



A study on the cementation interface of bio-cement

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ABSTRACT

X-ray diffraction (XRD) analysis was carried out on a new kind of bio-cement based on phosphate-mineralized microbes to confirm the composition of phosphate products, i.e. $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ and BaHPO_4 . Meanwhile, the bio-cement was researched in terms of the cementation interface. The results show that the interfacial bonding strength of bio-phosphate cement is lower than bio-calcite cement in the scratch experiment. The microstructure between calcite and loose sand particles was analyzed by X-ray photoelectron spectroscopy (XPS) and nuclear magnetic resonance (NMR). The results of XPS and NMR analysis confirm that the microbially-induced precipitation of minerals in loose sand particles means they interact with other particles to generate bonding effects, which plays a role in the binding function between loose sand particles and bio-cement.

1. Introduction

Conventional cementitious materials mainly include Portland cement, lime, gypsum, special cement, sodium silicate, etc. [1]. However, most of these materials are hazardous, except for sodium silicate. Portland cement is the most commonly used cementitious material, but it has high energy consumption, causes serious environmental pollution and has other shortcomings. Therefore, researchers have carried out basic research into cement production equipment, process technology, clinker composition optimization, supplementary cementing materials, prolonging cement service life and other aspects to understand direct energy-saving emission reduction during the cement preparation process, and the indirect energy-saving emission reduction when cement is in a high efficiency application. On the other hand, researchers are attempting to find new, alternative cementitious materials which are energy-saving, less polluting and have excellent durability.

Bio-cement, based on microbially-induced mineralization, has drawn much attention because of lower energy consumption and more abundant raw materials. Bio-calcite has been widely studied, i.e., the microbe cement can improve the mechanical properties and permeability of porous materials [2–5] or repair cement-based material and limestone [6,7], modify the properties of soil and sand [8,9], enhance oil recovery from oil reservoirs and can be used in bio-remediation [10,11]. However, more attention has been paid to the application of bio-cement, rather than the essence of cementation via bio-cement [12–15]. What is it about the microbially-induced precipitation in loose sand particles that makes the bio-carbonate/phosphate cement able to bind loose particles to sandstone? Can a chem-carbonate/phosphate

cement bind loose sand particles? There are currently no answers to these questions. Therefore, in this paper, loose sand particles are cemented by a microbial method and a chemical method to compare the loose particle binding effect. The bio-cement interface was taken into consideration, and the interfacial characteristics were investigated via an interface mineralization experiment using sheet glass. Meanwhile, the microcosmic interface between sand particles and bio-cement was also researched by XPS and NMR analysis, which aimed to shed light on the bonding effects of bio-cement.

2. Materials and Method

2.1. Bio-phosphate Cement

All raw materials were of analytically pure grade without further purification, and the deionized water was self-made. A kind of phosphate-mineralized microbe (P-bacteria for short) was selected to prepare the bio-phosphate cement.

A given amount of the substrate was completely dissolved in 100 mL of deionized water to the concentration of $0.2 \text{ mol}\cdot\text{L}^{-1}$, and its pH value was adjusted to about 9.0 using $0.1 \text{ mol}\cdot\text{L}^{-1}$ NaOH aqueous solution. 100 mL of $0.1 \text{ mol}\cdot\text{L}^{-1}$ CaCl_2 solution and 100 mL of $0.1 \text{ mol}\cdot\text{L}^{-1}$ BaCl_2 solution were also prepared prior to use. Then, 50 mL of substrate solution and 100 mL of saline solution (CaCl_2 or BaCl_2 solution) were successively mixed in the P-bacteria medium. After being aged for 24 h, the products were filtrated and washed three times with deionized water and ethanol, and then dried at 60°C for 12 h.

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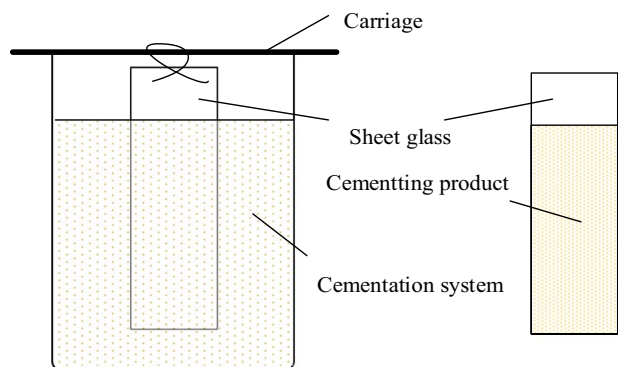


Fig. 1. Schematic of the interface mineralization experiment.

Table 1

The classification of different cementation system.

Cementation system	Serial number	Bacteria
Bio-calcite cement	BC	C-bacteria
Bio-calcium phosphate cement	BCP	P-bacteria
Bio-barium phosphate cement	BHP	P-bacteria
Chemical calcite cement	CC	Unused

2.2. Interface Mineralization Experiment

Smooth sheet glass (SiO_2) was used as a carrier to conduct the interface mineralization experiment. A schematic of the interface mineralization experiment is shown in Fig. 1. The sheet glass was firstly hung on the carriage and then vertically immersed in the cementation system. Three different kinds of bio-cement were researched and calcium carbonate synthesized by the chemical method was set as a control group, as shown in Table 1. After a certain deposition time, the glass surface was coated with cementing product and further investigated by the disbanding experiment of sonic oscillation and the scratch experiment.

