FEASIBILITY STUDY OF WATER BASED / POLYMER MODIFIED EICP FOR SOIL IMPROVEMENT INVOLVING RECYCLED GLASS AGGREGATE

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FEASIBILITY STUDY OF WATER BASED / POLYMER MODIFIED EICP FOR SOIL IMPROVEMENT INVOLVING RECYCLED GLASS AGGREGATE

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Thesis

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ABSTRACT

Glass is one of the principal waste products generated in the US. The use of these glass cullet in the construction of shoulder section could reduce the quantity of waste glasses that goes to the landfill. Certain type of cementing agent is required to bind these glass particles in shoulder. Enzyme induced carbonate precipitation (EICP) has shown early promise as a viable and sustainable ground improvement method. Water based EICP leads to faster infiltration of cementation solution due to high permeability, thus limiting the amount of available reaction substances to produce $CaCO_3$ precipitate at desired locations. This problem may be solved to some extent by the use of high viscosity polymer as a carrier of cementation solution in place of water.

Laboratory tests performed on the recycled glass cullet showed the possibility of using them in the construction of shoulder section to prevent erosion. Moreover, a series of laboratory experiments performed showed that EICP worked well on the Ottawa sand but did not work well on recycled glass cullet. However, it was successful on the samples containing mixture of glass particles and Ottawa sand. The samples consisting up to 20% of recycled glass in the mixture were brittle and strong. The results of UCS testing showed the compressive strength of the intact sample decreases with increase in amount of recycled glass in the mixture. The pull out test carried out on the glass surface showed the possibility of application of EICP on the surface treated glass particles.

SEM, XRD and TGA results on the samples treated with polymer modified EICP verify the presence of $CaCO_3$ and the strength of the samples were tested at different moisture contents. The treated sand columns were organic-inorganic

composites with sand cemented by a $CaCO_3$-PVA mixture. Unlike low molecular weight PVA, medium molecular weight PVA forms complex matrix with the $CaCO_3$ precipitate which does not dissolve in water at room temperature. The unconfined compression tests revealed that the strength and ductility of the soil columns treated with MMV PVA are moisture sensitive: the strength decreases but ductility increases with moisture content.

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1 INTRODUCTION

1.1 Problem Statement

10.7 million tons of waste glass was generated in the U.S in 2003 .Among them, only 2.35 million tons, or 22 %was recycled (Robinson 2006) .The clear glass cullet is primarily used in the manufacturing of new glass container .As a result, the colored glass cullet has a relatively low market value since they require color sorting, which could be expensive .So, some of these glasses are sent to the landfills instead of recycling, which adds to the extra cost. This glass cullet can be used in local roadway applications that do not adversely affect the performance or durability of the pavement structure. The use of glass cullet are not incorporated in highway and interstate applications due to the propensity of glass particles to strip off the surface course in the presence of moisture and lack of consistent supply of product. However, there may be an appropriate application of these materials in local system. [1]

The glass cullet may be used in the construction of shoulder sections in local applications. Shoulders are the critical components of the roadway systems, the quality of which directly affects the health condition of the pavement and safety of the traveling public. The erosion of shoulder has been identified as the major cause for crashes on rural two lane undivided highways [2]. Pavement edge drop-off is the problem with the shoulders that refers to the vertical elevation difference between the pavement surface and the shoulder surface [3]. Rain- or wind-induced erosion, irregular slopes caused by granular material degradation, vehicle off-tracking, or insufficient bearing capacity of subsurface soil under shoulders are the major factors causing pavement edge drop-off [4]. The erosion of shoulder materials occur when

the erosive forces surpass the resistance forces. Particle shape, particle size, cohesion and compactness of the shoulder materials are important parameters to ensure resistance of the shoulder materials to erosion. Proper aggregate mix design, compaction techniques and construction methods are the key factors for achieving a high-quality aggregate shoulder [5].

Certain type of cementing agent is required to bind these glass cullets in the shoulder section. Enzyme induced carbonate precipitation (EICP) has shown early promise as a viable and sustainable ground improvement method. However, water based EICP leads to faster and non-uniform infiltration of cementation solution in the subsurface region due to high permeability of water, thus limiting the amount of available reaction substances to produce $CaCO_3$ precipitate at desired locations. This limits the implementation of water based EICP method as a one-shot treatment [6]. This may be solved to some extent by the use of high viscosity polymer as a carrier of cementation solution in place of water.

1.2 Research Objective

The main objective of this study is to find the application of recycled crushed glass aggregates in roadway application, that would otherwise go to landfill for disposal. Enzyme induced carbonate precipitation (EICP) has shown early promise as a viable and sustainable ground improvement method. The feasibility of application of EICP for soil improvement involving recycled glass is checked. The specific objectives of this study are:

- Identify various reconditioning methods to prevent the erosion of roadside shoulder materials, caused by flowing water during the rainy season.

- Collect recycled crushed glass cullet from recycling company and determine their properties. Compare the properties of these glass cullet with those of natural aggregates, to check the possibility of using them in place of or along with natural aggregates in shoulder construction.
- Check the feasibility of application of EICP for soil improvement involving recycled glass.

This thesis is an exploratory research work to find out a technique to stabilize the shoulder section involving recycled glass and the resulting section should have sufficient strength and ductility.

1.3 Thesis Outline

This thesis is the result of combination of two project works funded by Ohio Department of Transportation (ODOT). The phase I of projects titled "Evaluation of post flood shoulder reconditioning" and "Use of crushed recycled glass in the construction of local roadways" were completed. Literature study was carried out to check whether glass cullet can be used in the unbound roadway shoulder as construction material. Moreover, EICP method is included to check the feasibility of their application to bind the particles together and to study the behavior of treated samples at different condition.

This thesis comprises of six chapters. The first chapter includes the purpose of carrying out the research i.e. the problem statement, research objective and thesis outline. The second chapter comprises of literature review on shoulder erosion, use of glass aggregates on roadways and application of EICP. The third chapter consists of various tests on recycled glass aggregates to determine their engineering properties and compare those properties with that of natural aggregate.

The fourth chapter includes various EICP testing carried out on graded Ottawa sand and glass particles. The fifth chapter comprises of polymer modified EICP method. Materials, experimental procedures, observations and results, and discussions are covered in these chapters. The sixth chapter covers summary and conclusions, and future work.

2 LITERATURE REVIEW

2.1 Recycled Glass Aggregates

Any types of material that has no lasting value and comes out as byproduct of human activities or from the industries is defined as waste materials[7]. The increase in the quantity of such waste generation, shortage of landfill area, and lack of natural materials has increased the necessity of recycling such wastes[8]. Glass is one of the principal waste products generated in the US [9]. The glass cullets are obtained from crushing the waste glass collected in industrial and municipal waste streams to smaller size. The clear glass cullets are used in manufacturing of new glass containers. But, colored glasses require color sorting that increases the cost of production[1]. So, they are sent to the landfill for disposal, which adds to the extra cost. However, this cullet could be used for various engineering applications. They can be mixed with aggregates for the construction of roadway and parking lot base or leveling courses, pipe bedding, drainage system, embankment, landfill and concrete [10]. The use of reclaimed glass offers the advantage of reducing a portion of material that would otherwise go to a landfill. That benefit matches well with the commitment of counties and cities to environmental and economic responsibility.

In 1991, Sibley County found for the first time the use of glass that had nowhere to go but to a landfill at a cost of $60 a ton. Analysis and testing of the samples at the Minnesota Department of Transportation)Mn/DOT (showed that adding glass to gravel actually helped increase the quality of gravel.[11] These glass culets include the mixed-color container glass from consumer foods and beverages, ceramic and china dinnerware, and windows from buildings. However, glasses

containing toxic materials cannot be used [12], which include car windshields, other car glass, light bulbs, porcelain, laboratory glass, and glass from television sets and computers. The reclaimed glasses may contain debris like non-glass material such as paper, foil, plastics, metal, corks, wood debris, food residue, or other deleterious materials. The debris content in the reclaimed glass shall not be more than 5 percent. The toxic chemicals, if present in the glass cullet, may leach when water flows through this cullet. It will have some negative impacts on the environment. However, the study on the glass cullet obtained from municipal wastes, curbside and industrial sectors showed that the chemical properties of glass and glass cullet leachate are all within the acceptable ranges [13]. Hence, they do not pose environmental risk for construction aggregate users.

The study by Arulrajah et. al. [14] showed the potential of using of recycled glass aggregates in unbound pavement base/subbase applications. Water absorption and roughness of the surface were found to be the controlling factors for compaction curve. The smooth surface and low water absorption capacity of the glass particles result in water insensitive behavior of glass particles. The test results from LA abrasion, Direct Shear test, CBR test, CD triaxial test showed that the glass particles meet the physical and shear strength property requirements for aggregates in pavement base/subbase applications.

Certain percentage of recycled crushed glass is mixed with natural aggregate to produce hot-mix asphalt, which is termed as glassphalt. Some gradation requirements should be met while using glass aggregate for the roadway construction. Particles smaller than 3/8-inch are recommended for the surface course. During placement, particles larger than 3/8-inch have a tendency to align themselves parallel to the road surface, thereby lowering the skid resistance. Larger particles also have

tendency to strip out of the surface more easily. However, particles larger than ¾-inch can be used for the base course [15].

2.2 Shoulder Drop-off and Erosion

[This section is included in the report published on "Evaluation of Post Flood Shoulder Reconditioning" [3]]

The shoulder drop-off problem results from the displacement of shoulder materials by one or more forces, such as traffic loads or water. The extent of the shoulder drop-off depends on the composition of the shoulder materials. In Ohio, shoulders are either paved or unpaved: paved shoulders have the same or similar composition as the roadway, while unpaved shoulders are either composed of unstabilized earth materials or stabilized materials. Unstabilized or granular shoulders, which are typically used for low-volume roads, are made of aggregates and onsite earth materials and are most prone to be damaged or eroded. Although a granular shoulder has a lower initial construction cost (by up to 70% compared to a paved shoulder [16]), it typically adds more expense during its service life due to the need for more frequent maintenance. Key factors for achieving a high-quality aggregate shoulder include proper aggregate mix design, compaction techniques and construction methods [5]. The ability of a granular shoulder to resist deterioration, which can result in shoulder erosion and drop-off, depends on its surface stability and strength. According to an unpublished survey of Virginia DOT maintenance managers concerning aggregate shoulder maintenance activities, it is believed that a shoulder should undergo maintenance approximately once every six months [17]. This is consistent with the ODOT Holmes County Garage's practice of about twice per year.

The figure below illustrates common problems with granular shoulders, which include:

- Erosion by wind, rain and pavement surface drainage;
- Vehicle off-tracking;
- Settlement of soft underlying subgrade; and
- Irregular slope caused by granular material degradation.

Most relevant to this study is the rain- or flood-induced erosion of granular aggregates, which will be discussed in more detail in the next section. However, it should be noted that these factors can be correlated and can influence one another.

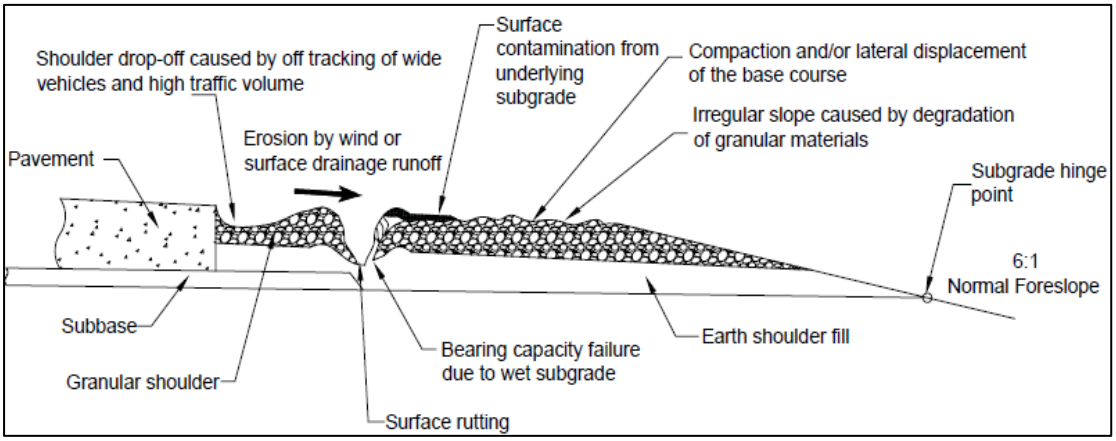


Figure 2-1 : Common Granular Shoulder Problems (D. J. White et al., 2007)

Reports from previous state or federal DOT projects on shoulder reconditioning were reviewed to identify alternative strategies. In addition, research papers or theses on this topic were reviewed. The literature review revealed that six main shoulder reconditioning techniques have been studied or adopted by state DOTs: 1) Reshaping (pulling) shoulders involves using equipment (usually a motor grader) to pull materials from the base of the shoulder slope back up to the pavement edge. The materials are then compacted using compactors such as a pneumatic tire roller.

2) Replenishing is a process similar to reshaping, but it is performed when there is more than a two-inch drop-off and when there are not enough materials left on the shoulder to reestablish its original shape and slope.

3) Promoting growth of vegetation is beneficial because it increases the shoulder's resistance to wind and water erosion. The root system of the vegetation helps to hold the aggregate in place under all climate and soil conditions.

4) Chemical stabilization of shoulder materials is another potential strategy for increasing resistance to erosion. The unbounded materials can be stabilized with chemical agents including cement, emulsion, salts, asphalt, and so on to increase its stability and extend the longevity of the pavement.

5) Mechanical stabilization mainly involves placing geosynthetic products to hold the aggregates in place. Different types of geosynthetic products such as geo-fabrics, geo-grids or three-dimensional geocells can be used.

