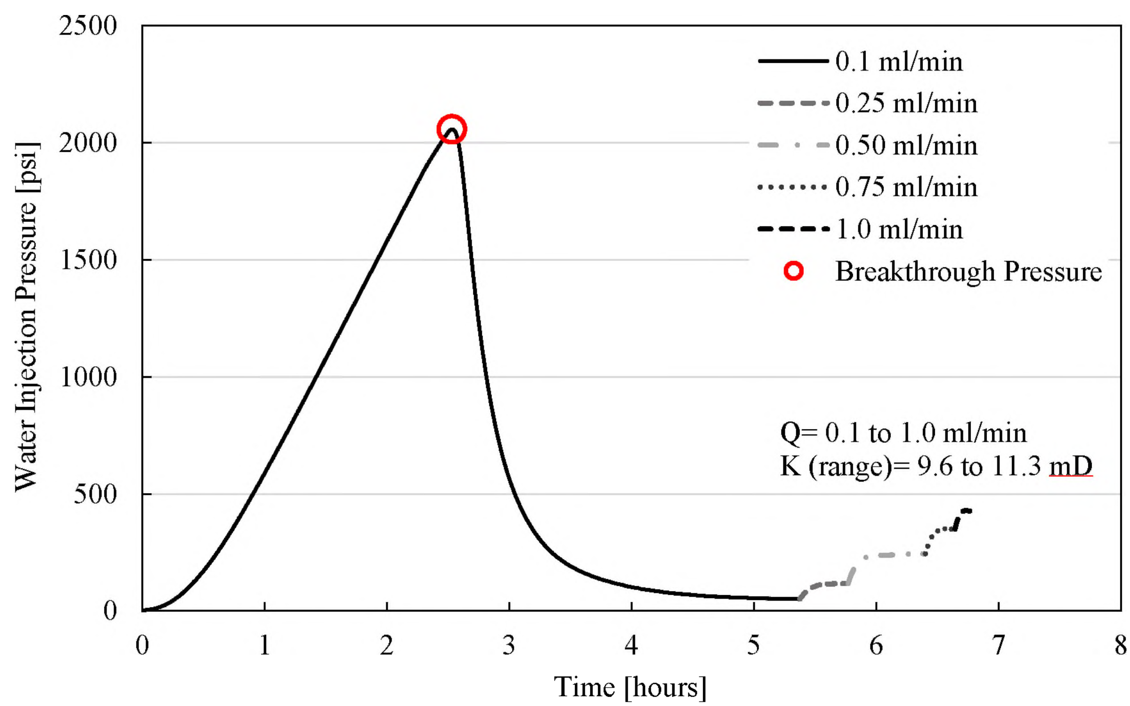

Figure 21. Plugging performance of Epoxy resin B against water.

For Epoxy resin C, the injection started at 0.1 ml/min constant flow rate and pressure kept increasing to a maximum of 1747.5 psi with an estimated breakthrough pressure of 1207.4 psi. The reduction in the permeability after initiating a new crack in the core were in a range of 0.078 to 0.255 mD at several constant pressures. Figure 22 shows the flow rate and permeability at each pressure. Overall, the three sealants were able to withstand a differential pressure higher than 1000 psi. In the case of sealants A and C, the permeability reduction was high even after the breakthrough. Epoxy resin B was able to withstand a differential pressure of 2000 psi but the reduction in permeability after the breakthrough was relatively low. For the CO_2 experiments, Epoxy resin C showed the highest resistance to the gas as the breakthrough pressure was around 775 psi.

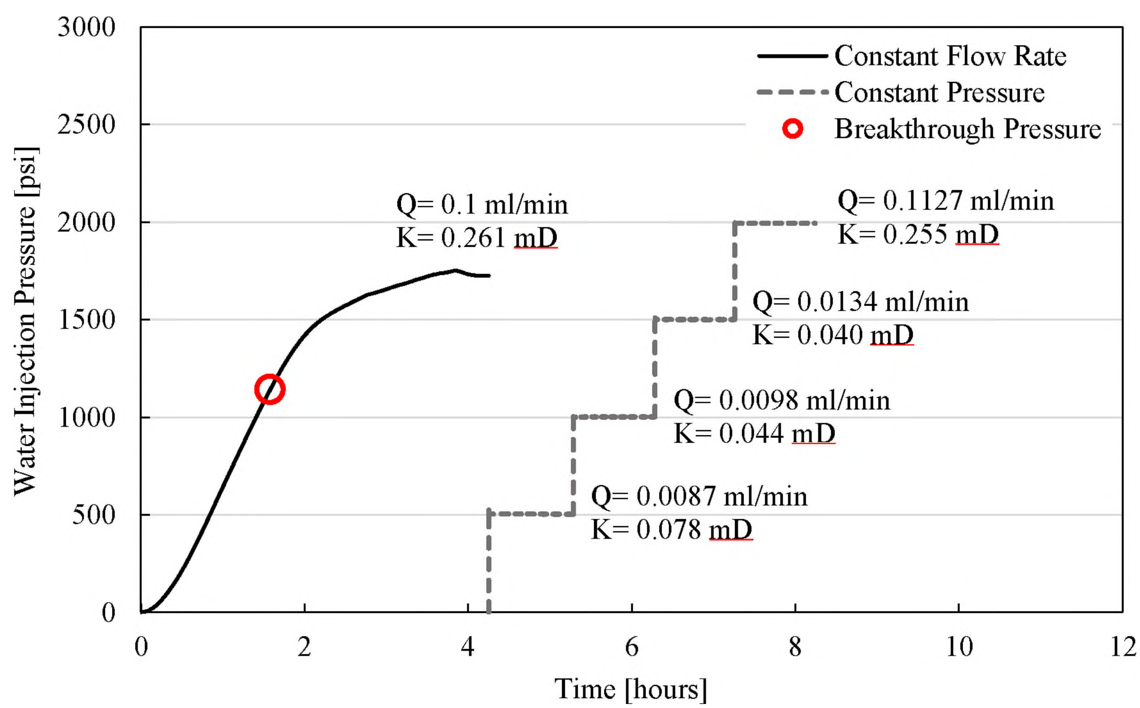

Figure 22. Plugging performance of Epoxy resin C against water.

For all the three epoxy resin sealants, the measurements were conducted three times and the results were close. The results presented here are for the lowest values obtained to avoid overestimating the plugging performance of the sealants.

3.6. MECHANICAL PROPERTIES RESULTS

In this study, two mechanical properties were investigated, the compressive and the tensile strength. Epoxy resin A showed high compressive strength after 24 hours curing at room temperature. The compressive strength was measured to be approximately 10111 psi as shown in Table 5. The elastic modulus was around 2.16 Gpa, which is low value compared to cements, which have elastic modulus in the range of 10 to 30 Gpa. This shows that the Epoxy resin is more flexible than the cement as the stiffness of the cement yield to radial cracks in the sheath, which creates pathways for the formations fluids to migrate. Figure 23 shows the stress vs strain of Epoxy resin A sample 2. The tensile strength of Epoxy resin A was around 3543 psi as shown in Table 6, which was higher than that of the cement as the cement develops tensile strength less than 500 psi in 24 hours.

Table 5. The compressive strength results of Epoxy resin A.

Epoxy resin A	Avg Diameter [mm]	Avg Length [mm]	Elastic modulus [Gpa]	Poisson's ratio	Compressive Strength [Mpa]	Compressive Strength [psi]
Sample 1	50.46	96.2	2.5	0.37	72.613	10531.62
Sample 2	50.59	98.34	2.2	0.33	69.251	10044.008
Sample 3	50.67	97.35	1.8	0.36	67.283	9758.57
Average	-	-	2.16	0.35	69.715	10111.31

Table 6. The tensile strength results of Epoxy resin A.

Epoxy resin A	Avg Diameter [mm]	Avg Thickness [mm]	T/D Ratio [0.2-0.75]	Max Force [kN]	Tensile Strength [Mpa]	Tensile Strength [psi]
Sample 1	50.59	25.59	0.51	56.2524	27.67	4013.2
Sample 2	50.40	25.51	0.51	45.5511	22.56	3272.1
Sample 3	50.47	27.43	0.54	49.6204	22.81	3308.3
Average	-	-	-	-	24.43	3543.3

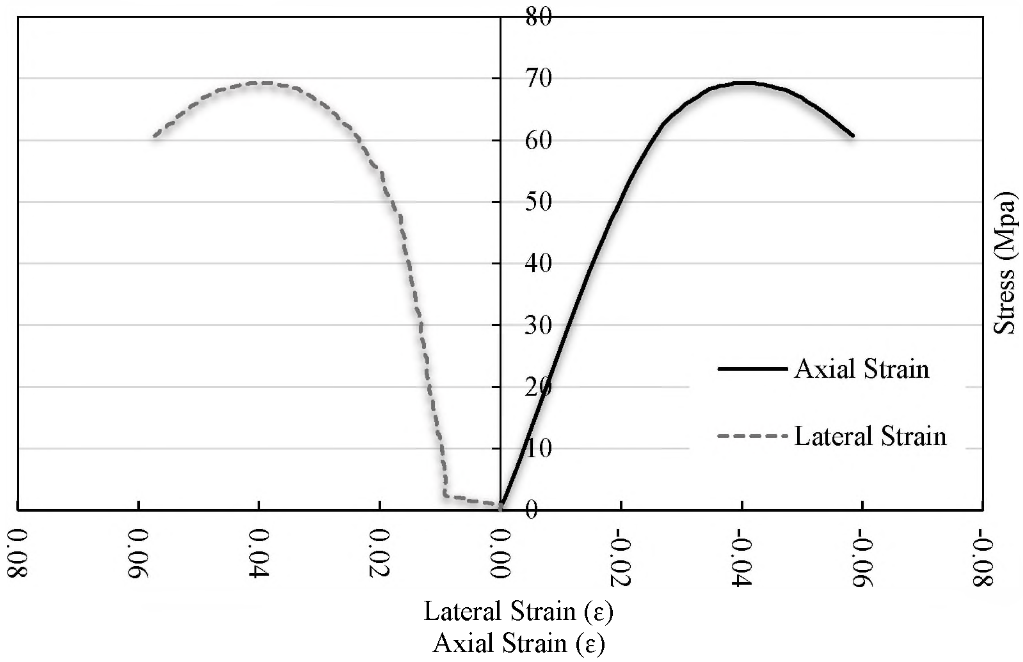

Figure 23. The axial and lateral strain of Epoxy resin A.

For Epoxy resin B, the compressive strength was found to be around 10409.7 psi as shown in Table 7. The tensile strength was around 2509 psi, which is relatively lower than that of Epoxy resin A, but higher than the tensile strength of cement. The results are listed in Table 8.

Table 7. The compressive strength results of Epoxy resin B.

Epoxy resin B	Avg Length [mm]	Avg Width [mm]	Elastic modulus [Gpa]	Poisson's ratio	Compressive Strength [Mpa]	Compressive Strength [psi]
Sample 1	49.89	51.52	2.54	0.361	72.034	10447.64
Sample 2	50.12	49.19	2.61	0.314	70.447	10217.47
Sample 3	50.15	48.02	2.46	0.321	72.836	10563.97
Average	-	-	2.54	0.332	71.177	10409.69

Table 8. The tensile strength results of Epoxy resin B.

Epoxy resin B	Avg Diameter [mm]	Avg Thickness [mm]	T/D Ratio [0.2-0.75]	Max Force [kN]	Tensile Strength [Mpa]	Tensile Strength [psi]
Sample 1	50.60	25.79	0.51	21.8464	10.66	1546.1
Sample 2	50.63	25.74	0.51	36.8466	18.00	2610.7
Sample 3	50.39	27.28	0.50	33.2188	16.60	2407.63
Average	-	-	-	-	17.3	2509.1

For Epoxy resin C, the compressive strength was found to be around 15101 psi as shown in Table 9. The tensile strength was around 2988 psi, which is relatively lower than that of Epoxy resin A, but higher than the tensile strength of cement and Epoxy resin B. The results are listed in Table 10. The use of aromatic hardener with bisphenol A as base resin resulted in the highest compressive strength. All the three sealants developed flexibility higher than the cement. This flexibility may play a major role in the wellbore, avoiding radial cracks that are created in the cement sheath due to its stiffness. These results can be used as a base for future work where these types of sealants can be added to the cement slurry in primary cementing. The use of such additive may enhance the mechanical properties of the cement.

Table 9. The compressive strength results of Epoxy resin C.

Epoxy resin C	Avg Length [mm]	Avg Width [mm]	Elastic modulus [Gpa]	Poisson's ratio	Compressive Strength [Mpa]	Compressive Strength [psi]
Sample 1	50.79	48.09	2.73	0.328	113.508	16462.94
Sample 2	51.12	50.97	2.87	0.372	98.322	14260.4
Sample 3	51.73	51.29	2.67	0.317	100.529	14580.5
Average	-	-	2.75	0.339	104.11	15101.28

Table 10. The tensile strength results of Epoxy resin C.

