

Laboratory Study of Using Temperature Activated Epoxy Resin Sealant for Wellbore Integrity Applications: Rheology and Plugging Performance

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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.

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 from 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 details the preparation of temperature triggered epoxy resin sealant and investigates its performance through an experimental evaluation. The evaluation include 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.

Theoretical Background

Resins are divided into two categories (Wasnik et al., 2005):

1. Thermosetting resins: are resins that change irreversibly under heat (from liquid to solid). Thermosetting polymers consists of chains with a relatively low molecular weight (<10000).
2. Thermoplastic resins: 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 ($T=150-200\text{ }^{\circ}\text{C}$) (Marfo et al., 2015). Fillers or/and diluents may be added to modify the properties of the thermoset.

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.

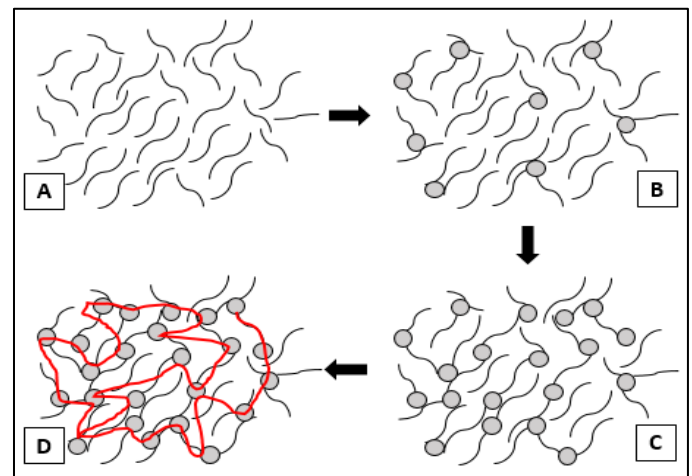


Figure 1. (a) The system in liquid state, (b) cure reaction takes place in continues liquid phase, (c) a cross-linking reaction occurs at some point called gel point, (d) the epoxy resin changes from liquid to solid state.

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).

$$\text{Phr of amine} = \frac{\text{AHEW}}{\text{EEW}} \times 100 \dots \dots \dots (1)$$

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) and the epoxy molar mass (EMM).

$$\text{EEW} = \frac{\text{MW of epoxy resin}}{\text{number of epoxy groups}} \dots \dots \dots (2)$$

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).

$$\text{AHEW} = \frac{\text{MW of amine}}{\text{number of active hydrogens}} \dots \dots \dots (3)$$

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**.

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

Where, weight of part A and weight of part B represent the amount of the resin and the diluent, respectively.

The information about the EEW and AHEW may be given by the chemicals providers.

Experimental Materials

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.

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.

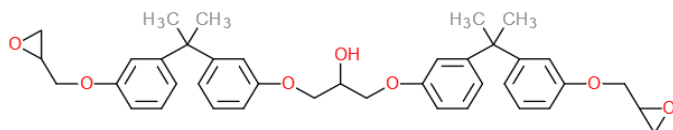


Figure 2. The chemical structure of Bisphenol A diglycidyl ether.

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**.

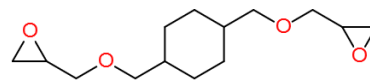


Figure 3. The chemical structure of cyclohexane dimethanol diglycidyl ether.

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.

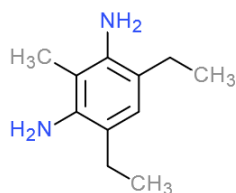


Figure 4. The chemical structure of diethyltoluenediamine.

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 Resin 828	185-192 (187.6)	11000-15000	9.7	-
Heloxy 107	155-165 (158)	55-75	9.1	-
Ethacure 100 DETDA	-	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.

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).

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.

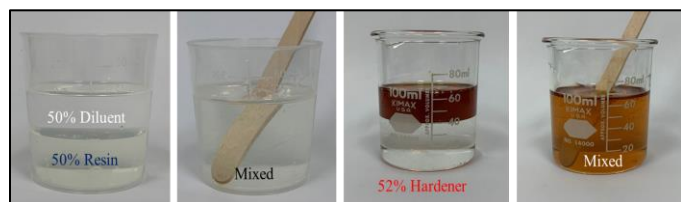


Figure 5. The chemical structure of diethyltoluenediamine.

