Enhancing potential of hydrofracturing in mylonitic coal by biocementation

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Funding information
State Key Laboratory Cultivation Base for Gas Geology and Gas Control (Henan Polytechnic University); Natural Science Foundation of Chongqing, Grant/Award Number: cstc2018jcyjJA2664; National Natural Science Foundation of China, Grant/Award Number: 51604051

Abstract
Mylonite coal is a representative of tectonically deformed coal and is a result of crushing original coal into fine coal grains under strong shear or long-low tectonic stress. Because of its granular nature and the resultant inferior mechanical property, it is difficult to initiate fluid-driven fractures within mylonitic coal reservoirs for enhancing coalbed methane recovery. The following explores a biomineralization method of microbially mediated calcium carbonate precipitation (MICP) to enhance the structural integrity and mechanical strength of mylonitic coal, enabling potential success for hydrofracturing. The experimental results indicate that the mechanical properties of mylonite coal are significantly enhanced after a short period of MICP treatment with ten cycles of treatments yielding a maximum uniaxial compressive strength (UCS) of 8.7 MPa and a maximum brittleness index of 0.218 approaching that of the hard coal. The increments in UCS and brittleness of biocemented mylonite coal show a positive correlation with the generated calcium carbonate content. Scanning electron microscopy (SEM) imaging indicates that the generated calcium carbonate precipitates first randomly occur on the particle surfaces, and then occupies the interstitial space until particle-particle bonds are developed. The irregular morphology of coal particles results in two contact relations between particles, point contact and planar contact, causing a significant difference in biocementation effectiveness. Two microfailure patterns of biocemented coal with uniaxial compression are observed. One is that the coal particles are crushed, and the other occurs at the biocemented interface between the coal particles and calcium carbonate crystals.

KEYWORDS
biomineralization, hydraulic fracturing, microbial cementation, microbially induced calcium carbonate precipitation, mylonitic coal

1 INTRODUCTION

Hydraulic fracturing has been employed to enhance coalbed methane (CBM) recovery for several decades.1-3 The primary goal of hydraulic fracturing is to initiate the fluid-driven fractures that can propagate continuously within reservoirs, providing permeable flow paths for methane.4 More importantly, the fluid-driven fractures can effectively reduce the in situ
stress on the coal matrix and generate gas pressure gradient from the coal matrices to the fracture pathway, thereby promoting both the gas desorption and convection. The exploitation of coalbed methane faces many technical challenges due to various reasons. One challenge is a considerable portion of CBM resources are associated with the tectonically deformed regions.\textsuperscript{5-7} Tectonically deformed coal is a type of coal that has undergone single-phase or multiphase tectonic stress and whose original structure and texture have been significantly damaged with a new structure and tectonic characteristics.\textsuperscript{8,9} Among them, the highly tectonically deformed coal is termed as mylonitic coal. Mylonitic coal is the natural result of intact coal being crushed into fine particles (generally less than 1 mm in diameter) by cyclic and continuous tectonic events.\textsuperscript{10} Figure 1 indicates the structures of mylonite coal and some other representative tectonically deformed coals with different magnitudes of tectonic deformation. Many field applications and laboratory experiments have indicated that the higher mechanical strength and greater structural integrity are beneficial in inducing fluid-driven fractures with higher conductivity, as a result, gaining higher CBM recovery rate.\textsuperscript{11,12} However, for mylonitic coal, the fluid-driven fracture is difficult to be initiated because of its granular attribute and the resultant inferior mechanical strength.\textsuperscript{13} Hence, artificial enhancement of the structural integrity and mechanical properties of mylonitic coal is a critical step for the successful application of hydraulic fracturing in mylonitic coal reservoirs.

One potential approach to enhance the integrity of mylonitic coal is via microbially mediated calcium carbonate cementation. The progress of microbial metallogeny demonstrates that certain strains of bacteria are able to induce calcium carbonate precipitation by enzymatic hydrolysis of urea. This microbially mediated calcium carbonate has high mechanical strength, enables function as a cementing agent, and is fundamentally different from chemical synthesis.\textsuperscript{15} This biomineralization is often referred to as microbially induced calcium carbonate precipitation (MICP).\textsuperscript{16}

The following explores microbially mediated carbonate precipitation to cement and strengthen mylonitic coal—and thereby enable potentially successful hydrofracturing operations. We have investigated and evaluated the biocemented mylonitic coal for strength, brittleness, permeability, and associated microstructure evolution.

