SOIL STABILIZATION WITH GEOPOLYMERS FOR LOW COST AND ENVIRONMENTALLY FRIENDLY CONSTRUCTION

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ABSTRACT: In this paper, a geopolymer synthesized from olivine and an alkaline solution made from sodium hydroxide which incorporates the carbonation process was utilized to stabilize silty clay soil, in order to improve its expediency for different cases of construction. The effect of alkaline molarity, for alkali activation and carbonation processes, on the Unconfined Compression Strength (UCS) of the soil-olivine mixture is important in enhancing the treated soil properties. In this study, the use of 4, 6, 8, 10, and 12 mols of NaOH affected the UCS and toughness of the soil mixture. The highest strengths were obtained at 8 and 10 molarities for the carbonated alkali activation process, and 10 and 12 molarities for the alkali activation process only. Micro structural analysis using X-ray diffraction (XRD) and scanning electron microscopy (SEM) tests show the benefits of carbonation in terms of decreasing the molarity of alkaline agent. This was due to the crystallization processes which accounts for strength gain after carbonation. This paper highlights a more environmentally friendly procedure of stabilizing soils compared with the traditional binders such as cement and lime.

Keywords: Geopolymer, Olivine, Mineral carbonation, Soil stabilization, Unconfined compression strength

1. INTRODUCTION

Soil firmness is one of the widest range methods used for ground refinement. However, for many cases it is substantial to refine or improve the in-situ soils, with different types of binders, building different kinds of structures on top. Cement, lime and other additives are ordinarily used in construction owing to enhance soil strength, control shrinking and swelling, reduce settlement and reduce permeability. For soil stabilization, cement and lime are the most important and exceedingly used binders (Pourakbar et al., 2015a; Pakbaz and Alipour, 2012; Horpibulsuk et al., 2011). However, cement and lime produce expends fossil fuels which are the main cause in carbon dioxide (CO2) emissions to the atmosphere. However, cement and lime production has intense environmental influence, consuming large quantities of fossil fuels and being accountable for the emission exceeds than 5% of all the carbon dioxide worldwide (Provis & Deventer, 2014). New researches in ground amelioration have raised the need for utilizing sustainable materials (e.g. zeolite, reactive magnesia and olivine) or Industrial waste (e.g. ground-granulated blast furnace slag, palm oil fuel ash, cement kiln dust, rice husk ash, recycled glass foam, recycled concrete aggregate and crushed brick, calcium carbide residue and fly ash) for fractional or full surrogate of cement or lime as a result of the considerable environmental influences (Fasihnikoutalab et. al., 2017; Fasihnikoutalab et. al., 2016; Arulrajah et. al., 2015; Cai et. al., 2015; Pourakbar et. al., 2015; Disfani et. al., 2014; Yi et. al., 2013a; Du et. al., 2011; Jegandan et. al., 2010; Basha et. al., 2005).

Broadly, alkaline activation is chemical synthesis in which alumina-silicate materials and alkali or alkali earth materials, which are alkaline ions like potassium or sodium, or alkaline earth ions like calcium are reacted. It can be substantive as a polycondensation reaction, which contains the process of silica and alumina units connect with each other and engage the oxygen ions. At the molecular level, substances formed by the interaction between silica and alumina on the one hand and alkaline cations such as sodium or potassium share the characteristics of sharing their stiffness, strength and durability with natural rocks (Davidovits, 1991).

Governments around the world recognize a rising demand for reducing the (CO2) levels of the atmosphere. CO2 confinement by utilizing reactive metals has been exceedingly suggested by several researchers (Haug et. al., 2011; Andreani et. al., 2009; Prigiobbe et. al., 2009b; Chen et. al., 2006). Olivine, with chemical composition (Mg,Fe)2SiO4, was considered by many researchers as an important candidate in the sequestration of (CO2) and as a participant in alkali activation (Fasihnikoutalab et. al., 2017; Olaire, 2013; Olsson et. al., 2012; Hänchen et. al., 2006). All (CO2) generated by the burning of one liter of fossil fuel can be trapped using less than one liter of olivine; and more accurately 140 g of olivine can trap more than 176 g
of (CO₂) (Schuiling and Praagman, 2011).

