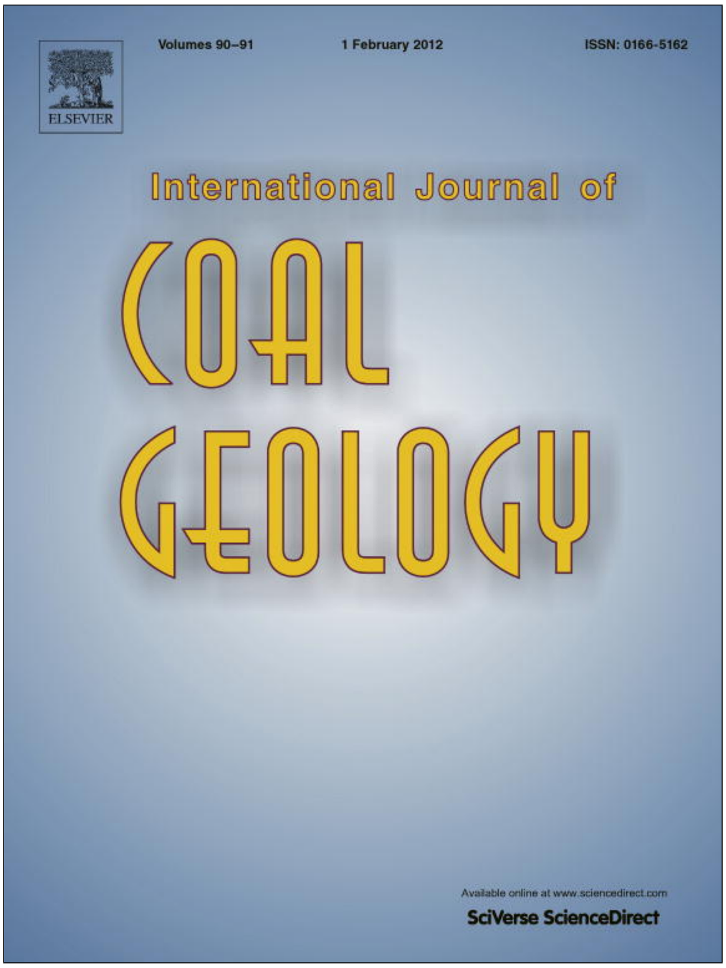
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Microbial production of methane and carbon dioxide from lignite, bituminous coal, and coal waste materials

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ABSTRACT

The aim of this study was to examine microbial methane and CO_2 production from bituminous coal waste, lignite, and bituminous coal materials. Bituminous coal and coal waste material were obtained from a Utah mine and lignite was obtained from a commercially available North Dakota sample. Microbial populations were cultured from hydrocarbon-rich environments and locations where natural methanogenesis was occurring. Various pulverized coal and coal waste materials were combined with selected microbial inocula and different types and levels of nutrient amendments. After a 30-day reaction period at about 23 °C, headspace methane and CO_2 were analyzed using gas chromatography. With increasing nutrient concentrations (0, 10, and 50%), coal waste generated an extrapolated equivalent of 36, 53, and 16,000 scf of CH_4/ton/year and 1870, 4400, and 8000 scf of CO_2/ton/year. Methane produced from native and nutrient-amended bituminous coal waste materials was the same order of magnitude as that produced from bituminous coal but lower than that produced from lignite. CO_2 generation from coal waste materials, with no nutrient addition was over twice as high as that produced from analogous bituminous coal. The results of this study suggest that coal waste products can be converted to useful fuel at volumes that may be commercially viable.

1. Introduction

Biogenic gas, produced from anoxic decomposition of organic matter by microorganisms, is considered an unconventional natural gas resource. For many years, biogenic gas (or biogas) was connected mostly with the decomposition of organic matter in shallow anoxic sediments (e.g., wetlands, landfill gas, marsh gas, methane associated with or produced from municipal wastewater treatment facilities and landfills, or from rice paddies). Recently, it has been realized that biogenic methane production is ongoing in many hydrocarbon reservoirs. Coalbed methane (CBM), for instance, has been believed for many years to have thermogenic as well as biogenic origins through decomposition of organic matter occurring during early stages of coalification (Thomas, 2002). Recent studies show, however, that coalbed methane may also be of a more recent biogenic origin, produced through microbial degradation and utilization of complex carbon compounds (Scott and Kaiser, 1995; Butland and Moore, 2008; Flores et al., 2008; Formolo et al., 2008). Similarly, large biogenic shale gas plays, such as the Antrim Shale of the Michigan Basin or the Colorado shale in Alberta, have been recently described (Curtis, 2002; Jarvie et al., 2007).

