

**SAND CONSOLIDATION USING
ENZYME-INDUCED CARBONATE
PRECIPITATION**

BY

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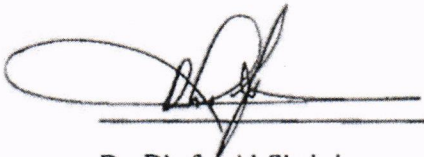
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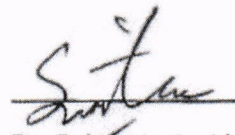
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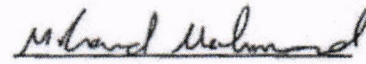
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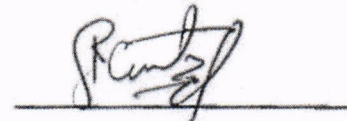
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To my family and friends who supported me throughout my degree journey.

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LIST OF ABBREVIATIONS

EICP- Enzyme-Induced Carbonate Precipitation

PR – Precipitation Ratio

EGMBE - Ethylene Glycol Monobutyl Ether

EC – Electrical Conductivity

XRD – x-ray diffraction

DI water – deionized water

UCS – Unconfined Compressive Strength

MSFR - Maximum Sand Free Rate

MICP – Microbially Induced Carbonate Precipitation

OES – Optical Emission Spectroscopy

OPC – Ordinary Portland Cement

ABSTRACT

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Sand production problem is one of the common problems confronted in upstream oil & gas industry. Formation vulnerability to sand production will limit recovery potential of hydrocarbon bearing zones and once initiated, will damage both surface & subsurface infrastructure. The approach to the problem can vary from mitigation to prevention techniques. This study focuses on one of mitigation techniques which is chemical treatment of productive zone by induced chemical reaction. Compared to other methods, EICP is environmentally friendly cementation technique which consist of biodegradable catalyzer to hydrolyze urea and yield carbonate minerals to the formation.

This work emphasizes the application of EICP to underground hydrocarbon bearing formation which are characterized by high temperature, pressure and complex chemical interactions. Initially, high-temperature behavior of EICP reaction has been investigated by means of ionic consumption studies. The work concluded that in spite of degrading catalyzer performance the reaction can still progress and yield adequate amount of precipitation in the temperature up to 90 °C. Besides, results showed that high-temperature environment can favor economic feasibility of treatment by decreasing amount of high-cost catalyzer.

The second part of study focuses on feasibility of EICP reaction to consolidate different sand samples. The addressed issue was to achieve spatial control of reaction products in the sand sample. Since vertical distribution of precipitates will be especially important for both lost-circulation mitigation and sand production, the addition of polymers has been tested. Results show that for different reactant concentration, xanthan gum can achieve at least 2 times stronger consolidation rather than base solution. The strength has been enhanced from 40 psi to 324 psi for lower and from 133 to 388 psi for higher reactant concentration. In terms of uniformity, polymer added high concentration EICP solution was successful to uniformly consolidate sand sample with average 2500 psi.

خلاصة

عنوان الرسالة: تقوية الرمال باستخدام ترسيب الكربونات الناتج عن الإنزيم

مجال التخصص: هندسة البترول

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مشكلة إنتاج الرمال هي إحدى المشاكل الشائعة التي تواجه صناعة النفط والغاز في المنبع. ستحد قابلية التكوين لإنتاج الرمال من إمكانات الاسترداد لمناطق تحمل الهيدروكربون ، وبمجرد البدء ، ستلحق الضرر بالبنية التحتية السطحية والجوفية. يمكن أن يختلف نهج المشكلة من أساليب التخفيف إلى أساليب الوقاية. تركز هذه الدراسة على إحدى تقنيات التخفيف وهي المعالجة الكيميائية للمنطقة الإنتاجية عن طريق التفاعل الكيميائي المستحث. بالمقارنة مع الطريقة الأخرى ، فإن EICP هي تقنية تدعيم صديقة للبيئة تتكون من محفز قابل للتحلل الحيوي لتحلل اليوريا وإنتاج معادن كربونات للتكوين.

يركز هذا العمل على تطبيق EICP على تكوين محمل الهيدروكربون تحت الأرض والذي يتميز بارتفاع درجة الحرارة والضغط والتفاعلات الكيميائية المعقدة. في البداية ، تم فحص سلوك درجة الحرارة العالية لتفاعل EICP عن طريق دراسات الاستهلاك الأيوني. خلص العمل إلى أنه على الرغم من تدهور أداء المحفز ، لا يزال من الممكن أن يتقدم التفاعل وينتج كمية كافية من الترسيب في درجة حرارة تصل إلى 90 درجة مئوية. إلى جانب ذلك ، أظهرت النتائج أن البيئة ذات درجة الحرارة المرتفعة يمكن أن تفضل الجدوى الاقتصادية للمعالجة عن طريق تقليل كمية المحفز عالي التكلفة.

يركز الجزء الثاني من الدراسة على جدوى تفاعل EICP لتوحيد عينات الرمل المختلفة. كانت المشكلة التي تم تناولها هي تحقيق التحكم المكاني في منتجات التفاعل في عينة الرمل. نظرًا لأن التوزيع الرأسي للرواسب سيكون مهمًا بشكل خاص لكل من التخفيف من التدوير الضائع وإنتاج الرمال ، فقد تم اختبار إضافة البوليمرات. تظهر النتائج أنه بالنسبة لتركيز المواد المتفاعلة المختلفة ، فإن صمغ الزانثان قادر على تحقيق تماسك أقوى مرتين على الأقل بدلاً من المحلول الأساسي. تم تعزيز القوة من 40 رطل / بوصة مربعة إلى 324 رطل / بوصة مربعة للخفض ومن 133 إلى 388

رطل / بوصة مربعة لتركيز المادة المتفاعلة الأعلى. من حيث التوحيد ، أضاف البوليمر محلول EICP عالي التركيز
نجح في دمج عينة الرمل بشكل موحد بمتوسط 2500 رطل لكل بوصة مربعة.

CHAPTER 1

INTRODUCTION

1.1 Background

A major issue in chemical consolidation is its environmental effects. Many chemical methods are characterized by their potential to pollute underground environment [1]. However, EICP method uses biodegradable source enzyme different than both ordinary Portland cement (OPC) and other chemical method which can be considered as green choice for application of oil and gas industry. The progress of reaction and its expected application is described in Figure 1. Once treated formation fluid contains sufficient amount of metal ions, it will shrink amount of introduced chemicals to underground environment and will minimize environmental effects.

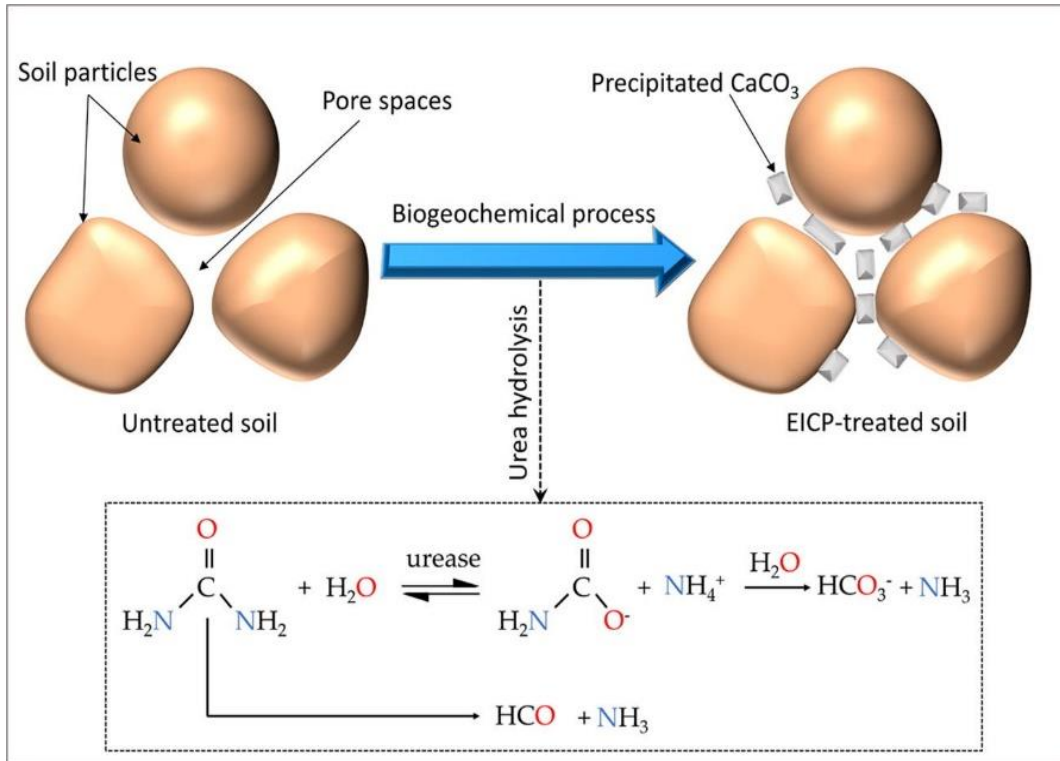


Figure 1: Sand consolidation with EICP [2]

Hydrocarbon-bearing underground environment is characterized by harsh conditions such as high temperature, pressure and complex chemical interactions. Therefore, application of EICP to upstream oil and gas operations requires detailed research on how this reaction will behave in high temperature and whether reaction products will meet the requirements.

Spatial control of reaction production is also essential depending on the purpose of operation. If EICP is injected to mitigate lost-circulation control [3] then vertical distribution of precipitation along the porous media is a factor to be considered. It is equally important for sand consolidation process where additional requirement, retention of permeability is introduced. The permeability can be retained only once the precipitation is distributed uniformly in the reservoir.

Another challenge of EICP application is to achieve controllable reaction rate which can induce precipitation in the area of interest. Especially for underground applications, this control is of great value. Oil and gas wells can achieve up to 25 0000 feet depth where delivery of chemical reactants and catalyzer can be unmanageable. Therefore, during selection of optimum condition, an emphasis should be put not only on maximum reaction yields but also a combination which can easily controlled and induced.

1.2 Suggested Solution

1.2.1 EICP Reaction Progress at High Temperature

There are wide range of literature showing how the effectiveness of EICP can degrade in high temperature environment. However, for underground sand consolidation process, the treatment of near well bore area with preflush (EGMBE) will favor the condition for the reaction [4]. Once injected cement reached cooled area of interest, it will be subjected to incremental temperatures reaching to actual temperature of reservoir. Although, there are numerous studies showing degrading enzyme performance after 70 °C, few investigated how this performance is changing up to this temperature[5]

According to study by Hengxing Wang et al. the urea hydrolysis by urease are positively changing with incremental temperature up 65 °C [6]. In other word, higher temperature favors the EICP reaction by yielding more calcium carbonate and dolomite minerals. In addition, urea hydrolysis can progress even without urease if the temperature raised to above 100 °C [7]. These increment in urease performance followed by urea hydrolysis also gives stability to treatment process. Since one of the challenges is to induce

precipitation in area of interest, an optimum concentration can be designed which can be simulated by high temperature in the unconsolidated zone.

1.2.2 Uniform Consolidation

Non-uniform distribution of precipitation for EICP is expected due to natural buoyance forces and connectivity of porous media. The effect of buoyance force will be mitigated by introduction of viscosity modifiers such biopolymers.

However, the main challenge here is determination of approximate particle size of precipitation. In this research, it will be addressed by implementing sensitivity analysis on sand particle size. Different particle sizes from fine to coarse in addition to mixed, will give indirect reflection of main factors affecting distribution of consolidation.

1.3 Problem Statement

Sand production problem is one the most common problems that upstream oil and gas industry faces. Its effect can be analyzed in terms of financial, operational and safety aspects [8]. The operational challenges which have negative impact on productivity are but not limited to accumulation of sand in different parts of downhole infrastructure starting from nearwellbore zone, tubing, pipelines and at the end on surface facilities. Onset of sand production will then erode pipelines, pumps and their moving components. In addition to their repair cost, non-operative time of producing well will put operators to financial hardships. If it is not controlled the process can proceed to erosion of subsurface safety valve which will give rise to safety concerns [7]. The last but most important aspect will environmental concern. Vast amount of hydrocarbon coated sand produced to surface can have significant effect on environment and need careful consideration to manage.