For soil refinement implementations, there was a successful attempt to fully replacement of traditional Portland cement with geopolymeric gel formed by alkaline activation of fly ash (Bernal, et al., 2011). Recently, researches have been done on utilizing the alkaline activation of olivine to form a geopolymeric gel to increase soil strength (Fasihnikoutalab et. al., 2017). Those researchers utilize the carbonation process in addition to the alkal activation process to increase soil strength. The investigation for alternate binders recognizes materials that are would impact the surrounding soil to a minimum range upon treatment and cost-effective.

However, no works have used the alkali reaction method in addition to carbonation for the purpose of soil stabilization using olivine with different molarities of alkaline solutions for use in low cost (e.g. low-cost roads in some countries) and fast construction.

Nonetheless, few works have studied the use of geopolymeric binders in soil improvement applications and none as soil stabilization technique for low-cost roads (Sara Rios, et al. 2016). In previous work (Rios, et al., 2015), the authors have explored few mixtures and very promising results were found namely in terms of its stress-strain behaviour by means of triaxial tests. In this work, many mixtures of soil-olivine and sodium hydroxide (NaOH) solution, as an alkaline solution, in different molarities were studied and unconfined compression strength (UCS) tests were have been conducted to assess the possible application of new mixture as a construction material.

1. MATERIALS AND ETHODOLOGY

1.2. Materials

2.1.1. Soil
The soil used in this research contained 60% silt, 30% clay, and 10% sand with a mean particle size (D50) of 11.759 μm and a specific surface area (SSA) of 1.04 m²/g. Table 1 shows the physical and engineering characteristics of host soil. Table 2 shows the chemical characteristics of the untreated soil. Depending on the Unified Soil Classification System (USCS), the soil used in this research can be classified as CL.

2.1.2. Olivine
The olivine used in this study was provided by the Maha Chemicals Company of Malaysia. The olivine as-received had a large grain size, which wished for ball milling for 24 h. The speed of ball milling was 60 rpm to decrease the grain size and to give the uniformity to the particle size previously to addition to the soil. The particle size distribution shows that the D50 is around 2.3. The analysis of the specific surface area (after ball milling) shows it is around 6.07 m²/g. Fig. 2 demonstrates the particle size allocation for olivine sample after ball mailing.

A laser diffraction particle size analyses (Mastersizer 2000E, ver. 5.52) was used to determine the specific surface area of olivine. The specific surface area and D50 (mean particle size) of the olivine sand were 6.07 m²/g and 2.24 μm, respectively. Table 2 shows the chemical composition of olivine sand according to the supplier in this study.

<table>
<thead>
<tr>
<th>Table 1 Physical and engineering characteristics of soil</th>
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<tbody>
<tr>
<td>Basic soil property</td>
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<tr>
<td>Specific gravity (GS)</td>
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<tr>
<td>Liquid limit (LL) (%)</td>
</tr>
<tr>
<td>Plasticity index (PI) (%)</td>
</tr>
<tr>
<td>OMC (%)</td>
</tr>
<tr>
<td>MDD (Mg/m3)</td>
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<tr>
<td>UCS (kPa)</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 2 Chemical characteristics of host soil</th>
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</thead>
<tbody>
<tr>
<td>Constituent</td>
</tr>
<tr>
<td>Silica (SiO₂)</td>
</tr>
<tr>
<td>Alumina (Al₂O₃)</td>
</tr>
<tr>
<td>Iron oxide (Fe₂O₃)</td>
</tr>
<tr>
<td>Calcium oxide (CaO)</td>
</tr>
<tr>
<td>Potash (K₂O)</td>
</tr>
<tr>
<td>Magnesia (MgO)</td>
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<tr>
<td>Loss on ignition (LOI)</td>
</tr>
</tbody>
</table>

2.1.3. Alkali activator
Sodium hydroxide (NaOH) containing Na⁺ was selected as activator because of its well-known efficiency (Fasihnikoutalab et. al., 2017). The activator was supplied in pellet form by “R&M Chemical” company and was previously dissolved in
distilled water to acquire a predesigned concentration of 10 Molar. The reaction of the activator (NaOH) with water is strongly exothermic, as well as with all strong Alkalis. Consequently, the alkaline activator was left to cool down to room temperature before use.

