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Soil Stabilization using Calcium Carbonate Precipitation via Urea Hydrolysis

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Abstract **-** Soil improvement techniques of granular soils via Microbially Induced Calcite Precipitation (MICP) and Enzyme Induced Calcite Precipitation (EICP) techniques have attracted an increasing attention over the past decade. MICP and EICP rely upon the hydrolysis of urea catalyzed by the urease enzyme. However, the MICP is a process in which living organisms produce the urease enzyme. These techniques use biogeochemical reactions to precipitate calcium carbonate ($CaCO_3$) especially in granular soils. Calcite precipitation improves the soil shear strength by introducing calcite to fill pores and bind soil particles. In this study, a review on successful development and implementation of the urea hydrolysis techniques is presented. In addition, discussion on potential advantages and limitations of urea hydrolysis techniques is included.

Keywords**:** Urea hydrolysis; Enzyme Induced Calcite Precipitation, EICP; Microbial Induced Calcite Precipitation, MICP; Soil biocementation; Soil improvement; Bio-grouting.

1. Introduction

In order to enhance properties of weak soil formations (for example: deep loose sand deposits), a wide range of ground improvement techniques have been introduced over the past decades. Majority of these ground improvement techniques utilize mechanical energy and/or man-made binders like Ordinary Portland Cement (OPC), both of which require substantial energy for material production and/or installation. Moreover, injection and grouting techniques have been used to improve soils by injecting chemical materials into the pore space to bind soil particles together [1].

Nowadays, there is a high demand for new sustainable methods to improve soils. Over the last decade, extensive research has been undertaken to find alternative soil binders to replace OPC for soil improvement. Among other alternatives, calcite precipitation has been intensively investigated as a sustainable alternative for soil improvement and soil grouting. There are several techniques used to induce calcite precipitation, including urea hydrolysis, microbial denitrification, and sulfate reduction [2].

The hydrolysis of urea is the most advanced mechanism to induce calcite precipitation in terms of development, and most often discussed in the literature due to its simplicity [3].

With this context, this research presents an overview of induced calcite precipitation soil improvement techniques via hydrolysis of urea. First, a conceptual framework is highlighted to provide an overview of the primary components of calcite precipitation soil improvement techniques and to emphasize the interdisciplinary nature of this field. Second, the basic bio-chemical background behind the urea hydrolysis in general is also presented with emphasis on microbial and enzyme induced calcite precipitation techniques. More focus is placed on calcite precipitation of sands, since research on granular soils is currently more advanced than the cohesive soils. Then, various laboratory examples used to assess how the pore space volume is altered by calcite precipitation are demonstrated. Next, successful experimental examples are presented showing the potential of using these techniques for enhancement of soil engineering properties.

2. Hydrolysis and Calcite Precipitation

Urea hydrolysis is a process in which urea molecule decomposes in presence of water to form carbonate and ammonium irreversibly. The urea hydrolysis can occur without the presence of catalyst, however in presence of catalyst (urease Enzyme) it is 10^{14} times faster than chemical (un-catalyzed) urea hydrolysis [4]. Urease is a nickel-containing enzyme of high molecular weight [5]. The urease enzyme can be found in many organisms, including many bacteria, some fungai, invertebrates and several higher plants [6]. The urease activity, which is measure of its activity in urea hydrolysis, depend mainly of the source of the enzyme [7, 6]. For example, Jack Bean (Canavalia ensiformis) is the most common source of commercially available urease that produces high activity enzymes up to 3,500 U/g [6]. While, the Sporosarcina pasteurii, formerly known as Bacillus pasteurii, is the most used bacterial source of urease enzyme [8].

