Major applications of MICP sand treatment at multi-scale levels: A review.

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ABSTRACT
Microbially Induced Calcite Precipitation (MICP, or biocalcification) is a biochemical process governed by microbial activity to induce the precipitation of calcite between soil particles. The mechanical and hydraulic behaviors of bio-treated materials are thus significantly enhanced. The potential application of MICP to deal with problems such as liquefaction, internal erosion, settlements and frost damages has been demonstrated and this promising technique offers an environmentally friendly alternative to traditional soil improvement approaches. However, since it emerged in the beginning of the 21th century and despite extensive demonstration of the process at laboratory scale, few field applications have been completed to evaluate the performances and understand the biochemical process at larger scale. This article reviews the main large scale applications available in the published literature up to now. The major contribution of this research is to assess the main parameters restricting the implementation of this method on site. A summary of improvements that should be considered to meet industry needs and match the promises of this technique is also established.

RÉSUMÉ
L'amélioration des sols en place par voie biologique est un procédé basé sur le contrôle de l'activité bactérienne pour cimenter les particules d'un sol par le biais de précipités de calcite. Les performances mécaniques et hydrauliques des sols traités s'en trouvent ainsi améliorées. Cette technique, aussi connue sous le nom de biocalcification, possède un potentiel d'application pour divers problèmes géotechniques tels que la liquéfaction, l'érosion interne, les tassements ou encore le gel/dégel et elle constitue une alternative aux techniques traditionnelles d'amélioration des sols en place. Bien que la technique ait démontré son efficacité au laboratoire depuis son apparition au début du 21ème siècle, son application à grande échelle pour évaluer les performances et comprendre les processus biochimiques reste restreinte. Le présent travail recense les différents essais de terrain réalisés jusqu'à présent. La principale contribution de cet article est d'évaluer les contraintes limitant l'implantation de la biocalcification sur le terrain. À la fin de cet article, les recommandations et améliorations, à mettre en place dans le futur, seront détaillées pour une meilleure insertion de cette technologie dans l'industrie géotechnique.

1 INTRODUCTION

Historically, ground improvement techniques counted two main sorts: (1) mechanical compaction or preloading, and (2) injection of cement or other chemicals. While the first one was energy consuming and not suitable for urban areas, the second type needed the use of synthetic materials that could be toxic for natural environment and people's health (DeJong et al. 2010). However, soils constitute a niche for biological activity, even though ignored for centuries, and there is an opportunity to exploit those natural processes (Dejong et al. 2013). For instance, the influence of plant roots on slope stability has been recognized and exploited (Gray and Sotir 1996). Mitchell and Santamarina (2005) were the leaders in the application of biological processes in geotechnical engineering. Since then, researchers in the geotechnical field have undertaken discussions and multidisciplinary research programs to develop strategies for advancing this emerging field and identify primary challenges and opportunities (DeJong et al. 2006, Ivanov and Chu 2008, Montoya and Dejong 2013, Mujah et al. 2016, Wang et al. 2017). One of these biological techniques is the Microbially Induced Calcite Precipitation (MICP) which has been widely investigated in laboratory and has demonstrated its efficiency to tackle number of geotechnical problems (Montoya et al. 2013, Amin et al. 2017, Ning-Jun et al. 2017). Various methods were employed to enhance and improve the performances of this technique at meter scale. Although this research field has jumped forward thanks to laboratory investigations, some issues are stifling its development at field scale.

This paper provides an exhausted review of the technique, the role of biological processes in geotechnical engineering, the process and factors of influence including examples of their application at larger scale and salient issues encountered. The major purpose of this review is to assess the main parameters restricting the implementation of this method into field. Several recommendations of authors are also considered to highlight the gap between research and practice for different fields of application and a summary of improvements that should be considered to meet industry needs is established.

2 MICP BACKGROUND

Naturally, cementation is created through chemical or biochemical processes (diagenesis) associated with weathering. For instance, sandstone formation is directly attributed to calcite precipitation. Within the same deposit, natural cementation varies depending on controlled characteristics of environmental conditions (Saxena and Lastrico 1978). Various factors either inhibit or facilitate the process of cementation including pore-water chemistry (degree of supersaturation), ability to transport Ca^{2+} and/or HCO_3^- to the precipitation site, the presence of pre-existing carbonate substrate, and the permeability as well as texture (Molenaar and Venmans 1993, Hall et al. 2004, Mozley and Davis 2005). Calcite precipitation in nature follows two different mechanisms, either by deposition from supersaturated water with carbonates ions or from chemical exchanges at the water-soil interface (Ismail et al. 1999). The earth's crust counts various places where naturally cemented sands are encountered such as stromatolites in shallow high saline water in Australia (Figure 1).