2.2. Methodology

2.2.1. Mix composition and sample preparation

Table 3 shows the mix compositions of the materials used in this study (soil, olivine, and NaOH). The molarity of NaOH was changed during samples preparation. The first step to prepare the samples was dissolving the alkali activator in distilled water at the desired concentration. The compaction test was done in order to attain the optimum water content (OWC) with maximum dry density (MDD) of the alkaline-activated soil-olivine mixture (British Standards Institution 2002). After drying the host soil for 24 h, the soil was mixed with 30% olivine and alkali activator (NaOH) at different molarities. Control samples of the pure soil were also tested as a reference. Samples were set in cylindrical (50cm diameter and 100cm height) templates onto which suitably modified compaction was done and extruded. Samples, for alkali-activated with and without carbonation, were prepared for UCS at OWC and MDD. After samples extrusion, (CSO30-4, CSO30-6, CSO30-8, CSO30-10 and CSO30-12) samples were permeated with CO2 at 300 kPa pressure and for 7 days curing time (Table 3). Samples without carbonation (ASO30-4, ASO30-6, ASO30-8, ASO30-10 and ASO30-12) were wrapped in polythene covers to prevent water loss and cured under ambient temperature in different curing periods prior to UCS testing (Table 3). The soil+30% olivine cured for 7 days, while alkaline activated and carbonated alkaline activated soil+30%olivine cured for (7, 28 and 90 days).

Table 3 Mix composition of the study samples

<table>
<thead>
<tr>
<th>Mix</th>
<th>O (%)</th>
<th>OWC (%)</th>
<th>MDD (g/cm3)</th>
<th>NaOH (molarity)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S</td>
<td>0</td>
<td>24</td>
<td>1.58</td>
<td>0</td>
</tr>
<tr>
<td>S030</td>
<td>30</td>
<td>18</td>
<td>1.82</td>
<td>0</td>
</tr>
<tr>
<td>ASO30</td>
<td>4,6,8,10,12</td>
<td>30</td>
<td>18</td>
<td>1.82</td>
</tr>
<tr>
<td>CSO30-4, 8, 10, and 12</td>
<td>30</td>
<td>18</td>
<td>1.82</td>
<td>4, 6, 8, 10, and 12</td>
</tr>
</tbody>
</table>


2.2.2. Carbonation

Instantly after molding, carbonated samples were submitted to carbonation with a confining pressure of 400 kPa, meanwhile, CO2 permeated upward. A triaxial cell was utilized to permeate CO2 through the carbonated samples CSO30. The pressurized gaseous CO2 was subjected at 300 kPa through the alkali-activated soil-olivine samples for 168 h. The outflow tube was placed under water to detect the samples saturation with gaseous CO2.

2.2.3. Unconfined compressive strength

The UCS test was implemented on carbonated (at 168 h) at different molarities (4, 6, 8, 10 and 12) and uncarbonated (at 7, 14, 28, and 90 days) at different molarities (4, 6, 8, 10 and 12M) samples instantly after predesigned curing procedure according to BS 1924: Part 7 (British Standards Institution 2003).Instron 3382, with 100 kN capacity, the device was used for UCS testing (Seri Kembangan, Malaysia).

2.2.4. Microstructural analysis

XRD, SEM/EDX and FTIR tests were conducted on samples before and after carbonation at different molarities. Crystalline phases were examined using a Philips (Seri Kembangan, Malaysia) XRD from 20 to 75° 2θ. For an SEM analysis, a JSM 5700 device was used (Seri Kembangan, Malaysia) coupled with an EDX spectrometer. Before the SEM/EDX test, samples were sputter covered with gold before test conducting to increase the electrical conductivity of the surface as well as reduce charging. FTIR via a PerkinElmer Paragon 100 spectrometer within a spectrum of 4,000−500 cm−1 (Seri Kembangan, Malaysia) was utilized to investigate the composition of samples before and after carbonation.

