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Sealant technologies for remediating cement-related oil and gas well leakage

A state-of-the-art literature review

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Acknowledgments

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Executive summary

Wells play a central role in unconventional gas development and ensuring well integrity during their entire life and beyond is a significant challenge. Leaking wells are an ongoing risk for the oil and gas industry, resulting in loss of production, safety concerns and environmental damage. There are many potential solutions and technologies that can be applied for remediating well leakage. Some of the technologies widely applied in industry are summarised below:

- *Conventional oilfield cements*. Portland based cement has been the option of choice for the majority of oil and gas well remediation treatments. Squeeze cementing has been applied to remediating leaking wells related to poor primary cement jobs and has been mostly successful. However, due to their particle sizes, the conventional oilfield cement cannot penetrate and seal small fractures or defects with an aperture less than approximately 400 μm (micrometre);
- *Micro fine cements*. Micro fine cement systems have been developed and applied to seal small well leaks, such as microannuli, casing leaks, where the conventional oilfield cements failed. However, laboratory and field case studies have demonstrated that the micro fine cement systems cannot penetrate and seal a micro fracture with an aperture less than approximately 120 μm;
- *Polymer resins*. Polymer resins have numerous advantages over cementitious sealants including the perceived ability to penetrate deeper into smaller fractures that have aperture less than 120 μm due to its solid free nature. In addition, with proper formulation the polymer resin can be managed in terms of curing time and viscosity. It has also been proved that polymer resins are highly stable after set at elevated temperature (> 80°C). These properties make the polymer resins favoured for remediating tight well leaks that would not be possible using conventional oilfield cements.

In addition, there are many other novel materials and commercially available products that have the potential to replace oilfield cements as alternative well remediating sealants. These include polymer gels, geopolymers and low melting point alloys. Nano technology has been applied to improve the performance of existing well sealants, such as cements and polymer resins. These sealants are currently being studied in laboratory and/or under field trials, and are not as widely applied in oilfields.

Despite the progress in sealant technologies made over the years, significant technology gaps remain in meeting industry needs. In particular, sealing micro fractures with an aperture less than approximately 120 μm is a significant challenge. Solid free polymer resin is perceived to be able to penetrate deeper and seal narrow fractures. However, the commercial products of the polymer resins have high viscosities which would make them difficult to be injected into small leaking pathways. Furthermore, there are limited studies on their long-term durability at harsh downhole environments, particularly the durability of their bonding strength to downhole surrounding materials, such as casing, cement sheath and formation rocks.

1 Introduction

Australia has abundant unconventional gas resources with estimates of over 200 trillion cubic feet (TCF) of all identified coal seam gas (CSG) resources and more than 1000 TCF of recoverable shale gas across the country (Cook et al., 2013). The impact of developing the unconventional resources on the environment remains a significant concern in Australia.

Wells play a central role in unconventional gas development and ensuring well integrity during their entire life and beyond is a significant challenge. Leaking wells are an ongoing risk for the oil and gas industry, resulting in loss of production, safety concerns and environmental damage. A substantial amount of work has been dedicated to identify well failure mechanisms and failure rates (Davies et al., 2014; King, 2014; King and King, 2013; Wu et al., 2016). A recent literature review on well integrity submitted to the NT Hydraulic Fracturing Inquiry showed that the rate of total well integrity failures that have the potential to cause environmental contamination is about one in 1000 wells, whilst the rate for single-well barrier failures which may not lead to environment contamination is higher at approximately 1-10 in 100 wells (Huddlestone-Holmes, et al 2017).

There are many different types and severity of well leaks. The causes of well leaks are often related to aging well equipment and operation errors during the well life cycle. These types of leaks can be relatively easy to mitigate once identified. The other causes for well leakage are related to well cementing or well operations after the cement is set. For example, gas migration can take place along micro fractures and microannuli in the cement sheaths behind the well casing and/or on the interfaces with the casing or formation rock due to debonding. This type of compromised well integrity is recognised as the most common well integrity risk (APPEA, 2017), and a challenge to remediate.

This report is a state-of-the-art literature review on sealant technologies that are applied for mitigating and remediating cement-related well leakage. The objective of the review is to develop a comprehensive understanding on existing technologies in mitigating and remediating compromised well integrity, on both relatively widely applied products and technologies and those under development, with an aim to develop new well sealing materials and technologies.

The report is organised as follows:

Section 2 provides a background to well leakage, including concept of well barriers, leakage pathways and causes, their occurrence rate and severity, and performance criteria required to meet for well sealing materials and technologies.

Section 3 reviews existing well leaking remediation technologies using cementitious materials. This includes conventional oilfield cements and micro fine cements. Cement squeezing technologies, field tools and practices in remediating well leakage are briefly described.

Section 4 reviews application of thermal activated polymer resins, a relatively new well leaking remediation technology. This includes the resin type (with initiator or accelerator), typical physical and mechanical properties of the polymer resins and some case studies in remediating well leakage.

Section 5 provides a brief review on new well sealing technologies under development and/or field trial, including alternative materials such as nano technology enhanced well sealants, polymer based gels, geopolymers and low melt point metal alloys.

Section 6 provides a review on laboratory methods for assessing performance of new well sealing materials based on the performance criteria described in Section 2.

Finally, technology gaps in mitigating and remediating well leakage will be discussed in Section 7 followed by a summary in Section 8.

2 Background – well leakage

Well integrity failure is defined as unfavourable outcomes at which all the barriers within a wellbore system fail and leakage paths created regardless of the potential for contaminating events (King and King, 2013). Well barrier failure can occur at any stage during the lifetime of a well when leakage pathways might be created at different locations within a wellbore. To maintain the integrity of the wellbores, a wellbore barrier system should be designed in a way to endure the mechanical and thermal operational procedures imposed by production and recovery phases during a well lifetime.

2.1 Well barriers

Wellbore barrier failure might occur due to the failure of the individual or multiple barriers even if there are no indications of detectable leakage into the wellbore surroundings (King and King, 2013). If a barrier fails, an assessment must be done to evaluate the imposed risk of fluid leakage and repair procedures should be planned. A barrier failure might happen during different stages of a well lifetime, i.e. pre-production phases / and production phases (Teodoriu et al., 2013; Watson and Bachu, 2009).

Well-cementing (cementation) is an influential stage of a wellbore construction since the cement sheath is responsible for providing complete zonal isolation, therefore a key barrier of the well barrier system (Teodoriu et al., 2010). The cement sheath should meet both short-term and long-term required characteristics to overcome all pressure and temperature variations imposed to a well during well lifetime and beyond (Ravi et al., 2002b). Accordingly, it is of utmost importance to comprehend the cement mechanical failure mechanisms. The cement sheath may experience different types of mechanical damage as a result of exposing to different wellbore operational procedures (Bois et al., 2012).

2.2 Well leakage

2.2.1 Causes

Some of the well operational procedures may lead to a barrier failure in the pre-production phase, i.e. pressure integrity tests (or leak-off tests) (Postler, 1997). Pressure integrity tests (PIT) are performed after cementing each casing, and impose pressure upon the set cement (Mueller & Eid, 2007). Drilling practices may also damage the unstable formations (caving) due to the imposed vibrations and pressures which may lead to formation failure. In addition, some formations are naturally weak and not stable enough or may have some faults and cracks. These faults can threaten the integrity of the wellbores even before the commencement of production procedures (Teodoriu et al., 2013).

The casing centralization should be executed properly. Otherwise, the cement would not be able to displace the mud from the annulus completely during cementing procedures and leads to the formation of eccentric cement sheath and non-uniform cement sheath thickness or possibly not fully covers the created gap between the casing and formation rock. This deviation of the casing from the center can cause unbalanced concentration of stress on the one side of the wellbore which results in additional shear stress to the cement sheath (Nabipour et al., 2010).

The existence of mud cake and grease deteriorates the bond strength between the cement and casing or the formation during cement pumping procedure. Additionally, contamination of cement by mud or formation fluid may weaken the cement mechanical properties as well, which may lead to compromising the wellbore integrity (Teodoriu et al., 2013). Muds have thixotropic behaviour and tend to build a gel-structure under low shear circumstances. The gelled pockets should be broken up and cleaned to achieve stronger cement bonding. Another reason could be related to the improper composition (cement slurry formulation) of the cement slurry, in terms of its compatibility with the formation which results in weak bonding properties (Teodoriu et al., 2013; Zhang & Bachu, 2011).

Cement shrinkage leads to a volumetric reduction and can consequently cause de-bonding between cement and casing or formation. This can also result in tensile cracks and increased permeability which provides pathways for undesired fluid and gas migration (Reddy et al., 2007; Zhang & Bachu, 2011).

Due to high overbalance conditions (high gradient of pressure between the well and the formation), the fluid in the cement slurry could be filtrated. This lack of water during the hydration process will decrease the cement strength (Teodoriu et al., 2013).

During production phases, the mechanical and thermal stress state of a wellbore is subjected to different pressure and temperature variations for different reasons (Ravi et al., 2002a). These include the alteration in induced pressure and temperature originating from casing expansion / contraction (Goodwin & Crook, 1992), hydraulic fracture stimulation (Bellabarba et al., 2008), tectonic stress, subsidence and formation creep, normal well production (Zhang & Bachu, 2011), injection of hot steam or cold water (Bois et al., 2011). These operational procedures have significant effects on the integrity and the failure mechanism of the cement sheaths.

2.2.2 Pathways and sources of fluids

Leakage incidents occur provided that a source of fluid, failure of one or two well barriers, and driving forces for fluid movement such as fluid buoyancy or excessive pore pressure are present simultaneously (Davies et al., 2014).

The leakage pathways can be categorized into two different groups; primary and secondary (Weideman & Nygaard, 2014). The primary category is more related to the time of primary cementing and secondary are associated with the events and conditions after cementing is complete.

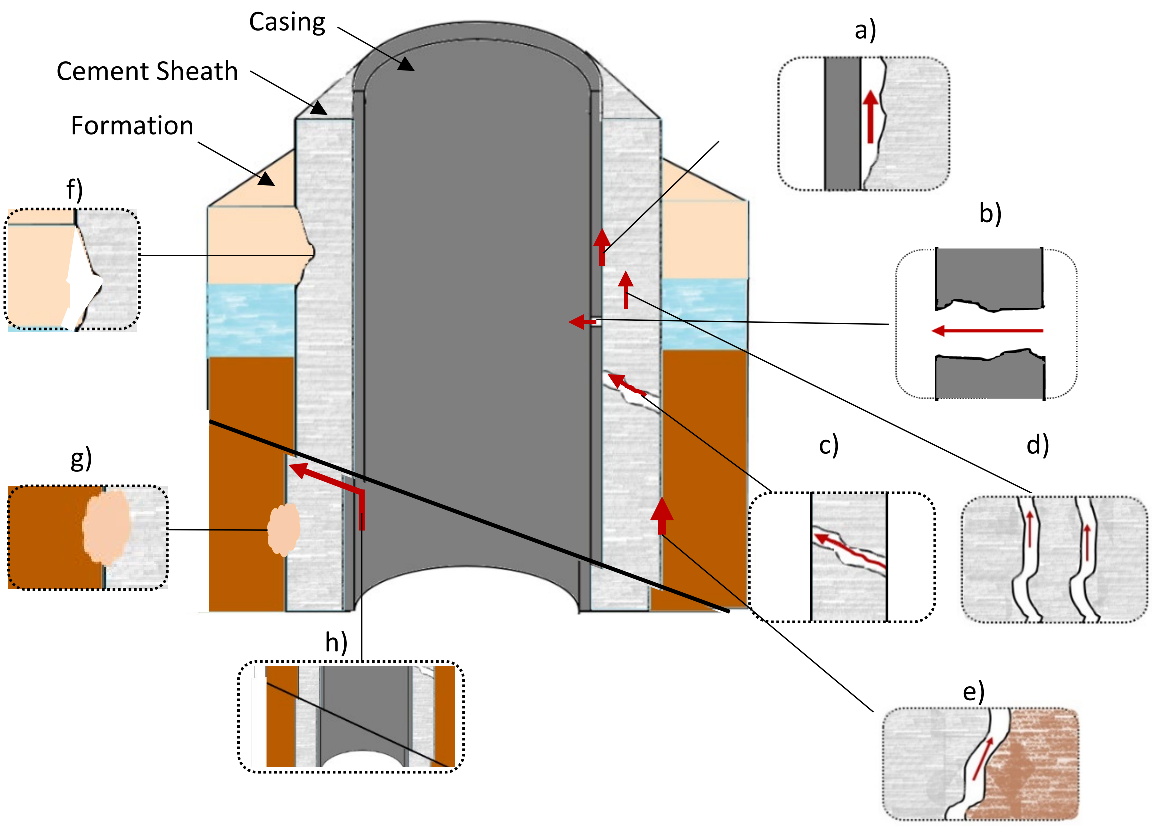

Figure 2.1: Potential leakage pathways locations along a wellbore after (Celia et al., 2005)

Figure 2.1 illustrates the possible locations of primary and secondary leakage pathways along a wellbore. The primary leakage pathways can be created due to mechanical failure of the casing (casing burst/collapse) or corrosion (Figure 2.1b) (Crow et al., 2010; Weideman & Nygaard, 2014).

Figure (2.1f) shows an unsatisfactory annular cementing job when the cement does not fill the annulus entirely, poor bonding due to the existence of mud cake is shown in Figure (2.1g), and the development of channels in the cement is shown in Figure (2.1d).