Epoxy resin C	Avg Diameter [mm]	Avg Thickness [mm]	T/D Ratio [0.2-0.75]	Max Force [kN]	Tensile Strength [Mpa]	Tensile Strength [psi]
Sample 1	55.62	23.88	0.43	45.5429	21.83	3166.2
Sample 2	57.40	25.41	0.44	41.1882	17.98	2607.8
Sample 3	55.46	22.83	0.41	43.5883	21.92	3179.2
Average	-	-	-	-	20.6	2987.78

4. CONCLUSIONS

This paper investigated comprehensively the applicability of three thermoset materials to be reliable alternatives to Portland cement in wellbore integrity applications. The main conclusions of this work are summarized below:

- The viscosities of the sealants were highly dependent on temperature as increasing the temperature reduces the viscosity significantly and at the same time reduces the curing time of the sealants. The sealants behaved like Newtonian fluids and the shear rate had no significant impact on viscosity.
- Epoxy resin C showed high injectivity and ability to penetrate small gaps when compared to conventional Portland cement.
- The three sealants had thermal degradation temperatures and Tg higher than the proposed applications.
- The three sealants showed high capability of plugging cement channels and reduces the permeability to zero. In addition, the permeability reduction after initiating new cracks in the cement was high. The sealants were able to stop water leakage and CO_2 leakage.
- This work covered the rheology and plugging performance of the sealants but the workability of the sealants (thickening time) needs to be investigated in the future.
- All the sealants showed great ability to withstand chemicals, but Epoxy resin A was the only sealants that was able to withstand the 98% sulfuric acid. Thus, epoxy resins formulated similar to Epoxy resin A can be implemented in applications where sulfuric acid is present.
- The results of this study show that Novolac based resins are suitable for low temperature applications as the sealant tested here was able to develop high strength and was able to plug cement channels and reduce the permeability. In addition, the use of aliphatic curing agent for moderate temperature applications require further study as the material reacted volatilely in presence of heat. Lastly,

the aromatic curing agent was the most suitable for application where temperatures are high as Epoxy resin C was able to plug the cement gaps and develop high strengths in short time.

NOMENCLATURE

Gram per milliliter	*gm/ml*
Pound per gallon	*lbm/gal*
Radian per second	*rad/s*
Dynamic Shear Rheometer	*DSR*
Degree Celsius	*°C*
Degree Celsius per minute	*°C/min*
Centipoise	*cp*
Pascal	*pa*
Differential scanning calorimeter	*DSC*
Glass transition temperature	T_g
Millidarcy	*mD*
Pound per square inch	*psi*
Milliliter per minute	*ml/min*

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V. EVALUATION OF AN ULTRA-HIGH-PERFORMANCE EPOXY RESIN SEALANT FOR WELLBORE INTEGRITY APPLICATIONS

ABSTRACT

After drilling each section of a well, cement is placed in the annulus of the casing and the formation. The cement integrity must be ensured during the life cycle of the well or after abandonment. If for any reason, the cement lost its integrity, the consequences could be severe for personnel, equipment, and the environment. When the cement fail, leakages may occur through the cement pathways and sealant materials are used to plug these pathways. This study investigates a temperature activated epoxy resin sealant to evaluate the potential use of this sealant as an alternative to Portland cement in oil and gas wells. This study focuses on analyzing the rheological behavior of the sealant, the effect of temperature on the rheology and the curing time of the sealant, the penetrability of the sealant into small voids, and the blocking efficiency of the sealant. Experimental tests were conducted to evaluate the epoxy resin sealant including rheological measurements, density, injectivity, blocking efficiency, and mechanical properties. The findings of this study show that this sealant has low viscosity and Newtonian rheological behavior, low density as low as water, high injectivity and penetrability even in small gaps, ability to resist differential pressure higher 1000 psi, and extremely high compressive strength. This work demonstrates that epoxy resin sealant can be used effectively and safely in sealing cement voids.

1. INTRODUCTION

Gas leakage, gas migration, gas seepage, and many other terms are synonyms to a problem that exists in hydrocarbon wells. This problem occurs due to a failure to achieve full zonal isolation and may result in high maintenance costs and threats to surrounding communities and the environment. Gas migration can be flow between zones, flow into shallow sands, and/or flow to the surface. Flow to the surface would occur within minutes or hours after completing the well while flowing between zones may not be noticed for weeks or even months. Many studies and field operations have been conducted throughout the history and well-integrity failures are still occurring through the wells' life from drilling to plug and abandonment (Santos, 2015). Gas migration is reported through pressure buildup, referred to as "sustained casing pressure (SCP)," and can be a significant safety hazard. The Mineral Management Service of the United States reported in 2003 that SCP affects more than 8,000 wells in the Gulf of Mexico (Rusch, 2004). The Norwegian standard defines well integrity as "application of technical, operational, and organizational solutions, to reduce risk of uncontrolled release of formation fluids throughout the life cycle of a well" (Norsok, 2013). For these reasons, primary cement designs must be optimized in such a way that accomplish short and long-term zonal isolation (Sauer, 1987).

Portland cement have been used mostly for primary cementing after drilling, for secondary cementing in remedial operations, and for plugging and abandonment. When the cement is placed in the annulus as a barrier between the casing and the formation, it must be able to protect and support the casing, and to isolate production zones. The cement must prevent the wellbore fluids from migrating in an annular flow path so as to allow the well to be utilized without any control problems (Alkhamis and Imqam, 2018). Mature wells need to be permanently plugged and abandoned. Plugging and abandonment operations usually consist of placing several cement plugs in the wellbore to isolate the reservoir and other fluids bearing formations. It is essential to ensure that no leaks are developed after abandonment. During the life cycle of a well, the cement is exposed to pressure and temperature variations, high mechanical loads, and corrosive fluids attacks. These harsh conditions may compromise the cement integrity and the cement may fail in delivering full zonal isolation (Ahdaya and Imqam, 2019). If this happened during the active life of the well, remedial jobs must be performed to restore the integrity of the well. Squeezing cement is usually the method of repair (Shryock and Slagle 1968). In which cement is forced in the cement voids and/or between the cement and its surroundings. However, this method is limited by the size of the voids (Jones et al., 2014) as the cement slurry consists of liquid and fine particles. The particles may start bridging in narrow voids. Micro cement may be used as an alternative but it can be limited in penetrating a gap of less than 300 microns in size (Wasnik et al., 2005) in addition to the thickening time of the micro cement that gets affected by contamination (Dahlem et al., 2017). For these reasons, a solids-free material should be designed to overcome the drawbacks of using cement for remedial operations. One of the alternatives is the use of polymer resins. This sealant material can also be used effectively in plug and abandonment operations.

In the petroleum industry, epoxy resins have been used for sand consolidation, which strengthen the formation by binding the grains of the unconsolidated formation together at their contact points (Marfo et al., 2015). Consolidated formations have higher elasticity after hardening than that of the rocks glued to (Milkowski and Szwedzicki, 1973). Epoxy resin was also used in enhance oil recovery applications (Hakiki et al., 2015; Tiwari et al., 2017). It can also be used as a lost circulation material (Knudsen et al., 2014) and for fluid losses mitigation (Teixeira et al., 2014).

Polymer resin systems consists of base resin and curing agent, also known as hardener. Polymer resins can be defined as “free flowing polymer solutions that can be irreversibly set to hard, rigid solids.” (Morris et al., 2012). Many studies have been conducted to investigate the properties of polymer resins. These properties includes the tunable rheological behavior of the material (Alsaihati et al., 2017) and the fact that the material is solids-free, which allows it to penetrate very small gaps (Todd et al., 2018). The flexibility in density, which is an advantage in case of narrow fracture pressure gradient, and the tunable setting time that was mentioned by Sanabria et al., 2016, and the resistance to contamination studied by Perez et al., 2017. In addition, to these pre-curing properties, cured sealant provides high mechanical strength (Elyas et al., 2018), can resists significant strain (Khanna et al., 2018), and develops good bonding (Jimenez et al., 2016; Genedy et al., 2017). Also, during the polymerization reaction of epoxy resin, no by-products are formed (Muecke, 1974). Epoxy resins provides high stability and durability at high temperatures, indicating reliability in the long term (Bertram et al., 2018). Based on the literature, no scientific laboratory work has been conducted to study the injectivity of epoxy through small voids as well as no work has investigated the plugging efficiency of this material.

This study presents in detail the preparation of an ultra-high-performance epoxy-resin sealant for wellbore integrity applications. The paper also evaluates the injectivity and blocking efficiency of the sealant, which to the author's best knowledge are two tests that have not been conducted for epoxy-resin in the petroleum industry. In addition, the rheological behavior under different temperatures, curing kinetics, and compressive strength have been evaluated.

2. BACKGROUND AND EXISTING TECHNOLOGY

In this section of the paper, a summary of seven successful field jobs are presented. This summary of field case studies prove that epoxy resin sealants work effectively. Table 1 lists seven cases where the wellbore integrity were compromised and remedial job was needed. These cases occurred in six different countries on both offshore and onshore locations. In some of these cases, several attempts to remediate the failure were conducted using conventional technologies but failed to successfully solve the problem. Epoxy resin sealants were the solution for all of these cases and the remediation jobs were conducted successfully. Like the case of Brazil offshore well (Perez et al., 2017), where casing leakage was observed in the 9-5/8" x 13 3/8" casing. The investigation tests concluded that this leakage is due to cement channels behind the casing and the solution was pumping 15 barrel of epoxy resin at 5 bbl/min rate to seal off the leakage as shown in Table 2, which lists the properties of the epoxy resin used in these field jobs.

Table 1. A summary of seven wellbore failures.

Paper	Locatio n	Well Type	Total Depth (ft)	BHST (F)	BHCT (F)	Well problem	Location of failure
(Alsaihat i et al., 2017)	Saudi Arabia	Gas Well (XA)	12450	210	178	Casing-casing annulus pressure of 4614 psi, Tubing-casing annulus pressure of 141 psi	9-5/8x13 3/8"
(Khanna et al., 2018)	India	Well (RXY)	8300.5	182	167	Channels in a cement packer (0.3875")	7" liner
(Davis, 2017)	Gulf of Mexico	Offsho re Well	20413	-	-	Obstruction in the casing requires special treatment in-tubing	7-3/4"x5-1/2"
(Perez et al., 2017)	Brazil	Offsho re Well	10823. 5	137	108	Casing Leakage due to channels in the cement	9-5/8"x13 3/8"
(Pardeshi et al., 2017)	New Zealand	Pilot Hole	10285. 5	160	120	Pilot hole needs to be permanently plugged	12-1/4"
(Sanabria et al., 2016)	Saudi Arabia	UTNM N Well	7335	175	-	Casing Damage/Leakage	7" Liner
(Ali et al., 2016)	Egypt	Produc tion Well	11224	-	188	Leak with 1 bbl/min rate	4.5" Liner

Table 2. The properties of the sealant used in the field jobs.

Paper	24 hr Compressive Strength (PSI)	Viscosity @ 300 rpm	Gelling time (Hrs)	Density (ppg)	Volume (bbl)	Rate (bbl/min)
(Alsaihati et al., 2017)	1500 @ 190 F	34 @ 178 F	8 @ 178 F	9.11	20	4
(Khanna et al., 2018)	12455 @ 182 F	50 @ 60 F	-	9.17	15.73	4.5
(Davis, 2017)	-	-	-	14	35	1-4
(Perez et al., 2017)	8750 @ 137 F	-	-	9.28	15	5
(Pardeshi et al., 2017)	7400 @ 185 F	230 @ 120 F 300 @ 80 F	-	9.3	27	3
(Sanabria et al., 2016)	-	-	3 @ 175 F	-	20	0.3
(Ali et al., 2016)	> 6000 @ 188 F	170 @ 188 F > 300 @ 80 F	2.5 @ 188 F	-	12	-

Although, the epoxy resin sealants have proved their capability to seal off cement failures, one might ask:

- Why do we need to use epoxy resin instead of other technologies?
- How about the cost of this type of materials?

To answer the first question, some of the drawbacks of other sealing materials and technologies will be highlighted. Starting by conventional Portland cement, which is the first choice of squeezing materials. Portland cement has the advantage of low cost. However, Portland cement is highly susceptible to bridge when squeezing through tight restrictions due to the large size of the cement particles (Abdulfarraj & Imqam, 2019). In addition, Portland cement properties such as thickening time get affected by brine. Micro fine cement has similar mechanical properties to Portland cement, but its smaller particle size reduces the risk of bridging across tight restrictions (Dahlem et al., 2017). Micro fine cement does not have problem with bridging but still get affected with contamination.

One other technology is the use of casing patches or scab liners, which are common solutions to recover the integrity of casings. Casing patches work well for short distances, but the major drawback is the reduction in the diameter of the casing. In addition, any restrictions in the wells must be removed before running the patches. If the reason behind the sustained casing pressure is micro-annuli or channels, casing patches cannot seal the leakage (Todd et al., 2018).

To answer the second question, the importance of planning the remedial job and the importance of sealing the leakages will be pointed out. The cost of the remedial job is directly related to waiting time (economic rig time) that is why planning the remedial job is the most important aspect. The cost also depends on the zone; the zone determines the amount of shut off material needed. In addition, the equipment needed to perform remediation job is important (pump truck, plugs, spacers, and packers) (Sufall, 1960). The remedial job when planned and executed carefully, the outcome is success.

Successful remedial job means production safely and effectively. Safely for both personnel and the environment.

3. EXPERIMENTAL MATERIALS

3.1. CLASS-H CEMENT

American Petroleum Institute (API) Class-H oil well cement was used in this study to prepare cement cores. The specific gravity of the cement was measured, using gas Pycnometer, to be 3.18. The chemical composition of Class-H cement, which was obtained utilizing X-ray fluorescence spectroscopy (XRF) is listed in Table 3.

Table 3. The chemical composition of class-H cement.