6) Paving the shoulder is the ultimate solution to increase the bearing capacity and erosional resistance of the shoulder. Since the function of the shoulder is different from that of the pavement, the paved shoulder does not need to possess the same structure as the pavement. Based on traffic volume and the original bearing capacity of the subgrade, a different design on the shoulder section can be proposed. In order to save cost, reclaimed material such as recycled asphalt pavement (RAP) materials can be used.

A brief summary of the different methods for shoulder reconditioning is summarized in Table 2-1.

Two additional measures can also be considered:

1) Hydraulic measures

The above methods are all used for the reconditioning of the shoulder from the perspective of improving the material strength or the erosional resistance. As mentioned earlier, erosion occurs when the flow-induced erosive force exceeds the resistive strength of the material. There, it is equally important to reduce the erosive force, or the flow velocity, acting on the shoulder. The most efficient approach is to divert water away from the shoulder by means of ditches or other drainage measures. Such methods can be categorized as hydraulic measures.

2) Safety edge

The ultimate goal of shoulder reconditioning is to ensure the safety of the traveling public. Other structural methods, such as a safety edge, have proven to be a relatively easy and inexpensive countermeasure to steep pavement edges or drop-off. A safety edge is a 30- to 35-degree tapered asphalt wedge or fillet installed along pavement edges. Safety edge provides stronger and more stable pavement edge by eliminating the abrupt drop-off and makes it easier for drivers to maneuver their vehicles back onto the roadway safely. [18]

Table 2-1: Summary of existing post-flooding shoulder reconditioning techniques

Technique	Material	Equipment	Effectiveness	Cost, time, labor	Ref.*
Reshaping	On-site materials	Motor grader, tractor broom, roller, etc.	Effective only for a few weeks up to a year under optimal conditions.	Quick, require minimum personnel and equipment.	[19]
Replenishing	Additional aggregates	Motor grader, dump trucks, tractor broom, front end loader, etc.	Lost granular materials should be added regularly (about once every 6 months).	Initial cost: $13,376/3ft-mile; Maintenance cost: $259/3ft-mile-year [2]; $2,370/3ft-mile-year [3]	[20], [17]
Vegetation	Vegetation		Vegetation increases shoulder stability in all climates and is the most practical and economical method available for reducing soil erosion.		[2]
Chemical Stabilization	Polymer emulsion	Road reclaimer, rollers, etc.	The stabilized granular section performed inadequately.		[4]
	Foamed asphalt (FA)	Road reclaimer, rollers, etc.	Effective for a short period of time.		[4]
	Soybean oil soapstock	Road grader, trucks for soapstock, water and sand	Not successful [5]; successful under certain conditions [8].		[4], [21]
	Portland cement	Road reclaimer, rollers, etc.	Edge drop-offs and erosion were observed after four months.		[4]

Technique	Material	Equipment	Effectiveness	Cost, time, labor	Ref.*
Chemical Stabilization (Continued)	15% to 20% Class C fly ash with the upper 12 in. of subgrade clay	Road reclaimer, pad foot roller, smooth wheel roller, etc.	Successful in improving both the short- and long-term performance; both California bearing ratios (CBR) and modulus increase significantly.		[2]
	Soiltac and Centrophase AD	Road reclaimer, rollers, etc.	Do not increase shoulder stiffness or bearing strength (but were only tested on crushed run stone). Insufficient data to determine their effectiveness to improve a shoulder's short-term resistance to wind or water erosion.		[17]
	Lignosulfonates	Road reclaimer, rollers, etc.	No significant difference in performance between stabilized and control section.		[22]
	Reclaimed asphalt pavement (RAP) and Portland cement (PC)	Motor grader, dump trucks, rollers, etc.	Successfully stabilized the shoulders. The rehabilitated section held up for a period of 6 years.		[23]
Mechanical Stabilization	Geogrids were placed at the interface between subgrade and granular layer	Rollers, etc.	Considerably improved the performance of the shoulder test section and eliminated rutting.		[4]
	Artificial soil reinforcement (geosynthetic mesh and grid)	/	/		[2]

Technique	Material	Equipment	Effectiveness	Cost, time, labor	Ref.*
Mechanical Stabilization (continued)	Geo-cellular materials such as geo-block or geo-web	Staple driver, roller side compactor	Sediment erosion reduced by 200% (geo-block) or 47% (geo-web).		[24]
Paved Shoulder	Portland cement/asphalt, aggregates, etc.	Motor grader, dump trucks, rollers, etc.	Reliable performance and require much less maintenance effort.	Initial cost: $53,469/3ft-mile ; Maintenance cost: $259/3ft-mile-year; $76/3ft-mile-year	[20]

2.3 Enzyme Induced Carbonate Precipitation (EICP)

Recent researches on EICP have demonstrated the improvement in strength and stiffness of the soil by the precipitation of $CaCO_3$ [25]. The urea, calcium chloride and urease are combined together in an aqueous solution to induce calcium carbonate precipitate, which coats the soil particles, bind them together and fill the pores between particles. Thus, carbonate precipitate improves the mechanical properties of soil [26]. In EICP, the free urease enzyme derived from the plant source is used for enzymatic hydrolysis. It catalyzes the hydrolyses of urea ($CO(NH_2)_2$)) into carbon dioxide (CO_2) and ammonia(NH_3). The carbon dioxide so released combines with calcium obtained from calcium chloride to form calcium carbonate. The overall reaction is given as [25]:

$$CO(NH_2)_{2(aq)} + 3H_2O = CO_{2(aq)} + 2NH_4{}^{+}{}_{(aq)} + 2OH_{(aq)}.$$

$$H^{+}{}_{(aq)} + HCO3{}^{-}{}_{(aq)} + 2OH_{(aq)} + Ca^{2+}{}_{(aq)} = CaCO_{3(S)} + 2H_2O.$$

The net reaction can be written as:

$$CO(NH_2)_{2(aq)} + 2H_2O + CaCl_{2(aq)} = CaCO_{3(s)} + 2NH_4Cl_{(aq).}$$

EICP has the potential to be used as an alternative to Portland cement for various ground improvement techniques. The manufacture of cement is one of the major causes for climate change, as it accounts for emission of about 5% of global man-made CO_2[27]. Ground improvement via inter-particle cementation by calcium carbonate can address variety of geotechnical problems by means of mass stabilization including slope stability, water and wind erosion, bearing capacity, scour, tunneling in flowing and running sand masses, liquefaction and seismic settlement [25], [28], [29].

The research work performed at Arizona State University has shown that stabilization of surficial erodible soils, mass soil stabilization and columnar soil stabilization can be achieved through EICP method [26]. Authors used coarse silica sand (Ottawa 20-30), medium-fine silica sand (F-60) and native AZ sand for columnar stabilization. Tests were performed at various concentrations of cementation media. Carbonate precipitate was observed in the soil columns up to cementation media concentration of 1.6M, while above that concentration, no precipitate was formed. Columns (0.2M and 0.5M) remained intact upon extraction, column (0.8M) broke into several chunks, and columns (1.1M, 1.3M and 1.6M) had small randomly occurring intact chunks soil that were held together by a cementing agent which was confirmed to be carbonate later by acid digestion. Although the actual reason for these variances is not known, the method of sample preparation and the poor mixing environment can concentrate solute and make EICP process complicated.

The higher value of Unconfined Compression Test (UCS) was found for 0.5M (peak strength = 220 kPa). The acid digestion showed certain differences in the maximum amount of $CaCO_3$ precipitate for different concentration of cementation media. It may be because $CaCO_3$ precipitate form in large range of sizes. The smaller precipitate (<40mm), which are not bound to the soil particles or other large $CaCO_3$ crystals, may be lost during initial drainage or subsequent rinse cycles. The columnar samples were prepared in two ways. The first approach consists of pouring dry sand into the column from drop height of 127mm up to a desired height followed by adding EICP solution. The second method was mix and compact method. In this method, the column was filled with EICP

solution followed by pouring sand in the column. The latter sample gained more strength for Ottawa 20-30 sand when tested with the UCS machine (peak strength = 529 kPa).

Zhao et. Al. tried to find experimental factors that affect MICP process catalyzed by bacteria and urease [30]. Authors used urease enzyme from Fisher Scientific and Ottawa sand. Full contact flexible molds were used to prepare the sample and were immersed in cementation solution for several days. Concentration of urease enzyme, cementation solution concentration, reaction time and curing conditions were altered to see the effects. The Unconfined Compression Test (UCS) performed on the treated sample showed an increase in compressive strength of sample when the concentration of urease enzyme increased from 5 to 15 g/L. The sample treated by 2.5 g/L urease could not form an intact sample.

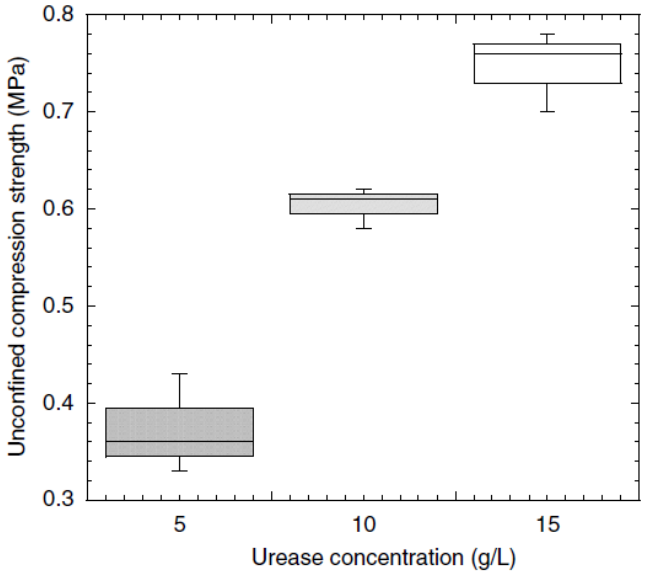


Figure 2-2: Box plot of UCS of EICP treated sample with urease concentration

Source [30]

When sample was treated with 0.25M cementation media, it could not form an intact sample. The tests were repeated with cementation media concentration of 0.5, 1 and 1.5M. The treated sample remained still loose when cementation media concentration was 0.25M and the median UCS of treated sample was 0.6MPa at 0.5M concentration. This increase in strength was higher as compared to the change in strength of treated sample when cementation media concentration was increased from 0.5M (0.6MPa) to 1.5M (0.8MPa). This shows that when the concentration of media is above 1M, the concentration of urea and calcium ions in the solution is excessive and not fully utilized.

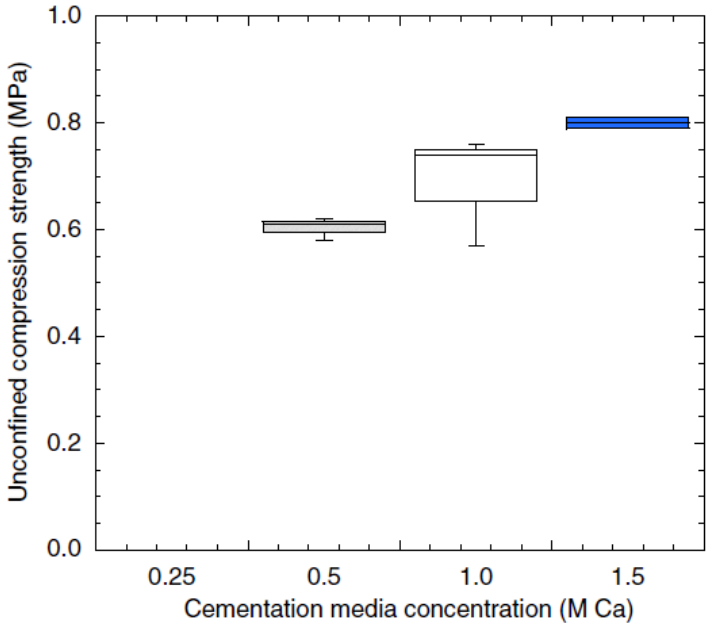


Figure 2-3: Box plot of UCS of EICP treated sample with cementation media concentration

Source [30]

The high permeability of water leads to faster and non-uniform infiltration of cementation solution in the subsurface region, thus limiting the amount of available reaction substances to produce $CaCO_3$ precipitate at desired locations. This limits the implementation of water based EICP method as a one-shot treatment [6]. This difficulty seems to be addressed by the use of proper hydrophilic polymer instead of deionized water as a carrier of enzyme and cementation media. PVA has high viscosity and provides appropriate reaction time and environment for the hydrolysis of urea. Polyol-cellulose, guar gum, and xanthan gum were used to enhance the performance of the enzyme-induced carbonate precipitation (EICP) approach for surface soil stabilization [31].

3 CHARACTERIZATION OF RECYCLED GLASS PARTICLES

3.1 Laboratory Testing

Laboratory testing was performed to determine the engineering properties of the recycled glass cullet.

Materials

Two types of glass cullet were selected for testing having different gradations. They are named as type 1 (relatively finer) and type 2 (coarser). All the tests were performed on 100% glass cullet.

(a) (b)

Figure 3-1: Recycled glass aggregates (a) Type 1 (b) Type 2

3.1.1 Gradation Analysis

Sieve analysis was performed to determine the gradation of the glass cullet. Testing was in accordance with ASTM C136/C136M - Sieve Analysis of Fine and Coarse Aggregates.