The secondary category included the leakage pathways created along micro-annuli at the cement sheath interfaces with the casing and the formation respectively (Crow et al., 2010; Weideman & Nygaard, 2014) as shown in Figure 2.1a and Figure 2.1e and degraded or cement fractures (Figure 2.1c) (Celia et al., 2005; Weideman & Nygaard, 2014).

The secondary leakage pathways might be created due to many reasons including but not limited to deterioration of cement bond strength which leads to the creation of micro-annulus at cement interfaces with the casing and the formation (Figure 2.1a and 2.1e), cement shrinkage, and cement mechanical failure (Figure 2.1c) (Dusseault et al., 2000). Shear failure mechanisms may happen in specific geological conditions due to reservoir compaction in the production period which can lead to the creation of shear failure zone in the formation and casing above the reservoir (Figure 2.1h).

It is worth noting that the cement used in the oil and gas industry has very low permeability, usually less than a 0.2 mD (Lecampion et al., 2011; Maharidge et al., 2016) which indicates that hydraulic isolation is accomplished straight forwardly, and any possible leakage can only occur through mechanical failures of the cement sheath (Lecampion et al., 2013). Therefore, the integrity of the cement sheath may be compromised mostly because of the creation of cracks and micro-annulus within the cement sheath (Saidin et al., 2008).

Figure 2.2 schematically demonstrates the different types of cracks that may occur within the cement sheaths. Radial cracks (Figure 2.2a) might be created due to the difference in pressure between the inner wall of the cement sheath with the outer wall which leads to the cement sheath expansion/contraction (Bois et al., 2012). The cement sheath may experience a large deviatoric state of stress which leads to shear damage (Figure 2.2b) (Bois et al., 2012). Disking cracks might be created due to axial sliding / disking of the cement sheath (Figure 2.2c) (Bois et al., 2012). The cement sheath interfaces debonding may occur due to the uneven expansion/contraction of the cement sheath in comparison with the displacement of the surrounding wellbore components which leads to the creation of micro-annulus within the wellbores (Figure 2.2d) (Bois et al., 2012). Consequently, understanding of cement failure mechanisms under different operating conditions is of the utmost importance for the better evaluation of wellbore integrity

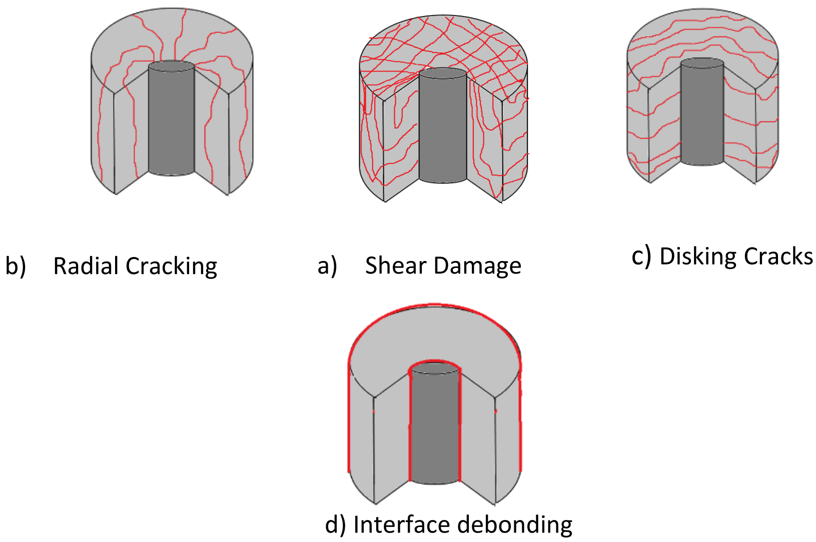

Figure 2.2: Different types of cracks within the cement sheath after (Bois et al., 2012; Weideman & Nygaard, 2014)

2.2.3 Occurrence and severity

A substantial amount of work has been dedicated to identify well failure mechanisms and failure rates (Davies et al., 2014; King, 2014; King & King, 2013; Wu et al., 2016). For recent shale gas wells, Vidic et al. (2013) derived a figure of 3.4% well barrier leakage for shale gas production sites in Pennsylvania (219 violations for 6466 wells) between 2008 and 2013. Using the same database, Ingraffea (2012) argued that 211 (6.2%) of 3391 shale gas wells drilled in Pennsylvania in 2011 and 2012 had failed. Considine et al. (2013) identified 2.58% of 3533 individual wells as having some form of barrier or integrity failure. This consisted of 0.17% of wells having experienced blowouts (4 wells), venting or gas migration (2 wells), and 2.41% having experienced casing or cementing failures. Measurable concentrations of gas were present at the surface for most wells with casing or cementing violations (Davies et al., 2014).

2.2.4 Detection technologies

Detection technologies prior to decommissioning

Technical or mechanical integrity in the context of Enhanced Gas Recovery (EGR) is determined based on the definition of pressure tight and fluid loss free which the former means there is no "significant leak in the subsurface system" and the latter determines that there is no "significant fluid movement into a higher underground source of the drinking water and the biosphere" (Hou et al., 2010).

To examine the pressure integrity of wells (pressure tight) a casing pressure test (annular pressure test) is typically run. The other accepted methods are annular pressure monitoring and radioactive tracer survey. The verification of loss free can be detected by running radioactive tracer surveys, temperature surveys such as fiber optic temperature surveys, noise-log, oxygen activation log and the cementation logs such as cement bond logs, ultrasonic imager tool (USIT), and isolation scanner (Hou et al., 2010).

To examine the long-term mechanical integrity of wells and test the state of the wells by the passage of time, the state of corrosion and the quality of the cementation should be tested. Table 2-1 summarises the options to assess the damage induced into the wellbore components (Hou et al., 2010).

Table 2-1 Options to examine the damage of the wellbore components (modified from Reinicke & Fichter, 2010)

System	Damage	Survey
Pipe	Corrosion internal	Caliper survey, isolation scanner
	Corrosion	Isolation scanner, elektromagn. wall
	Weight loss	Isolation scanner, elektromagn. wall
	Deformation	Caliper survey, isolation scanner; elektromagn. wall thickness survey
	Leakage	Caliper survey, isolation scanner
Cement	Annulus content	CBL, isolation scanner
	Micro annuli	CBL, isolation scanner
	Channels	Isolation scanner
	Cracks	(isolation scanner)

Detection technologies after decommissioning

Efficient monitoring of decommissioned wells plays a vital role in order to detect and subsequently remedy methane leakage, however, the options are limited due to the removal process of the wellhead (Schout et al., 2019). Two different approaches can be undertaken:

- Surface monitoring technologies/ direct measurements include shallow groundwater monitoring, surface water monitoring, soil gas sampling, soil flux, eddy covariance atmospheric monitoring, surficial scanning, and static chamber measurements (Feitz et al., 2014).

- Subsurface monitoring /indirect measurement including distributed fiber optic sensors, distributed acoustic sensing, and distributed temperature sensing measurements (Mawalkar et al., 2019; Reinsch et al., 2015; Wu et al., 2019) and indirect measurements which highlight the occurrence of CO_2 leakage by the ecosystem stress monitoring, groundwater monitoring, etc.(McMahon et al., 2018; Schout et al., 2019).

2.3 Performance criteria of sealant materials

The conventional Portland cement has been predominantly utilised in oil and gas industry to seal the wellbores and provide zonal isolation (Øyan, 2017). However, the hydraulic and mechanical integrity of the cement may be compromised as a result of exposed to pressure and temperature variations, and corrosive fluids within a wellbore. This emphasises the necessity to look for alternative solutions. The Oil & Gas UK's "Guidelines on qualification of materials for the abandonment of well" (Oil and Gas UK, 2015) provides six categories of functional requirements of permanent well barriers as follows

- *Sealing*. The main function of a permanent well barrier is to provide a seal against movement of fluids, including liquids and gases. The rate of permeation through the barrier should be acceptably low. For example, good quality cement, typically with a permeability of 10 microdarcy, is deemed acceptable;
- *Position*. Once placed, the position of the barrier should not move. The barrier material is required to remain attached to interfaces it has been placed against. This is achieved through sealing stresses normal to the casing, friction stress, bonding at the interface, weight and dimensional stability or a combination of them;
- *Placeability*. It is a requirement that the barrier material can be placed in a wellbore at depth and is subsequently able to perform its required function. The materials should have appropriate properties that allow it to displace the existing fluids and form a continuous sealing medium, even when taking its inevitable contamination into account;
- *Durability*. The barrier materials should not degrade such that its sealing capability or position is compromised. In order to define testing criteria against a quantified service life, a service life of an arbitrary number of a million days (circa 3,000 years) is proposed in this guideline;
- *Removal options and "reparability" concepts*. A key objective of permanent well abandonment is that re-entry into the well should be unnecessary, unless in the rare event that a leak through the barrier developed, there should be a method to remove the barrier in order to remedy the leak; and
- *Absence of environmental harm*. Materials used in the barrier should not be harmful to the environment as deemed by local regulatory requirements. Furthermore, they should not generate substances harmful to the environment when they undergo physical or chemical changes in-situ, either as the result of intended deployment processes (such as curing) or as a result of deterioration.

Alternative materials should outperform the oilfield cements, such as class G, in many aspects. However, there is not any specific functional requirement or performance criteria for well leaking sealants to the authors' knowledge. Following aspects on sealant material performance are often suggested in various publications:

- Ability to penetrate deep and seal the micro-cracks to reduce their permeability. (Todorovic et al., 2016; Tongwa et al., 2013);
- Minimal shrinkage (Genedy et al., 2017);
- Ability to bond with the surrounding materials (Genedy et al., 2017);
- Compatible with other fluid: mud/spacer/formation fluids (Rostoshanshaya, 2019);
- Ability to flow under pressure (desired pumpability and injectivity) (Tongwa et al., 2013); and
- Long-term thermal and chemical stability (Tongwa et al., 2013).

3 Cementitious materials and squeeze cementing

This section reviews cementitious materials that have been applied to rectify well leaking problems. The review covers conventional oilfield cements, micro cement and engineered and optimized micro-cements (collectively defined as micro fine cements in this report), including their advantages and limitations. Furthermore, operational procedure and technologies for conducting well sealing, i.e., squeeze cementing will be briefly described.

3.1 Conventional oilfield cements

Once well leaking is identified to be related to poor primary cementing, such as mud channels, voids, debonding, cement degradation, the conventional approach to rectify the problems is to conduct a squeeze cementing operation. This is accomplished by forcing conventional oilfield cement slurry into an isolated target casing/wellbore annular space through perforations or cut slots (Nelson & Guillot, 2006). Squeeze cementing operation starts with wellbore preparation. If the slurry needs to be injected bottom off, a plug must be installed below the squeeze interval to prevent the slurry flowing deeper downhole. The slurry is pumped through a drill pipe or coiled tubing until the wellbore pressure reaches a predetermined value. In most cases, the tubing is pulled out of the cement slurry before or during the setting period. The next step is to remove excess cement slurry from the wellbore, which is usually performed by reverse circulation.

Squeeze cementing is a dehydration process. When the slurry is pressured against a permeable formation, the solid particles filter out onto the formation face as the aqueous phase (cement filtrate) enters the formation matrix (Figure 3.1). The cement filter cake allows the well to withstand the squeeze pressure. A properly designed squeeze job causes the resulting cement filter cake to fill the openings between the formation and the casing (Nelson & Guillot, 2006). Upon curing, the cake forms a nearly impenetrable solid (Suman and Ellis, 1977).

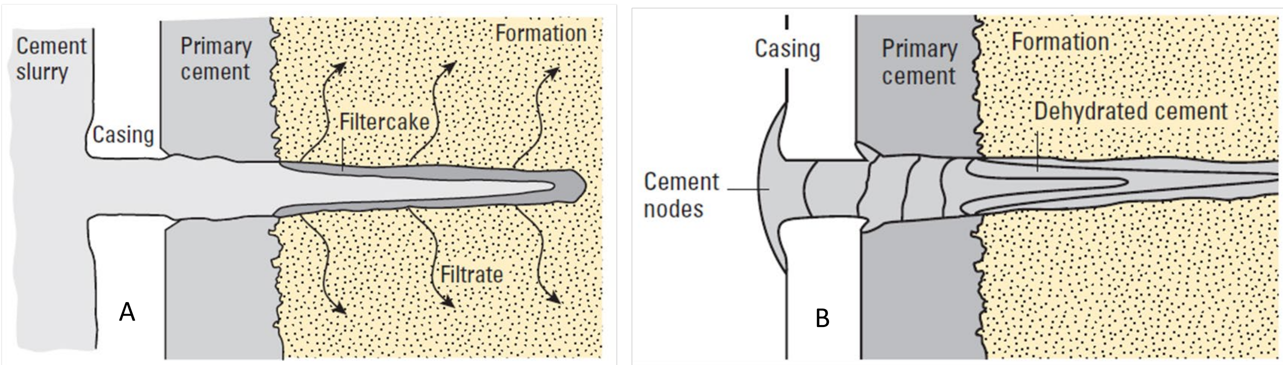

Figure 3.1 Filtercake buildup into a perforation channel (a) and perforation channel properly filled with dehydrated cement from (Nelson & Guillot, 2006)

In addition to remediating the defect primary cementing jobs, squeeze cementing is applied to repair casing/liner leaks, seal lost circulation zones during drilling, water shut off, isolation of gas or water zones and well abandonment.

