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Bio-cementation through controlled dissolution and recrystallization of calcium carbonate

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HIGHLIGHTS

• Limestone dissolved in lactic acid was used as calcium source for bio-cementation.

Consolidation of sand was achieved through enzyme induced carbonate precipitation.

• Solutions containing lactate gave rise to spherical shaped calcite crystals.

• Limestone in aggregate resulted in small, scattered precipitated calcite crystals.

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ABSTRACT

We present an approach to bio-cementation of sand where the calcium source is prepared by dissolving powdered limestone (chalk) in lactic acid. Cementation is achieved through enzyme induced carbonate precipitation (EICP) with Jack Bean urease. The real-time nucleation and growth of crystals, crystal morphology and mechanical strength of consolidated samples was studied for dissolved chalk solution as well as calcium chloride (CaCl₂) and calcium lactate solutions. Solutions containing lactate were found to yield spherical calcite crystals. The compressive strengths of consolidated samples ranged from 0.06 to 2.8 MPa, increasing with the amount of precipitated CaCO₃.

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1. Introduction

Cement is an important component in construction materials. It is used as a binder in concrete and as a component in mortar for masonry. The cement industry accounts for approximately 5% of global anthropogenic carbon dioxide (CO₂) emissions [1,2]. The calcination of limestone and the combustion of fossil fuels are responsible for about 90% of CO₂ emitted from the cement industry. During the calcination process, limestone (CaCO₃) is thermally decomposed into lime (CaO) at high temperatures. For example, Portland cement and magnesium cement are produced by heating limestone to 1450 °C and 750 °C, respectively [3]. This process not only releases a lot of CO₂ from the material itself, but also involves high energy consumption.

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https://doi.org/10.1016/j.conbuildmat.2018.02.059 0950-0618/© 2018 Elsevier Ltd. All rights reserved. Due to the increase in public and industrial environmental awareness, several approaches have been introduced in recent years to minimize the environmental impact caused by the cement industry. One of the most effective methods for reducing emissions and energy use in cement production today is to replace a portion of the Portland cement with pozzolanic materials of natural (volcanic) or industrial (fly ash, blast furnace slag) origin [4]. New cement materials such as energetically modified cement [5] and bio-cement [6] have also been introduced over the last few decades, in an effort to reduce the energy consumption and environmental pollution by producing cement via alternative routes.

Bio-cement is a material that is produced via a biological approach. To date, the most commonly reported system of biocement is based on ureolytic bacteria that produce the enzyme urease as a metabolic product. In the presence of urea and a calcium source, urease hydrolyzes urea to form ammonia and CO_2 . The produced ammonia increases pH and CO_2 is transformed into carbonate ions. This leads to precipitation of calcium carbonate







 $(CaCO_3)$. The mechanism is known as microbially induced carbonate precipitation (MICP) [7,8]. When this takes place inside a granular material, such as sand, the formed crystals can act as a binder between grains to achieve cementation. The resulting material is a porous, sandstone-like material that can be used as bricks, for ground stabilization and could be considered for other application where low-strength concrete is used today, but should not be in direct contact with steel reinforcement due to the low pH (around 8.6) of the calcium carbonate binder.

Calcium chloride (CaCl₂) is often used as the calcium source in MICP. However, a major drawback of CaCl₂ is the excessive production of chloride ions that may lead to corrosion of the steel reinforcement used in concrete. MICP can also be achieved using other calcium salts such as calcium lactate [9,10], calcium nitrate [9,11,12] and calcium acetate [10,12], which reduce the unfavorable effects of chloride ions on concrete durability.

In search for low cost alternatives to pure calcium salts, some studies have used calcium ions from sources such as limestone [13,14] and eggshells [15], dissolved using organic acids, for the MICP process. Limestone, which is primarily composed of CaCO₃, is one of the major components in conventional cement. Due to its low cost and high global abundance, it would be a great advantage if it could be used as a major binder in concrete without the need to first decompose it at high temperatures.

In this paper, we present a two-step process to achieve cementation by dissolution and recrystallization of limestone, as illustrated in Fig. 1. Our final aim is to use bacteria to both produce organic acids for limestone dissolution and urease enzyme for carbonate precipitation. However, in order to investigate the feasibility of the chemical processes involved, we present a simplified setup where we use reagent-grade lactic acid and commercial plant-derived urease from Jack Bean (*Canavalia ensiformis*).

The use of purified urease to precipitate $CaCO_3$ is known as enzyme induced carbonate precipitation (EICP). EICP offers several advantages over MICP. It eliminates the need for cultivation of, and effort to sustain, the bacteria. The use of plant-derived urease makes the system less susceptible to bio-plugging, due to its smaller size (\sim 12 nm) [6,7]. Furthermore, plant-derived urease is readily available in the market and will degrade after use, while in situ mirobial production of urease will leave the microorganisms behind in the material.

The process is outlined as follows (Fig. 1): First, the calcium source is obtained by dissolving powdered limestone (chalk) in lactic acid to form a dissolved chalk solution (DCS):

$$CaCO_3 + Lactic \ acid(HLact) \rightarrow Ca^{2+} + HCO_3^- + Lact^-$$
(1)

Next, the DCS is mixed with equivalent amounts of urea and added into a column with sand and urease. The urease starts to hydrolyze the urea, producing bicarbonate ions and increasing pH:

$$(NH_2)_2CO + 3H_2O \rightarrow 2NH_4^+ + HCO_3^- + OH^-$$
 (2)

This causes the dissolved CaCO₃ to re-precipitate:

$$Ca^{2+} + HCO_3^- + OH^- \rightarrow CaCO_3 + H_2O$$
(3)

In order to investigate the performance of the DCS as an alternative calcium source for bio-cementation, we also performed experiments with solutions made by dissolving commercial salts, i.e. CaCl₂ and calcium lactate.

In the first part of the paper, we describe real-time in situ monitoring of CaCO₃ precipitation and crystal growth from different calcium sources using an optical microscope. The structure and morphology of the CaCO₃ crystals were characterized via Raman spectroscopy and scanning electron microscopy (SEM). In the second part of the paper, we report on consolidation of sand using the same approach. Different processing parameters can have substantial impact on the properties of the final consolidated product. Thus, the effects of different calcium sources and number of injections during the consolidation experiment were studied. Powdered limestone was also added in some of the samples to investigate the

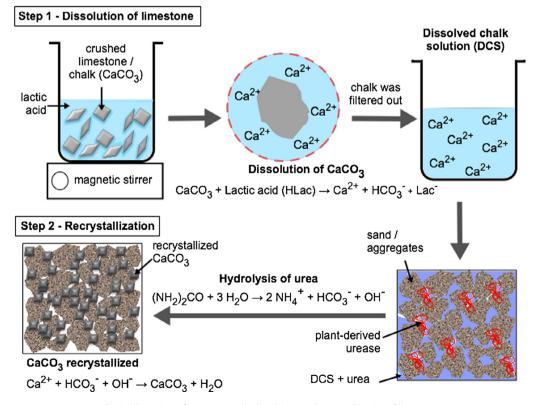


Fig. 1. Illustration of cementation by dissolution and recrystallization of limestone.

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