



Loose sand particles cemented by different bio-phosphate and carbonate composite cement



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HIGHLIGHTS

- A new bio-cement was applied to bind loose particles into bio-sandstone.
- Loose particles can be cemented by different bio-phosphate and carbonate composite cement.
- Ammonia/ammonium can be effectively become into struvite in injection process.
- Permeability, porosity, strength and microstructure of the bio-sandstones were investigated.

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ABSTRACT

Loose sand particles could be cemented by the microbial induced phosphate and carbonate precipitation into a bio-sandstone with a certain mechanical strength when the *Sporosarcina pasteurii* contained $K_2HPO_4 \cdot 3H_2O$ and the mixture solution of urea and $MgCl_2$ in the 1:1 of volume ratio were injected by a peristaltic pump into quartz sand mold. The result shows that loose quartz sand particles are well cemented into a bio-sandstone, and the compressive strength of bio-sandstones is greater than 1.0 MPa. Ammonia/ammonium was produced during the hydrolysis of the urea and could be effectively become into environment-friendly struvite in the cementation process. In addition, the ingredients of the bio-sandstones were mainly quartz sand, struvite and hydromagnesite through XRD analysis. The internal microstructure of bio-sandstone was observed by SEM and the bio-phosphate and carbonate composite cement between sand particles indicated the shape was mainly irregular particles. The solidified layer of the surface of dust and sand particles was formed after spray bio-phosphate and carbonate composite cement, and wind erosion rate of dust and sand is $0 \text{ g/m}^2/\text{h}$ and $0 \text{ g/m}^2/\text{h}$, respectively.

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1. Introduction

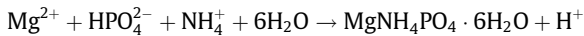
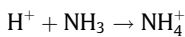
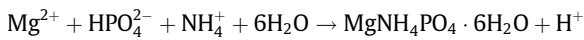
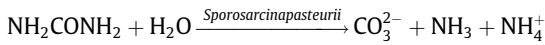
Loose particles are cemented by the microbial induced carbonate precipitation has been widely studied via scientists [1–6]. Compared with conventional cementitious materials (cement, lime, gypsum, sodium silicate, epoxy), microbe cement has low viscosity, permeability, and reaction rate, crystal size and compressive strength are controllable. Moreover, eco-compatibility between microorganisms and sandy soil is good, and microorganisms are relatively inexpensive. Urea can be rapidly hydrolyzed by urease which can be secreted via microorganisms, and then ammonia/ammonium and carbonate ions are obtained [7,8]. Different carbonate precipitation has been synthesized by calcium, magnesium

and other ions reacting with carbonate ions, and bacterial cells may also be provided as a nucleation site. In carbonate deposition process, a large number of ammonia are released, and then an alkaline environment is formed which can increase the dissolution of inorganic carbon [9,10]. Two moles of ammonia can be produced by hydrolyzing one mole of urea in theory, and the environment will be polluted because released ammonia cannot be completely dissolved in the solution. Therefore, a lot of money will be taken to handle effluent that is not conducive to large-scale application in the actual project [11].

Loose particles can be cemented by bio-phosphate and carbonate composite cement into a whole, and which has a certain mechanical properties. Ammonia/ammonium (NH_3/NH_4^+) can be changed into environment-friendly struvite in the cementation process. The formation mechanism of struvite ($NH_4MgPO_4 \cdot 6H_2O$ or $MgNH_4PO_4(H_2O)_6$) can be explained by the following steps:

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Therefore, struvite and carbonate are used to bind loose particles. Based on cementation property of struvite and carbonate, the microbe cement can be applied to dust-fixing, foundation reinforcement, slope, consolidation of loose sandy soil, etc [12–14].

2. Experiments and methods

2.1. Materials

Sporosarcina pasteurii was selected to apply for cementation experiment. Microbe having OD₆₀₀ value of 1.2 and enzyme urease activity value of 2.2 μmol urea/min was used in this study. Cultivation of the organism was conducted in a medium containing 15 g/l peptones, casein and 5 g/l soya peptone. In general, the harvest microorganism was stored at 4 °C prior to use. The mixture solution of urea (1 mol/l) and MgCl₂ (2, 2.5 and 3 mol/l) was prepared before use. 1, 1.5 and 2 mol of K₂HPO₄·3H₂O was completely dissolved in 1 L of *Sporosarcina pasteurii* prior to use. The aggregate used in this study was less than 420 μm of quartz sand.

2.2. Microbial induced different magnesium carbonate and struvite precipitation

100 ml of mixed solution of urea (1 mol/l) and MgCl₂ (2, 2.5 and 3 mol/l) was respectively poured into 100 ml of *Sporosarcina pasteurii* liquid contained K₂HPO₄·3H₂O (1, 1.5 and 2 mol/l), and the mixture solution was allowed to stand under static conditions for 6 h at ambient temperature of 25 ~ 30 °C. Finally, three of different mixture of struvite and magnesium carbonate were obtained by removing away the supernatant of precipitations.

2.3. Cementation experiment

PVC pipes (internal diameter 5.0 cm, length 15.0 cm) were used in this study. Loose quartz sands were added to PVC pipe, and a layer of approximately 1.0 mm of gauze was placed at the bottom and top of the PVC pipe. The tube was closed and positioned vertically. Cementation solution was injected into sand column by a peristaltic pump. All experiments were performed at ambient temperature of 25–30 °C.