3. RESULTS

3.1. Unconfined Compressive Strength

Fig. 1 presents the UCS values for natural soil (S), alkali-activated soil-olivine mixture (ASO) at different molarities of NaOH (4, 6, 8, 10 and 12M) at (7days) curing time. Natural soil samples (S) appeared an UCS value of 77.9 kPa. Alkali activation for soil-olivine mixture increased the strength of the mixture to 392.8, 421.4, 583.9, 1082.8 and 904.3 kPa at 7days, and for 2, 4, 6, 8, 10 and 12M respectively. Fig. 2 shows the effect of aging on the UCS values for the alkali-activated soil-olivine mixture at 10M of NaOH. Fig. 3 represents the behavior of carbonated alkali-activated soil-olivine samples at different molarities of NaOH (4, 6, 8, 10 and 12M) at 7days and 300 kPa CO2 pressure.

The UCS values slightly increased for soil-olivine specimens due to the presence of NaOH.
This might be attributed to the role of NaOH in elevating the dissolution of Si, Al and Mg within the soil (Fasihnikoutalab et al. 2017). The increase in the strength of ASO was significant with a molarity of 10M NaOH. These findings are inconsistent with that of Cristelo et al. (2011, 2012b). A cumulative increase in UCS values was observed with increasing the curing time from 7 to 90 days. ASO, for 10M, achieved the highest strength within all alkali-activated soil-olivine samples, resulting in 1082, 1728, and 2275 kPa at 7, 28, and 90 days, respectively.

Fig. 1. UCS of the alkaline-activated soil-olivine mixture at 7 days curing time for different molarities

Fig. 2. UCS of the alkaline-activated soil-olivine mixture at 7, 28 and 90 days curing time for 10M

Fig. 3. UCS of the carbonated alkaline-activated soil-olivine mixture at 7 days curing time for different molarities

Fig. 3 presents the UCS values of the carbonated alkali-activated soil-olivine mixture after 7 days of curing and at 300 kPa CO₂ pressure. The alkali activator molarity was changed from 4 to 12M, and subjected to CO₂ at a pressure of 300 kPa for 7 days. The increase of alkali activator molarity was accompanied by an increase in the UCS values. A significant increase, in strength, was achieved by increasing the NaOH molar value by 8 and 10 M. While the increase was noticeable by increasing the molarity by values 10 and 12 in uncarbonated alkali-activated soil-olivine samples. The results show that carbonation technique is a promising technique for soil stabilization and to increase the environmental concerns for alkaline agents. This may be attributed to alkali-activated soil-olivine samples were sufficiently carbonated within a few days to achieve strengths required for ground improvement.

3.2. SEM Characterization

The clusters shape particles in Fig. 4(a) actually refer to the soil particles. The microstructure of olivine particles is of irregular shape which can be seen in Fig. 4(b). Fig. 4(c) clarifies the soil-olivine mixture after 7 days of curing. By comparing Figs. 4(a and c) it’s clear that the olivine gives a more dense structure for the host soil as a result of its hydration and pozzolanic reaction within the host soil. Fig. 5(d) represents the microstructure of host soil mixed with 30% olivine in the existence of 10 M of NaOH after 90 days of curing. SEM images show a dense morphology with less major discontinuities, which is symmetrical with the
mechanical characteristics obtained. The raise of a new amorphous phase is obvious in Fig. 4(e), which detect the forming of a gel mass and crystals on the surface of the particles. Fig 4(f) shows the SEM images of carbonated alkali activated samples which subjected to carbonation at a CO₂ pressure of 300 kPa for 7 days. Fig 4(f) reveals a denser and more homogenous microstructure in the presence of NaOH.

Fig. 4. SEM images of (a) soil; (b) olivine; (c) soil olivine mixture; (d) alkaline-activated soil-olivine mixture; (e) carbonated alkaline-activated soil-olivine mixture

3.3. FTIR Characterization

Fig. 5 (a and b) demonstrates the FTIR of seven samples in the range of 500–4,500 cm⁻¹ containing 8, 10, and 12M of NaOH soil-olivine mixture after 90 days for (a) and 6, 8, 10 and 12M of NaOH carbonated alkali activated soil-olivine mixture. From Fig. 5(a), the band at 3,693 cm⁻¹, existing in all samples, is related to O─H stretching which come from brucite. Previous researches showed that the attribute Mg─OH stretch of phyllosilicates near 3,690 cm⁻¹ and a low stretch in the Mg─OH zone (Lee and van Deventer 2002b).
Fig. 5. FTIR of: (a) alkaline activated soil-olivine mixture for different molarities. (b) the carbonated alkaline-activated soil-olivine mixture at 300-kPa pressure for 7 days at different molarities

