

**INVESTIGATION OF MODIFIED ENZYME-INDUCED
CARBONATE PRECIPITATION FOR SAND PRODUCTION
CONTROL APPLICATIONS**

BY

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I would like to dedicate my thesis to the wonderful people who have been there for me throughout my academic journey – my family, friends, and teachers. Their unwavering support has been the cornerstone of my success, and I am deeply grateful for the encouragement and guidance they have provided me with over the years.

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LIST OF ABBREVIATIONS

EICP	:	Enzyme Induced Carbonate Precipitation
MEICP	:	Modified Enzyme Induced Carbonate Precipitation
AM-AMPS	:	Acrylamide and 2-Acrylamido-2-Methylpropane Sulfonic Acid
MEICP	:	Modified Enzyme Induced Carbonate Precipitation
XRD	:	X-ray Diffraction
SEM	:	Scanning Electron Microscope
CT Scan	:	Computed Tomography Scan
XG	:	Xanthan Gum

ABSTRACT

Full Name : [Abdul Rehman Baig]
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Sand production is one of the major problems that can occur in an oil or gas well. Enzyme-induced carbonate precipitation (EICP) methods have recently emerged as possible environment-friendly solutions for enhancing loose sand consolidation and prevent it from being produced with the fluids to the surface. This work explores increasing the consolidated sand strength and its treatment procedure using a modified EICP. The study also examines the characterization of precipitation generated by microorganisms using computed tomography (CT scan). To consolidate the sand specimen, fourteen different solutions were prepared. The solutions are a mixture of urea, urease, CaCl_2 , MgCl_2 , Bio-polymers and Copolymer of Acrylamide and 2-Acrylamido-2-Methylpropane Sulfonic Acid (AM-AMPS) in varying quantities. X-ray diffraction (XRD) analysis was conducted to determine the type of calcium carbonate polymorph. The morphology of calcium carbonate precipitation, in the sand sample was visualized through scanning electron microscopy (SEM) imaging. Strength of consolidated samples were determined by scratch test. Thermogravimetric analysis (TGA) analysis to find the degrading temperature of polymers, and permeability was measured via sand pack flooding.

In our research, we explored the effectiveness of different EICP (Enzyme-Induced Carbonate Precipitation) solutions by subjecting them to various curing temperatures.

Notably, samples cured at 70°C demonstrated the highest strength, while those cured at 25°C exhibited the weakest. This underscores the critical role of temperature control in determining the strength of the samples. Moreover, a combination of CaCl₂ and MgC₂ salts in the solution resulted in greater strength compared to solutions with only one type of salt. The use of CT scans proved valuable in characterizing microbially induced precipitation, such as calcite and dolomite. The study highlights the importance of assessing how different curing temperatures impact specimen performance and emphasizes the need for precise temperature control in experimental setups.

In another aspect of our investigation, we mixed the EICP solution with bio-polymers (Xanthan Gum and Cellulose) and synthetic copolymers of AM-AMPS (AN 113, AN 113 SH, AN 125, AN 125 SH). Curing these mixtures at 70°C and 120°C revealed that Cellulose, a bio-polymer, exhibited the highest strength at both temperatures. Conversely, Xanthan Gum showed weakened strength at 70°C and no strength at 120°C due to its breakdown at high temperatures. Among the synthetic copolymers, AN 125 consistently demonstrated the highest strength, while AN 113 SH was the weakest. Surprisingly, the weakest synthetic copolymer had strength comparable to the strongest bio-polymer. Additionally, thermographic examination indicated that all copolymers degraded at temperatures exceeding 300°C, resulting in comparable strength for all EICP solutions at 120°C. Permeability tests with sand pack flooding revealed that the solution prepared with AN 125 exhibited almost a 30% reduction in permeability, making it a promising candidate for EICP applications in oil and gas wells. This research underscores the importance of a thorough methodology in evaluating the suitability of both bio-polymers and synthetic copolymers of AM-AMPS for EICP applications.

ملخص الرسالة

الاسم الكامل: عبد الرحمن بيغ

عنوان الرسالة: تحقيق تعديل ترسيب الكالسيت المحفز بالإنزيم لتطبيقات التحكم في إنتاج الرمال

التخصص: هندسة البترول

تاريخ الدرجة العلمية: ٢٠٢٣ ديسمبر

يعد إنتاج الرمال أحد المشاكل الرئيسية التي يمكن أن تحدث في آبار النفط أو الغاز. ظهرت مؤخرًا طرق ترسيب الكربونات المستحثة بالإنزيم (EICP) كحلول صديقة للبيئة لتعزيز عملية توحيد الرمال السائبة ومنع إنتاجها مع ظهور السوائل على السطح. يستكشف هذا العمل زيادة قوة الرمال المدمجة وإجراءات معالجتها باستخدام EICP المعدل. تبحث الدراسة أيضًا في توصيف الهطول الناتج عن الكائنات الحية الدقيقة باستخدام التصوير المقطعي المحوسب (CT). لدمج عينة الرمل، تم إعداد أربعة عشر محلولًا مختلفًا. المحاليل عبارة عن خليط من اليوريا واليورياز و $CaCl_2$ و $MgCl_2$ والبوليمرات الحيوية والبوليمر المشترك من الأكريلاميد و2-أكريلاميدو-2-ميثيلبروبان حمض السلفونيك (AM-AMPS) بكميات متفاوتة. تم إجراء تحليل حيود الأشعة السينية (XRD) لتحديد نوع متعدد أشكال كربونات الكالسيوم. تم تصوير شكل ترسيب كربونات الكالسيوم في عينة الرمل من خلال التصوير بالمجهر الإلكتروني الماسح (SEM). تم تحديد قوة العينات المجمعة عن طريق اختبار الصفرة. تحليل قياس الحرارة الحراري (TGA) للعثور على درجة حرارة التحلل للبوليمرات، وتم قياس النفاذية عن طريق غمر العيوب الرملية.

في بحثنا، اكتشفنا فعالية حلول EICP المختلفة (ترسيب الكربونات الناتج عن الإنزيم) من خلال تعريضها لدرجات حرارة معالجة مختلفة. ومن الجدير بالذكر أن العينات المعالجة عند 70 درجة مئوية أظهرت أعلى قوة، في حين أظهرت العينات المعالجة عند 25 درجة مئوية الأضعف. وهذا يؤكد الدور الحاسم للتحكم في درجة الحرارة في تحديد قوة العينات. علاوة على ذلك، فإن مزيج أملاح $CaCl_2$ و $MgCl_2$ في المحلول أدى إلى قوة أكبر مقارنة بالمحاليل التي تحتوي على نوع واحد فقط من الملح. أثبت استخدام الأشعة المقطعية قيمته في توصيف هطول الأمطار الناجم عن الميكروبات، مثل الكالسيت والدولوميت. تسلط الدراسة الضوء على أهمية تقييم كيفية تأثير درجات حرارة المعالجة المختلفة على أداء العينة وتؤكد على الحاجة إلى التحكم الدقيق في درجة الحرارة في الإعدادات التجريبية.

في جانب آخر من تحقيقنا، قمنا بخلط محلول EICP مع البوليمرات الحيوية (صمغ الزانثان والسليولوز) والبوليمرات المشتركة الاصطناعية من AN 113 SH، AM-AMPS، AN 113 SH، AN 125، AN 125 SH. كشفت معالجة هذه الخلائط عند درجتني 70 درجة مئوية و120 درجة مئوية أن السليولوز، وهو بوليمر حيوي، أظهر أعلى قوة في كلتا درجتني الحرارة. وعلى العكس من ذلك، أظهر صمغ الزانثان قوة ضعيفة عند 70 درجة مئوية وانعدام القوة عند 120 درجة مئوية بسبب تحلله عند درجات حرارة عالية. من بين البوليمرات المشتركة الاصطناعية، أظهر AN 125 SH استمرار أعلى قوة، في حين كان AN 113 SH هو الأضعف. والمثير للدهشة أن أضعف كويوليمر صناعي يتمتع

بقوة مماثلة لأقوى بوليمر حيوي. بالإضافة إلى ذلك، أشار الفحص الحراري إلى أن جميع البوليمرات المشتركة تتحلل عند درجات حرارة تتجاوز 300 درجة مئوية، مما يؤدي إلى قوة مماثلة لجميع محاليل EICP عند 120 درجة مئوية. كشفت اختبارات النفاذية مع غمر العبوات الرملية أن المحلول الذي تم إعداده باستخدام AN 125 أظهر انخفاضاً بنسبة 30% تقريباً في النفاذية، مما يجعله مرشحاً واعدًا لتطبيقات EICP في آبار النفط والغاز. يؤكد هذا البحث على أهمية اتباع منهجية شاملة في تقييم مدى ملاءمة كل من البوليمرات الحيوية والبوليمرات المشتركة الاصطناعية ل-AM-AMPS لتطبيقات EICP.

CHAPTER

INTRODUCTION

1.1 General Introduction

Sand production is one of the major problems that can occur in an oil or gas well. Enzyme-induced carbonate precipitation (EICP) methods have recently emerged as possible environment-friendly solutions for enhancing loose sand consolidation and prevent it from being produced with the fluids to the surface.

1.2 Problem Statement

Previous research on enzyme-induced carbonate precipitation for sand consolidation has primarily focused on testing at room temperature (25°C). However, these studies have demonstrated that the consolidated samples did not have the desired strength. Therefore, this thesis seeks to conduct practical experiments on an altered process called enzyme-induced calcite precipitation. The goal is to create a formula that works effectively at elevated temperatures, specifically 120°C, while ensuring strong consolidation of samples and maintaining sufficient permeability for efficient oil production. By conducting thorough testing, this research seeks to address the shortcomings of previous studies and provide a more effective and reliable method for sand consolidation using modified enzyme-induced calcite precipitation.

1.3 Objective

This study's ultimate objective is to offer a MICP formulation that may be applied as a dependable and effective sand consolidation technique in the oil and gas sector. To achieve this, create a formulation for Modified Enzyme Induced Carbonate Precipitation (MICP), that may successfully consolidate sand formations with good strength at high temperatures while preserving the permeability of the consolidated sand. This will entail carefully controlling the temperature and additives of the MICP solution to encourage carbonate precipitation. Scratch tests and sand pack flooding will be used to determine the consolidated sand's strength and permeability

1.4 Types of Sand Consolidation Methods

In petroleum industry, numerous wells face the issue of sand flowing into the wellbore alongside the reservoir fluids during production. This can cause various problems, like blocking up liners and leading erosion in valves and pipelines (Alarifi et al., 2022). Given these challenges regarding sand production in oil and gas operations, various approaches have been adopted to control them as they go on within the respective facilities. Such measures involve carrying out utilizing chemical interventions, mechanical adjustments, controlling production rates, and introducing innovations like the Enzyme-induced Carbonate Precipitation (EICP).

1.4.1 Mechanical Methods and Well Completions

Mechanical management of sand production in well completion comprise a number of methods like downhole tools as well as Frac & Pack technique designed to decrease velocities around the sand face or stop sand from entering towards the wellbore. These include slotted liners, screens, gravel packs, etc., either separately or combined. However, while effective, such measures, especially wetting, can increase skin as well as reduce oil production rates. An example of such filters is slotted liners or screens that are placed opposite the sand-producing interval and allow formation fluids to flow but prevent sand. The second type of completion involves installing a gravel pack; whereby the formation is first packed with gravel and then a screen is installed in direct contact with it. Using frac and pack technique, productivity and sand control are improved simultaneously (Weirich et al., 2013).

1.4.2 Controlling Production Rate

In this case, reaching maximum sand-free production could entail limiting the well's production at an optimum flow rate that affects both productivity as well as revenue. The rate is found out through trial and error, with parameters like reservoir pressure or water cut dictating the optimum rate. This can be seen by reviewing some previous research which also highlighted potential downsides of using such an approach (Ben Mahmud et al., 2020; Saurabh Mishra, 2015).

1.4.3 Chemical Methods

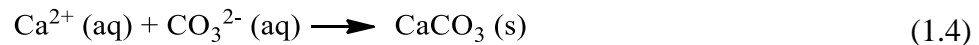
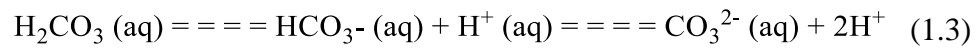
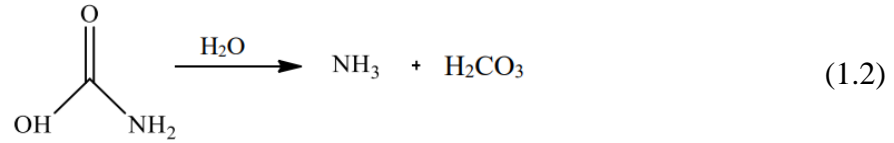
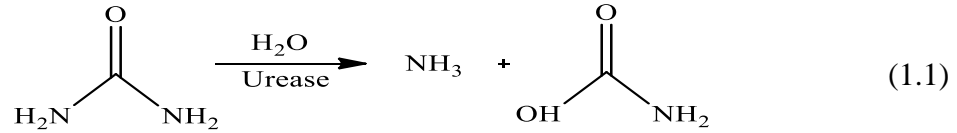
Chemical consolidation as a sand control method involves injecting liquid chemicals, such as plastic resins, polymers, and nanoparticles, into loose formations to bind sand grains together. One advantage of this technique is that it can be applied in small-diameter

wellbores without the need for a rig. Additionally, it does not require the installation of downhole tools, making it a cost-effective option for sand control in primary and remedial completions (Osisanya, 2010). Commercially available polymers, such as epoxy, furan, and phenolic resins, are commonly used for sand consolidation. Typically, these resins are in liquid form when injected into the formation, and a catalyst or curing agent is needed for hardening (Economides et al., 1998). There are two types of resin systems: in essence, internally activated and externally activated (over-flush system). Internally activated systems entail application of resin solution by mixing it with catalyst over the surface where the resins begin to harden. However, it takes time and may be rapid in case of very high temperature wells or high clay formations hence limiting the success of that system. However, it is necessary to mention that, externally activated systems consist of injecting the resin into the formation and then pouring the catalyst after the resin has been placed (Economides et al., 1998).

Other chemical consolidation systems have been formulated for reservoirs that are prone to high temperatures in order to overcome the shortcomings associated with resins such as epoxies, furans, and phenolics. For example, water-soluble organosilane could be used as a chemical sand control system. Organosilane reacts with water molecules trapped in formation pores and the hydroxyl groups on the sand grain surface, forming a network between sand grains (Morkved et al., 2014). Another method is the Zeta potential system, which alters the surface charge on sand particles, creating an ionic attraction and inducing their agglomeration (Saurabh Mishra, 2015).

1.4.4 EICP Method

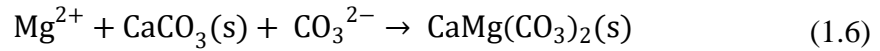
A chemical technique that is proposed for sand consolidation by in-situ precipitation of calcium carbonate between loose sand grains in the reservoir uses enzymes to help calcium carbonate (CaCO_3) precipitation for grain cementation (Larsen et al., 2008). Engineers were worried about potential blockages in the reservoir using previous methods that relied on resin-mediated sand consolidation chemicals. The uniqueness of this solution lies in its reversibility. As calcium carbonate (CaCO_3) is soluble in acid, if the enzyme-based solution causes a significant reduction in permeability during the chemical placement process, we can address it by administering acid. This allows us to regain some of the lost permeability and maintain the effectiveness of the solution. The concept behind the theory is that calcium carbonate (CaCO_3) precipitates in the gaps between sand grains. This is achieved by introducing sufficient calcium and carbonate into the water solution, allowing for controlled precipitation. To ensure a more controlled and delayed reaction, carbonate is introduced through a more intricate chemical process, in addition to calcium, which is provided in the form of a salt in the mixing solution. The breakdown of urea, a component in the solution, involves several steps (as shown in Equations 1.1 and 1.2). With the help of a bio-catalyst enzyme called urease, urea undergoes hydrolysis, resulting in the production of ammonia and carbonic acid. The hydrogen carbonate and carbonate ions then reach equilibrium with the carbonic acid (as shown in Equation 1.3). Once a critical concentration is achieved, the produced carbonate reacts with calcium, leading to the precipitation of calcium carbonate (CaCO_3) as depicted in Equation 1.4 (Larsen et al., 2008):



When calcite forms in the soil, it acts like nature's glue, sticking soil particles together. This not only stops them from moving around too much but also makes the soil stronger and better for construction (Harkes et al., 2010; Yasuhara et al., 2012). Calcite as a handy filler that plugs up the gaps in the soil, making it less porous and permeable (Harkes et al., 2010; Yasuhara et al., 2012). However, using microorganisms in this method comes with its share of challenges. It requires careful control of how bacteria are grown (Neupane et al., 2013; Putra et al., 2016a). Also, the large amount of stuff used could get in the way of the bacteria doing their job of breaking down urea (Nemati et al., 2005). This could possibly make the bacteria less effective in the soil (L. van Paassen et al., 2010; Yasuhara et al., 2011).