Comp.	CaO	SiO_2	Fe_2O_3	Al_2O_3	SO_3	MgO	K_2O	SrO	TiO_2	Other
Wt %	65.72	20.36	6.19	3.17	2.26	1.32	0.43	0.21	0.16	0.18

3.2. CEMENT PASTE PREPARATION

All cement slurries mixed in this study had a water/cement ratio (WCR) of 0.38 in accordance to API specification 10A (API, 2010). The mixing was at room temperature in a two-speed bottom-drive laboratory blender. Dry cement was added to the water in the blender at a uniform rate while mixing at low speed for around 15 seconds. Then, the blender was covered and the mixing continued for 35 seconds at high speed (API RP 10B-2 2013).

3.3. DILUTED RESIN

The resin used in this study is Bisphenol A diglycidyl ether resin (BADGE), which is one of the most widely used epoxy resins. Figure 1 shows the chemical structure of BADGE. It was selected to be the base polymer in the formulation of this temperature activated epoxy resin due to its ability to produce a sealant with very good mechanical, adhesive, and chemical resistance when cured with appropriate curing agent.

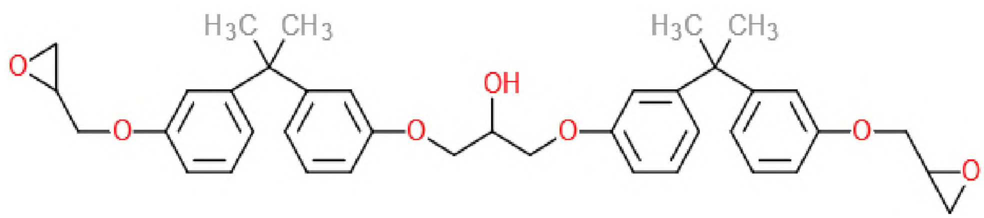

Figure 1. The chemical structure of Bisphenol A diglycidyl ether.

Since the base resin has a very high viscosity between 11000 – 14000 cp, it was essential to dilute the resin using a diluent. Cyclohexane dimethanol diglycidyl ether (CHDGE) was used as a diluent. This reactive diluent is a difunctional modifier that gives moderate viscosity reduction with minimum loss in properties. The chemical structure of CHDGE is shown in Figure 2.

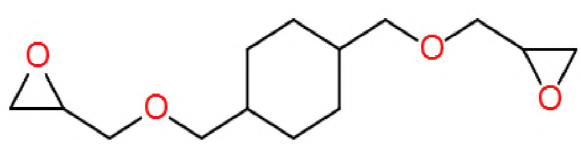

Figure 2. The chemical structure of cyclohexane dimethanol diglycidyl ether.

3.4. CURING AGENT

Diethyltoluenediamine (DETDA), which is an aromatic amine, was used as a curing agent for the diluted resin. This curing agent is less reactive and require longer time and higher temperature to cure. The decrease in the reactivity can be due to steric hindrance by alkyl groups adjacent to the amino group (Dewprashad and Eisenbraun, 1994). The low reactivity is important to minimize the rate of heat released (Pardeshi et al., 2016). Figure 3 shows the chemical structure of DETDA.

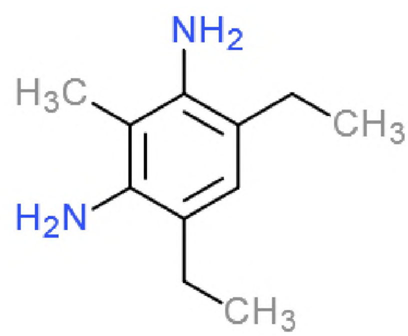

Figure 3. The chemical structure of diethyltoluenediamine.

3.5. EPOXY RESIN PREPARATION

To prepare the epoxy resin mixture, an amount of the resin and the reactive diluent were weighted and mixed at room temperature by hand and/or using a magnetic stirrer until a homogenous fluid was obtained. Then, a calculated amount of curing agent was added to the blend and mixed at low shear until the mixture. For the elevated temperature experiments, the mixture was heated until the desired temperature while the stirring continued using the magnetic stirrer.

4. EXPERIMENTAL METHODOLOGY

In this study, the rheological behavior of the sealant was studied first including measuring the viscosity of the undiluted resin, the diluted resin, and the final mixture (resin and curing agent). Then, the curing time of the sealant was measured. Lastly, the injectivity, blocking efficiency, and compressive strength of the material was discussed. In this section, the details of each experiment is presented, along with the procedure and the required outcomes from each experiment.

4.1. RHEOLOGICAL MEASUREMENTS

Measuring the viscosity of the base resin and the effect of changing the shear rate on the resin is important for this work. The effect of adding the diluent to the resin on the viscosity is also important to study as adding diluent to the base resin may change the behavior of the material. For these measurements, a dynamic shear Rheometer (DSR) with parallel plates system was used to characterize the rheological behavior of the base resin and the diluted resin. Samples of around 1.0 ml of the resin and diluted resin were placed on the lower plate of the instrument and the upper plate was lowered to a gap of around 1.0 mm. The readings were taken in both ascending and descending order in a range of 0.1 1/s to 1000 1/s. After that, the curing agent was added to the diluted resin samples and were preheated to several temperatures to obtain the effect of temperature on the rheology of the sealant.

4.2. DENSITY MEASUREMENT

The density of the epoxy resin was measured using a simple weighting method. A specific volume of the sealant was placed on a high accuracy balance and the density was calculated by dividing the mass of the sealant by its volume. The value was recorded in [gm/ml].

4.3. ISOTHERMAL CURING MEASUREMENTS

These measurements are executed to estimate the gelling time of the epoxy resin to define the workability of the system at different temperatures. For a successful placement of the sealant inside the cement's fracture without premature curing the equipment, the gelling time must be estimated. For these measurements, sinusoidal oscillatory tests using the DSR at angular frequency of 10 rad/s were performed and the viscosity increase with respect to time was recorded while the preheated sealant samples were curing under different temperatures.

4.4. INJECTIVITY MEASUREMENTS

The injectivity test in the field is performed to establish the rate and pressure at which fluids can be pumped into the treatment target without breaking the formation. Prior to any remedial job, this test is performed to determine the key parameters of the treatment and the limitations. In this study, a setup consists of syringe pump, accumulator, core holder, and hand pump for confining pressure was used as shown in Figure 4. The pressures were recorded using transducers. For this test, two cement core were prepared with artificial channels of different sizes 0.3 mm and 0.51 mm. For the injectivity measurement, water was first injected in the channels followed by the sealant. The injectivity of the sealant was calculated by dividing the injection flow rate by the pressure and values were recorded in [ml/psi*min]. The 0.51 mm channel was also used to test the conventional cement injectivity.

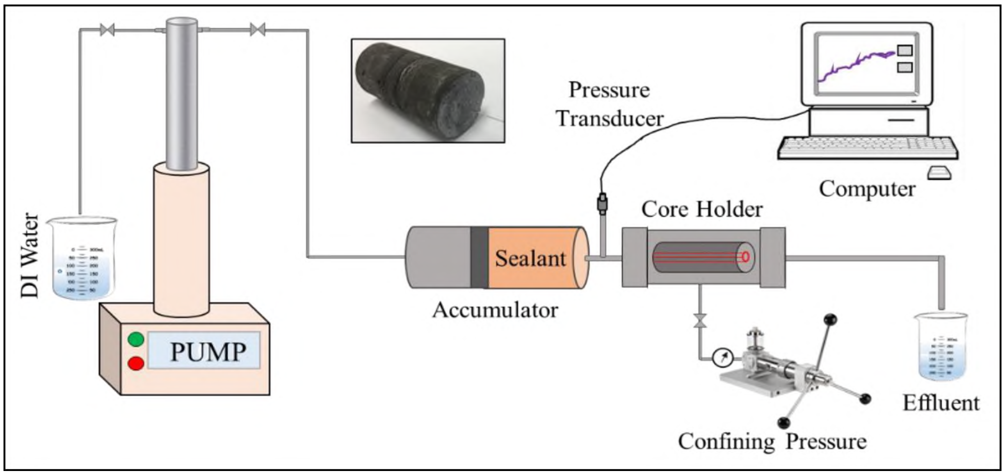

Figure 4. Injectivity and blocking performance setup.

4.5. BLOCKING PERFORMANCE MEASUREMENTS

The blocking performance measurements were conducted to test the sealant blocking efficiency. The sealant was placed in the channels of the cement and left in an oven for 24 hours to cure at 80 ˚C. Then, the cement cores were placed in the core holder of Figure 4 and water was injected.

4.6. COMPRESSIVE STRENGTH MEASUREMENT

The compressive strength measurements determine the strength of the sealant. Determining the compressive strength of the sealant is essential to ensure its ability to withstand downhole conditions. This test simply measures the force needed to crush a sample of material. For this test, the sealant was preheated to 80 ˚C prior to pouring it into 2”×2”×2” cubic molds and place it in an oven at 80 ˚C for 24 hours. Then, the cured cubes were carefully removed from the molds. The specimen height and width were measured using a caliper, and the minimum surface area was calculated. A hydraulic press was used to measure the force required to crush the samples.

5. RESULTS AND ANALYSIS

This part of the paper presents the results of each experiment and their analysis to point out their importance in the application of the sealant. The results include the rheological behavior of the sealant, the density, the curing time at different temperatures, the injectivity, the blocking efficiency, and the compressive strength of the sealant.

5.1. RHEOLOGICAL RESULTS

At low shear rate, the viscosity of the neat resin was found to be around 14764 cp. The viscosity decreased at higher shear rates with the lowest viscosity at 1000 1/s, which was found to be around 4000 cp as shown in Figure 5. These results indicate the shear-thinning behavior of the sealant under accelerated shear rate. The measurements were conducted at room temperature and atmospheric pressure. The overall viscosity of the neat resin is high, and it was essential to dilute the resin. Generally, there are two types of diluent, reactive and unreactive diluent. The later one may have one big disadvantage that diluent may be lost during the curing reaction resulting in shrinkage in the material and loss of adhesion. So, CHDGE, which is a reactive diluent, was added to the resin at different concentrations 25 and 50%. Adding 50% reactive diluent reduced the viscosity by around 97% to be around 400 cp at room temperature. In addition, adding reactive diluent at concentrations higher than 25% eliminated the structure change that can be seen at high shear rates for the neat resin. The structure change was observed by taking the viscosity measurements of two ramps up and down. Figure 5 shows these results. In this study, 50%-diluted resin was selected to be the base resin for the sealant in the next experiments. The viscosity of the 50% diluted resin is in the range of 388 to 399 cp.

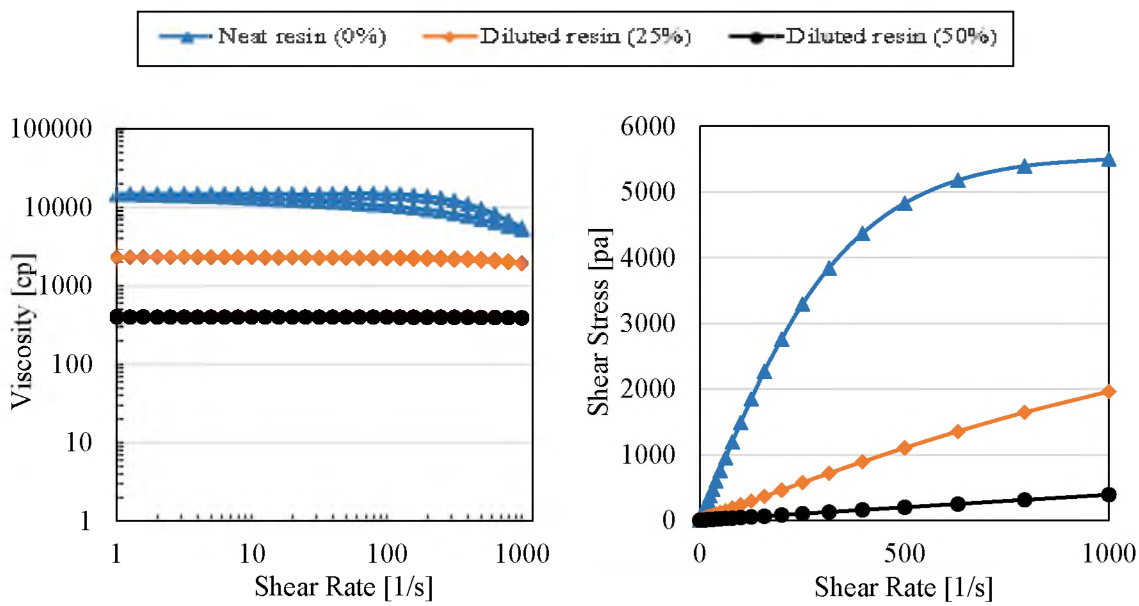

Figure 5. The rheological results for neat and diluted resin.

Furthermore, the diluted resins showed Newtonian rheological behavior with no or very low yield stress. These results suggest that the material can flow under very low forces. It is important to point out that these results are for the resin part of the material prior to adding the curing agent. The results of adding the curing agent are presented in the next section along with the effect of increasing temperature on the viscosity of the sealant.

5.2. VISCOSITY OF THE SEALANT AT DIFFERENT TEMPERATURES

In this part of the work, the viscosity of the sealant was measured. The curing agent was added to the diluted resin at stoichiometric ratio. The viscosity of the diluted resin was as mentioned earlier around 400 cp. The viscosity result of adding the curing agent to the diluted resin at 24, 60, 80, 100, and 120 °C are presented in Figure 6. Increasing the temperature decreased the viscosity of the sealant. The viscosity of the sealant at 60, 80, 100, and 120 °C was around 45, 23, 15, and 9 cp, respectively, which are very good viscosities for sealant planned to be used for remedial and/or plug and abandonment operations. The low viscosity helps ensuring successful placement of the sealant in very tight clearances with very low pumping rates needed.