1.4 Research Objectives

1. To achieve environmentally friendly method of chemical sand consolidation
2. To achieve optimum EICP reactant and catalyzer concentration in terms of
 - a) temperature stability
 - b) efficiency
 - c) strength

CHAPTER 2

LITERATURE REVIEW

2.1 Sand Consolidation

2.1.1 Cause of Problems and Effects

There are numerous challenges and constraints that the oil industry is affected by. Sand production is one of them which is one of common problems that has continued since almost a century ago and exists all over the world. According to studies [9], 70% of oil fields are either poorly consolidated or suffer from sand production issues. Most of formations can be naturally unconsolidated and prone to sanding issues. This is especially case for geologically young rocks where lack of mineral deposition acting as cement can form unstable formation [10]. However, there are other factors that can directly lead to this problem. Primary reason is flowing fluid through porous media which exerts friction force on surface of sand. In more detail, for each geologic formation, there is threshold value of differential pressure for producing fluid to give rise to sand production [11]. Besides, water cutting further increases possibility of this issues by altering pore pressure supporting the formation and due to its sand wetting property. In contrast, resisting factors will be strength of formation, capillary action, and cohesion [1]

After onset of sand production, both surface and subsurface infrastructure will be exposed to some issues such as erosion, wearing of downhole equipment, separators and plugging of tubes. When there is no adequate response for a long time, it can even trigger subsidence

of strata and walls demolition. From operational aspect, sand production will damage recovery of hydrocarbons by decreasing permeability and increasing pressure drop adjacent to near wellbore zone. Once formation is exposed, significant time and efforts will be needed to design and intervene to ongoing production operations [12]

2.1.2 Remedies and Selection Criteria

Mitigation methods have different approach to sand consolidation. Conventional methods to deal with this problem can be categorized as mechanical, which are stand-alone screen, gravel pack, chemical methods or by controlling production parameters such as differential pressure. It is quite common in industry to define maximum sand free rate (MSFR) which is considered as safe and operationally feasible amount of produced sand to sustain production of hydrocarbons [13]. Before considering various sand control techniques, it is worth to consider which criteria is followed when decision is made on type mitigation approach. Firstly, the economic aspect of treatment will be considered. Those are total cost and benefits of operation and examination of different performance metrics to draw maximum benefit. Another main point is feasibility of treatment method which is proved in different or similar formation. Success chance will be especially high when those treatments are not only limited to lab scale which cannot mimic real conditions. Last and foremost consideration is sustainability of process. The treatment method should be durable and their interactions with subsurface formation should be properly analyzed. For example, the process can have a tendency to exert negative effect to subsurface environment.

Despite mitigation of sand production problem, mechanical methods are still suffering from some drawbacks such as their negative effect on productivity index, isolation issues,

damage during implementation and timely well intervention to ensure filtering properties of screens. Chemical consolidation, on the other hand, is not as costly and time-consuming as mechanical methods[13]. Their initial application dates back to 1940 where plastics were used to control and mitigate sand issues. In simple terms, chemicals will be injected to desired interval for the sake of increasing strength of formation and performance metrics are taken as Uniaxial Compressive Strength in most of studies. It is expected for chemical treatment to provide at least 147 psi strength for formation and thanks to introduction of polymers, when properly applied this threshold value can be easily achieved [14]. Regardless of the type of chemicals, there are constraining factor for successful implementation. Those are pore volume, formation temperature, permeability, curing time and treatment rate.

2.1.3 Constraints of Chemical Sand Consolidation Methods

The degree of success for chemical sand consolidation is not only a function of compressive strength but striking a balanced approach among numerous factors. The primary issue with practices is maintaining permeability of formation. It is quiet desirable when formation doesn't suffer from wellbore instability, sand production problem and at the same time has ability to produce hydrocarbons with incremental pressure difference. To the question of whether there is chemical consolidation method capable of increasing strength without any modification to permeability, a study [10] concluded that almost all conventional methods (resins) degrade the permeability to different extend. When analyzing treatment methods based on percentage change of permeability, care should be taken to consider initial permeability of formation. For low permeability reservoirs, even

1 mD drop can reflect huge percentage values but those can be negligible for very permeable formation. Hence, different authors offer threshold value in order for formation to be applicable to chemical treatment. It is 10 mD for gas and 50 mD for oil bearing reservoirs. Another limiting factor is thickness of formation. Since the range of interval also depends on injected chemical's properties, different authors have different suggestions. Cholet et al [15] offered 10 m while Carlson and Kuncoro [16] limit this value to 3 to 4 m. In addition to thickness of treated zone, care also should be taken to decide both interval and density of perforations. Numerous field experiences reflect 10-15 ft [17] perforation interval as threshold and advice to limit length of perforated interval and number of perforations. For formation to consolidate properly, there should be adequate pore volume for cement to be injected and stored. As surface area between grains and cement slurry is increased, efficiency of cementing process will be improved. Particle size distribution reflects non-direct estimation of contact area and it is desirable for geologic formation to have less particle size. However, this concluding remark can create compromise when strata has high clay content which is in most cases characterized by low particle size. Swelling phenomena is studied by numerous authors and it is clearly observed that mineralogy of clay especially montmorillonite has tendency to swell and plug permeable medium when in contact with aqueous solution [18]. In more detail, each clay minerals have cation exchangeable capacity to hydrate when it confronts with different polarity solution. Other minerals such as kaolinite and illite has propensity to cause fine migration problems. Allen et al and Carlson et al. concluded that formation possessing 10% fines or clay will significantly deteriorate sand consolidation efficiency. This instability phenomena are mitigated by introduction of surfactants such as 1,3-diaminopropane

dihydrochloride ethylenediaminedihydrochloride and hexadecyltrimethylammomium bromide [19]. Numerically, each surfactant leads approximately 50 % reduction in clay swelling. Some studies only emphasize on montmorillonite with an addition of KCl, NaCl and polyethylene glycol coated silica nano-particles [18]. Their effectiveness is validated by different concentration of NaCl and KCl and synergetic effect was observed with an addition of nanoparticles. Those additives are also offered to be applied to sand consolidation practices. Another essential factor to consider is temperature. Phase transformation from liquid to solid phase and flow behavior of injected fluid are both function of temperature [20]. By several studies, it was observed how viscosity of fluid can change with respect to surrounding temperature. Regarding curing time of injected slurry which is in most cases, polymers, there is a process called crosslinking. It is a transformation from liquid state to three-dimensional network. Initially, injected phase only carriers' small monomers and by the time it is converted into more and larger clusters. The end point for this progress is generation of big network of clusters (gel point) and its viscosity is defined by Stoke's law. Characterized by infinite viscosity, the point is sensitive to different operational parameters. Generally, applicable range of temperature for chemical sand consolidation is between 100 and 225 °F [21]. However, thanks to different catalyst enhancement methods, this range can be extended depending on temperature of reservoir. Wasnik et al. [22] offered different catalysts to curb this limitation such as AlCl_3 , $\text{Al}(\text{SO}_4)_3$, NiCl_2 , ZrOCl_2 , NH_4Cl , $\text{Cr}(\text{CH}_3\text{COO})_3$ and o-Phosphoric acid as best candidates. However, their field scale application is not tested yet.

2.1.4 Chemical Sand Consolidation Methods

In this section, different chemical methods other than EICP will be discussed, and lessons learned from each will be analyzed in detail. Main metrics for comparison of different chemicals is the compressive strength of treated formation, percentage change of preserved permeability and finally range of temperatures that the method is applicable. Besides, the section will include field scale practices and procedures.

Formaldehyde Resin is a highly reactive gas and lead to polymerization with phenol, urea and melamine. Condensation of this gaseous phase which is followed by water and other by products evaporation will consolidate the treated interval. They are namely phenol-formaldehyde resin, urea-formaldehyde resin, melamine-formaldehyde resin. The application of phenol-formaldehyde dates back to 5 centuries and is characterized as infusible and insoluble polymer network. Other properties are flash point and boiling point which are 72.5 and 181.8 °C respectively. Formaldehyde Resins are distinguished by thermal stability up to 149 °C.

Regarding application procedure, Spain et al. utilized three-stage treatment procedure. Firstly, formation is flushed with agent to improve sand grain adhesion. Next step comprises injection of polymer solution which is followed by diesel injection to clean the near wellbore zone. This study achieved compressive strength of 1000 psi with 50% reduction of permeability while Talahjat et al. [23] managed to achieve 3 times more in Ahwaz and Mansoori oil field. His chemical content was a modified phenol-formaldehyde resin with post-treatment permeability of 1500-3000 mD. Mishra and Ojha [24] further improved permeability retention up to 64.65% but with comparatively lower strength of 1300 psi. The polymer was Urea-formaldehyde with an addition of curing agents such

potassium silicate and ammonium chloride. In spite of lower strength, authors highlighted low cost and availability of inorganic polymers.

As the summary, this type of polymers has potential of yielding high tensile strength, thermal stability and disadvantaged by high percentage permeability loss and difficulty with application.

Furan Resin is heterocyclic organic compound comprising a five membered aromatic ring. The raw materials to obtain is furfuryl alcohol and furfural. It possesses a lower flash point at around 25 and 65°C, viscosity of 100-300 cP and boiling temperature of 170°C.

In of the study by Young et al, furan resin was used with combination other performance enhancers. Those were silane bonding agent to improve adherence to sand particles, furfuryl alcohol to decrease viscosity, stabilize clay particles and increase efficiency in high temperature and finally combination of surfactant and water to prevent existence of emulsions. Similar to other methods, diesel was used as spacer solution. Youngs studies include experimental investigation of this resin where he reached 3000 psi strength at temperatures of 15.5 to 149 °C. However, the treatment was disadvantaged by high viscosity and low flash point of polymer. His field scale application (40) comprises modified versions of treatment chemicals such as Urea formaldehyde (UF-85) concentrate, furfuryl alcohol, urea, gamma-aminopropyltriethoxysilane with compressive strength of 1072 psi. In his next studies [25], he offered less viscous form which is mixing of tetrahydrofurfuryl methacrylate and a furan resin. Further modification of resin content was realized by Fader et al. where oligomer furan resin with organic diluent, ester and a catalyst [26]. Preflush slush for this treatment included ester and sulphuric acid to remove oily

residues. Negative environmental effects, limited temperature application and longer time for injection was negative side of this polymer. However, with some modification to content which is chemical system comprises of furfuryl alcohol with ethylacetate, methanol and sulfuric acid, Fader et al managed to 550 psi strength with permeability reduction of 10% at elevated temperatures of up to 160 °C. In spite of lab scale application, Parler et al. [27] managed to find combination which can improve strength to 2000 psi and 80-85 % permeability retention. His cement solution comprises oil-wetting agents, non-emulsifiers, coupling agents and even applicable to the formation of higher 68% clay content. Interestingly, Todd et al. [28] validated possibility of reaching even to 3000 psi with coupling agent of aminosilane, surface active agent of ethoxylated nonyl phenol phosphate ester, diluent of methanol or butyl alcohol and viscous fluid carrier breaker of peroxides/ester.

Nguyen et al. provided comprehensive procedure of chemical sand consolidation. The procedure included 3 stages. Initially, the interest zone was perforated and cement slurry containing furfuryl alcohol resin, furfuryl alcohol, a silane coupling agent was injected. This was followed by injection fracture fluid which contained proppant particles coated with resin composition. In his next studies, he modified the composition with surfactants to improve contact with proppants and ester to break gelled fracturing fluid films. Temperature applicability was between 27 to 149 °C with compressive strength of 80 to 1642 psi.

Epoxy Resins are reactive polymers with epoxide group and differentiated with resistance to heat and chemical exposure. In spite of its positive aspects such as high boiling and flash point of 150 and 164 °C, it is toxic to aqueous environment and human eyes/skin.

Penberty et al [29] achieved 2000 psi compressive strength overcoming the problem of wetness in formation where water wetness of particles resists adequate resin displacement. Chemical composition of ethylene glycol isopropylether solvent and isopropyl alcohol (IPA) with ethoxylatedhexyl alcohol solvent was used as preflush of treatment. Dee et al [30] even reported 5000 psi and 67 %permeability retention with diverting agent which has explosiveness to push the solvent into perforated zone. Dee et al [31] showed further improvement potential using gas generator to achieve more pressurized injection and managed to reach 7000 psi strength between 10 to 121 °C. In spite of curing time, longer periods of consolidation eases injection procedure to the formation. Dewprashad et al [32] experimentally offered new HT epoxy resin with this positive aspect. Marfo et al. [21] investigated field scale injection of both internally and externally catalyzed resin. In spite of low achieved strength of 57 and 235 psi for without and with 1% bentonite clay, they observed positive effect of increased surface area. Riyanto et al. [33] applied different pre and post flush composition of KCl and aqueous surface modification agent in Sarawak, East Malaysia. Their studies reflect good retention of permeability from 73 % to 100 % in 63°C. Other authors such as Hadi et al [34] and Palencia et al. achieved 614–1816 and 217.21–694 psi strength with comparatively lower temperature.

Amino-Aldehyde polymer is acquired from polymerization of amino and aldehyde. Amino Aldehyde is easily accessible, low-cost material and characterized by easy transformation at both low and high temperature. In the experimental study by Larsen et al.[35] it was used with urea/Ca²⁺ in sand pack. Sample was consolidated up to 1531 psi with 25 % permeability reduction. However, the temperature was between 25-65C. Lahalih and Ghloum [14]use these polymers with inorganic and organic crosslinkers and used KCl

brine to implement experimental work. The strength was improved to 1422 psi with elevated temperatures of 60, 80 and 120 °C.

Silicon Polymer composition basically contains silica and oxygen but can exist in very wide range of structures. To treat the formation, they will be injected together with alcohol and water. Water helps polymers to hydrolyze, and it is desirable to have strong acid water with rich protons. Due to low surface tension with water, alcohol will help to sweep the residual formation from water. However, high acidity of solution makes it practically unsafe treatment. Shue et al [36], on the other hand, directly injected aqueous solution of ammonium silicate which is followed by two slurry, immiscible hydrocarbonaceous phase and water miscible phase. High retention of permeability is observed without specific reported value. In other studies, ammonium is displaced by sodium silicate and experimental values of 0-396 psi at temperature range of 20 and 60 °C is achieved. Cobianco et al. [37] test field scale application with the following treatment procedure. Operation was started by preflush of KCl to improve contact points and postflush of diluter and organic acid to stabilize amorphous silica network. At final stage, nitrogen is injected to dry treated interval and remove extra silicate solution. Experimental studies show 70-80% permeability retention and 287-427 psi strength with the following procedure.