Enhancing methane recovery and stimulating methanogenesis in low production and depleting gas wells has received increased interest in recent years. Proven conventional natural gas reserves in the United States amount to about 244 TCF (trillion cubic feet) (EIA, 2008), while the annual domestic consumption is about 23 TCF (EIA, 2010). Assuming no imports, conventional reserves will last for roughly 10 years. Since the American economy depends nearly as much on natural gas as it does on crude oil, the search for unconventional resources of natural gas within the United States has a high priority. Due to the developments in hydraulic fracturing in the last decade, shale gas, one of the unconventional natural gas resources, is now responsible for 20% of the American natural gas production (Kerr, 2010). Development of other unconventional natural gas resources will be crucial for the sustainable future. The technology described in this document has potential application to at least some liquid-bearing shales, oil shale, various un-minable coals, other waste hydrocarbon materials, and methane-depleted coal deposits.

From a different perspective, mining of coal and other hydrocarbon sources (e.g., tar sands and oil shales) results in mountainous waste heaps of mineral waste and lower grade coal materials, a potentially useful fuel source. In the United States alone, accumulated culm and gob (waste products of anthracite and bituminous coal mining, respectively) are estimated to be about two billion tons (Akers and Harrison, 2000). Annually, about 55 million tons of waste coal

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are generated (Tillman and Harding, 2004) and pile up on mine sites as unprofitable mountains or valley fills that can potentially contribute to generation of metal contaminated waters, or are gravitationally unstable and a risk for slope failure. Successful conversion of even a fraction of this waste material into useful fuel could prove advantageous to the mining industry and the environment.

This study examines the ability of microbial consortia, alone and combined with simple and complex nutrient amendments, to generate methane and carbon dioxide from various coal ranks and coal waste materials under diverse conditions. Unlike many other studies, this research does not focus exclusively on enhancing methane recovery from depleting coalbed methane wells using added nutrient materials (Jones et al., 2010; Ulrich and Bower, 2008). It leans towards methane production, which can be achieved through utilization of surface (ex situ) or subsurface (in situ-coal waste piles or valley fill) bioreactors. Moreover, it examines the potential for methane and CO_2 generation from coal waste materials compared to lignite and bituminous coal.

2. Materials and methods

2.1. Solid hydrocarbon samples

Coal and coal waste samples used in this study were provided by PacifiCorp Energy from the Deer Creek Mine in Utah. The samples came from the same mining operation and types of mined materials to permit a more direct comparison of the results obtained. The coal sample had a total moisture content of 4.28% and 6.15% ash content as received. With over 76% carbon (dry) and a caloric value of about 14,000 BTU/lb it is classified as a bituminous coal. The coal waste product, as received, contained over 50% ash, 28% carbon (dry), and had a caloric value of about 4400 BTU/lb. More specific elemental composition, as well as ash analysis for these coal and coal waste samples, is given in Tables 1 and 2.

A commercially available North Dakota lignite sample was purchased as a bulk pack from Ward's Natural Science (#47-2133). Chemical composition of characteristic North Dakota coals is presented in Table 1, as given by Tang et al. (1996). All coal samples were pulverized to −200 mesh particle size in a ball grinder to provide maximum surface area. It is known that particle size will influence the extent of methanogenesis and the results presented in this paper represent the best-case scenario (Green et al., 2008). Moreover, the coal materials were exposed to air during sample storage and preparation, which might have influenced their biodegradability.

2.2. Microbial samples

Microbial consortia containing methanogens were collected from various environments and characterized. These environments included coal (C), waste coal (WC), high-salt oil seeps (OS), natural gas wells (GW), high-salt lake sediments (LS), wetland sediments (WS), river sediments (RS), and anaerobic digester sludge (DS). Microbes from the coal and coal waste materials were obtained from the coal samples used in this study. Microbes from lake sediments and wetland sediments were sampled from the Great Salt Lake area. Microbes from river sediments were collected from the Jordan River, UT in the Legacy Nature Preserve. Consortia containing methanogens were obtained from a 500 mL sample of anaerobic digester sludge collected from the Central Valley Wastewater Treatment Plant, UT. Additionally, microbes from eight samples from the north arm of the Great Salt Lake; the Rozel Point oil seeps and from the produced waters of six Conoco-Phillips' Drunkard's Wash coalbed methane wells were collected. All microbial consortia collected were cultured, using classical aseptic microbiological techniques, in six liquid media containing a combination of direct methane precursors (e.g., acetate), nitrogen containing nutrients (e.g., urea), phosphorus containing nutrients (e.g., phosphate), and a balanced nutrient solution (trypticase soy broth). No pH buffer was added to the media and pH of all used media was close to neutral.