Once again, the material showed Newtonian behavior with no or very low yield stress. This suggests that the material can flow under very low forces. Unlike cement that behaves like Bingham plastic or Herschel Bulkley, this sealant behaves like Newtonian fluid. The Newtonian behavior and the low viscosity makes the sealant easy to pump (Al-Ansari et al., 2015). The results herein agree with the results presented by (Perez et al., 2017 and Alsaihati et al., 2017).

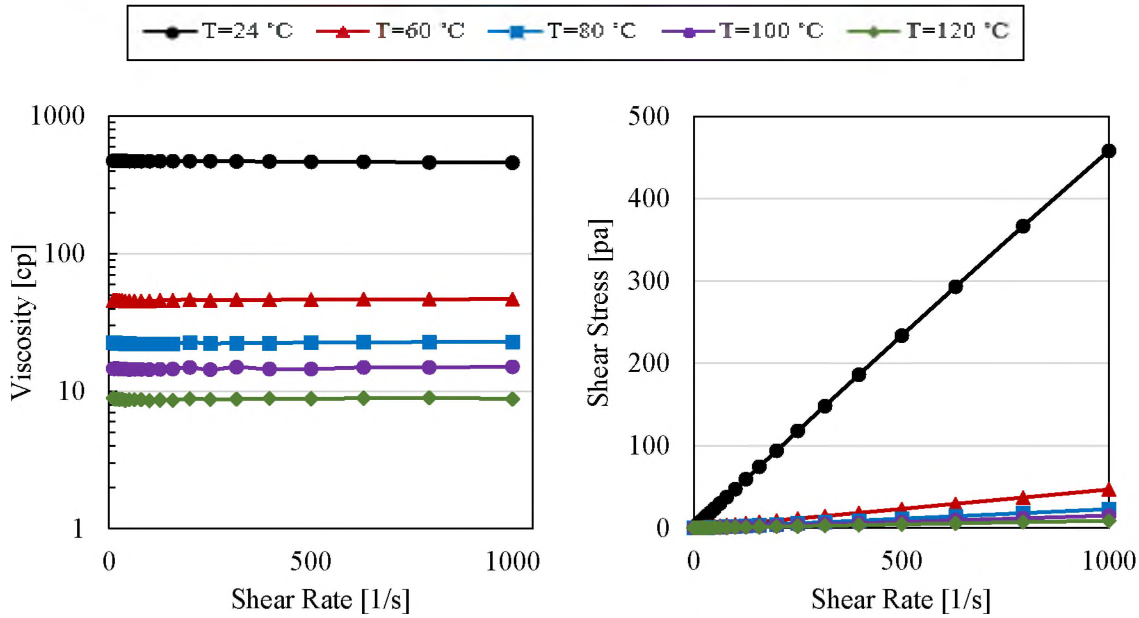

Figure 6. The effect of temperature on the viscosity of the sealant.

These are the results of the sealant in its liquid state. At elevated temperatures and with time the curing reaction will take place in continues liquid phase. Then, a cross-linking reaction will occur at some point. This point is called gelling point. At this point, the epoxy resin changes from liquid to rubber state. Then, to solid state. This can be determined by a rheological analysis of the isothermal curing process.

5.3. DENSITY RESULT

The density of the sealant was measured by simple weighting method. 0.20 ml of the sealant was taken and its weight was found to be 0.21 gm. Using these information, the density of the sealant was calculated to be 8.76 lb/gal. This density is higher than the density of water and lower than most of the fracture gradient pressures of formation. This is good to prevent fracking the formation while placing the sealant in the cement voids and good for displacing and remaining water in the cement fractures and/or between the cement and its surroundings.

5.4. ISOTHERMAL CURING RESULTS

The curing measurements were conducted at two constant temperatures 80 ˚C and 120 ˚C. The results obtained from these measurements are important to determine the workability time of the sealant at different temperatures and to evaluate the effect of temperature on the curing process of the sealant. At 80 ˚C, the sealant's complex viscosity was increasing steadily for around 6 hours. This time could be the gelling time. Then, the material started to transfer to solid after around 8 hours. After 10 hours, there was a rapid increase in the complex viscosity reaching around 24,000 cp. When the system cured for around 14 hours, the complex viscosity was around 9,000,000 cp as shown in Figure 7. The test was stopped at this point and the parallel plates were removed.

The parallel plates shown in the same figure were placed in a pure acetone and left for 24 hours. After 24 hours, the sealant was still able to adhere the plates together even though some small pieces of the cured material were observed around the plates. At 120 ˚C, the same behavior was seen except for the curing time, which became shorter as the sealant was fully cured after 3 hours. Overall, the temperature increase speeded up the curing process.

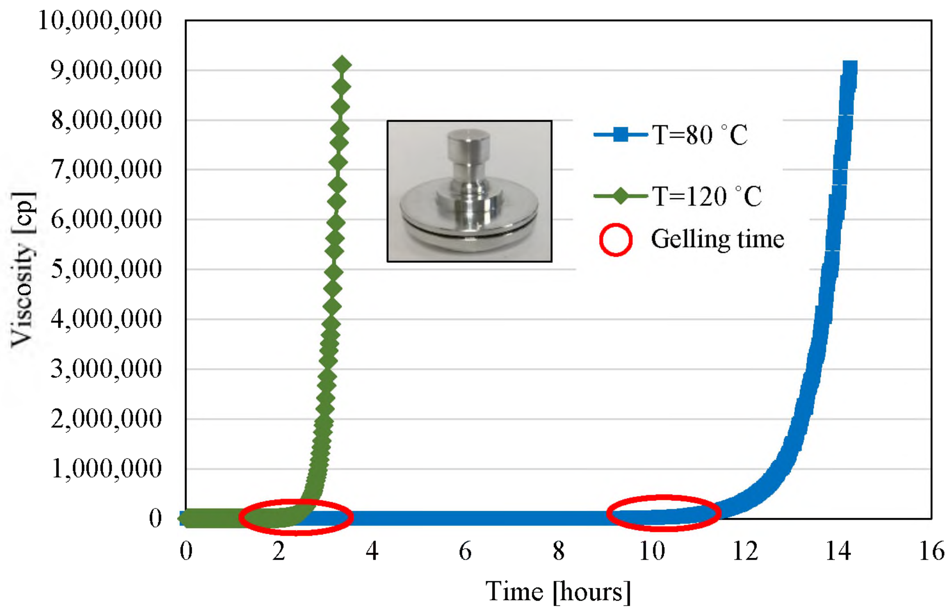

Figure 7. The isothermal curing process of the sealant at two different temperatures.

5.5. INJECTIVITY RESULTS

Prior to any remedial job, injectivity test may be conducted by pumping a solids-free material through the annulus of the wellbore to determine the injectivity factor to increase the success rate of the remedial job (Alsaihati et al., 2017).

In this study, several cement cores with two channel sizes 0.3 mm and 0.51 mm were used to measure the injectivity of the sealant compared to water and conventional cement. The sealant injectivity in the core with the channel size of 0.51 mm was found to be around 0.4228 ml/psi*min. The injection pressure was between 9 psi to 10 psi at constant flow rate of 4 ml/min as shown in Figure 8 (left side). The test was conducted for the sealant at 80°C. The same test was performed on the cement core with the smaller size and the injectivity was as expected smaller 0.2521 ml/psi*min showing that narrowing the size of the channel increases the injectivity factor and hence lowering the injectivity as shown in Figure 8 (right side). The water injection pressure was around two psi while the epoxy injection pressure was 16 psi. The flow rate was constant during the injection at 4 ml/min.

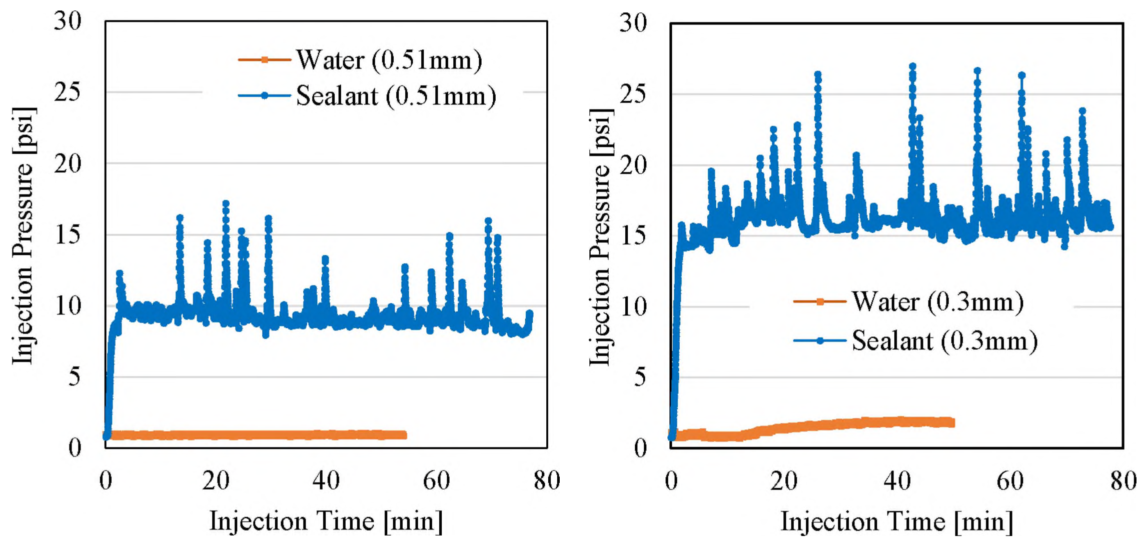

Figure 8. Injection pressure with time for 0.51 mm channel (left side) and injection pressure with time for 0.3 mm channel (right side).

Another experiment was conducted where cement was used as the injection material in the large channel (0.51 mm). The cement was injected at 4 ml/min flow rate. The injection pressure of the cement increased sharply to around 1000 psi in a few minutes as shown in Figure 9. The test was stopped at this pressure. This indicates that the cement was not able to propagate inside the cement's channel. The injectivity using the cement was calculated to be 0.004 ml/psi*min assuming that the cement penetrated the channel at that pressure (1000 psi). The reduction in the injectivity is around 99% between injecting the sealant and the cement.

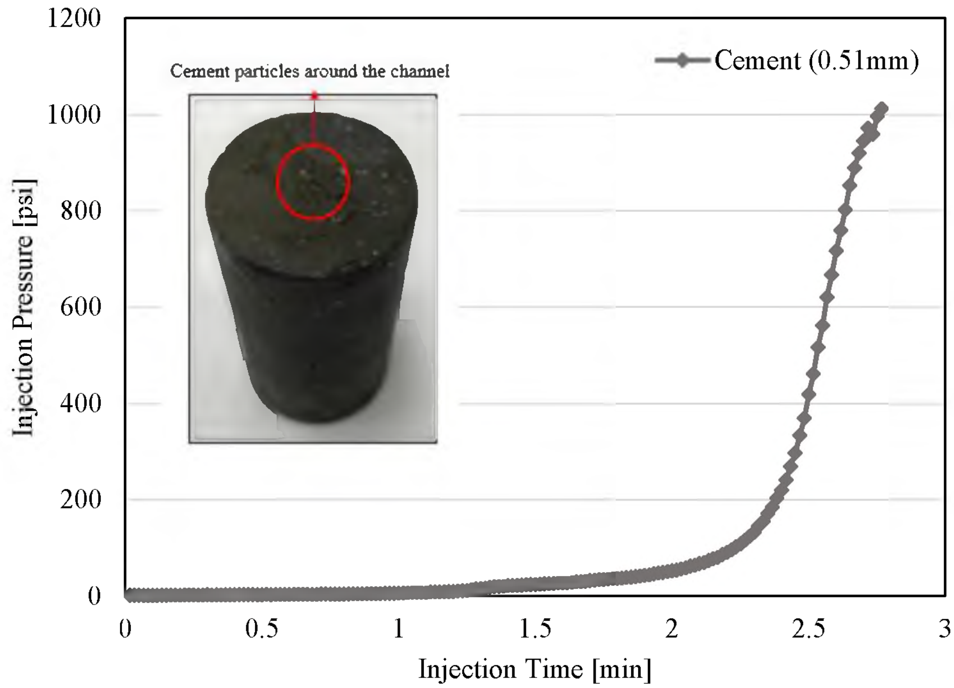

Figure 9. Injection pressure of the cement in 0.51 mm channel.

5.6. BLOCKING PERFORMANCE RESULTS

To evaluate whether the sealant is able to seal cement's channel, the sealant was placed in the channels of the cement cores and left in an oven at 80 °C to cure for 24 hours. Then, the core was placed in the core holder and tested against water flow. The injection pressure of the water was at first 500 psi. At this pressure, no water was produced from the outlet for around 20 minutes. Then, the pressure was increased to 1000 psi and left for around 15 minutes. Again, no water was observed at the outlet of the core holder. However, when the pressure increased to 1500 psi, only few drops of water appeared at the outlet of the core holder. The pressure was kept for around 40 minutes in which around 0.21 ml of water was produced as shown in Figure 10. At this pressure, the sealant started to debond from the cement. After that, the pressure was increased to 2000 psi to see whether the amount of produced water will increase or not but the amount of produced was similar. Similar results were obtained when the smaller channeled cement core was used. This experiment proves the ability of this sealant to completely seal cement channels in a differential pressure up to 1000 psi and to reduce the amount of produced water under higher pressures.