When there are magnesium ions (Mg^{2+}) and calcium carbonate around, the expected steps to create dolomite are shown in Equation (1.5–1.6).





In this research, we'll be using a technique that involves enzymes to make calcium carbonate and address the issue of lost circulation. This in-place formation of calcite is a cutting-edge biotechnology with practical uses in various scientific and engineering areas, like enhancing soil quality (DeJong et al., 2010; Neupane, Yasuhara, Kinoshita, & Ando, 2015; Neupane, Yasuhara, Kinoshita, & Putra, 2015; Putra et al., 2016b; L. A. van Paassen et al., 2010; Whiffin et al., 2007) and Boosting the strength of concrete (Achal & Pan, 2011; De Muynck et al., 2008).

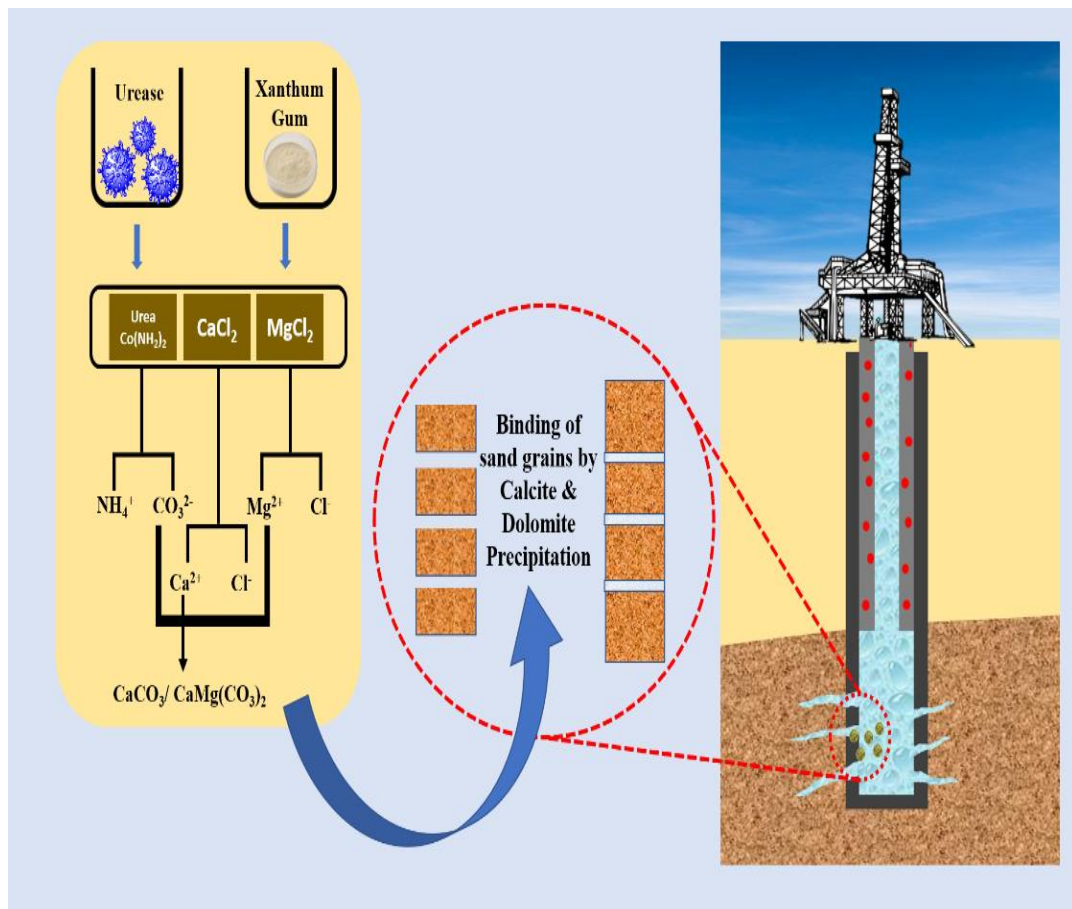


Figure 1-1 Schematic of EICP in Sand Production Mitigation

CHAPTER

LITERATURE REVIEW

There hasn't been much research on sand consolidation using EICP solutions, and the existing studies have mainly focused on lower temperatures. The strength achieved so far is not sufficient for effective sand consolidation in petroleum wells.

2.1 Previous Work

Unconfined compressive strength was tested in 2012 (Yasuhara et al., 2012). resilience and Toyoura sand's permeability after solution treatment with urea and calcium chloride values that are equimolar. The sand was combined with an enzyme named Urease with a 2,950 U/g stated activity, before adding solutions of urea-CaCl₂ of 0.50M and 1 M. At precipitated carbonate contents of 6% and 5%, they measured strengths of 0.75 and 1.6 MPa. (Hamdan et al., 2013 used two cycles of EICP percolation to treat Ottawa F60 sand to of 1.6% content of carbonate and a content of carbonate of 2 % for Ottawa 20-30 sand. They employed an EICP solution containing 1.4 gram/Liter urease enzyme of low activity (200 U/g activity), 0.38 M urea, and 0.27 M CaCl₂ (calcium chloride dihydrate). They noted that after isotropic consolidation to 50 kPa, samples that are treated are examined in drained triaxial compression tests showed a rise in the cohesiveness of 4.35 Psi for the Ottawa F60 sand and 1.30 Psi for the Ottawa 20-30 sand.

Using EICP percolation at a pair of distinct temperatures (23.5°C and 5°C), A one-time treatment cycle to coarse sand of 1m columns by (Neupane, Yasuhara, Kinoshita, & Putra, 2015), and the carbonate precipitation distribution was assessed. At 5°C (lower

temperature), they saw a very equal scattering of carbonate precipitation down the coarse sand column (2% on average). However, the carbonate content of the specimen that was treated at a temperature of 23.5°C declined from around 5% to 0% along the depth. These studies utilized 1 M concentration of CaCl₂, 1 M concentration of urea, and 15 grams/Liter of urease enzyme.

Organic soil, Sand, and silty soil were all treated by Oliveira et al., utilizing a mix and compact method of EICP (Oliveira et al., 2017). They performed their initial round of testing using an Enzyme Induced calcite precipitation solution containing 0.250 M concentration of CaCl₂, 0.250 M concentration of urea, and 4.0 KU/L urease (equivalent to around 0.120 grams/Liter enzyme with high activity). Then, they carried out additional tests using calcium chloride, 0.5 M urea and 8.00 KU/Liter of urease (equivalent to 0.230 grams/Liter enzyme with high activity). They discovered that whereas EICP treatment has a negative effect on organic soil, on the contrary, it strengthens sand and silty soil.

Magnesium chloride was added to the Enzyme Induced Calcite precipitation (EICP) solution by Putra et al. The amount of Urease was 1 g/l, and 0.50 M concentration of urea was set. MgCl₂ concentration was adjusted in a complementary way while CaCl₂ concentration was altered from 0.25 to 0.50 M, ensuring that the sum of these two chlorides' concentrations was always 0.50 M. According to their findings, adding magnesium chloride to test tube experiments increased the precipitation ratio to concentrations of 0.20 M by encouraging aragonite growth and minute calcite crystals. Further, they discovered that treated sand specimens had higher unconfined compressive strength than untreated specimens when compared to the treatment solution without magnesium chloride (Putra et al., 2016a).

In another study by Putra et al., 0.50 M concentration of urea was fixed and the amount of urease was varied from 1 to 5 grams/Liter. The concentration of MgSO_4 was adjusted in a complimentary way while CaCl_2 concentration vary from 0.50 to 0.40 M, ensuring that the combined concentration of these two substrates was always 0.50 M. A low MgSO_4 concentration encouraged the development of aragonite in test tubes, that with the increase in magnesium sulfate concentration precipitated mass increased, and that a sizable amount of gypsum was created at a MgSO_4 concentration of 0.10 M. A silica sand sample that had been reacted with a solution accommodating 0.10 M concentration of MgSO_4 showed that with the increase in precipitation, mass causes the increase in unconfined compressive strength, reaching to roughly 87.2 Psi (Putra, Yasuhara, & Kinoshita, 2017).

The impact of MgCl_2 on the EICP solution was investigated by Hu et al., in their study they utilized a solution containing 0.30 mol/L urea, 5 KU/L urease enzymes, and varying concentrations of CaCl_2 and MgCl_2 . The results showed that the modified EICP solution, treated with higher concentrations of MgCl_2 , exhibited the highest carbonate precipitation. The mechanism behind this enhancement was explained as an increase in the activity of the urease enzyme due to the introduction of magnesium ions, as interpreted from the study (Hu et al., 2021).

The influence of Mg ions on the polymorph of carbonate crystals in the MICP process, using $\text{Ca}(\text{CH}_3\text{COO})_2$ and urea as a cementation solution, was examined by Xu et al., Findings demonstrate that the inclusion of Mg ions enhances the properties of bio-cemented samples, resulting in higher UCS values. The addition of 0.01 M Magnesium ions in a 0.5 M urea and $\text{Ca}(\text{CH}_3\text{COO})_2$ solution leads to a 40% increase in UCS, while 0.5 M Mg ions double the average UCS. However, samples treated with MgCl_2 and urea

containing poorly crystalline magnesium carbonate exhibit significantly lower UCS. XRD and SEM-EDS analysis reveals altered crystal polymorph with increased aragonite precipitation but inhibited the growth of calcite. The effectiveness of $\text{Ca}(\text{CH}_3\text{COO})_2$ and urea with minor amounts of Magnesium ions in the MICP process is noteworthy (Xu et al., 2020).

Natural zeolite was incorporated into the EICP solution by Putra et al., in order to eliminate the ammonium by-product. They employed high-activity urease enzymes at concentrations of 1.00 and 2.00 g/L in EICP solutions of 0.5 and 1 M for CaCl_2 and urea. Depending on the reagent concentrations, they claimed that using 10 grams/Liter zeolite leads to NH_4Cl removal efficiency of approx. 75%. (Putra, Yasuhara, Kinoshita, et al., 2017).

Another research investigated Enhanced-Inorganic Carbon Precipitation (EICP) using alpha-amylase enzyme. Various parameters were studied to maximize calcite precipitation, including enzyme concentration, volume, CaCl_2 concentration, temperature, MgCl_2 and CaCl_2 synergy, Xanthan Gum, and solution pH. The study revealed that pH, temperature, and salt concentrations had significant impacts on precipitation, with higher enzyme concentrations leading to increased precipitation when sufficient salt was available. The optimal conditions for precipitation were observed at pH 12, with 2.5 g/L of Xanthan Gum as a stabilizer, and a temperature of 75°C , while the highest CaCO_3 precipitation occurred with a 0.6:0.4 molar ratio of CaCl_2 to MgCl_2 (Albenayyan et al., 2023).

AlKuroud et al., considered into the impact of Xanthan Gum and Magnesium chloride on an EICP solution. A standard solution account for 1M urea, 0.5M CaCl_2 , and 2g/L urease was discovered to be the perfect setting for the EICP procedure. The outcomes exhibit that

the precipitation ratio diminished with increasing MgCl_2 concentration, indicating that 0.5M MgCl_2 was the ideal concentration for precipitating calcium carbonate. Furthermore, increasing the Xanthan Gum concentration raised the precipitation of CaCO_3 , with 5 g/L of XG being the perfect level. Calcite and dolomite were the significant minerals that precipitated, according to XRD examination, with a substantial percentage of dolomite being seen when the solution consist of 0.4M CaCl_2 and 0.6M MgCl_2 (AlKuroud et al., 2022).

Tariq et al., investigated and tested a solution called EICP to discuss the issue of lost circulation in drilling operations. They evaluate variable compositions and quantities of reagents and identified the favorable combination for the EICP method, which consist of 0.1M MgCl_2 , 1M urea, 0.9M CaCl_2 , 1 g/L Xanthan Gum, and 3 g/L urease. This mixture resulted in the elevated amount of thermally stable calcite, very minute amount of aragonite, and highest precipitation efficiency. Lost circulation studies using this optimized EICP solution on a limestone sample with high permeability at both ambient and elevated temperatures was also conducted. Analysis of porosity and permeability measurements before and after the EICP treatment exhibit a notable reduction of 20% in porosity and 99% in permeability and. The findings of this study focus on the effectiveness of the optimized EICP solution in mitigating lost circulation issues in drilling operations (Tariq et al., 2022).

In highly saturated EICP solutions, elevating the temperature to levels as high as 90 °C has been observed to promote the reaction. ICP analysis has revealed that solutions containing metal ions such as Ca^{2+} and Mg^{2+} at concentrations exceeding 50,000 ppm, along with urea levels of 110,000 ppm, exhibit heightened sensitivity to high-temperature environments. In terms of efficiency, a critical threshold of 35,000 ppm for metal ions is essential to

achieve nearly complete 80% conversion into precipitation. Beyond this threshold, the conversion rate plateaus at 50%, but this higher concentration offers improved controllability, which can have a positive impact on the application of EICP solutions (Omarov et al., 2023).

CHAPTER

Material and Methodology

3.1 Materials for EICP Solution

The solution is prepared by mixing Deionized water, urea ((NH₂)₂CO, >99.5% purity), calcium chloride dihydrate (CaCl₂·2H₂O, >99% purity), magnesium chloride Hexahydrate, urease enzyme (extracted from jack beans), xanthan gum, cellulose and synthetic Copolymer of Acrylamide and 2-Acrylamido-2-Methylpropane Sulfonic Acid (AM-AMPS). To improve the heat resistance of a water-soluble anionic polymer, we combine a thermally stable monomer called 2-acrylamido-2-methylpropane sulfonic acid (AMPS) with acrylamide (AM) through copolymerization. This process produces a copolymer known as AM-AMPS, and you can see its structure in Figure 2. Difference between these AM-AMPS is molecular weight and anionicity (Table1). Chemicals were taken from Sigma Aldrich. Fig. 2 summarizes the different experiments and conditions followed in this study.