Table 1
Analysis of bituminous coal and coal waste samples.

Parameter	Coal	Waste coal	Lignite
Total moisture (as received)	4.28%	6.89%	3.90%
Ash (dry)	6.43%	57.61%	5.58%
Volatile matter (dry)	48.13%	29.30%	44.83%
Fixed carbon (dry)	45.44%	13.09%	45.69%
Carbon (dry)	76.60%	28.24%	61.20%
Gross calorific value [BTU/lb] (dry)	13,949	4,370	NA
Sulfur (dry)	0.38%	0.30%	0.25%
Organic sulfur (dry)	0.37%	0.16%	NA
Oxygen (dry)	9.15%	11.40%	27.98%
Hydrogen (dry)	6.02%	1.99%	3.97%
Nitrogen (dry)	1.42%	0.46%	1.01%

Table 2
Ash analysis of bituminous coal and coal waste samples.

Component	Coal	Waste coal
SiO_2	52.72%	62.05%
Al_2O_3	13.16%	8.72%
Fe_2O_3	5.27%	2.30%
CaO	12.10%	16.88%
MgO	1.50%	6.34%
K_2O	0.18%	1.55%
Na_2O	4.19%	0.35%
SO_3	8.89%	1.11%
P_2O_5	0.75%	0.18%
TiO_2	0.90%	0.42%

All collected consortia were exposed to the atmosphere during the collection and cultivation steps. It was decided to handle microbial samples in aerobic conditions, in order to create a microbial consortium that can withstand low oxygen concentrations. This is a crucial component of any technology that wishes to be applied on a large scale, outside the laboratory settings.

2.3. Experimental

Glass serum bottles (20 mL, Wheaton #223742) were used as bioreactors for methane and CO_2 generation. Four gram aliquots of pulverized hydrocarbon materials (bituminous coal, waste bituminous coal, and lignite) were placed in separate serum bottles; 5 mL of either sterile normal saline or nutrient solution and 1 mL of washed microbial consortia were added, resulting in approximately 13 mL of free headspace. Microbial inocula were centrifuged and washed with saline solution three times, in order to remove any remaining culture media. The headspace was not sparged with nitrogen gas and initially contained the atmospheric gases for the same reasons why the microbial samples were collected and cultured under aerobic conditions.

Three levels of nutrient amendments were selected for this study: 0%, 10%, and 50% concentration of one of several common growth nutrient preparations. Thus, the liquid solution added to the various samples contained either normal saline (0.85% NaCl) solution (corresponding to 0% nutrient amendment) or normal saline solution with added nutrients. The sample nutrient volume was equal to 5 mL; therefore, 10% nutrient amendment contained 0.5 mL of stock nutrient solution and 4.5 mL of normal saline solution. The composition of nutrient amendments in each test was identical to the liquid media initially used to culture a specific consortium. Serum bottles were capped with Teflon silicone septa (Wheaton #224173) and

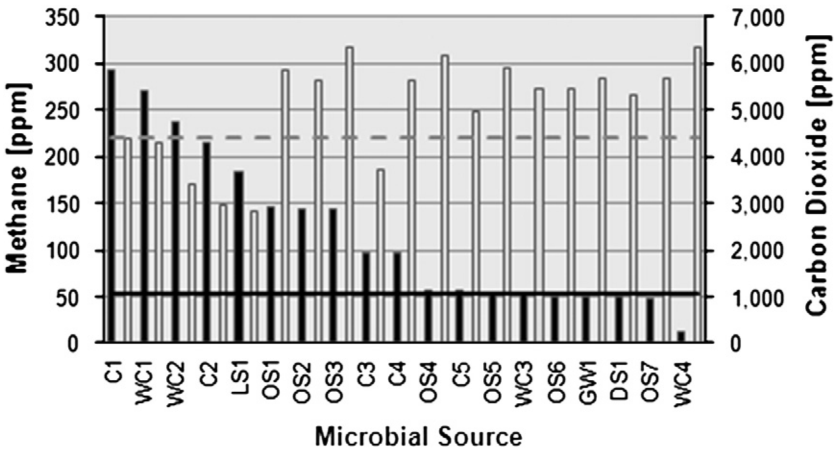

Fig. 1. Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal samples inoculated with various microbial consortia (represented by the *x*-axis) after 30 days incubation period with no nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