While squeeze cementing using conventional oilfield cements has successfully applied to repair defect primary cement jobs, it has been proved ineffective in sealing microfractures/microannuli which often have an aperture much less than 150 μm (Normann, 2018). For example, due to cement shrinkage, the induced aperture of the microannuli between cement sheath and formation rock could typically be in the order of 10 – 20 μm (Dusseault et al., 2000). Despite their small sizes, these fractures are sufficiently large to be conduits for gas flow, resulting in potential well leakage. The Portland cement slurries are suspensions with the largest particle size in the range of 100 μm to 150 μm (Figure 3.2), as such they have at best small penetration into narrow fractures due to aperture bridge-off and dehydration at or close to the point of entry. Ewort et al. (1990) demonstrated that conventional oilfield cement slurries will not penetrate fractures narrower than about 400 μm.

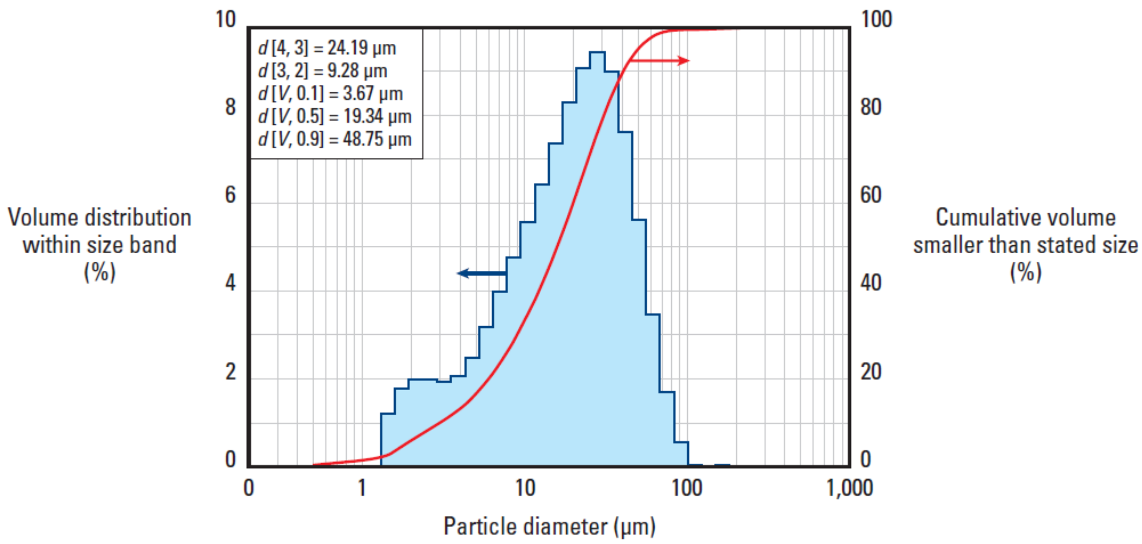

Figure 3.2 Typical Portland cement particle size distribution (Nelson & Guillot, 2006)

3.2 Micro fine cements

3.2.1 Conventional micro-cements

Small particle size Portland cement or micro-cements have been commercially available for a long time, however, it was not until early 1990's the micro-cement was applied to oilfield production operations. The development of the micro-cement technology was based on the simple premise that minimizing particle sizes in a cement slurry would maximize the slurry's penetration capability. In this manner, tight leaks in a production well previously inaccessible to conventional oilfield cement slurry can now be repaired by micro-cements that can enter the leak (Meek & Harris, 1993). Figure 3.3 shows particle size distribution of a typical micro-cement and compared with the Class H cement.

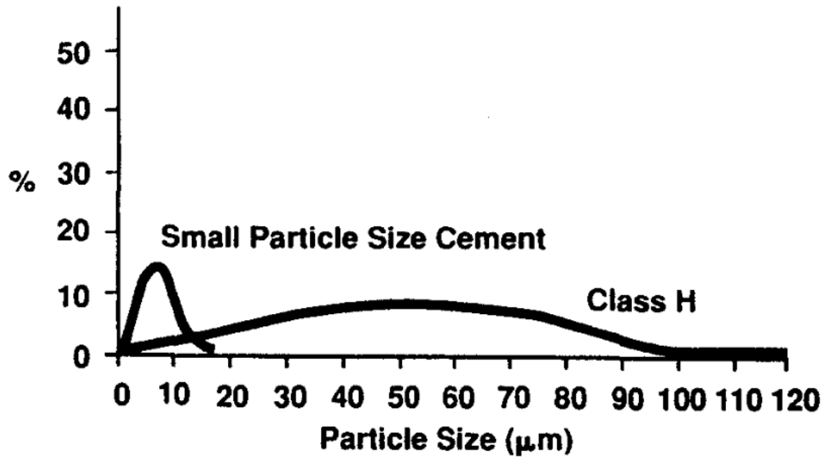

Figure 3.3 Particle size distribution: small-particle size cement vs Class H cement (Meek & Harris, 1993)

Laboratory injectivity tests on sand packs by Ewort et al. (1990) demonstrated that the micro-cement slurry could penetrate and seal a 40/60 gravel pack while the conventional oilfield cement slurry failed. Further laboratory gap penetration experiments reported by Meek & Harris (1993) showed that micro-cement slurry with density of 12ppg could penetrate a slot width of 0.01” (250 μm) without bridge or form a filter cake at the slot.

Tongwa, et al. (2013) conducted an experimental study to evaluate potential fracture sealing materials for remediating CO_2 leakage pathways in well cement. The study compared the performance of the four sealant materials, i.e., micro-cement, polymer, paraffin wax and silica gel in sealing fractures. The performance of the materials was assessed in terms of sealed fracture permeability, long term thermal and chemical stability, sealed fracture integrity after CO_2 injection and sealed fracture strength. The fracture aperture ranged from 0.25mm up to 1mm. Whilst all the four sealant materials could reduce the fracture permeability significantly, however, the micro-cement was the most effective sealing materials and the only sealant that was able to withstand the large differential pressure due to CO_2 or brine injection. Based on the performance, micro-cement was recommended as the material of choice to seal CO_2 leakage pathways for fractures with a width larger than 0.5mm (Tongwa et al., 2013).

Table 3-1 summarises some of the laboratory studies on micro-cement slurries and their performance in sealing fractures.

As shown in Figure 3.3, the micro -cements for oilfield application have a maximum particle size in the range of 20 – 30 μm and a median diameter of 10 μm or less (Nelson & Guillot, 2006). Conventional wisdom suggested that a slurry will penetrate a porous medium or fracture aperture without bridging if the maximum particle size in the slurry is 1/3 to 1/5 or less than the size of the aperture (Farkas et al., 1999). However, this experience is only valid for the inert materials that have no particle to particle interaction other than that of physical bridging. Cement powders exhibit substantial surface interaction between particles due to electrostatic charges along the particle surfaces. Furthermore, in addition to the small particle sizes, high resistance to slurry dehydration, low fluid loss and low rheology are required to penetrate and seal narrow fractures (Morris et al., 2006). Figure 3.4 shows a schematic of slurry fluid loss in both radial and axial directions in penetrating a narrow fracture.

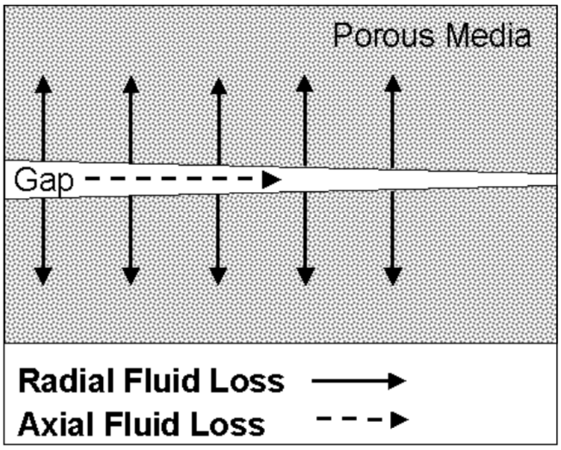

Figure 3.4 Schematics of slurry fluid loss in penetrating a narrow fracture (Slater et al., 2001)

These interactions, together with viscosity increase due to fluid loss while penetrating a small fracture, could create an effective particle size much larger than the actual grain size. The gap injection testing by Farkas et al. (1999) demonstrated that cement slurries may prematurely bridge and dehydrate in openings that are 10 times wider than the largest particle size. Considering the surface charge interactions between cement particles, this means that even conventional micro cements can be limited in penetrating a gap of less than 300 μm in width (Farkas et al., 1999).

3.2.2 Engineered and optimized micro-cements

Engineered micro-cement slurry (EMS) was developed to address difficult squeeze cementing operations through small restrictions. The EMS slurries are simply composed of cement that has been ground to a finer particle size that is 3-10 times smaller than the standard oilfield cement. Furthermore, the EMS slurry addresses issues related to fluid loss and slurry rheology (Farkas et al., 1999). The slurry performance is enhanced by increasing the packing volume fraction of the solids. This is achieved by using an engineered particle size distribution, smaller than the cementitious material. A properly selected particle size distribution allows the smaller particles to fit inside the void spaces of the larger particles. As a result, the EMS slurry has a less porosity and permeability for a given density. The other improvements in the performance of EMS slurry over the conventional micro-cement slurry include more stable slurry, an almost zero free water, better control on fluid loss, lower rheological properties, resulting in easier placement and better injectivity. The laboratory injection tests demonstrated that the EMS slurry is capable of penetrating gaps as small as 120 μm with ten-times deeper penetration than conventional micro-cements (Farkas et al., 1999).

Based on the same physics of particle packing as the EMS, the optimized micro-cement system (OMS) was developed to obtain slurry and set cement properties that are suitable for sealing extremely narrow voids (Slater et. al., 2001). The cement particles used in the OMS has a maximum particle size of 7 to 10 μm. Instead of filling the pore space between the cement particles with water for conventional micro-cement slurries, the pore space is filled with solid non-cement particles with two distinctly smaller particle sizes. The first constituent of solid particles has a size in the range of 0.5 to 1.0 μm and the second has a maximum particle size of 1 nanometre. The OMS slurry is normally mixed at a density of 1.68 SG with an API fluid loss of less than 15 mL/30min., a plastic viscosity less than 50 cP and yield stress less than 2.5 Pa. The injectivity tests using a laboratory fracture model demonstrated that the OMS can penetrate fractures with an aperture of 120 μm (Slater et. al., 2001).

Table 3-1 summarises some of the laboratory studies on engineered and optimized micro-cement slurries and their performance in sealing fractures.

3.3 Remediating well leakage using micro fine cements

The early application of the micro fine cement technology to oilfield included sealing gravel packed annuli to shut off steam migration, unwanted water flow and depleted reservoir zones (Ewort et al., 1990). Subsequently, the technology was applied to repair small casing leaks (Meek & Harris, 1993). These leaks were so restrictive that repairs by conventional oilfield cement slurry squeeze often failed. A large number of successful field case histories on application of micro-cements or micro fine cements were reported (Meek & Harris 1993, Heathman & East, 1992), demonstrating the advantages of using such cement slurry to repair tight casing leaks in comparison with the conventional oilfield cement slurries.

The EMS technology had numerous field applications, including isolating old and non-productive perforations, sealing open channels behind the casing, water control and mitigating annulus gas migration (Farkas et al., 1999).

The OMS together with the ultra-low-rate placement technique was specifically developed for sealing gas vent flows outside the surface casings in Western Canada. Low squeeze rates are usually considered in the range of 0.25 to 0.5 barrel/min (BPM), whilst the ultra-low-rate is in the range of 10 litre/min (0.06BPM). Several successful field case histories were reported (Slater et al., 2001). Often, prior to the OMS squeeze, conventional squeeze cementing was carried out, but failed to seal the gas migration outside the casings.

Table 3-2 provides a summary of field case histories on application of micro fine cements to remediate well leakage.

Figure 3.5 shows a typical field example of gas migration behind casing and remedial cementing required to provide annular isolation for an offshore leaking gas well (Morris et al., 2006). After several years of production, gas bubbles appeared in various locations from seafloor in the Duyong field offshore east of peninsular Malaysia. Shallow seismic study showed gas charging in several shallow gas layers throughout the field. Duyong B-4 well was identified as one of the probable contributing wells to the shallow gas. Both cement bond logs and ultrasonic imaging logs detected gas and fluid channels behind the 9 5/8” production casing extended from a depth of 1047 to 1072 m AH. and the R7 sand gas reservoir was the source for the gas flow behind the casing. Consequently, the production casing was perforated between 1055 and 1056 m AH with the bridge plug set at 1058 m AH. The total OMS slurry volume squeezed into the microchannel was 0.85 m^3. Following the squeeze cementing operation, the cement integrity behind the casing from 1071 to 1047 m AH was re-evaluated by cement bond logs and ultrasonic imaging logs. The well logs revealed that the water in the identified channel was totally displaced by the OMS, hence demonstrated the success of the treatment.