100 ml of *Sporosarcina pasteurii* liquid contained K₂HPO₄·3H₂O (1, 1.5 and 2 mol/l) was firstly injected in sand column. Next, 100 ml of the mixed solution of urea (1 mol/l) and MgCl₂ (2, 2.5 and 3 mol/l) was injected into the sand column. After standing 6 h, 100 ml of *Sporosarcina pasteurii* liquid contained K₂HPO₄·3H₂O (1, 1.5 and 2 mol/l) was injected to sand column, and then 100 ml of mixture solution of urea (1 mol/l) and MgCl₂ (2, 2.5 and 3 mol/l) was injected. Above steps were repeated until no fluids leak out, indicating that the cementation process was over. The number of injections of bio-sandstones cemented by mixture solution of urea (1 mol/l) and MgCl₂ (2, 2.5 and 3 mol/l) and *Sporosarcina pasteurii* liquid contained



Fig. 1. Loose sand particles to bio-sandstone cemented by bio-phosphate and carbonate composite cement.

K₂HPO₄·3H₂O (1, 1.5 and 2 mol/l) is 6, 4 and 3, respectively. After casting, specimens were kept at 30 ± 2 °C until the test ages were reached and the state before and after cementing loose sand particles are given in Fig. 1.

2.4. Characterization

The phase purity of products was examined by powder XRD with Bruker D8-Discover diffractometer by graphite-monochromatized high-intensity Cu Kα radiation (λ = 1.5406 Å). Scanning electron microscope (SEM, FEI Company, Netherlands, operating voltage 20 kV) was used to conduct morphological studies of the samples. The compressive strength of bio-sandstones was tested by computer-controlled electronic universal testing machine (SANS CMT 8502) with a loading rate of 1 mm/min.

The permeability coefficient change of the sand was characterized by Darcy's law [15],

$$Q = \frac{k\Delta h S}{L}$$

where Q is the volumetric flow rate (cm³/s), k the material permeability (cm/s), Δh the constant water head (m), S the sample section presented to the flow (cm²) and L the length of the flow path (cm, in this case the sample height).

The average porosity of bio-sandstones was estimated using the formula [16],

$$P = \frac{M_{sat} - M_{dry}}{\rho_w V_{vol}}$$

where P is the porosity of bio-sandstones, M_{sat} the weight of the bio-sandstones as it was fully saturated with water (g), M_{dry} the weight of the dried bio-sandstones (g), V_{vol} the volume of bio-sandstones (cm³), ρ_w the volume density of water (1.0 g/cm³).

3. Results and discussion

3.1. XRD patterns and SEM images of the mixture of struvite and different magnesium carbonate

Fig. 2 shows XRD patterns of different mixture of struvite and magnesium carbonate. The precipitates are mainly the mixture of MgCO₃(H₂O)₃ and NH₄MgPO₄·6H₂O when 100 ml of *Sporosarcina pasteurii* liquid contained K₂HPO₄·3H₂O (1 mol/l) reacted with 100 ml of the mixture solution of urea (1 mol/l) and MgCl₂ (2 mol/l). The standard XRD patterns of the reported structures of MgCO₃(H₂O)₃ and NH₄MgPO₄·6H₂O used are JCPDS No. JCPDS No. 70-1433 and 15-0762, as shown in Fig. 2a. XRD of samples analysis confirm that ingredients of products are mainly MgCO₃·2H₂O (JCPDS No. 18-0768) and MgNH₄PO₄(H₂O)₆ (JCPDS No. 71-2089) when 100 ml of *Sporosarcina pasteurii* liquid contained K₂HPO₄·3H₂O (1.5 mol/l) (Fig. 2b). When 100 ml of *Sporosarcina pasteurii* liquid contained K₂HPO₄·3H₂O (2 mol/l), the precipitates are mainly the mixture of MgHPO₄(H₂O)₃, MgCO₃·2H₂O and MgNH₄PO₄(H₂O)₆, which corresponding to the standard card is JCPDS No. 70-2345, 18-0768 and 77-2303 (Fig. 2c). Above results show that ammonia can be effectively changed by K₂HPO₄·3H₂O into environmentally friendly struvite. Therefore, the mixture solution of urea and MgCl₂ and *Sporosarcina pasteurii* liquid contained K₂HPO₄·3H₂O are used to bind loose sand particles in the next experiment.

SEM images show that the morphology of different mixture of struvite and magnesium carbonate (Fig. 3). The shape of mixture of MgCO₃(H₂O)₃ and NH₄MgPO₄·6H₂O, MgCO₃·2H₂O and MgNH₄PO₄(H₂O)₆, and MgHPO₄(H₂O)₃, MgCO₃·2H₂O and MgNH₄PO₄(H₂O)₆ is irregular particles with nonuniform size. The length and width of mixture of MgCO₃(H₂O)₃ and NH₄MgPO₄·6H₂O, MgCO₃·2H₂O and MgNH₄PO₄(H₂O)₆ are less than 100 μm (Fig. 3a–d). However, the size of the mixture of MgHPO₄(H₂O)₃, MgCO₃·2H₂O and MgNH₄PO₄(H₂O)₆ is 100–200 μm, as shown in Fig. 3e and f. Therefore, the size of the mixture of MgHPO₄(H₂O)₃, MgCO₃·2H₂O and MgNH₄PO₄(H₂O)₆ is bigger than mixture of MgCO₃(H₂O)₃ and NH₄MgPO₄·6H₂O, MgCO₃·2H₂O and MgNH₄PO₄(H₂O)₆.

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