cramped with aluminum seals (Wheaton #224178). Over 650 samples, containing solid hydrocarbons, microbial consortia from various environments, and saline or nutrient solution amendments, were created. Since it was a large microbial screening test, many of the collected microbial samples did not generate higher methane and carbon dioxide concentrations than the control samples, and are therefore not included in this paper. Control samples were created by adding sterile normal saline solution to the respective pulverized samples without the addition of microbes. The samples were analyzed after a 30-day reaction period at about 23 °C. Samples were not exposed to agitation.

2.4. Gas chromatography

A Hewlett Packard gas chromatograph (model HP6890) with a GS-GasPro PLOT column containing a proprietary, bonded silica-based stationary phase was used to determine headspace gas concentrations in each sample bottle. A flame ionization detector (FID) and thermal conductivity detector (TCD) were connected in series to analyze organic compounds and inorganic gases, respectively. The temperature program of the system began with 35 °C for 3.8 minutes to allow for carbon dioxide and ethane elution and was then increased by 25 °C min^{-1} to 260 °C. Methane and carbon dioxide standard gases (Scotty Analyzed Gases) were used for calibration. GC ChemStation (Agilent Technologies) computer software was used.

3. Results

Headspace methane and carbon dioxide concentrations in the samples were analyzed after 30 days at approximately 23 °C. Carbon dioxide generation is important; it is one of the direct methane precursors and can increase the overall methanogenic potential in the samples. Results were measured and presented here in parts per million (ppm).

Control samples are represented on the following graphs (Figs. 1 through 9) by solid black and dashed gray lines for methane and CO_2, respectively. Any gases detected from control samples were a result of desorption from coal under atmospheric conditions and/or generation by the native microbial population present in the respective coal materials.

Additional control samples included the same concentration of microbial inocula suspended in normal saline solution (0.85% NaCl) without any coal material. Gases generated from these controls came from degradation of the dead cells by the remaining populations. Depending on a microbial inoculum source, 3.89 ppm to 5.40 ppm methane and 1210 ppm to 1597 ppm carbon dioxide were generated. These were insignificant in comparison to the obtained results and are not included on the presented figures.

3.1. No nutrients added

Generation of methane and CO_2 with no nutrient amendment is one of the more interesting parts of this study, since it indicates

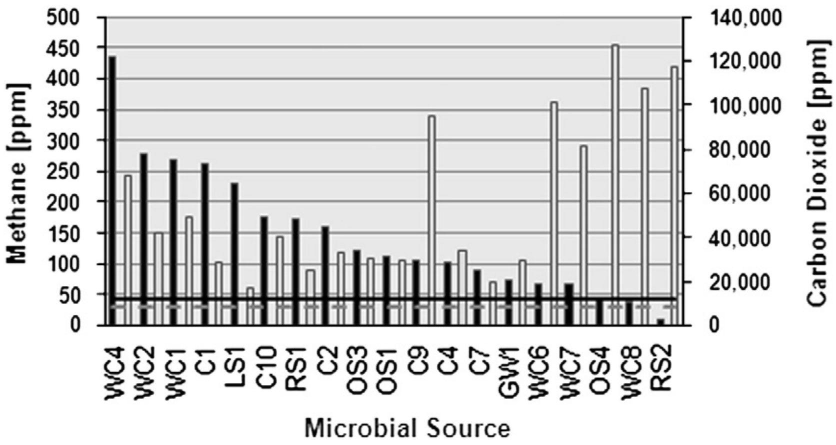

Fig. 2. Methane (black bars) and carbon dioxide (gray bars) generated from lignite samples inoculated with various microbial consortia (represented by the *x*-axis) after 30 days incubation period with no nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