Table 3-1: Sieve Analysis

Sieve Size (mm)	Percentage Passing	
	Type 1	Type 2
25	100.00	100.00
19	100.00	92.51
12.5	100.00	46.61
9.5	99.34	13.94
4.75	72.76	0.83
2.36	38.03	0.40
1.18	18.22	0.27
0.6	8.84	0.15
0.3	3.41	0.07
0.15	0.98	0.00
0.075	0.10	0.00

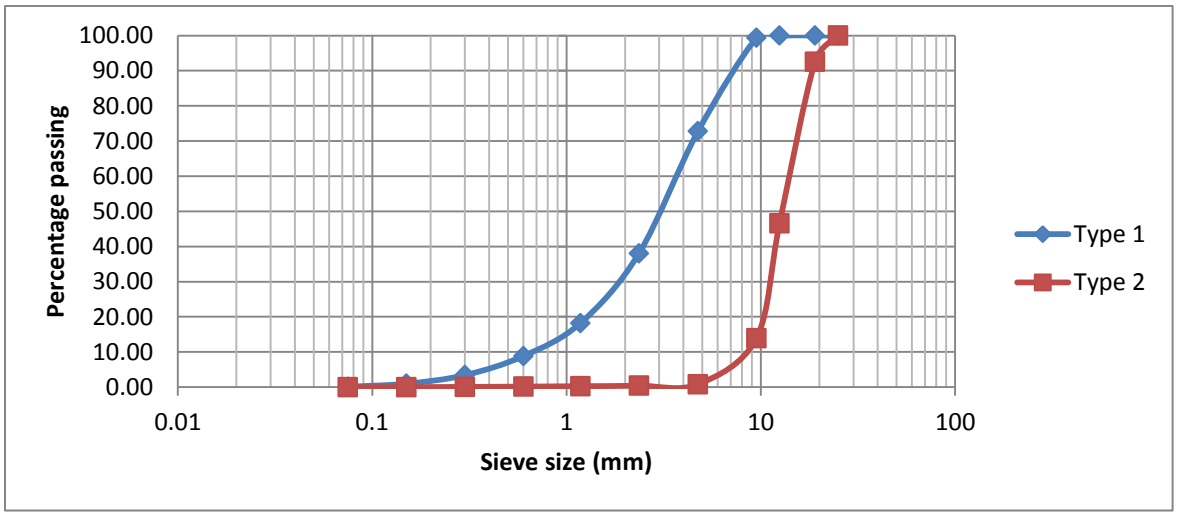


Figure 3-2: Gradation curve for recycled glass aggregates

Table 3-2: Classification of recycled glass aggregates

Type 1	Type 2
D_{60} = 3.7	D_{60} = 12.5
D_{30} = 1.9	D_{30} = 10.7
D_{10} = 0.63	D_{10} = 8.5
Cu = 5.87	Cu = 1.47
Cc = 1.55	Cc = 1.08
USCS Classification: Poorly Graded Sand (SP)	USCS Classification: poorly Graded Gravel (GP)

The type 2 cullet satisfies the requirement for ODOT No. 57 specifications.

3.1.2 Specific Gravity

Specific gravity is the measure of density of the solid. The specific gravity of glass cullet was determined following AASHTO T85. The specific gravity of type 2 glass cullet was measured.

Procedure:

- The sample was sieved and any materials passing through No. 4 (4.75mm) was discarded.
- The aggregate was then washed.
- The sample was then placed in the oven regulated at 110±5 °C for about 24 hours. Then the sample was allowed to cool at room temperature. The dry weight of the sample was determined (A).

- The sample was immersed in water for about 15 hours.
- The entire sample was placed in the container and the weight of sample under water was determined. (C)
- The sample was then removed from the container and the water present on the surface of aggregate was removed using an absorbent cloth. The saturated Surface Dry (SSD) weight of the sample was determined. (B)

Observations

Wt. of oven dried sample (A) = 1019.6 gm

Wt. of SSD sample (B) = 1022.2 gm

Wt. of sample under water (C) = 646.2 gm

Calculations:

Bulk Specific Gravity = $\frac{A}{B-C}$

= 1019.6 / (1022.2 – 646.2)

= 2.71

The bulk specific gravity was found to be 2.71, which is comparable to the specific gravity of gravel.

3.1.3 Compaction characteristics

It includes the relationship between moisture content and the density. Standard proctor compaction test was done in accordance with ASTM D698 and a curve was obtained.

Type 1Glass material

The sample consists of more than 25% of the particles being retained on 4.75 mm sieve and less than 25% of the particles retained on 9.5 mm sieve. So, method B of standard compaction test was selected.

Size of mould = 4 inches diameter

No. of layers compacted = 3

No. of blows per each layer = 25

Weight of rammer = 5.5 lbf

Height of fall of rammer = 12 inches

The standard compaction test was carried out at different water contents (2%, 4%, 6%, 8%, 10% and 12%) and the dry density of the sample was determined after oven drying at all these moisture contents. The graph was plotted between the moisture content and the dry density.

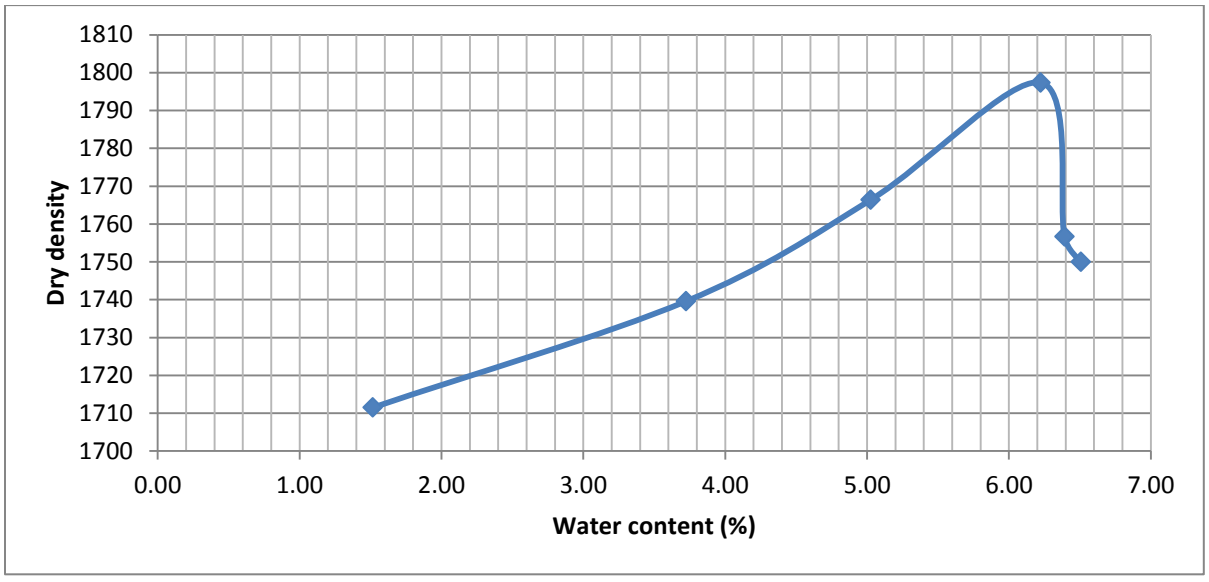


Figure 3-3: Standard proctor compaction curve

From the graph,

Maximum dry density = 1797.36 Kg/m^3

Optimum Moisture Content (OMC) = 6.22 %

Hence, the aggregate can be compacted to a maximum dry density of 1797.36 Kg/m^3 at the water content of 6.22%.

Type 2 Glass material

The sample consists of less than 30% particles by mass retained on 19 mm sieve. Hence, method C of standard compaction test was selected.

Size of mould = 6 inches diameter

No. of layers compacted = 3

No. of blows per each layer = 56

Weight of rammer = 5.5 lbf

Height of fall of rammer = 12 inches

absorb enough water However, the larger particles present on type 2 cullet cannot rst water content ofand the water started to segregate at the bottom at fi2 .%Hence ,the test was discarded.

The sieve analysis was conducted on the compacted sample and the curve was compared with that of gradation curve before compaction. The objective of this was to determine how much of the particles are crushed into finer particles after the test.

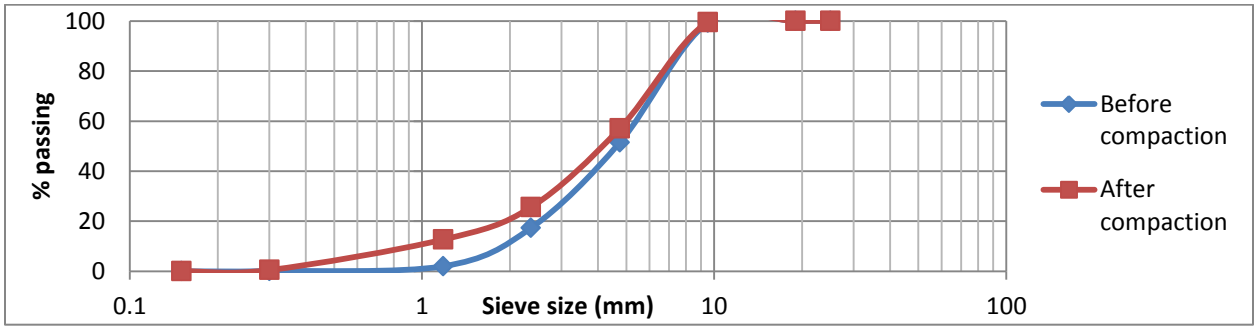


Figure 3-4: Gradation curve – Type 1, for compaction characteristics

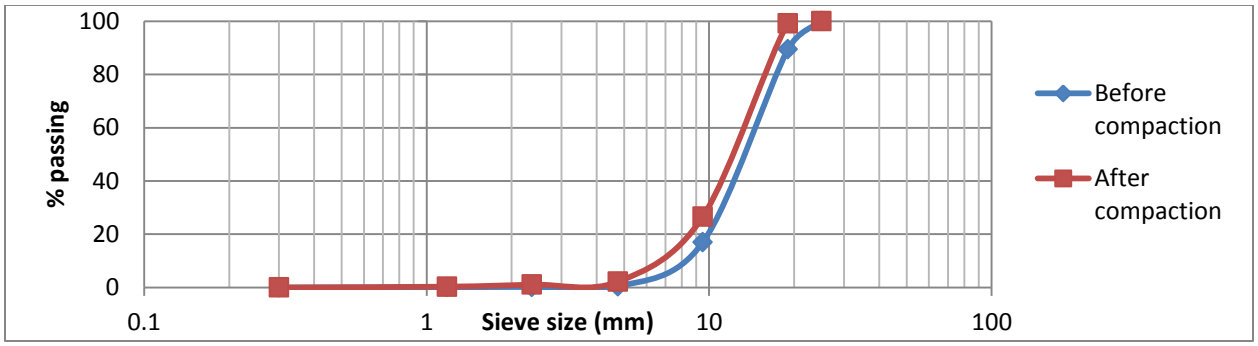


Figure 3-5: Gradation curve – Type 2, for compaction characteristics

The quantity of fine was increased after compaction but the classification of cullet after compaction still remained the same for both samples. This implies that the sample can sustain the amount of compaction force from standard proctor compaction with little crushing.

3.1.4 Permeability

The constant head permeability test method was adopted to determine the permeability of glass cullet following ASTM D2434.

We have,

Coefficient of permeability (K) = $\frac{QL}{Ath}$

Where,

Q = Volume of discharge in cc.

L = Height of sample in cm.

A = Cross-sectional area of sample in cm^2

t = time for discharge in seconds

h = hydraulic head difference across length L in cm of water = vertical distance between the constant funnel head level and the chamber overflow level.

Observations and calculations:

Three tests were performed at different hydraulic head by varying the position of the funnel. For each test, three sets of data for time required for the discharge of 1000cc are recorded.

Figure 3-6: Permeability test setup

Height of sample (L) = 172 mm = 17.2 cm

Diameter of sample (d) = 100 mm = 10 cm

So, A = $\frac{\pi d^2}{4}$ = $\frac{\pi * 10^2}{4}$ = 78.53 cm^2

Hence, K = (1000*17.2) / (78.53*t*h) = 219.02/ (t*h)

Table 3-3: Permeability test records

Test	Head (cm)	Time required (sec)	K (cm/sec)	K * 10 (cm/sec)
1	94.5	50.7	0.0457	4.6
		49.95	0.0464	
		50.5	0.0459	
2	89.5	50.05	0.0489	4.83
		50.6	0.0484	
		51.2	0.0478	
3	84.5	53.1	0.0488	4.9
		52.9	0.049	
		52.5	0.0494	

Hence,

Value of K = (K1+K2+K3)/3 = (4.6+4.83+4.90)/3 * 10^{-2} = 4.77* 10^{-2} cm/sec.

The obtained value of coefficient of permeability for type 1 was 4.77 *10^{-2} cm/sec .

3.1.5 Soundness

The resistance to the forces of weathering such as drying and wetting is soundness. Soundness of cullet was evaluated in accordance with ASTM C 88.

Procedure

- Immerse the samples in the prepared solution of sodium sulfate or magnesium sulfate for not less than 16 h nor more than 18 h in such a manner that the solution covers them to a depth of at least 1⁄2 in at the temperature of 21 +/- 1 °C.
- Allow the sample to drain for 15-20 mins after immersion and place it in drying oven at the temperature of 110+/- 5°C.

- Check the weight losses of test samples by removing and weighing them, without cooling, at intervals of 2 to 4 h.
- Repeat the process of alternate immersion and drying until the required number of cycles was obtained.
- After the completion of the final cycle and after the sample has been cooled, wash by circulating water at 43+/- 6 °C.