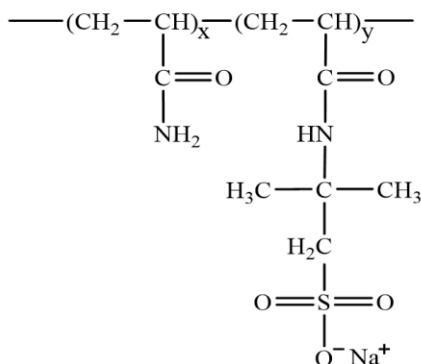


Figure 3-1 Structure of copolymer of AM-AMPS

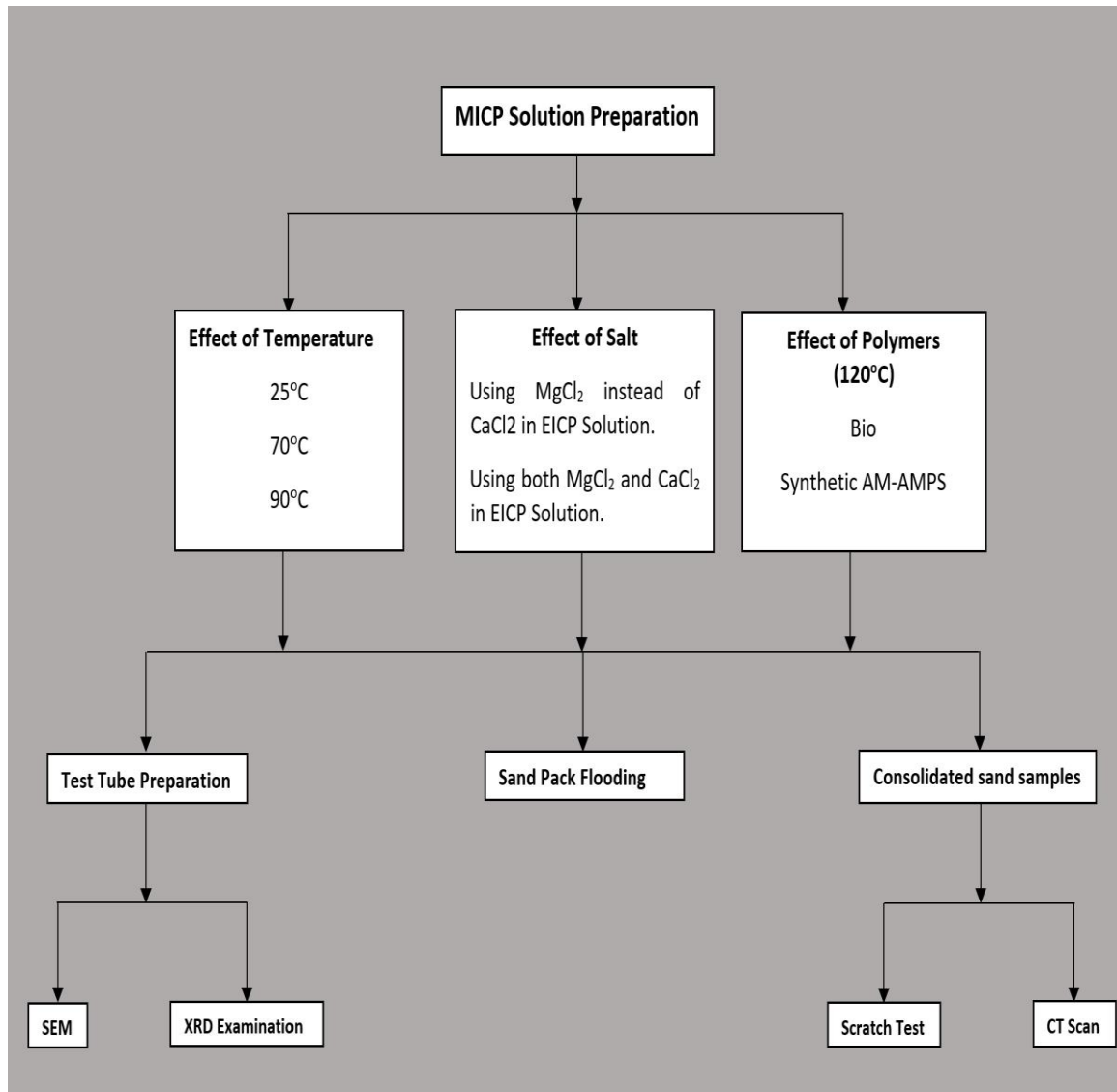


Figure 3-2 Methodology of EICP Experiment

3.2 Solutions preparation

To examine the impact of the Urease enzyme, xanthan gum CaCl_2 and MgCl_2 on CaCO_3 precipitation, seven distinct solution samples were made in Plastic tubes. Solution 1 (Baseline EICP solution) is prepared by adding 3 g/L enzyme, 0.67M calcium chloride, and 1.0M urea dissolved in Deionized water (AlAhmari et al., 2020; Almajed et al., 2019), Solution 2 is prepared by adding 3 g/L enzyme, 0.67M Magnesium chloride, and 1.0M urea dissolved in Deionized water, solution 3 is prepared by adding 3 g/L enzyme, 0.33M

calcium chloride, 0.33M Magnesium chloride, and 1.0M urea dissolved in Deionized water, solution 4 is prepared by adding 3 g/L enzymes, 0.67M calcium chloride, 0.67M Magnesium chloride, and 1.0M urea dissolved in Deionized water, Solution 5 is prepared by adding 3 g/L enzymes, 0.67 M calcium chloride, 0.67 M Magnesium chloride, 1.0 M urea and 1 g/L xanthan gum dissolved in Deionized water, solution 6 is prepared by adding 3 g/L enzymes, 0.67 M calcium chloride, 0.67 M Magnesium chloride, 1.0M urea and 2 g/L xanthan gum dissolved in Deionized water, solution 7 is prepared by adding 3 g/L enzymes, 0.67 M calcium chloride, 0.67 M Magnesium chloride, 1.0M urea and 3 g/L xanthan gum dissolved in Deionized water, Solution 8 is prepared by adding 3 g/L enzymes, 0.67 M calcium chloride, 0.67 M Magnesium chloride, 1.0 M urea and 1 g/L cellulose dissolved in Deionized water, solution 9 is prepared by adding 3 g/L enzymes, 0.67 M calcium chloride, 0.67 M Magnesium chloride, 1.0M urea and 2 g/L cellulose dissolved in Deionized water, solution 10 is prepared by adding 3 g/L enzymes, 0.67 M calcium chloride, 0.67 M Magnesium chloride, 1.0M urea and 3 g/L cellulose dissolved in Deionized water, solution 11 is prepared by adding 3 g/L enzymes, 0.67 M calcium chloride, 0.67 M Magnesium chloride, 1.0M urea and 3 g/L AN 113 dissolved in Deionized water, solution 12 is prepared by adding 3 g/L enzymes, 0.67 M calcium chloride, 0.67 M Magnesium chloride, 1.0M urea and 3 g/L AN 113 SH dissolved in Deionized water, solution 13 is prepared by adding 3 g/L enzymes, 0.67 M calcium chloride, 0.67 M Magnesium chloride, 1.0M urea and 3 g/L AN 125 dissolved in Deionized water and solution 14 is prepared by adding 3 g/L enzymes, 0.67 M calcium chloride, 0.67 M Magnesium chloride, 1.0M urea and 3 g/L AN 125 SH dissolved in Deionized water Table 2 summarizes formulation of all the solutions.

Table 3-1 Solution Formulations

Solution	Formulation
1	3 g/L enzyme, 0.67M CaCl ₂ , and 1.0M urea
2	3 g/L enzyme, 0.67M MgCl ₂ , and 1.0M urea
3	3 g/L enzyme, 0.33M CaCl ₂ , 0.33M MgCl ₂ , and 1.0M urea
4	3 g/L enzymes, 0.67M CaCl ₂ , 0.67M MgCl ₂ , and 1.0M urea
5	3 g/L enzymes, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 1.0 M urea and 1 g/L xanthan gum
6	3 g/L enzymes, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 1.0 M urea and 2 g/L xanthan gum
7	3 g/L enzymes, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 1.0 M urea and 3 g/L xanthan gum
8	3 g/L enzymes, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 1.0 M urea and 1 g/L cellulose
9	3 g/L enzymes, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 1.0 M urea and 2 g/L cellulose

10	3 g/L enzymes, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 1.0 M urea and 3 g/L cellulose
11	3 g/L enzymes, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 1.0 M urea and 3 g/L AN 113
12	3 g/L enzymes, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 1.0 M urea and 3 g/L AN 113 SH
13	3 g/L enzymes, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 1.0 M urea and 3 g/L AN 125
14	3 g/L enzymes, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 1.0 M urea and 3 g/L AN 125 SH

3.3 Test Tube Preparation

The 17 test tubes were prepared with the solution mentioned above. The plastic tubes were filled with 40 ml of the solution. To reduce solution evaporation, solution samples were then closed with plastic caps (Fig. 4). Test tubes with solution 1, was cured for 72 hours at temperatures 25°C, 70°C, and 90°C respectively. Test tubes with solutions 5-14 were cured for 72 hours at 70°C, each test tubes were allowed to be dried at a temperature they were cured for 72 hours and tubes prepared with solution 5-14 were also overdried at a temperature 90°C for 168 hours because solution prepared with polymers takes more time to get dried. In order to check effect of high temperature (120°C) six HPHT glass tubes were prepared from polymer solution (Fig. 6). Each test tube's precipitation was then filtered, dried in the oven, and submitted to XRD examination and SEM Analysis. With

the exception of the xanthan gum solution, all the other solutions produced a white precipitate when exposed to a high temperature of 120°C (Fig. 4c). In contrast, the xanthan gum solution underwent degradation at this temperature, resulting in the formation of a dark precipitate, as depicted in Fig. 7. Table 2 provides a summary of the mass of precipitation for all 14 solutions after the curing and drying stages.



Figure 3-3 Plastic tube with a closed cap containing EICP Solution



Figure 3-4 Precipitation at the bottom of the tube



Figure 3-5 White precipitation for Baseline, cellulose and AM-AMPS at 120°C



Figure 3-6 Dark precipitation of degraded xanthan gum at 120°C

Table 3-2 Mass of Precipitation

Solution	Temperature	Mass of precipitation (grams)
1	25°C	2.56

1	70°C	3.57
1	90°C	2.64
2	70°C	2.87
3	70°C	4.12
4	70°C	5.86
4	120°C	2.33
5	70°C	11.03
6	70°C	11.55
7	70°C	12.1
7	120°C	4.5
8	70°C	8.53
9	70°C	12.61
10	70°C	13.14
10	120°C	13
11	70°C	13.36
11	120°C	13.11

12	70°C	11.53
12	120°C	11.53
13	70°C	12.42
13	120°C	12.35
14	70°C	11.71
14	120°C	11.56

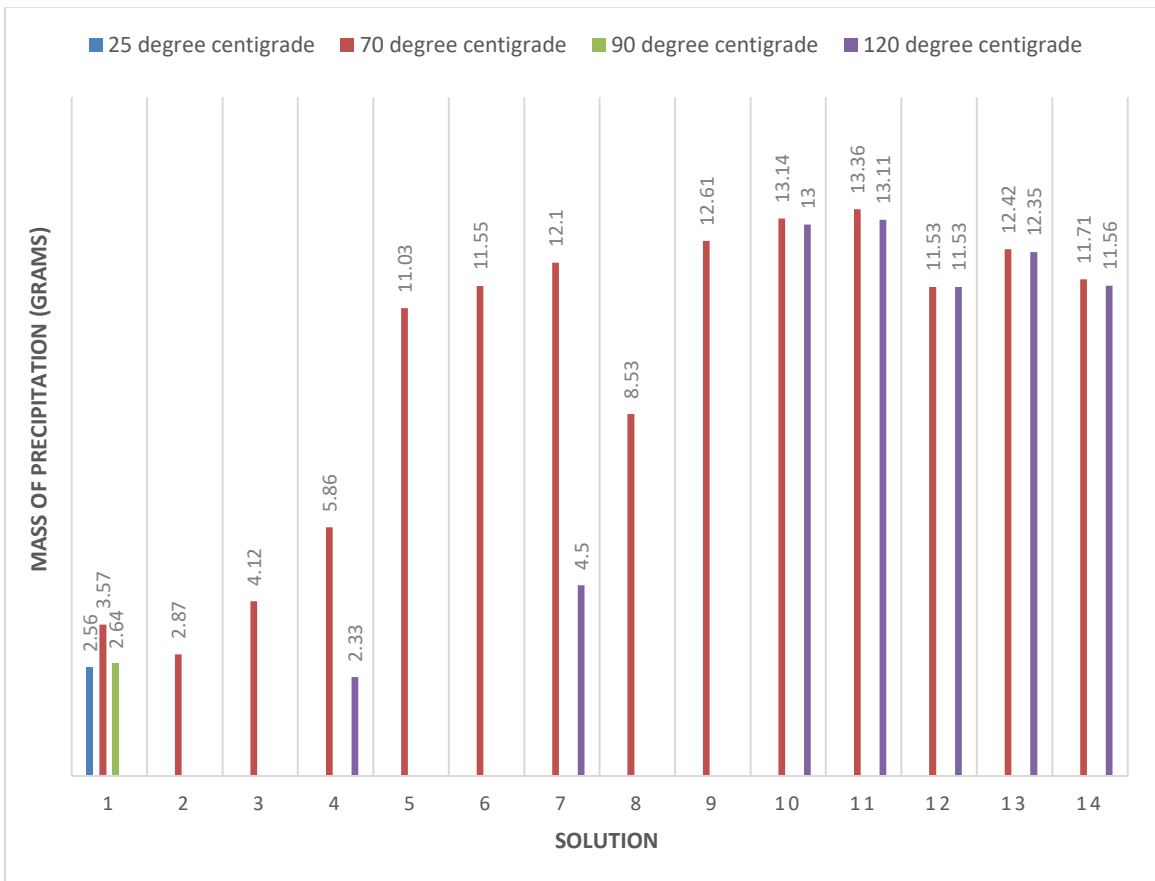


Figure 3-7 Mass of Precipitation

The amount of precipitation that forms when the Baseline EICP solution is cured at 70°C is the largest, measuring 3.57 gm. On the other hand, 25°C has the lowest precipitation levels. However, compared to the EICP solution made with only one salt, the modified EICP solution, made with a combination of salts (CaCl₂ and MgCl₂), exhibits even more precipitation of 6.16 gm. Additionally, it has been noted that the precipitation rises with xanthan gum and cellulose content, peaking at 12.10 gm and 13.14 gm respectively when the modified EICP solution is made with 3 g/L of xanthan gum and cellulose. In all the EICP solutions prepared using AM-AMPS polymers, there's a substantial amount of solid material that precipitates out. Cellulose solutions also exhibit a significant amount of solid precipitation, while Baseline EICP and xanthan gum solutions have the least amount of solid material settling out compared to the other solutions. Since AM-AMPS polymers break down at high temperatures, as indicated in Table 2, the EICP solutions prepared with them maintain their mass of solid precipitation even at elevated temperatures. In contrast, EICP solutions made with cellulose experience a slight reduction in the amount of solid material at 120°C. Xanthan gum solutions, on the other hand, undergo a significant reduction in the mass of solid precipitation, nearly halving at 120°C. These results point to the possibility of further EICP process modification in various applications by indicating that the improved EICP solution.

3.4 XRD Analysis

The utilization of X-Ray Diffraction (XRD) Analysis as a laboratory method enables the identification of the mineral constituents of precipitation. Table 3 summarizes the chemical composition for all the 7 solutions

Table 3-3 Chemical composition of precipitation

Solution	Composition
1 (25°C)	87.9% Calcite, 1.5% dolomite, and 10.6% Salammoniac
1 (70°C)	90.4% Calcite and 9.6% Vaterite
1 (90°C)	69.1% Calcite, 0.1% dolomite and 30.8% Salammoniac
2 (70°C)	29.1% Calcite, 62% Dolomite, and 8.9% Argonite
3 (70°C)	33% Calcite, 57.6% Dolomite, 6.3% Aragonite, and 3.1% Vaterite
4 (70°C)	53.5% Calcite, 44.6% dolomite, and 1.9% Salammoniac
4 (120°C)	50.2% Calcite, 31.8% dolomite, 1.1 % Aragonite, 16.9% Salammoniac
5 (70°C)	20.2% Calcite, 1.6% dolomite, 14.5% Salammoniac, 0.5% Argonite, 0.1% Vaterite, 63.1% Carnallite
6 (70°C)	17.8% Calcite, 0.3% dolomite, 17.9% Salammoniac, 0.9% Argonite, 3.5% Vaterite, 59.7% Carnallite
7 (70°C)	14% Calcite, 1% dolomite, 6.2% Salammoniac, 0.6% Vaterite, 78.3% Carnallite

7 (120°C)	59.7% Calcite, 30.3% dolomite, 2.9 % Aragonite, 7.1% Salammoniac
8 (70°C)	53.5% Calcite, 44.6% dolomite, 39.4% Carnallite, 13.7 % Salammoniac and 2.3% Vaterite
9 (70°C)	42.1% Calcite, 4.8% dolomite, 47.7% Carnallite and 5.4% Vaterite
10 (70°C)	34.3% Calcite, 0.3% dolomite, 65.1% Carnallite and 0.3% Vaterite
10 (120°C)	16.6% Calcite, 10.5% dolomite, 73% Salammoniac
11 (70°C)	85.1% Calcite, 53.8% dolomite, 0.6% Aragonite, 10.6% Salammoniac, 0.6% Vaterite
11 (120°C)	0.8% Calcite, 0.3% dolomite, 17.9% Salammoniac, 0.9% Argonite, 3.5% Vaterite, 59.7% Carnallite
12 (70°C)	33.2% Calcite, 4.8% dolomite, 21.5 % Aragonite, 33% Salammoniac, 7.6% Vaterite
12 (120°C)	1.7% Calcite, 0.5% dolomite, 0.2% Salammoniac, 19.1% Argonite, 12.2% Vaterite, 66.4% Carnallite
13 (70°C)	22.9% Calcite, 3.9% dolomite, 14.7 % Aragonite, 49.9% Salammoniac, 8.6% Vaterite

13 (120°C)	46.8 % Salammoniac, 3.6% Argonite, 1.4% Vaterite, 48.1% Carnallite
14 (70°C)	10.5% Calcite, 8.3% dolomite, 9.8% Aragonite, 69% Salammoniac, 2.4% Vaterite
14 (120°C)	40.7 % Salammoniac, 0.1% Argonite, 59.2% Carnallite

The baseline EICP solution, when cured at 70°C, exhibits a substantial proportion of Calcite. In contrast, the modified EICP solution, which is prepared using a combination of salts (MgCl₂ and CaCl₂), contains noteworthy amounts of both Calcite and Dolomite. Baseline EICP solution cured at 90°C shows significant amount of salammoniac along with calcite. In the baseline EICP solution, we observe a substantial presence of calcite and dolomite in the precipitated materials, both at 70°C and 120°C. When it comes to the EICP solution prepared with xanthan gum, we find a notable amount of carnallite at a temperature 70°C, but at 120°C, there's a notable presence of calcite and dolomite. For the EICP solution made with cellulose, noteworthy amounts of calcite and carnallite appear at a temperature 70°C, while at 120°C, Salammoniac becomes a significant component. Similarly, the EICP solution prepared with AN 113 shows significant levels of calcite and dolomite at a temperature 70°C, but at 120°C, it displays a noteworthy presence of carnallite. In the case of EICP solution using AN 113 SH, calcite is notable at a temperature 70°C, but at 120°C, carnallite becomes prominent. For the EICP solution prepared with AN 125, both Salammoniac and calcite are significant at 70°C, and at 120°C, Salammoniac

and carnallite are prevalent. Finally, the EICP solution prepared with AN 125 SH exhibits a substantial amount of Salammoniac at 70°C, and at 120°C, it displays significant amounts of both Salammoniac and carnallite.

