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Full Length Article

Sustaining biogenic methane release from Illinois coal in a fermentor for one year

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ABSTRACT

To evaluate coal biogasification in a larger reactor over a longer duration as compared to studies reported so far, a 3-liter fermentor was established. During the one-year study, a nutrient recipe was added three times to sustain methane release from Illinois bituminous coal. The cumulated methane production was 5171 ft^3/ton with a methane content of 75.4% on day 365. After the fermentation was terminated, the residual coal and fermentation broth were characterized in detail. Compared to the untreated coal, the treated coal residue appeared to be finer and highly degraded with less carbon but more ash. Based on mass balance, volatile and fixed carbon decreased 15.9% and 29.6%, respectively, using the untreated coal as the baseline. According to GC/MS analysis, the fermentation broth contained mainly three groups of compounds: fatty acids and their derivatives, aromatics, and hydrocarbons. In addition, the fermentation broth was found to have effect on flocculation and contained compounds that possessed surface-active properties. Further investigations are needed to identify these chemicals responsible for these activities and develop ways to further enhance coal biogasification based upon results obtained then.

1. Introduction

Coalbed methane (CBM) is an important natural gas resource that has attracted increasing attention worldwide [1]. Generally, CBM is contributed by two processes, geological and biological. Accumulated geological data has shown that the secondary biogenic source is a more important origin of CBM [2]. Generation of biogenic methane is due to microbial activities after coalification, which indicates that coal has the potential to be converted to methane under normal ambient conditions [3]. Recently, great efforts have been extended to enhance biogenic methane production from coal in view of promising results reported in the literature [1,4].

For the purpose of enhancing methane production from coal both in situ and ex situ, different biological approaches have been tested, including adding external microbial sources- bioaugmentation and supplementing chemicals and nutrients- biostimulation. These approaches could be used separately or in combination to achieve continued generation of biogenic methane from existing CBM installations. For bioaugmentation [5], microorganisms may be added if they have demonstrated greater capability in methane production than the existing microorganisms in the coal beds, or the target coal beds lack microbial activities toward methane release. Intuitively, it seems that native microbial communities would be optimally adapted to their environment in the presence of coal and would provide higher methane production compared with the foreign microbial consortia [1]. However, in some cases, the opposite is true as evidenced by reports that some foreign microbial communities were able to produce similar or more methane from coal than native communities [5–8]. But, if legal aspects are considered, such as getting permits for injecting microbes to a given environment, bioaugmentation may face daunting challenges. Thus, a better niche for this may be for it to be used ex situ. In terms of biostimulation, numerous studies have evaluated various recipes including MS medium, trypticase soy broth, commonly used anaerobic medium, and different solvents [9–13]. However, the majority of these studies focused on short-term evaluation of methane production from different ranks of coal in small reactors. The study periods normally were 30–45 days and the reactor volume was generally less than 250 mL. Thus, at this point, it is unknown whether results obtained from short-term studies in small vessels can be extrapolated to longer term and in large scales.

In addition, even though a great number of studies have been published in the domain of coal biogasification, only a few have

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evaluated the residual coal after bioconversion. According to Barnhart et al. [14], after 1169-day bioconversion with the addition of algal extract or yeast extract, the British Thermal Unit (BTU) content of the treated coal was 99.5% of that of the untreated coal. In addition, several parameters, such as total coal moisture, coal ash and coal sulfur did not vary significantly among different treatments and controls. Thus, the coal quality remains largely unchanged following a long term stimulated microbial methane production. In another study [15], however, compared to the untreated coal, the coal residues after 30- and 60-day biogasification were found to have lower carbon content, higher sorption capacity, more pore surface area, higher gas storage capacity, and significantly enhanced diffusion rates as a result of continued bioconversion. Further test of similar samples revealed that after biotreatment, the mesopore surface area and pore volume decreased with increased average pore diameter, while the micropore surface area increased with decreased pore volume. After bioconversion, both inaccessible meso-/micropore size distributions decreased while the accessible micropore size distribution increased, making a portion of closed micropore network accessible. In addition, the methane adsorption capacities increased after bio-treatment, which was confirmed by the increase of micropore surface area [16].

Considering different results published by different research groups, the effect of bioconversion on coal structure remains to be elucidated, in particular at relatively larger scale. To fill this critical knowledge gap, this study was designed to evaluate coal biogasification in a 3-liter fermentor for a one-year duration. Besides measuring and computing methane yield, we have specifically focused on: (1) evaluating the residual coal with regard to particle size, elemental composition, and morphology and (2) studying the fermentation broth with respect to their chemical composition and potential functions as bioflocculant and biosurfactant. It needs to be noted that this study is an extension and scale up of what we have extensively studied in the past several years at the microcosm level [9–13,17,18]. At those levels, we have demonstrated through delicate experimental designs that coal is the dominant carbon source for methane detected even though the microcosms are supplemented with suitable nutrient solutions.