\section{OVERVIEWS OF MICROBIALLY INDUCED CARBONATE PRECIPITATION (MICP)}

Microbially induced carbonate precipitation (MICP) is a typical biomineralization, by which organisms produce inorganic minerals.\textsuperscript{17,18} \textit{Sporosarcina pasteurii} is a nitrogen-circulating gram-positive bacterium that is a representative microorganism capable of precipitating calcium carbonate given a calcium ion source and urea.\textsuperscript{19} This bacterium can secrete highly active urease at its metabolic process. This enzyme is able to catalyze the hydrolysis of urea into carbon dioxide (CO$_2$) and ammonia (NH$_3$). These hydrolyzed products then diffuse into the solution around the cells and promptly hydrolyze to carbonate (CO$_3^{2-}$) and ammonium (NH$_4^+$). If the hydrolysis reaction occurs in a calcium-rich environment, the CO$_3^{2-}$ generated from the hydrolysis will be converted to calcium

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure1.png}
\caption{Structures of mylonite coal and some other representative tectonically deformed coals: (A) primary structure coal (no deformation), (B) cataclastic coal (the slightest deformation), (C) granular coal (moderate deformation), and (D) mylonitic coal (the intensest deformation)\textsuperscript{14}}
\end{figure}
carbonate (CaCO$_3$). As the concentration of calcium carbonate around the bacteria exceeds its saturation point, supersaturated calcium carbonate will first transform into amorphous precipitation (CaCO$_3$·H$_2$O), and then spontaneously turning into vaterite or calcite.\textsuperscript{20,21} The biochemical process of microbially induced calcium carbonate precipitation is shown in Figure 2. This microbologically mediated mineral precipitation can act as a biological cement agent to strengthen the granular medium.

3 | MATERIALS AND METHODS

We reported the experiments of microbially induced carbonate precipitation (MICP) for different durations in mylonitic coal columns to examine the levels of permeability, strength, and brittleness evolution.

3.1 | Bacterial cultivation

*Sporosarcina pasteurii* used in this study was obtained from the American Type Culture Collection (freeze-dried, ATCC 11859). The growth medium is the NH$_4$-YE liquid medium (ATCC 1376). The bacterial cultures were cultured at 30°C in the shaking water bath (200 rpm) for 36-48 hours before harvesting at OD$_{600}$ = 1.4-1.6. OD$_{600}$ is an abbreviation standing for the optical density of a sample measured at a wavelength of 600 nm. It is a standard measurement method for estimating the concentration of bacterial or other cells in a liquid.\textsuperscript{23} Then, bacteria solution was centrifuged at 4000 g for 30 minutes, removed the supernatant, and supplemented the fresh NH$_4$-YE growth medium. After repeating the above centrifugation process once, the bacteria solution was stored at 4°C before use.

3.2 | Mylonitic coal column set assembly

Mylonitic coal was collected from the Jiulishan coal mine, near Jiaozuo, China. Jiulishan coal mine is located at the central part of the Jiaozuo coal basin and the sampled coal is No. 2, coal seam, which is the primarily mined layer of Jiulishan mine and also the typical mylonitic coal seam.\textsuperscript{24}

A cylindrical PVC tube [1-in. (25 mm) in inner diameter and 6-in. (150 mm) in length] was used as the experimental core holder with the raw mylonitic coal about 3-in. (75 mm) in length. The aggregate of coal particles was encapsulated in the PVC tube and then reconstituted by pressing at 10 MPa for 10 minutes so that the structure of the coal sample is close to the in situ behavior while ensuring the original characteristics of coal particles. No water nor binder was mixed during the molding process, so the molded sample did not have any significant initial strength. A total of twelve samples were prepared for MICP treatment, and the initial permeability of these coal samples ranges from 0.47 to 0.59 Darcy.