As the reaction rely on the activity of urease, the process is termed urease-aided $CaCO_3$ mineralization. This process, in contrast to typical chemical techniques, mimics the formation of $CaCO_3$ in nature. Therefore, the process has several potential applications in multiple engineering fields, particularly geotechnical, environmental and construction. This section emphasis on the principles of urease-aided calcium carbonate mineralization. This section will discuss the two main pathways of urease-aided calcium carbonate mineralization either through enzymes produced by microbial activity or agricultural driven enzyme sources. The calcium carbonate mineralization relies on the pH and the availability of crystal nucleation sites. The process requires alkaline pH as the most significant condition to shift the bicarbonate equilibrium to form carbonate ions [8].

Even though various biological pathways have been proposed in the literature to precipitate calcite biologically (for example, denitrification, photosynthesis, autotrophic pathway, and etc.). The method that is most widely utilized for the precipitation of $CaCO_3$ for technical applications, is arguably the one based on the hydrolysis of urea [8]. In this approach, the reaction as mentioned earlier is catalysed by the enzyme urease in general regardless of the enzyme source.

2.1. Urease-Aided Calcite Precipitation (UACP)

The process of urease-aided calcium carbonate mineralization in general (irrespective of the enzyme source) is activated by the urease catalysing the hydrolysis of urea. Hydrolysis of urea, result in producing carbon dioxide (CO_2) and ammonia (NH_3). Following, ammonia form ammonium (NH^{4+}) in aqueous environment and thereby increasing the pH of the solution (creating favourable conditions to carbonate precipitation). In the presence of dissolved Ca^{2+}, the ions merge forming calcium carbonate precipitates. The overall process summarized in Fig.1. As shown, the overall reaction takes place by the supply of carbonate ions resulted from hydrolysis of urea and of alkalinity generated by the reaction. The calcium carbonate precipitate bind soil particles together as it accumulates at soil contact points. There are two sources investigated in the literature for as enzyme source. The first in the enzyme produced through the ureolytic bacterial activity. This technique is usually referred to as Microbial Induced Calcite Precipitation (MICP), the other technique is Enzyme derived from agricultural source usually called Enzyme Induced Calcite Precipitation (EICP).

2.3. Hydrolysis of Urea using Microbial Activity

In MICP, the ureolytic bacteria activity produce the urease enzyme as catalyst for the reaction. Which means that the reaction follows the same hydrolysis procedure illustrated earlier with the exception that enzyme source is coming from the bacteria activity. Moreover, the deposition of the calcium carbonate precipitates mostly takes place on bacterial cell walls, which serve as nucleation sites. Because bacterial cell walls are negatively charged, they attract and bind Ca^{2+} ions, resulting in their accumulation around the bacteria cell [9]. Consequently, carbonate crystals cultivate on the external surfaces of bacteria cells by successive layers encasing the bacteria within the precipitation.

There are two different approaches for MICP: bio-augmentation in which external bacteria are grown in the lab and then injected into the soil, and bio-stimulation in which indigenous bacteria within soil pores are used for soil treatment [10]. The MICP process is similar to the general summary shown earlier for the hydrolysis except that in the end of the reaction carbonate ions react with calcium ions present either in the surrounding environment. Three-phase injection plan is usually adopted in bio-augmentation approach where the ureolytic bacterial is injected in the beginning into the soil pores. Followed by injection of solution of $CaCl_2$, which work as a fixation agent for the bacterial cells to the soil particles [11]. Finally, a cementation fluid containing urea, calcium chloride, and in some cases additional nutrients and a pH stabilizer is injected [10]. On the other hand, injection of bio-augmentation has shown significant practical difficulties, including clogging near the pumping inlet ports, uneven distribution of the cementation within the soil pores and the complexity of managing an on-site bioreactor prior to injection [11]. More recently, Cheng et al. [12] proposed one-phase low-pH mixture of bacterial culture, urea, and $CaCl_2$. The proposed acidic buffer provided lag period, which is a function of several parameters (i.e. pH, biomass concentration, and urease activity), to allow even distribution within the soil pores and reduction of clogging in inlets pores [12]. Moreover, Cheng et al. [12] showed that the use of this one-phase approach reduced the production of the ammonia gas into the environment. Gomez et al. [13] have shown that efficient MICP treatment can be activated in situ using a bio-stimulation approach [13]. However, in both bio-augmentation and bio-simulation, the ureolytic bacteria is an aerobic bacteria which means it needs oxygen to sustain, this may restrict their activity for deep soil treatment. As mentioned earlier, S.pasteurii are the most widely used species of bacteria in MICP due to high urease activity, and resistance to high concentration of the ammonium by-product of urea hydrolysis [10]. Since the MICP sand treatment depends on calcite precipitation, the effectiveness of soil treatment is directly dependent on the distribution of calcite [14]. DeJong et al. [14] showed the results of SEM scanning for treated sand specimen prepared using urease-aided calcium precipitation using specimens prepared by Sporosarcina pasteurii bacteria as source of enzyme and three-phase injection routing. The samples were fixed with epoxy fixation and then the specimen surface was polished. The results shown in Fig. 2 confirm the calcite distribution around sand particles with calcite concentration at particle/particle contacts [14].