Shape memory polymers are intelligent materials capable of returning their original shape which can be controlled by temperature. Positive aspect of these polymers is that they have permeable medium 60-160 μm and can allow passage of less than 43 microns sand particles. They are applied for the sand formation having 5-600 mD with temperature range of 37-93 °C.

Polyacrylamide Polymer is characterized by environmentally friendliness, water solubility, swelling properties, low viscosity, and favorable high flash point. Numerous studies have been realized using Polyacrylamide Polymer such as its combined utilization with aldehyde/phenol (106) and used to wellbore total shut-off treatments with composition of acrylamide-polymer/chromium (III)- carboxylate. One of significant improvement was achieved by Salehi et al. where their team managed to increased strength 30 times. Crosslinker such as chromium triacetate are added to hydrolyzed acrylamido propyl sulfonated acid to improve the performance.[38]

Consolidation of sand can be achieved directly from reaction of crude of oil with oxygen which will yield aldehydes and alcohols. [39]. In the study by Jennings et al. [40] consolidation was achieved heating penetrated pay zone. The reaction can be achieved in-situ combustion and or combustion-supporting oxygenated foam which can be drawn from diesel. 3000 psi strength and 70% permeability retention were achieved by this type of ignition. To achieve more combustion, either oxygen concentration can be increased or can be initiated by ignitor such electrical ignitor ([33]) and gas fired burner. In spite of lower strength of 375-1264 psi, Agguor and Osman achieved low temperature oxidation with permeability loss of 22 %. Nevertheless, Aggour [41] improved these results by using asphalt-reformate solution, heavy oil residue and aged samples by improving up to 1800-2300 psi with 95.5 permeability retention.

PEA will be used as an additive to decrease viscosity of cement slurry and can prevent permeability plugging by its liquid-phase presence. Kalgaonkar [42] have conducted experimental studies where injected different type of resin in combination with PEA. His studies offered 1:1:0.8 ratio of combination with 4-6 times more compressive strength. In

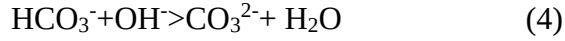
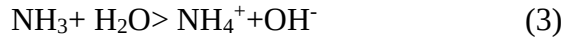
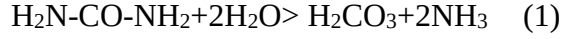
addition, this study highlighted other advantages such as controllable curing time, high permeability retention and easier mixing methodology.

2.2 Enzyme Induced Carbonate Precipitation (EICP)

2.2.1 Introduction

In upstream oil and gas operations, Ordinary Portland Cement has been used extensively from sand consolidation to different segments of drilling operations. However, massive production of it causes environmental concerns such as emission of big volumes of CO₂ to the atmosphere. Therefore, interest to more environmentally friendly methods has risen significantly. One of the alternatives are biomediated methods which are Enzyme Induced Carbonate Precipitation and Microbially-Induced Carbonate Precipitation. Both methods use biodegradable catalyzer to stimulate urea hydrolysis to produce carbonate ions. In the presence of metal ions such as Ca²⁺ and Mg²⁺, carbonate ions will give rise to different types of carbonate minerals. Since MICP is more sensitive to environmental condition and technically difficult to deliver, EICP is more suitable and feasible for different applications.[9][10]

The sequence of EICP reaction can be summarized as follows. Initially, catalyzed urea hydrolysis will yield carbamate and ammonia. Both products are reversible and can be shifted to carbonic acid, bicarbonate, hydrogen, ammonium and hydroxide ions. Hydroxide ions will react with carbamate to produce carbonate ions and will keep the pH of solution high. [43]



In the presence of sufficient amount Ca ions, the system will yield calcium carbonate precipitation.



Cementation process will happen when the solutions of urea and metal ions will be injected to pore space of sand before reaction progress. The products, mostly CaCO_3 and MgCaCO_3 , will improve contact area between sand particles and enhance geomechanical properties

2.2.2 EICP Reaction Performance Metrics and Optimization

Main aim of EICP is to achieve to maximum precipitation that at the end it leads to improved geomechanical properties of rock. The performance of this technique can be evaluated by different factors such as UCS, permeability reduction of treated rock and PR (precipitation ratio), pH, electrical conductivity of cement solution.

There are different approaches and metrics to optimize and improve performance of EICP for sand consolidation purposes. The most common ones used in the literature is Precipitation Ratio (PR), Unconfined Compressive Strength, pH and electric conductivity. A mathematical expression of PR ca be found in Equation 9 and 10 [3].

$$PR = \frac{m_p}{m_t} \quad (9)$$

where m_p -mass of precipitated material, m_t -theoretical mass

$$m_t = C \times V \times M \quad (10)$$

C-concentration of solution (mol/L), V-volume of solution,

M-molar mass of precipitant

USC – one of the common techniques to measure rock mechanical properties. It is derived from stress/strain relationship of exposed sample to uniaxial load.

pH - is non-direct reflection of OH^- concentration with respect to H^+ . It is commonly used by different authors to monitor how urea is hydrolyzed and its conversion to precipitates. As it can be seen from Figure 2, continuous hydrolysis of urea lead to sharp increase of pH value and followed by instant drop. This is the reflection of reaction equilibrium which give rise to carbonate ion generation.

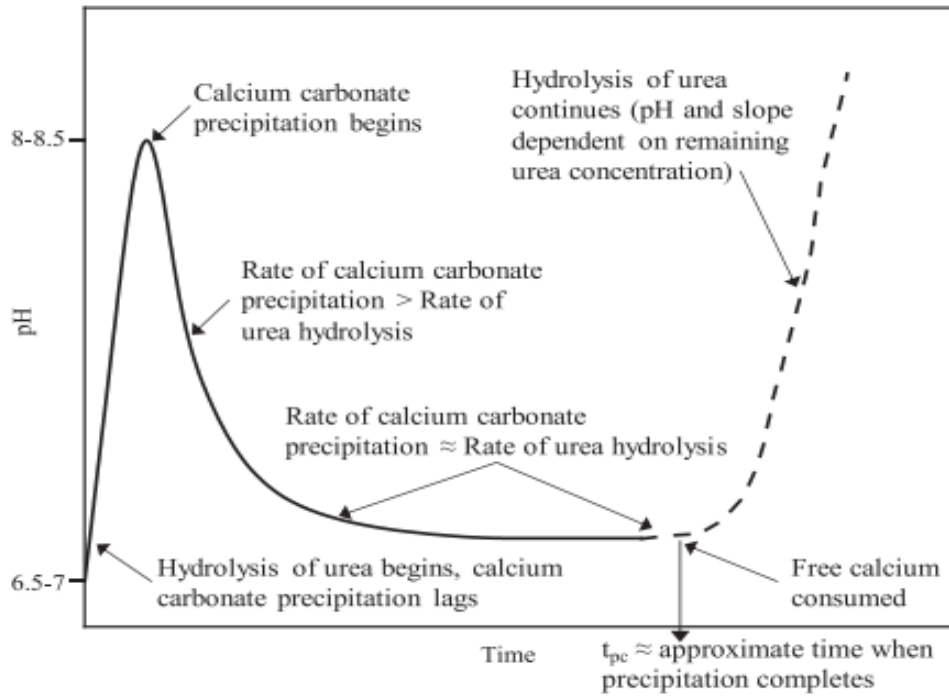


Figure 2: pH dynamics of EICP reaction [44]

Electrical Conductivity - can numerically shows overall ionic concentration of solution. Since precipitation process consumes ions in the solution, conductivity will be sensitive to reaction progress. [45] [46][47] Almajed et al. used this metric as in Figure 3 to clearly define best concentration for precipitation efficiency.

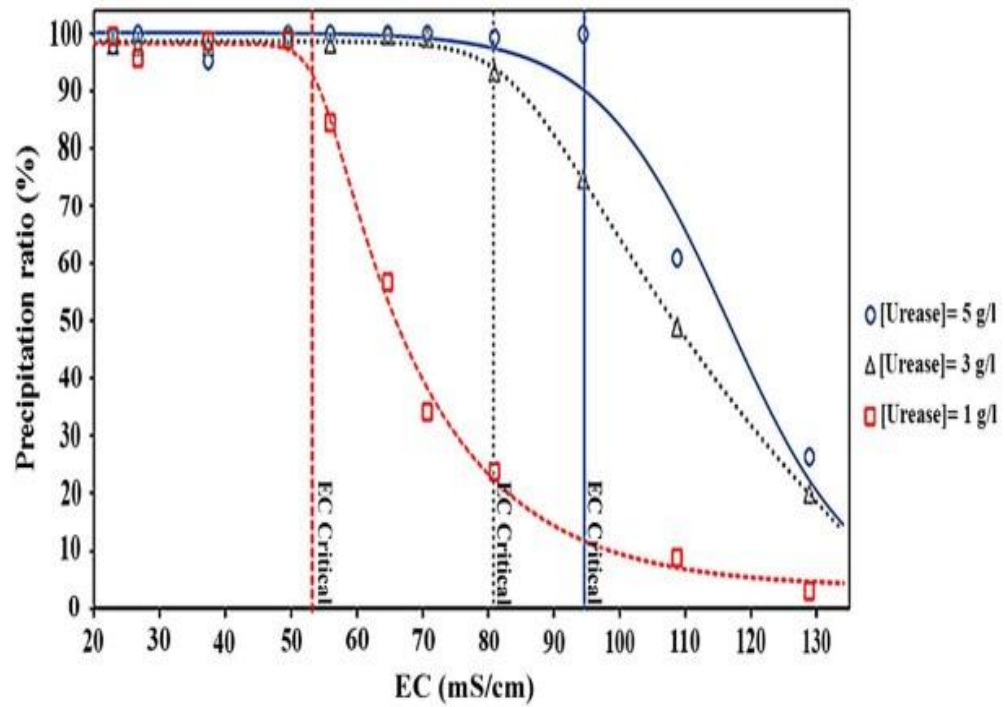


Figure 3: PR vs EC relationship [48]

Most studies in literature follows similar procedure to optimize EICP reaction. Extensive test-tube experiments have been performed by many authors to find the best combination. Almajed et al. and Yasuara et al. shows that high precipitation amount can be improved by enzyme concentration until 1 M/L cement concentration [49] [46][50]. Threshold value can even be lower if the activity of enzyme is lower (4 kU/L) [51]

The effect of temperature is also a worthy factor to investigate. Nemati et al. observed stimulated production of calcium carbonate from 0.038 to 0.34 g/L/h by incremental temperature [52]. In case of even higher temperature, precipitation process can happen even without catalyzer [7]. However, it has been experimentally shown that some temperatures limits exist for different types of catalyzers. Among different source enzymes, JBM outperforms other enzymes and its performance worsened after 70 C temperatures

[5]. On the other hand, Neupana et al. showed that temperature increment can only increase reaction rate but not the final reaction products [53]

The necessity of pH value can be explained by different reasons. Firstly, calcium carbonate dissolves in pH value lesser than 7 while higher values can degrade enzyme activity. [54][55]

2.2.3 Sand Consolidation Using EICP

The effectiveness of EICP is not only a function of EICP reaction but also its behavior in the pore space of loose or partly consolidated sand media. By optimizing reaction, massive amount of precipitates can be achieved but if the reaction is not happening in contact points of grains, the effectiveness can be low. There are different methods of sand treatment using EICP cement surface pluviation, injection and Mix and Compact which is illustrated in Figure 4. Therefore, there will be a different correlation between precipitated amount and degree of consolidation.