3.3 | Experimental apparatus and procedure

The experimental apparatus is shown in Figure 3. The *Sporosarcina pasteurii* suspension, fixation solution
SONG et al. (0.05 mol/L CaCl₂), and cementation solution (1.0 mol/L CaCl₂ and 1.0 mol/L urea) were sequentially injected into the sample from the bottom inlet of the core holder by a peristaltic pump. Calcium ions in the fixation solution can enhance the adhesion of bacteria to the surface of the coal particles as well as bacterial flocculation. A standard injection procedure was as follows:

a. 10 mL of the bacterial suspension is first injected into the sample at a rate of 0.25 mL/min.

b. The sample is then stood for 2 hours to allow the bacteria to attach to the particle surfaces.

c. 10 mL of fixation solution is injected at a rate of 0.25 mL/min.

d. 50 mL of cementation solution is injected at a rate of 0.25 mL/min.

e. Disconnect the flow lines, reverse the sample (sample is turned end-over-end), reattach the flow lines so that the inlet is now at the former outlet and repeat procedures a-d.

The steps (a)-(e) constitute a single MICP cycle. The injection is performed continuously, with no interval between the two subsequent injections. The coal samples were individually performed to four, six, eight, and ten cycles of MICP treatment, and each group injection experiment contained three parallel specimens.

FIGURE 3 Schematic of the experimental apparatus

3.4 | Measurements for permeability, strength, brittleness, and calcium carbonate content of MICP-treated coal cores

After the MICP injection experiment, these cores were dried at 80°C for 24 hours and trimmed into standard samples (2-in./5.08 cm in height and 1-in./2.54 cm in diameter in the center of the original core) for permeability test, uniaxial compression experiment, and calcium carbonate content measurement in sequence. Since it is difficult to conduct hydraulic fracturing in such small-scaled coal samples, we measure the hydraulic fracability of biotreated mylonitic samples using uniaxial compressive strength (UCS), brittleness, and permeability, as well as microscopical observations.

The experimental apparatus for permeability measure is illustrated in Figure 4. The MICP-treated coal core was installed inside a polyvinyl chloride (PVC) rubber jacket and confined within a triaxial core holder (Temco) capable of applying independent loading in the radial and axial directions. A dual cylinder syringe pumps (ISCO 500D) with a control accuracy ±0.1 kPa were used to apply the radial and axial stresses with distilled water as confining fluids. The coal core was sandwiched between two cylindrical stainless-steel loading platens with throughgoing flow connections and flow distributors. The coal core and the platens were isolated from the confining fluid by the polyvinyl chloride (PVC) rubber jacket. The platen at one end was connected to a syringe pump (ISCO 500D) for inputting fluid to measure the permeability. The other end of the platen was connected to the atmosphere.

The coal core was loaded with a constant stress of 200 kPa in the radial and axial directions during the permeability test. Distilled water, which was used as the injected fluid for the permeability test, is injected into one end of the core holder via the syringe pump (ISCO 500D) at a constant pressure of 10 kPa higher than the other end (the other end is connected to the atmosphere). The permeability was measured by reading the flow data of the inputting pump and bringing it into the permeability formula based on Darcy’s law, which is shown in Equation (1).

FIGURE 4 Schematic of experimental apparatus for permeability measure

(0.05 mol/L CaCl₂), and cementation solution (1.0 mol/L CaCl₂ and 1.0 mol/L urea) were sequentially injected into the sample from the bottom inlet of the core holder by a peristaltic pump. Calcium ions in the fixation solution can enhance the adhesion of bacteria to the surface of the coal particles as well as bacterial flocculation. A standard injection procedure was as follows:

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The steps (a)-(e) constitute a single MICP cycle. The injection is performed continuously, with no interval between the two subsequent injections. The coal samples were individually performed to four, six, eight, and ten cycles of MICP treatment, and each group injection experiment contained three parallel specimens.
where $k$ is the permeability, $Q$ is the volumetric flow rate, $\mu$ is the dynamic viscosity of the fluid, $L$ is the length of the specimen, $\Delta P$ is the small pressure difference, and $A$ is the cross-sectional area of the sample.

The dimensionless relative permeability ($k/k_0$) was used for comparison between the normalized permeability reduction with increasing MICP injection cycles, where $k_0$ represents the initial permeability of the untreated coal core.