3.5 SEM Analysis

SEM images of solution 1 cured at 25°C and 70°C (Fig. 3-8) show fine calcite polymorph while the SEM image of solution 1 cured at 90°C shows the flat surface of Salammoniac. SEM images of solution 2-4 show precipitates of Dolomite along with calcite. SEM images of solution 5-7 shows flat surface of Carnallite. From the SEM images (Fig. 8) it is evident that dolomite crystals coexist with calcite when the EICP (Enzyme-Induced Calcite Precipitation) solution is prepared using both magnesium chloride (MgCl₂) and calcium chloride (CaCl₂) salts, specifically in solution 2, 3, and 4. The baseline EICP solution, when cured at a temperature of 90°C, displays a smooth surface characterized by the presence of salammoniac. The SEM Analysis results reveal that Solution 1 exhibits the precipitation of calcite and dolomite at both 70°C and 120°C. When we look at Solution prepared with xanthan gum, the SEM image at 70°C displays flat surfaces of carnallite, while at 120°C, it exhibits the formation of calcite and dolomite crystals. Similarly, in the case of Solution prepared with cellulose, at 70°C, we observe calcite, and at 120°C, we can see both calcite and salammoniac in the SEM images. For Solution prepared with AN 113, SEM images at 70°C show the presence of calcite and dolomite, while at 120°C, they primarily display calcite. In Solution prepared with AN 113 SH, the SEM images reveal calcite at 70°C and carnallite at 120°C. Solution prepared with AN 125, under SEM analysis, displays calcite and salammoniac at 70°C, and at 120°C, it shows both carnallite and salammoniac. Finally, when examining solution prepared with AN 125 SH, SEM images show salammoniac at

70°C, and at 120°C, they reveal the presence of both carnallite and salammoniac.

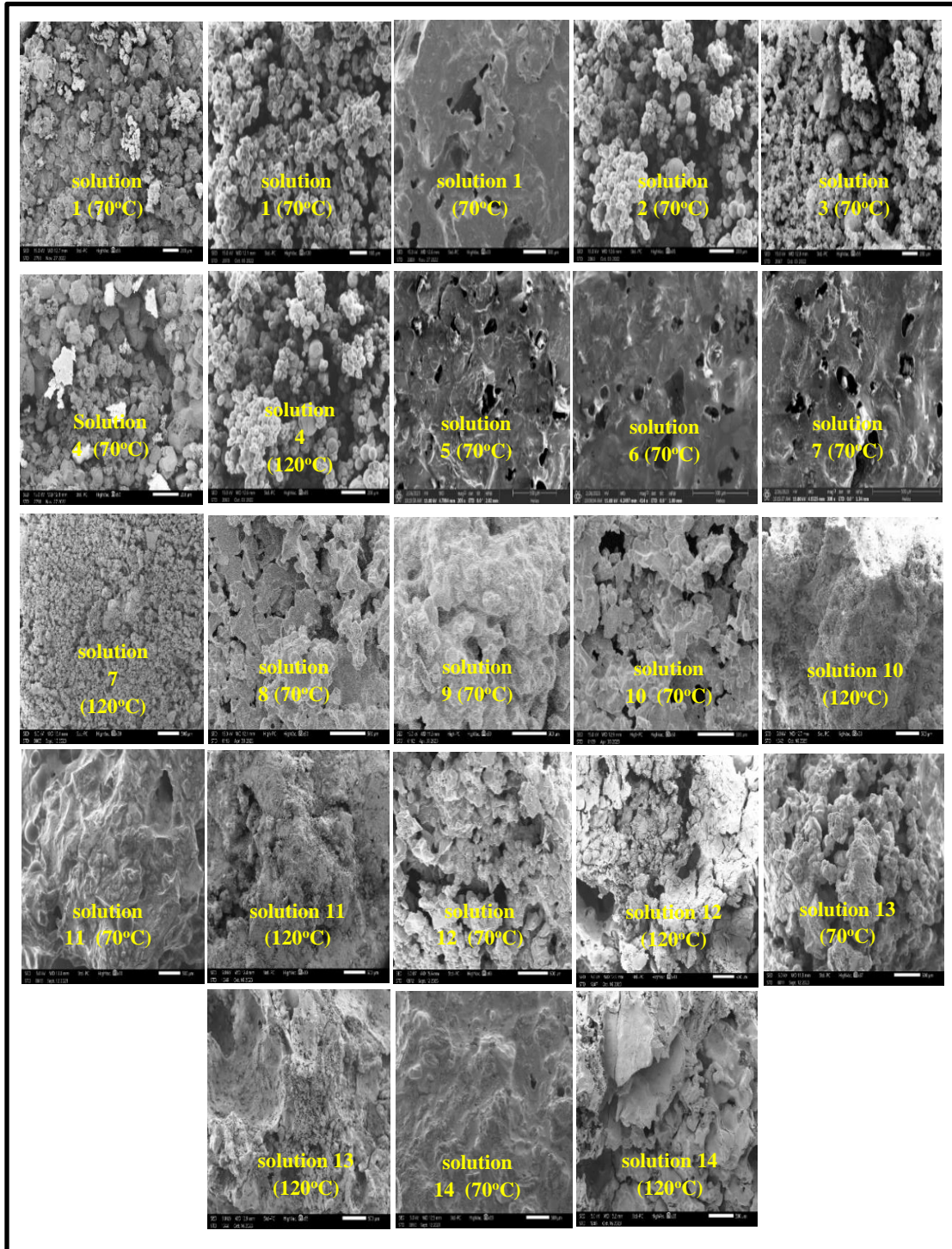


Figure 3-8 SEM Images

3.6 Sand Column Experiment

To study how EICP solution affects Carbonate and Dolomite precipitation, specimens were made in plastic container with sand inside. The test samples were prepared by mixing 150g of sand with 40 ml of EICP solution (approximately one pore volume). The sand and solution were then mixed in plastic containers, containers were carefully tapped until a solution rose a few millimeters above the soil's surface indicating that the packed soil was almost saturated. The plastic caps were then placed on the plastic containers to prevent the fluid from evaporating too much. The container prepared from solutions 1 was given a minimum of 72 hours to cure at a temperature of 25°C, 70°C, and 90°C. The container prepared from solutions 2-14 was given of 72 hours to cure at a temperature of 70°C. In order to check effect of high temperature (120°C) six specimens were prepared in Teflon container from the polymer solutions (Fig. 3-9). An illustration of the sand column experiments is shown in Fig. 3-11.



Figure 3-9 Teflon container for 120°C



Figure 3-10 Plastic container for 70°C

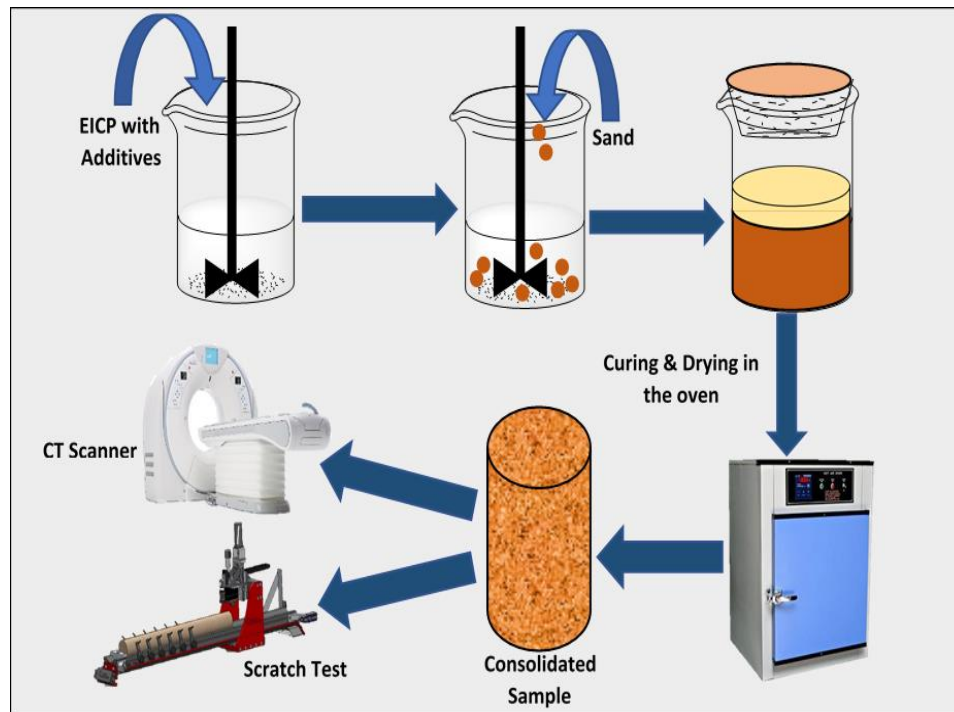


Figure 3-11 Procedure to Consolidate loose sand

After drying, the consolidated samples were gently tapped from the back side of the container to facilitate their removal. Fig. 3-12 shows all the samples after drying.



Figure 3-12 Consolidated samples after drying

3.7 Scratch Test

The strength of consolidated sand samples can also be evaluated using a scratch test. In this scenario, the surface of the consolidated sand sample would be intentionally scratched or grooved while measuring the force needed to do so using the scratch test machine. The adhesive strength of the consolidated sand, a measurement of its capacity to withstand the stresses and strains involved in oil and gas production, can then be calculated using this force. Researchers can assess how well sand consolidation methods, like Modified EICP, work to increase the strength and durability of sand formations by employing a scratch test equipment. We conducted a scratch test on all the samples to assess their strength. Notably, the sample prepared with xanthan gum at 120°C exhibited no strength at all and was completely damaged during the scratch test. Following the scratch test, we observed the presence of white precipitation within the sample prepared with polymers except xanthan gum at both 90°C and 120°C, as illustrated in Figure 3-13,3-14.



Figure 3-13 Scratch test machine (Epslog)

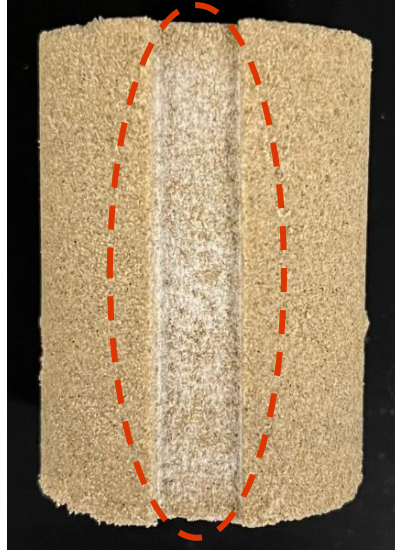


Figure 3-14 Scratch test samples after 70°C (All polymers except XG)

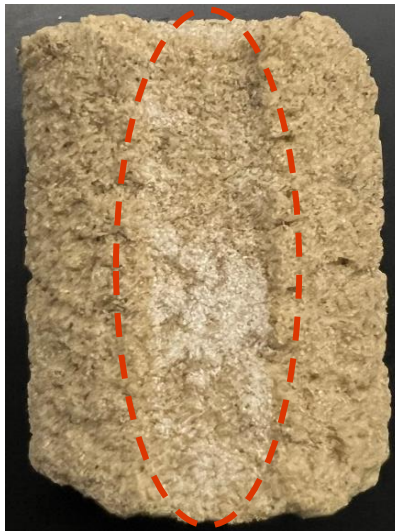


Figure 3-15 Scratch test samples after 120°C (All polymers except XG)

3.8 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA), which is widely used in chemistry, helps to understand what happens with the mass of a material when the temperature varies. In simple terms, there is a loss or gain of mass with respect to time as the sample is heated. TGA is easy to apply where we are looking at why materials break down, disintegrate, or burnout.

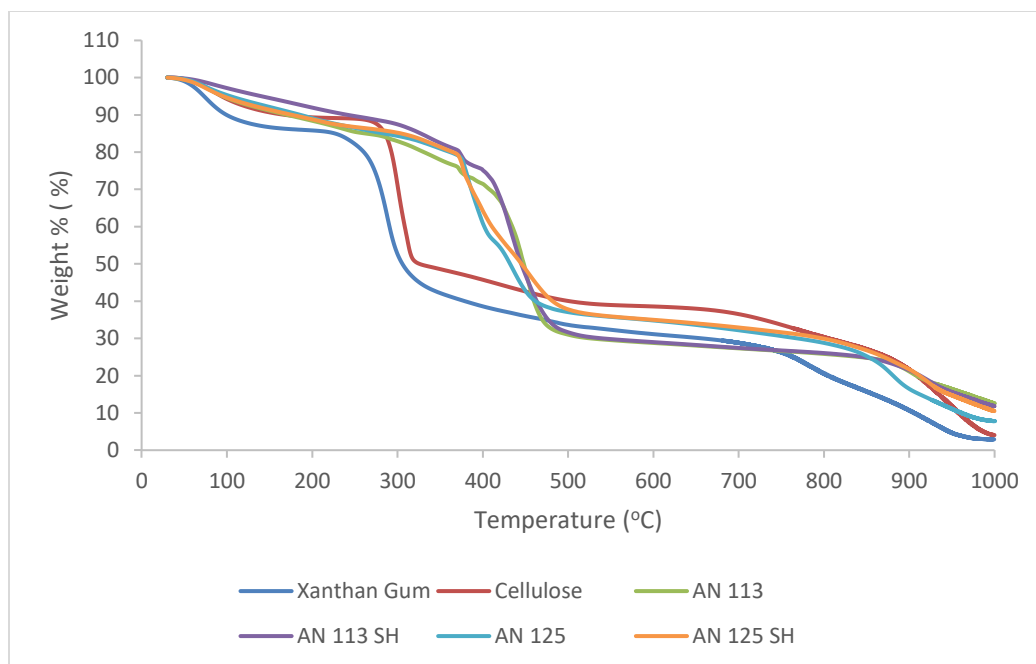


Figure 3-16 Thermogravimetric analysis of Bio and Synthetic AM-AMPS copolymers

We used TGA to examine the thermal stability of the both Bio and synthetic polyacrylamide polymers. The TGA curves for six samples are displayed in Figure 3. In each case, we conducted TGA tests by heating a set amount of the precipitated material to 1000°C at a rate of 20°C per minute. What we observed was that the Synthetic Copolymer of AM-AMPS has better thermal stability when compared to the Bio-Polymers. For instance, when we looked at xanthan gum, it began losing mass noticeably around 250°C, while cellulose showed significant mass loss after reaching 300°C. In contrast, for all the synthetic polyacrylamide polymers, mass degradation became significant only after they exceeded 400°C. Figure 3-16 illustrates that the loss of mass with increasing temperature is more pronounced for Bio-polymers than for synthetic Copolymers, making Synthetic Copolymer of AM-AMPS a more suitable choice for high-temperature applications.