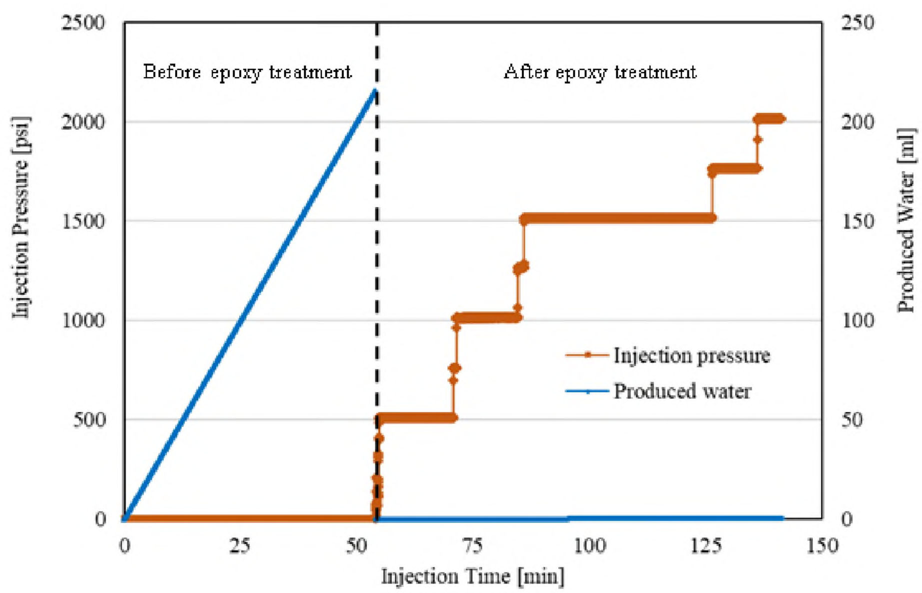

Figure 10. Water injection after placement of sealant in 0.51 mm channel.

5.7. COMPRESSIVE STRENGTH RESULT

For the compressive strength of the sealant material, a load was applied to the cubic mold in a constant rate until the load reached a value greater than 22,067 lbf as shown in Figure 11. The cubic mold did not fail under this high load and returned to its original shape showing the high ductility and strength of the material. Figure 12 shows on the right side one of the molds that were prepared for the compressive strength test, on the left side the epoxy resin after mixing in its liquid state, and in the center the cured epoxy resin, which was left in the flask for couple of days.

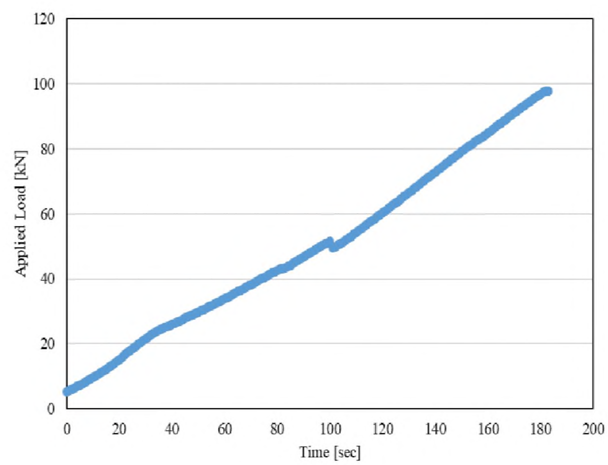

Figure 11. The load vs time in compressive strength measurement.

Figure 12. Pictures of the sealant in liquid, solid state and cured sealant in cubic form.

6. CONCLUSIONS

By studying the epoxy resin sealant, several findings were obtained. These findings are based on the results of the analysis of the rheological measurements, density measurements, isothermal curing results, the injectivity and blocking performance results, and the compressive strength result. The main conclusions are summarized below:

- The temperature activated epoxy resin sealant studied in this work exhibits Newtonian behavior with no or very low yield stress, which indicates the ease of pump of this sealant.
- The density of the sealant is higher than that of water and low enough to be used for narrow fracture pressure gradient formations.
- The temperature of the curing plays a major role in the curing time of the sealant. The system is temperature activated which means that the sealant can be mixed in-house before transferring it to the remedial job location, which would save costs on equipment and time.
- The injectivity of the sealant is much higher than that of the cement and this sealant can penetrate very tight channels.
- The sealant has the ability to withstand differential pressure as high as 1000 psi and resists loads higher than 22,067 lbf after only 24 hours of curing.

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VI. LABORATORY STUDY USING TEMPERATURE ACTIVATED EPOXY RESIN SEALANT FOR WELLBORE INTEGRITY APPLICATIONS: RHEOLOGY AND PLUGGING PERFORMANCE

ABSTRACT

Cementing is one of the most important procedures conducted during drilling of oil wells. If the cement integrity is compromised at any point during drilling or production operations, the consequences could be severe for both personnel and equipment. Cement may fail to provide zonal isolation at any point of its life, which would create pathways for fluids to migrate. Sealant materials are used to plug these pathways. This work studies a temperature triggered epoxy resin sealant to be used as a sealant material for oil and gas wells. The focus of this work is on studying, the rheological behavior of the sealant, the effect of temperature on the viscosity and the curing time of the sealant, and the ability of the sealant to block water and carbon dioxide (CO2). Experimental tests were conducted to evaluate the properties and the performance of the epoxy resin sealant. The lab tests include rheological measurements and analysis, and blocking efficiency measurements. The chemicals used to develop the epoxy resin sealant were chosen based on rigid criteria. The criteria was based on environmental impacts, ease of chemical handling, and the mechanical strength of the final product. The findings of this study show that this sealant exhibits Newtonian rheological behavior and ability to resist differential pressure higher than 2000 psi against both water and CO2. This work demonstrates that epoxy resin sealant can be used effectively and safely in sealing the cement migration pathways.

1. INTRODUCTION

Gas, oil, and water are natural resources that can be found in subterranean formations. Recovering these valuable resources usually require drilling a wellbore into the pay zone formation. During the drilling and completion phases of a wellbore, a casing (steel pipe) is run in the wellbore to provide an annulus for cementing. Next, primary cement is placed in the annulus as a barrier between the casing and the penetrated formations. The cement main functions are to protect and support the casing, and to isolate production zones (Jimenez et al., 2016). The cement must prevent the wellbore fluids from migrating in an annular flow path so as to allow the well to be utilized without any control problems. The cement must restrict any fluid communication during the life of the well among various formations and the surface. If the primary cement failed to deliver full zonal isolation at any period of its life, remedial job must be performed to restore the integrity of the cement.

Despite the huge amount of research and the numerous field operations that have been conducted throughout the world, cement failures are still occurring within the life of wells from the drilling phase to the abandonment of the well (Santos, 2015). During the life of the well, the cement may be exposed to the leftover of the drilling fluids, high mechanical loads, variations in pressure and temperature, chemical degradation due to carbonic acid and other corrosive fluids presented in the formations. Therefore, cement can fail and fluids leakage may happen. The cement failures include formation of micro-annuli at the interfaces on each side of the cement, channels through the cement, and fractures within the cement matrix. These failures provide pathways for fluids (mainly gases) to migrate either form one formation to another and/or from one formation to the surface (Alkhamis and Imqam, 2018 and Ahdaya and Imqam, 2019). The consequences of these failures may be blowouts or leaks that can cause material damage, personnel injuries, loss of production and environmental damages resulting in costly and risky repairs. Often, these losses exceed the cost of the well's repair (Sanabria et al., 2016).

For remedial jobs, squeezing cement is usually the method of repair (Shryock and Slagle 1968 and Perez et al., 2017). Squeezing cement is the process of forcing cement slurry into a hole in the casing and the cavities behind casing. However, this method may require more than one squeeze to achieve shut-off (Alkhamis et al., 2020) and is limited by the size of the leakage (Jones et al., 2014) as the cement contains solids that may increase the risk of particles bridging in narrow clearances (Davis, 2017). Even micro cement can be limited in penetrating a gap of less than 300 microns in width (Wasnik et al., 2005) in addition to the thickening time of the cement that gets affected by contamination (Dahlem et al., 2017). Generally, squeezing cement require casing perforations. One of the alternative techniques used to overcome the drawbacks of using cement for remedial jobs is the use of cross-linked polymers and polymer resins (epoxy resin), which can easily penetrate small gaps.

A combination of polymer and cross-linker can be optimized on the surface to transform from liquid form to semisolid mass at reservoir temperature. Although, polymer gel can penetrate micro pores and channels, at high temperature the 3D network structure of the gel breakdown and lose its ability to trap fluids such as water in water shut off applications. The other limitation is their lack of mechanical strength (Wasnik et al., 2005).

On the other hand, polymer resin systems can provide a sealant with superior properties for wellbore integrity applications. Polymer resin systems can be defined as "free flowing polymer solutions that can be irreversibly set to hard, rigid solids." (Morris et al., 2012). The properties of polymer resins include but not limited to good and tunable rheological behavior (Alsaihati et al., 2017), solids-free material that can penetrate small gaps (Todd et al., 2018), good wetting and adhesive properties for mineral surfaces (Brooks et al., 1974), especially, silica surfaces (Shaughnessy et al., 1978), flexibility in density which is good for areas of narrow fracture pressure gradient, tunable setting time (Sanabria et al., 2016), and exceptional resistance to contamination (Perez et al., 2017). In addition, to these liquid properties, solid cured sealant provides high mechanical strength (Ali et al., 2016; Elyas et al., 2018), resists significant strain (Khanna et al., 2018), develops good bonding properties (Genedy et al., 2017), forms no by-product during the polymerization reaction (Muecke, 1974), and has stability and durability at high temperatures, which indicates its reliability in the long term (Bertram et al., 2018). For these reasons, this type of sealant is being used as an alternative to Portland cement for remedial jobs. One of the drawbacks of polymer resins is the incompatibility of the resin with water and this downside has been discussed and solved by the use of systems that is based on aliphatic resins (Eoff et al., 2001). The other important drawback is the limited shelf life of the epoxy resin; limited shelf life would require delivering raw materials to be mixed at the well site, which would add undesirable step to the remedial operation. Hence, the system should be delivered to the well site ready to be injected (Treadway et al., 1964). Shelf life is important also in the case of using the epoxy resin in remote areas (Shaughnessy et al., 1978). This can be solved by using sealant that is activated by temperature.

Epoxy resin was first offered commercially in 1946 (Dewprashad and Eisenbraun, 1994). The applications of epoxy resins are wide including protective coatings, adhesives, electrical laminates, reinforced plastics, and commercial flooring. In the oil and gas industry, epoxy resin has been used for wellbore strengthening (consolidation). Resin consolidation strengthen the formation by binding the grains of the unconsolidated formation together at their contact points (Marfo et al., 2015). It has higher elasticity after hardening than that of the rocks glued to (Milkowski and Szwedzicki, 1973). Epoxy resin has been also used to reduce gas oil ratio to enhance oil recovery (Tiwari et al., 2017). The applicability of epoxy resin to be used for conformance control has been investigated by (Hakiki et al., 2015). It can be used for fluid losses mitigation (Teixeira et al., 2014) and as a lost circulation material (Knudsen et al., 2014).

This study presents in detail the preparation of temperature triggered epoxy resin sealant and investigates its performance through an experimental evaluation. The evaluation includes conducting lab experiments such as rheology to study the factors that affect rheological behavior of the sealant. This research also evaluates the curing kinetics of the epoxy resin system under different temperatures.

2. THEORETICAL BACKGROUND

Resins are divided into two categories (Wasnik et al., 2005): The first one is thermosetting resins, which are resins that change irreversibly under heat (from liquid to solid). Thermosetting polymers consists of chains with a relatively low molecular weight (<10000). The second one is thermoplastic resins, which are those, which soften and flow when heat and pressure are applied. They consist of chains with a relatively high molecular weight (>10000).

Epoxy resins are thermosetting polymers that contain in their unset phase (before curing) one or more epoxide groups. Epoxide group is one or more three-membered rings, known also as oxirane, epoxy, or ethoxyline group. The molecular weight of epoxy resins varies greatly. They exist in the forms of solids and liquids with wide range of viscosities.

Chemically, the epoxide groups in the resin may react with many types of curing agents (Also, called hardeners) that contain hydroxyl, carboxyl, amine, amine group. The result of the reaction is a hard three-dimensional cross-linked network. Some other types of epoxy resins may be reacted (cross-linked) by themselves through catalytic homopolymerization. Some of the epoxy resins will cure at ambient temperature but many require heat to cure (T=150-200 ˚C) (Marfo et al., 2015). Fillers or/and diluents may be added to modify the properties of the thermoset.

2.1. EPOXY RESIN CURING MECHANISM

Figure 1 shows the curing process of epoxy resins. The flow behavior of the system is similar and related to the cure process. The system at the beginning is in liquid state and cure reaction takes place in continues liquid phase. Then, a cross linking reaction occurs at some point called gel point. At this point, the epoxy resin changes from liquid to rubber state. The gel time can be determined by a rheological analysis of the cure process. After this point, the system starts to build 3D structure and become solid.