After the permeability tests, the samples were examined the pre-peak strength and post-peak strength response with the increasing MICP treatments by uniaxial compressive strength (UCS) test. The UCS test process referred to the ASTM D7012-14 (ie, Standard Test Methods for Compressive Strength and Elastic Moduli of Intact Rock Core Specimens under Varying States of Stress and Temperatures). The axial load was applied at a constant strain rate of 0.1 mm/min throughout the UCS testing.

Brittleness is a term used to identify and describe the potential failure characteristics of the rock materials. And it is also a crucial index for evaluating the ability of initiating fluid-driven fractures of reservoirs.\(^{27}\) To date, nearly twenty quantitative expressions of rock brittleness have been proposed considering brittleness characteristics of different rock and practical use. The use of the stress-strain curves to derive the brittleness index (BI) is a straightforward and credible approach.\(^{28}\) We referred to the reference\(^{29}\) which proposed a valuation methodology of rock brittleness considering the relative stress drop and the stress drop velocity. Brittleness index is defined as follows,

$$BI = \frac{\tau_p - \tau_r}{\tau_p} \log|m|$$

where $\tau_p$ represents uniaxial compression strength, $\tau_r$ is residual strength, and $m$ is the slope of the line from the initial yielding point to the starting point of the residual strength. The absolute value of $m$ is adopted as slope here is always negative.

The meanings of the above nomenclatures in the typical stress-strain curves of the rock sample are shown in Figure 5. The value of BI ranges from 0 to 1, and a higher BI value indicates a higher brittleness.

The calcium carbonate (CaCO\(_3\)) content of the MICP-treated sample was measured by immersing the sample fragment into 5.0 mol/L chlorhydric acid to fully remove the CaCO\(_3\) and then measured the mass difference before and after soaking. The calcium carbonate content was expressed as follows,

$$W_{CaCO_3} = \frac{M_{coal+CaCO_3} - M_{coal}}{M_{coal}}$$

Figure 6 shows the mylonitic coal samples with four, six, eight, and ten cycles of MICP treatment. All the biotreated coal cores can keep intact after demolding the PVC core holder—suggesting that the coal particles were effectively cemented by microbially induced calcium carbonate precipitation.

Measured permeability of microbially treated samples is shown in Figure 7. Overall, the permeability indicates decreasing changes with the increase of MICP treatments. After ten cycles of injection, the permeability for three parallel samples dropped to 8.4%, 11.2%, and 16.6% of its initial permeability, respectively.
Subsequently, all of the MICP-treated coal cores were performed the uniaxial compressive test to determine the uniaxial compressive strength (UCS) and brittleness. The images of coal samples after uniaxial compressive failure are shown in Figure 8. It can be clearly seen that the macroscopic failure behavior of coal evolves from the plastic failure to brittle failure as the increase of biotreatments, suggesting the potential improvements to allow successful fluid-driven fractures.

Both the results of stress-strain curves and peak strengths for the biomineralized coal samples are shown in Figures 9 and 10, respectively. The stress-strain curve characteristics of coal samples exhibited significant differences with increasing durations of microbial treatment. Peak strength increases with increasing MICP treatments. After ten cycles of treatments, the UCS of these three paralleled samples reaches to 6.91 MPa, 7.89 MPa, and 8.68 MPa, respectively.

The calculated brittleness index (BI) values of the coal samples and its corresponding UCS are shown in Table 1. For comparison, the coal samples from the Qinshui Basin of China and Illinois, suitable for hydrofracturing, were tested for UCS and BI. The process of the uniaxial compression test also refers to the ASTM D7012-14. The axial load was applied at a constant strain rate of 0.1 mm/min throughout the UCS testing, which was identical with the UCS testing for MICP-treated coal samples.