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.

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 reading were taken in both ascending and descending order in a range of 0.1 1/s to 1000 1/s.

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-centipoise.

Density Measurement

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].

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.

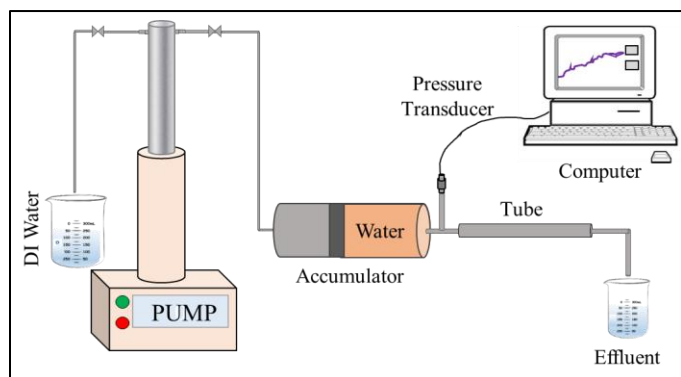


Figure 6. Blocking performance setup.

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.

Rheological Measurements Results

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.

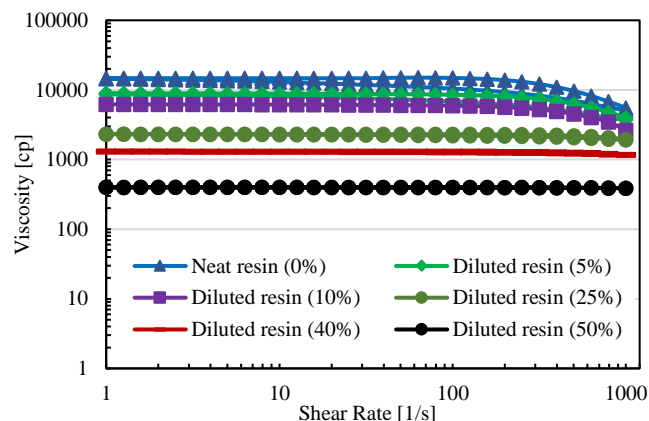


Figure 7. The viscosity results for neat and diluted resin.

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.

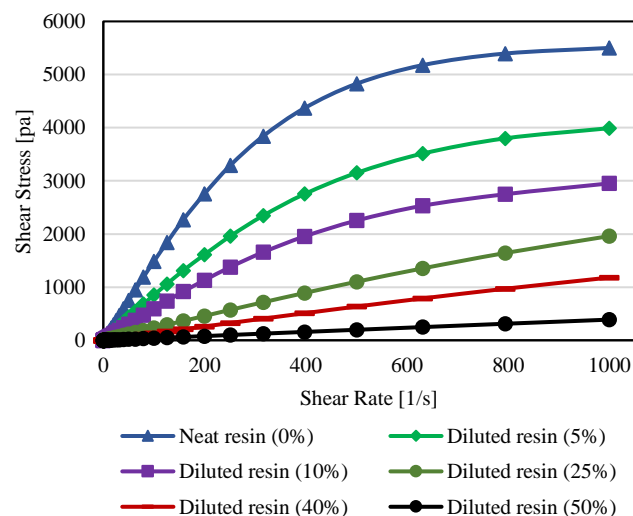


Figure 8. The shear stress vs shear rate results for neat and diluted resin.