2. Materials and methods

2.1. Coal samples

For this study, the coal sample used was the same as what has been investigated and reported before [9–11,13,15,18]. Briefly, coal blocks were collected from the Herrin Seam, # 6 in the Illinois basin. This coal contained 70.1% of carbon, 1.4% of nitrogen, 5.2% of hydrogen, 0.6% of sulfur, 15.4% of oxygen, and 7.5% ash (dry weight basis). Contents of volatile matter and fixed carbon were 49.9% and 42.6% (dry weight basis), respectively. Immediately before use, a block of coal was broken into lumps approximately 1.3 cm in size. The coal lumps were subsequently ground and sieved to obtain coal samples less than 200 mesh (74 μm). This particle size was chosen based upon our previous observation that among different particle sizes, biogasification of coal < 74 μm led to the highest methane yield for this Illinois coal [10]. Ground coal samples were stored in re-sealable ziploc bags at room temperature in order to prevent moisture loss and oxidation.

2.2. Formation water collection

Formation water used in this study was collected from an established coal-bed methane (CBM) well as described in our reported study [10,17]. At the sampling site, the formation water was retrieved from a depth of around 850 ft. The in situ temperature was measured immediately after the formation water came to the surface. For those dedicated to experimental setup as described below, the water samples in half-gallon containers were supplemented with sodium sulfide (Na_2S) at 0.25 g/L and resazurin at 1 mg/L to maintain anaerobic conditions. Once sealed tightly, these containers were brought back to our laboratory where they were immediately stored in a −20 °C freezer for later use. Fresh formation water without the addition of these two chemicals was analyzed thoroughly in terms of its chemical composition as reported already [17].

2.3. The microbial community

The microbial community used in this study was that initially present in the formation water aforementioned above. Upon arrival in our laboratory, the formation water was concentrated 80 times through high-speed centrifugation at 10,000*g* force for 30 min. The resulting concentrate was used to make glycerol frozen stocks. Based on next-generation 16S rDNA sequencing, this community comprised a total of 231 Bacterial species and 33 species of Archaea [18]. The Bacteria were distributed among 24 phyla. The dominant three were Proteobacteria, 40.8 ± 0.0%; Bacteroidetes, 22.9 ± 2.0%; and Firmicutes, 17.9 ± 0.1%. In terms of Archaea, the majority (89.8 ± 0.7% of the total) fell within the order of Methanobacteriales within the phylum of Euryarchaeota.

2.4. Experimental setup and monitoring

Most biogasification studies have been conducted in small serum bottles lasting for a few months or shorter. To understand how biogasification performs in a larger reactor over a longer duration, a 3-liter fermentor (Eppendorf, Hauppauge NY, USA) was used. The testing conditions were the same as the optimal conditions gained from our previous study [10]. Specifically, the coal loading was 200 g/L, the temperature was 32 °C, and the coal particle size was < 200 mesh (74 μm). The recipe used in this study was developed from our previous work targeting in situ biogasification [17]. This recipe contained Fe-powder at 74 mM (particle size: 80 nm–100 nm); methanol at 97.9 mM; ethanol at 100 mM, and a trace mineral solution at 100%. For the trace mineral solution, a 100% supplement was used to ensure that the formation water, after external trace minerals were added, had the same composition of trace metals as in a standard MS medium [19]. Specifically, a trace mineral stock solution was made containing $CoCl_2{\cdot}6H_2O$ at 1.3 mg/L, $ZnCl_2$ at 0.76 mg/L, $Na_2WO_4{\cdot}2H_2O$ at 0.26 mg/L and H_2SeO_3 at 0.01 mg/L. For each recipe addition, 10 mL of the trace mineral stock solution was added to one liter of formation water. It needs to be noted that the formation water used in this study was filtered through 0.45 μm filters to minimize impacts of suspended solid in the water. The fermentation system was started by adding 100 g of coal samples together with 500 mL filtered formation water, and 50 mL of inoculum developed from the glycerol frozen stocks. In light of the fact that yeast extract and trypticase peptone are important nitrogen sources and their demonstrated effect on stimulating coal bioconversion [9], these two ingredients were added at 2 g/L for each. After all ingredients were added, the fermentor was sealed and purged with N_2 completely to remove oxygen. It is noteworthy that the developed recipe was not supplemented on day 0 and the fermentor had an approximately 2-liter headspace at the beginning together with the fermentor, three replicate uninoculated control microcosms were established. These microcosms included coal at 200 g/L, the filtered formation water and the same amendments as those added to the fermentor and at the same concentrations, but not the inoculum. These controls were set up in the same way as described in Zhang et al. [17].

Starting from day 10, the headspace gas in the fermentor was released and collected in a 3-L airbag. The fermentor was then purged entirely by at least six liters of N_2 gas to ensure a zero concentration of methane in the 1-atm headspace. The volume of the released gas together with gas content measured by a Gas Chromatography (GC) at different time points were recorded. On day 31, day 121, and day 300, the developed recipe described above was injected into the fermentor following nitrogen purging to supplement what was consumed by the

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