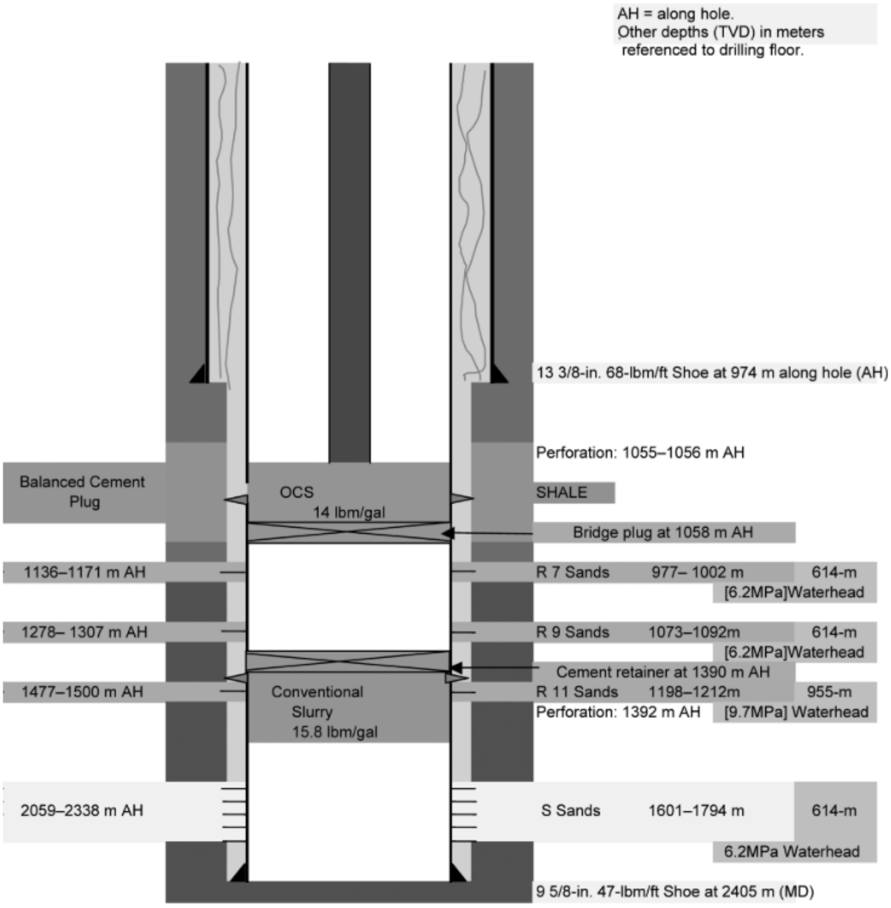

Figure 3.5 An example of the OCS field application to seal gas flow behind casing – Duyong B-4 well schematic (TVD=true vertical depth) (Morris et al., 2006)

The advantage of using of micro-cement and its variations, such EMS and OMS is that Portland cements are used so commonly and are so widely accepted that this method will require almost no familiarization other than product awareness, laboratory quality control (QC) and testing procedures, and general availability. Because no new equipment or specialized training of service personnel is necessary, the micro fine cement technology is expected to remain one of the options for remediating well leakage.

Table 3-1 Laboratory studies of micro fine cement slurries

Micro fine cement type	Particle size Max, D50	Slurry density (SG)	Viscosity (cP)	Yield strength (Pa)	Fluid loss (ml/30min)	Leak size or Fracture aperture (μm)	Reference
Micro-cement	23.6, 8.5	1.52	6	1.4		40/60 mesh gravel packs	Ewort et al., 1990
Micro-cement	27, 9.2	1.38-1.5		2.0-2.6		254	Meek & Harris 1993
Fondu micro-cement						250 - 1000	Tongwa et al., 2013
Microfine cement – 1 (slog material)	11-15, 4,	1.32-1.5				40/60 mesh gravel pack	Heathman & East, 1992
Microfine cement – 2 (Portland cement)	8-15, 8	1.32-1.5					Heathman & East, 1992
EMS	Max particle size 3-10 times smaller than oilfield cement	1.68	< 40	<1.43	< 20	160	Farkas et al 1999
OMS	Max particle size 7 to 10 μm with non-cement particles 0.5 to 1 μm and nano particles	1.68	<50	<2.5	15	120	Slater et al., 2001

Table 3-2 Case histories of micro-cement application to repair leaking oil and gas wells

Micro fine cement type	Field application	Reference
Microfine 1 & microfine 2	Casing leak repair; channel repair behind casing to reduction water production; seal 40/60 gravel packs to reduce gas cut; seal water encroaching formation during drilling; seal old perforations to block water production. Over 500 remediation jobs reported	Heathman & East, 1992
Conventional micro-cement	Seal tight casing leaks. Over 100 successful applications in US reported	Meek & Harris, 1993
EMS	Seal non-productive perforations; Seal channel behind casing connecting producing interval with water producing zone; seal microannuli behind casing to reduce water production. The technology was successful in more than 40 wells in New Hope field, East Texas. Good success in shallow gas fields in South Eastern Alberta, heavy oil fields in North Eastern Alberta, Canada	Farkas et al 1999
OMS	Seal microannuli outside surface casing to stop gas vent flow and gas migration in Western Canada where tens-of-thousands of wells were leaking gas between the surface and production casings.	Slater et al., 2001
OMS	Remediating sea floor gas bubbling due to annulus gas flow behind production casing, offshore, peninsular Malaysia	Morris et al., 2006
Special fine cement	Seal gas leaks in different casing annuli. The integrity problem resulted in oil/gas bubbles observed on the seabed around the well conductor and high sustained casing pressure, offshore UAE	Ibrahim et al., 2011
EMS	Improve zonal isolation behind casing between oil producing and gas bearing zones in Tiung Biru field in East Java, Indonesia	Prasetyo et al., 2014
Micro-cement	Intermediate casing shoe squeeze, Marlim field at the Campos Basin, Brazil	Freitas et al., 2019

3.4 Squeeze cementing operation

Prior to squeeze cementing, a series of decisions must be made to determine a) if a problem exists; b) the magnitude or severity of the problem; c) if the squeeze cementing will correct the problem; d) the risk factors associated with the squeeze job; and e) if it is economically feasible ("Remedial cementing - PetroWiki," n.d.). Once the squeeze cementing is deemed necessary, the wellbore and perforations are prepared for the operation. A cement slurry is designed, and prepared on the surface, and pumped down the wellbore via either a drill pipe or coil tubing to the area of squeeze target. The area is isolated, and pressure is applied from the surface to effectively force the cement slurry into all voids. Excessive cement slurry is cleaned out of the wellbore by reverse circulation. The slurry is designed specifically to fill the type of void identified in the wellbore, such as small cracks or micro-annuli, casing split or large vug, formation rock or another kind of cavity.

3.4.1 Squeeze pressures

There are many variations on the general squeeze cementing procedure and they are specifically tailored to the operational objectives, available equipment and local practice. In terms of bottomhole treating pressures, the procedure is broadly categorised as low pressure squeeze and high pressure squeeze (Nelson & Guillot, 2006).

The aim of a low pressure squeeze operation is to fill perforation cavities and interconnected voids with cement filter cake. The cement-slurry volume is usually small, because no slurry is actually pumped into the formation. Precise control of the pump pressure and the hydrostatic pressure of the cement column is essential because excessive pressure could result in formation breakdown. In low pressure squeeze operation, it is essential that perforations and channels be clear of mud or other solids. This may be the case if the well has been producing; however, for newly completed wells, it may be necessary to clean the perforations before performing the squeeze job.

In some cases, a low-pressure squeeze of the perforations may not accomplish the job objective. For example, the channels behind the casing may not be directly connected to the perforations. Small cracks or microannuli that may allow gas flow do not allow the passage of a cement slurry. Such channels must be enlarged to accept viscous solids carrying fluid. In addition, many low-pressure operations cannot be performed if it is impossible to remove plugging fluids or debris ahead of the cement slurry or inside the perforations. In such cases, cement-slurry placement behind the casing is accomplished by breaking down, or fracturing, the formation at or close to the perforations. Fluids ahead of the slurry are displaced in the fractures, allowing the slurry to fill the desired spaces. Further application of pressure dehydrates the slurry against the formation walls, leaving all channels (from fractures to perforations) filled with cement cake.

3.4.2 Pumping methods

There are two pumping methods during a squeeze cementing job, i.e., running squeeze method and hesitation squeeze method (Nelson & Guillot, 2006). During a running squeeze procedure, the cement slurry is pumped continuously until the final desired squeeze pressure is obtained, which can be lower or higher than the formation fracturing pressure. The pressure is monitored after pumping stops. If the pressure falls due to additional filtration at the cement/formation interface, more slurry is pumped to maintain the final squeeze pressure. This continues until the well maintains the squeeze pressure for several minutes without additional cement-slurry injection. The volume of slurry injected is usually large, ranging from 10 to 100 bbl (Rike & Rike, 1982). A modified running squeeze technique, using pumps that can deliver several barrels of slurry at rates as low as 0.06 bbl/min (10 L/min) to avoid formation fracturing, has been used successfully to seal narrow microannular gaps (Slater et al., 2001).

During a squeeze cementing job, the rate at which cement filtrate leaks into the formation can be lower than the minimum pump rate of most field equipment. Therefore, maintaining a constant differential pressure is nearly impossible, especially for low pressure squeeze. A solution to this problem is the hesitation squeeze pumping method. This procedure involves the intermittent application of pressure—by pumping at a rate of 0.25 to 0.5 bbl/min - separated by an interval of 10 to 20 min for pressure falloff caused by filtrate loss to the formation. The initial filtrate leakoff is normally fast because there is no filtercake on the formation wall. As the cake builds up and the applied pressure increases, the filtration periods become longer and the differences between the initial and final pressures during the filtration period become smaller. At the end of the squeeze job, the pressure falloff becomes negligible. The slurry volumes necessary for this technique are usually much less than those required for a running squeeze job.

3.4.3 Squeeze equipment

When there is no concern on the casing's ability to withstand the squeeze pressure, a low pressure squeeze may be performed using the Bradenhead squeeze technique (no packer) (Nelson & Guillot, 2006). No special tools are involved, although a bridge plug may be required to isolate other open perforations farther downhole. Figure 3.6 illustrates the principal steps of the Bradenhead squeeze technique.

Prior to the squeeze operation, a bridge plug is installed below the perforation interval to ensure the cement slurry is spotted in the target zone and to isolate the deeper section of the well from the squeeze pressure. An open ended tubing is then run to the bottom of the target zone (Figure 3.6a). Blowout preventer (BOP) rams are closed over the tubing. Following an injection test with a clean wellbore fluid, the cement slurry is spotted in perforated zone. Once the required volume of cement slurry is in place, the tubing is pulled out to a point above the top of the cement slurry and the BOP rams are closed. The squeeze operation is then performed using either the running squeeze or hesitation squeeze method. Once the designed squeeze pressure has been achieved and the squeeze operation is complete, the excessive cement slurry is cleaned out of the wellbore by reverse circulation. The Bradenhead squeeze method is popular because of its simplicity and no special tools required.

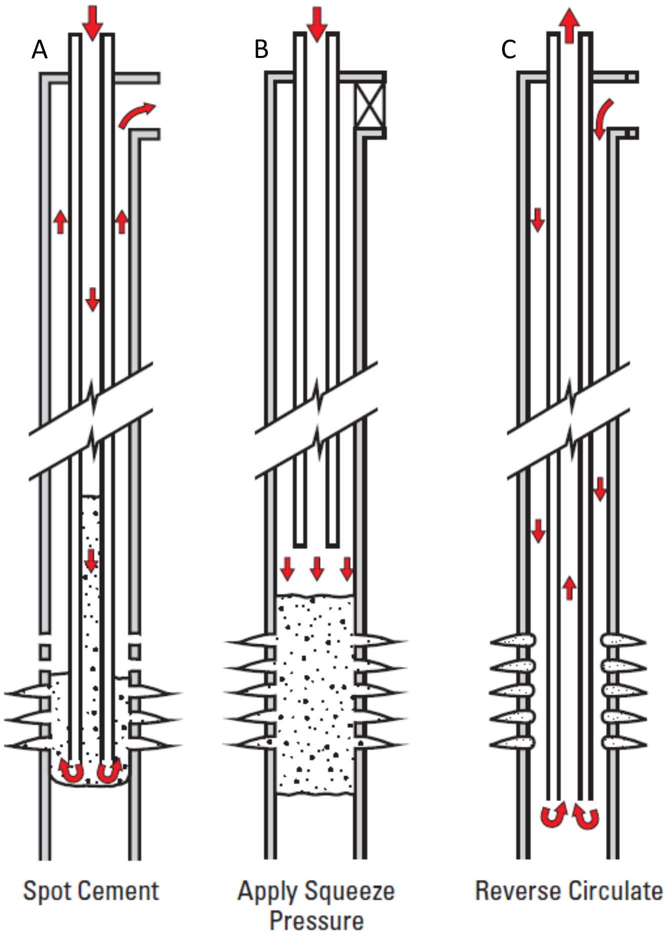

Figure 3.6. Bradenhead squeeze technique, a) spot cement; b) apply squeeze pressure; and c) reverse circulation (Nelson & Guillot, 2006)

When there is a concern on the casing's ability to withstand the squeeze pressure, the squeeze-tool placement technique can be employed (Nelson & Guillot, 2006). The main objective of using squeeze tools is to isolate the casing and wellhead from the squeeze pressure while applying high squeeze cementing pressure downhole. This technique can be subdivided into two parts, i.e., the retrievable squeeze packer method and the drillable cement retainer methods. The details of the two methods are provided in (Nelson & Guillot, 2006).