The UCS for the samples from both the United States and China (Figure 11) is greater than that of biocemented mylonitic coal. However, the brittleness index (BI) of the coal specimens after ten cycles of treatments fairly approaches that of the hard coal, implying the biotreated sample for the potential success of hydrofracturing based on the evaluation of brittleness (Table 2). Furthermore, it is worth noting that a comparatively higher peak strength of UCS does not absolutely represent a higher brittleness property. As shown in Figure 12, we sort the UCS magnitudes for all biotreated samples from left to the right, and their corresponding BI values do not show a monotonous increase. Therefore, the evaluation of initiating fluid-driven fractures for reservoirs should be based on its brittleness property and cannot be evaluated solely by the compressive strength.

Following uniaxial compression tests, the calcium carbonate content of each sample was examined. Figure 13A,B is the correspondence between CaCO$_3$ content—peak strength, and CaCO$_3$ content—brittleness index, respectively. In terms of calcium carbonate content, the CaCO$_3$ mass within the coal sample instinctively increases with increasing MICP treatments. Specifically, within the range of measured calcium carbonate content, the peak strength shows an exponential increase with the increase of calcium carbonate content, while the brittleness index indicates a linear growth.

5 | MICROSCOPIC ANALYSIS AND DISCUSSION

In this section, we have examined the micromorphology of the generated CaCO$_3$ as well as its distribution within the intergranular pores by scanning electron microscopy (SEM) and discussed the microscopic mechanism of cementation for coal particles, which explains the macroscopic behaviors of coal samples after the biotreatment.

5.1 | Crystal characteristics of microbial calcium carbonate

The SEM image (Figure 14A) shows the morphologies of CaCO$_3$ crystals including hexahedron and spheroid which are calcite and vaterite, respectively. The generated calcite and vaterite are mixed with an overlapped distribution. In addition, the early stage of microbial calcium carbonate is observed (Figure 14B). The rod-shaped bacteria are surrounded by precipitated calcium carbonate. This confirms that the role of microorganisms acts as nucleation sites for the crystallization process of calcium carbonate precipitation.
5.2 | The cumulative process of calcium carbonate precipitation in interstitial space

We explore the microscopical process of generated calcium carbonate within intergranular spaces and resultant particle bondings by comparing the SEM images with four cycles of MICP treatment (Figure 15A) and ten cycles of MICP treatment (Figure 15B). Microorganisms first adhere to the surfaces of coal particles where they gradually induce calcium carbonate precipitations. As this mass accumulates continuously, the interstitial spaces are gradually occupied by precipitated calcium carbonate. Meanwhile, the particle-particle contacts are bonded sporadically by the generated calcium carbonate, and the bonding strength between the particles is continuously enhanced with the increase in the cementation area. Notably, the irregular morphology of coal particles results in contact relation between particles taking two different forms: point contact bond (Figure 16A) and planar contact bond (Figure 16B). The latter forms platy void space that provides a larger effective area for cementation; thus, calcium carbonate can exert greater effectiveness in bonding strength. “Point contact bond” means the contact area between particles is nonplanar, and the potential biocemented strength and biocemented area are greatly limited.

5.3 | Microfailure characteristics of MICP-cemented coal under uniaxial compression

We also investigated the micromorphology of the fracture plane of MICP-cemented coal under uniaxial compression. The axial compression deformation mainly causes the compaction of intergranular space and the dislocation between the biocemented coal particles. The coal particle crushing and the resultant deformation combinedly lead to macroscopic failure.
Two microfailure forms were observed. One is that the coal is crushed (Figure 17I), and the other occurs at the biocemented interface between the coal parties and calcium carbonate crystals (Figure 17II). We hardly observed the failure of calcium carbonate crystals. Although the breakdown mechanism of the fluid-driven fracture is somewhat different from that of the fracture with uniaxial compression, the observed microfailure forms of biotreated coal after uniaxial compression suggested that the strength of calcium carbonate crystals is higher than that of coal particles, and the bonding strength between calcium carbonate crystals is higher than that between calcium carbonate and coal particles. Therefore, the fluid-driven fractures of MICP-treated coal sample are more prone to occur on the coal particles themselves and the cemented surface of the coal particles and calcium carbonate, which can expose more fresh surfaces for gas transport and desorption.