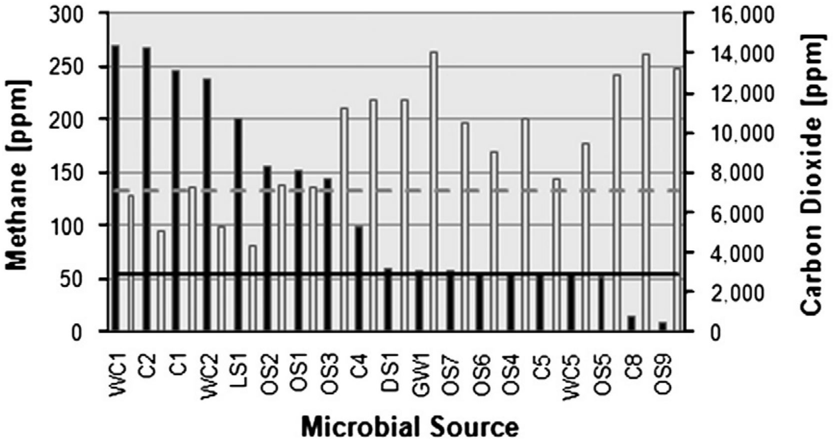

Fig. 3. Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal waste samples inoculated with various microbial consortia (represented by the *x*-axis) after 30 days incubation period with no nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

microbial breakdown of coal materials and utilization of intermediate coal degradation compounds to generate these gases.

The highest concentration of methane produced from the tested bituminous coal sample was nearly 300 ppm, while carbon dioxide exceeded 6000 ppm (Fig. 1). Generation of both methane and carbon dioxide from the lignite was considerably higher, reaching nearly 450 ppm and over 100,000 ppm, respectively (Fig. 2). Waste coal samples generated less methane than either bituminous coal or lignite (over 250 ppm) but doubled the amount of CO_2 in comparison to bituminous coal samples (Fig. 3).

3.2. 10% nutrients

Realizing that some nutrient addition might be necessary to stimulate the methanogenic population, nutrient amendments were examined at 10% and 50%. Amendment with 10% nutrient solution did not result in significantly higher methane production from bituminous coal, with one exception in which over 550 ppm was generated (Fig. 4). However, carbon dioxide concentrations in all bituminous coal samples were significantly increased, ranging from 5,000 to 30,000 ppm, corresponding to a 1.2-10 fold increase, relative to the samples with no nutrient amendments. A similar situation was observed with the lignite and coal waste samples. Total headspace methane in the lignite samples reached about 300 ppm, with one exception of 2000 ppm, while CO_2 generation was stimulated in all samples to 20,000–90,000 ppm, corresponding to a 1- to 2-fold increase (Fig. 5). Bituminous coal waste material produced up to 400 ppm CH_4, while carbon dioxide production was stimulated by 1–7 times, yielding 10,000-30,000 ppm (Fig. 6).

3.3. 50% nutrients

The increase of nutrients to 50% resulted in a significant increase in generated gases from the bituminous coal material, where up to 200,000 ppm of methane (160- to 660-fold increase) and 50,000 ppm carbon dioxide (3- to 8-fold increase) were produced (Fig. 7). One microbial consortium produced 110,000 ppm of methane in the 50% nutrient solution, while most of the lignite samples did not generate methane above 500 ppm (Fig. 8). Carbon dioxide generation was not significantly stimulated, yielding 40,000 to 120,000 ppm, similar to the 0% nutrient samples. Good microbial stimulation was achieved in the bituminous coal waste samples, where 120,000 ppm methane and up to 60,000 ppm carbon dioxide were produced, corresponding to up to 480-fold and 10-fold increases, respectively (Fig. 9).

4. Discussion

The results given in this paper represent a one-time measurement after a 30-day testing period. Consequently, it is not known whether the maximum conversion to methane or CO_2 was achieved or whether partial gas pressures in the reaction vessels limited the amount produced. Furthermore, only limited conclusions on conversion kinetics can be drawn from the results obtained.

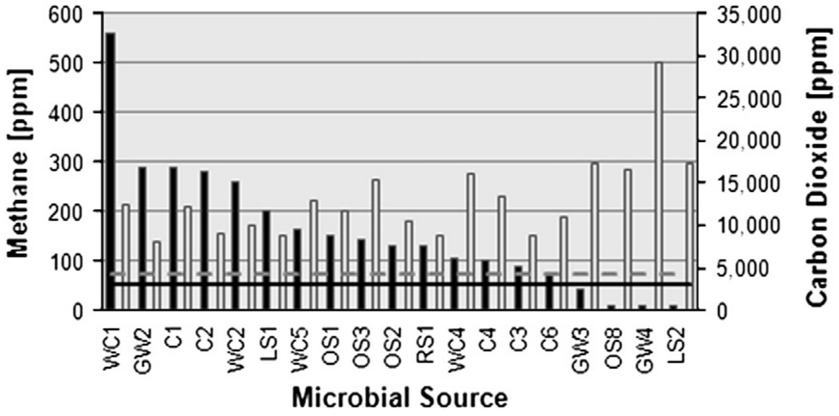

Fig. 4. Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal samples inoculated with various microbial consortia (represented by the *x*-axis) after 30 days incubation period with 10% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