The preferred placement method for squeezing gas migration and vent flows is the Bradenhead method because it minimizes slurry contamination during placement and is simple to apply. The drawback of the method is that the squeeze pressure is imposed along the entire length of the casing above the bridge plug. Should the gas migration path be a microannulus, as is often the case, then the pressure applied to the inside of the casing during squeezing will act to expand the casing (Figure 3.6), closing-off the channel and limiting penetration by the slurry. The ultra-low-rate technique (in the range of 10L/min or less) minimizes the pressure on the inside of the casing and therefore minimizes the degree to which the microannular gap is narrowed.

4 Temperature-activated polymer resins

As demonstrated in Section 3, conventional oilfield cements and micro fine cements have long been applied to repair cement related leaking wells. However, they present limitations in sealing small annuli behind the casing. Polymer resins can be more advantageous comparing to the cementitious materials in regards to the following aspects (Beharie et al., 2015):

- More flexible and gas tight;
- Solid free and perceived ability to penetrate deeper and seal much narrower fractures;
- Manageable curing time and tuneable viscosity;
- Higher compressive, tensile, shear and bonding strengths;
- Higher tolerance to contamination by wellbore fluids; and
- Higher stability and durability at elevated temperature

Polymer resins also have some benefits in terms of field placement considering its solid free formulation which allows penetration into the geometries that may not be possible for cement slurries. In addition, the controllable setting time and in some cases controllable viscosity prevents premature setting and allows pumpability (Beharie et al., 2015).

This section reviews some of the important properties of the temperature-activated polymer resins and their field applications in sealing leaking oil and gas wells.

4.1 Performance of temperature-activated polymer resins

Polymer resins can be physically characterized as free flowing polymer solutions that can be irreversibly set to hard, rigid solids (Morris et al., 2012). The polymer resin systems consist of base resin and curing agent (or hardener). Temperature-activated polymer resins are formulated to start curing at a specific curing temperature. Several factors affect the reaction between the resin and hardener (Rostoshanshaya, 2019):

- Concentration and type of both the resin and the hardener will affect the setting time and the speed of the reaction; and
- Curing temperature affects the curing reaction. The higher the temperature, the faster the reaction, and in general stronger the final product.

There are various additives that can be used to control the properties of the temperature-activated polymer resins based on a specific field application (Knudsen et al., 2014) as follows

- Curing initiator: a liquid chemical added to the resin system that ensure the reaction occur at certain temperature;
- Accelerator: a liquid chemical added to speed up the curing process, usually when the temperature is low;

- Inhibitor: a liquid chemical added to slow the curing process time;
- Viscosifier: a chemical added to increase the viscosity. It is a heavy material mixed with the resin system that provides the rheological properties required;
- Diluent: a chemical added to decrease viscosity of the resin if required (Alsaihati et al., 2017); and
- Weight fillers: solid materials can be selectively added to achieve a specific density. They could be used to either increase or decrease the density of the system as required.

Many studies have been conducted to investigate the properties of polymer resins. Alsaihati et al. (2017) conducted a laboratory experimental study on the rheology, thickening time and mechanical properties of the two epoxy resin systems deployed for remedial operations in Saudi Arabia. The study demonstrated that the viscosity of the resin systems can be modified by using a reactive diluent based on downhole conditions. As the resin system transfers from liquid to solid, the pressure gradient in the resin system is maintained until it becomes completely solid when there will be no hydrostatic pressure transmission through the resin solid. This capability of hydrostatic pressure transmission as the resin system transfers from liquid to solid is particularly important to prevent formation gas influx into the annulus and the resin system, which can sometime occur during the curing of conventional oilfield well cements (Kolstad & Mozil, 2004). The tuneable rheological property of epoxy resin and the fact the material is solid-free, allow the epoxy resin to penetrate deep into very small fractures (Todd et al., 2018 and Alkhamis et al., 2019). Furthermore, the density of epoxy resin slurries can be controlled by adding weight fillers to reach a specific weight as demonstrated by Knudsen et al. (2014). The flexibility to formulate different densities of epoxy resin system based on application is an advantage for sealing a wellbore in the formation with a low formation fracture pressure gradient (Sanabria et al., 2016).

In addition to the pre-cured properties, the cured solid polymer resin typically has a high mechanical strength (Elyas et al., 2018) (Al-Yami et al., 2019), can resist significant strain prior to failure (Khanna et al., 2018) and develop good bond strength with casing (Jimenez et al., 2016, Genedy et al., 2017 and Morris et al., 2012), in comparison with conventional oilfield cements. The property range for some of the important properties of the temperature-activated resins are summarised in Table 4-1, whilst a comparison of the properties between the epoxy resins and cements are provided in Table 4-2.

Furthermore, solid epoxy resin can provide high stability and durability at high temperature, therefore maintaining long term sealing reliability. The ageing and verification tests reported by Beharie et al. (2015) demonstrated that the properties of cured resins have key elements that allow them to withstand the chemical compositions typically found in a wellbore environment, and still maintain a reliable integrity. The ageing tests were conducted by exposing the cured resin samples to crude oil, methane gas, CO_2, and H_2S at a temperature up to 130 ^oC and pressure up to 7250 psi over a period of 12 months (Beharie et al., 2015). The resin properties evaluated on post-ageing samples included permeability, compressive and flexural strength with typical properties summarized in Table 4-3 (extracted from Beharie et al., 2015).

The study on thermal degradation kinetics of epoxy resins by Al-Yami et al. (2019) further showed that reaching 10% mass loss by thermal degradation can take more than 160 years, which is beyond the operational life of typical oil and gas wells. It should be noted the presence of any chemicals in the environment of the well could increase the rate of degradation so careful studies looking at the interaction of the polymer resin with reservoir fluids are required.

In terms of field placement of the epoxy resins in remediating leaking wells, the controllable setting time and viscosity of the epoxy resin system will prevents premature setting and allows pumpability (Beharie et al., 2015). Furthermore, the epoxy resins can tolerate high contamination by wellbore fluids (Perez et al. 2017 and Ziashahabi et al., 2019). For example, the epoxy resin system developed for deepwater application was compatible with up to 20% of synthetic based drilling fluid (Morris et al., 2012).

Table 4-1. Properties of thermal activated resins (modified from Knudsen et al., 2014)

Property	Range
Specific gravity (SG)	1.03 – 1.05
Density (g/cm^3)	0.75 – 2.5
Viscosity (cP)	10 – 2000
Setting time (min)	3 to as long as required
Miscible with water or well fluids	No
Pumpable through tubing/drill pipe/BHA/Bit	Yes
Target temperature (^oC)	9 – 135
Temperature resistance up to (^oC)	480

Table 4-2. Comparison of hydraulic and mechanical properties between thermal activated resin and traditional cement (modified from Knudsen et al., 2014 and Beharie et al., 2015)

Property	Thermal activated resin	Traditional cement
Water permeability (mD)	$0.5x10^{-6}$	$1.6x10^{-3}$
Compressive strength (MPa)	77	58
Flexural strength (MPa)	43	10
Failure flexural strain (%)	1.9	0.32
E-modulus (MPa)	2,240	3,370
Tensile strength (MPa)	60	1
Density (g/cm^3)	0.75 – 2.5	1.5 +
Right angle setting	Yes	No

Table 4-3 Ageing test results of cured epoxy resin after exposed to different chemicals at 130 ^{o}C for 12 months

Property	Initial value	Crude oil 38^{o}API (7250psi)	Methane gas (7250psi)	CO_2 5% in N_2 (7250psi)	H_2S 5000ppm (145psi)
Permeability (nD)	<0.5	<20	Not possible	Not possible	666
Comp. strength (MPa)	77±5	37±1	84±9	76±3	50±1
Flexural strength (MPa)	43±3	20±2	60±3	38±2	29±6

4.2 Remediating well leakage using temperature- activated resins

Despite the ability of polymer resins to offer superior properties over the conventional oilfield cements, such as superior bonding, resistance to corrosive and caustic environments, enhanced mechanical properties, their application prior to 2010's was quite limited as well sealing materials. Recent technological advances in optimal control of rheology, density and curing time required for proper downhole placement have accelerated the application of the resin to remediate leaking wells (Morris et al., 2012). The liquid form of neat resin free of any solid particles, together with excellent thermo-chemo-mechanical and bonding properties provided by this advancement makes the epoxy resin suitable for repairing tight well leaks that would not be possible using conventional cements (Urdaneta et al., 2014 and Beharie et al., 2015). Since then, epoxy resin applications have significantly increased for remediation and mitigation in leaking oil and gas wells. Table 4-4 provides a summary of some of the case studies using the temperature-activated epoxy resin to repair tight leaking oil and gas wells and the associated properties of the resin systems. The case studies demonstrated the effectiveness of the materials in sealing micro fractures and channels in cement sheath, sustained casing pressure (SCP), gas migration behind casings and casing leaks.

Table 4-4 Case studies of temperature-activated epoxy resin application to repair leaking oil and gas wells

Case study by	Field application	Bottomhole temp. (°C)	Viscosity (cP)	Density (SG)	Gelling time (hour)	Compressive strength (MPa)	Injection volume (bbl)	Pump rate (bbl/min)
Jones et al., 2013	SCP behind intermediate casing	66	~28 Bc		3 hours 40 mins	10.3 @24 hours	10	
Urdaneta et al., 2014	SCP in annuli behind 13 3/8" and 9 5/8" casings	72		1.14		86	5	2
Al-Ansari et al., 2015	Micro channels in annulus behind 9 5/8" casing	40	-	1.03 – 1.05SG	2 hours at 40 °C	77 MPa	1.13	
Sanabria et al., 2016	Leakage in 7" casing	79			3 hours @ 80 °C		20	3
Ali et al., 2016	Leakage in 4.5" liner hanger assembly	87	170 @ 87 °C & 300 @ 27 °C		2.5 hours @87 °C	> 41.3 @ 87 °C	12	
Perez et al., 2017	Channels in cement sheath behind 7" production liners	58		1.11	10 hrs to 100 Bc		15	5
Alsaihati et al., 2017	SCP, channels and de-bonding behind 9 5/8" casing in gas wells	99	34	1.1	8 @ 81 deg C	10.3 @ 87 °C cured for 1 hr	20	4
Elyas et al., 2018	Gas migration in micro channels behind surface casing	24	164 at 24 °C		2.5 hours at 24 °C & 2 hours at 93 °C respectively			
Khanna et al., 2018	Channels in cement packer behind production tubing (aperture 9.8mm)	83	50	1.1		80 @ 24 hrs	15.73	4.5
Alanqari et al., 2019	Leakage in 7" casing	57	25 @ 21 °C	1.28	3 @ 57 °C		20	4

Blanc & Brunherotto, 2019	Leakage behind 10 ¾” liner	53	-	-	9 hours to 100 Bc	52.8 cured for 24 hours	27	4
Sun et al., 2019	Leakage in 3.5” casing in gas wells	54		1.1	6 hours to 100 Bc	34.5 -103	1.9	0.63

5 Other materials

As demonstrated in Sections 3 and 4, the conventional oilfield cements and micro fine cements, and more recently polymer resins are the main types of sealant materials that have been applied to mitigating and remediating leaking wells in the oil and gas industry. However, there are many novel materials and commercially available products that have potential to replace wellbore cement as an alternative material for remediating well leakage due to cement sheath failure (Todorovic & Cerasi, 2019). Based on the performance criteria discussed in Section 2, the requirements for such alternative materials are low or negligible permeability, long-term durability, non-shrinkage, ductility and chemical stability. This section provides a brief review on some of the alternative materials. Table 5-1 presents a summary of such sealant materials and their laboratory performance.

5.1 Nano-technology enhanced sealant materials

5.1.1 Epoxy-based composite materials

Thermoset polymers, in particular Novolac-based epoxies, can improve the bond strength when used as well repair materials. In addition, by adding Aluminium nanoparticles (ANPs) to the polymer resin matrix, the mechanical properties and durability of the repair materials can be enhanced massively (Genedy et al. 2014 and 2017). Their experimental study on the incorporations of ANPs into the resin matrix demonstrated that:

- Using ANPs-epoxy nanocomposites (5% ANPs by weight of the epoxy resin) as repair materials improve the bond strength significantly;
- ANPs-epoxy nanocomposite can penetrate the porous space of the shale surface and builds an epoxy network which enhances the sealability between cement and shale; and
- Microstructural analysis including Dynamic Mechanical Analysis (DMA) and Fourier Transform Infrared (FTIR) showed that the limited cross-linking makes the epoxy to be more flexible which is very beneficial facing shock waves, temperature and moisture variations.

Though nanoparticles confer additional mechanical benefits to the resin care must be taken with nanoparticles which pose health risks under certain circumstances so this must be factored in when designing any resin.

5.1.2 Graphite nanomaterials for enhanced performance of oilfield cement

Low-cost graphite nanoplatelet (GnP) and carbon nanofiber (CNF) can be utilised as nano-scale reinforcement to improve the tensile strength and toughness resistance of the oilfield cement.

The addition of GnP and CNF can also develop the shrinkage resistance which leads to less microcracks and consequently less well leakage (Peyvandi et al. 2017).

5.1.3 Nanosealant

A nanosealant material has been developed as an alternative to in-situ polymerization technologies, such as polymer resins and monomers for sealing leaking pathways smaller than 120 μm (Todd et al., 2018). The nano-technology based sealing fluid is a low viscosity, single component, nanoparticle fluid. Some of the advantages of the nanosealant material are:

- It can enter and effectively seal pathways as small as 20 μm with the ability to work in gaps up to 1000 μm. The sealed gaps were able to sustain a hydraulic pressure gradient greater than 1,000psi/ft;
- The material is a single component and activates only upon contacting with divalent ions, which includes set cement. This reduces the complexity of fluid formulation and simplifies the on-location deployment; and
- The material has a low HSE foot print and is ease to drill out in comparison with polymer resins.