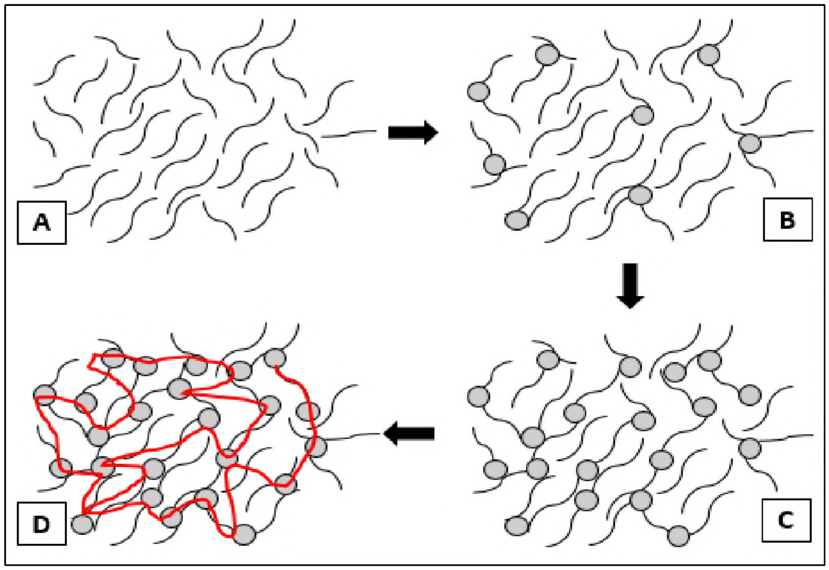

Figure 1. (a) The system in liquid state, (b) cure reaction takes place in continues liquid phase, (c) a cross-linking reaction occurs at some point called gel point, (d) the epoxy resin changes from liquid to solid state.

2.2. MIXING RATIO CALCULATIONS

The epoxy resin system can perform its best at stoichiometric ratio of 1:1. To determine the amount of curing agent to be added to the resin to reach stoichiometric ratio of 1:1 simple calculation must be done. The calculation includes finding the phase ratio of the amine (phr) which is expressed in parts by weight per 100 parts by weight of epoxy resin. Phr can be determined using Equation 1 (Petrie, 2006).

$$Phr\ of\ amine = \frac{AHEW}{EEW} \times 100 \tag{1}$$

where, AHEW is the active hydrogen equivalent weight of the curing agent and EEW is the equivalent epoxy weight.

• Equivalent epoxy weight or epoxide equivalent weight (EEW) is the ratio of the molecular weight of epoxy resin over the number of epoxy groups as shown in Equation 2 (Petrie, 2006). It is also known as the weight per epoxy (WPE).

$$EEW = \frac{MW\ of\ epoxy\ resin}{number\ of\ epoxy\ groups} \tag{2}$$

where, MW is the molecular weight expressed in [grams/mole]. Most of the resins used in the formulations of adhesives have EEWs in the range of 180 to 3200 (Petrie, 2006).

• Active hydrogen equivalent weight (AHEW) of curing agent is defined by the ratio of the molecular weight of amine over the number of active hydrogens per molecule see Equation 3 (Petrie, 2006).

$$AHEW = \frac{MW\ of\ amine}{number\ of\ active\ hydrogens} \tag{3}$$

where, MW is the molecular weight expressed in [grams/mole] and number of active hydrogens is the number of available hydrogens per molecule.

If the resin is to be diluted like the case of this study, then the EEW of the diluted resin shall be calculated using Equation 4.

$$EEW\ of\ mixture = \frac{Total\ weight\ of\ mixture}{\left(\frac{weight\ of\ part\ A}{EEW\ of\ part\ A}\right)+\left(\frac{weight\ of\ part\ B}{EEW\ of\ part\ B}\right)} \tag{4}$$

3. EXPERIMENTAL MATERIALS

3.1. CLASS-H CEMENT

The cement systems used in this study were prepared using American Petroleum Institute (API) Class-H oil well cement, which was provided by Haliburton, and distilled water. The specific gravity of the cement was measured, using gas Pycnometer, to be 3.18.

3.2. BASE RESIN

The resin used in this study is an undiluted difunctional Bisphenol A/epichlorohydrin derived liquid resin known as Bisphenol A diglycidyl ether resin (BADGE). The resin was purchased from Miller-Stephenson Chemical Company, Inc. Figure 2 shows the chemical structure of BADGE. This is one of the most widely used resins. It was selected to be the base polymer in the formulation due to its ability to produce a sealant with very good mechanical, adhesive, and chemical resistance when cured with appropriate curing agent. BADGE can be produced by reacting Bisphenol A and excess epichlorohydrin in the presence of sodium hydroxide (Dewprashad and Eisenbraun, 1994). The degree of polymerization is controlled by the ratio of the reactants.

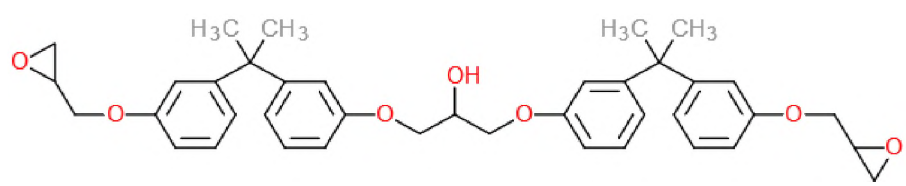

Figure 2. The chemical structure of Bisphenol A diglycidyl ether.

3.3. REACTIVE DILUENT

Since the base resin used in this study had a very high viscosity (in the range of 11000 – 14000 cp), it was essential to dilute the resin using a diluent. Diluents are low viscosity liquids added to an epoxy resin system to reduce its viscosity. They generally provide a plasticizing effect on the final product. One of their disadvantages is that they may be lost during curing which will result in shrinkage and loss of adhesion. To avoid this drawback, a reactive diluent known as cyclohexane dimethanol diglycidyl ether (CHDGE) was chosen to be utilized. This diluent is a difunctional modifier that gives moderate viscosity reduction with minimum loss of properties. It is also good for chemical resistance according to the manufacturer. The reactive diluent was purchased from Miller-Stephenson Chemical Company, Inc. The chemical structure of CHDGE is shown in Figure 3.

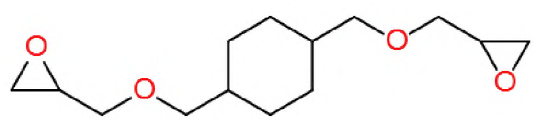

Figure 3. The chemical structure of cyclohexane dimethanol diglycidyl ether.

3.4. CURING AGENT

The curing agent of the epoxy resin was selected based on several criteria such as reactivity, toxicology, and quality of final product. Diethyltoluenediamine (DETDA), which is an aromatic amine, was selected as a curing agent for the epoxy resin. This curing agent was obtained from Albemarle chemical company. DETDA is a liquid curing agent with alkylated aromatic nuclei. It is toxicologically safe. This curing agent is less reactive and require longer time and higher temperature to cure. The decrease in the reactivity according to (Dewprashad and Eisenbraun, 1994) can be due to steric hindrance by alkyl groups adjacent to the amino group. The low reactivity is important to minimize the rate of heat released during the chemical reaction (Pardeshi et al., 2016). Figure 4 shows the chemical structure of DETDA. Table 1 is a summary of the chemicals used in this study.

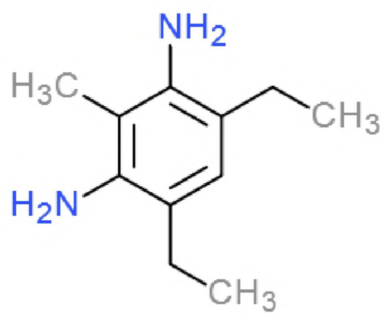

Figure 4. The chemical structure of diethyltoluenediamine.

Table 1. The chemicals used in formulation the sealant.

Materials	Weight per epoxide [g/eq]	Viscosity [cp] at 25 °C	Density [lb./gal] at 25 °C	Active hydrogen equivalent Weight [g/eq]
EPON 828	185-192 (187.6)	11000-15000	9.7	-
Heloxy 107	155-165 (158)	55-75	9.1	-
Ethacure 100	-	158.4-286.16	8.5 at 20 °C	89.1

The base resin, the diluent, and the curing agent used in this study can cause skin corrosion/irritation, eye damage/irritation, and may cause damage to organs through prolonged or repeated exposure. Wearing suitable protective gloves and eye or face protection is essential as precautionary action.

3.5. CEMENT PASTE PREPARATION

All cement slurries were mixed at room temperature in a two-speed bottom-drive laboratory blender. Dry cement was added to the blender at a uniform rate while mixing at low speed for around 15 seconds. Then, the blender was covered while the mixing continued for 35 seconds at high speed (API RP 10B-2 2013). All cement systems had a water/cement ratio (WCR) of 0.38 in accordance to API specification 10A (API 2010).

3.6. EPOXY RESIN PREPARATION

To prepare the epoxy resin system, a specific amount of base resin and reactive diluent were weighted and mixed at room temperature by hand and/or using a magnetic stirrer until a homogenous fluid was obtained. Then, a calculated amount of curing agent was added to the blend and mixed at low shear rate until the mixture was clear and homogenous Figure 5. For the high temperature experiments, the mixture was heated until the desired temperature while stirring using the magnetic stirrer.

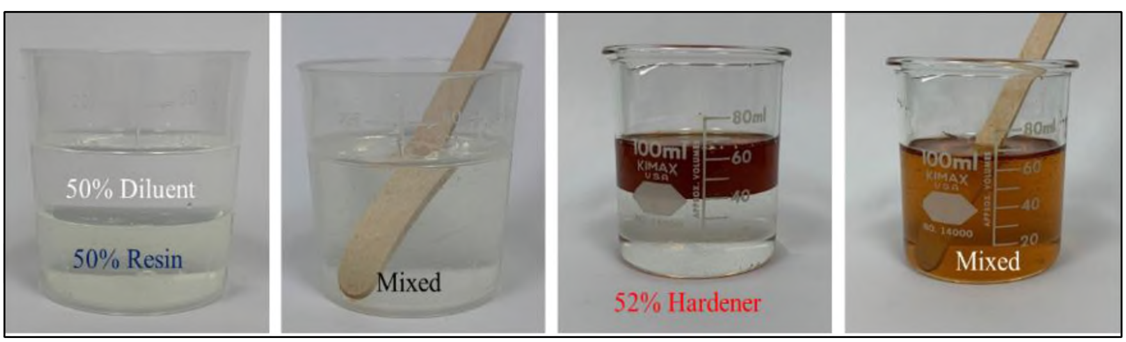

Figure 5. The chemical structure of diethyltoluenediamine.

4. EXPERIMENTAL METHODOLOGY

This section provides a detailed description of each experiment conducted in this work, along with the procedure and the required outcomes from each experiment. The experiments include rheology, curing measurements, density measurement, and blocking performance. For the rheological part of this study, two sets of experiments were conducted. First, measuring the viscosity of the resin. The effect of adding reactive diluent was investigated in this part. Second, measuring the viscosity of the system after adding the curing agent to the resin and the reactive diluent. These measurements were taken under different shear rates to mimic the placement of the sealant in the targeted zones. Then, the curing measurements were taken under steady dynamic oscillatory to monitor the viscosity development of the system without deforming it simulating the curing time of the sealant inside the targeted zones.

4.1. RHEOLOGICAL MEASUREMENTS

These measurements are important for this study to optimize the amount of reactive diluent to be added to the base resin and to characterize the viscosity of the epoxy resin system and to measure the effect of temperature on the system. For these measurements, the epoxy resin samples were preheated to the desired temperatures. An advanced Anton Paar Rheometer, which is a dynamic shear Rheometer (DSR) with parallel plates system, was used to characterize the rheological behavior of the base resin and the epoxy resin. Samples of 0.5 to 1.0 ml of the epoxy resin were placed on the lower plate of the instrument and the upper plate was lowered to a gap of 0.5 to 1.0 mm. The readings were taken in both ascending and descending order in a range of 0.1 to 1000 1/s.

4.2. ISOTHERMAL CURING MEASUREMENTS

These measurements are executed to estimate the gelling time of the epoxy resin to define the workability of the system. This information is essential to be known to protect the downhole equipment and to ensure a safe and successful placement of the sealant inside the fracture. If this measurement was not optimized, it may lead to a sealant with premature curing. For these measurements, sinusoidal oscillatory tests using the DSR were performed at an angular frequency of 10 rad/s and the complex viscosity increase with time was monitored while the preheated epoxy resin samples were curing under different temperatures. In these tests, disposal parallel plates of 25 mm in diameter were used as the tests were run until the material reached a viscosity of nine-million-cp.

4.3. DENSITY MEASUREMENTS

The density of the sealant is another critical property to be studied. Generally, the density of the sealant should be higher than that of water to displace it and lower than that of the formation fracture pressure gradient to avoid breaking it. The density was measured using simple weighting method. A specific volume of the sealant was placed on a high accuracy balance and the density was calculated by dividing the mass of the sealant by its volume. The value was recorded in [gm/ml].

4.4. BLOCKING PERFORMANCE MEASUREMENTS

In this study, a setup consists of syringe pump, accumulator, back pressure regulator, and stainless-steel tubes were used as shown in Figure 6. The pressures were recorded using transducers.

For this test, several stainless-steel tubes were prepared with different inner diameters 1.753 mm and 4.572 mm and different lengths 3 inches and 12 inches. The epoxy resin was mixed and placed inside the tubes and left in an oven for 24 hours to cure at a temperature of 80 ˚C. Then, the tubes were removed and placed in the testing setup. The ability of the epoxy sealant to seal the tubes were tested against both water and CO2.