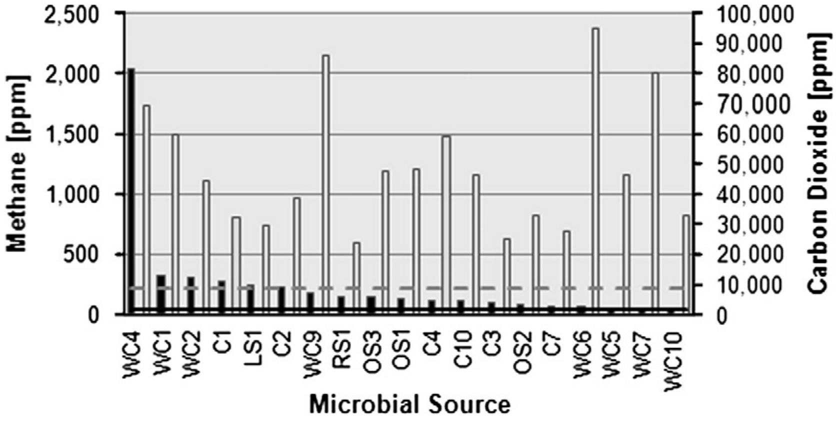

Fig. 5. Methane (black bars) and carbon dioxide (gray bars) generated from lignite samples inoculated with various microbial consortia (represented by the *x*-axis) after 30 days incubation period with 10% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

Analysis of duplicate samples showed standard deviation in methane concentration of 0.02 ppm at low concentrations to 20.82 ppm at high concentrations, or about 0.16% to 8.34%. Carbon dioxide measured from duplicate samples indicated a standard deviation between 1.2% and 20.4%. Only 3.89 ppm to 5.40 ppm methane and 1,210 ppm to 1597 ppm carbon dioxide were generated from no coal controls.

The highest concentrations of methane were obtained from samples inoculated with consortia cultured from coal or coal waste environments. This was expected, since coal populations contain native methanogens that are adapted to this environment. Interestingly, the highest concentrations of carbon dioxide were produced from samples inoculated with consortia cultured from other environments. These included oil seeps, natural gas wells lake sediments digester sludge and river sediments. This is an important finding, indicating that introduction of non-native species could increase the rate of hydrocarbon biodegradation, with CO_2 as an end product. It can be assumed that under proper environmental conditions (e.g., sufficient amount of hydrogen ions, appropriate temperature, etc.), a part of generated carbon dioxide would ultimately be converted to methane. Furthermore, the performance of tested consortia depended strongly on the composition of the media in which they were cultured.

Moreover, concentrations of both methane and carbon dioxide obtained from samples with no nutrient amendments were higher than in control samples, containing only normal saline solution and lower concentrations of native microbial populations. This strongly indicates that introduction of non-native species and/or higher concentrations of native species into solid hydrocarbon materials could potentially enhance the rates of gas production. Production of methane directly from coal sources showed a 2- to 7-fold increase over control samples.

Table 3 shows extrapolated gas generation potential. Gas concentration values produced in the headspace from 4 g of coal materials in 30 days were extrapolated into standard cubic feet of gas (scf) per ton of coal per year. This extrapolation is presented for ease of comparison with other published results. Nevertheless, scf/ton/year data extrapolations, in Table 3, as well those presented in other literature are based on many assumptions and have many limitations, and therefore should be considered with caution. From the results obtained, it is calculated that about 40 scf, 60 scf, and 36 scf of methane annually could be generated per ton of bituminous coal, lignite, and coal waste, respectively, using no nutrient amendments. To put this into perspective, commercially operated CBM wells may contain an order of magnitude of 100 scf adsorbed CH_4/ton of coal (Mastalerz et al., 2004).