5.2 Gels

Gel treatments are mainly incorporated in oilfield applications to control the conformance and to prevent water or gas challenging within reservoirs (Bai et al. 2004). Preformed particle gels (PPG) is an advanced super adsorbent polymers (SAPs) with the capabilities to absorb liquid more than a hundred times of their weight and will not release the absorbed liquid under pressure (Bai et al. 2008).

5.2.1 Polymer-based gels

Tongwa et al.,(2013) evaluated the performance of polymer-based gels and silica based-gels in terms of their ability to seal the fractures and its permeability, long-term thermal and chemical stability, the integrity of the sealed fractures after CO_2 injection, and the strength of the sealed fractures. According to this study the aforementioned materials have the ability to seal fractures with widths from 0.25 mm to 1 mm to reduce permeability significantly, and the strength of the intact samples was not achievable when compared to the samples with sealed fractures.

5.2.2 pH-triggered polymer gelant

pH-triggered polymers are aqueous solutions with low viscosity at low pH, comprising pH-sensitive microgels (polyacrylic acid) which viscosify consequent to neutralization and turns into highly swollen gels with substantial yield stress that can block fluid flow (Tavassoli et al., 2018). The alkalinity of cement (pH≈13) in wellbore provides the suitable environment for this gelant to start transitioning from water like structure to a gel structure.

According to the study by Tavassoli et al., (2018), the decrease in fracture aperture can lead to an increase in holdback pressure gradient (expect two regions in which the aperture size is 100‑200 µm and >400 µm) and the polymer concentration should be modified based on the fracture aperture size, which may be difficult to obtain in the field.

5.2.3 Micro-sized crosslinked polymer gel

Crosslinked polymer gels are mainly applied for conformance control in oilfield. The potential for employing the micro-sized crosslinked polymer gel to seal fractures in cement sheath was investigated by Abdulfarraj & Imqam (2019a,b). According to this study, the polymer gel particles show an acceptable injectivity performance through small fracture width and crack features. However, their plugging performance to water leakage is limited to less than 100 psi for a fracture width of 0.5 mm. This failure pressure could be controlled by managing the gel strength, but only to a certain level. The gel particles propagate piston-like in a fracture when the gel particle size to fracture width ratio was larger than 1. However, it propagates with different angles where this ratio was equal to or below 1. Also, the existence of brine makes the gel particles to propagate with smaller angles and due to the swelling ratio, the gels develop higher strength. Therefore, design a sealant material with micro-sized crosslinked polymer gels requires considering different factors and tailoring for each purpose including gel strength versus gel injectivity, brine concentration and fracture width size.

5.3 Geopolymer

Geopolymers are classified as polymers due to the chains and repeating units in their structures with cementitious base and also as inorganic polymers since in their structure carbon elements are replaced by aluminosilicate minerals. The geopolymers are produced by mixing tetrahedral aluminosilicate minerals with hardener. The hardener is a mixture of alkali silicate solution and alkali solution (Khalifeh et al., 2019).

The geopolymers are shown to be very advantageous because of their high compressive strength, high durability encountering corrosive environment, low shrinkage, tolerance to contamination of oil-based muds, high ductility, and their stability at high temperature (Salehi et al., 2017; Khalifeh et al., 2019).

Geopolymers are required to be activated utilising a range of alkali activators such as liquid sodium hydroxide (LSH), liquid sodium silicate (LSS), solids sodium silicate (SSS), and liquid potassium silicate (LPS) which leads to different effects on the geopolymer slurry properties, setting behaviour and strength development. The geopolymers produced through activation of fly ash with potassium and sodium silicates might be very advantageous in terms of long-term thermal well integrity. Incorporation of LPS in geopolymers is shown to be very effective in higher temperature fields (temperature > 200˚F) including geothermal wells (van Oort *et al.*, 2019).

Nasvi *et al.*, (2014) investigated the effect of temperature on the permeability of the geopolymers. They concluded that despite the fact that geopolymers permeability increases with curing temperature due to increased pore diameter and heterogeneity in the porous structure of the geopolymers at elevated temperature, the maximum achieved permeability is 0.04 µD (micro-Darcy) which is massively lower than the limits suggested by API.

Salehi (2017) and Salehi et al., (2017) investigated the effect of contamination of oil-based and water-based drilling fluids on strength of geopolymers and cement mixtures. According to their experiments, cement is massively damaged due to the oil-based contamination, whereas geopolymer mixtures are a lot less damaged. They observed an 88% decrease in the strength of cement class H samples when contaminated with 10% oil-based mud (OBM), while, the strength of the geopolymers samples decreased by 25% encountering the same dosage of OBM contamination.

The Scanning Electron Microscopy (SEM) images of fly ash base-geopolymer samples shows a well-compacted structure formed because of Aluminosilicate gel which helps achieving ultra-low permeability sample and makes an appropriate material to be incorporated in plug and abandonment applications (Salehi, 2017).

5.4 Low melting point alloys

The low melting point Bismuth alloy has been suggested as a well sealant material due to its following properties (Spencer et al., 2015)

- The melting point can be altered by adding small amount of other metals, such as tin. The Bismuth-tin alloy has a melting point of 138^{o}C;
- The molten Bismuth has a viscosity very similar to water with a low surface tension and specific gravity of 10, which allows the liquid metal to penetrate inside small cracks(Nygaard, 2010);
- Upon cooling and solidification, Bismuth expands by 3% volumetrically, generating a tight seal against casing to avoid cement shrinkage induced micro-fractures;
- The solid Bismuth is CO_2 resistant and has a high compressive strength (55 MPa); and
- The Bismuth-tin alloy can be molten in-situ (downhole) using a heating tool. The low melting temperature required means little damage to the surrounding well elements.

Table 5-1 Summary of other sealant materials and their performance

Sealant material	Performance	Measurements	Merits and limitations	Reference
Nanocomposite Polymer (Novolac epoxy incorporating ANP)	• Ability to flow during injection to penetrate micro-annulus cracks and minimal shrinkage (low viscous) • Ability to bond with the surrounding shale materials	• Viscosity with 0.5% ANPs - appr. 216 cP • Shear bond strength – 2.4 times higher than cement • Shear displacement at peak and toughness were 3.7 and 7 times higher than micro cement respectively	• Novolac epoxy nanocomposite incorporating 0.5% ANPs was shown to be a good repair material • ANPs interfere with epoxy polymerization process and limitations in epoxy reactivity with other interfaces (which epoxy is stuck to) leading to increasing the bond strength	Genedy et al., 2017

Graphite nanometerials (Incorporating GnP & CNF in oilfield cement)	• Addition of 0.2vol% of cement lead to improvement in
	  ○ flexural strength by 20%
	  ○ tensile strength by 10%
	  ○ shrinkage resistance by 50%
	• Ability to control inception of microcracks and leaks in cement sheath
		• Cement slurry viscosity
		  ○ 131 cP with GnP 0.2vol%
		  ○ 201 cP with GnP 0.4vol%
		  ○ 282 cP with GnP 0.8vol%
			• GnP & CNF relatively low-cost
			• Improvement in physical & mechanical properties of cement
			• Particularly effective in resisting microcracks, microannulus cracks
			• May not be suitable for sealing micro fractures due to cement particle size
				Peyvandi et al., 2017

Nanosealant	• A single component low viscosity nano particle material • Activate only upon contact with divalent ions, which includes set cement	• Can enter and seal gaps as small as 20 μm, as well as up to 1,000 μm • The sealed gaps can withstand a pressure gradient greater than 1,000psi/ft	• Reduce complexity of fluid formulation • Simplify deployment in field • Low HSE foot print, ease to clean up and drill out in comparison with resins	Todd et al., 2018
Polymer-based gels	• Ability to seal fractures with widths from 0.25mm to 1mm to reduce permeability • Long term thermal and chemical stability	• Permeability measurement before & after sealing fracture • Thermal and chemical stability using reaction tube & bottle method • Hydraulic fracturing test to measure sealed fracture strength • Measurement on compressive & shear strengths	• The strength of sealed fracture will be lower than that of intact sample • The gels cannot be expected to withstand large differential pressures	Tongwa et al., 2013
pH-triggered polymer gelant	• Low viscosity at low pH & becoming highly swollen upon neutralization with substantial yield stress, therefore block fluid flow	• The gel exhibits non-Newtonian properties with shear thinning behaviour • Apparent viscosity ranges from 10 cP to lower than 1 cP depending on shear rate, polymer concentration and pH	• Long term applicability depends on the dynamic geochemical environment of the wellbore	Tavassoli et al., 2018

	• The high alkalinity of cement (pH ~ 13) provides required neutralization	• Hold back pressure for sealed fracture in cement ranges from 82 to 104 psi/ft of pressurized fluids depending on fracture aperture		
Micro-sized crosslinked polymer gel	• Change in concentration of brine has clear effect on gel particle swelling • The gel particles show acceptable injectivity performance through small fracture width • Gel with higher strength has a higher plugging efficiency	• Gel swelling capacity decreases with brine concentration • Gel strength increases with brine concentration • Gel plugging efficiency	• Plugging pressure to water leakage limited to 100psi for the tested fracture geometry. This pressure could be controlled by managing gel strength, but to a certain level	Abdulfarraj & Imqam, 2019a &b
Geopolymer	• Compared to conventional oilfield cement, geopolymer has ○ high compressive strength ○ high durability & stability exposed to corrosive environment and high temperature ○ low shrinkage & high ductility	• All the measurements for oilfield cement apply to geopolymer	• Geopolymer has a particle size distribution similar to oilfield cement which renders it not suitable for sealing micro fractures	van Oort et al., 2019 and large volume of other literature available
Bismuth-tin alloy	• Bismuth has a melting point 273*ºC,* which can be altered by adding small amount of other metals, such as tin • Melton Bismuth has a viscosity similar to water and specific gravity of 10 • Non-corrosive and not affected by H_2S or CO_2 • Bismuth expands by 3% on solidification	• Alloy bond with metal and rocks • Alloy rock bond shear strength • Alloy rock bond tensile strength • Alloy rock interface permeability	• The alloy does not wet solids • Poor bond to metal and rock • Special tool needed to melt the alloy • Expensive compared to cement	Carragher & Fulks, 2018 and Zhang et al., 2019

6 Laboratory methods for sealant performance evaluation

Potential sealant materials often need to be assessed for their suitability in sealing fractures and other defects in well cement in laboratory prior to their field trial and application. This assessment is conducted based on the performance criteria for well barrier materials as discussed in Section 2.3. This section reviews laboratory testing methods in evaluating the performance of the potential sealant materials.

6.1 Permeability of sealed fractures

The main function of a sealant material is to provide a seal against movement of fluids in the fracture and restore the hydraulic integrity of the well barriers which refers to cement sheath in this report. The seal ability of the sealant is assessed by conducting permeability measurements of a fracture or defect before and after applying the sealant material. The fracture geometry could be planar or annulus (Tongwa et al., 2013, Todorovic et al., 2016 and Stormont et al., 2015). The flowing media could be either water, nitrogen or air. However, when quantifying sealant materials, it is considered necessary to test the sealed fracture in terms of gas (nitrogen or air) permeability, since it is likely to be the escape of gas which is the key issue for the sealed fracture.

As an example, Tongwa et al., (2013) measured the ability of their sealing agents to seal a planar fracture and reduce the permeability of the cores through using a core flooding apparatus. Firstly, the permeability of the intact core was measured under high pressure-high temperature in the apparatus. A fracture with a given height and width was created along the entire sample length by cutting the core into two halves and machining a grove on both cutting surfaces. The two halves were then combined to form a fractured core which was inserted back into the coreholder for the measurement of the permeability of the fractured core. In the next stage, the sealant materials were injected into the fracture and the permeability measurement was repeated. By comparing the permeabilities of the intact core, core with open fracture and core with sealed fracture, the seal ability of the sealing agents and the degree of restored hydraulic integrity of the core could be assessed.

6.2 Bond strengths of sealed fractures

Whilst seal ability of a sealant material in reducing fracture permeability is important, it is equally important for the sealed fracture to possess sufficient bond strength to ensure the seal can be maintained when exposed to changes in temperature, pressure and stresses which could take place in the wellbore and formation. There are two aspects of bond strength of the sealed fracture, i.e., mechanical and hydraulic. The mechanical bond strength includes shear bond and tensile bond strengths on the interface between the sealant material with cement, casing steel or formation rock, whilst the hydraulic bond strength refers to hydraulic pressure required to allow fluid to penetrate at the interface, effectively causing crack propagation at that interface leading to failure of the seal (Oil and Gas UK, 2015).

6.2.1 Mechanical bond strength

Shear bond strength

Shear bond strength can be determined as the maximum shear stress that can be sustained by the material interface (Salehi et al., 2017). There are a few methods that can be used to measure interfacial shear bond strength between materials, such as push-out test and direct shear test (Genedy et al., 2017 and Tongwa et al., 2013).