Observations and Test Results

Table 3-4: Soundness test for type 1 glass

Sieve Size (mm)	Grading of original sample	Weight of Test Fractions before Test (gm)	Percentage passing designated sieve after test	Weighted percentage loss
19	0			
9.5	0.21			
4.75	32.36	100	1.9	0.61
2.36	38.85	100	1.6	0.62
1.18	17.76	100	1.8	0.32
0.6	7.03	100	3.3	0.23
0.3	2.24			
0.15	1.2			
0.075	0.2			
Pan	0.15			
	100			1.79

Table 3-5: Soundness test for type 2

Sieve size (mm)	Wt of sample taken (gm)	Designated sieve size to determine loss (mm)	Weight passing designated sieve after test (gm)	Percentage passing designated sieve after test
19	500	16	5.8	1.16
12.5	670			
9.5	330	8	4	0.4
4.75	300	4	0.9	0.3

Sieve Size (mm)	Grading of original sample	Percentage passing designated sieve after test	Weighted percentage loss
19	7.49	1.16	0.09
9.5	78.58	0.4	0.31
4.75	13.11	0.3	0.04
			0.44

The calculated soundness values of cullet were 1.79 and 0.44 for type 1 and type 2 respectively.

The mean particle size (D_{50}) for type 1 and type 2 were 3.05 mm and 12.5 mm respectively. The result of the soundness test shows that smaller the particle size, larger will be the exposed surface area and more will be the loss.

3.1.6 Direct Shear Test

The direct shear test gives the measure of shear strength parameters of glass particles. The test was performed in Digi shear device (GEOTAC, Texas) at constant rate of shearing of 0.006 inch/min.

Table 3-6: Direct shear test results

Material	Compaction level	Void ratio	Normal stress (psi)	Shear stress (psi)	Angle of friction
Type I glass	Loose	0.67	10	10.997	47.35
			15	17.624	
			20	21.854	
	Dense	0.5	10	15.907	52.65
			15	22.649	
			20	29.01	
Graded Ottawa sand	Dense	0.51	10	8.15	36.5
			20	15.55	
	Loose	0.64	10	7.06	30.37
			20	12.92	
Graded Ottawa glass	Dense	0.75	10	7.97	40.2
			20	16.42	
	Loose	0.83	10	7.19	32.13
			20	13.47	

The dry sample was placed inside the shear box. The diameter of cylindrical sample inside the box was 6.35cm and the height was 2.85 cm. The shear box consists of two halves, the bottom half is fixed while top portion can move in lateral direction under application of shearing force. The lateral force was applied on the top half at constant rate of shearing after application of normal stress. The failure surface develops along the plane of movement of two halves.

3.2 Observations and Discussions

The measured engineering properties of glass particles were compared with those of aggregates/sand obtained from literature and online sources.

Table 3-7: Test results comparison

	Glass		Aggregate		
Test Name	Fine	Coarse	Fine	Coarse	Remarks
Gradation	Poorly Graded Sand (SP)	Poorly Graded Gravel (GP)	Poorly Graded Sand (SP)	Poorly Graded Gravel (GP)	
Specific Gravity	2.71		2.4 - 2.9		[32]
Sodium sulfate Soundness (% Loss)	1.79	0.44	0 to 12 (Most typical: 5 to 12)		[33]
Permeability (cm/sec)	4.77* 10-2	….	2.55*10-3 to 5.35*10-2	….	[34]

Direct Shear Test

Table 3-8: Test results comparison

	Friction angle (void ratio)				Remarks
Material	Glass		Aggregate/Sand		
Compaction Level	RG (Gradation : SP)	FRG	Aggregate	Ottawa sand	
Loose	47.35 (0.67)	32.13 (0.83)	30 to 39	30.37 (0.64)	[35]
Dense	52.65 (0.50)	40.2 (0.75)		36.5 (0.51)	

The specific gravity and permeability of the glass particles are in the range of the aggregates having same classification. However, the values of sodium sulfate soundness for glass particles are pretty low as compared to that of aggregates. It shows that the loss/disintegration of glass particles after alternate freezing and thawing cycle is less as compared to natural aggregates. Moreover, the glass particles are angular in shape and the angle of friction obtained for these are higher than for the natural sand or aggregates. These properties of glass cullets showed the potential of using them in place of or along with natural aggregates in local roadway construction. However, the issues related to the abrasive nature of the glass particles, lower skid resistance, propensity to strip off the surface course in presence of moisture, etc. should be taken into account.

Relation between material properties and erosion**:** [36]

The erosion of shoulder materials occur when the erosive forces surpass the resistance forces. The erosion potential by flowing water can be evaluated in terms of

rational correlation between energy dissipation of flowing water and erodibility index of the materials (K_h).

Table 3-9: Mass strength number for granular soil (Ms). (Annandale, 1995)

Consistency	Identification in Profile	SPT Blow Count	Mass Strength Number (M.)
Very loose	Crumbles very easily when scraped with geological pick	0-4	0.02
Loose	Small resistance to penetration by sharp end of geological pick	4-10	0.04
Medium dense	Considerable resistance to penetration by sharp end of geological pick	10-30	0.09
Dense	Very high resistance to penetration of sharp end of geological pick - requires many blows of pick for excavation	30-50	0.19
Very dense	High resistance to repeated blows of geological pick - requires power tools for excavation	50-80	0.41
Note: Granular materials in which the SPT blow count exceeds 80 to be taken as rock			

The rate of energy dissipation (P) is given as the function of erodibility index as;

$$P = f(K_h)$$

If $P > f(K_h)$, material is expected to erode and if $P < f(K_h)$, material is expected not to erode.

The index is given as the scalar product of indices of individual constituent parameters.

$$K_h = M_s K_b K_d J_s$$

Ms = mass strength number; refers to material shearing strength of intact representative sample [37].

K_b = particle/block size number; depends on mean grain size (D_{50}) for granular materials

$= 1000\ D_{50}^3$

For cohesionless granular material, Value of K_b can be less than 1.

Kd = discontinuity or shear strength of inter-particle bonds in granular soil ; depends on tan(ϕ), where ϕ is the equivalent residual friction angle [36], [38]

Js = relative ground structure number; it depends on shape of material unit that determines how easily stream can penetrate ground and dislocate individual particles.

The higher value of specific gravity and the results of compaction characteristics of glass cullets show glass as a pretty hard material. The results of direct shear test shows that glass cullets has higher friction angle than that of natural aggregates. So, for the same gradation of materials having same mean particle size, glass seems to possess more resistance to erosion. This has shown that the glass cullet has the potential to be used in the construction of shoulder section to resist erosion caused by flowing water. However, certain type of cementing agent is required to bind these glass particles together. To provide cementation, a technique called Enzyme Induced Carbonate Precipitation was applied on these glass aggregates.

4 WATER BASED EICP FOR GLASS/SAND STABILIZATION

EICP is a cementation technique in which a mixture of cementation solution and dissolved urease enzyme solution is mixed with sand. The cementation solution is the mixture of urea and $CaCl_2$ in deionized water. The urease enzyme catalyzes the precipitation of $CaCO_3$, which binds the soil particles together and fill the pores between them. [26]

4.1 Materials

4.1.1 Sand and Recycled Glass

The feasibility study of the application of EICP method for the stabilization of Ottawa sand and glass particles is carried out. The study was carried out on the pure Ottawa graded sand, Recycled glass particles, glass particles with gradation corresponding to that of Ottawa sand and the mixture of sand and glass particles. The particle size distributions of these materials are:

a) Ottawa graded sand (OS)

Particle diameter at 10% finer by mass (D_{10}) = 0.26 mm

Particle diameter at 50% finer by mass (D_{50}) = 0.33 mm

Coefficient of uniformity (C_u) = 1.35

Coefficient of curvature (C_c) = 0.9.

b) Recycled glass particles (RG) having particle size distribution as

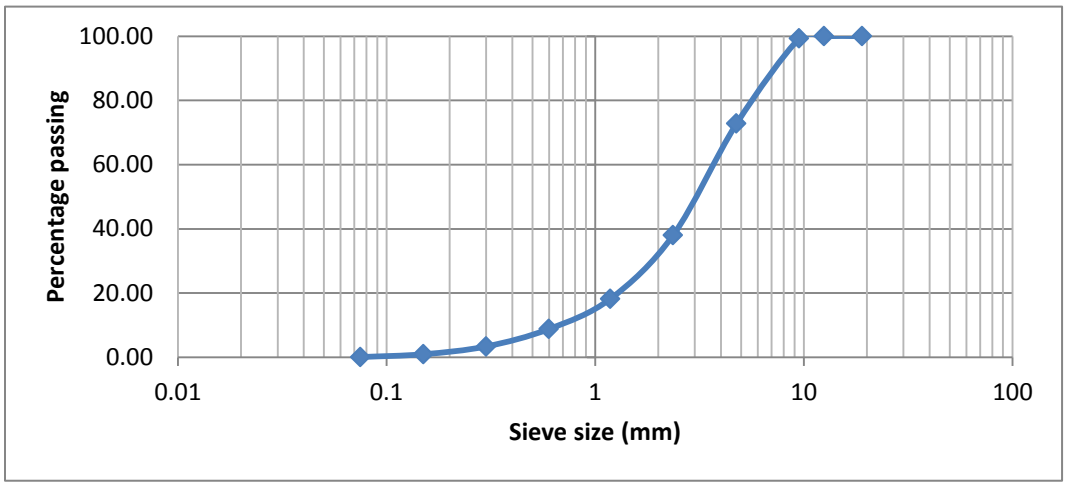


Figure 4-1: Particle size distribution for recycled glass

D_{10} = 0.63 mm, D_{50} = 3 mm, C_u = 5.87, C_c = 1.55

Classification (USCS): Poorly Graded sand (SP)

c) Fine glass particles with gradation corresponding to Ottawa graded sand (FRG).

4.1.2 Enzyme

Low activity urease enzyme obtained from Fisher Chemicals was used for the experiment. The enzyme was obtained in the powder form and was mixed thoroughly with water using magnetic stirrer. It catalyzes the hydrolyses of urea ($CO(NH_2)_2$)) into carbon dioxide(CO_2) and ammonia(NH_3) [39].

The conductivity probe was used to determine the activity of the urease enzyme. Urease activity was calculated from the slope of conductivity changes versus time under standard conditions of 1.5 M urea at 25^oC. The quantity of enzyme that catalyzes the formation of 1 μmole of ammonia per minute at the pH of 7 refers to one unit of urease [40].

Conversion of conductivity (mS) to urea hydrolysed (mM)

The slope of the conductivity change versus time was converted to the enzyme activity by multiplying with suitable conversion factor. The conversion factor was determined for the cases when enzyme was used with water. The conversion factor is the slope of the conductivity reading when the urea was completely hydrolyzed versus corresponding concentration of solution.

For this, the urea solutions of six different concentrations were prepared in different glass vials. The selected concentrations of urea were 0mM, 75mM, 150mM, 225mM, 300mM and 375 mM. The urease enzyme solution was introduced in these vials so as to obtain desired concentration of urea and 0.5gm/L of urease concentration in total volume. The solution was allowed to stand for sufficient time for complete hydrolyses of urea and the conductivity reading was taken. The conductivity readings were also taken at intermittent times to track the change with time.

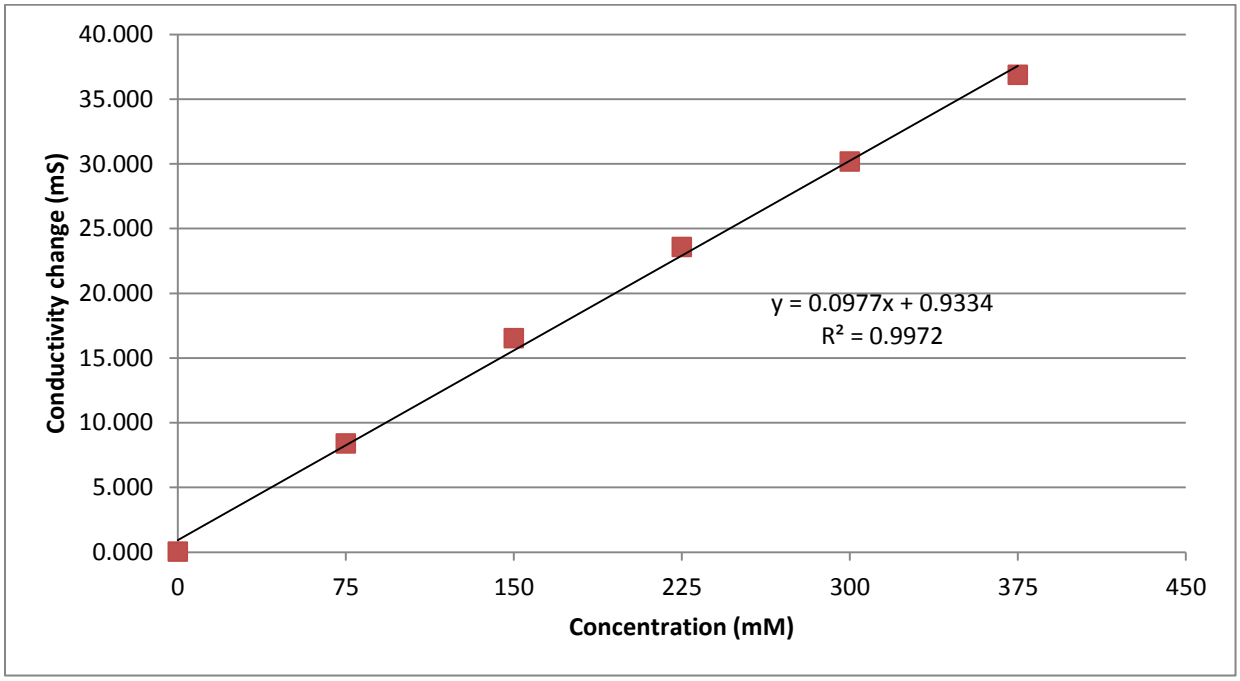


Figure 4-2: Conductivity change vs concentration for water based enzyme solution

The conversion factor was calculated based on the slope of conductivity change versus concentration as 10.24.