Using an equation from Buswell and Neave (1930), the maximum theoretical conversion of nutrient organic content to methane and carbon dioxide can be calculated (Eq. (1)).

$$C_cH_hO_oN_nS_s + yH_2O \rightarrow xCH_4 + (c-x)CO_2 + nNH_3 + sH_2S \tag{1}$$

where

$$x = 0.125(4c + h-2o-3n + 2\,s) \tag{1a}$$

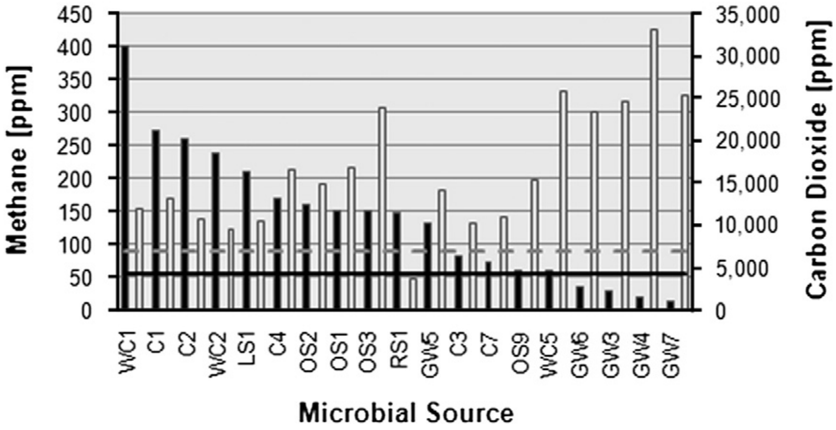

Fig. 6. Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal waste samples inoculated with various microbial consortia (represented by the *x*-axis) after 30 days incubation period with 10% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

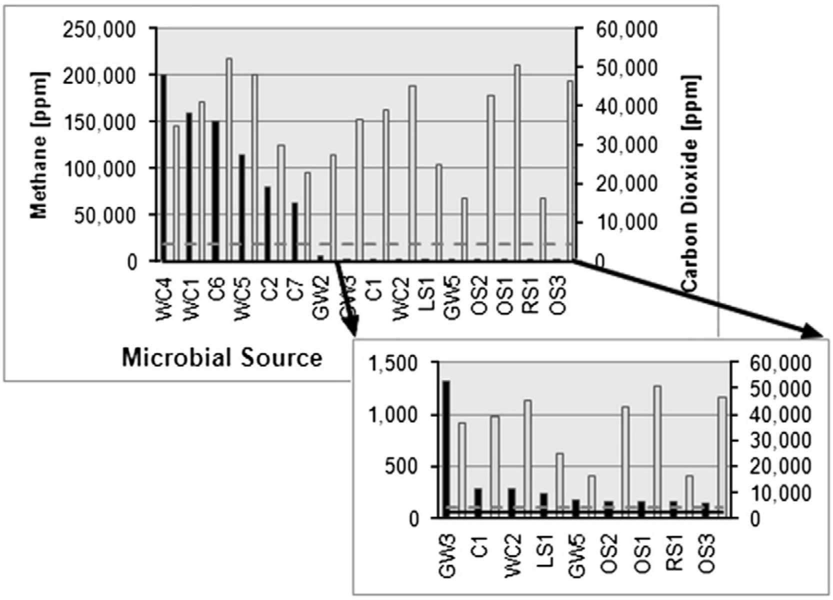

Fig. 7. Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal samples inoculated with various microbial consortia (represented by the *x*-axis) after 30 days incubation period with 50% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

$$y = 0.25(4c–h–2o + 3n + 2\,s) \qquad (1b)$$

Table 4 summarizes the best methane and carbon dioxide generation from the evaluated coal materials and compares them to the maximum theoretical nutrient conversion calculated from Eq. (1).

Addition of 10% nutrient solution increased the degradation of hydrocarbon material and subsequent methanogenesis. With addition of a 50% nutrient solution, potential methane generation increased to about 26,700 scf, 14,670 scf, and 16,000 scf per ton of the tested bituminous coal, lignite, and coal waste, respectively. Only a small fraction of methane (1–2%) and carbon dioxide (1–3%) generated from these samples was a direct result of nutrient conversion from the samples amended with 50% nutrients (Table 4).

The highest carbon dioxide concentrations were generated from lignite samples at all nutrient amendment levels. Lignite is the lowest metamorphosed and the softest hydrocarbon rock used in this study. High concentrations of carbon dioxide indicate the most rapid biodegradation of lignite as compared to other samples. Since methane generation from lignite at 0% and 10% nutrient amendment was also the highest of all of the coal sources considered, it is suspected that direct CO_2 conversion was responsible, as long as there was availability of hydrogen ions. Physicochemical analysis of the liquid phase of the samples after 30 days of incubation showed that pH of lignite was below 4.0, while that of bituminous coal and coal waste was circum-neutral. Carbon dioxide is a direct methane precursor and its high concentrations in the lignite samples could have resulted in increased methane generation. Moreover, the significant increase in