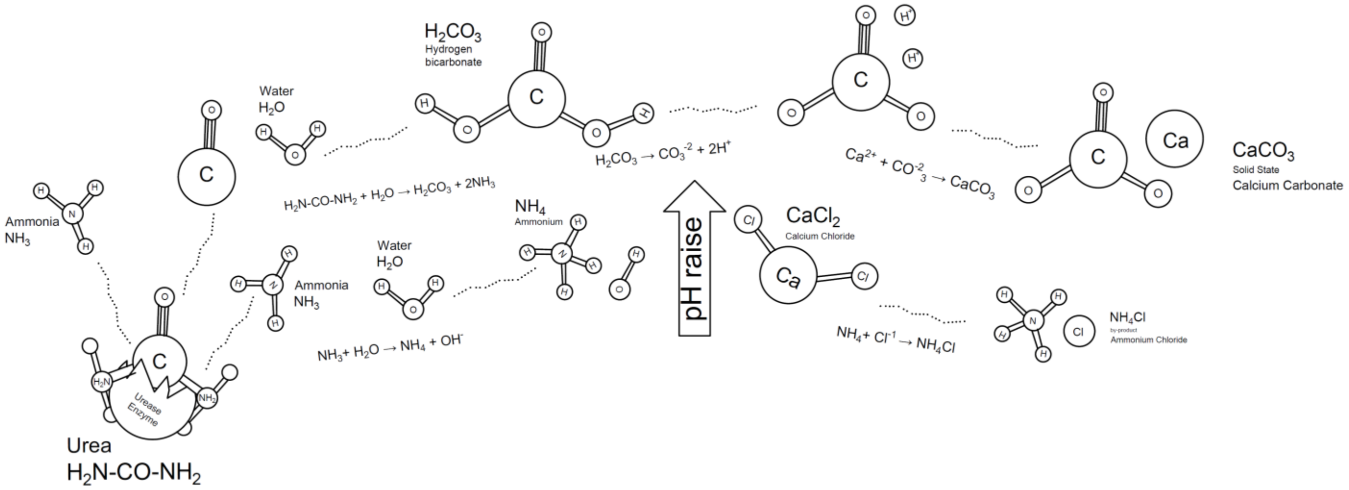

Fig. 1: Overall urea hydrolysis reaction irrespective of the Enzyme Source.

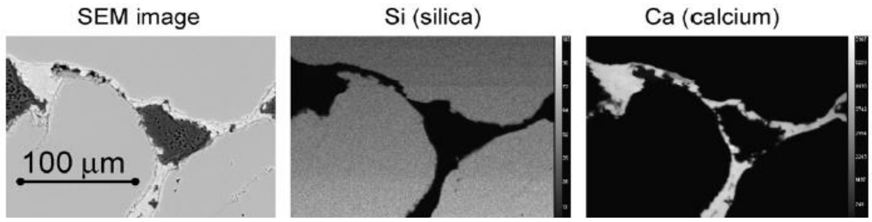

Fig. 2: High-resolution SEM images showing calcite precipitation distribution around sand particles for MICP treated sands [14].