CHAPTER

Results and Discussions

4.1 Effect of Temperature on baseline EICP

Scratch test was performed on specimens prepared with solution 1 for both that were cured for 72 hours and 48 hours. Specimen prepared with solution 1 cured at 70°C for 48 Hours and 72 hours shows the maximum strength of 216 Psi and 273 Psi respectively and Specimen prepared from solution 1 cured at 25°C for 48 Hours and 72 hours shows the weakest strength of 71 Psi and 80 Psi respectively. Specimen prepared with solution 1 cured at 90°C for 48 Hours and 72 hours shows the strength of 171 Psi and 179 Psi respectively. Baseline EICP solution shows best result at 70°C. In Fig. 8, it is evident that the strength of the consolidated specimen improves with longer curing time. This positive correlation between curing time and intrinsic specific energy (ISE) strength is observed across all specimens. The scratch test was conducted on consolidated sand samples, and the results revealed that the strength of the samples was not uniform across their length. Specifically, the highest strength was observed at the point where there was maximum precipitation, whereas the lowest strength was observed where there was minimum precipitation, as shown in Fig. 4-1. Additionally, it is noteworthy that the variation of strength was not significant for the samples cured at 70°C using solution 1, compared to those consolidated using solution 1 and cured at 25°C or 90°C. In comparison to the other samples, the specimen that was created using solution 1 and cured at 90°C showed the greatest strength variation. This outcome is explained by the substantial amount of

salammoniac in the sample, which led to less calcite precipitation in comparison to the other solutions. The results indicate that the salammoniac concentration can significantly affect how well the modified enzyme-induced carbonate precipitation approach consolidates sand forms. In order to maximize the consolidation process and guarantee dependable and consistent results, it is crucial to carefully manage the concentration of all the chemical components in the solution.

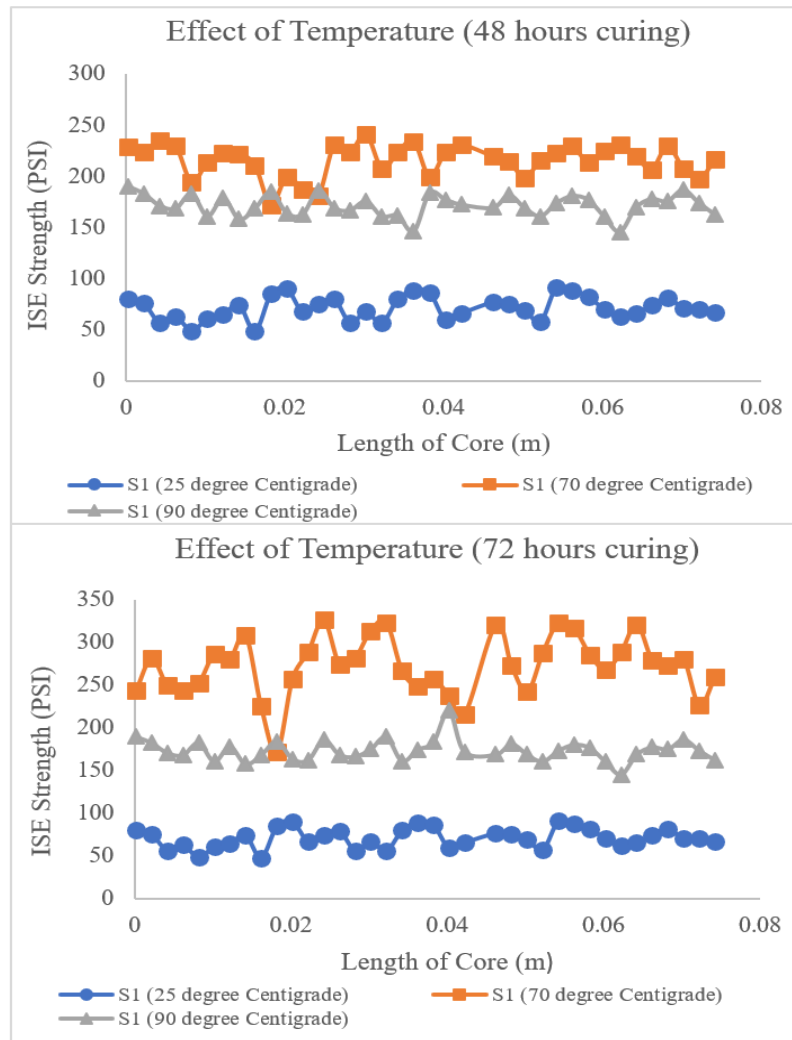


Figure 4-1 ISE strength vs Length of core (Effect of Temperature)

4.2 Effect of salts on EICP solution

Scratch test was performed specimens prepared with solutions 2-4 for both that were cured for 72 hours and 48 hours. Specimen prepared with solution 4 prepared with using both salts (CaCl_2 and MgCl_2 , 0.67 M concentration each) cured at 70°C for 48 Hours and 72 hours shows the maximum strength of 350 Psi and 370 Psi respectively and Specimen prepared from solution 2 (0.67 M MgCl_2 as a salt) cured at 70°C for 48 Hours and 72 hours shows the weakest strength of 167 Psi and 185 Psi respectively. Specimen prepared with solution 3 prepared with using both salts (CaCl_2 and MgCl_2 , 0.33 M concentration each) cured at 70°C for 48 Hours and 72 hours shows the strength of 268 Psi and 273 Psi. Strength of specimen prepared with solution 1 cured at 70°C already discussed previously, that is 216 Psi and 273 Psi for 48 hours and 72 hours respectively. EICP solution shows best result when we use both salts in a solution. In Fig. 4-2, it is also evident that the strength of the consolidated specimen improves with longer curing time. This positive correlation between curing time and strength is observed across all specimens. Fig. 4-2 further demonstrates that, although the strength was not consistent throughout the core, the specimen created with solution 4 demonstrated the maximum strength. It is interesting to note that Dolomite precipitation was responsible for the specimens made from solutions 3 and 4 having the highest strength point on the graph. The specimen made with solution 2 showed the least amount of strength variation over the length of the core, and overall, it was less strong than the other specimens.

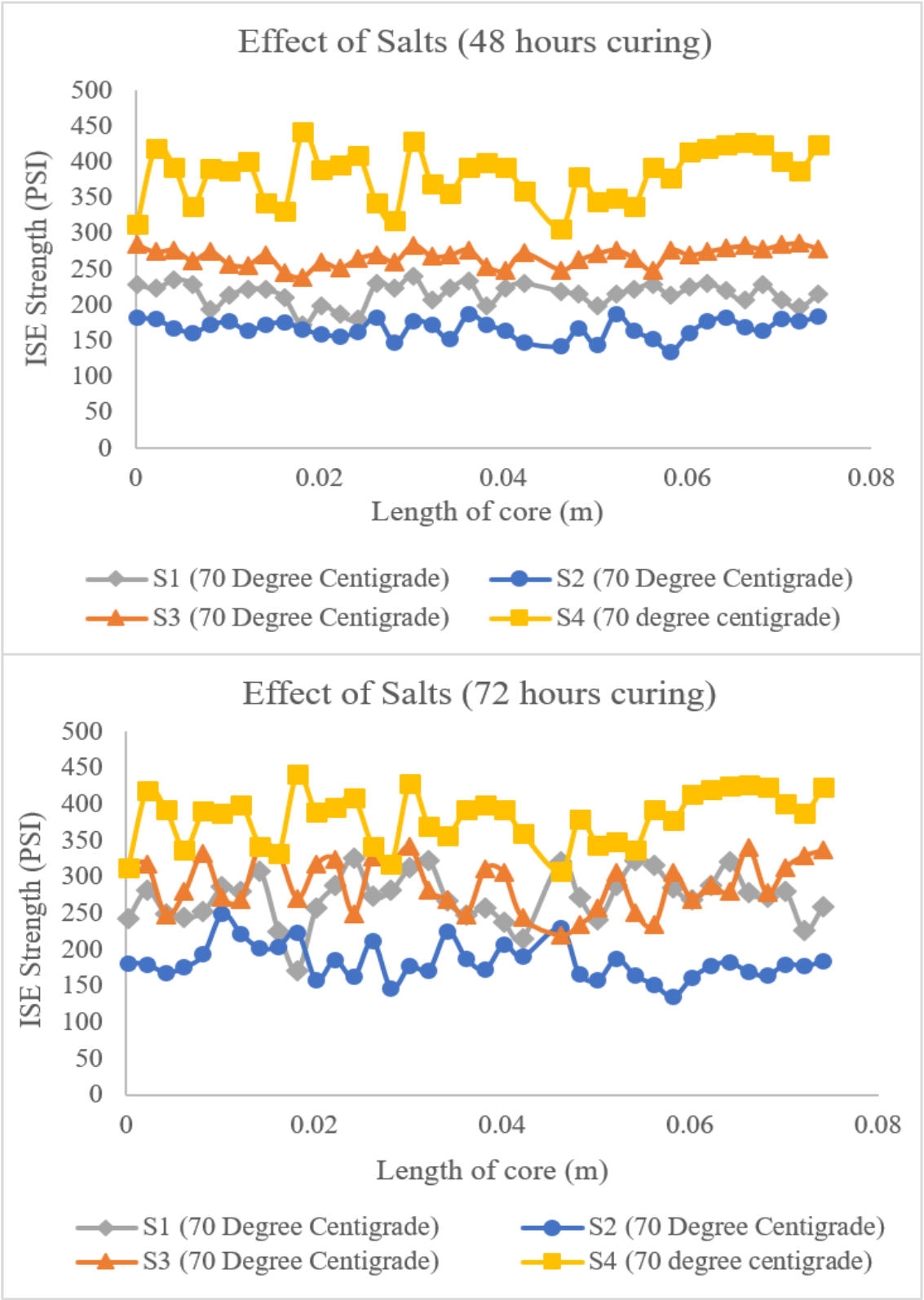


Figure 4-2 ISE strength vs Length of core (Effect of salts)

4.3 Effect of Xanthan gum on EICP solution

The scratch test was conducted on specimens that were prepared using solutions 5-7, that was cured for 72 and 48 hours. The specimen prepared with solution 7, which had a concentration of 3 g/L xanthan gum cured at 70°C for 48 Hours and 72 hours exhibited the highest strength of 520 Psi and 550 Psi respectively, while the specimen prepared with solution 5, with a concentration of 1 g/L xanthan gum cured at 70°C for 48 Hours and 72 hours showed the lowest strength of 400 Psi and 410 psi respectively. The specimen prepared with solution 6, which had a concentration of 2 g/L xanthan gum cured at 70°C for 48 Hours and 72 hours exhibited the moderate strength of 460 Psi and 475 Psi respectively. Notably, there was a clear trend of increasing strength with higher concentrations of xanthan gum. Additionally, Fig. 4-3 revealed that longer curing times were associated with improved strength of the consolidated specimen. This positive correlation between curing time and strength was observed consistently across all specimens. As the concentration of xanthan gum rose, Fig. 4-3 clearly shows, the strength of the cemented sand samples also increased. Because xanthan gum can condense into a sticky, glue-like substance that contain significant amount of carnallite, all three samples made with it showed great strength. Despite the fact that these samples' strengths did vary along the length of the core, the difference was not very large, it was around 100 Psi. These findings imply that xanthan gum concentration may be a significant factor in determining the strength of the consolidated sand samples, and that great thought should be made to maximizing the concentration to attain the desired strength and uniformity.

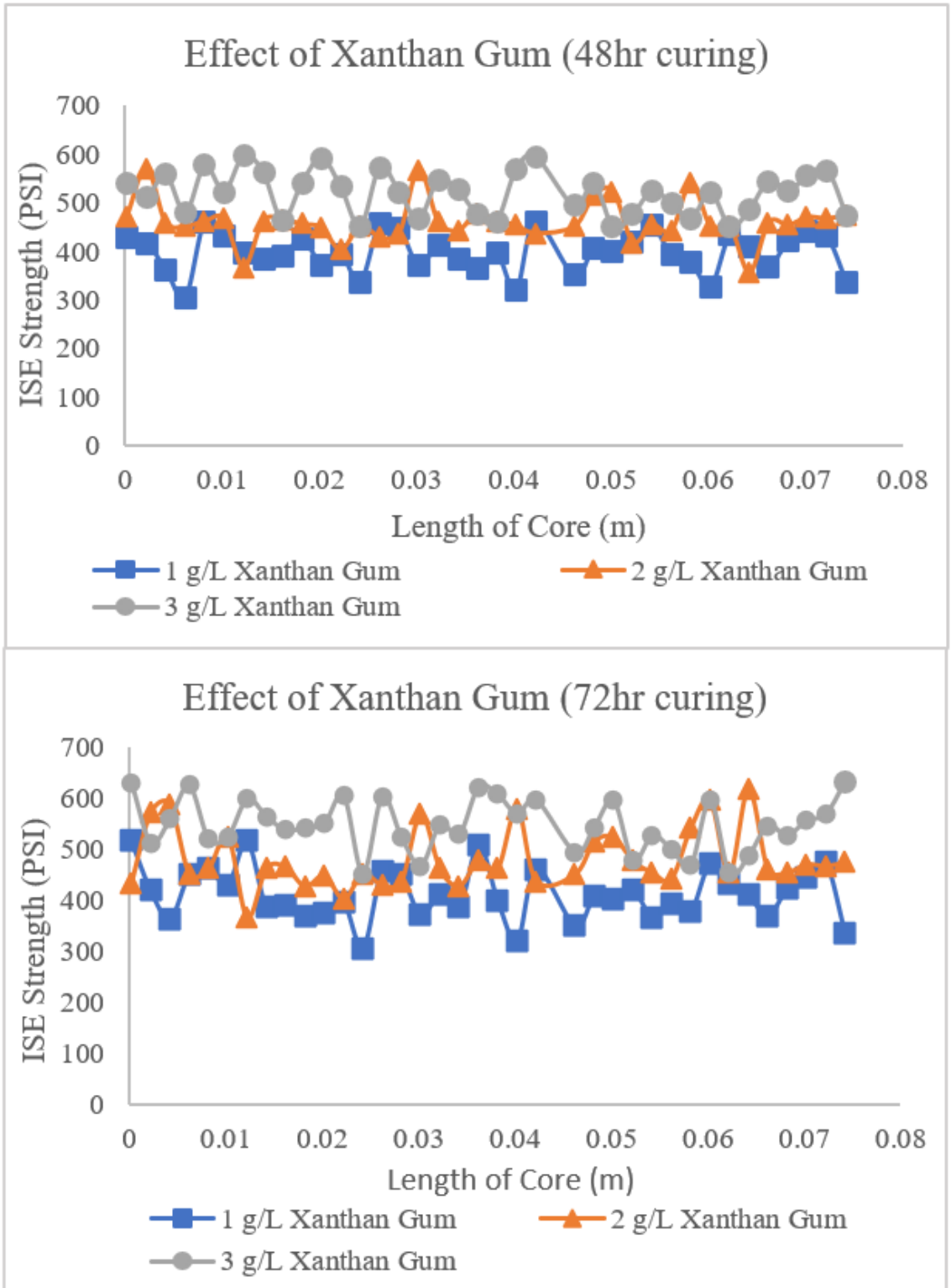


Figure 4-3 ISE strength vs Length of core (Effect of XG)

4.4 Effect of Cellulose on EICP solution

The scratch test was conducted on specimens that were prepared using solutions 8-10, that was cured for 72 and 48 hours. The specimen prepared with solution 10, which had a concentration of 3 g/L cellulose cured at 70°C for 48 Hours and 72 hours exhibited the highest strength of 1081 Psi and 1100 Psi respectively, while the specimen prepared with solution 8, with a concentration of 1 g/L cellulose cured at 70°C for 48 Hours and 72 hours showed the lowest strength of 460 Psi and 485 psi respectively. The specimen prepared with solution 9, which had a concentration of 2 g/L cellulose cured at 70°C for 48 Hours and 72 hours exhibited the moderate strength of 848 Psi and 876 Psi respectively. Notably, there was a clear trend of increasing strength with higher concentrations of cellulose. Additionally, Fig. 4-4 revealed that longer curing times were associated with improved strength of the consolidated specimen. This positive correlation between curing time and strength was observed consistently across all specimens. As the concentration of cellulose rose, Fig. 4-4 clearly shows, the strength of the cemented sand samples also increased. Because cellulose precipitate contain significant amount of calcite, all three samples made with it showed great strength. Despite the fact that these samples' strengths did vary along the length of the core, the difference was not very large, it was around 100 Psi. These findings imply that cellulose concentration may be a significant factor in determining the strength of the consolidated sand samples, and that great thought should be made to maximizing the concentration to attain the desired strength and uniformity.