A schematics of push-out test is shown in Figure 6.1. The sealant material is injected into the microannulus between the cement and shale core. Upon curing of the sealant, the shale core is pushed downwards slowly by applying an axial load via a piston to top of the shale core (not showing) until the debonding occurs while the cement ring is fixed. The test load and the shale core displacement are recorded, and the bond strength can be determined based on the maximum test load and the bonding surface area.

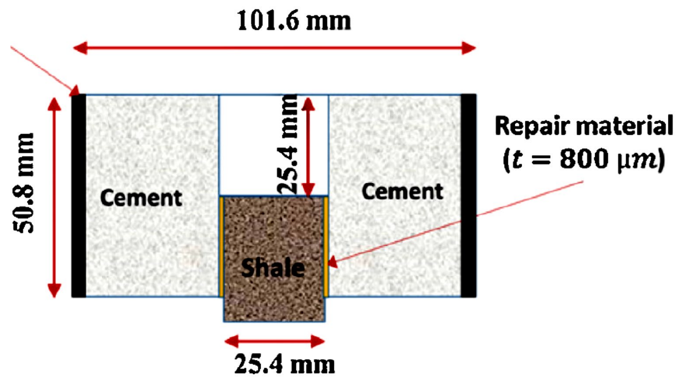

Figure 6.1 Schematic of the push-out test specimen showing measurement on shear bond strength between shale and cement (Genedy et al., 2017)

Tensile bond strength

Whilst tensile failure is a possible failure mechanism for a sealed fracture, for example, a sealed microannulus between the casing and cement sheath subject to changes in wellbore pressure, there is no standard measurement method to determine the tensile bond strength of a sealed fracture (Oil and Gas UK, 2015). However, for intact materials, there are standard methods for tensile strength measurements, including direct tensile and indirect tensile strength tests.

Direct tensile tests are performed on a classical dog-bone samples according to ASTM standards (ASTM C307-08, 2008) (Figure 6.2a) and indirect splitting tensile tests are including Brazil test performing on cylindrical samples (ASTM C496-04, 1993) and three-point bending test performing on prismatic samples (Figure 6.2c) according to (ASTM C348-02, 2002) (Quercia et al., 2016).

In the indirect splitting or Brazil test, a diametral compressive force is applied along the length of the cylindrical sample at a constant specified rate until splitting failure occurs as shown in Figure 6.2b (ASTM C496-04, 1993). Based on the theory of elasticity, this loading condition imposes uniform tensile stress along the diameter which instigates the failure of the specimen by splitting along a vertical plane (Arioglu et al., 2006). The indirect tensile strength of the test material can be obtained from the maximum compressive force and the dimensions of the test sample.

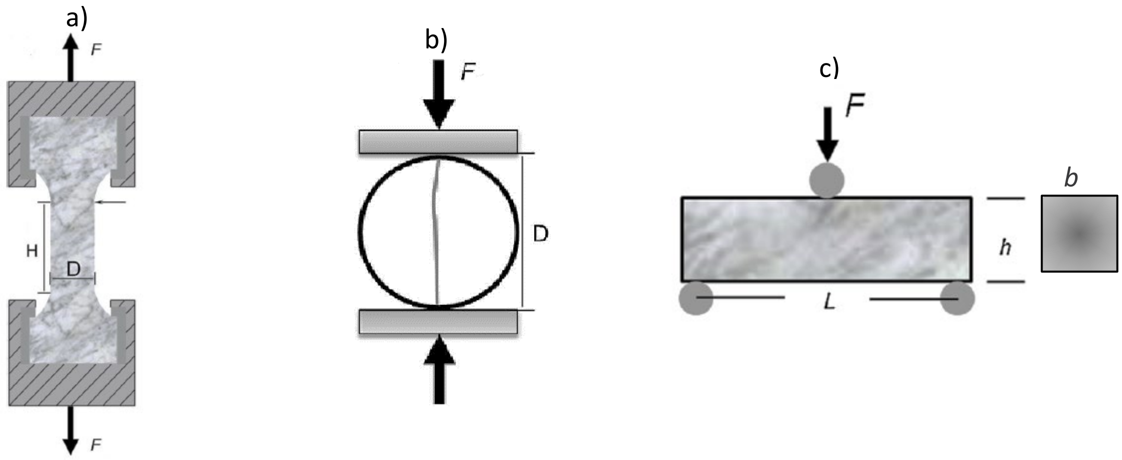

Figure 6.2 Tensile strength measurements: direct tensile strength measurements (a), splitting test (b), and three-point bending test (c)

The three-point bending test consists of applying load/displacement at the central point between the two supports. The tensile strength can be computed based on the maximum load and the test sample dimensions.

6.2.2 Hydraulic bond strength

Figure 6.3 shows a schematic of a laboratory test set up to measure the hydraulic bond strength between a cement plug and casing (Edgley et al., 2005). The cement or resin slurry is placed inside a 2” heavy steel pipe with a sand bed at the bottom so that the sealant would not plug off the incoming water line. After curing, high pressure water was pumped into the sand bed until the bond between the steel pipe and the cement or resin plug bloke, allowing water to flow from the open end of the pipe. The maximum water pressure is taken as the hydraulic bond strength of the plug.

Hydraulic bond strength or sealant blocking performance on sealed fracture in cement cores was measured by (Alkhamis et al., 2019). Artificial channels were created in cylindrical cement cores and sealed by injecting the sealant in the artificial channels. Upon curing, the cement cores were placed in a coreholder with a given confining pressure. Water was then injected into the cement cores from one end, while the other end of the core was left open to atmosphere. The hydraulic bond strength or the blocking pressure was taken as the injection pressure when the sealant started to debond from the cement core and water broke through from the other end of the core.

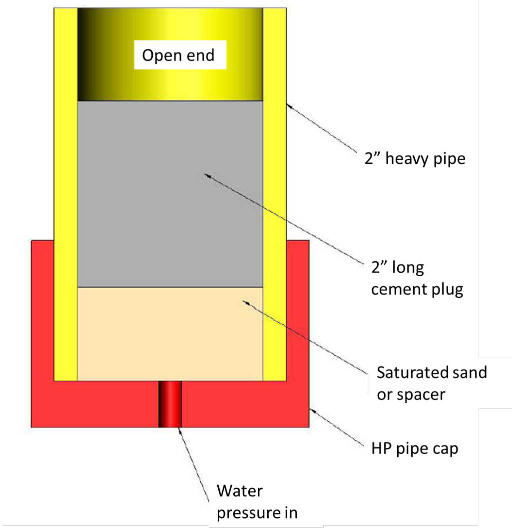

Figure 6.3 Schematic of a test set up for measurement of hydraulic bond strength between case and cement plug

6.3 Sealant slurry injectivity into fractures

When sealing a fracture in cement sheath or microannuli along the interfaces of cement sheath with the casing and formation, it is a prerequisite that the sealant material must be able to penetrate deeply into the fracture or microannuli with an injection pressure as low as possible. Injectivity, therefore, forms an important aspect in assessing the suitability of a potential sealant slurry.

Injectivity tests are often performed using an artificial fracture with a known aperture. The tests may be conducted at an ambient condition or conducted using a coreholder under a simulated down hole pressure and temperature condition (Meek & Harris, 1993, Farkas et al., 1999, Slater et al., 2001 , Todorovic et al., 2016, Todd et al., 2018).

Figure 6.4 shows a relatively simple bench top injectivity experimental apparatus. Narrow fractures are created by placing spacers between a porous plate and a transparent Plexiglas plate. The two plates are then clamped together and the sealant slurry is injected into the artificial fracture with a known fracture aperture. Penetration of the sealant slurry can be observed visually. Whilst such injectivity tests are quick and provide a qualitative information in screening different sealant slurries, more detailed information such as injection pressure and flow rate cannot be obtained.

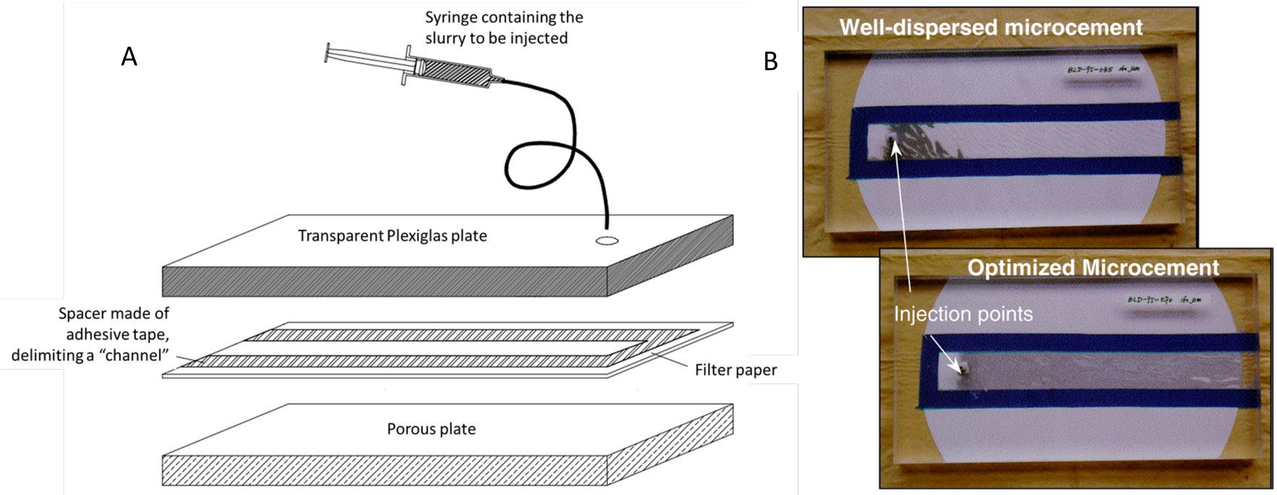

Figure 6.4. Apparatus for evaluating narrow gap penetration of cement slurries (A) and photos of injectivity tests showing a well-dispersed microcement bridging in a fingered pattern after minimal penetration with the optimized microcement penetrated the entire length of the model in a fully fluid state modified from (Todd et al., 2018)

Figure 6.5 shows a schematic of an experimental set up for injectivity and blocking performance tests. An artificial fracture with a known fracture aperture and height is created in cement core along the entire core length (can be other materials if required). The fractured core is then inserted into a coreholder which can apply a simulated downhole temperature and pressure condition. The injection pressure and flow rate of the sealant slurry can be measured accurately. Using the pressure and flow rate, an injectivity index can be calculated for the sealant slurry (Alkhamis et al., 2019). The injectivity index can be used to make quantitative comparison for different sealant slurries.

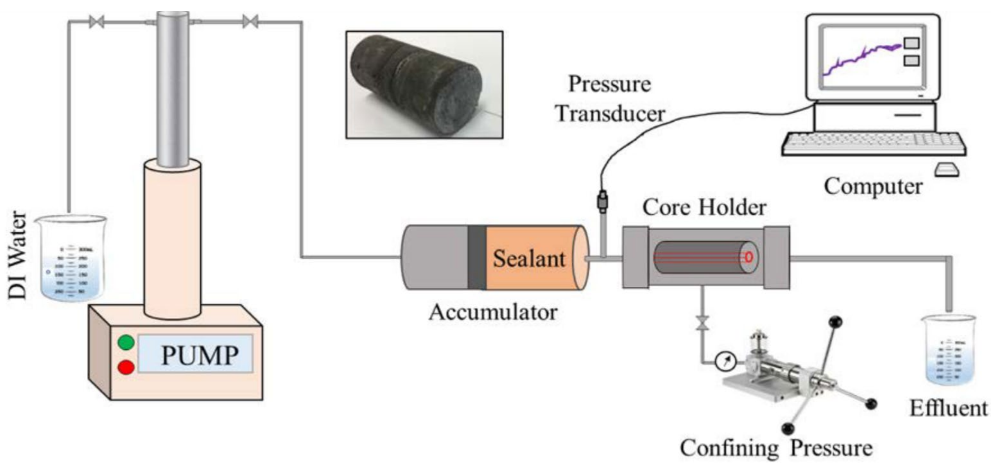

Figure 6.5 Injectivity and blocking performance test set up (Alkhamis et al., 2019)

6.4 Ageing tests

A key test in the guidelines by Oil and Gas UK (Oil and Gas UK, 2015) for qualifying sealant material is ageing test. This test involves exposing the sealant material to likely worst-case downhole conditions, periodically measuring changes in selected properties, and employing extrapolation techniques to establish likely rates of deterioration over longer timescales. For some sealant materials, temperatures in excess of those encountered in reality may be used to achieve accelerated ageing during the testing programme, which can provide further useful data for the extrapolation process.

6.4.1 Cement

Satoh et al., (2013) reported a comprehensive ageing study by exposing cement cores and casing-cement and rock-cement composites to a simulated downhole condition of CO_2 injection well in titanium reaction vessels. Two types of laboratory experiments were conducted;

- Static tests. Conventional batch-reaction experiments of cement cores in the system of CO_2 and simulated formation water at different temperature and pressures. In this experiment, individual cement cores are allowed to react with wet CO_2 and CO_2-rich NaCl solution, and
- Dynamic tests. CO_2-injection reaction involving casing (API Grade J-55)-cement (API class A) and cement (API class A)-shale composites which were saturated with simulated formation water. Supercritical CO_2 injection was carried out at elevated temperature and pressure with a constant differential pressure.