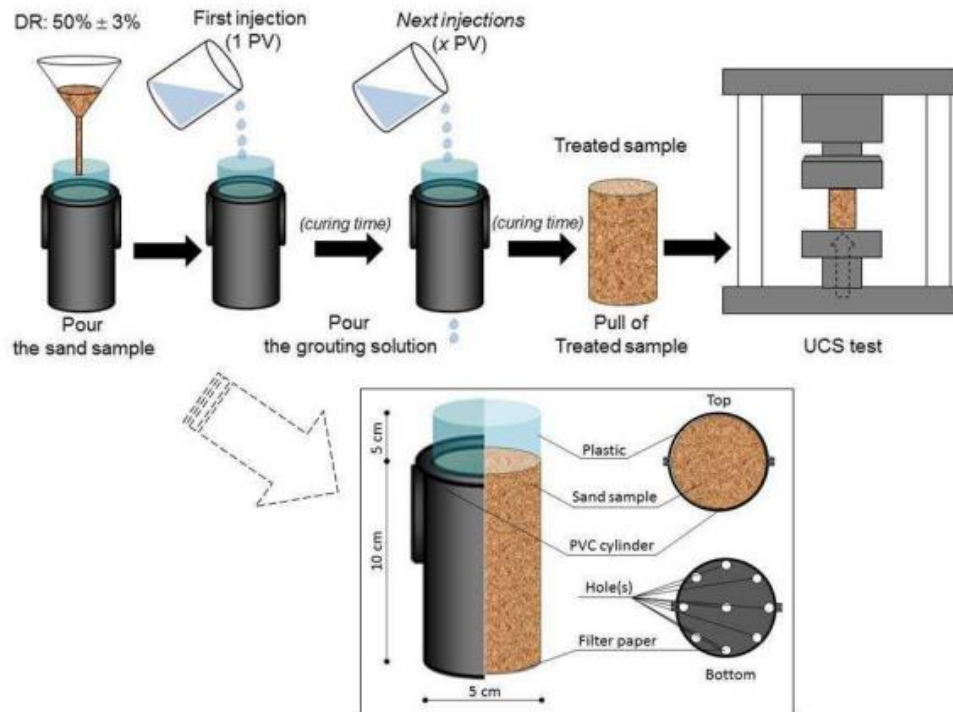


Figure 4: EICP treatment procedure[56]

a. Carbonate Precipitation and Unconfined Compressive Strength (UCS)

In the literature, there are numerous ways of treating sand to EICP solutions. Some authors recommend achieving initial compression of sample before sample injection. The performance can be further upgraded by injecting multiple pore volumes. Almajed et al. reported 10 times higher UCS from 4 treatment cycle for Ottawa 20-30 sand. with their solution of 1M Urea and 0.67 CaCl_2 [49]. Ahmed Miftah et al. validated multiple injection effectiveness also with seawater presence where strength increased up to 0.263 and 0.224 MPa [45]. Another challenge in evaluating consolidation process is to measure precipitation amount in porous media. It has been achieved by acid leaching methods but the exact correlation between precipitation and strength will be sensitive to distribution of

reaction products among the sample. A study by Almahed et al. showed that the strength can vary for different amount of precipitation if some stabilizers such as non-fat milk is added. They achieved strength value of 0.5 MPa which requires multiple treatment to achieve [57]. It is interesting that selecting appropriate curing time has a significant effect on UCS. Another factor affecting consolidation process is investigated by Kee-Lim et al. They showed that moisture content of environment can deteriorate the consolidated sample. [58]. The presence of fine particles, roundness and organic matter is also one them which can negatively affect EICP performance. [59]

b. Effect of Additives

Considering extensive studies which show positive effect of additives, it is worthy to investigate which additives has been used and how effective they for consolidation process. Other than non-fat milk, use of unpurified enzymes can bear similar content with milk which are rich with polymers. Polymers not only stabilize enzymes but are also used to improve nucleation points [66]. Since precipitation in treated samples are prone to accumulate on the bottom of sample, addition of some polymers can enhance retention abilities and stability of treatment solution. Comparing to glycerol, xanthan gum illustrates promising results [60]. Nasser Hamdan validated this fact by addition of vapor pressure measurement of modified solution [61]. He also studied how addition of polymers can add water retention abilities. This is reflected in Figure 5, where vaporization of water content in EICP solution significantly reduced thanks to addition of Guar polymer without effecting precipitation efficiency.

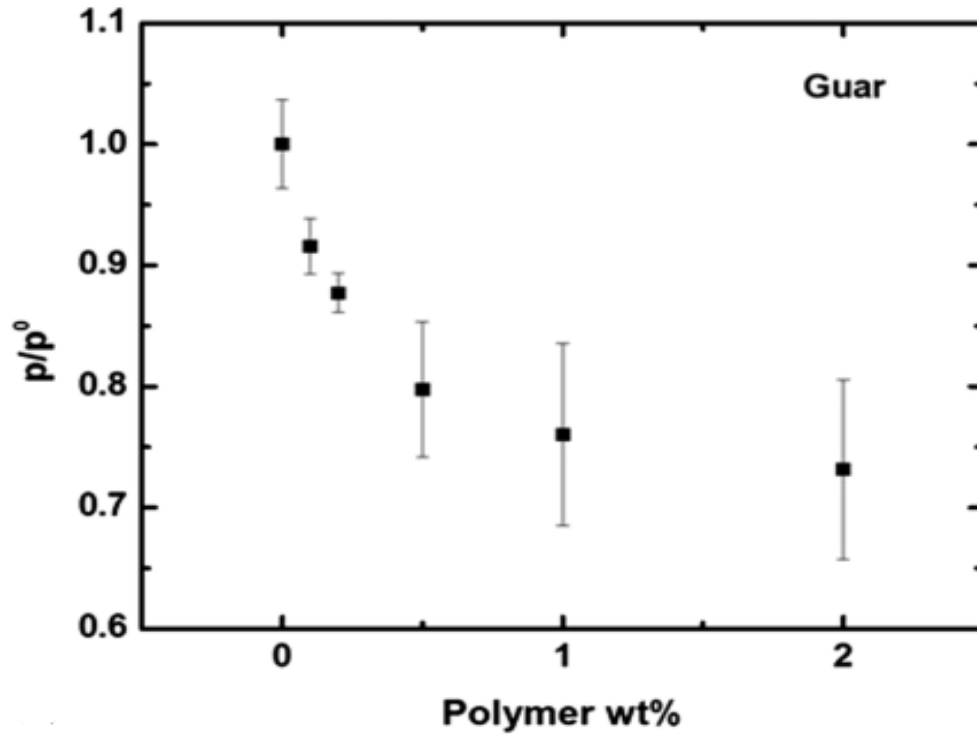


Figure 5: Vapor pressure of different polymers [62]

Another use of polymers is to upgrade thermal stability of solutions. Xanthan gum was also used to increase thermal stability of cement solution. Study by Tariq et al. showed only 10% weight loss of modified EICP solution which is exposed to 300 C temperature [3]. Addition of other types of polymers is also investigated and they have positive effect on strength of sample. Thanks to their crosslinking abilities both Sodium Alginate, up to 1711 kPa UCS, and Na-Mt has induced strength improvement [3] [63]

Another method of improving efficiency is to modify reactions inputs. Putra el., in their studies, shows that replacing CaCl_2 partially with MgCl_2 increases resultant strength of treated samples. There is also possibility of MgSO_4 instead MgCl_2 . 555 kPa has been achieved by substitution of 0.04 CaCl_2 in an overall 1 M cement solution [64]. In his next

study, more extensive analysis shows that this modification makes morphology of aragonite rather than calcite and 2.5 times increment has been achieved [56]. Crystal size during calcium carbonate precipitation is sensitive to the presence of divalent, trivalent ions and molar ratio of reactants. Addition of Mg^{2+} in the ratio of 1: 1000 with Ca^{2+} can yield 2 times larger crystal size. Similar effect can be achieved using deficit amount of $CaCl_2$ in EICP solution [65]. The introduction of Ca^{2+} in different salts can also affect the final yield of reaction. Due to its higher solubility, $CaCl_2$ is selected as best candidate for EICP reactions [66].

Since most of studies have used similar methodology of sand treatment, the questions arise on the efficiency of EICP for partially consolidated samples. There, performance metric will not be strength but permeability sample. Zeeshan et al. found that 0.9M $CaCl_2$, 0.1 $MgCl_2$ and 3 g/L urease with an addition of xanthan gum can plug permeability to 99 % [3].

CHAPTER 3

METHODOLOGY

3.1 Temperature and Concentration Sensitivity Analysis of EICP Reaction

3.1.1 Materials

a. Reactants and Catalyzer

Jack Urease Bean has been used as catalyzer in these reactions. Technically, their presence lower down activation energy between reactants and transition state. They exist most commonly in either microorganisms or higher odder plants. The chemical is product of SIGMA-ALDRICH with specification Type III, 25920 units/g solids and U1500-20KU.

The sources of carbonate ions are from urea, which is characterized by high solubility in water, colorless, odorless and neither acidic or basic. Its chemical structure consists of 2 amino groups and carbonyl functional group. Urea hydrolysis catalyzed by enzymes can be explained by multiple steps. Initially, urea's carbonyl oxygen implements nucleophilic attack on nickel atom of urease. It is followed by the release of NH_3 and carbamate group sequentially.

Source of metal ions are derived from $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ which have 99% purity and the product of SIGMA-ALDRICH and VWR Chemicals as described in Figure 6.



Figure 6: EICP reactants and catalyzer

b. Coupled Plasma Optical Emission Spectroscopy

Optima 8000 ICP-OES (Figure 7) has been utilized for the purpose of detecting ionic concentration of solutions. By means of high energy plasma, injected sample solutions are exposed to high energy sources. Since each element atom has unique form of energy level transition and a way of bouncing back to their ground level for their electron, emitted lights from the solution can be used to detect ions and their concentration.

The equipment is capable of measuring up to 150 ppm solutions so that each sample has been diluted 1000 times before measurement.



Figure 7: Optima 8000 ICP-OES

c. Malvern Panalytical (XRD)

XRD is a nondestructive technique to derive mineralogy without chemical analysis. Injected X-rays will interact with mineral surfaces, and they will diffract in different manner based on crystal structure of each mineral. By analyzing diffraction patterns, it gives abundance of different mineralogy. Based on these principles, the assessment has been done using Malvern Panalytical in Figure 8.



Figure 8:Malvern Panalytical

3.1.2 Procedure

To perform ICP analysis, different solutions of Ca, Mg and Urea have been prepared. The concentration of reactants is increasing from M4 to M2 (Table 1). The preparation of solution follows the steps of:

1. Mix different concentration of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and Urea using mixer for at least one hour
2. Filter the solution
3. Cure the solutions for at least one day

Table 1: EICP reactant concentration

	Ca ²⁺	Mg ²⁺	Urea
	ppm	Ppm	ppm
M2	57,087	26,655	180,205
M3	53,000	23,085	150,171
M4	49,457	19,885	120,137

The second step is to prepare enzyme solution and preparation of final EICP solutions as seen in Table 2.

1. Prepare 1 g/L solution from jack urease bean
2. Prepare final EICP solutions which contain different volume ratios from 1 to 7 but with overall constant volume of 40 ml.
3. Mix them thoroughly

Table 2: Volume ratio and ionic concentration of EICP solutions

	Volumes		Ca ²⁺	Mg ²⁺
	Reactants	Catalyzer		
	ml	ml	ppm	ppm
M2V1	20	20	27,446	13,327
M2V2	30	10	41,169	19,991
M2V3	35	5	54,892	23,323
M3V1	20	20	26,500	11,542
M3V2	30	10	39,750	17,314
M3V3	35	5	46,375	20,199
M4V1	20	20	24,728	9,942
M4V2	30	10	37,092	14,914
M4V3	35	5	43,275	17,399

To perform temperature sensitivity analysis, the following steps has been followed.

1. Cure the solutions for 24 h at each temperature from 25 °C to 90 °C with an increment of 10 °C
2. Take 0.1 ml sample solution at each step
3. Dilute the collected sample 1000 times using DI water
4. Weight and collect precipitation for mineralogical analysis

3.2 Sand Consolidation using EICP

3.2.1 Materials

a. Sand Samples

The sand sample used in this study has been characterized by mineralogy and sand particle size distribution. Initially raw sand has been washed and fine particles has been removed from sample. Then, the following sand samples described in Table 3 have been prepared for analysis.

Table 3: Particle size of filtered sands

	Fine	Medium	Coarse	Mix
	μm	μm	μm	μm
Particle size	$125 < r < 250$	$250 < r < 425$	$425 < r$	$31 < r < 253$

According to mineralogical analysis in Figure 9, the main constitutes are quartz and feldspar with 64.8 % and 8.5 % respectively. There is a negligible amount of clay particles so that it can be characterized as sandstone.

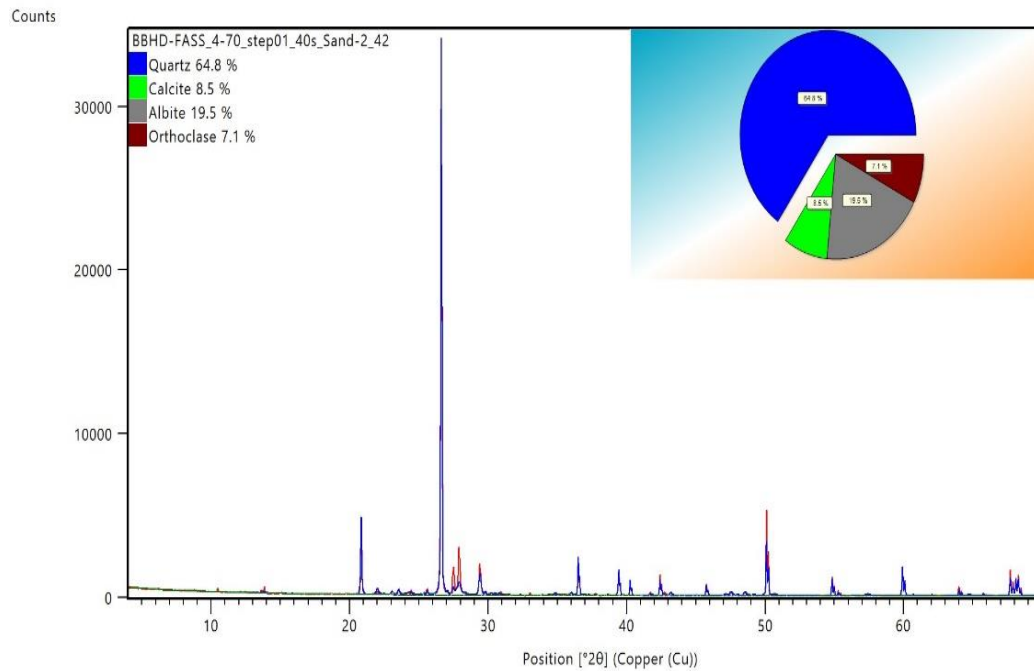


Figure 9: Mineralogy of sand sample

b. WOMBAT Scratcher

It is considered as quasi nondestructive test and sensitive to both tangential and normal components of force during scratching. During measurement, relative velocity, depth of cut and cutting angle are adjustable and can be changed based on application.