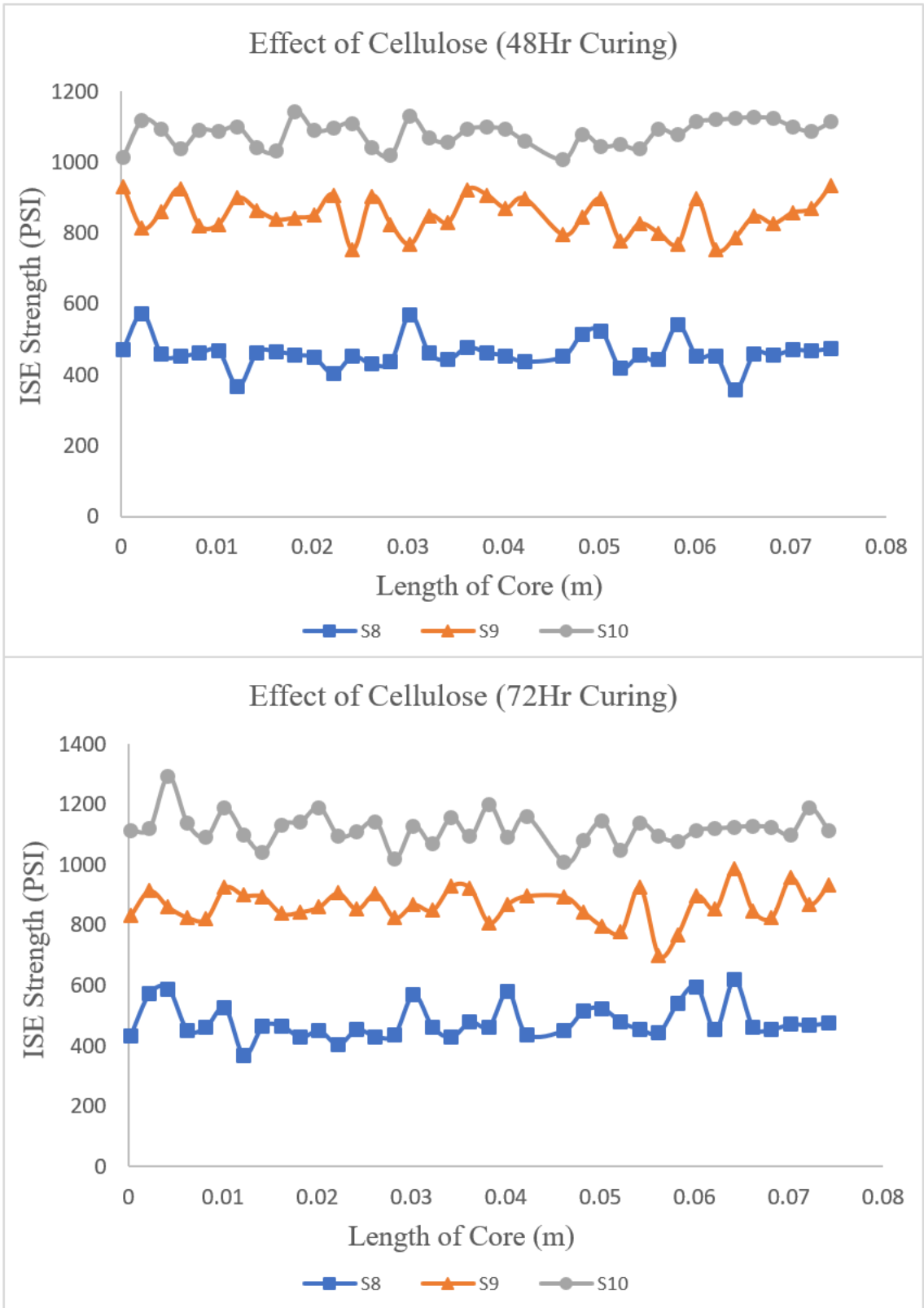


Figure 4-4 ISE strength vs Length of core (Effect of Cellulose)

4.5 Effect of high temperature on EICP solution

4.5.1 Strength of solution 4 EICP Solution

We conducted a scratch test on the samples prepared with Solution 4, which were cured at both 70°C and 120°C. The results showed that at 70°C, the samples exhibited a strength of 370 Psi, while at 120°C, their strength decreased to 21.7 Psi (Fig. 4-5). The reason for the decrease in sample strength at high temperatures can be attributed to the lower mass of precipitation at 120°C when compared to 70°C, as indicated in Table 2. Interestingly, at high temperatures, the strength remained consistent throughout the core length. In contrast, at lower temperatures, the strength distribution along the core was not uniform, as depicted in Figure 4-5.

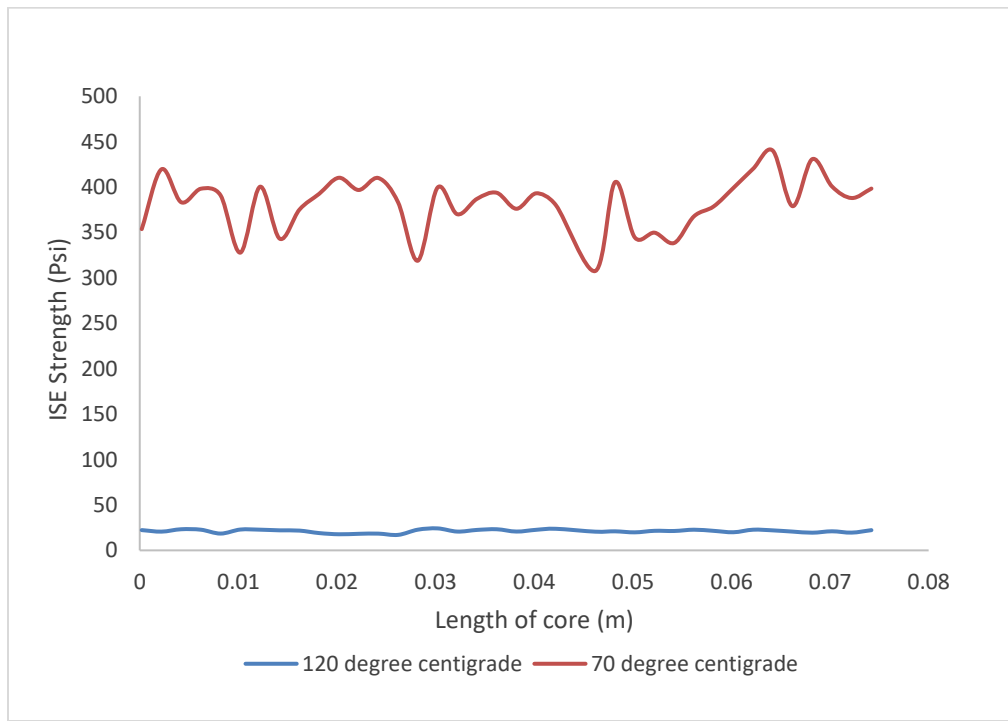


Figure 4-5 ISE strength vs Length of core (Solution 4)

4.5.2 Effect of Bio-Polymers on EICP Solution

We conducted a scratch test on specimens prepared with Solution 7, containing xanthan gum, and Solution 10, which consists of cellulose. These specimens were cured at both 70°C and 120°C. At 70°C, the solution with xanthan gum displayed a substantial strength of 475 Psi. However, at the higher temperature of 120°C, it exhibited no strength at all. On the other hand, the specimen prepared with Solution 3 (Cellulose) showed a strength of 1100 Psi at 70°C, which slightly decreased to 918 Psi when tested at 120°C (Fig. 4-6).

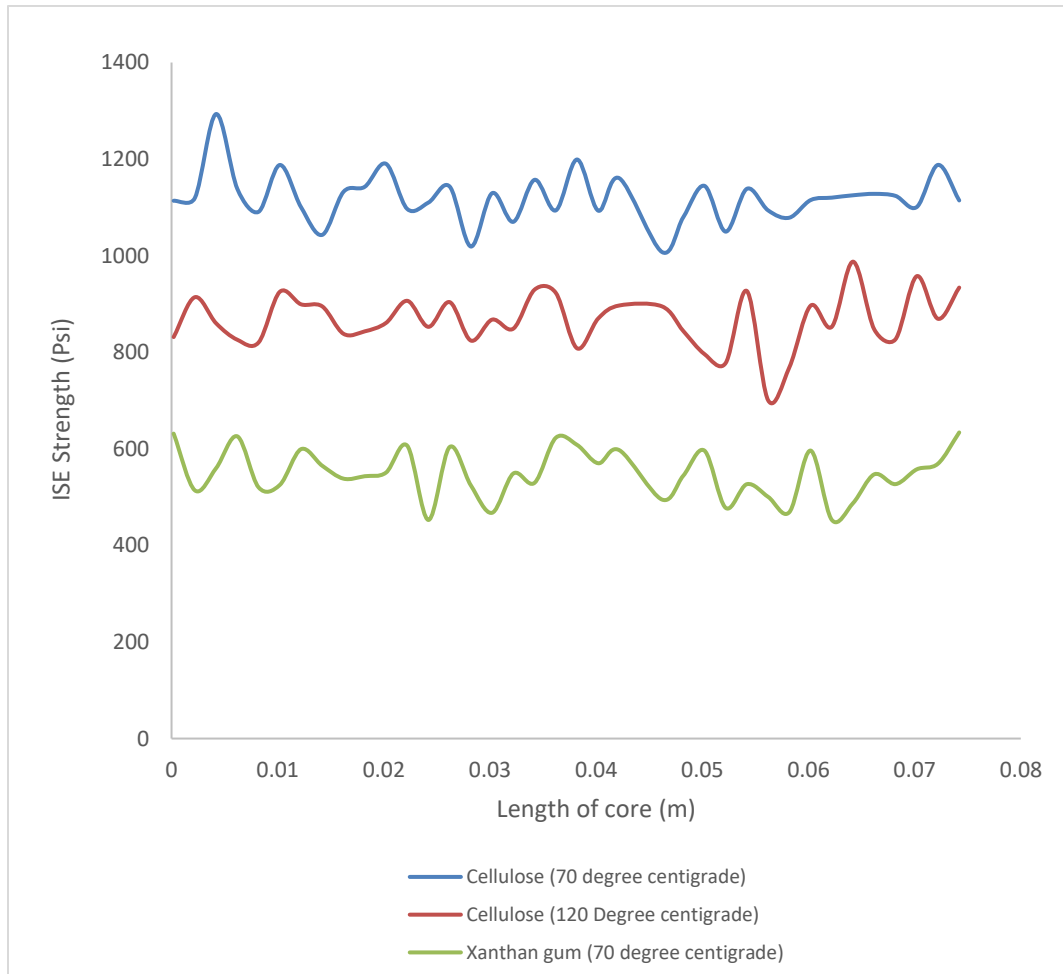


Figure 4-6 ISE strength vs Length of core (Polymers)

The drop-in sample strength at higher temperatures can be linked to the fact that there's less solid precipitation at 120°C, as illustrated in Table 2, compared to what we see at 70°C. It's noteworthy that all the specimens exhibit uneven distribution of strength along the length of the core. The impressive strength observed in the cellulose specimen is attributed to the precipitation of carnallite at 70°C and Salammoniac at 120°C (Table 3). These mineral formations significantly contribute to the sample's overall strength.

4.5.3 Effect of Synthetic AM-AMPS on EICP Solution

We conducted a scratch test on specimens prepared with AM-AMPS copolymers that had been cured at both 70°C and 120°C. The results showed some interesting findings. For the specimens prepared with AN 113, we observed a strength of 1607 psi at 70°C, and remarkably, the strength at 120°C remained almost the same at 1597 psi. Similarly, for the specimens prepared with AN 113 SH, the strength was 1030 psi at 70°C, and it showed minimal change at 120°C, registering at 1025 psi. Moving on to the specimens prepared with AN 125, they exhibited a strength of 2175 psi at 70°C, and at 120°C, the strength slightly decreased to 2155 psi. The specimens prepared with AN 125 SH displayed a strength of 1800 psi at 70°C, with a minimal drop to 1790 psi at 120°C, specimen prepared with AN 125 shows the maximum strength on both temperatures as depicted in Figure 4-7. It's noteworthy that we observed no significant change in strength for both 70°C and 120°C in the case of these synthetic AM-AMPS co-polymers. This lack of change can be attributed to their high degradation temperature, as shown in Figure 3-16. Among all the specimens, AN 125 consistently demonstrated the highest strength, whether at 70°C or 120°C. Conversely, AM 113 SH exhibiting the weakest strength at both temperatures. Furthermore, it's important to note that all the specimens displayed an uneven distribution

of stress along the core sample. The impressive strength observed in AN 125 is primarily due to the presence of a significant amount of Salammoniac at 70°C and a substantial amount of carnallite at 120°C. These minerals play a crucial role in contributing to the overall strength of the specimen.

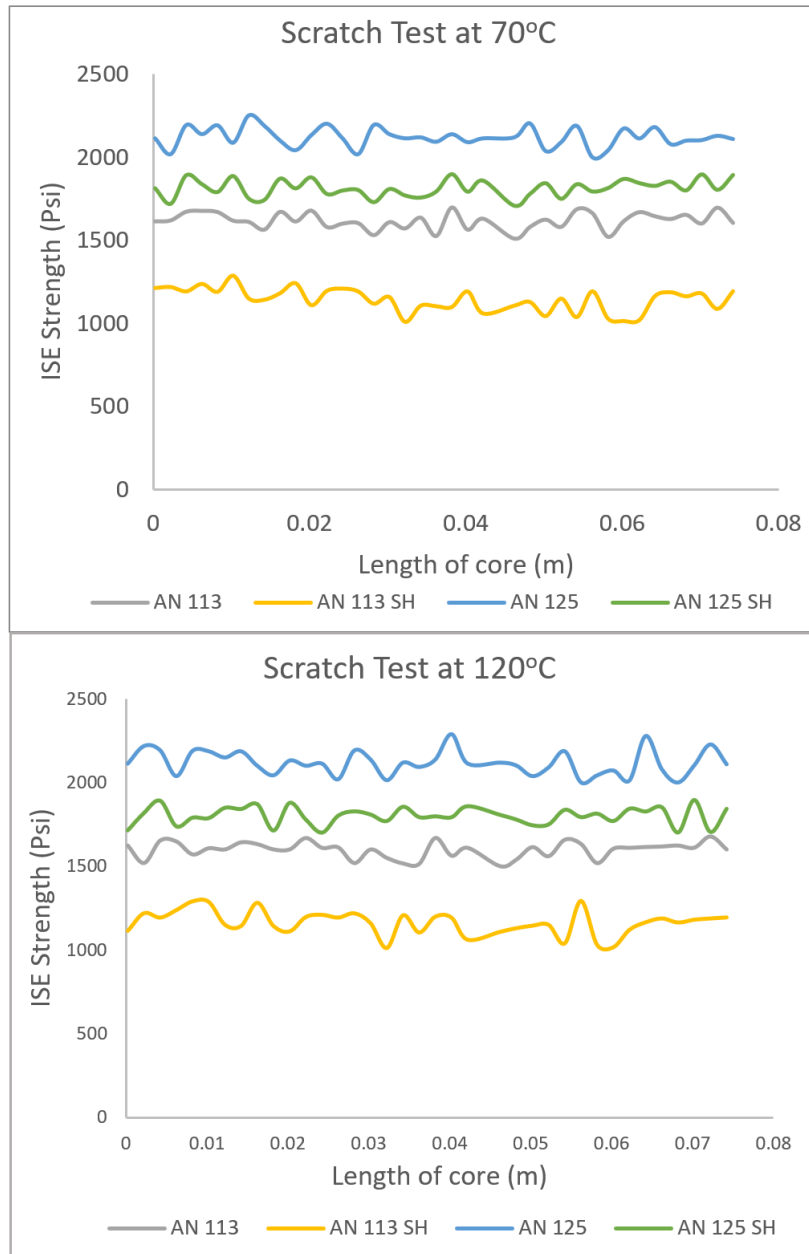


Figure 4-7 ISE strength vs Length of core (Effect of AM-AMPS)