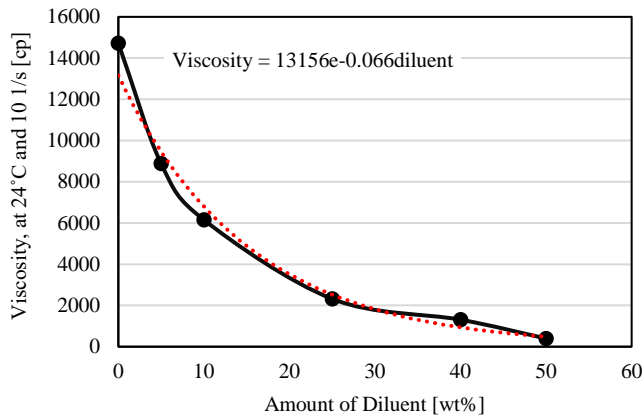


Figure 9. Effect of diluent on viscosity at low shear rate.

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).

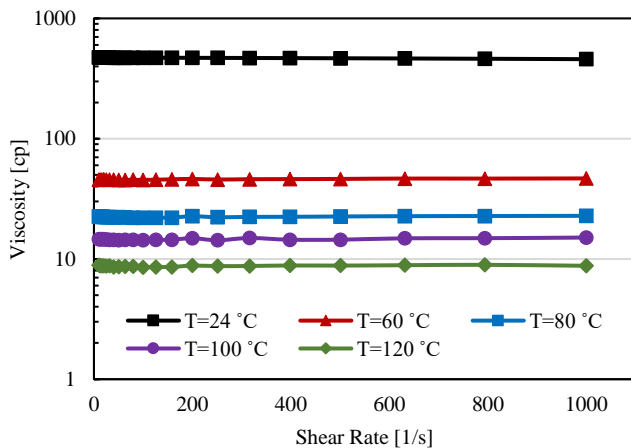


Figure 10. Effect of temperature on the viscosity of the sealant.

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.

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.

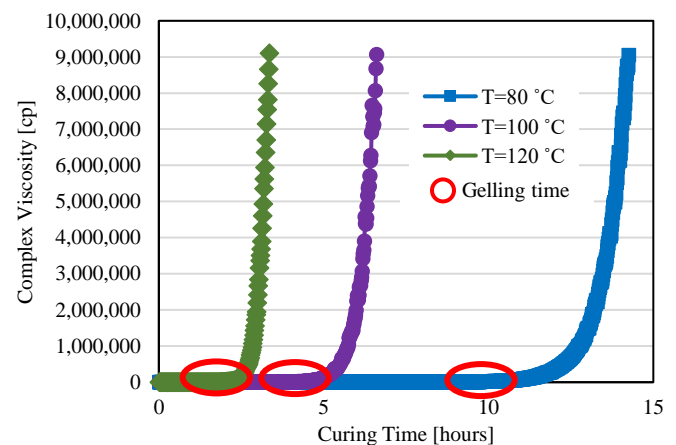


Figure 11. The isothermal curing process of the sealant at different temperatures.



Figure 12. The cured sealant between the parallel plates.

Density Measurement Result

The density of the sealant studied in this paper 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 fracturing 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

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.

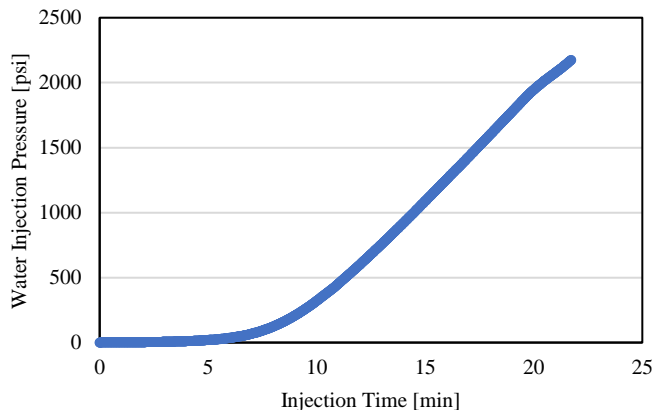


Figure 13. Injection pressure of water in sealed tube (4.572 mm).

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. This modifications were done to the setup to capture the CO₂ gas in case of any leakages occur. The CO₂ 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.