Figure 1. Stromatolites at shark Bay Western Australia (Photo taken by Stuart Lilley Photography).

Observations from nature leaded into exploring a new branch in geotechnical engineering called biogeotechnology. This multidisciplinary field aims to transform natural sands into biosandstones using microbiological processes while improving their engineering properties (Achal and Mukherjee 2015). MICP is the consequence of such microbial metabolic activities and aims to transform sand into sandstones (Stocks-Fischer et al. 1999, Ramakrishnan et al. 2001). Various processes including urea hydrolysis, denitrification, sulphate reduction inducing dolomite precipitation, and iron reduction inducing ankerite or even other minerals precipitation were investigated (Ciurli et al. 1999, Roden et al. 2002, Karatas et al. 2008, van Paassen et al. 2010, Dejong et al. 2013). The most efficient process in terms of energy is enzymatic hydrolysis of urea by microbes (DeJong et al. 2010). It is also straightforward, easily controlled and generates up to 90% of chemical conversion efficiency of the precipitated calcite amount in less than 24 hours (Al-Thawadi 2011, Dhami et al. 2013).

Number of bacteria species could be used for their urease enzyme production in biomineralization process (Kucharski et al. 2006). Those are not hazardous for environment as they are natural (Fritzges et al. 2006). The most reported bacteria in literature is *Bacillus pasteurii* (ATCC 6453) that was reclassified as *Sporosarcina pasteurii* (ATCC 11859). It is an alkalophillic bacterium able to hydrolyze urea within a short period due to its high urease activity (Ciurli et al. 1996, Bachmeier et al. 2002, Ng Wei et al. 2014).

Biogrouting procedure is usually based on three main steps including (1) introduction of bacterial suspension solution, (2) injection of calcifying solution containing urea and calcium ions and (3) recovery of by-products by flushing (Whiffin et al. 2007, Van Paassen 2011, Cheng and Cord-Ruwisch 2012, Cheng et al. 2013), Esnault-Filet et al. (2016). The bacterial metabolic activity uses urea as a source of energy and raises the pH locally as result of ammonia production. MICP occurs according to chemical reaction completed in few hours as a result of enzymatic hydrolysis of urea in the presence of calcium salts following two stages (Kroll 1990, Stocks-Fischer et al. 1999, Bang et al. 2001, Ramakrishnan et al. 2001):

1. **Urea hydrolysis stage:** 1 Mole of urea is hydrolyzed to produce 1 Mole of carbonates and 2 Moles of ammonium ions (Equation 1).
2. **$CaCO_3$ precipitation stage:** Calcium ions (Ca^{2+}) (derived from calcium chloride) reacts with carbonate ions (CO_3^{2-}) to form 1 Mole of calcium carbonates ($CaCO_3$) crystals (Equation 2).

$$CO(NH_2)_2 + H_2O \rightarrow 2NH_4^+ + {CO_3}^{2-} \qquad [1]$$

$$Ca^{2+} + {CO_3}^{2-} \rightarrow CaCO_3 \downarrow \qquad [2]$$

3 FACTORS CONTROLLING MICP EFFICIENCY

As the effectiveness of this biogeotechnology depends directly on the spatial distribution of the precipitated calcite and $CaCO_3$ crystallographic patterns (DeJong et al. 2010), factors influencing MICP treatment such as urease activity, availability of nucleation sites, pH level, temperature, degree of saturation, concentration of reagents solutions, and soil gradation curve must be controlled and well understood.

Geometric compatibility between microbes and the soil in which they are injected is a key factor. The lower bound limit of particle size is relative to microbe size (between 0.5 and 3 µm) and was set as silt (Mitchell and Santamarina 2005). Moreover, ex-situ mixing of microbes and nutrients with soil might extend the application of the technique to clays (Fritzges et al. 2006).