Table 4-1 Overall summary

S No.	Formulation	Temperature	Mass of precipitation (gm)	ISE Strength (PSI)
1	1 M Urea, 0.67 M CaCl ₂ , and 3 g/L Urease	70°C	3.57	273
2	1 M Urea, 0.67 M MgCl ₂ , and 3 g/L Urease	70°C	2.87	185
3	1 M Urea, 0.33 M CaCl ₂ , 0.33 M MgCl ₂ , and 3 g/L Urease	70°C	4.12	273
4	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , and 3 g/L Urease	70°C	5.86	370
5	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , and 3 g/L Urease	120°C	2.33	21.7
6	1 M Urea, 0.67 M CaCl ₂ , 0.33 M MgCl ₂ , 3	70°C	11.03	410

	g/L Urease and 1 g/L Xanthan Gum			
7	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3 g/L Urease and 2 g/L Xanthan Gum	70°C	11.55	475
8	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3 g/L Urease and 3 g/L Xanthan Gum	70°C	12.10	550
9	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3 g/L Urease and 3 g/L Xanthan Gum	120°C	4.56	Fail
10	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3 g/L Urease and 1 g/L Cellulose	70°C	8.53	485
11	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3	70°C	12.61	876

	g/L Urease and 2 g/L Cellulose			
12	1 M Urea, 0.33 M CaCl ₂ , 0.33 M MgCl ₂ , 3 g/L Urease and 3 g/L Cellulose	70°C	13.14	1100
13	1 M Urea, 0.33 M CaCl ₂ , 0.33 M MgCl ₂ , 3 g/L Urease and 3 g/L Cellulose	120°C	13	918
14	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3 g/L Urease and 3 g/L AN 113	70°C	13.36	1607
15	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3 g/L Urease and 3 g/L AN 113	120°C	13.11	1597
16	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3	70°C	11.53	1030

	g/L Urease and 3 g/L AN 113 SH			
17	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3 g/L Urease and 3 g/L AN 113 SH	120°C	11.53	1025
18	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3 g/L Urease and 3 g/L AN 125	70°C	12.42	2175
19	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3 g/L Urease and 3 g/L AN 125	120°C	12.35	2155
17	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3 g/L Urease and 3 g/L AN 125 SH	70°C	11.71	1800
17	1 M Urea, 0.67 M CaCl ₂ , 0.67 M MgCl ₂ , 3	120°C	11.56	1790

	g/L Urease and 3 g/L AN 125 SH			
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4.5.4 Computed Tomography Scan

To establish a baseline for comparison, a container filled solely with sand was included in the study and subjected to CT scanning. This container served as a reference point for analyzing the CT numbers of the consolidated specimens prepared in separate containers. By comparing the CT numbers of the consolidated samples with the CT number of the sand-only container, we were able to detect the presence of calcite, dolomite, and carnallite precipitation in the specimens. Specifically, an increase in the average CT number from the sand-only container indicated the precipitation of calcites, dolomites and other precipitation. All the scans were conducted using a using Toshiba Alexion TSX-032A Medical CT scanner (spatial resolution =1 mm). Voxclc software was used for analyzing the CT tomograms to obtain the CT number of each sample. Table 6 shows the average CT number of all the dried consolidated specimens and the average CT number subtracted by the base case CT number.

A study was conducted in which they used a chelating agent, glutami-N,N-diacetic acid(GLDA) to stimulate the sandstone cores. In the core flood tests, several GLDA solution pH levels (ranging from 1.7 to 13) were used. To find out how GLDA affected the core, the sandstone cores were scanned both before and after the treatment. CT number of all cores that were treated with GLDA was decreased showing that the porosity of each sample increased. This decrease in porosity indicates that sandstone is stimulated and the

precipitates of calcite, dolomite, and quartz inside the sandstone are removed from it (Mahmoud et al., 2011). The precipitation of minerals such as calcite, dolomite, and quartz can be inferred from an increase in CT number from the base case of only sand. As the CT number of calcites is around 2000 and that of dolomite is approximately 2200, a greater increase in average CT number from the base case indicates a higher degree of calcite and dolomite precipitation.

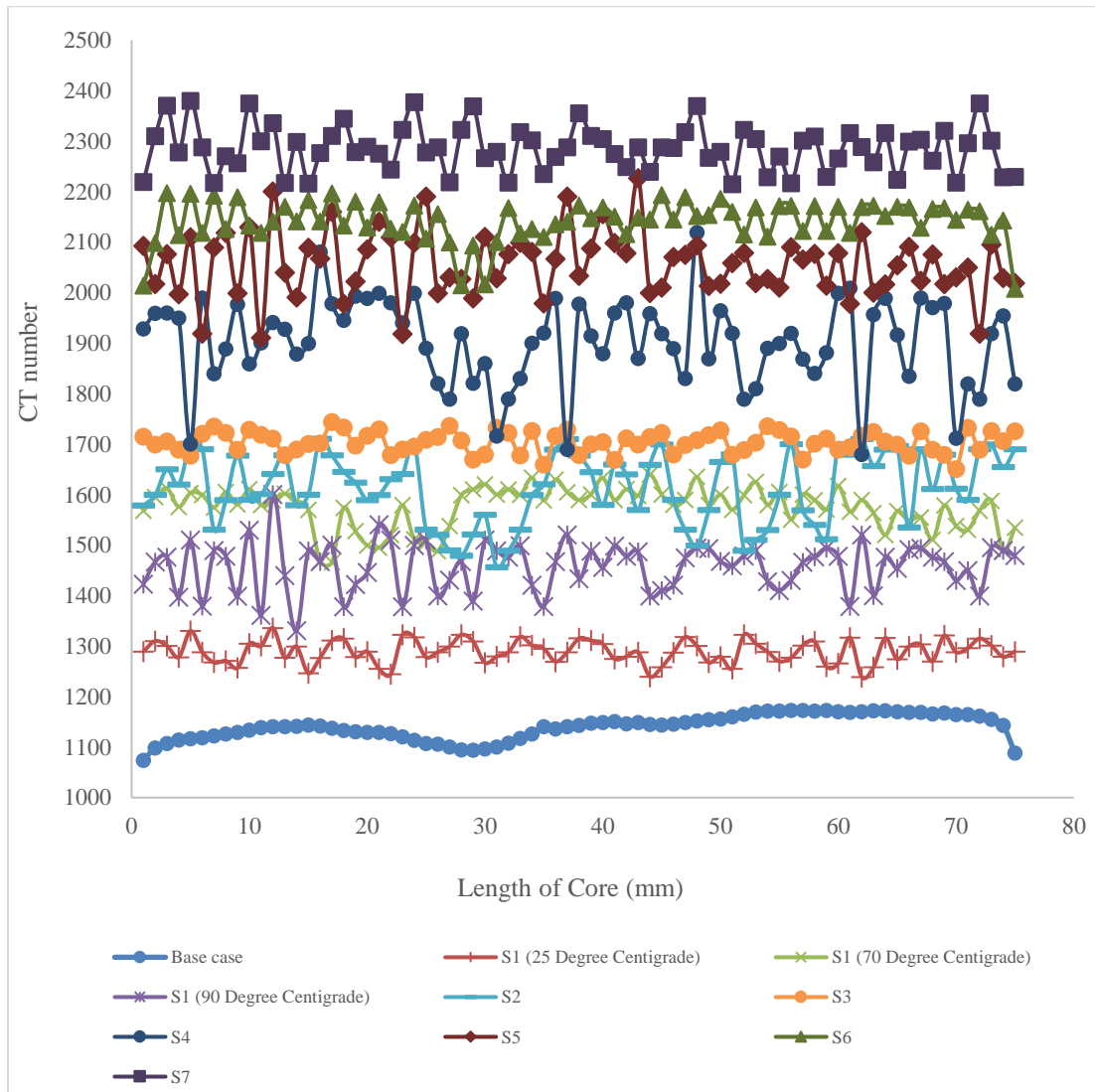


Figure 4-8 Plot between CT number and Length of the core

As evidenced by Table 5, specimens prepared from solutions 2 and 3 exhibit a significant increase in average CT number due to the presence of not only calcites but also a substantial amount of dolomite. Similarly, the specimen prepared with solution 1 (70°C) displays a marked increase in average CT number, owing to its high calcite content. Conversely, the tube prepared with solution 1 (25°C) shows only minimal increase in average CT number because mass of precipitation is quite low in it (Table 2). The specimen prepared from solution 1 (90°C) also shows an increase in the average CT number, which can be attributed to the high precipitation of salammonic in it. Furthermore, the specimen prepared with xanthan gum displays a substantial increase in average CT number, likely due to the precipitation of carnallite. Notably, the specimen prepared with 3 g/L xanthan gum exhibits the highest increase in average CT value (Table 5). CT number increases with decreasing porosity, meaning greater amounts of minerals such as calcite, dolomite, and another minerals precipitate are present. Fig. 4-8 clearly shows that an increase in CT number corresponds to a decrease in porosity. The specimen that was prepared with xanthan gum had the greatest reduction in porosity, while the MICP solution that was prepared with both magnesium chloride and calcium chloride salts had the second-highest decrease. Table shows average CT number for all samples.

Table 4-2 Average CT number of Dried Consolidated Specimens subtracted by base case

Sample	Average CT number	Average CT No. – Base case
Solution 1 (25°C)	1289.58	149.51
Solution 1(70°C)	1581.22	435.11

Solution 1(90°C)	1458.54	318.48
Solution 2 (70°C)	1613.67	473.60
Solution 3 (70°C)	1704.64	564.58
Solution 4 (70°C)	1880.23	740.16
Solution 5 (70°C)	2058.67	918.61
Solution 6 (70°C)	2141.53	1001.46
Solution 7 (70°C)	2286.12	1145.94

4.6 Sand-pack Flooding

We set up a sand pack in a plastic container filled with loose sand. The container has an inlet and outlet connected to a pressure transducer. We have a pump to inject the water at different rates after pumping the water we monitor the pressure drop on a screen (see Fig. 4-11,4-12). We also use accumulator after the pump in order to easily pump the viscous polymer fluid (Fig. 4-10). For our EICP solution, we chose two polymers: cellulose and AN 125. Cellulose is known for its strength among biopolymers, and AN 125 is the strongest among copolymers of AM-AMSH. We tested three injection rates (4 cc/minute, 8 cc/minute, and 12 cc/minute) for both cellulose and AN 125. Initially, we injected DI water into loose sand samples to calculate their initial permeability. After that, we injected cellulose solution and AN125 solution into separate containers. These containers were sealed and placed in an oven for curing at 70°C for 72 hours. After three days, we injected

DI water again at three different rates to calculate the permeability after the treatment. To determine permeability, we used Darcy's law (eq. 7):

$$K_1 = \frac{q\mu L}{A\Delta P} \quad (7)$$

Here: $q = \text{cc/sec}$, $\Delta P = \text{atm}$, $A = \text{cm}^2$, $L = \text{cm}$ and $\mu = \text{cP}$

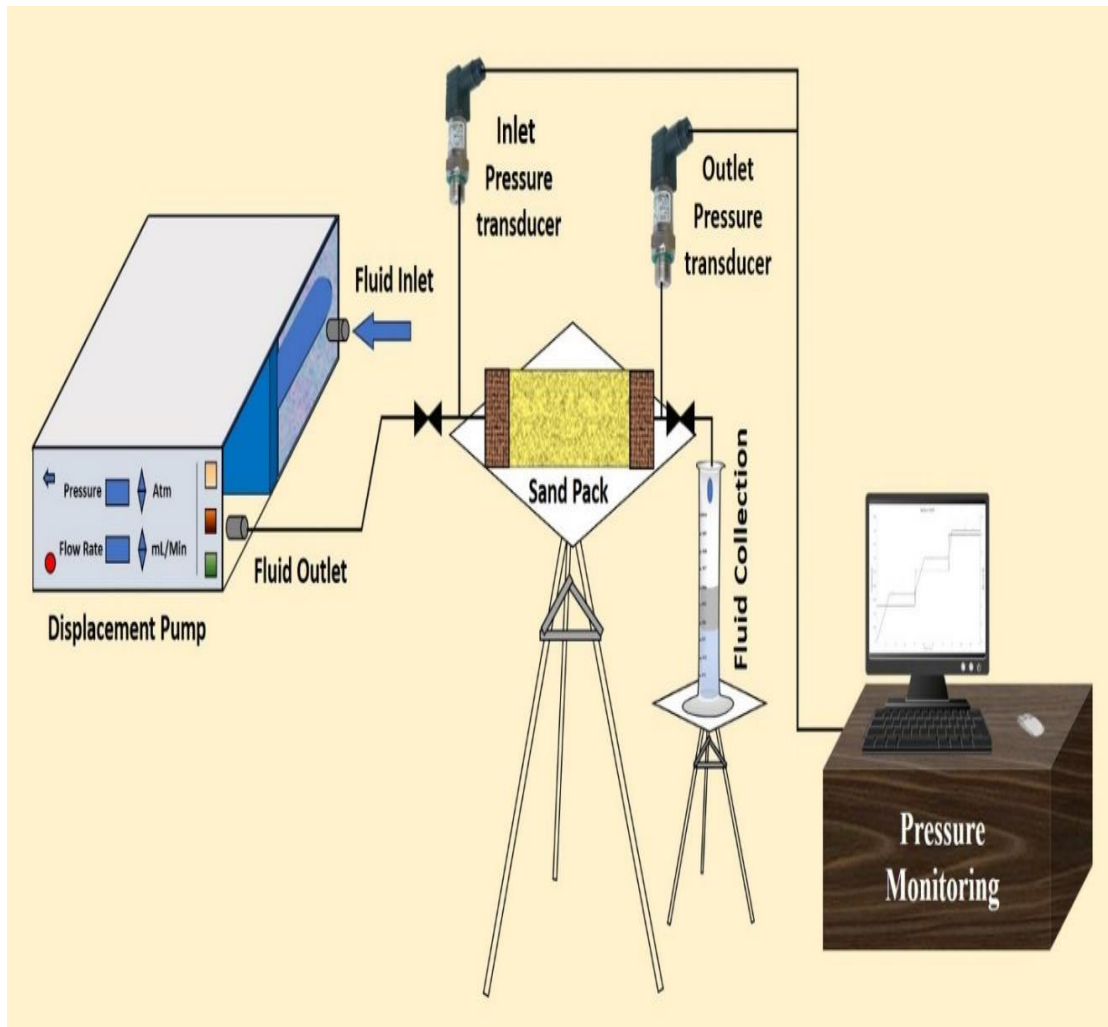


Figure 4-9 Sand-pack schematic diagram

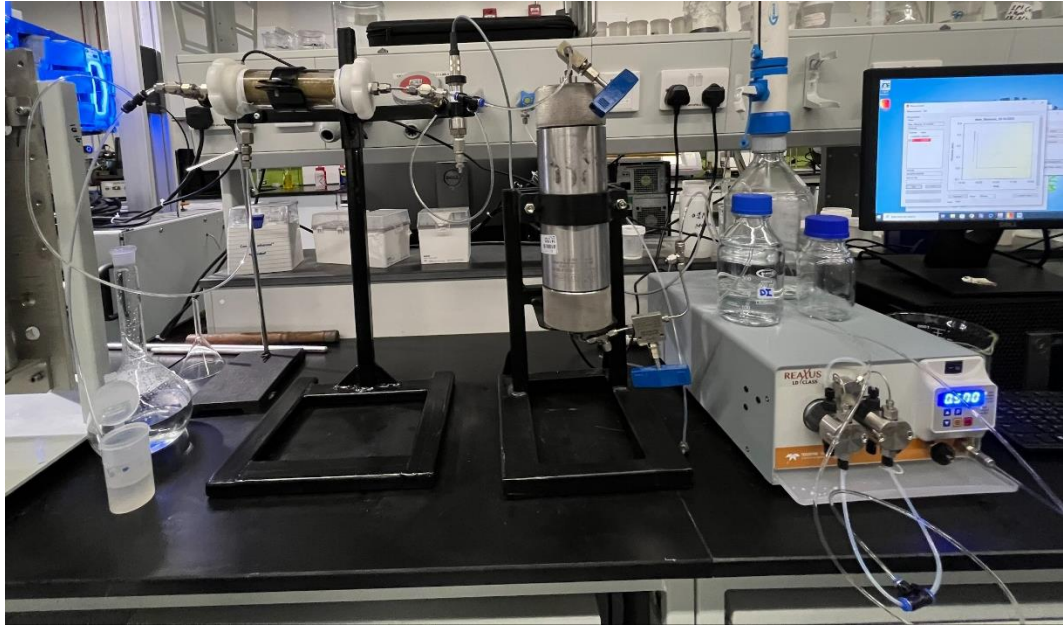


Figure 4-10 Sand Pack flooding setup

For the Cellulose solution (Solution 10), the permeability before the EICP treatment was roughly 16 darcy. After treating it with the EICP solution containing cellulose, the permeability dropped to 8.2 darcy, marking about a 50% reduction in permeability. These calculations were based on the pressure drop observed during sand flooding (see Fig. 4-10). As for the AN 125 solution (Solution 13), the initial permeability was around 20 darcy. After the EICP treatment with the AN 125 solution, the permeability decreased to 14.2 darcy, indicating a roughly 30% reduction. These permeability values were determined from the pressure drop observed in the sand flooding experiments (see Fig. 4-11). These results were obtained at 70°C, but it's anticipated that similar outcomes would be observed at higher temperatures, such as 120°C. This expectation is based on the fact that both polymers are not significantly affected by temperatures up to 120°C (see Fig. 3-16). Additionally, the precipitation mass is consistent for both polymers at 120°C (Table 2). In

practical terms, AN 125 seems to be more suitable for petroleum field applications due to its maximum strength and minimal reduction in permeability compared to cellulose.