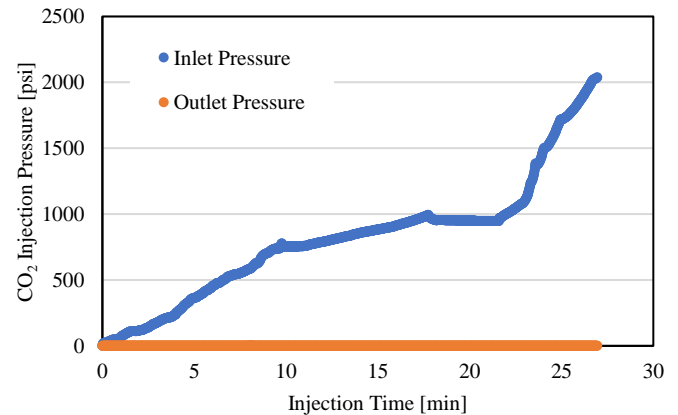


Figure 14. Injection pressure of CO₂ in sealed tube (4.572 mm).

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.

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Nomenclature

g/ml = Gram per milliliter.

psi = Pounds per square inch.

lb/gal = Pounds per gallon.

$^{\circ}C$ = Degree celsius.

$BWOC$ = By weight of cement.

ρ = Density, Pounds per gallon.

Pa = Pascal.

Cp = Centipoise.

μ = Viscosity.

gm = Gram.