A relationship between the initial soil pH and the solubility of $CaCO_3$ crystals was pointed out by Cheng et al. (Cheng et al. 2014). MICP begins at pH level of 8.3 and increases to 9 where urease activity is high (Stocks-Fischer et al. 1999). Moreover, stable and continuous $CaCO_3$ production is directly linked with the cell growth and urease enzymatic activities (Hammes et al. 2003, De Muynck et al. 2008).

As temperature affects urease activity of microorganisms, nucleation, growth rates of calcium carbonates crystals and $CaCO_3$ solubility (Nemati and Voordouw 2003, Rebata-Landa 2007), Cheng et al. (2014) investigated the impact of room temperature on the strength of biocemented sand samples. Although, the amount of produced $CaCO_3$ crystals was higher at 50 ˚C, biotreated sand specimen strength was greater at 25 ˚C. This observation demonstrated that the localization and form of the precipitates is as important as their quantity.

Moreover, the rate of calcite precipitation is controlled by biochemical aspects such as the injection procedure and the concentration of chemical reactants (Kakelar et al. 2016). To ensure a successful ground improvement by biomineralization, the injection and retention of bacteria inside soil matrix are important. Indeed, only the retained bacteria in the soil can induce $CaCO_3$ precipitation from a solution of cementation. Three main treatment methods were investigated in the literature: (1) alternate injection of reactant solutions in saturated soils (Whiffin et al. 2007, Harkes et al. 2010, Al Qabany et al. 2012), surface spraying or percolation in non-saturated soils (Stabnikov et al. 2011, Cheng and Cord-Ruwisch 2012, Chu et al. 2012), and (3) premixing method (Yasuhara et al. 2012, Zhao et al. 2014). Figure 2 illustrates an example of a scanning electron microscopy (SEM) image of sand specimen treated by surface percolation where 6.7% of $CaCO_3$ was precipitated (Waldschmidt 2017).

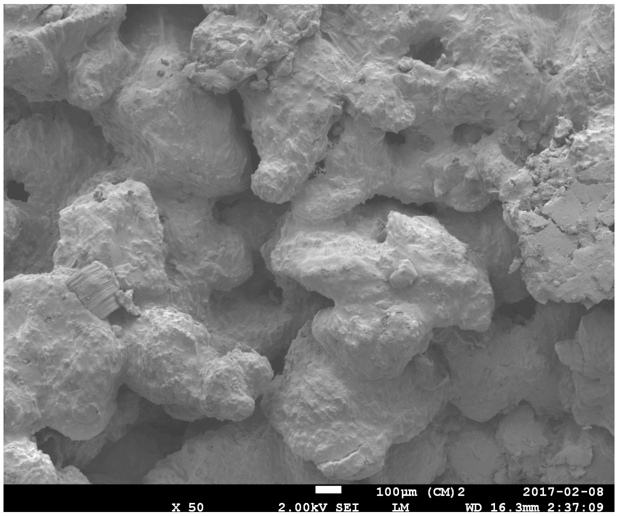


Figure 2. Scanning electron microscopy (SEM) image of sand treated by MICP using pre-mixing method (Waldschmidt 2017).

Homogeneous crystal distribution along sand specimen was observed at lower cementation solution concentration (Al Qabany and Soga 2013, Cheng et al. 2014, Ng Wei et al. 2014).

Cheng et al. (2013) showed that MICP works better at lower degree of water saturation, i.e. 20%, as the calcite crystals are formed at effective locations of particle to particle contacts.

4 IMPROVED ENGINEERING PROPERTIES OF SOILS

The biocalcification process relies on the creation of bonds at particle to particle contacts. This mechanism helps to strengthen and improve the mechanical performances as calcite precipitation results in a decrease in the pore space and an increase in solid content (DeJong et al. 2010). Several characteristics of biotreated soils are modified namely strength, rigidity, permeability and resistance to liquefaction.

In laboratory, biocalcification has demonstrated its efficiency to improve strength of loose sand and silt (Montoya and DeJong 2015). Ng Wei et al. (2014) demonstrated that this parameter is increased by a factor of 1.4 to 2.6 for silty soils treated using *Bacillus megaterium*. Geotechnical soil parameters such as cohesion (c) and internal friction angle (φ) are a function of the calcite content (Chou et al. 2012, Cheng et al. 2013, St-Onge 2016).