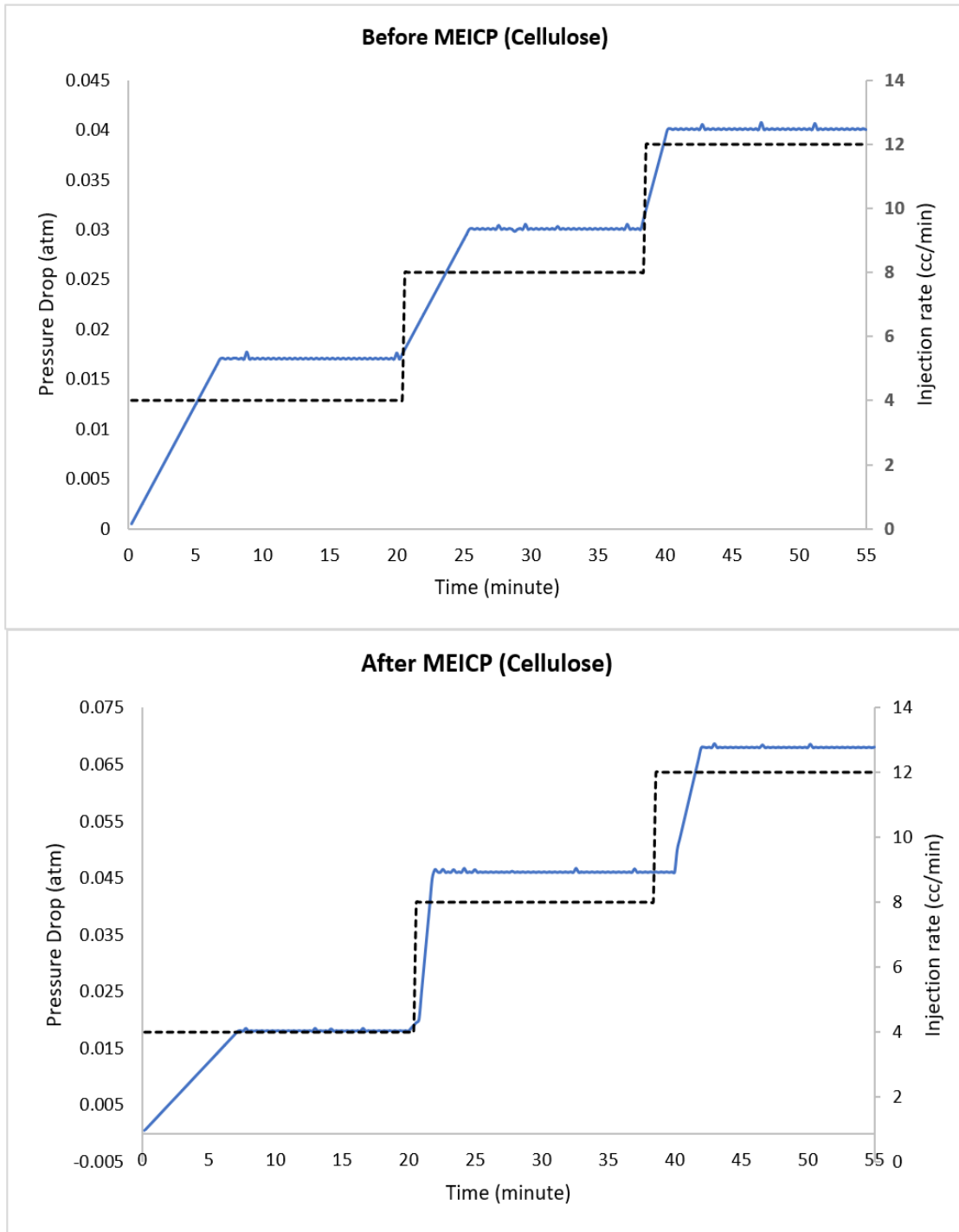


Figure 4-11 Pressure drop profile vs time before and after treatment with EICP solution (Cellulose)

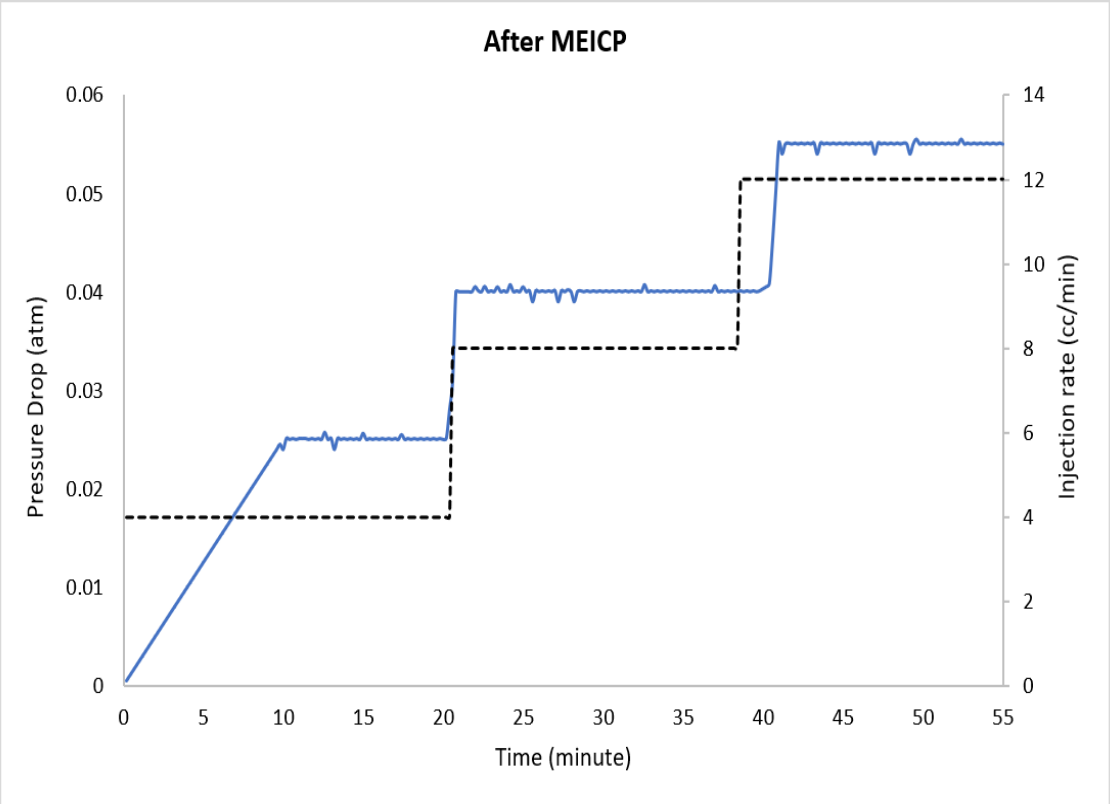
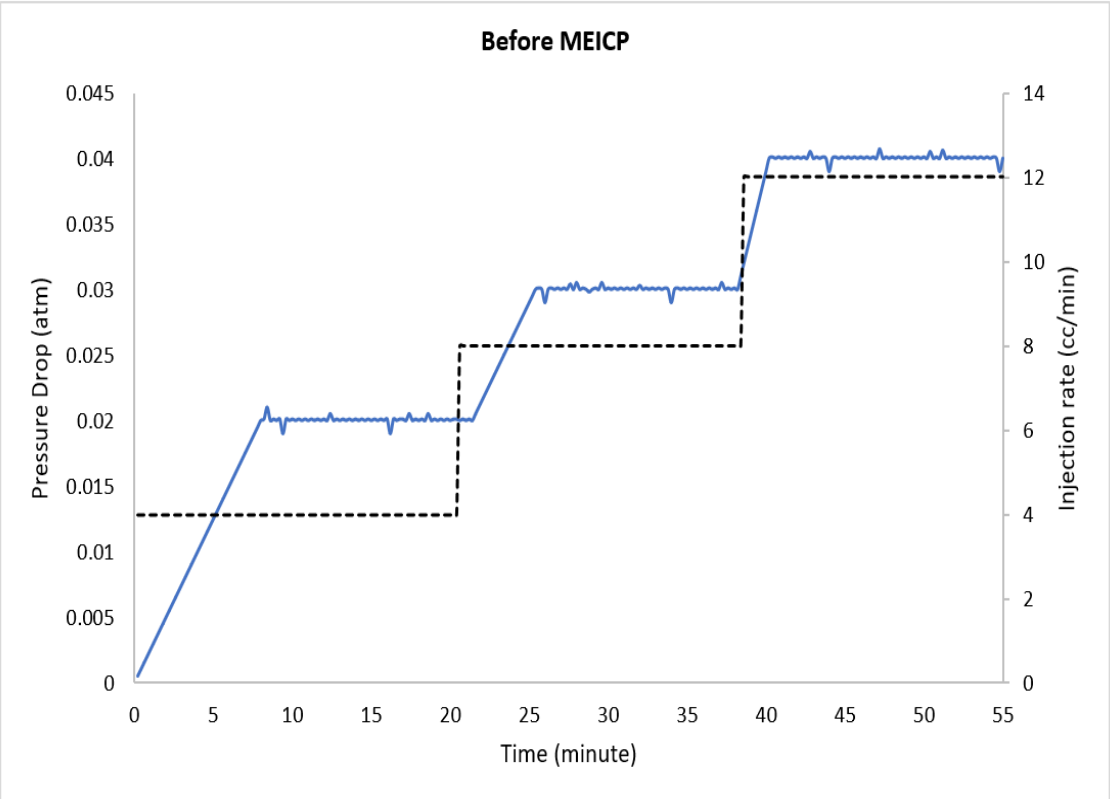


Figure 4-12 Pressure drop profile vs time before and after treatment with EICP solution (AN 125)

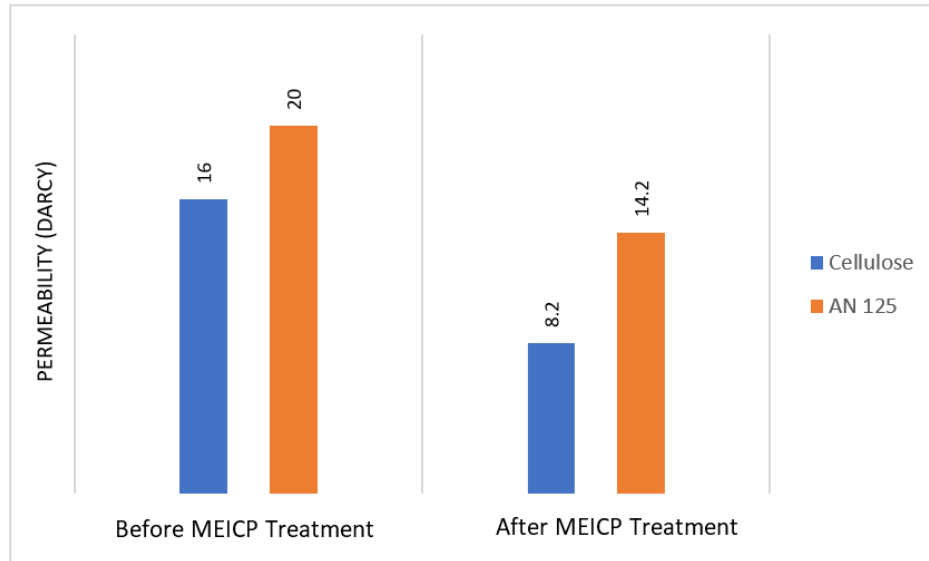


Figure 4-13 Permeability before and after EICP treatment (Cellulose & AN 125)

Following the earlier discussion, it's noted that AN 125 causes less reduction in permeability compared to Cellulose (Fig. 4-12). To assess sand consolidation, after calculating the pressure drop, one side of the container was opened, and injection was done from the other side at a rate of 12 cc/minute. The results showed that around 60% of sand was consolidated for Cellulose, while for AN 125, it was 75% (see Fig. 4-13).

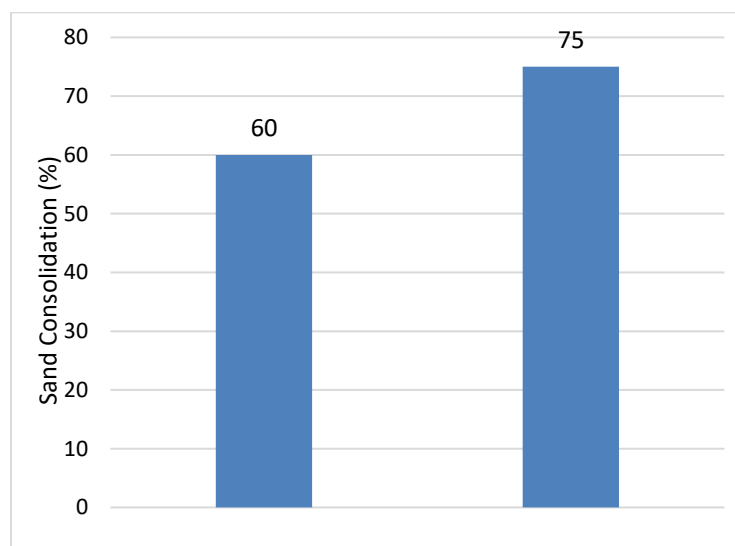


Figure 4-14 Sand production after MEICP treatment (Cellulose & AN 125)

CHAPTER

Conclusion

In this work, a modified EICP solution was developed using two types of salts (CaCl_2 and MgCl_2) with urea, urease, bio-polymers and copolymers of AM-AMPS. This solution showed promising results in consolidating sand to help prevent sand production in oil or gas wells. Loose sand has been consolidated using the developed modified EICP solution and showed an ISE strength up to 2175 psi at a high temperature of 120°C . Through various experiments, we uncovered some significant findings:

1. An effective sand consolidation can be achieved using the modified EICP solution developed in this work.
2. Laboratory results indicate that EICP solution exhibits maximum strength at 70°C and weakest at 25°C , indicating that curing at 70°C is preferable.
3. When preparing EICP solution, a combination of CaCl_2 and MgCl_2 salts shows the highest strength, suggesting that it is preferable to use a mixture of both salts.
4. Xanthan gum enhances strength in EICP solutions up to 70°C but degrades and loses effectiveness at 120°C .
5. Among bio-polymers, cellulose emerged as the strongest option at both 70°C and 120°C .
6. Synthetic copolymers of AM-AMPS are all stable at 120°C .
7. Among these synthetic copolymers, AN 125 stands out for providing the maximum strength (2175 PSI).

8. Sand pack flooding experiments revealed that treating loose sand with an EICP solution containing AN 125 resulted in less permeability reduction and sand production. This establishes AN 125 as the best additive for EICP solutions, justifying its use in the petroleum field.

Future Recommendation

1. Reducing curing time of MEICP solution.
2. Find alternative of urease enzymes.
3. Use MICP solution on the produced sand from oil well.
4. Use of MICP at very high temperature (200°C).
5. To make MICP solution more cost effective.

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- Investigation of Modified Enzyme-Induced Carbonate Precipitation for Sand Production Control Applications (Submitted in SPE Journal-Minor reviews received).
- Assessing the Viability of Different Bio-Polymers and Synthetic-Copolymers with Modified Enzyme-Induced Carbonate Precipitation (MEICP) Solutions for Sand Consolidation Applications (To be Submitted in Fuel).
- Using a new kind of Bio-polymer in Modified Enzyme-Induced Carbonate Precipitation for Sand Consolidation to control sand production (Patent Filed).
- Enhancing Sand Consolidation and Production Control through Novel Synthetic Polymers in Modified Enzyme-Induced Carbonate Precipitation (Patent Filed).