References

- Ahdaya M, Imqam A, Fly ash Class C based geopolymer for oil well cementing, *Journal of Petroleum Science and Engineering*, Volume 179, 2019, Pages 750-757, ISSN 0920-4105, <https://doi.org/10.1016/j.petrol.2019.04.106>.
- Ahdaya M, Imqam A, Investigating geopolymer cement performance in presence of water based drilling fluid, *Journal of Petroleum Science and Engineering*, Volume 176, 2019, Pages 934-942, ISSN 0920-4105, <https://doi.org/10.1016/j.petrol.2019.02.010>.
- Al-Ansari, A. A., Al-Refai, I. M., Al-Beshri, M. H., Pino, R. M., Leon, G. A., Knudsen, K., & Sanabria, A. E. (2015, September 8). Thermal Activated Resin to Avoid Pressure Build-up in Casing-Casing Annulus (CCA). *Society of Petroleum Engineers*. doi:10.2118/175425-MS.
- Ali, A., Morsy, A., Bhaisora, D., & Ahmed, M. (2016, November 7). Resin Sealant System Solved Liner Hanger Assembly Leakage and Restored Well Integrity: Case History from Western Desert. *Society of Petroleum Engineers*. doi:10.2118/183295-MS.
- Alkhamis, M., & Imqam, A. (2018, August 16). New Cement Formulations Utilizing Graphene Nano Platelets to Improve Cement Properties and Long-Term Reliability in Oil Wells. *Society of Petroleum Engineers*. doi:10.2118/192342-MS.
- Alkhamis, M., Abdulfarraj, M., & Imqam, A. (2020, January 13). Solids-Free Epoxy Sealant Materials; Injectivity through Channels for Remedial Job Operations. *International Petroleum Technology Conference*. doi:10.2523/IPTC-20110-MS.
- Alkhamis, M., Imqam, A., & Milad, M. (2019, December 2). Evaluation of an Ultra-High Performance Epoxy Resin Sealant for Wellbore Integrity Applications. *Society of Petroleum Engineers*. doi:10.2118/199184-MS.
- Alsaihati, Z. A., Al-Yami, A. S., Wagle, V., BinAli, A., Mukherjee, T. S., Al-Kubaisi, A., Alsafran, A. (2017, June 1). An Overview of Polymer Resin Systems Deployed for Remedial Operations in Saudi Arabia. *Society of Petroleum Engineers*. doi:10.2118/188122-MS.
- API RP 10B-2, Recommended Practice for Testing Well Cements, second edition. 2012. Washington, DC: API.
- API Specification 10A/ISO 10426-1, Specification for Cements and Materials for Well Cementing, Twenty-third Edition.
- Bertram, F., Tuxen, A., & Nielsen, T. B. (2018, February 7). Development of Environmentally Friendly Epoxies for Well Conformance. *Society of Petroleum Engineers*. doi:10.2118/189475-MS.
- Brooks, F. A., Muecke, T. W., Rickey, W. P., & Kerver, J. K. (1974, June 1). Externally Catalyzed Epoxy for Sand Control. *Society of Petroleum Engineers*. doi:10.2118/4034-PA.
- Dahlem, J. E., Baughman, T., James, T., & Kelly Rives, R. (2017, May 1). Intervention and Abandonment - Riserless Productive Zone Abandonment Using Epoxy Resin. *Offshore Technology Conference*. doi:10.4043/27847-MS.
- Davis, J. E. (2017, September 5). Using a Resin-Only Solution to Complete a Permanent Abandonment Operation in the Gulf of Mexico. *Society of Petroleum Engineers*. doi:10.2118/186113-MS.
- Du, J., Bu, Y., Liu, H., & Shen, Z. (2018, July 30). Experimental Feasibility Study of a Novel Organic-Inorganic Hybrid Material for Offshore Oil Well Cementation. *International Society of Offshore and Polar Engineers*.
- Elyas, O., Alyami, A., Wagle, V., & Alhareth, N. (2018, August 16). Use of Polymer Resins for Surface Annulus Isolation Enhancement. *Society of Petroleum Engineers*. doi:10.2118/192266-MS.
- Eoff, L., Chatterji, J., Badalamenti, A., & McMechan, D. (2001, January 1). Water-Dispersible Resin System for Wellbore Stabilization. *Society of Petroleum Engineers*. doi:10.2118/64980-MS.
- Elturki, M., Imqam, A. (2020, June 28). Application of Enhanced Oil Recovery Methods in Unconventional Reservoirs: A Review and Data Analysis. *American Rock Mechanics Association*.
- Ewert, D. P., Almond, S. W., & Bierhaus, W. M. (1991, May 1). Small-Particle-Size Cement. *Society of Petroleum Engineers*. doi:10.2118/20038-PA.
- Fundamentals of Epoxy Formulation Brahmadeo Dewprashad and E. J. Eisenbraun *Journal of Chemical Education* 1994 71 (4), 290 DOI: 10.1021/ed071p290.
- Hakiki, F., Salam, D. D., Akbari, A., Nuraeni, N., Aditya, W., & Siregar, S. (2015, October 20). Is Epoxy-Based Polymer Suitable for Water Shut-Off Application? *Society of Petroleum Engineers*. doi:10.2118/176457-MS
- Jimenez, W. C., Urdaneta, J. A., Pang, X., Garzon, J. R., Nucci, G., & Arias, H. (2016, April 20). Innovation of Annular Sealants During the Past Decades and Their Direct Relationship with On/Offshore Wellbore Economics. *Society of Petroleum Engineers*. doi:10.2118/180041-MS.
- Jones, P. J., Karcher, J., Ruch, A., Beamer, A., Smit, P., Hines, S., Day, D. (2014, February 25). Rigless Operation to Restore Wellbore Integrity using Synthetic-based Resin Sealants. *Society of Petroleum Engineers*. doi:10.2118/167759-MS.
- Khanna, M., Sarma, P., Chandak, K., Agarwal, A., Kumar, A., & Gillies, J. (2018, January 29). Unlocking the Economic Potential of a Mature Field Through Rigless Remediation of Microchannels in a Cement Packer Using Epoxy Resin and Ultrafine Cement Technology to Access New Oil Reserves. *Society of Petroleum Engineers*. doi:10.2118/189350-MS.
- Knudsen, K., Leon, G. A., Sanabria, A. E., Ansari, A., & Pino, R. M. (2014, December 10). First Application Of Thermal Activated Resin As Unconventional LCM In The Middle East. *International Petroleum Technology Conference*. doi:10.2523/IPTC-18151-MS.
- Marfo, S. A., Appah, D., Joel, O. F., & Ofori-Sarpong, G. (2015, August 4). Sand Consolidation Operations, Challenges and Remedy. *Society of Petroleum Engineers*. doi:10.2118/178306-MS.
- Milkowski, W., & Szwedzicki, T. (1973, January 1). Use of Epoxy Resin for Strengthening Walls of Narrow Workings. *International Society for Rock Mechanics and Rock Engineering*.
- Moneeb Genedy, Usama F. Kandil, Edward N. Matteo, John Stormont, Mahmoud M. Reda Taha, A new polymer nanocomposite repair material for restoring wellbore seal integrity, *International Journal of Greenhouse Gas Control*,