2.4. Enzyme Induced Calcite Precipitation (EICP)

Unlike MICP, EICP techniques uses free urease enzyme derived from agricultural source to catalyse the hydrolysis of urea in the solution, which results in carbonate ion production. In the presence of calcium ions from any salt source, the carbonate ions precipitate as calcium carbonate as a result of increase in carbonate concentration beyond the level of supersaturation. Unlike ureolytic bacteria, the free urease enzyme used in EICP has a size on the order of 12 nm and is soluble in water, which increases groutability of the enzyme inside soil pores [10]. Moreover, in EICP there is no need to provide nutrients for bacterial activity [10]. Hamadan and Kavazanjian [15] showed the results of SEM scanning for treated sand using EICP solution. In their research, Hamadan and Kavazanjian [15] used EICP solution that have enzymes derived from (Jack Bean) and organic material as stabilizing agent (dry milk). The SEM images shown in Fig. 3 confirm the carbonate precipitation around sand particles with calcite concentration at particle/particle contacts even in the absence of bacteria comparable to the results in MICP [15].

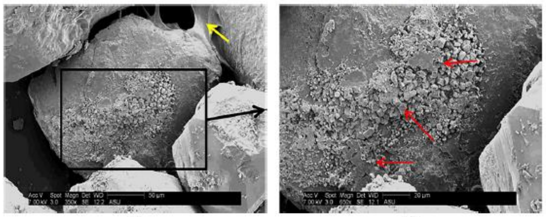

Fig. 3: High-resolution SEM images showing calcite precipitation distribution around sand particles for EICP treated sands [14].

3. Applications of the Calcite Precipitation for Soil Improvement

3.1. Improvement of Sand Soil Mechanical Properties Using MICP

MICP treatment technique has shown potential to improve variety of soil properties including permeability, stiffness, compressibility, shear strength, and volumetric behaviour [16]. DeJong et al. [14] conducted experimental program to investigate the mechanical properties of sandy soils treated with MICP technique. The soils were treated using three-phase treatment protocol illustrated earlier. DeJong et al. [14] observed that the improvement occurred in shear strength of the treated soil tested in undrained conditions is attributed to both cementation as well as densification of the loose sand soil specimen. Both cementation and densification are result of the precipitation of the carbonate in the soil pores. In this experiment, the untreated loose sand specimen exhibits monotonic collapse while the MICP treated specimen experience brittle failure where the sample experience a large peak and then degrades to slightly higher residual shear strength compared to untreated soil [16].

Van Paassen et al. [3] conducted a large laboratory experiment to investigate the feasibility of MICP treatment for field applications. In this experiment, Van Paassen et al. [3] built a five meter long PVC tube (internal diameter of 66 mm) filled with sand of dry density of 1.65 g/cm^3. In this experiment, Sporosarcina pasteurii was used as source of enzyme with three-phase injection program. The strength and stiffness measurement of the treated sand showed a significant improvement up to 4 m. While at the end of the column the sand was not improved and the calcium precipitation was minimal. Van Paassen et al. [3] attributed this to the clogging occurred and the consumption of the urea along the column. Moreover, Van Paassen et al. [3] found out that there is a threshold for calcium carbonate content which below no significant improvement in sand was observed (see Fig.4). Clear correlation was observed between the calcium carbonate content and the sand shear strength (Fig.4). Residual strength was found to be comparable with untreated sand, which mean that once the bonds were broken, the increase in strength of the material was almost completely lost [3].