Derivation of strength parameter, UCS, are non-direct where the correlation between UCS and intrinsic specific energy will be used. The correlation is stronger for the rock performing ductile behavior. In addition to non-destructiveness, it offers advantages of minimal sample preparation and log of spatial distribution of strength. Figure 10 illustrates the ongoing Scratch Test on of sample studied in this research.

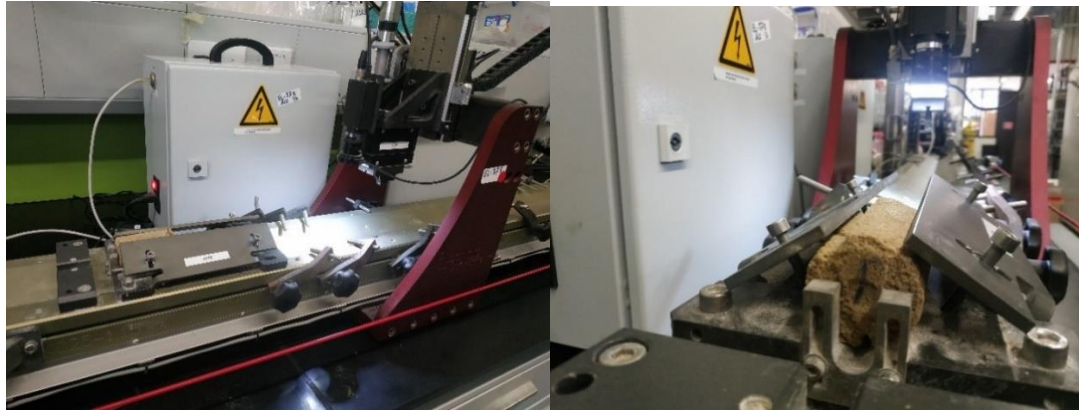


Figure 10: WOMBAT scratcher

c. Retsch Vibratory Sieve Shaker, Mixer and Oven

Sand is filtered using Retsch Vibratory Sieve Shaker. For each treatment, 10 minutes is given for vibrating and reaching desired sand particle size. During the treatment of sand, mixer used to achieve efficiency contact of EICP solution and sand. All treated samples are cured at 90 °C. Equipments used for sand processing can be found in Figure 11.



Figure 11: a) oven b) Retsch Vibratory Sieve c) Mixer

3.2.2 Consolidation Enhancement with Biopolymers

According to numerous literature resources, sand consolidation using EICP is sensitive to many factors. Firstly, it has been monitored how concentration is affecting the

consolidation process. This study has been followed by the addition of biopolymers and different methodologies.

1. Take 40 ml of different concentration of EICP
2. Treat 150 g sand sample (Mixed) with EICP/modified EICP solution using mixer for 10 minutes
3. Cure the solutions for at least 3 days at room temperature and 7 days at 90 °C

The concentration of tested EICP solutions has been summarized in Table 4.

Table 4: EICP concentration for polymer studies

	CaCl ₂ *2H ₂ O	MgCl ₂ *2H ₂ O	Urea	Urease	Xanthan gum	Sand type
Units	M/L	M/L	M/L	g/L	g/L	Mixed
M	0.5	0.25	0.25	3	0	Mixed
N	1	0.5	0.5	3	0	Mixed
O	1.5	0.75	0.75	3	0	Mixed
J	0.5	0.25	0.25	3	3	Mixed
K	1	0.5	0.5	3	3	Mixed
L	1.5	0.75	0.75	3	3	Mixed

In the second part of study, different methodologies have been used to achieve more effective consolidation.

1. Take xanthan gum with weight of 3 % of sand and mix/shuffle with sand
2. Treat the sand with different EICP solutions
3. Cure the solutions for at least 3 days at room temperature and 7 days at 90 °C

Concentration has been summarized in Table 5.

Table 5: EICP concentration for polymer studies

	CaCl ₂ *2H ₂ O	MgCl ₂ *2H ₂ O	Urea	Urease	Xanthan gum	Sand type
Units	M/L	M/L	M/L	g/L	g	Mixed
P	0.5	0.25	0.25	3	3	Mixed
Q	1	0.5	0.5	3	3	Mixed
R	1.5	0.75	0.75	3	3	Mixed

3.2.3 Effect of Sand Particle Size

To implement sand particle size studies, there type of sand has been treated with 3 solutions. Treatment methodology is similar to the procedure in the Section 3.2.2. The difference is concentration of EICP solutions, sand and curing temperatures (Table 6)

Table 6: EICP concentration for sand particle size studies

	Volumes		Ca ²⁺	Mg ²⁺
	Reactants	Catalyzer		
	ml	ml	ppm	ppm
M2V1	20	20	27,446	13,327
M3V1	20	20	26,500	11,542
M4V1	20	20	24,728	9,942

Treated sands has particle size range described in Table 7. Approximate diameter of sand particle has been named “d”.

	Fine	Medium	Coarse
	μm	μm	μm
Particle size	125<d<250	250<d<425	425<d

Table 7: Treated sand particle sizes

All samples can be characterized with Table 8.

Table 8: Sample identification

	Volumes		Ca ²⁺	Mg ²⁺	Sand
	Reactants	Catalyzer			
	ml	ml	ppm	ppm	
M2V1F	20	20	27,446	13,327	Fine
M2V1M					Medium
M2V1C					Coarse
M3V1F	20	20	26,500	11,542	Fine
M3V1M					Medium
M3V1C					Coarse
M4V1F	20	20	24,728	9,942	Fine
M4V1M					Medium
M4V1C					Coarse

CHAPTER 4

RESULTS AND CONCLUSION

4.1 Temperature Sensitivity Analysis

Initial concentrations of solutions can be seen in Table 9 below. The experiment methodology is designed in a way that allows determination of optimum concentration reactants. Ca and Mg ionic concentration will be measured with an assumption that consumptions of metal ions are reflection of precipitation process. Initial metal ion concentration can be found in Table 9.

Table 9: EICP ionic concentration for temperature studies

	Volumes		Ca	Mg
	Reactants	Catalyzer		
	ml	ml	ppm	ppm
M2V1	20	20	27,446	13,327
M2V2	30	10	41,169	19,991
M2V3	35	5	54,892	23,323
M3V1	20	20	26,500	11,542
M3V2	30	10	39,750	17,314
M3V3	35	5	46,375	20,199
M4V1	20	20	24,728	9,942
M4V2	30	10	37,092	14,914
M4V3	35	5	43,275	17,399

From the progress of ionic concentration of Figure 12 and 13, it can be observed all solutions show decreasing trend of Ca ions except the solutions which contain lower amount of catalyzer (jack urease bean). Regarding Mg consumption, these metal ions are less reactive than Ca ions. According to other studies, the presence of Mg ions should contribute significantly to precipitation process.

Figure 12 and 13 illustrate information about elemental concentration of EICP solutions Table 9 with respect to temperature. It is clear from the both figure that each solution from M2V1 to M4V3 shows a different pattern of downward trend. The solutions can be categorized from high concentration M2 combinations to low concentration M4 concentration. Each solution contains the same overall volume but with shrinking catalyzer volume from V1 (20 ml) to V3 (5ml). Starting from high concentration combination, they showed most pronounced sensitiveness to the temperature while least concentrated M4 combinations are less vulnerable. In more detail, for example, M4V3 which contains most catalyzer with respect to reactants has achieved its chemical equilibria at 40 °C without a need of further time and heat flow. On the other hand, other solutions which contain at most 25 % catalyzer (V2, V3) needs high temperature environment and exposure time for their reaction to proceed. Another interesting behavior is from M3V3 which contains only 12.5 % catalyzer. It is only 60 °C temperature that can stimulate urea hydrolysis and corresponding carbonate precipitation. The fluctuation of Ca concentration can be due to urea hydrolysis after that temperature which introduces carbonic acid to the solution. Finally, M2 solution combinations are distinguished with their high temperature behavior. Although initial rate of progress is slow, it accelerates as they are exposed to continuous

heat flow for considerable time. Taking M2V3 as example, it shows further potential of reaction progress at 90 °C in spite of its negligible catalyzer content.

Mg ions, on the other hand, are less competitive to Ca ions in terms of carbonate precipitation. From Figure 12, it is clear that none of the solutions shows clear sign of consumption. Despite the fact that there are many studies showings positive effect of Mg on carbonate precipitation efficiency [64], for high concentration, their effectiveness is debatable. However, their presence can affect on morphology of other precipitation.