Conductivity as a measure of urease activity

The measurement of conductivity was performed for the solution in the glass vial. 5 ml of the urease solution was introduced in the vial containing 15 ml of urea solution thereby making overall concentration of urea and urease 1.5M and 0.5 gm/L respectively. The reading of conductivity was taken at times 0, 1, 2, 3, 4, 5, 6, 7 and 8 minutes. Zero minute reading refers to the point at which urease solution was introduced to the urea solution. The reading of these conductivity tests is plotted in a graph against time. The so obtained slope is converted to the activity by multiplying with suitable conversion factor.

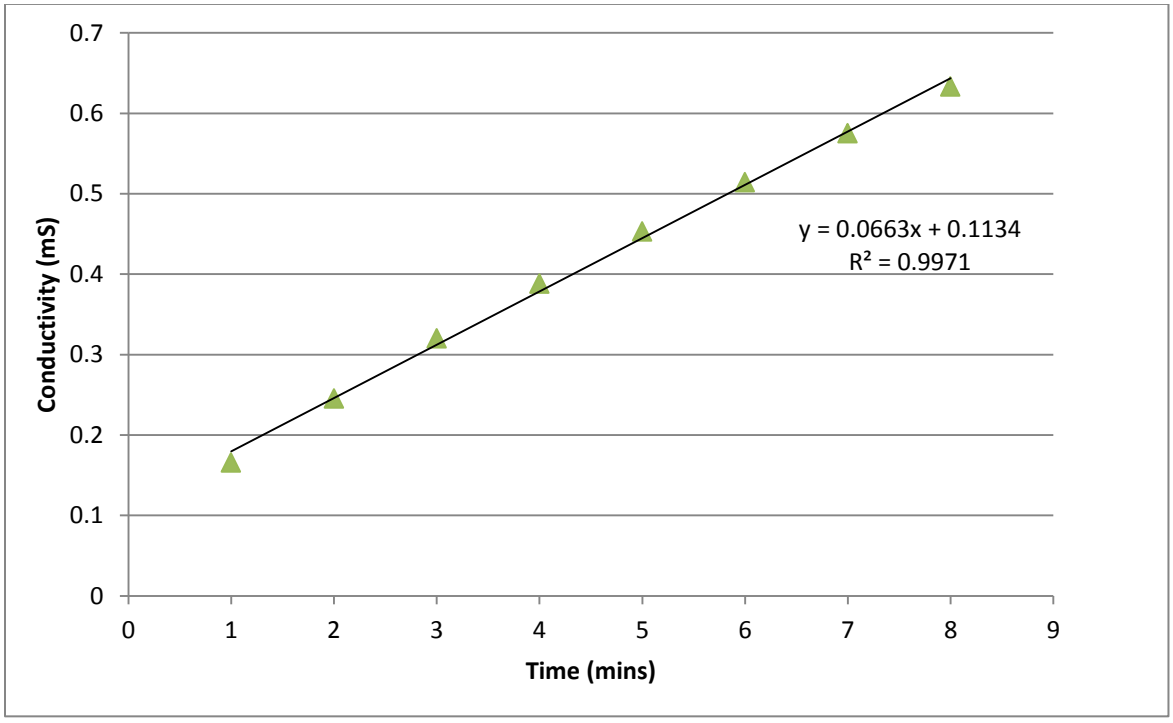


Figure 4-3: Conductivity vs time for water based enzyme solution

The slope of conductivity versus time was 66.28 as shown in graph. So, the activity and specific activity is calculated as;

Urease activity = 66.28 x 10.24 = 678.70 µM urea/min

Specific urease activity = 678.70 / 0.5 = 1357.41

4.1.3 Cementation Solution

The low activity (LA) urease enzyme is used for the tests. The cementation solution consists of mixture of urea and calcium chloride ($CaCl_2$) in deionized water. The ratio of Urea to $CaCl_2$ is maintained as 1.5:1 for all tests. The concentrations of the chemicals used are listed in table 4-1.

Table 4-1: concentration of chemicals in cementation solution

Chemical	Concentration (g/L)
Urea	60
$CaCl_2$	111
Urease Enzyme	5

4.2 Experimental Procedures

4.2.1 Specimen Preparation

The test is performed in a clear plastic cylinder. It is divided into two catagories based on method of sample preparation.

a) Method A

The dry material (glass/sand) is mixed with the urease enzyme prior to placing in the cylinder. The prepared cementation solution is then placed in the cylinder and the mixture of enzyme and material is pluviated into the cylinder in installments and compacted using a glass rod after each filling.

b) Method B

The cementation solution and the urease enzyme are poured in the cylinder and stirred thoroughly for uniform mixing. The dry material (glass/sand) is then immediately pluviated into the cylinder with gentle stirring by glass rod.

A series of specimens were prepared in 1 inch acrylic cylinders. The experiment was conducted on the pure OS, RG, FRG, mixture of OS and RG, and mixture of OS and FRG as mentioned in table 4-2 and the void ratio achieved for these samples were about 0.56, 0.52, 0.74, 0.57 and 0.57 respectively. The samples were prepared; top of column was covered with aluminum foil and allowed to remain undisturbed for 7 days. The samples were disassembled after 7 days and further tests on the samples were carried out.

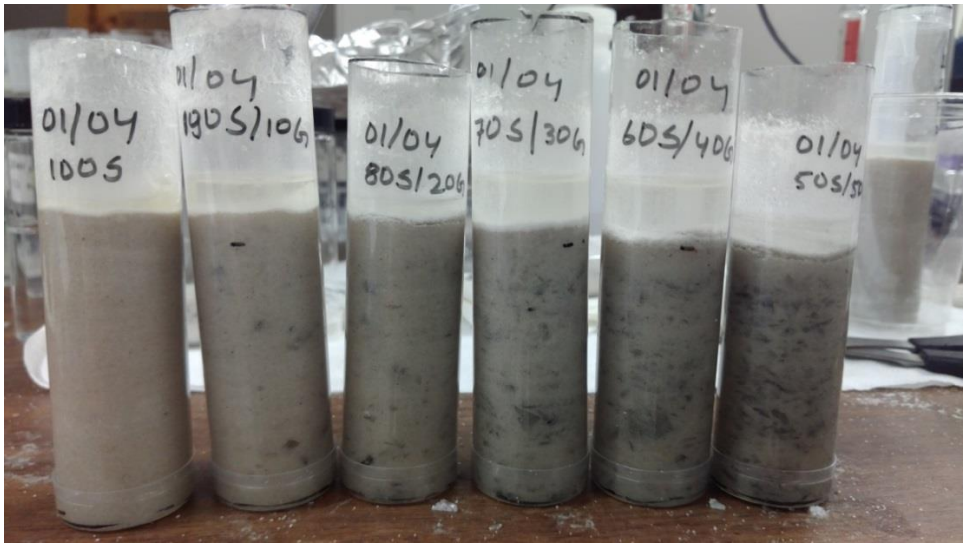


Figure 4-4: Sample preparation of EICP treated specimen

Table 4-2: Summary of Water based EICP test methods

Sample preparation Method	Sample		Chemicals	Concentration
Method A	i) Pure material	100% OS	Urea, $CaCl_2$, Urease(5 g/L)	1M Ca Urea:$CaCl_2$= 1.5:1
		100% RG		
		100% FRG		
	ii) Mixture of OS & RG	100% OS		
		90% OS + 10% RG		
		80% OS + 20% RG		
		70% OS + 30% RG		
		60% OS + 40% RG		
		50% OS + 50% RG		
Method B	i) Pure material	100% OS		
		100% RG		
		100% FRG		
	ii) Mixture of OS & RG	100% OS		
		90% OS + 10% RG		
		80% OS + 20% RG		
		70% OS + 30% RG		
		90% OS + 10% FRG		
		80% OS + 20% FRG		
		70% OS + 30% FRG		

4.2.2 $CaCO_3$ content determination

The $CaCO_3$ content was determined by acid digestion with HCl. The washed sample is dried in the oven at 105^oC for at least 24 hours to completely remove the moisture. The sample is then washed with excess of HCl to dissolve all the precipitate. The remaining sample after acid digestion is washed, drained and dried in the oven. The difference in dried weight of the sample before and after acid digestion is the weight of $CaCO_3$.

4.2.3 Unconfined Compression Tests

The unconfined compressive strength testing of the intact samples were performed in Sigma-1 Automated Load Test System (GEOTAC, Texas) under strain controlled conditions at a uniform loading rate of 1.0%/min. The specimens were trimmed at both ends so as to make height to diameter ratio between 2 and 2.5.

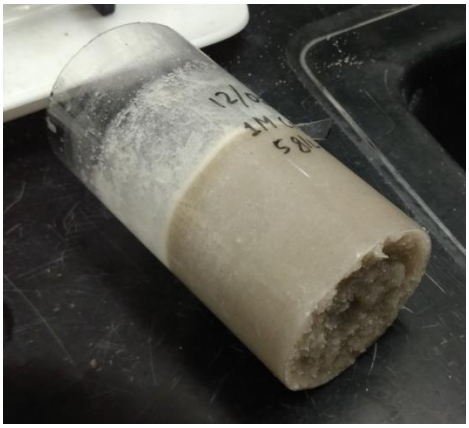
4.3 Observations and Results

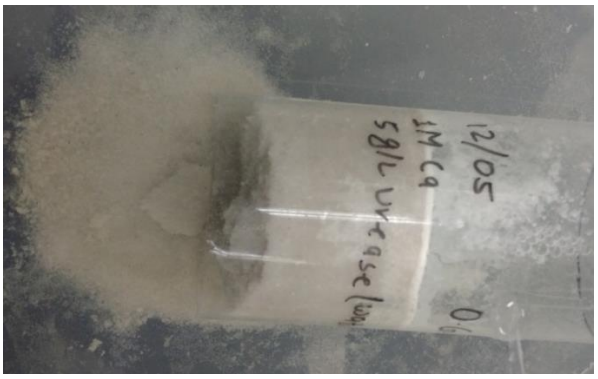
4.3.1 Observations of Cylindrical Sand Specimens

i) Method A

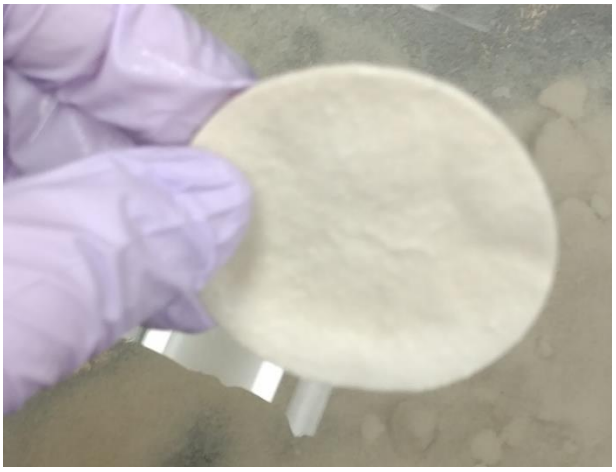
a) Pure sand or Pure Glass

A layer of white precipitation was observed on the top surface of all samples. The Ottawa graded sand sample was hard enough when felt with fingers from outside along the circumference and top. However, the RG and FRG samples were not stiff enough when felt even after a month.





(a) **(b)**



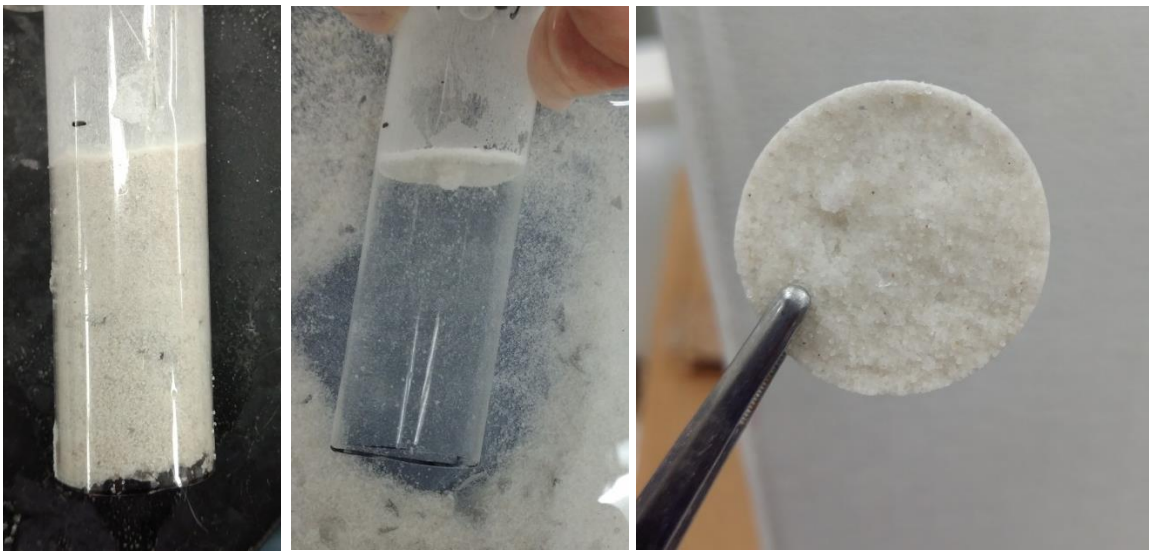
(c) **(d)**

Figure 4-5: Dis-assembling of Ottawa graded sand sample after 7 days.