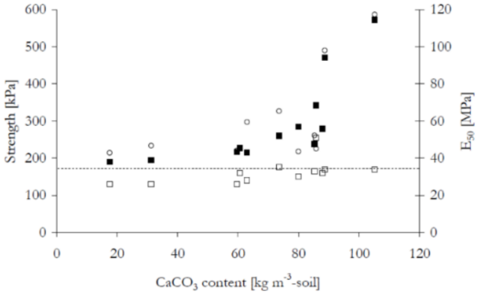

Fig. 4: Unconfined compressive strength (■), stiffness, E50 (○) Young's modulus at 50% of the peak stress and residual strength of material after failure (□) versus calcium carbonate content [3].

As mentioned earlier usually a three-phase program is used in MICP to treat soil in the laboratory to enhance the soil strength. Also, cycles of treatment (up to 4 cycles) is usually utilized to increase the calcium carbonate precipitation and hence increase the soil shear strength. This approach limits the applicability of using MICP in field applications. However, recent results from Cheng et al. [12] shows that low-pH one phase solution may be used to enhance the soil with no need for three phase approach. The one phase approach proposed is shown to have a better distribution of the carbonate precipitation in case of lower urease activity solution however; higher strength is achieved in case of higher urease activity solution (see Fig. (5)). This new approach enhance the applicability of the MICP for grouting application, however multiple cycles is still needed to reach the desired strength.

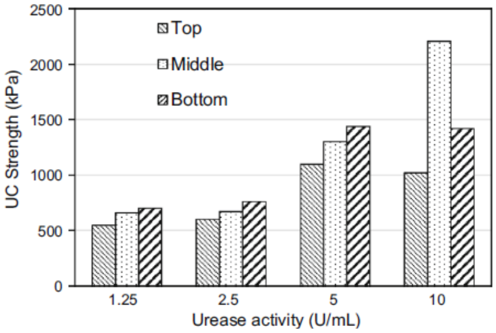

Fig. 5: Unconfined compressive (UC) strength of sand treated with one-phase low pH-solution MICP solution versus urease activity (distribution of carbonate precipitation along treated soil column is shown) [12].

3.2. Improvement of Sand Soil Mechanical Properties Using EICP

Almajed et al. [17] used EICP cementation solution composed of 1 M urea, 0.67 M calcium chloride, and 3 g/L enzyme to treat sandy soils. In their research they compared two different treatment techniques. First treatment technique was simply by mixing the treatment solution with the sand soil and compacting soil specimen in mold (mix and compact). The second technique i by percolating the cementation solution from the top of the soil specimen. Both methods are suitable for surficial soil treatment. Unlike the MICP no need for three phase treatment scenario but rather all the components of the cementation solution are added together and added to the soil in one step. This enhance the practicality of this technique for field applications. Almajed [17] investigated adding one, two and three cycles of EICP solution treatment to investigate the effect of the calcium carbonate content on the treated sand shear strength. Almajed et al. [17] found out that samples treated with mix and compact (without adding subsequent cementation solution) did not have sufficient strength to be extruded from the tube. This suggest that the percolation facilitated nucleation around sand solid particles and consequently growth of bridges between particles, providing better binding between particles (see Fig. 6). Fig.7 shows the results of the unconfined strength conducted by Almajed et al. [17] plotted versus the mass of the calcium carbonate precipitation for soil samples treated with percolation of EICP cementation solution. Almajed et al. [17] found that there is a threshold of carbonate content exists for a significant increase in the Unconfined Compressive Strength (UCS) of specimens treated using EICP. This threshold exists also in case of MICP as shown by Van Paassen et al. [3] to be around 60 kg/m^3 compared to 30-35 kg/m^3 in case of EICP treated soils. Moreover, soil treated with EICP solution with the same calcium carbonate content yield a higher UCS compared to soils treated with MICP.

3.3. Fugitive Dust control using Urease-Aided Calcium Carbonate Precipitation

Due to rapid economic growth, a notable growth has come in the construction of large-scale infrastructure, including new industries, transportation networks, and cities. All this contribute to the air quality and the amount of the particulate matter (PM) in the air. Dust rises from soils due to construction activities considered one of the main sources of air pollution. The air pollutants may infiltrate inside houses and homes contributing to indoor air pollution.