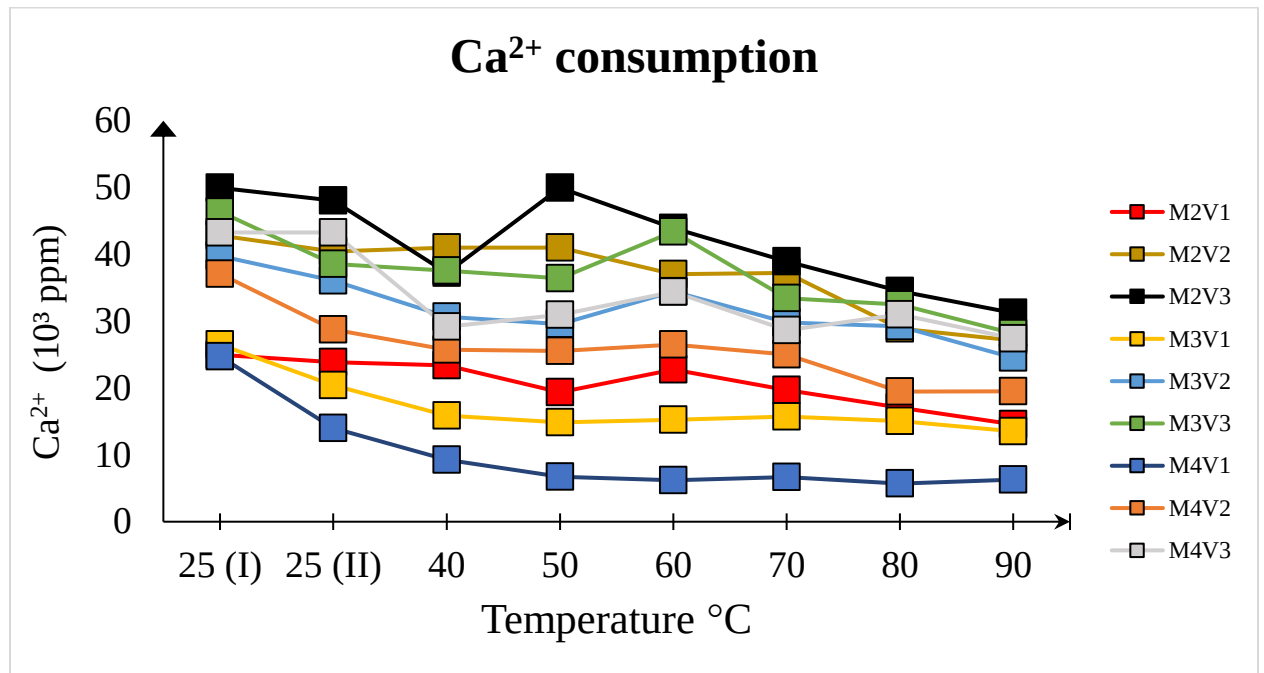


Figure 12: Ca²⁺ consumption

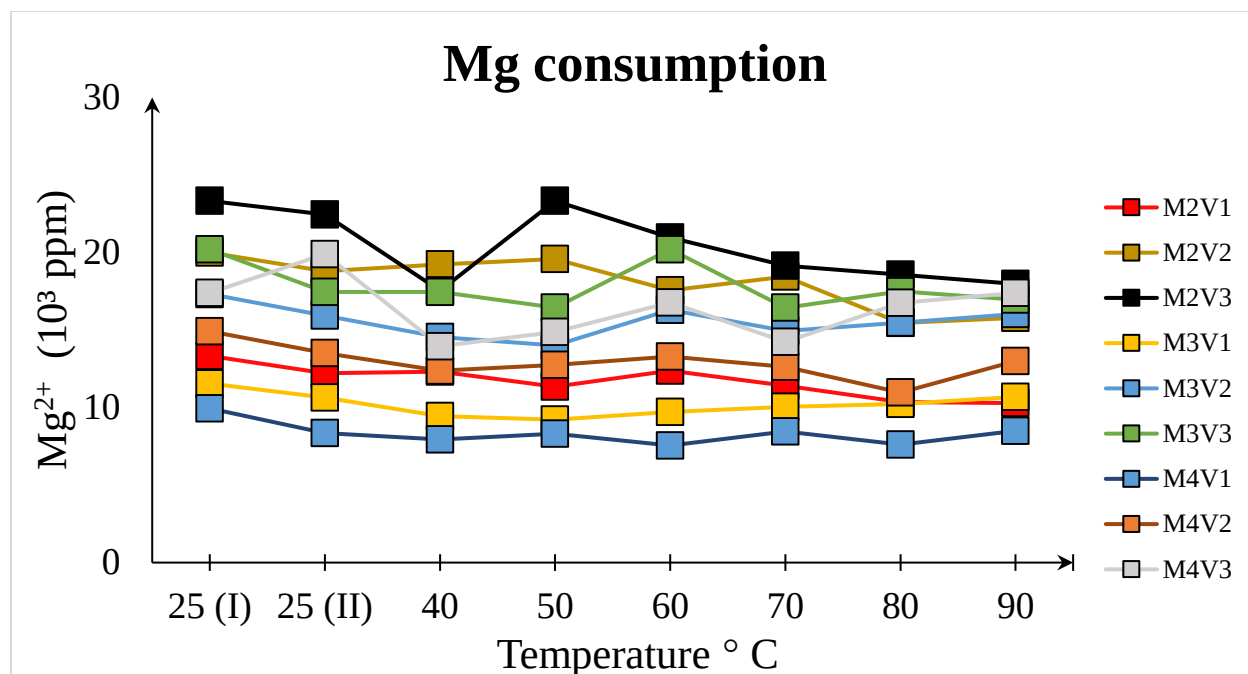


Figure 13: Mg^{2+} consumption

The aim of the study is to show that high temperature environment can favor the precipitation process. Different studies have shown that enzyme activity degrades at high temperatures especial after 70 °C [5]. However, if solutions are saturated with a high number of reactants, the reaction can still be proceeded. Figure 14 below shows the difference between cumulative ion consumption for 70 and 90 °C. It is worthy to highlight that with regard to lesser order of catalyzer from V1 to V3, Ca consumption follow opposite upward trend for both M2 and M3 combinations except for M4 solutions. However, the optimum concentration is not only based on cumulative conversion. For all M2 and M3 cements, less than 50 % of introduced ions are converted to precipitation. However, when the reactant concentration is lower, for example M4V1, the efficiency jumps to almost 80%. This is again followed by a drop to 50 % efficiency as the reactant concentration with respect to catalyzer increased.

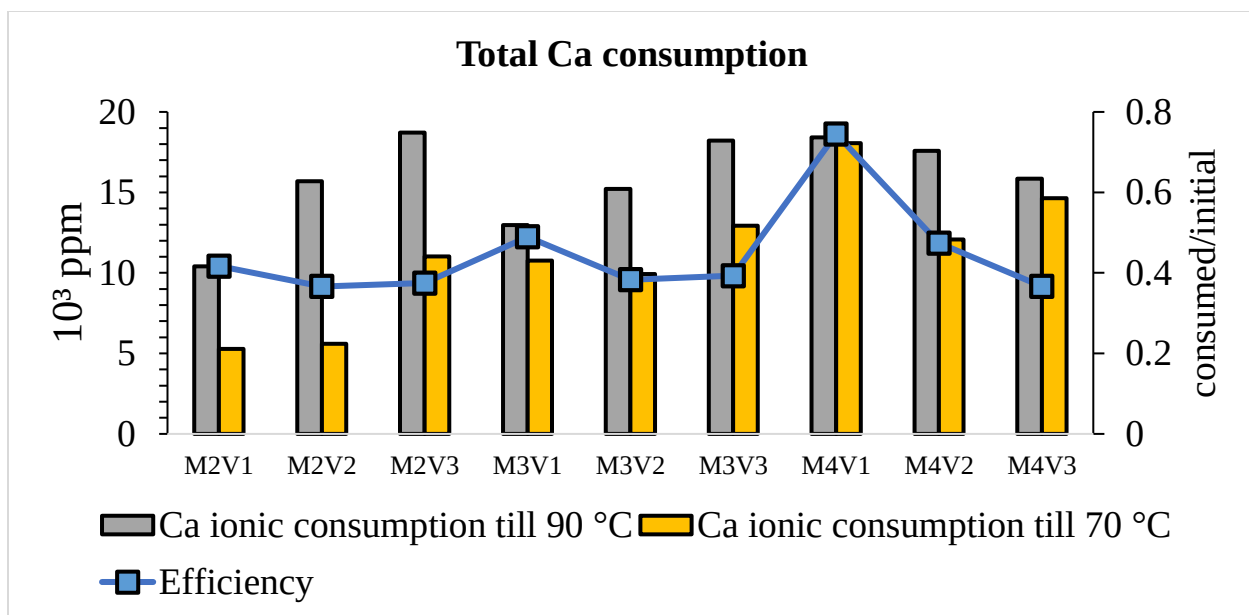


Figure 14: Comparison of final consumptions

Almost in all solutions from M2V1 to M4V3, ionic consumption for 90 C is higher than 70 C. However, this trend in Figure 12 is not followed by Mg ions which is characterized by its less reactive nature.

At the end of study, the mass of precipitation has been measured and mineralogy of precipitate has been determined. Due to low reactivity of Mg, there is no significant trace of dolomite or other minerals related to that chemical (Figure 15). However, as in numerous studies, the presence of Mg gives rise to presence of aragonite which is one of polymorph of calcium carbonate. Another interesting fact is when volume of jack urease is significantly lower than reactants, there is more chance for generation of calcite minerals rather than aragonite.

In terms of mass of precipitation, the amount ranges between 2.5 and 4 g. The combination with M2 solutions yields the highest amount. M3V2 combination which has less enzyme than M3V1 gives 3.7 g precipitation.

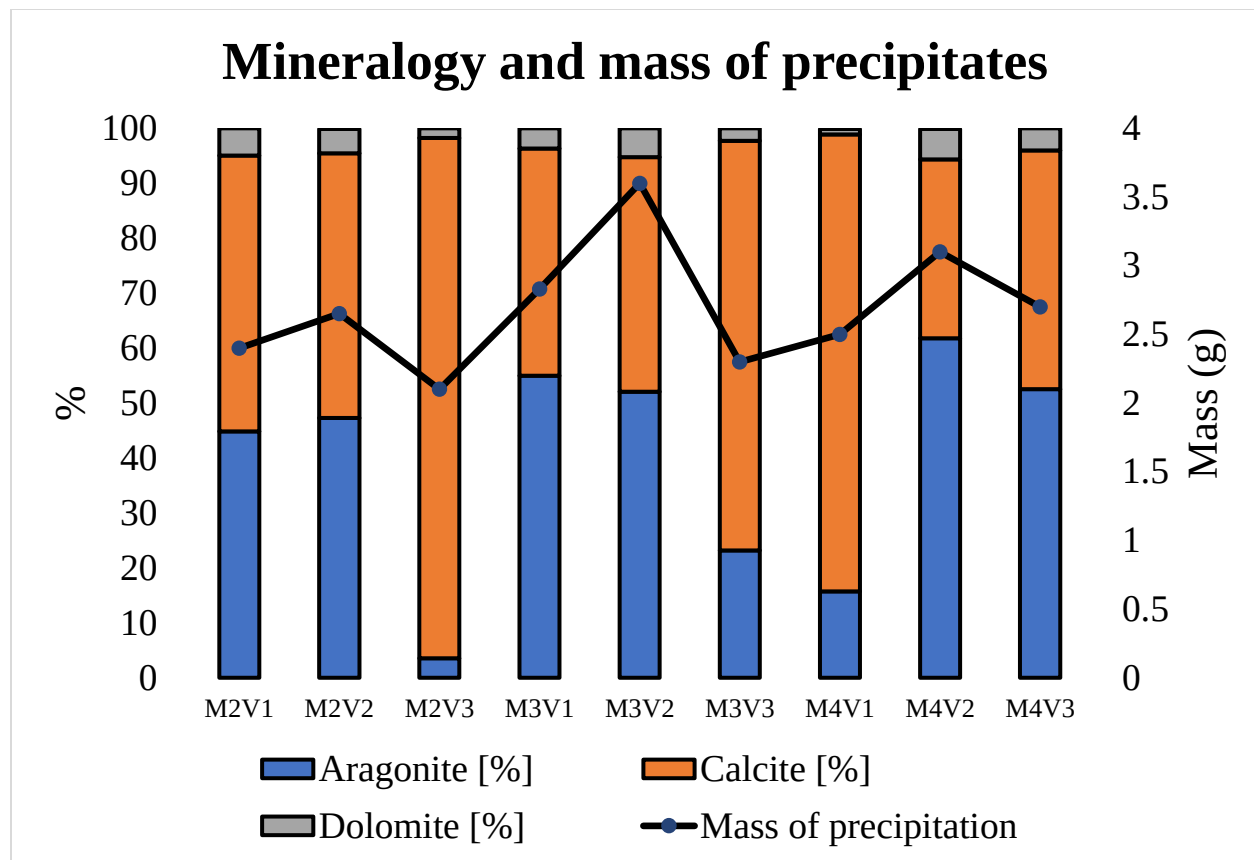


Figure 15: Mineralogy and mass of precipitates

4.2 Sand Consolidation using EICP.

4.2.1 Consolidation Enhancement using Biopolymers.

Prior to consolidation experiments, precipitation sensitivity test has been implemented to choose optimum solutions. To do that, overall cement concentration has been changed from 1M/L to 3M/L by keeping molar ratios of urea to metal ions constant. In terms of mass of precipitation, from A to C, the average mass of precipitation is increasing. However, the efficiency of reaction yield follows different trends. (Table 10)

Table 10: Results of test/tube experiments

Urea	CaCl ₂ *2H ₂ O	MgCl ₂ *6H ₂ O	urease	Theoretical mass	Actual Mass	PR	Sample name
M/L	M/L	M/L	g/L	g	g		
0.5	0.25	0.25	1	1.5	1.01	0.67	A
0.5	0.25	0.25	2	1.5	1.02	0.68	
0.5	0.25	0.25	3	1.5	1.12	0.73	
1	0.5	0.5	1	3	1.09	0.36	B
1	0.5	0.5	2	3	1.1	0.36	
1	0.5	0.5	3	3	1.55	0.52	
1.5	0.75	0.75	1	4.5	1.13	0.25	C
1.5	0.75	0.75	2	4.5	1.17	0.26	
1.5	0.75	0.75	3	4.5	1.26	0.28	

From Figure 16, it is clear that enzymes from jack urease bean is improving precipitation efficiency but with different rates. For lower concentration A, the efficiency is higher 0.65 and improved up to 0.72 by incremental increase. Efficiency is dropping from A solutions to C, almost reaching below 0.3 when the solutions are saturated with 3 M/L cement constituents.

To sum up, it can be generalized that higher concentration solutions can give more precipitation while lower concentration will give lesser but with more efficiency. It should also be noted that those solutions are cured at room temperature. Since they will be exposed

to pore space of loose sand at higher temperature (sample curing), the reaction behavior can differ significantly.

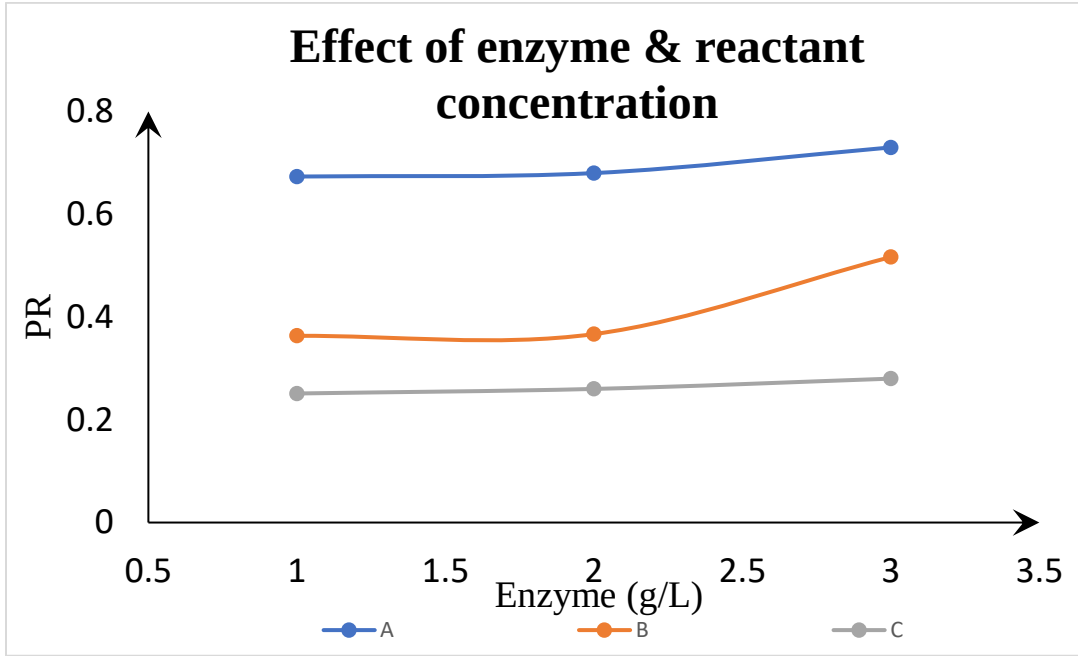


Figure 16: Reaction and catalyzer concentration sensitivity analysis

Figure 17 illustrates the strength of consolidated samples which are treated with concentration discussed above. All the samples were sensitive to reactant concentration and exhibit different rate of increase for their average strength along the sample. Except MNO, JKL and PQR consist of modified EICP solutions with biopolymer (xanthan gum). The performance of other polymers has been discussed in detail in literature [67] . There is an interesting behavior of EICP where 3 M/L cement concentration gives significantly higher strength than others. This solution concentration efficiency was below 0.3 and yielded almost similar weight of reaction products with B solutions. However, it should be noted that the behavior inside pore space is different where higher concentration solutions

regardless of their efficiency gives exceptionally good consolidation. It yielded almost 3000 psi strength which is at least 3 time stronger than its nearest followers. On average, consolidation process has been improved from 179 psi to 503 psi when polymer has been introduced and it is further enhanced to 1036 psi with different methodology.