The bottom cap of the column was removed (fig. 4-5 a) and the sample were washed gently with tap water in a container (fig. 4-5 b). The particles started to erode slowly from the bottom when placed in the container leaving only some chunks as shown in figure 4-5 (d). These chunks does not deform when placed in the water for 24 hours. The formation of these chunks represents the precipitation of calcite that binds the particles together. Moreover, a thin layer of calcite precipitation was formed on the top surface of the sample. The deformed soil particles were also tested with hydrochloric acid

for the presence of calcite. Small quantities of calcite were observed at different locations. But, this was not sufficient to bind the whole sample as a block.

b) Mixture of Ottawa sand (OS) and Recycled Glass (RG) at different proportions



(a) **(b)** **(c)**

Figure 4-6: (a): 80% OS + 20 % RG sample after 10 days; (b): washing sample gently with water; (c): top layer that does not dissolve in water and remained intact**.**

The top portion of sample was felt stiff when squeezed with fingers from outside whereas the lower portion was relatively less stiff. The bottom cap was removed and the sample was washed with tap water in a container gently. The particles lost their binding and flowed except the top layer, which remained intact. The top layer was subjected to acid digestion to ensure the presence of calcium carbonate. 1M HCl was introduced which caused effervesce, indicating the presence of carbonate.

ii) Method B

The samples were dis-assembled after 7 days and washed gently with the water. The smell of NH_3 as observed while opening the samples are tabulated below.

Table 4-3: Qualitative measures and general observations of the samples. The odor strength of NH_3 was assessed as follows: ++ : Strong, + : Faint, -- : No smell

Sample	NH_3 odour	Sample intact?
100S	++	Yes
90S/10RG	++	Yes
80S/20RG	++	Yes
70S/30RG	+	Partially, some chunks
90S/10FRG	+	Partially, some chunks
80S/20FRG	+	No
70S/30FRG	--	No

- 100S, 90S/10RG, 80S/20RG: samples remained intact after dis-assembling.
- 70S/30RG, 90S/10FRG: Samples felt partially stiff before dis-assembling. However, they broke down into small chunks while opening the column.
- 80S/20FRG, 70S/30FRG: samples felt loose and particles lost binding while opening. No chunks were formed except a white disc at the top, which is presumed to be $CaCO_3$.

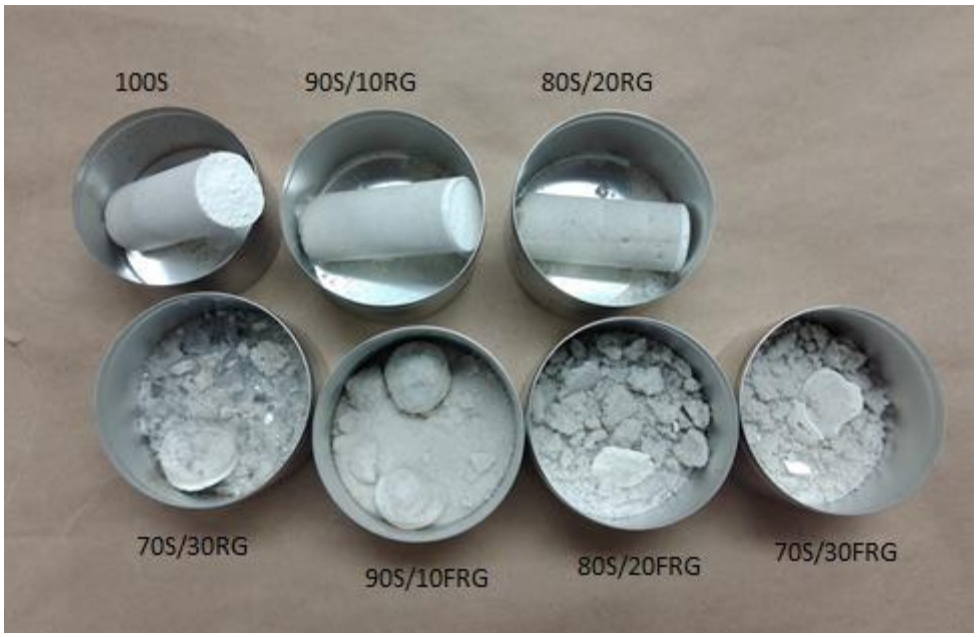


Figure 4-7: Disassembled samples after 7 days

- Two more replicates of each intact sample 100S, 90S/10RG, 80S/20RG were prepared. $CaCO_3$ content and Unconfined Compressive Strength (UCS) of these samples were determined.

4.3.2 $CaCO_3$ Content Measurement

The $CaCO_3$ content was determined by acid digestion with HCl. The washed sample is dried in the oven at 105^{o}C for at least 24 hours to completely remove the moisture. The sample is then washed with excess of HCl to dissolve all the precipitate. The remaining sample after acid digestion is washed, drained and dried in the oven. The difference in dried weight of the sample before and after acid digestion is the weight of $CaCO_3$.

The result for the $CaCO_3$ content on different samples shows that the precipitated amount of $CaCO_3$ was close enough. The same amount of recipe was used for all the

samples. This suggests that the amount of precipitated $CaCO_3$ should be same for all. The decrease in amount of observed $CaCO_3$ content was due to the loss of loose amorphous $CaCO_3$, during washing the sample.

Table 4-4: Measurement of $CaCO_3$ content

Sample	% of $CaCO_3$ as total weight	
	Method A	Method B
100S	3.04	2.9
90S/10RG	3.04	2.67
80S/20RG	1.94	2.62

4.3.3 Unconfined Compressive Strength (UCS) Test

Table 4-5: Unconfined compressive strength testing results

Sample	Stress at failure (psi)	Strain at failure (%)	Average stress (psi)
100S	190.38	1.92	192.41
	194.45	1.2	
90S/10RG	78.65	1.1	81.31
	80.38	0.22	
	84.9	0.69	
80S/20RG	63.68	1.2	63.94
	52.08	0.45	
	76.06	0.62	

The samples prepared with method B of columnar method were able to form the intact sample. Hence, 100S, 90S/10RG and 80S/20RG were subjected to UCS testing. The unconfined compressive strength testing of the intact samples were performed in Sigma-1 Automated Load Test System (GEOTAC, Texas) under strain controlled conditions at a uniform loading rate of 1.0%/min. The height to diameter ratio of all the samples was between 2 and 2.5.

4.4 Discussions

The urease enzyme is effective in formation of carbonate through hydrolysis of urea. For the EICP to occur effectively, the sample preparation method and test setup plays an important role. None of the methods employed were able to stabilize pure RG and FRG samples. However, method B was able to bind the particles as an intact sample for pure OS. The samples were able to stand on their own when the recycled glass was mixed up to 20% with the Ottawa sand.

The method A failed to bind the particles to form an intact sample for all the cases. A thick layer of carbonate precipitate was formed at the top but the precipitation in the void space was not enough to bind the particles. This could be due to lack of uniform distribution of enzyme, urea and $CaCl_2$ throughout the sample. The sample was mixed with the enzyme solution before pouring in the column. While pouring, the enzyme was washed by the cementation solution present in the column resulting in settling of sand at the bottom and enzyme at the top. However, for method B, the samples were able to stand on their own when the percentage of recycled glass was increased upto 20%. The enzyme solution was mixed with cementation solution before the sample was pluviated.

This caused the chemicals to distribute uniformly and as a result intact sample was formed.

The experiments performed showed that EICP worked well for the Ottawa sand but it did not work well for the glass particles. Method B was found successful on pure Ottawa sand. However, it did not work well for glass material. Also, for the mixture of sand and glass particles, this method was able to bind the particles as an intact sample when recycled glass (RG) was added to OS up to 20%. The identified reasons for EICP not being effective on glass are:

i) Surface characteristics of glass

The glass particles have smooth surface. It was found that the adhesion between carbonate precipitate and glass surface was not good enough. For this, a test was conducted on two glass plates. One glass plate has smooth surface while for the second one, the surface was roughened with sandpaper. The glass plates were kept in a container and the solution of urea, $CaCl_2$ and enzyme was introduced so as to immerse the plates. $CaCO_3$ precipitate was allowed to form on the surface for 3 days and the dishes were taken out. The dishes were allowed to dry in air for two days.

Figure 4-8: Sample preparation for pull out test

The pull out test was carried to determine the strength of bonding. The dolly is attached to the precipitate with a layer of resin and is allowed to cure for 48 hours. The pull out test was carried out on both the samples. For smooth surface, the failure of the bonding occurred at 51PSI. The failure was between the carbonate precipitate and the glass surface. However, for rough surface, the failure occurred within the calcite at 119PSI. This shows that the bond strength between glass surface and calcite precipitate is improved after surface treatment.

Figure 4-9: Pull out testing device to determine bond strength (Image source:[41])

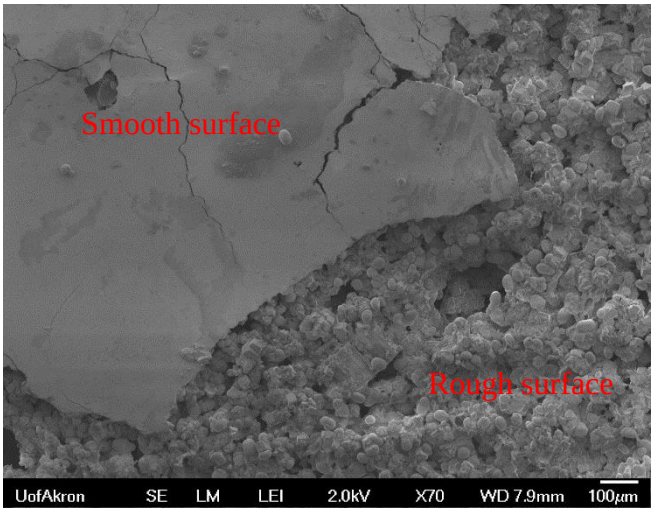


Figure 4-10: SEM image of $CaCO_3$ precipitate deposited on smooth and rough surfaces of glass

ii) Shape of the particle

The Ottawa sand and glass particles are observed under Scanning Electron Microscope (SEM). The images observed show that the sand particles are more rounded while those of glass particles are angular and platy.

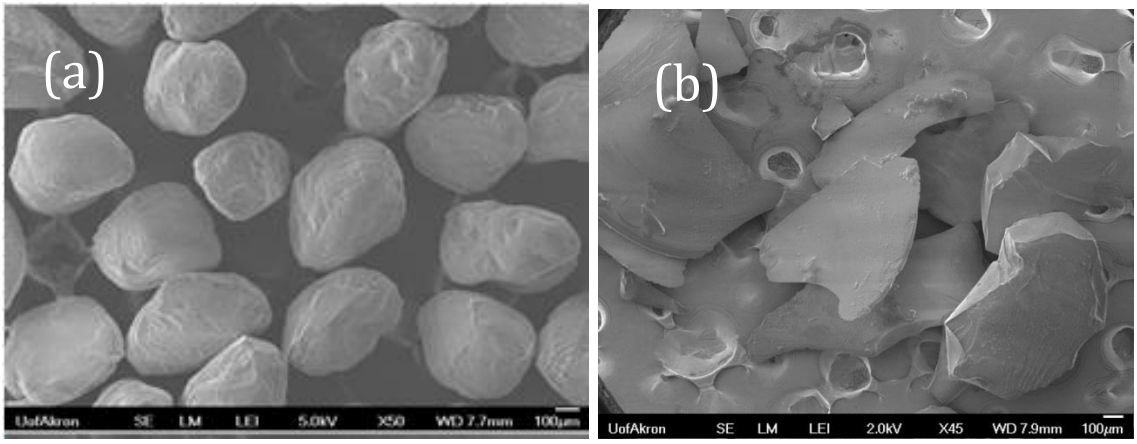


Figure 4-11: SEM images of Ottawa graded sand (a) and Fine Recycled Glass (b)

To further explain the effect of angularity, the natural gravel was crushed to the gradation of Ottawa sand and EICP was performed. The crushed gravel particles were angular in shape. It was found that the sample was not intact after a week. It shows that the precipitate was not enough to bind the particles together. The amount of $CaCO_3$ content was determined on the recycled glass sample and was found to be 1%, which is low compared to that of intact Ottawa sand (3%). This is due to the amorphous nature of the precipitate on the glass particles. The carbonate cannot form solid crystal and is washed away during washing of the sample with water.

The porosity of the sample increases with increase in angularity of the grain. The highest porosity samples are obtained with angular platy and needle like particles.

Permeability being the function of angularity, it increases as the shape of the particles depart from that of true sphere [42]. The mean coordination number of the particle decreases with increase in porosity[43]. The glass particles being angular and platy, have more voids in the sample. Thus, more amount of carbonate precipitate is required to bridge the particles together.

Water based EICP method was successful in forming intact sample for the specimens containing up to 20% of recycled glass aggregates, which shows the potential of using them on shoulder section. However, the samples treated with this method are brittle in nature. The UCS results revealed that the samples failed at lower strain level although the stress at failure was large for the treated samples. Moreover, Water based EICP will drain out rapidly when applied on the shoulder section due to low viscosity of water. So, it requires a number of percolation/injection steps to ensure the precipitation of sufficient amount of carbonate. The application of high viscosity solution as the carrier of cementation solution instead of water could address these problems.

5 POLYMER MODIFIED EICP FOR SAND STABILIZATION

Most existing EICP studies involve natural water as the carrier for the cementation solution. The low viscosity of water leads to faster and non-uniform infiltration of cementation solution in the subsurface region, which is one of the reasons why conventional EICP requires a number of percolation/injection steps to ensure the precipitation of sufficient amount of calcite. This limits the implementation of water based EICP method as a one-shot treatment [6]. This difficulty seems to be addressed by the use of proper hydrophilic polymer instead of deionized water as a carrier of enzyme and cementation media. PVA has high viscosity and provides appropriate reaction time and environment for the hydrolysis of urea. This chapter presents the results of implementation of hydrogel-based EICP for stabilization of Ottawa sand.