6 | DISCUSSION

The key to hydraulic fracturing that can effectively enhance the recovery of coalbed methane is to generate fluid-driven fractures that can provide permeable flow paths for methane. More importantly, the fluid-driven fractures can effectively reduce the in situ stress on coal matrix and generate gas pressure gradient from the
coal matrices to the fracture pathway, thereby promoting both the gas desorption and convection. The considerable gains on mechanical strengthening and brittleness via biotreatments ensure the potential success of hydraulic fracturing in mylonitic coal. Although the passages for gas transport (pore-to-pore and pore-to-fracture) are partially occupied by generated calcium carbonate, fluid-driven fractures may be readily initiated based on our observations, leading to the release of effective stress on the coal matrices and the formation of gas pressure gradient. Therefore, gas drainage will definitely increase after MICP treatment. In addition, the injection of the bacterial solution and nutrient solutions may change the wettability of the coal and thus affect the gas desorption and gas transport. This is also an issue worth considering. The following research will focus on the changes in gas desorption, gas seepage, and ensemble permeability of MICP-treated mylonitic coal. In this experiment configuration, the cost of biotreatment is relatively cheap, but the integrated economic feasibility for increasing gas production needs further exploration. Besides, Cunningham et al.\textsuperscript{30} proposed that using alternative urea sources and calcium ion sources like urea fertilizer and CaCl\textsubscript{2} ice melting products could enhance the economic viability of MICP technology. This will also be investigated in our following researches. To date, laboratory-scale MICP injection experiments are mostly one-dimensional column experiments that are quite different from the engineering scale. The following experiment will investigate the three-dimensional flow characteristics of MICP injection fluids and the effectiveness of microbially induced CaCO\textsubscript{3} cementation on large-scale mylonitic coal.

**FIGURE 13** Correspondence between (A) CaCO\textsubscript{3} content—peak strength, and (B) CaCO\textsubscript{3} content—brittleness index

**FIGURE 14** SEM images of microbially induced calcium carbonate precipitation

(A) Crystal Characteristics of calcium carbonate.  
(B) Bacteria surrounded by precipitated CaCO\textsubscript{3}.
FIGURE 15  The cumulative process of calcium carbonate within interparticle space

FIGURE 16  Contact bond relation between particles

FIGURE 17  Microfailure characteristics of MICP-cemented coal
7  |  CONCLUSION

We have explored a biomineralization method of microbially mediated calcium carbonate precipitation (MICP) to enhance the structural integrity and mechanical strength of mylonitic coal, enabling potential success for hydrofracturing. After ten cycles of biotreatments (several days), the maximum uniaxial compressive strength (UCS) reaches 8.7 MPa, and the brittleness index is quite close to that of the hard coal, implying the mylonitic coal after biotreatment for successful hydrofracturing. The increments in UCS and brittleness of biocemented mylonitic coal show a positive correlation with the generated calcium carbonate content.

SEM results indicate the morphologies of CaCO3 crystals including cuboidal calcite and spherical vaterite. Calcium carbonate precipitation occurs first on the surfaces of coal particles, irregularly occupying the interstitial space, before creating particle-particle bonds. The irregular morphology of coal particles results in contact relation between particles taking two different forms: point contact bond and planar contact bond. Two micro-failure patterns of biocemented coal with uniaxial compression were observed. One is that the coal particles are crushed, while the other type of failure occurs at the biocemented interface between the coal particles and calcium carbonate crystals.

This study emphasizes the potential of microbially mediated cementation to enhance mechanical strength and brittleness for mylonitic coal, with the latter index indicating that the improvement is sufficient to achieve the success of hydrofracturing. The following experiments will investigate the three-dimensional flow characteristics of MICP injection fluids and the effectiveness of microbially induced CaCO3 cementation on large-scale mylonitic coal.

ACKNOWLEDGMENTS

This paper is supported by the National Science Foundation of China (Grant No. 51604051), the Natural Science Foundation of Chongqing (Grant No. cstc2018jcyjAX0657), and State Key Laboratory Cultivation Base for Gas Geology and Gas Control (Henan Polytechnic University). This support is gratefully acknowledged.

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How to cite this article: Song C, Zhi S, Feng G, Lin J. Enhancing potential of hydrofracturing in mylonitic coal by biocementation. *Energy Sci Eng*. 2021;9:565–576. https://doi.org/10.1002/ese3.860