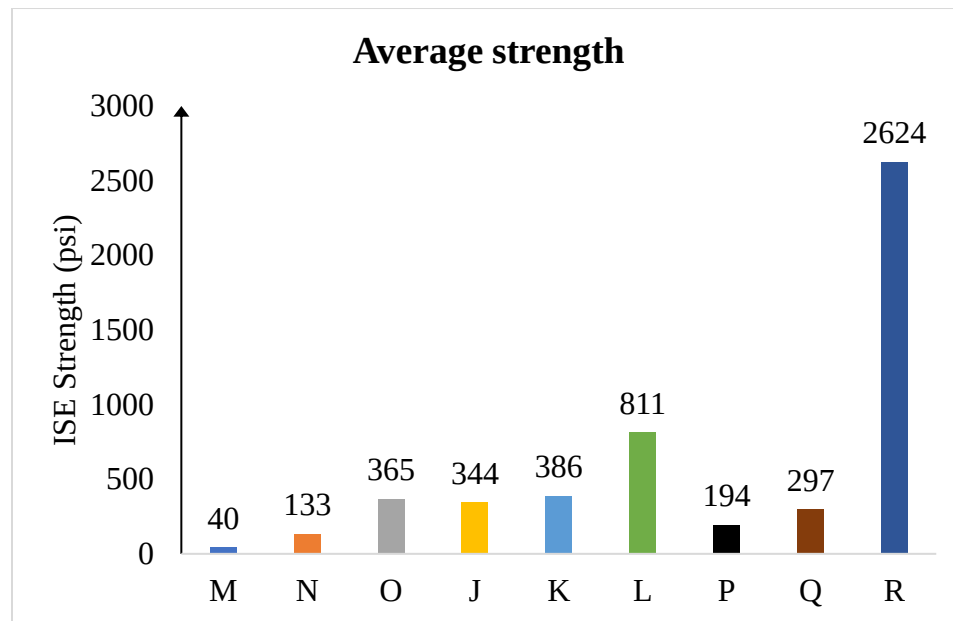


Figure 17: Strength improvement with biopolymers

The second point to address is how precipitations is distributed and how uniform the consolidation process along the sample. It can be observed from Fig 18 that samples treated without polymers have slightly non-uniform distribution at the bottom of sample. This non-uniformity is clearly pronounced when 3 g/L xanthan gum is added to the solution. The bottom half part of sample possess approximately 700 psi while upper stabilized at below 200 psi. To mitigate this effect, polymers are added and shuffled with sand before the treatment. Subsequently, sample P gives more uniform but less strength fluctuating around 200 psi.

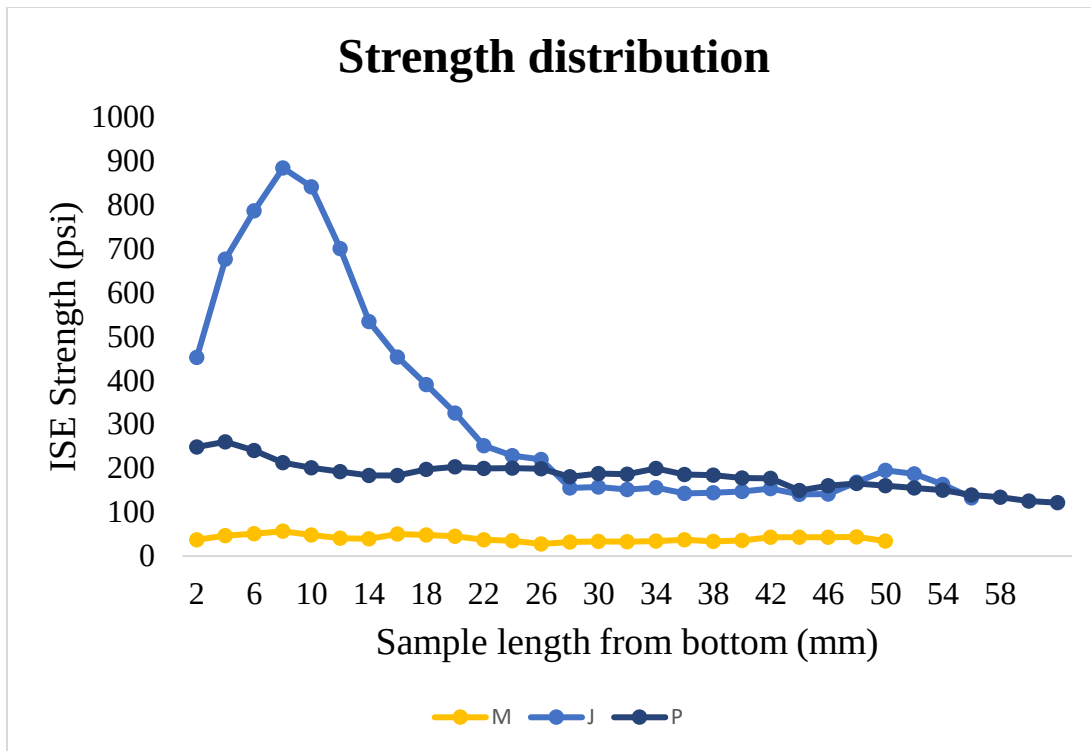


Figure 18: Strength distribution at A concentration

When overall concentration has been increased from 1 M/L to 2 M/L, significant strength improvement has been achieved while non-uniformity becomes more obvious (Figure 19). This concentration led to accumulation of reaction products at the bottom for all samples. The trend is similar to 1 M/L except for samples with mix/shuffle treatment. Although xanthan gum powder has uniform distribution along the sample, the peak at the bottom of sample indicate dominating effect of precipitation. The difference of strength along the sample differs based on polymer and treatment method. Sand treated with base solution exhibit 50 psi difference while this value jumped to approximately 200 psi in the presence of polymers.

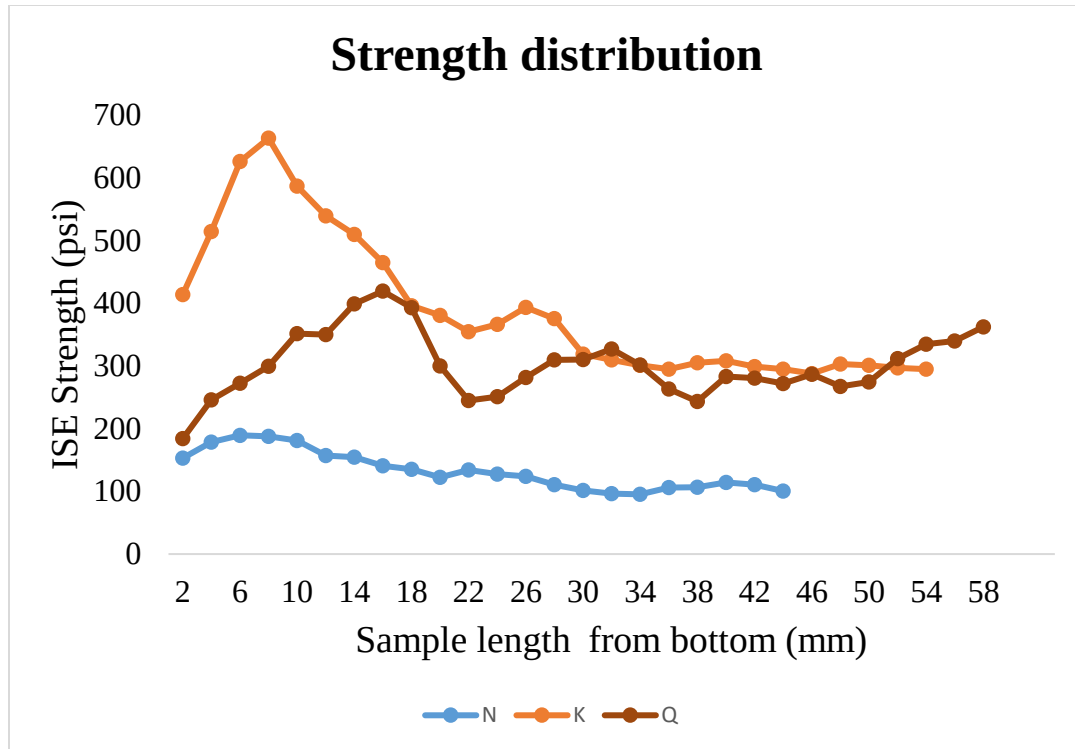


Figure 19: Strength distribution at B concentration

At the final part of study as described in Figure 20, the concentration is further increased up to 3 M/L. With its less efficiency and precipitation mass near to 2 M/L, highest strength has been achieved which was almost 3000 psi at different point of sample. Comparing to A and B concentration, it is more tended to give uniform distribution in spite of similar trend followed by sand sample L. Regarding treatment without polymer, accumulation of precipitation is observed in middle part of sample which is smoothed again near to the bottom of sample. In terms of both uniformity and degree of consolidation, R sample with highest concentration possessed strength value of at least 2000 psi which can be considered ideal for EICP application.

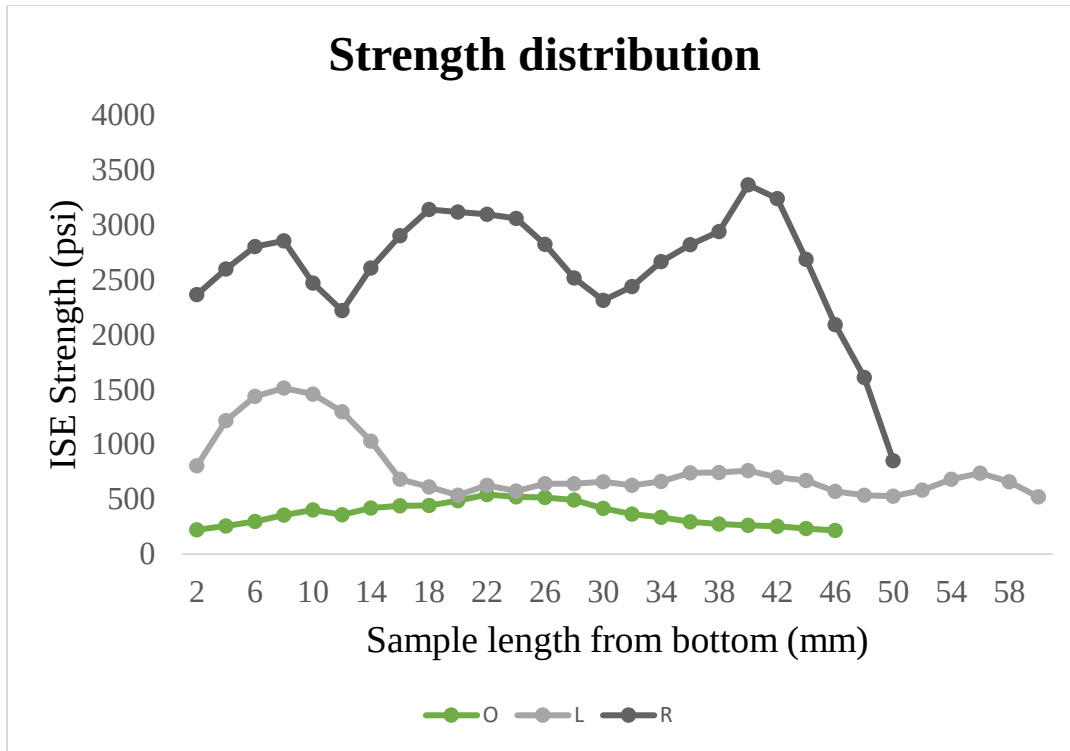


Figure 20: Strength distribution at C concentration

4.2.2 Effect of Sand Particle Size

The aim of this is to analyze how sand particle size and reactant concentration can affect the consolidation process. From the general Figure 21, which contains all samples with different concentration (from high to low) and different sand size (fine to coarse), it can be observed that consolidation process is not uniform through the sample. For all samples, the lower bottom part contains most precipitates, and it is the region where sample has higher strength. However, non-uniformity is different for each treated sand and there is a clear trend from coarse to fine sand.