Many researchers used the unconfined compressive strength (UCS) to describe the strength of biotreated sands (Whiffin et al. 2007, Harkes et al. 2010, Cheng et al. 2013, Chu and Ivanov 2014, Ivanov et al. 2015, Waldschmidt 2017). Results showed that the amount of calcium carbonates is related to the strength of the treated specimen. Moreover, the stiffness, or soil elastic modulus (*E*), is improved using MICP technique (Cheng et al. 2013) and the small-strain shear stiffness is increased as bio-mineralization occurs at particle to particle contacts (Martinez 2012).

Biomineralized sand soils better resist to liquefaction and show enhanced dynamic properties compared to untreated specimen (Mortensen 2012, Montoya and Dejong 2013, Zhang et al. 2015).

From a hydraulic point of view, MICP technique improves soil strength while preventing the development of excess pore water pressure as biotreated sands conserve good drainage abilities (Esnault-Filet et al. 2016). Finally, biocalcification inhibits leaching of finer particles within soil skeleton when submitted to water flow seepage which prevents internal erosion and suffusion to occur (Ning-Jun et al. 2017).

5 MULTI-SCALE APPLICATIONS

Several field trials and up-scaled experiments were performed to validate the effectiveness of MICP in site conditions.

The first full scale attempt was performed in the Netherlands to treat the Rotterdam port area in 2004 (Mujah et al. 2016). The application of MICP reduced successfully the permeability of a sandy material and the bio-treatment showed good long-term performances (Hongzhi 2007).

A step-wise approach was followed by Van Paassen et al. (2009) to scale up biocalcification from 1 m^3 to 100 m^3 sand specimen. First, a box container was set up to mimic an injection well. The dimensions of the container filled with sand were 0.9 m x 1.1 m x 1 m and the container had drainage filters on its sides. A bacterial suspension and 0.5 M urea/calcium chloride reagent solutions were injected at the center of the box at a constant flow rate. A total volume of 3 500 L of cementation solution was flushed sequentially through in 8 batches during 50 days. The cubic meter container is illustrated in Figure 3.

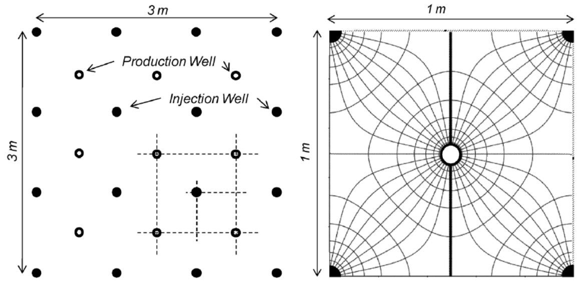
Afterwards, biocalcification was tested at larger scale as 100 m^3 of Itterbeck sand was biocemented in a large container using 100 L of inoculum. A total volume of 100 m^3 of a reagent solution containing 1 M of urea and calcium chloride were flushed during 12 days using injection/extraction wells. The implementation of the MICP technique was successful and 43 m^3 of the sand particles in the large container were bonded. The results also showed that the strength was remarkably increased following MICP treatment. However, the amount of the precipitated calcite was spatially dispersed. These observations were justified by several scenarios including heterogenous transport of reagents and preferential flow paths which leads to higher content of $CaCO_3$ compared to other areas.

Figure 3. The cubic meter bio-grout experiment performed by van Paassen(2009) (picture shared by van Paassen Leon).

Another field test was performed by Van Paassen (2011) to find a solution for borehole instability when installed in gravel. This field test was preceded by laboratory tests on a 3 m^3 container filled with gravel. Horizontal directional drilling in this container demonstrated the success of the treatment and an upscale in field was performed. A total soil volume of 1 000 m^3 was treated at depth varying between 3 and 20 m below the surface. The biotreatment required 200 m^3 of bacterial suspension and 300 to 600 m^3 of cementation solutions containing urea and calcium chloride. Note that the groundwater was extracted until ammonium concentrations measurements were equal to initial values. During this step, the pumped water was transferred to a local waste water treatment plant. The results showed that the gravel layer remained stable during drilling process, and laying gas pipeline was performed without any collapse (Figure 4).

Figure 4. Bio-cementing of gravel for borehole stability field project (picture shared by van Paassen Leon).