Figure 6.6 shows the outline of the static batch reaction system which consists of two water baths kept at two different temperatures. Each water bath contains 10 reaction vessels (Figure 6.6a). The CO_2 pressure in each vessel can be manually adjusted to the required pressure. Each reaction vessel contains 2 cores exposed to wet CO_2 and CO_2-rich NaCl solution respectively (Figure 6.6b). The duration of the static batch reaction ageing tests was varied from 100 to 4000 hours.

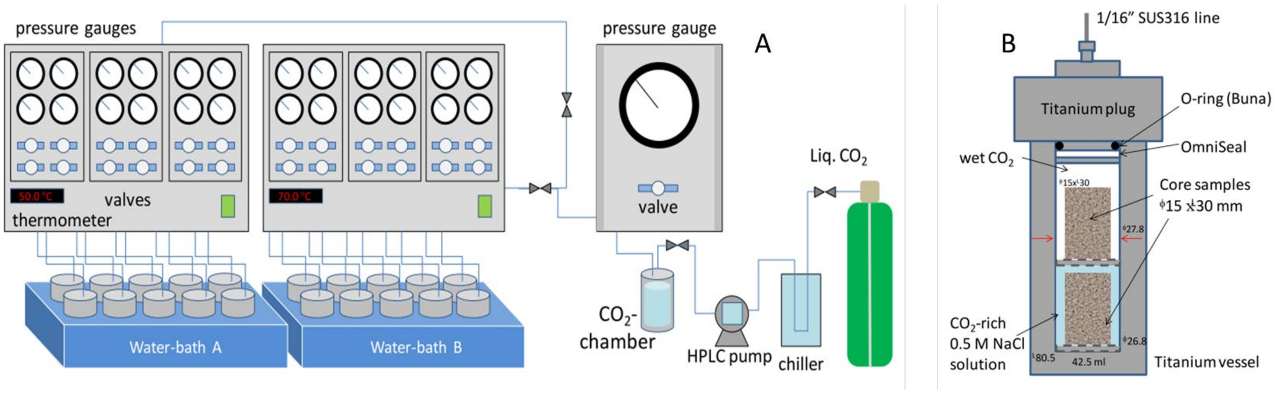

Figure 6.6. Outline of batch reaction system for cement core, and cement-casing and cement-shale composites

Figure 6.7 the outline of the dynamic CO_2 injection apparatus. The reaction vessel was placed inside an oven at an elevated temperature. The up-stream supercritical CO2 injection pressure was 8.5 MPa and the down-stream pressure maintained at 8.495 MPa, therefore applying a differential pressure of 5 kPa cross the core length. The CO_2 flow rate was measured using a mass flow meter. The duration of the dynamic CO_2 injection tests was varied from 50 to 60 hours.

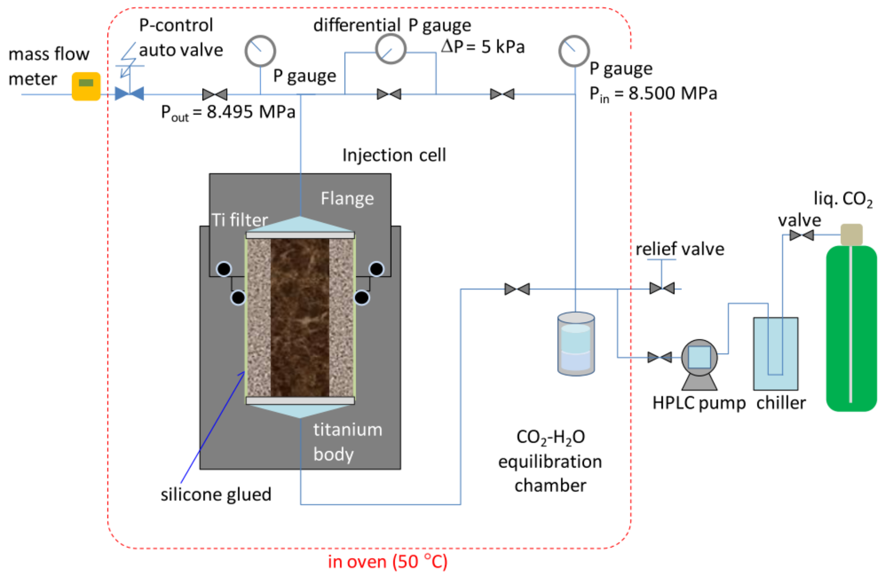

Figure 6.7 Outline of CO2 injection apparatus for composite cores

6.4.2 Polymer resin

In qualifying polymer resin as an alternative well barrier material, Beharie et al. (2015) reported an ageing study to ensure that the polymer resin is fit for purpose and can survive the anticipated life expectancy for the specific application. The simulated downhole conditions are summarised in Table 6-1. The cured prism-shaped resin samples with a dimension of 10 x 10 x 80 mm were used for the ageing tests. Permeability, compressive and flexural strengths of the polymer resin were measured by end of 1st month, 3rd month, 6th month and 12th month of the ageing tests respectively.

Table 6-1 Summary of simulated downhole conditions for ageing tests on polymer resin

Chemical	Pressure (psi)	Temperature (°C)
Crude oil (38° API)	7,250	100, 130
Methane (100%)	7,250	100, 130
CO2 5 % in N2	7,250	100, 130
H2S 5000ppm	145	100, 130

7 Technology gaps

Despite the progress in sealant technologies in remediating wellbore leakage made over the years, significant technology gaps remain in meeting oilfield challenges, as witnessed by the recent continued effort in adopting or developing new materials and solutions (Brunner et al., 2017), (Todd et al., 2018). The micro fine cements and polymer resins are the two types of main sealant materials for well sealing applied in recent years. Some of the technology gaps associated with their applications are summarised as follows

- Sealing small fractures or narrow leaking pathways, say, less than approximately 120 μm remains a significant challenge. There are very limited options available for sealing such small fractures:
    - The micro fine cement systems are unable to seal the fractures smaller than 120 μm; and
    - The solid-free epoxy resins are perceived to penetrate and seal a much smaller fracture. However, there are very limited studies on their injectivity into small fractures without the need to apply an excessive injection pressure (Alkhamis et al., 2019). Commercial products of the epoxy resin typically have a high viscosity, which would make them difficult to be injected in small pathways (Todd et al., 2018)
- Bond strength of sealants, particularly their tensile bond strength, are poorly quantified. For example, although debonding of the sealant from steel casing is a possible failure mechanism due to changes in pressure and temperature in the wellbore, which can lead to a well leakage, there are no standards for the measurement of the tensile bond strength which is extremely sensitive to the bonding surface conditions. As a result, at the present time, there is no requirement for tensile bond strength measurement when qualifying sealant materials (Oil and Gas UK, 2015);
- Limited ageing studies under simulated downhole conditions are reported on polymer resins, with the longest test duration up to 12 months. Such studies are limited to the measurements on permeability, compressive and flexural strength of the polymer resin. There have been no ageing test studies on sealant bonding strength, which is important as a fracture sealing material; and
- Durability of polymer resin as a well sealing material is, in general, poorly qualified. Very limited study on thermal degradation kinetics of epoxy resins showed that the life expectancy of the sealant (defined as 10% of weight loss by Jones et al. 2017) is very sensitive to downhole temperature. Theoretical prediction based on short term ageing test showed the expectancy of the resin system could be in the order of magnitude of hundred years. Whilst such life expectancy would be sufficiently long as a well repairing material, it may be far shorter than the durability as permanent well sealing material which is expected to be in thousands of years.

8 Summary

Wells play a central role in unconventional gas development and ensuring well integrity during their entire life and beyond is a significant challenge. Leaking well is an ongoing risk for the oil and gas industry, resulting in loss of production, safety concerns and environmental damage. As demonstrated in Sections 3 to 5, there are many potential solutions and technologies that can be applied for remediating well leakage. Appendix A provides a brief summary of the application range for available squeeze sealants for field remediation operations. Some of the technologies that are widely applied in industry are summarised below:

- *Conventional oilfield cements*. Over the years, Portland based cement has been the option of choice for majority of oil and gas well remediation treatments. Squeeze cementing has been applied to remediating leaking wells related to poor primary cement jobs, such as mud channels, voids, debonding, and has been mostly successful. However, due to the particle size, conventional oilfield cement cannot penetrate and seal small fractures or defects with an aperture less than approximately 400 μm;
- *Micro fine cements*. In recognizing the limitations of the conventional oilfield cements in well remediation treatments, micro fine cement system, including micro cements, and enhanced and optimized micro cements, has been developed and applied to seal small well leaks since early 1990's. Whilst micro fine cement system has been successful in remediating some tight well leaks, such as microannuli, casing leaks, where the conventional oilfield cements failed, laboratory and field case studies demonstrated that it remains a challenge for the micro fine cement system to penetrate and seal fractures or defects with an aperture less than approximately 120 μm;
- *Polymer resins*. Polymer resins have numerous advantages over cementitious sealants including the potential ability to penetrate deeper into smaller fractures that have aperture less than 120 μm due to its solid free nature. In addition, with proper formulation the polymer resin can be managed in terms of curing time and viscosity. It has also been proved that polymer resins are highly stable after set at elevated temperature (> 80°C). These properties make the polymer resins favoured for remediating tight well leaks that would not be possible using conventional oilfield cements.

Table 7-1 provides a summary of the nominal fracture aperture ranges that the sealant materials can seal.

Table 7-1. Summary of fracture apertures for various sealant materials

Sealant	Nominal fracture size (μm)	Reference
Portland cement	400	Ewort et al., 1990
Microcement	250	Meek & Harris, 1993
Enhanced microcement	160	Farkas et al., 1999

Optimized microcement	120	Slater et al., 2001
Polymer resin	310 – 500*	Alkhamis et al., 2019

* Fracture apertures used for injectivity tests, not necessary the smallest gap the epoxy resin can penetrate.

In addition to the conventional oilfield cements, micro fine cements and polymer resins, there are many other novel materials and commercially available products that have the potential to replace well cement as alternative well leaking sealants. They are currently being studied in laboratory or under field trials and are not as widely applied in industry as the oilfield cements, micro fine cements and polymer resins. Some of the other sealant technologies/solutions are summarised below:

- *Nano technology enhanced sealants*. Performance of polymer resins and oilfield cements can be improved significantly by adding nanoparticles to the sealants. By adding aluminium nanoparticles to the polymer resin matrix, the mechanical properties and durability of the sealant can be enhanced significantly. The addition of graphite nanomaterials to oilfield cement can improve the tensile and fracture strengths and reduce shrinkage of the cement. A recently developed nanosealant can effectively enter and seal leakage pathways as small as 20 μm;
- *Gels*. Preformed particle gel is an advanced super adsorbent polymer with a capability to absorb water more than hundred times of their weight and will not release the absorbed water under pressure. Whilst the polymer gel can reduce permeability of a fracture significantly, the hydraulic pressure differential the sealed fracture can sustain is typically significantly lower than the cementitious materials and polymer resins;
- *Geopolymer*. The geopolymers are very advantageous in comparison with conventional oilfield cements due to their high compressive strength, high durability exposed corrosive environment, low shrinkage, tolerance to contamination of oil-based muds, high ductility, and their stability at high temperature. Since the geopolymers have a particle size distribution similar to that of the conventional oilfield cements, it would be a significant challenge to remediate small fractures with an aperture less than approximate 400 μm; and
- *Low melting point alloys*. Bismuth-tin alloy has been proposed as a well sealing material due to its low melting point (138^{o}C), low viscosity (similar to water), low surface tension, and high specific gravity in liquid state, and volume expansion upon solidification.

Despite the progress in sealant technologies in remediating wellbore leakage made over the years, significant technology gaps remain. Particularly, sealing micro fractures with an aperture less than 120 μm is still a significant challenge. The solid-free polymer resins are perceived to be able to seal small fractures where the cementitious materials fail, however, commercial products of the polymer resins have high viscosities which would make them difficult to be injected in small leaking pathways. Furthermore, there have been limited studies on their long term durability, particularly the durability of their bonding strength with downhole materials, such as casing, cement sheath and formation rocks.

Appendix A Application range of available squeeze sealants in field remediating operation

Prior to a squeeze operation, it is a common industry practice to inject a clean and solids-free fluid into the interval to be squeezed to make sure the sealant slurry can be injected into the interval. The concept of injectivity factor was firstly introduced by (Cowan, 2007) after reviewing data from a large number of cement squeeze operations. The injectivity factor is defined as

INJECTIVITY FACTOR (psi-min/bbl) = Injection pressure (psi)/Injection rate (bbl/min)

and can be calculated from the injection rate and injection pressure from the prior squeeze injection. As the injection pressure increases for a given injection rate or as the injection rate decreases from a given injection pressure, the injectivity factor increases, indicating smaller particle size sealant systems are required to seal the leakage. However, a relationship between the injectivity factor and leakage pathway size has never been established, probably due to difficulties in quantifying fracture size downhole accurately.

Table A- 1 provides a summary of the application range for various sealants available in terms of injectivity factor

Table A- 1 The injectivity factor and summary of application range of available squeeze sealant materials, modified from (Todd et al., 2018)**.**

Injectivity range	< 2000	2000 - 4000	4000 - 6000	> 6000
Sealant materials	API cements Class C cement is typically the finest particle size of normal API cements	API cements – micro fine cement blend 50-80% API Class C, G or H cement + 20-50% micro fine cement	Micro fine cements Nano sealants Solid-free materials (resins/monomers)	Solid-free monomer or resin sealant (low viscosity materials generally preferred) Water/oil-based monomer blend Nanosealants

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