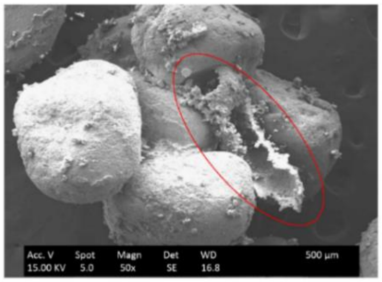

Fig. 6 : Precipitation from percolation method between particles and along flow path [17].

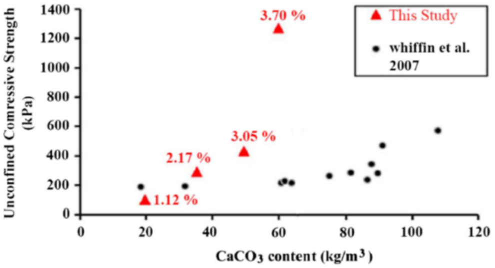

Fig. 7: Unconfined compressive strength versus carbonate precipitation for specimens treated by EICP using percolation and MICP-treated specimens [17].

Several researchers have investigated the feasibility of the control of dust using MICP, EICP and mix of both techniques [18, 19, 15 & 20]. In all these studies, Sporsarcina pasteurii ureolytic bacteria was used for the MICP solution. Bang et al. [18] compared three treatment options for surficial stabilization of cohesionless fine sand with ureolytic carbonate precipitation: (a) urease enzyme only (i.e. EICP); (b) ureolytic bacteria only (i.e. MICP); and (c) enzyme mixed with ureolytic bacteria (i.e. combined EICP and MICP). The outcome of the study was that the enzyme-only treatment produced the highest increase in strength and resistance to wind erosion even better than mix of both cementation agents. It is worth to mention here only one cycle of application was used in this study and the MICP solution was mixed and sprayed on soil sample. Fig. 8 shows a picture of the sandy soil treated with Enzyme from Bang et al. [18].

Fig. 8: Sand Particles 7 Days after Enzyme Treatment [18].

Maleki et al. [19] conducted wind erosion tested using MICP solution varying the concentration of the cementation solution (both urea and calcium chloride concentration). The soil resistance to erosion increased with the increase in the cementation solution. In this study, penetration test results for MICP treated soils were correlated to the wind erosion in wind tunnel.

Hamadan and Kavazanjian [15] conducted wind tunnel experiments to evaluate the use of EICP solution containing plant-derived urease to stabilize soil against fugitive dust emission. In their study, three different soils were tested: a native Arizona silty sand, a uniform medium-grained silica sand and fine sand-sized mine tailings from southern Arizona. Hamadan and Kavazanjian [15] compared two conventional treatment options to treat soils with EICP: a) water; (b) urea solution only; and (c) salt. The results show that application of a urea–calcium chloride solution without urease enzyme also provided similar erosion resistance (after drying). Hamadan and Kavazanjian [15] stated that due to the solubility of calcium chloride subject to precipitation the EICP treatments more favourable and more durable. The wind tunnel tests established the treatment concentrations at which EICP was more effective in suppressing fugitive dust than specimens prepared by either thoroughly wetting the soil or treatment with a salt–urea solution. The overall outcome of the study shows the feasibility of using EICP for fugitive dust control.

Recently, Tian et al. [20] used MICP for soil erosion control. The MICP solution was sprayed on the soil surface and multiple treatment was used up to seven treatments with the cementation solution. Tian et al. [20] found out that the increasing number of MICP treatments increased the soil resistance to wind erosion.

4. Limitations Associated with UACP

There are different limitations that were identified in the literature for the UACP treatment. These limitations are mainly related to economy, environmental impact, soil type and spatial distribution of the treatment. In addition, durability related issues have not extensively investigated in the literature.