De Jong et al. (2014) developed a three-dimensional treatment method to implement MICP at field scale. The method is based on a repeated five-spot injection/extraction well pattern for treatment of 3m by 3m by 0.15m experimental layout (Figure 5.a). Each spot pattern is made up of one injection well placed at the core and one production well at each corner of the targeted treatment zone (0.5m by 0.5 m by 0.15 m) as illustrated in figure 5.b. The experiment aims to treat Ottawa 50-70 sand and was performed by injection of solutions into a saturated sand. A two-phases MICP treatment was implemented. The first phase consisted on the re-circulation of 30 L of *S. Pasteurii* suspension in a urea-rich solution for 50 non-continuous hours (the treatment was stopped during the night). The second phase consisted in two stopped-flow cycles where a calcifying solution was injected at high flow rate during 1 hour followed by 2 hours of rest period. The first cycle was performed in the same flow direction as the injection of bacteria, while the second was performed in the reverse direction (Martinez 2012). A uniform treatment was achieved experimentally even under highly active microbial conditions as clogging at injection well was prevented using the two stopped-flow cycles.



(a) Well Pattern

(b) Stream and Potential Lines

Figure 5. The repeated five-spot well pattern designed by De Jong et al. (2014): a) Plan view of 3m by 3m layout and b)Theoretical stream and potential lines for target treatment zone of 0.5 by 0.5 by 0.15 m zone.

Gomez et al. (2015) performed a field study focusing on the surface application of MICP to prevent erosion of loose sand deposits at a mine site location in the province of Saskatchewan in Canada. A depth of 28 cm of soil was improved and the formation of cemented crust of about 2.5 cm thick increased resistance to erosion. MICP appears to be a promising technique to treat larger-scale fields. The results pointed out that low-concentration solutions achieved greatest improvement compared to high and medium concentrations.

Soletanche-bachy, a French contractor, developed its own method for industrial implementation of biomineralization process. The process is called Biocalcis®. In 2009, the concept of the industrial process was validated through a pilot test that was performed in the Netherlands, in partnership with VSF and Deltares (Figure 5). This pilot test led to feasibility method definition and costs estimation (Filet et al. 2012).

Figure 5.100 m^3 container provided by VSF company and used by Soletanche-bachy (Esnault-Filet et al. 2015).

Esnault-Filet et al. (2016) successfully used Biocalcis® to treat sandy-silt material at field scale in the south of France. Signs of corrosion started to appear in the reinforcements of a retaining wall located beneath the abutment of a motorway interchange bridge. The site was in a crowded urban area with a very difficult access under the bridge abutment due to the presence of a tramway passing just beneath and private buildings in the vicinity. The wall was constructed in the 70's and was made of compacted backfill reinforced by sub-horizontal steel tensile rods. The facing system was made of precast concrete panels equipped with embedded connections to fix the steel reinforcement rods. No interruption in motorway traffic flow or stopping the tramways was allowed during field works. Moreover, the site was in urban area which did not allow the implementation of conventional techniques. Soil nailing was prohibited as available space in front of the wall was insufficient for placing drilling machine. On the other hand, jet grouting couldn't be considered as it might generate stability risks on the structure in case of high pressure build up. Finally, the backfill was composed of a compacted material of very low permeability (Sandy silts matrix which permeability was lower than 10^{-6} m/s). Face with these constraints, Biocalcis was proposed. The feasibility was first confirmed after successful laboratory and pilot tests using real site material. The treatment was achieved by 23 horizontal walls having 5 m width, distributed over 3 lines of injection with a drainage line at the base of the injected zone (Figure 6). The total volume was equal to 100 m^3 over 3 m height and 6 m length. The final results were estimated by in-situ coring and pressiometric tests. These tests confirmed the feasibility of the procedure for the reinforcement of the wall, and allow proposals for the final solution which consisted of a biocalcified block working as a gravity wall.

All those experiments allowed to highlight advantages/challenges restricting the implementation of biogeochemical soil improvement processes into field.

Figure 6. Reinforcement of soil retaining wall using Biocalcis® at field scale (Esnault-Filet et al. 2015).