- Volume 58, 2017, Pages 290-298, ISSN 1750-5836, <https://doi.org/10.1016/j.ijggc.2016.10.006>.
29. Morris, K., Deville, J. P., & Jones, P. (2012, January 1). Resin-Based Cement Alternatives for Deepwater Well Construction. Society of Petroleum Engineers. doi:10.2118/155613-MS.
 30. Muecke, T. W. (1974, February 1). Factors Influencing the Deterioration of s Plastic Sand Consolidation Treatments. Society of Petroleum Engineers. doi:10.2118/4354-PA.
 31. Pardeshi, M., Wilke, A., Jones, P. J., Gillies, J., & Jedlitschka, V. (2016, March 22). Novel Use of Resin Technology for Offshore Pilot Hole Abandonment. Offshore Technology Conference. doi:10.4043/26565-MS
 32. Petrie, E. M. (2006). Epoxy adhesive formulations. New York: McGraw-Hill).
 33. Sanabria, A. E., Knudsen, K., & Leon, G. A. (2016, November 7). Thermal Activated Resin to Repair Casing Leaks in the Middle East. Society of Petroleum Engineers. doi:10.2118/182978-MS.
 34. Santos, O. L. A. (2015, January 1). Technology Focus: Well Integrity (January 2015). Society of Petroleum Engineers. doi:10.2118/0115-0100-JPT.
 35. Shaughnessy, C. M., Salathiel, W. M., & Penberthy, W. L. (1978, December 1). A New, Low-Viscosity, Epoxy Sand-Consolidation Process. Society of Petroleum Engineers. doi:10.2118/6803-PA
 36. Shryock, S. H., & Slagle, K. A. (1968, August 1). Problems Related to Squeeze Cementing. Society of Petroleum Engineers. doi:10.2118/1993-PA.
 37. Teixeira, G. T., Lomba, R. F. T., Fontoura, S. A. B., Melendez, V. A., Ribeiro, E. C., Francisco, A. D., & Nascimento, R. S. (2014, September 10). New Material for Wellbore Strengthening and Fluid Losses Mitigation in Deepwater Drilling Scenario. Society of Petroleum Engineers. doi:10.2118/170266-MS.
 38. Tiwari, S., Deo, A., Kumar, A., Anand, P., Singhal, A., Kumar, A., Li, J. (2017, March 6). Low Injection Squeeze to Reduce Gas Oil Ratio & Gain Significant Crude Production using Epoxy Resin Technology. Society of Petroleum Engineers. doi:10.2118/183988-MS.
 39. Todd, L., Cleveland, M., Docherty, K., Reid, J., Cowan, K., & Yohe, C. (2018, September 17). Big Problem-Small Solution: Nanotechnology-Based Sealing Fluid. Society of Petroleum Engineers. doi:10.2118/191577-MS.
 40. Treadway, B. R., Brandt, H., & Parker, P. H. (1964, January 1). Laboratory Studies Of A Process To Consolidate Oil Sands With Plastics. Society of Petroleum Engineers. doi:10.2118/906-MS.
 41. Vicente Perez, M., Melo, J., Blanc, R., Roncete, A., & Jones, P. (2017, October 24). Epoxy Resin Helps Restore Well Integrity in Offshore Well: Case History. Offshore Technology Conference. doi:10.4043/28124-MS.
 42. Wasnik, A. S., Mete, S. V., & Ghosh, B. (2005, January 1). Application of Resin System for Sand Consolidation, Mud Loss Control & Channel Repairing. Society of Petroleum Engineers. doi:10.2118/97771-MS.