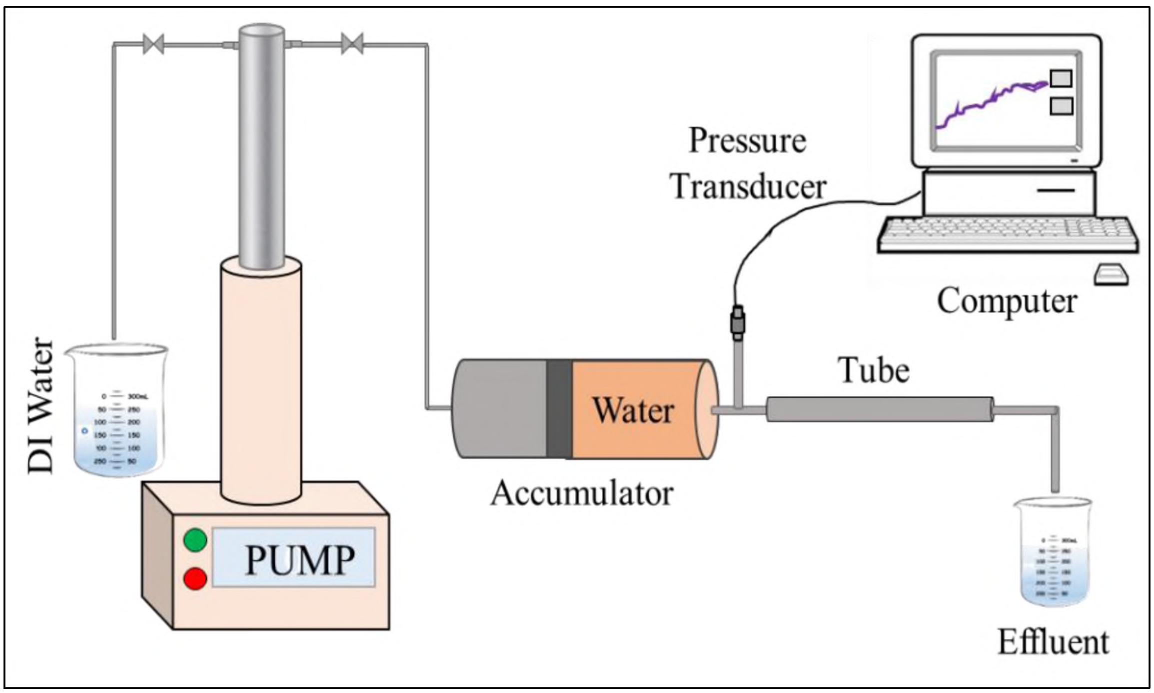

Figure 6. Blocking performance setup.

5. RESULTS AND ANALYSIS

In this section of the paper, the results of each experiment are presented and analyzed according to their importance in the application of the sealant.

5.1. RHEOLOGICAL MEASUREMENTS RESULTS

5.1.1. Viscosity of Neat and Diluted Resin. First, the viscosity of the neat resin was measured using the Rheometer. The viscosity of the neat resin was found to be in the range of 3991 cp at high shear rate to 14764 cp at low shear rate. The behavior of the sealant under accelerated shear rate was shear-thinning meaning that increasing the shear rate decreases the viscosity of the resin. The measurements were conducted at room temperature and atmospheric pressure. The overall viscosity is high, and it was essential to dilute the resin using diluent. In general, there are two types of diluent, reactive diluent and unreactive diluent. The later one may have one big disadvantage that it may be lost during curing which will result in shrinkage and loss of adhesion. For remedial application, this is not preferable as it contradict the main goals of sealing cement fractures and that is one of the reasons why reactive diluent was selected. The other reason is that reactive diluents impart flexibility and mechanical strength to the sealant (Du et al., 2018). CHDGE reactive diluent was added to the resin at different concentrations 5.0%, 10%, 25%, 40%, and 50% (by weight of resin). Adding 10% and 50%, reactive diluent reduced the viscosity by around 50% and 97%, respectively. In addition, adding reactive diluent at concentrations higher than 25% eliminated the structure change that can be seen at high shear rates for the neat resin, 5.0% diluted resin, and 10% diluted resin. The structure change was observed by taking the viscosity measurements of two ramps up and down. Figure 7 shows these results. In this study, 50% diluted resin was selected for further experiments. The viscosity of the 50% diluted resin is in the range of 388 cp and 399 cp at room temperature.

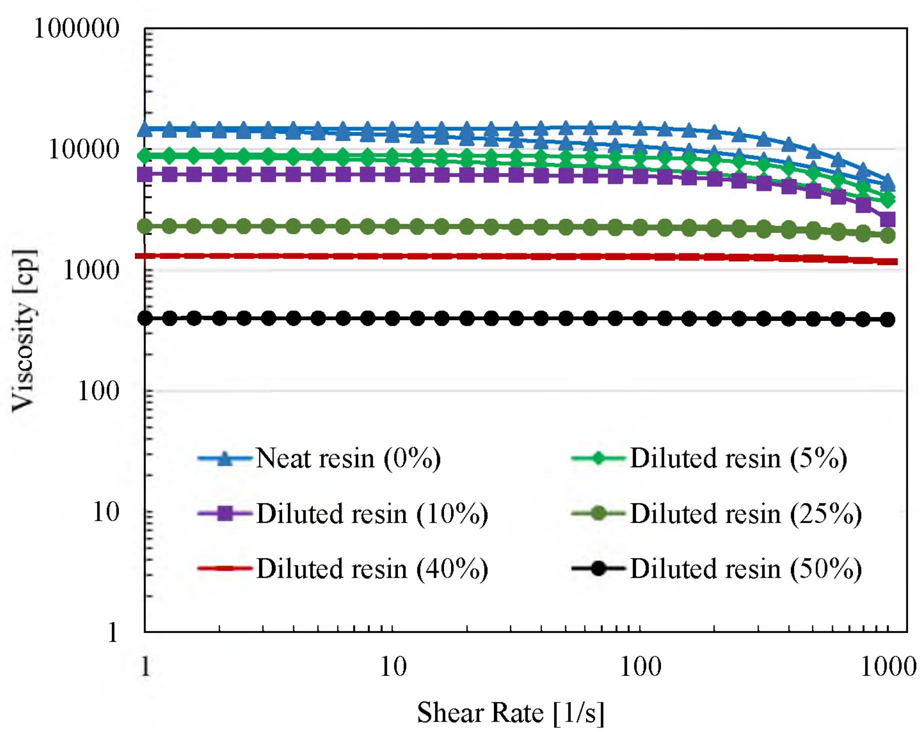

Figure 7. The viscosity results for neat and diluted resin.

Furthermore, the 25%, 40%, and 50% diluted resins showed Newtonian rheological behavior with no or very low yield stress as shown in Figure 8. This Newtonian-like behavior can also be observed in Figure 7, which shows that the viscosity of the material is independent of the shear rate. These results suggest that the material can flow under very low forces. It is important to point out that these results are for the resin part of the material prior to adding the curing agent. Figure 9 represents the effect of the amount of reactive diluent on the viscosity of the neat resin. This figure can be used in general to predict the viscosity of the diluted resin.

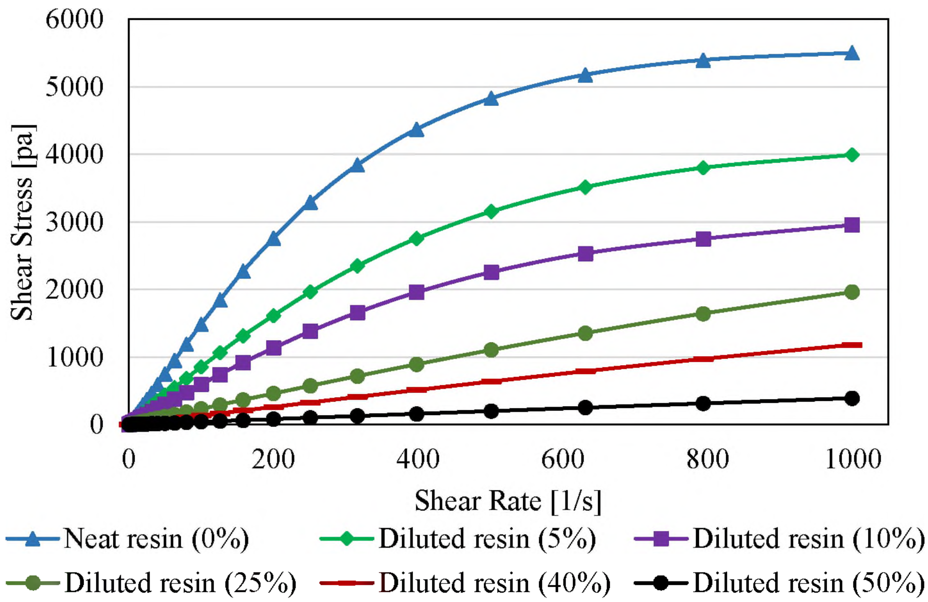

Figure 8. The shear stress vs shear rate results for neat and diluted resin.

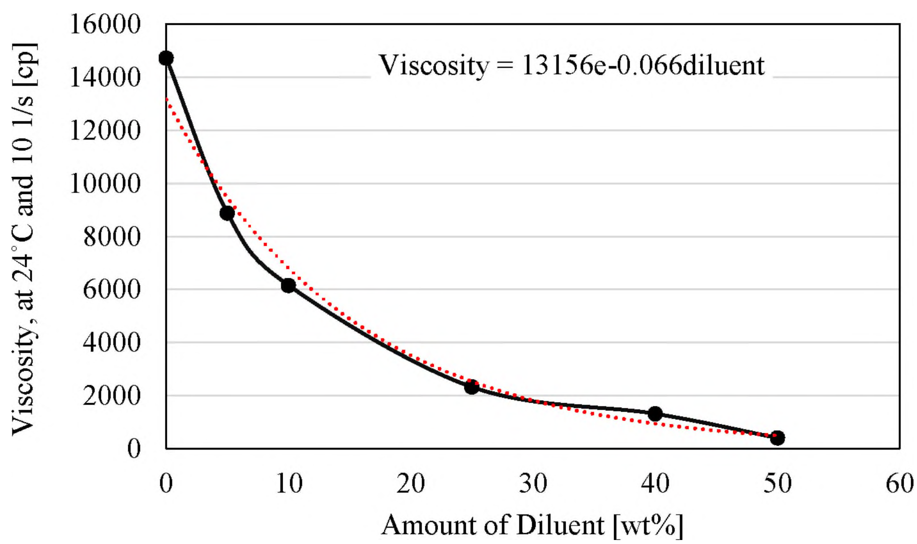

Figure 9. Effect of diluent on viscosity at low shear rate.

5.1.2. Viscosity of the Sealant System. In this part of the study, the viscosity of the epoxy resin sealant was measured. The curing agent was added to the diluted resin at stoichiometric ratio. The viscosity of the diluted resin was as mentioned earlier around 400 cp. The viscosity result of adding the curing agent to the diluted resin at 24˚C, 60˚C, 80˚C, 100˚C and 120˚C are presented in Figure 10. The viscosity of the sealant decreased with increasing the temperature. The viscosity of the sealant at 80˚C was around 23 cp, which is a very good viscosity. This low viscosity helps ensuring successful placement of the sealant in very tight clearances with very low forces needed (Alkhamis et al., 2019).

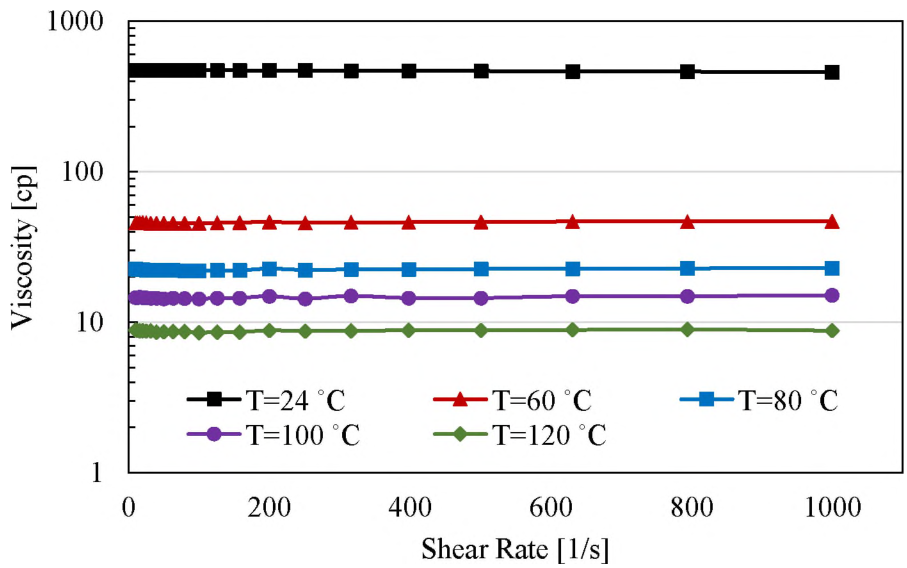

Figure 10. Effect of temperature on the viscosity of the sealant.

Again, the material showed Newtonian rheological behavior with no effects of shear rate on the viscosity of the material. These results suggest that the material can flow under very low forces. It is essential to point out that cement behaves like Bingham plastic or Herschel Bulkley while this sealant behaves like Newtonian fluid. The

Newtonian behavior and the low viscosity makes the sealant easy to pump (Al-Ansari et al., 2015). These are the results of the sealant in liquid state. At elevated temperature and with time the cure reaction takes place in continues liquid phase. Then, a cross linking reaction occurs at the gel point. At this point, the epoxy resin changes from liquid to rubber state. Then, to solid state. The gel point can be determined by a rheological analysis of the curing process presented next in this paper.