Bands harmonize to O─H stretching in the domain of 3,394–3,401 cm⁻¹ assist H─ bonds in all samples, as well as the water will presence even after 90 days of curing. This could clarify that greater strengths could be gain with prolonged curing and high molarity in terms of full reaction. However, very weak reductions near 1,220 cm⁻¹ assigned to Si─O stretching may be a sign of silica polymerization which indicates to M─S─H gels formation. These gels, which formed in an amorphous aluminosilicate gel, could be formed due to the dissolution of olivine with higher molarities of NaOH was apparent at 1,080–1,100 cm⁻¹ and 980–1,040 cm⁻¹ regarding to Si─O bond and asymmetrical stretching vibrations of Si─O/Al─O bonds, respectively (Criado et al. 2005). Bands at 680–650 cm⁻¹, and 914, 790–730 were related to Si─O vibration stretching, and Al─OH, Al─O bond stretching, respectively (Abdul Rahim et al. 2014; Weng and Sagoe-Crentsil 2007). Fig. 6(b) presents the FTIR characterization of carbonated alkaline activated soil-olivine mixture after carbonation at a CO₂ pressure of 300 kPa at different molarities of NaOH of 6, 8, 10, and 12M for 7 days curing period. The crests appeared are corresponding to the existence of an aluminosilicate gel in amorphous form. Bands at 950 and 797 cm⁻¹ were assigned to the symmetric and asymmetric stretching vibration of Al─O─Si and Si─O─Si bonds, showing that the magnitude of carbonation in these samples. After carbonation, there were changes at 1,220 cm⁻¹, and this refers to the chain structure modifications of M─S─H. These modifications may refer to the decomposition of M─S─H during the carbonation process and a higher degree of polymerization, as well as, it may refer to the lengthening of silicates within the residual gel. These results establish the results from previous researches on the contribution of carbonation in the slag geopolymerization. So, the role of Mg²⁺ is possible to be as the role of Ca²⁺ since it is present within the geopolymeric gel phase (Lee and van Deventer 2002a, b). The strong peaks present around 1,410 and 1,570 cm⁻¹ in Fig. 5(b) are imputed to C─O vibrations from the sequestration of CO₂ and forming of carbonates in solution, that might be in close combination with Na⁺ or Mg²⁺ cations (Abdul Rahim et al. 2014). The bands between 1,450 and 1,650 cm⁻¹ assigned to the asymmetric stretching of CO₃²⁻ earning strength with increasing NaOH molarity due to the forming of MgCO₃ and NaHCO₃. This is most noticeable as the molarity of NaOH increased from 6 to 10M, leading to the vanishing of the 3,690 cm⁻¹ band originally assigned to brucite.

### 3.4. XRD Characterization

Fig. 6 demonstrates the XRD of alkali-activated soil-olivine mixture after 90 days of curing. Brucite, mullite, serpentine, quartz, sodium silicate (Na₂SiO₃), and Mg peaks are revealed due to olivine dissolution through the sodium hydroxide in soil. Peaks of brucite (21, 36, and 48°), quartz (SiO₂) (24, 26, 28, 39, and 45°), and sodium silicate (30 and 35°) were revealed meanwhile the patterns (Komljnović et al. 2010; Yi et al. 2013b). Figs. 7(a–d) present the XRD patterns for carbonated alkali activated soil-olivine mixture for 6, 8, 10 and 12M of NaOH under 200 kPa CO₂ pressure for 7 days curing period.