2.3. Sand Particles- Bio-cement Interface

The loose sand column was respectively dealt with by the three different kinds of bio-cement, i.e., bio-calcite, bio-CP, and bio-HP cement. After being aged for the certain time, the consolidated sand was obtained. The respective chem-product was also investigated as a

control group. Then, the microcosmic interface between sand particles and bio-cement product was researched by the X-ray photoelectron spectroscopy and nuclear magnetic resonance.

2.4. Testing Method

The phase purity of products was examined by powder X-ray diffraction (XRD) with a Bruker D8-Discover diffractometer with $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$).

Scanning electron microscopy (SEM, FEI Company, Netherlands, operating voltage 20 kV) with an energy dispersive X-ray spectroscopy (EDS), were used to conduct morphological studies and to measure elemental compositions of the samples.

The scratch experiment using coating adhesion scratch tester (WS-2005) was used to determine the quantitative characterization of the interfacial bonding strength. A load of 10 N, a loading rate of 5 N/min, and a scratch rate of 5 m/min was set in the experiment.

All the XPS measurements were carried out on a VERSAPROBE PHI5000 with a resolution of 0.3–1.0 eV, using non-monochromatized $\text{Mg K}\alpha$ X-ray radiation as the excitation source. The calibration of binding energy scale for Si 2p photoelectron spectra was performed with the C 1s line (285.0 eV) from the carbon contamination layer. The XPS signals were analyzed using a peak synthesis program in which a nonlinear background is assumed and the fitting peaks of the experimental curve are defined by the Gaussian Method.

^{29}Si NMR experiment was performed on a Bruker Avance-400D spectrometer using 4 mm o.d. ZrO_2 rotors with a rotation speed of $5000 \pm 2 \text{ Hz}$. Proton high-power decoupling pulse sequences with $4.5 \mu\text{s}$ excitation pulses were employed to allow quantitative evolution of the ^{29}Al NMR spectra.

3. Results and Discussion

3.1. XRD Analysis of Bio-phosphate Product

The XRD patterns of the precipitation induced in the P-bacteria liquid are shown in Fig. 2a and b. Fig. 2a shows that all the peaks were in good agreement with the standard card of hydroxyapatite (JCPDS No. 09-0432). A relatively low crystallinity was obviously reflected by relatively broad and low intensity XRD peaks, which are marked by triplets or broad, intense diffraction bands ranging from 31.82° to 34.07° 2θ , and an individual intensive peak at 25.89° 2θ , characteristic of an apatite phase.

The barium hydrogen phosphate was confirmed by the XRD patterns as shown in Fig. 2b. By comparison, the characteristic peaks of BaHPO_4

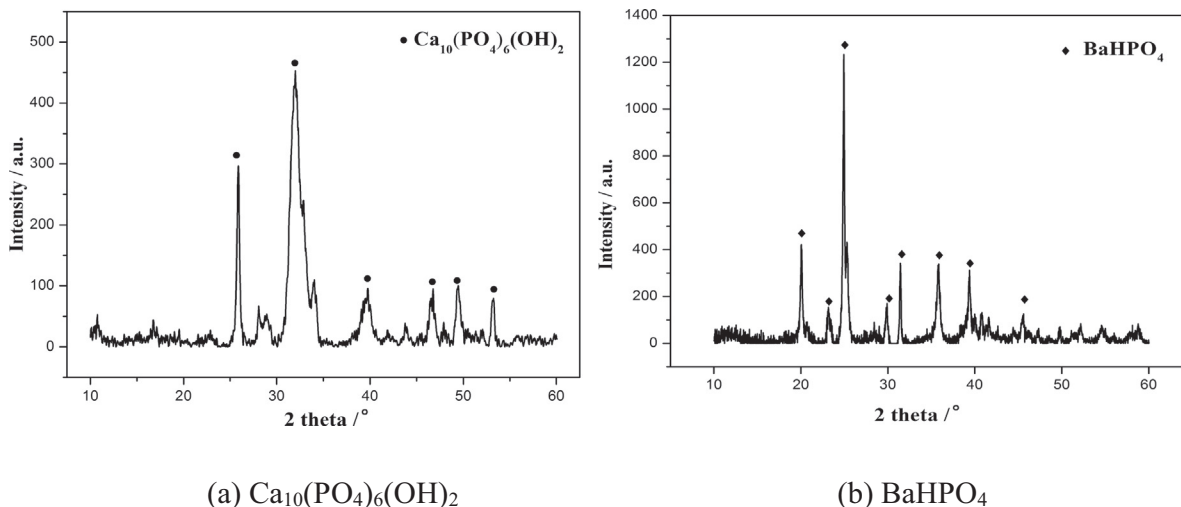


Fig. 2. The XRD patterns of phosphate precipitate obtained by microbial method.

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