4.1. Economy

As the UACP is mainly catalysed by urease enzyme, the enzyme source is the most expensive component in the UACP. In EICP, the commercially available urease is of high purity enzyme used for lab grade applications. Replacing the lab grade enzyme used in laboratory studies with cheaper crude extracted enzyme from food waste can also considerably reduce costs. Efforts to reduce the cost of EICP have included adjusting the concentrations of EICP constituents to optimize the solution by decreasing the enzyme and adjusting the other constitutions (urea and calcium chloride) [16]. In MICP, three phase grouting and the nutrition needed for the cultivation of the bacteria are the most expensive parameter. In this context, the use of one phase low pH grouting technique proposed by [12] and Bio-stimulation may provide cost-effective alternative.

4.2. Environmental Impact

Ammonium production is the main environmental concern with UACP. Most of the criticism received for these methods is related to the production of the ammonium, which is considered a pollutant to ground water. Flushing the ammonium from the soil is not feasible. As mentioned earlier, the use of low-pH one phase MICP solution may reduce the gaseous ammonium by-product [12]. Osman et al. [21] showed the potential of the use of nitrogen fixating bacteria, Rhizobium leguminosarium, with the MICP to reduce or eliminate of the ammonium by-product [21].

4.3. Spatial Distribution of the Precipitation

The main challenge for both EICP and MICP is obtaining uniform treatment in the soil mass. Due to the speed of hydrolysis process in presence of urease enzyme (start almost instantaneous), precipitation in UACP is usually non- and concentrated usually at point of cementation solution application. Therefore, controlling the rate of precipitation is of the key factors in achieving uniform treatment with UACP. Hamdan [22] utilized hydrogel to enhance EICP by reaction product (calcite) around the soil particles. This hydrogel-assisted enzyme-induced carbonate precipitation showed promise to have a uniform distribution of calcite around soil particles. The use of low urease enzyme in MICP along with low-pH solution have shown a potential to increase the lag period and enhance spatial distribution of the carbonate precipitation.

4.4. Durability

Few researchers have discussed durability issues related to calcite precipitation. One of the obvious durability issues related to calcite treated soil is the susceptibility of calcium carbonate to dissolve in acid environments. This may lead to sever deterioration of treated soil mechanical properties. However, wetting and drying cycles and temperature effects are factors that need to be investigated. Cheng et al. [23] observed the high durability of MICP treated soils against freeze thaw cycles and attributed this to abundant bridging points in the soil mass and high soil permeability.

5. Conclusions

The cementation process using urease-aided calcium carbonate precipitation offers a promising development in the soil improvement techniques. The main advantages behind the feasibility of this technique lies in several facts. (i) The technique avoids using any cementing agent, (ii) it avoids using cement, a material whose production greatly contributes to the emission of CO_2. In conclusion this technique is classified as environmentally-friendly approach to geotechnical engineering applications. However, this method has several limitations. These limitations include: (i) the production of by-products: ammonia which is harmful gas that is not recommended to mix with groundwater, (ii) limited cost effectiveness in the current stage of research due to the high price of enzymes and bacterial nutrients, (iii) the complexity of the process due to the multidisciplinary approach needed to design such treatments, (iv) the non-uniform distribution of the cementation especially in case of using bacterial activity as catalyst for the calcite precipitation and (v) the groutability issues related to clogging of tubes and grouting lines during grouting of the cementation solution.

There are two methods presented in this research for the soil stabilization via hydrolysis. The first technique uses the bacterial activity as catalyst to produce the urease enzyme needed for the hydrolysis. The second technique uses agricultural source for the urease enzyme as catalyst for the ureolysis reaction.

For both techniques, the results of the laboratory tests reported in this research indicate that these two methods have potential to replace conventional improvement techniques. While there are still challenges to overcome prior to commercialization of UACP, there clearly is a potential for bio-grouting to provide a sustainable method for improving the properties of granular soil.

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