### 4. DISCUSSION

The chemical bonds between olivine compositions will break down in the presence of NaOH, this will happen when olivine mixes within a soil mass. So that, MgO and SiO₂ will be free and this will lead to leaching the silicon from amorphous phases in olivine particles (Fasihnikoutalab et.al. 2017). So that, Mg(OH)₂ and Na₂SiO₃ are created as approved by FTIR and XRD analyses (Figs. 6 and 7). The Na₂SiO₃ created from the reaction between NaOH and olivine in an aqueous solution. Previous
studies show that using NaOH and/or Na$_2$SiO$_3$ as activators to raise phases dissolution and later alkaline activation (Abdul Rahim et al. 2014; Phoongernkham et al. 2015). The appearance of A-S-H gel could be a result of the use of NaOH and/or Na$_2$SiO$_3$, which could lead to the leaching of the Si and Al from the amorphous stage of soil. Adding Ca was demonstrated to have a positive impact on the mechanical characteristics of alkali-activated binders (Temuujin et al. 2009; Ryu et al. 2013). The high content of MgO (48%) in olivine makes it a good candidate to release of Mg ions, which may display a comparable behavior to Ca ions convenient to their similar charge. In addition, Al, Si with Mg play an important role in increasing the amount of crystal formed in the new matrix. In the existence of an alkali activator, Mg ions might give more nucleation.

Fig. 6. XRD of alkaline activated soil+30% olivine (ASO) after 90 days curing time in presence of: (a) 8M, (b)10M, and (c) 12M. of NaOH.
From FTIR and XRD results for carbonated alkali activated samples, the reaction between NaOH and olivine leads to the creation of Na₂SiO₃. Thereafter, Na₂SiO₃ reacts with CO₂ dissolved in water, leads to the formation of NaHCO₃ and SiO₂. These reactions will lead to the formation of nesquehonite, dypingite, and hydromagnesite, thereafter, to form well-divergent networks of huge crystals with high efficiency of binding ability (Yi et al. 2013c). As a result, this led to a denser and more homogenous microstructure for the new matrix of carbonated alkali activated soil-olivine mixture (Fig. 5(f)), converting into increased strength gain. Eitelite Na₂Mg(CO₃)₂ has been formed from the reaction between Mg(OH)₂ and Na₂CO₃ at a high pressure of CO₂, which is another advantage of using olivine in the presence of NaOH. Eitelite includes CO₂ inside its structure in the form of MgCO₃, that qualifies it to sequestrate of twice the quantity of CO₂ per ton of olivine Mg-rich rock [J. Blencoe and D. Palmer, “Carbonation of metal silicates for long-term CO₂ sequestration,” U.S. Patent No. 8,114,374 (2012)].

The tests have been conducted in different scales as needed to show the changes in the soil-olivine mixture and chemical bonds due to the chemical reaction.

The study emphasizes carbonating olivine is a potential technique to produce a sustainable binder through the sequestration of CO₂ in the presence of a range of molarities of NaOH. Results demonstrate that olivine can gain high strengths as a result of the sequestration of greater amounts of CO₂ in the presence of NaOH in a wide range of molarities, consequently offers environmental benefits. As well, from an economic viewpoint, this technique offers a cost-effective candidate (olivine and NaOH), as such candidates have a lower cost in comparable other binders used for soil stabilization such as cement and lime.

5. CONCLUSIONS

This paper presents some results of a silty clay soil stabilized with two techniques: firstly; geopolymer production technique, and secondly; carbonation technique. Both of these techniques were used with a range of molarities of NaOH. The source binder used was olivine. The strength of alkali-activated soil-olivine mixture with 10M of NaOH increased and curing time, achieving a strength gain up to 14 times higher than that of host soil. The carbonated alkali activated soil-olivine mixture at a 300 kPa CO₂ pressure at 7 days curing time, increased the strength of soil up to 65 times higher than the host soil and 4.6 times that of the alkali-activated soil-olivine mixture after 90 days of curing, in the presence of 10M of NaOH. From microstructural analysis, the strength development was assigned to carbonation with the formation of new crystals like nesquehonite, and hydromagnesite and dypingite. The FTIR results confirmed the formation of the final gel.

From the carbonation results, two molarities were selected which showed comparable strength values in contrast with the others, which are 8 and 10M.

The results of this paper highlighted the usefulness of the abovementioned techniques in decreasing the demand for traditional soil stabilizers (cement/lime).

From these hopeful results, further tests should be executed in this material, leaching tests and resistance to acidic attack to establish other characteristics of this material that may allow its application in different construction projects.

6. ACKNOWLEDGMENTS

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7. REFERENCES