5.2. ISOTHERMAL CURING MEASUREMENTS RESULTS

Curing measurements were conducted at three constant temperature 80 ˚C, 100 ˚C, and 120 ˚C. The objectives of these measurements are to determine the workability time of the sealant at different temperatures and to study the effect of temperature on the curing process of the sealant. At 80 ˚C, the sealant complex viscosity was increasing slightly for around 6 hours. This time could be the gelling time. Then, the material started to transfer to solid after around 8 hours. After 10 hours, there was a rapid increase in the complex viscosity reaching around 24,000 cp. When the system cured for around 14 hours, the complex viscosity was around 9,000,000 cp as shown in Figure 11. The test was stopped at this point and the parallel plates were removed as shown in Figure 12. The parallel plates shown in the figure were placed in a pure acetone. The plates were removed 24 hours later; the sealant was still able to bond them together. Some small pieces of the cured material were observed around the plates. At 120 ˚C, the same behavior was seen except for the curing, which became shorter as the sealant was cured after 3 hours. These measurements are crucial to optimize the placement of the sealant in the cement fractures. Short curing time may result in premature hardening; this may plug the coil tubing before reaching the targeted zone. Long curing time may affect the sealant negatively if the formation fluids bypassed the sealant and/or created channels in it. Therefore, optimum-curing time is desired for successful remedial job. Also, determining the bottom hole temperature is important prior to the remedial job to determine the exact gelling time of the sealant at that temperature to determine the waiting on sealant time. The temperature increase speeded up the curing process. The effect of this acceleration on the properties of the sealant are being studied currently and will be published later.

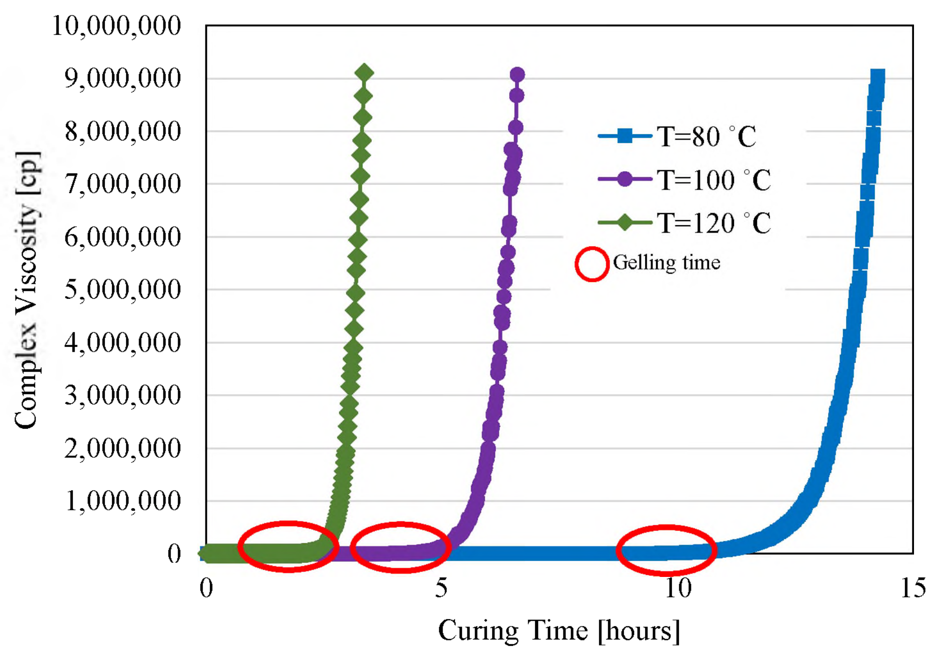

Figure 11. The isothermal curing process of the sealant at different temperatures.

Figure 12. The cured sealant between the parallel plates.

5.3. DENSITY MEASUREMENTS RESULTS

The density of the sealant was found to be 8.76 lb. /gal. This density is slightly higher than the density of water, which is good for displacing the remaining water in the cement fractures and possibly good to prevent fracking the formation that has narrow fracture gradient. The density measurements are listed in Table 2.

Table 2. The density measurements of the sealant.

Ratio	Volume [ml]	Mass [gm]	Density [gm/ml]	Density [lb./gal]
1:1	0.20	0.21	1.05	8.76

5.4. BLOCKING PERFORMANCE RESULTS

The ability of the epoxy resin to seal the tubes was tested by injecting water in one side of the tube while the other side was left open to collect water if any leakage occurs. The test started by injecting the water at constant flow rate while monitoring the pressure at the inlet. The pressure started to build up gradually at the inlet of the tubes. The injection pressure reached around 2160 psi in around 22 minutes as shown in Figure 13. After that, the injection stopped for safety reasons. At this high pressure the epoxy resin sealant showed an efficient blocking and no water was observed at the outlet of the tubes. This experiment was repeated using tubes with different diameters and different lengths and no leakages were observed in all the experiments. These results show the ability of the epoxy resin tested herein to bond with steel tubes. This an indication that this type of sealants can be used effectively in sealing the micro annuli that could form between the cement and the steel casing in oil wells.

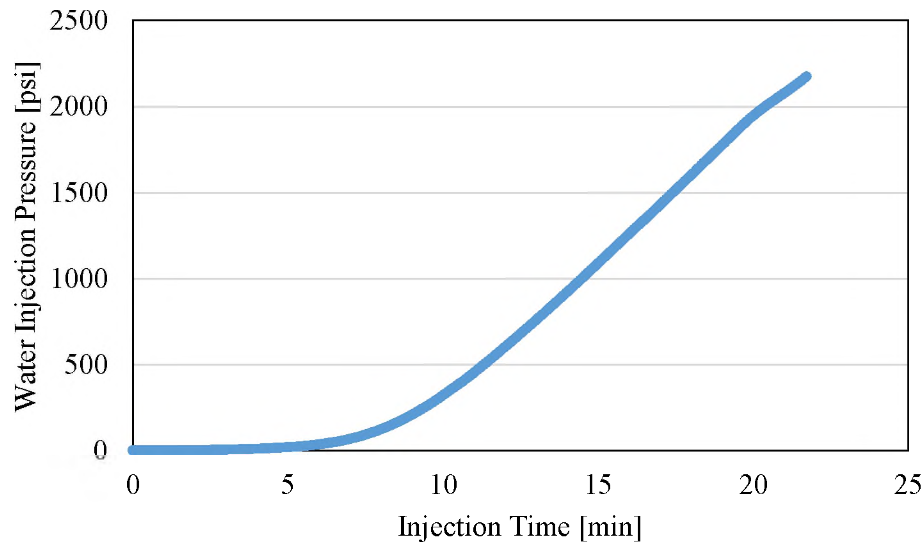

Figure 13. Injection pressure of water in sealed tube (4.572 mm).

After testing the sealant against water leakages, similar experiments were conducted to test its performance against gases. First, the tubes were placed in the setup but this time another pressure transducer was installed at the outlet of the tubes in addition to back pressure regulator. These modifications were done to the setup to capture the CO_2 gas in case of any leakages occur. The CO_2 was injected at the inlet of the tubes at constant flow rate until the pressure reached around 2000 psi as shown in Figure 14. The outlet pressure remained zero during these experiments indicating that the epoxy resin sealant is also efficient against gases.

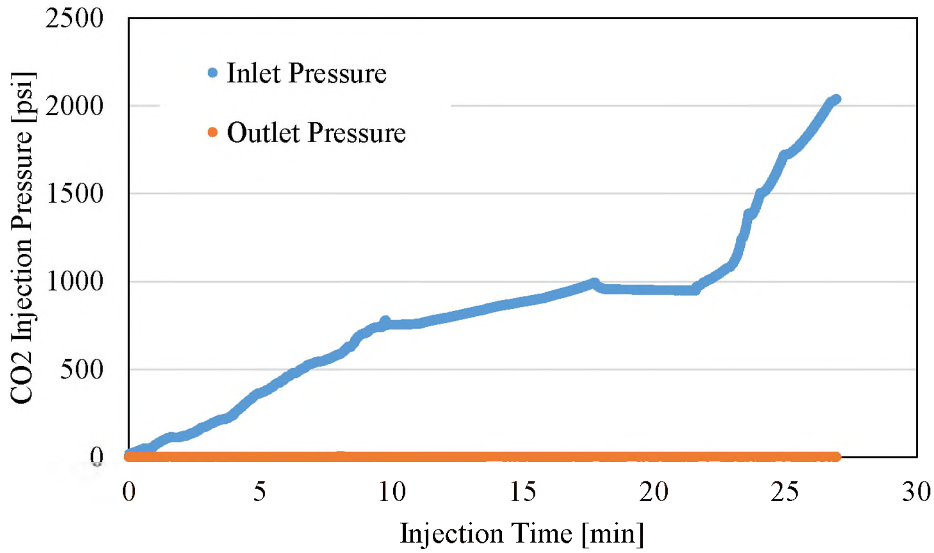

Figure 14. Injection pressure of CO_2 in sealed tube (4.572 mm).

6. CONCLUSIONS

By studying the epoxy resin sealant, several findings were obtained. These findings are based on the results of the analysis of the rheological measurements, density measurements, isothermal curing results, and blocking performance results. The main conclusions are summarized below:

- The temperature triggered epoxy resin sealant tested in this study exhibits Newtonian behavior with no or very low yield stress, which indicates the ease of pump of this sealant.
- The density of the sealant is higher than that of water and low enough to be used for narrow fracture pressure gradient formations.
- The temperature of the curing plays a major role in the curing time of the sealant.

- The system is temperature activated which means that the sealant can be mixed in-house before transferring it to the remedial job location, which would save money on equipment and time.
- The sealant has the ability to seal tubes with different sizes and withstand pressures as high as 2000 psi. The sealant was able to stop water and CO2 leakages.

NOMENCLATURE

$g/ml = Gram\ per\ milliliter.$

$psi = Pounds\ per\ square\ inch.$

$lb/gal = Pounds\ per\ gallon.$

$BWOC = By\ weight\ of\ cement.$

$\rho = Density, Pounds\ per\ gallon.$

$Pa = Pascal.$

$Cp = Centipoise.$

$\mu = Viscosity.$

$gm = Gram.$

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SECTION

2. CONCLUSIONS AND RECOMMENDATIONS

2.1. CONCLUSIONS

By studying the cement and the epoxy resin sealants, several findings were obtained. These findings are based on the results of the literature review regarding cement failures, the analysis of the rheological measurements, density measurements, isothermal curing results, injectivity measurements, chemical resistance measurements, blocking performance results, and mechanical measurements. The main conclusions are summarized below:

- Today, the petroleum industry focuses on the short-term properties, which is good for the first days after the cement placement. However, the long-term properties are as important as the short-term and tests should be conducted for the durability of the cement.
- The initial state of stress in the cement sheath must be studied to estimate the limitation of the cement downhole.
- To better evaluate the state of stress in the cement after setting, the hydration process of the cement and time effect should be analyzed.
- It can be concluded from this review that the sealant material for oil and gas wells must follow these criteria:
    - Has low environmental impact with desired density.
    - Low fluid losses and very little to zero free fluids.

    - High mechanical strength and experience low shrinkage.
    - Short transition time and chemical resistance.
    - Adhesive properties with steel and different types of formations.

- The viscosities of the sealants were highly dependent on temperature as increasing the temperature reduces the viscosity significantly and at the same time reduces the curing time of the sealants. The sealants behaved like Newtonian fluids and the shear rate had no significant impact on viscosity.
- Epoxy resin C showed high injectivity and ability to penetrate small gaps when compared to conventional Portland cement.
- The three sealants had thermal degradation temperatures and Tg higher than the proposed applications.
- The three sealants showed high capability of plugging cement channels and reduces the permeability to zero. In addition, the permeability reduction after initiating new cracks in the cement was high. The sealants were able to stop water leakage and CO_2 leakage.
- This work covered the rheology and plugging performance of the sealants but the workability of the sealants (thickening time) needs to be investigated in the future.
- All the sealants showed great ability to withstand chemicals, but Epoxy resin A was the only sealants that was able to withstand the 98% sulfuric acid. Thus, epoxy resins formulated similar to Epoxy resin A can be implemented in applications where sulfuric acid is present.
- The void size, viscosity of the sealants, injection flow rates, and heterogeneity of the voids played major roles in determining the injectivity of the sealants. Having

a sealant with Newtonian behavior was beneficial in eliminating the effect of the flow rate.

- Solids-free sealants exhibited the most potential to successfully remediate wellbores in terms of the injectivity of the material.
- Solids-free sealant demonstrated high injectivity and low degradation after injection.
- The epoxy resin showed Newtonian behavior, and the injectivity showed the effect that Newtonian materials have on the injectivity.
- The cement presented a huge limitation in terms of its ability to penetrate small voids.
- The PPG showed good injectivity, but unless this injectivity is correlated with the ability to develop enough strength to hold reservoir fluids in place, this injectivity is not useful.

2.2. RECOMMENDATIONS

Based on the results obtained from this study, it is recommended to investigate the use of additives for the epoxy resin sealants such as fly ash to reduce the cost of the sealants and to be used as primary cementing material. It is also essential to measure the thickening time of the sealant prior to any application.

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VITA

Mohammed Mousa M Alkhamis was born in Dammam, Saudi Arabia. He received his bachelor’s degree in mechanical engineering from King Fahd University of Petroleum and Minerals (KFUPM), Dhahran, Kingdom of Saudi Arabia in January 2014. He received his Master of Science in Petroleum Engineering from Missouri University of Science and Technology in May 2018. In August 2018, Mohammed started working on his PhD at Missouri University of Science and Technology under the supervision of Dr. Abdulmohsin Imqam. He received his Doctor of Philosophy in Petroleum Engineering from Missouri University of Science and Technology in May 2021.