6 LIMITATIONS ENCOUNTRED WHILE UPGRADING TO FIELD USE AND RECOMMANDATIONS

From these few large-scale experiments, several limitations of the MICP upscaling must be addressed:

- **By-products:** Ammonium and nitrate are by-products of urea hydrolysis. The generation of high concentrations of these compounds induces toxic effects on human health, vegetation, atmospheric nitrogen deposition (van Paassen et al. 2010, Tobler et al. 2011, Dhami et al. 2013). Terrestrial ecosystems are, by consequence, exposed to eutrophication and acidification. Those by-products must be properly controlled and eventually treated during the *in-situ* implementation of biogrounting to follow environmental legislative norms (Mujah et al. 2016, Wang et al. 2017). Substantial volumes of chemical reagents and microbial solutions are generated during MICP process especially for field applications. A treatment based on flushing is thought to get rid of those by-products taking in consideration the fate and transport of by-products. Note that the water is usually extracted until the electrical conductivity and ammonium concentrations are back to initial values (Van Paassen 2011). Some authors also suggest the reuse of ammonia-rich effluents as fertilizer for plants (Dejong et al. 2013, Wang et al. 2017).

- **Cost:** The biogeochemical process is material consuming as it could require about 88 kg of $CaCl_2$ and 96 kg of urea per 1 m^3 of sand to produce the content of precipitated calcium carbonates of 75-100 g/kg of sand, which can cost up to 41 $/$m^3$ (Ivanov and Stabnikov 2017). At a large scale, the technology is expensive but applicable to geotechnical applications (van Paassen et al. 2010). The cost of calcium reagent and urea are higher than conventional cement and implementation of the process requires the preliminary investigations at small and pilot scale before upgrading to field scale which inflates costs (Van Paassen 2011, Esnault-Filet et al. 2012, 2016). Moreover, injection and extraction wells could represent a non-negligible part of the final cost. The method total cost of MICP treatment (materials, equipment, and installation) in saturated soils ranges from 25-75 \$US/m^3 to about 500 \$US/m^3 depending on the quantity of $CaCO_3$ (Dejong et al. 2013, Wang et al. 2017).

- **Feasibility:** Parameters such as injection flow rate, number of treatments, volumes, concentrations are all key factors that control the success of MICP. These parameters must be analyzed in laboratory, which can be time- and cost consuming. Moreover, clients are easily prone to use conventional soil improvement techniques as all parameters are controlled and have shown their efficiency over years. The advantage to use bio-geochemical-based soil improvement technologies is that they are natural and non-intrusive/disturbing for existing structures (Filet et al. 2012, 2016). Nevertheless, some activities designed to raise awareness and industry training may be needed. Also, statistical studies must be conducted including rigorous assurance/quality control process, monitoring operations during treatment and maintenance norms should be considered for re-treatment/healing processes.

- **Performance:** Models for time- and cost effectiveness optimization have been assessed aiming *in-situ* implementation of biogrounting (Weil et al. 2012, Gomez et al. 2015, Terzis and Laloui 2017). The homogeneity of treatment along the soil matrix remains one of the weakness; although researchers have progressed a lot in this area. Nevertheless, a uniform treatment could be achieved when controlling variables will be fully understood including number of injections, method of injection, concentrations of reactants and flowrate of injection. All these parameters might be fixed depending on field conditions, targeted applications and preliminary results at laboratory scale.

- **Lifetime service**: The biochemical treatment for a specific application requires a durability in accordance with its service life requirements. MICP is expected to be stable for more than 50 years if alkaline conditions are provided (Montoya and Dejong 2013) and an occasional retreatment can be applied to extend this service life. Studies on longevity of treatment while in contact with acid rain precipitations demonstrated that no large erosion occurs. Only 0.7g of weight loss was measured for sand columns after being flushed with 12 L of acid rain volume corresponding to 5 years' rainfall (1000mm/year). Sand columns were treated under fully saturated conditions with $CaCO_3$ content of about 0.1-0.105 g/g sand (Cheng et al. 2013). Nevertheless, the calcite must be assessed to evaluate its long-term degradation. Also, the application of this technology might target regions of the world where factors such as temperature, pH, weather are favorable for its implementation such as deserts.

7 CONCLUSIONS AND SUMMARY

Soil improvement techniques based on biomineralization have grown over the past 15 years and innumerable results proved the applicability of these techniques to tackle geotechnical issues (optimization of the bio-geochemical treatment procedure, controlled key factors influencing the process, geotechnical applications, numerical modeling). Research should now focus on testing and modeling in-situ conditions and considering practical needs in terms of sustainability, costs, performance, feasibility and life service.

Opportunities and challenges for geotechnical applications were identified in this work to open discussions for future possibilities.

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