5.1 Materials

5.1.1 Sand

Ottawa graded sand was used for the experiment. The details are given in chapter IV.

5.1.2 Enzyme

Low activity urease enzyme obtained from Fisher Chemicals was used for the experiment. The enzyme was obtained in the powder form and was mixed thoroughly with PVA solution using magnetic stirrer. The concentration of enzyme used in the overall solution was 5 gm/L.

Conductivity probe was used to determine the specific activity of the enzyme in PVA solution. The detail of the procedure is described in chapter 4.

Conversion of conductivity (mS) to urea hydrolysed (mM)

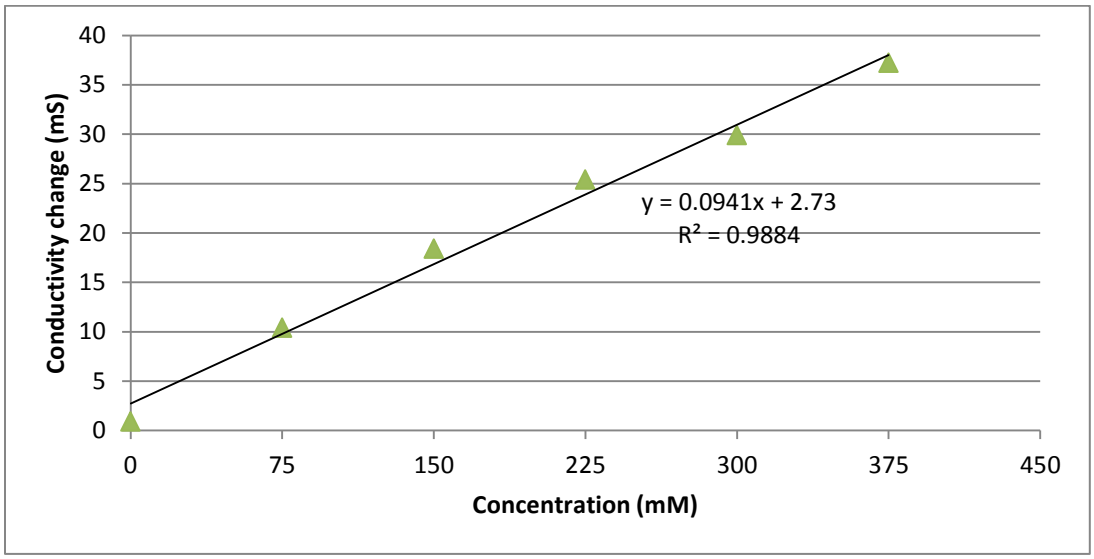


Figure 5-1: Conductivity change vs concentration for PVA based enzyme solution

The conversion factor obtained based on the slope of conductivity change versus concentration was 10.63

Conductivity as a measure of urease activity

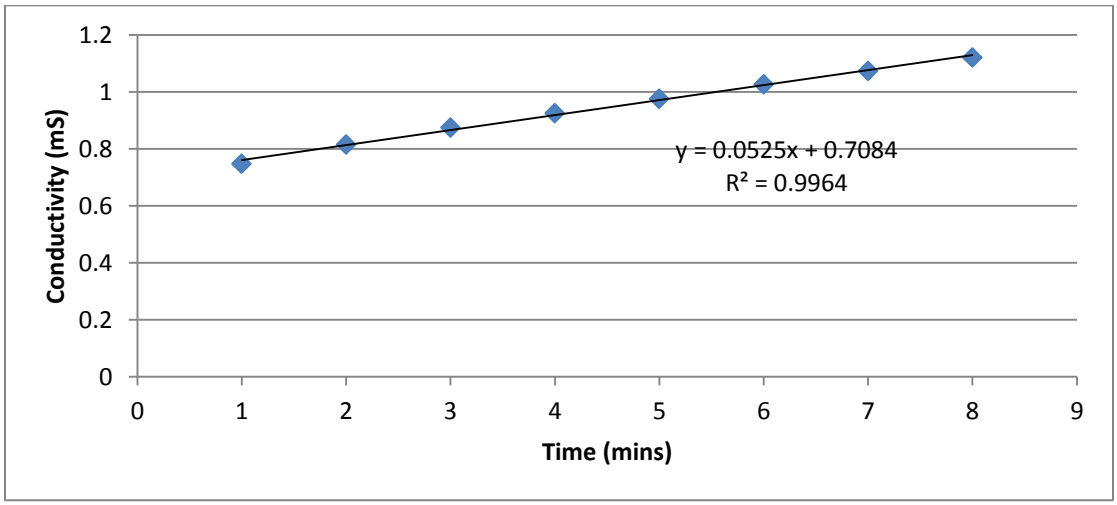


Figure 5-2: Conductivity vs time for PVA based enzyme solution

The slope of conductivity versus time was 52.47 as shown in graph. So, the activity and specific activity is calculated as;

Urease activity = 52.47 x 10.63 = 557.82 µM urea/min

Specific urease activity = 557.82 / 0.5 = 1115.64

5.1.3 Cementation Solution

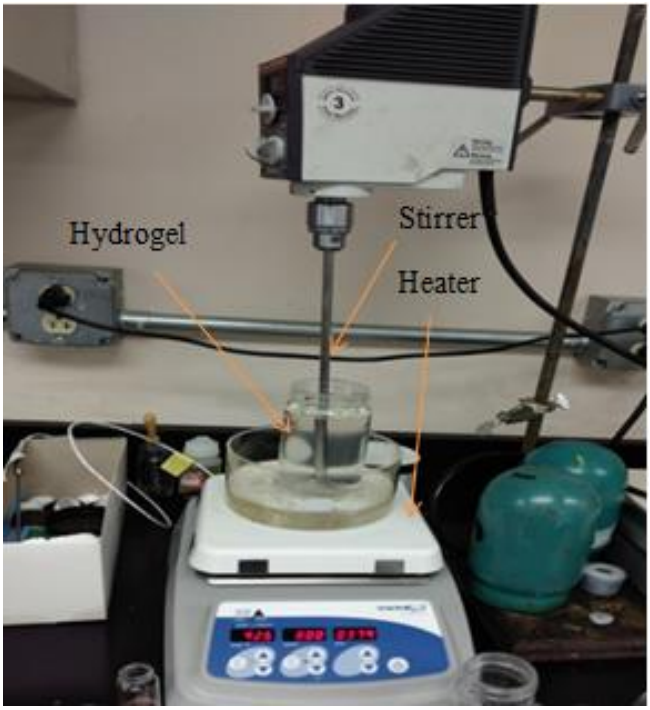


Figure 5-3: Preparation of PVA hydrogel cementation solution

The cementation solution consists of urea, $CaCl_2$ and PVA in deionized water. PVA is used because it has high viscosity and provides appropriate reaction time and environment for the hydrolysis of urea. 86-89 % hydrolyzed, Medium molecular weight PVA powder from Alfa Aesar was used and the concentration of PVA in the cementation solution was 7.5% weight per weight (w/w). A solution of urea and calcium chloride is prepared before adding PVA. The PVA hydrogel cementation solution is then prepared

by adding PVA powder in small installments to the previously prepared urea, $CaCl_2$ deionized water solution under high speed stirring at 60 ºC for 6 hours. The concentrations of chemicals used are as listed in table 4-1.

5.2 Experimental Procedures

5.2.1 Specimen Preparation

(a) (b)

Figure 5-4: (a) cylindrical clear plastic container; (b) Preparation of cylindrical specimen

The EICP treatment of sand specimen was conducted in clear plastic cylindrical container having internal diameter 51mm. The cementation solution and the enzyme solution are poured into the cylinder and stirred for about 30 seconds for uniform mixing.

Air dried sand/glass was then pluviated into the container in five stages while slightly stirring the mixture with glass rod. The entire container was vibrated gently so as to make upper level of solution slightly higher than the upper surface of the sand/glass column. This was done to ensure the sand/glass column was fully saturated. The prepared sand column has the void ratio of about 60%.

A total of three specimens were prepared for the testing. The containers were covered with the aluminum foil at the top and kept undisturbed for 2 months. The specimens were removed from the containers and immersed in the separate beakers filled with water. The first sample was taken out from the water at 3 days, second sample at 6 days and allowed to dry in air at room temperature. The third sample was left submerged in the water for 9 days. Further tests on the sample were performed at 9^{th} day.

5.2.2 Unconfined Compressive Strength (UCS) Tests

The sand specimens were trimmed at both ends to make aspect (diameter to height) ratio of 1:2. Sigma-1 Automated Load Test System (GEOTAC, Texas) was used to perform unconfined compression test. The uniform loading rate of 1.0%/min was applied under strain controlled conditions in accordance with ASTM D2166 (“Standard Test Method for Unconfined Compressive Strength of Cohesive Soil”) (ASTM, 1991).

5.2.3 Thermogravimetry Analysis and $CaCO_3$ content measurement

Thermogravimetry Analysis (TGA) was carried out to measure the change in the weight of material as a function of increasing temperature. A small portion of sample weighing about 15mg was picked with tweezer from three different locations of each sample for the test.

$CaCO_3$ content on the sample was determined from the results of TGA. $CaCO_3$ content is expressed as the percentage of loss in weight of carbonate to the total weight of sample taken for analysis over a specified range of temperature.

5.2.4 Microstructure Analysis

Scanning Electron Microscopy (SEM) and powder x-ray diffractometry (XRD) were used to examine the morphology and distribution of the calcium carbonate in the treated sand sample. For the SEM test, small amounts of treated sample consisting of approximately 50 particles were selected from different locations. The samples were mounted on the specimen holder and subjected to conductive coating. The prepared samples were then observed under the SEM. For the XRD test, the small portion of air dried sample was crushed to separate the individual particles and subjected to testing.

5.3 Observations and Results

The two immersed samples were taken out of the water at 3 days (#1) and 6 days (#2) and allowed to dry in air at room temperature. The third sample (#3) was taken out of the water at 9^{th} day. The samples become stiffer when allowed to dry. All the samples were subjected to unconfined compression test at 9^{th} day.

5.3.1 Unconfined Compressive Strength (UCS) Tests

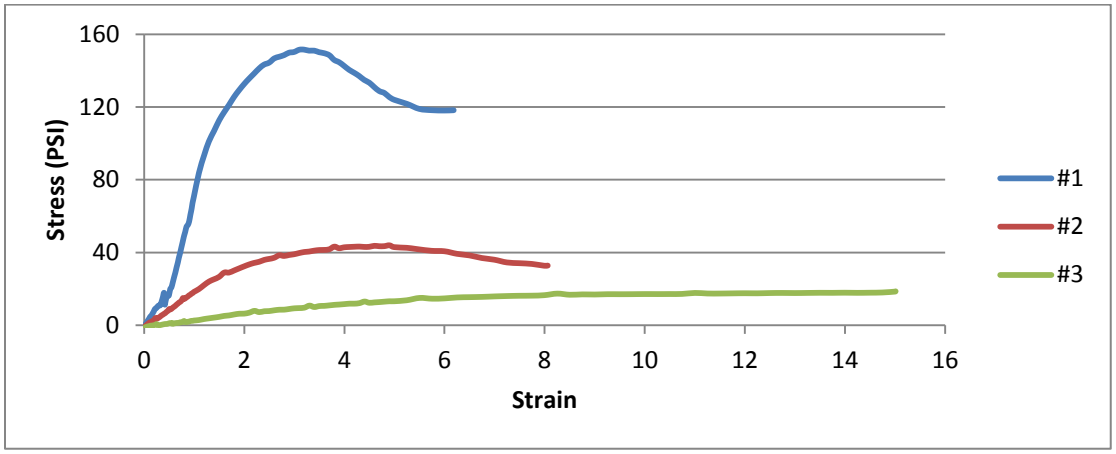
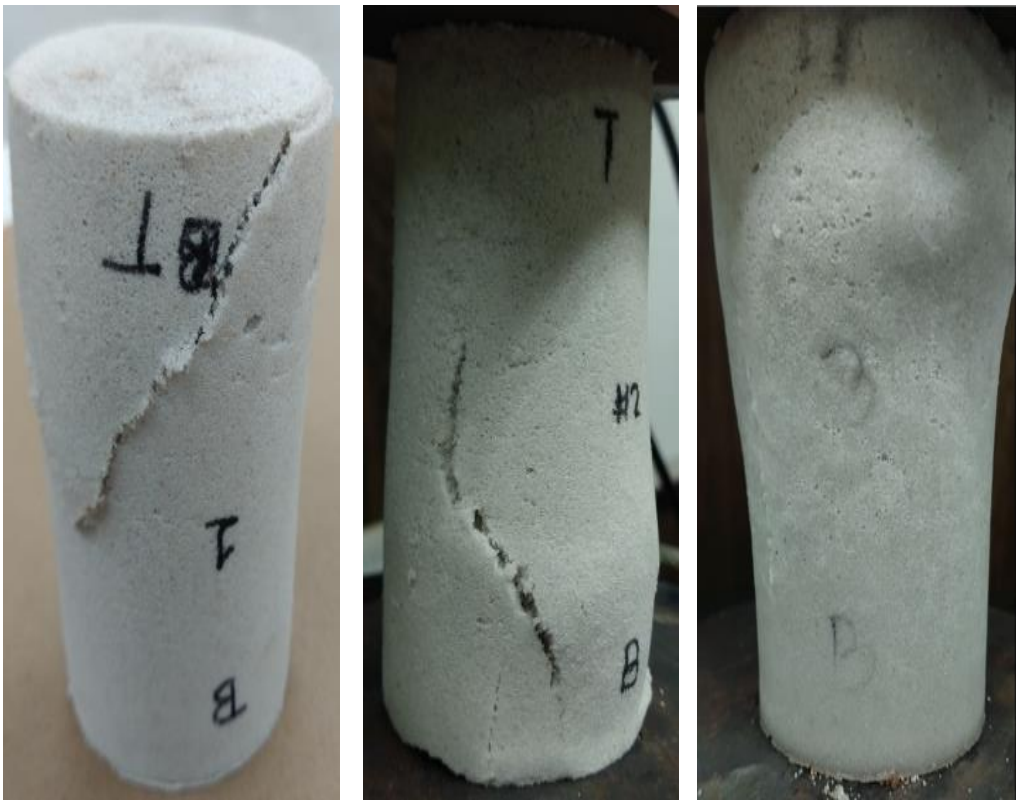


Figure 5-5: Unconfined compression test results

The sand specimens were trimmed at both ends to make aspect (diameter to height) ratio of 1:2. The top and bottom surface of the samples were made flat and smooth before testing to ensure the uniform distribution of pressure on both surfaces. The specimen #1 was stiffer and showed a distinct failure plane when subjected to UCS testing. For sample #2, the sample failed by bulging at the bottom initially followed by the development of failure plane as shown in figure 5-6 (#3). The ductility of the specimen increased with increase in moisture content. For sample #3, the sample kept on bulging when the strain was applied up to 15% but did not develop a distinct failure plane. On removal of stress, the sample almost returned to its original shape within few minutes.