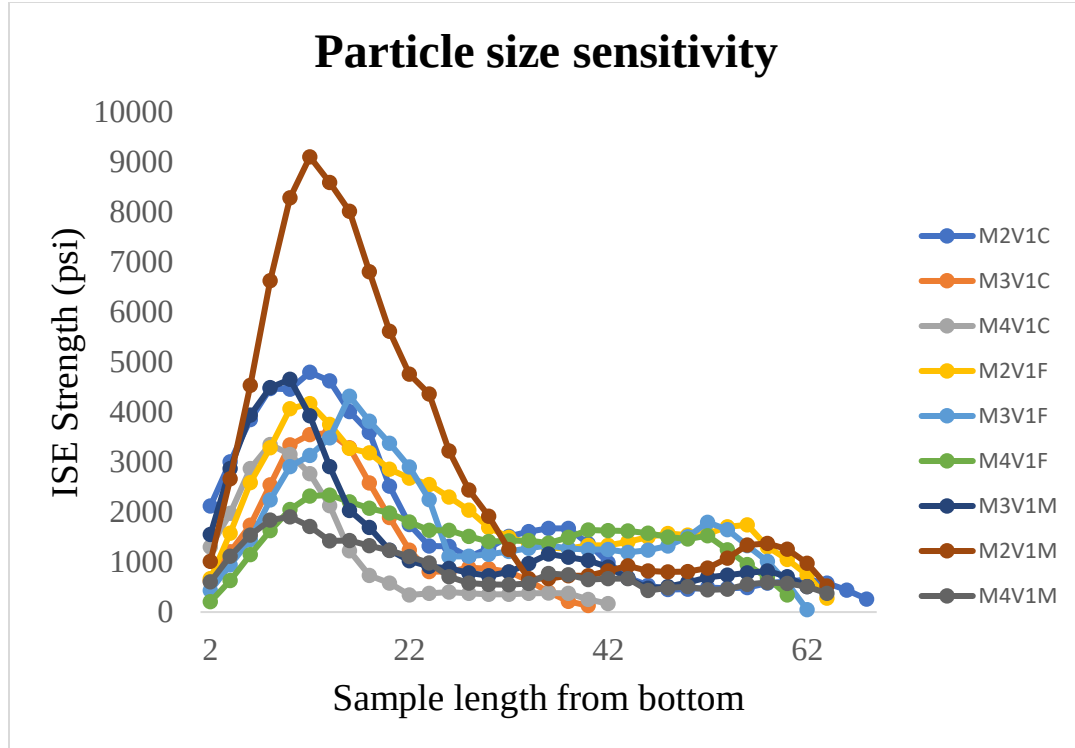


Figure 21: Particle size and concentration sensitivity

At the end of scratch test, high resolution camera is used to take the picture of scratched sample (Figure 22). Regarding coarse sand, 2 of 3 samples cracked during curing. Therefore, the length of those samples is different from others. It can be explained by the fact that large sand particles need uniform and high particle size precipitates to be binded. On the other hand, medium sand size samples are cured without cracking. The concentration from high to low can be observed from the white color accumulated at the bottom part of sample. Treated fine sand samples also showed interesting results. Due to smaller pore space, the distribution of precipitation is more uniform. It is clearly observed in high concentration sample (M2V1) and lower concentration (M2V3).

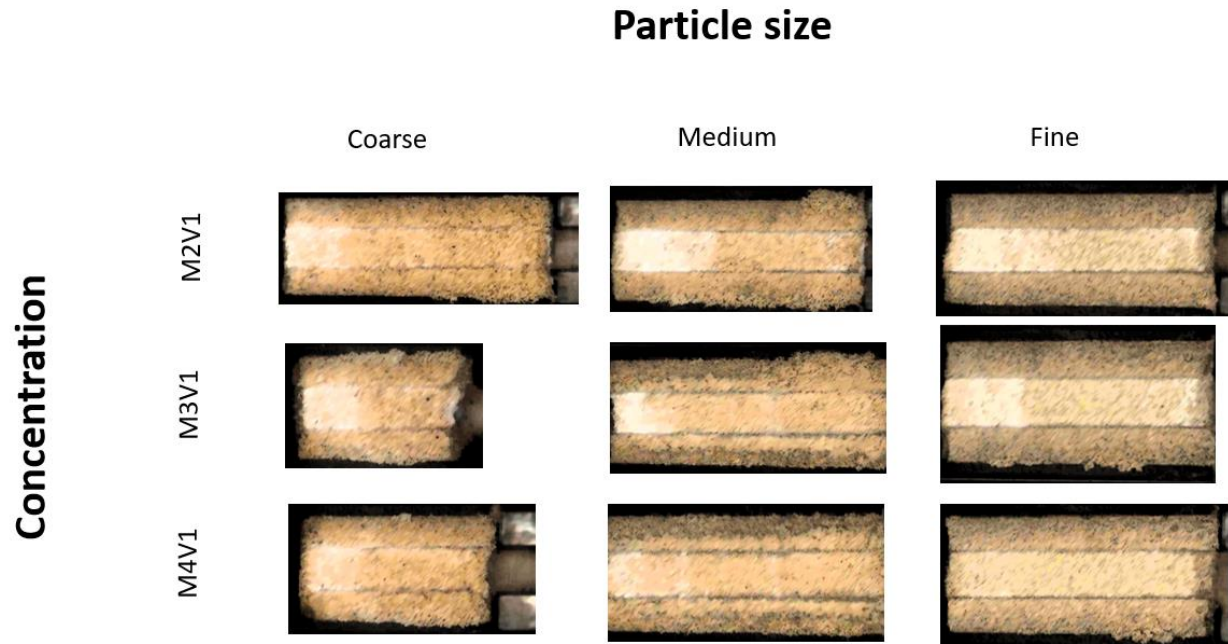


Figure 22: Visual inspection of treated samples

From the spatial strength distribution graph of Figure 23, some of data can be filtered to highlight the effect of concentration and particle size. If we concentrate specifically on high concentration treated (M2V1) ones, medium size sand particles are consolidated with highest strength. However, this strength is especially high for the bottom part and comparatively lower than other in bottom part. In terms of uniformity, both coarse and fine sand is better. Especially, fine sand resulted in both uniform and adequate strength of more than 1000 psi through the sample.

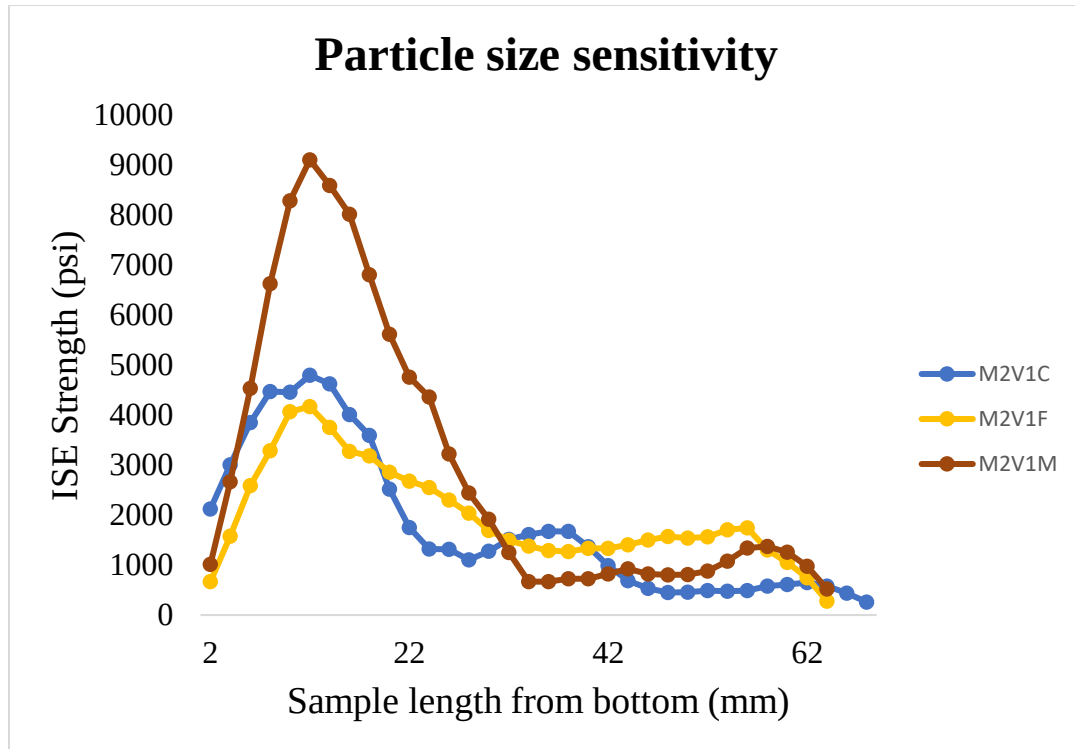


Figure 23: Particle size sensitivity

If emphasis is put on fine sand (Figure 24) which has comparatively more uniform consolidation, the effect of concentration can be observed with incremental strength. However, due to limited pore space, after one point of concentration the consolidation process cannot be progressed. It needs to be noted that low concentration sample with fine is only sand which gives uniform consolidation. This trend can be indirect reflection of particle size precipitation among the pore size.

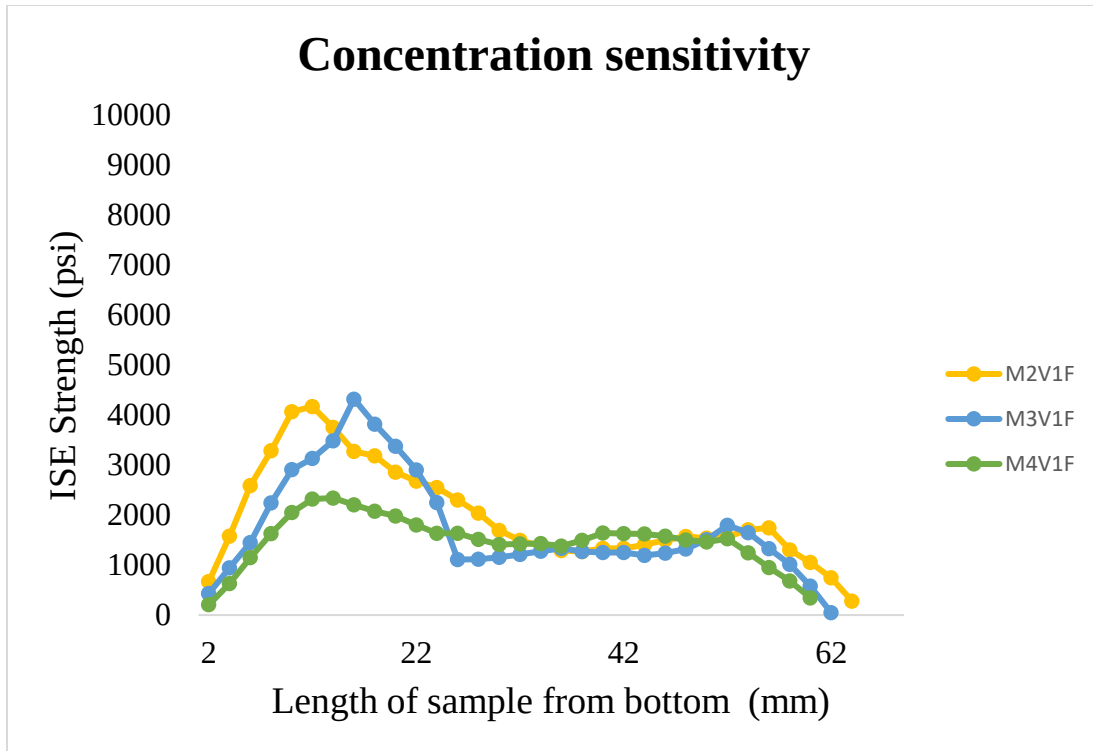


Figure 24: Concentration sensitivity

CHAPTER 5

CONCLUSIONS AND FUTURE WORK

5.1 Conclusions

In this research, the potential of EICP (Enzyme Induced Carbonate Precipitation) as an environmentally friendly sand consolidating agent has been investigated. An extensive study has been conducted on determination parameters that affect its performance and potential solutions to maximize its efficiency. An emphasis is put on to meet requirements of oil gas industry operations. Those are respective minimum strength of 147 psi and high temperature environment that can affect the EICP application. Based on baseline investigation and 2 phases of research regarding temperature sensitivity and sand particle size effect, the following conclusion has been drawn.

1. Incremental temperature from ambient to 90 °C can be favorable for high concentration EICP solutions. It is thanks to improving performance of jack urease bean till 70 °C and high chance of urea hydrolysis without catalyzer.
2. For high concentration solutions containing 240,000 ppm cement constituents, Mg shows a less reactive nature compared to Ca^{2+} . Therefore, it is not feasible to use both metal ions in high concentration to achieve effective precipitation.

3. High concentration EICP solutions have a controllable nature which can ease storage and injection procedures. The reaction progress showed temperature dependence until 50 °C.
4. In most samples, the consolidation process is not uniform along the sample. This is more pronounced in coarse and medium sand samples. In contrast, fine sands can achieve uniform but less strengthful consolidation.
5. Polymers (xanthan gum) exhibits high potential for consolidation enhancement. Their addition contributed to at least 2 times increment in strength. However, uniformity of consolidation has not been improved significantly despite viscosity increase.

5.2 Future Work and Recommendations

1. Design of controllable EICP reaction is necessary for successful sand consolidation. It is especially important if reactants and catalyzer need to be delivered over long distances.
2. Extensive research needs to be conducted on the surface charge studies. For highly porous media, precipitation quantity can be not enough to plug the pores. However, additional cohesive forces introduced by surface charges can enhance the plugging process.
3. Biopolymer exhibits promising enhancement for consolidation process. Further research needs to be conducted on different polymer types. An emphasis should be put on their hydrolysis rate which plays an important role in rock hardening. Besides, their performance with different (injection) methodology needs to be evaluated.

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