#1 #2 #3

Figure 5-6: Failed samples after UCS testing

The water content of samples #1, #2 and #3 were determined to be 0.4%, 4.4% and 20.4% respectively and the corresponding degree of saturation as 1.9%, 20.8% and 96.5%. The experimental results show that the stiffness of the treated sample depends on the moisture content.

Table 5-1: UCS results with degree of saturation

Sample	Peak stress (PSI)	Strain (%)	Degree of saturation (%)
#1	151.54	3.19	1.9
#2	44.0	4.9	20.8
#3	No definite peak		96.5

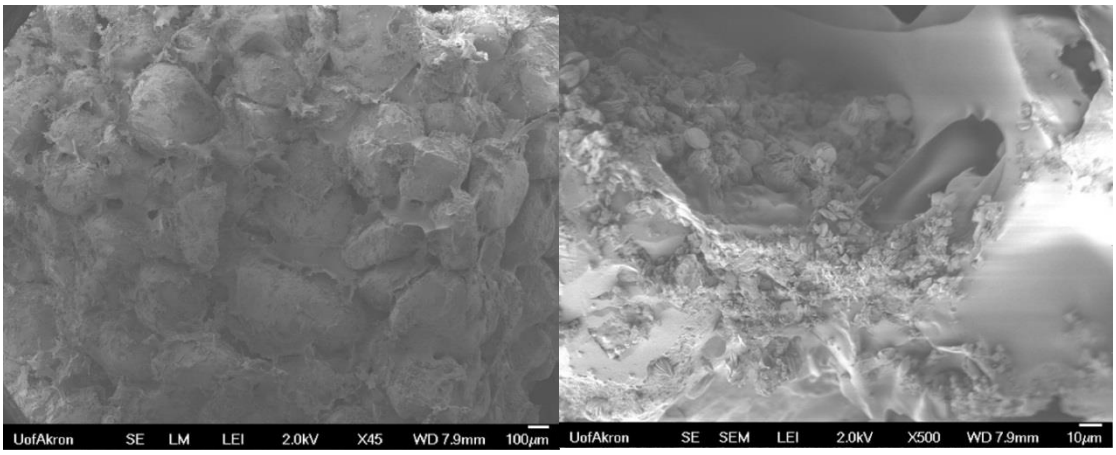
5.3.2 Thermogravimetry Analysis and $CaCO_3$ content measurement

TGA was carried out to measure the change in the weight of material as a function of increasing temperature. A small portion of sample weighing about 15mg was picked with tweezer from three different locations of each sample for the test.

$CaCO_3$ content on the sample was determined from the results of TGA. The loss in weight of $CaCO_3$ is calculated from the results of TGA and expressed as percentage of total mass of material used for the analysis. The $CaCO_3$ content for samples #1, #2 and #3 was found to be 0.95%, 0.97% and 1.17%. The maximum amount of $CaCO_3$ precipitate that can form on the sample was 2.52%. This value was calculated from the amount of Ca^{2+} ions present in the cementation solution. This lower amount of $CaCO_3$ precipitate observed from the TGA results could be due to: (i) a layer of $CaCO_3$ precipitate was formed on the top surface of the sample, which constitute of certain amount, and (ii) a very small amount of sample was used for the TGA, which may not be representative of the whole sample.

5.3.3 Microstructure Analysis

Scanning Electron Microscopy (SEM) and powder x-ray diffractometry (XRD) were used to examine the morphology and distribution of the calcium carbonate in the treated sand sample. For the SEM test, small amounts of treated sample consisting of approximately 50 particles were selected from different locations. The samples were mounted on the specimen holder and subjected to conductive coating. The prepared samples were then observed under the SEM. For the XRD test, the small portion of air dried sample was crushed to separate the individual particles and subjected to testing.



(a) (b)

Figure 5-7: SEM images of the MMW PVA based EICP treated sand sample at resolution (a) x45 (b) x500

The experiments performed demonstrate EICP can occur in PVA solution. SEM images of the sample give the direct evidence of presence of $CaCO_3$ precipitation. The images reveal that the PVA forms a matrix with precipitated calcium carbonate crystals in the pore spaces (figure 5-7). This matrix did not dissolve in the water at room temperature. PVA forms theta solutions over a range of temperatures with water including room temperature. The PVA tends to adsorb on all solid surfaces from the theta

solution and has the ability to hydrogen bond. The hydrogen bonding between PVA and calcium carbonate increases the number of segments that are attached to the solid and forms high density of polymers near the interface. [44] This matrix forms the bond between sand particles and provides treated sand with reinforced strength.

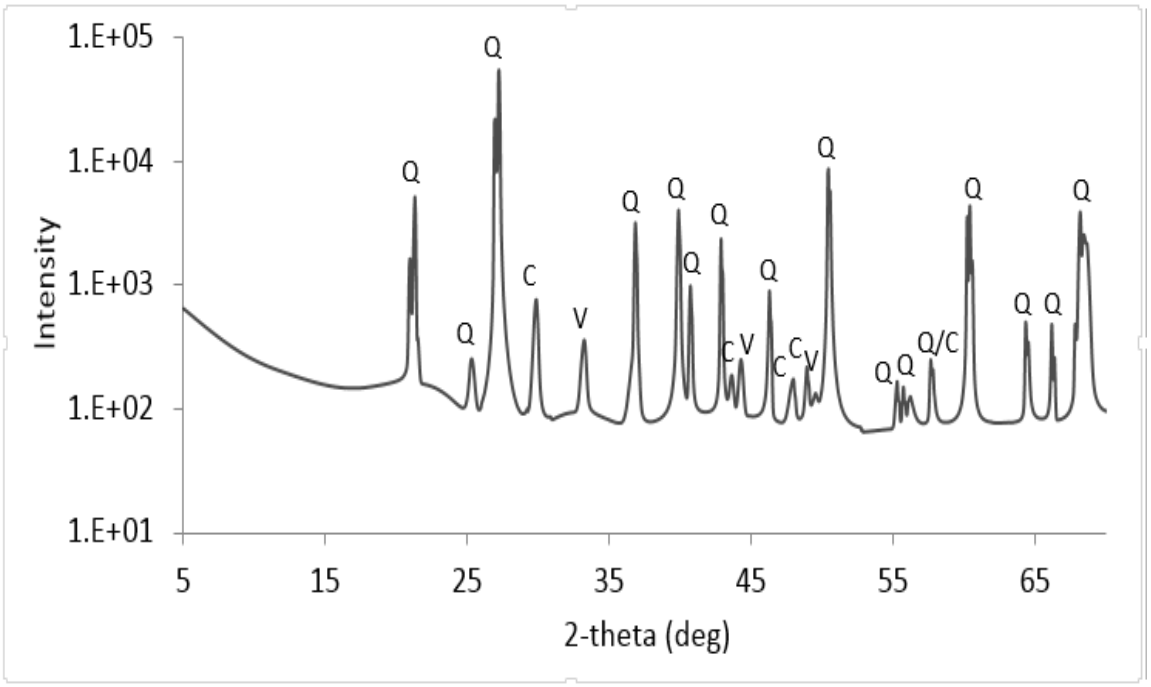


Figure 5-8: XRD analysis results for samples treated with MMW PVA based EICP approach. (Q= Quartz, C = Calcite & V = Vaterite)

XRD analysis was carried out to identify the polymorph of the precipitated $CaCO_3$ and the result of the analysis is shown in figure 5-8. Calcite and vaterite crystals were identified as shown in the figure. The first crystal to form is vaterite, which is metastable and transforms to calcite by dissolution and recrystallization. When enough PVA is used, it inhibits the formation of calcite from vaterite. [44] However, the formation of substantial amount of calcite and vaterite may be because of large number of nuclei/crystals that PVA should adsorb onto. It is also possible that calcite has formed very soon without going through the vaterite.

5.4 Discussions

Enzyme induced carbonate precipitation (EICP) showed early promise as a viable and sustainable ground improvement method. While previous studies proved that EICP significantly improves the strength of sands, the treated material is usually brittle. Inspired by natural tough materials like nacre which are organic-inorganic composites, this study aims to characterize the properties of sand treated by a polymer modified EICP process. The experiments carried out on the Ottawa graded sand showed the evidence that MMW PVA based EICP approach as efficient one-shot treatment scheme. It was found that the existence of PVA increases the viscosity of the solution and slightly reduces the specific enzyme activity. The high viscosity of PVA provides appropriate reaction time and environment for the hydrolysis of urea. The treated sand columns are organic-inorganic composites with sand cemented by a $CaCO_3$-PVA mixture. Unlike low molecular weight PVA, MMW PVA forms complex matrix with the $CaCO_3$ precipitate which does not dissolve in water at room temperature. The unconfined compression tests revealed that the strength and ductility of the soil columns treated with MMV PVA are moisture sensitive: the strength decreases but ductility increases with moisture content. More studies are needed to design tunable environment-responsive and resilient geomaterials.

6 SUMMARY, CONCLUSION AND FUTURE WORKS

6.1 Summary and Conclusion

In this study, feasibility study on the potential usage of recycled glass aggregates in the construction of shoulder section is carried out. The laboratory testing performed showed that the glass cullet has comparable properties to that of natural aggregates, which makes it possible to use in place of or along with natural aggregates. However, the issues related to the abrasive nature of glass, higher skid resistance and its propensity to strip off the surface course in presence of moisture when used as glassphalt leaves a serious concern. The literature study on the material properties and erosion further showed the possibility of using recycled glass aggregates on the shoulder section for erosion control. Enzyme Induced Carbonate Precipitation (EICP) technique was applied on these materials to provide cementation and bind the particles together when used on the shoulder section. The objective is to form a shoulder section incorporating recycled glass aggregates and the resulting section should have both strength and ductility,

Enzyme induced carbonate precipitation (EICP) showed early promise as a viable and sustainable ground improvement method. This method was applied on the recycled glass aggregates to check if it can stabilize them. The application of EICP on the pure glass cullet was not successful in binding the particles together. The smooth surface and angular shape of the glass particles were identified as the major factors behind this. However, EICP was successful on the samples containing mixture of glass particles and Ottawa sand. The samples treated with EICP, consisting upto 20% of recycled glass, were

brittle and strong. The results of UCS testing showed the decrease in compressive strength with increase in amount of recycled glass in the mixture.

The pull out test was carried out on the glass surfaces to check the bond strength of precipitated calcite with glass surface. Two types of glass surfaces were chosen; one has smooth surface and the other having rough surface. The test results showed that the bond strength was higher in rough surface than the smooth surface. The failure occurred between glass surface and the precipitate in smooth glass surface while the failure was within the calcite in case of rough glass surface. This implies that if surface treatment of glass cullets can be done before application of EICP, the better bonding could be achieved. This shows the possibility of application of EICP on the surface treated glass particles. However, more detailed study and research on this is still a need.

Moreover, the high permeability of water leads to faster and non-uniform infiltration of cementation solution in the subsurface region, thus limiting the amount of available reaction substances to produce $CaCO_3$ precipitate at desired locations. This limits the implementation of water based EICP method as a one-shot treatment [6]. This difficulty seems to be addressed by the use of proper hydrophilic polymer instead of deionized water as a carrier of enzyme and cementation media. PVA has high viscosity and provides appropriate reaction time and environment for the hydrolysis of urea. Medium molecular weight PVA was used in the preparation of cementation solution and the EICP was conducted on Ottawa sand. SEM, XRD and TGA results verify the presence of $CaCO_3$ and the strength of the samples were tested at different moisture contents. The treated sand columns are organic-inorganic composites with sand cemented by a $CaCO_3$-PVA mixture. Unlike low molecular weight PVA, MMW PVA forms

complex matrix with the $CaCO_3$ precipitate which does not dissolve in water at room temperature. The unconfined compression tests revealed that the strength and ductility of the soil columns treated with MMV PVA are moisture sensitive: the strength decreases but ductility increases with moisture content. These samples failed at higher strain level than the samples treated with water based EICP. This shows the ability of polymer modified EICP to form a sample having both strength and ductility. However, more studies are needed to design tunable environment-responsive and resilient geo-materials.

6.2 Future Works

- Perform the erodibility tests on the recycled glass aggregates to check the feasibility of using them in preventing surface erosion.
- Perform EICP on the surface treated recycled glass aggregates to check the strength.
- Perform polymer modified EICP on the samples involving recycled glass aggregates.
- More detailed study on polymer modified EICP to design tunable environment-responsive and resilient geo-materials.

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