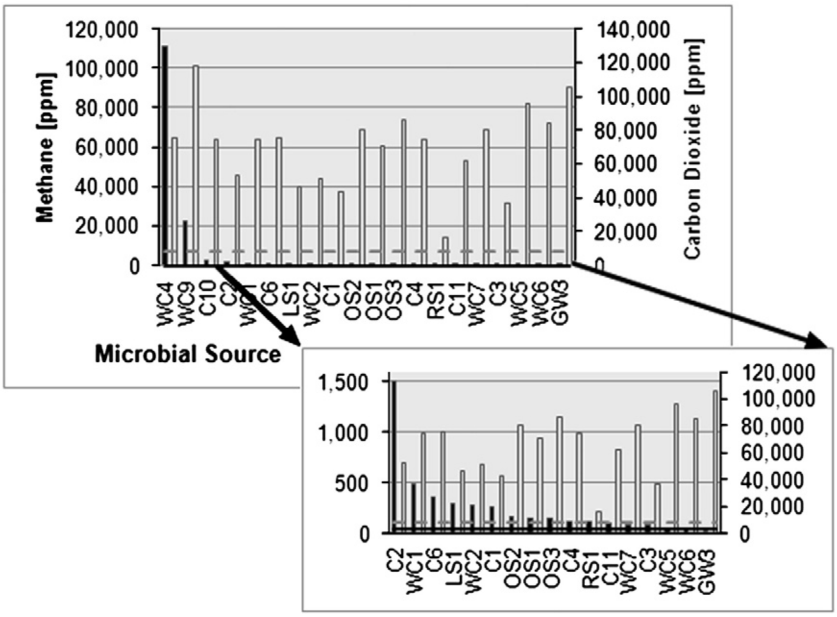

Fig. 8. Methane (black bars) and carbon dioxide (gray bars) generated from lignite samples inoculated with various microbial consortia (represented by the *x*-axis) after 30 days incubation period with 50% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

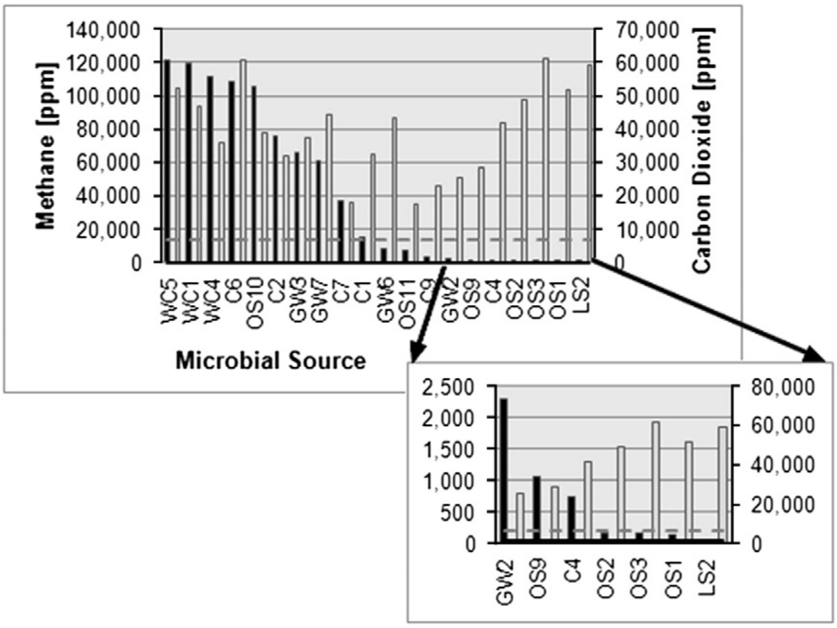

Fig. 9. Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal waste samples inoculated with various microbial consortia (represented by the *x*-axis) after 30 days incubation period with 50% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

Table 3
Values extrapolated from test results for maximum gas generation potential in scf/ton of hydrocarbon material/year.

	0% Nutrient		10% Nutrient		50% Nutrient	
	CH_4	CO_2	CH_4	CO_2	CH_4	CO_2
Bituminous coal	38.7	813.5	77.3	3867	26,671	6,668
Lignite	58.7	17,336	266.7	12,669	14,669	16,003
Coal waste	36.0	1,867	53.3	401	16,003	8001

methane concentrations with increasing nutrient levels could be responsible for no change in carbon dioxide concentrations.

Results from the literature report successful methane generation of between 10^1 and 8×10^3 scf per ton of coal per year (Jones et al., 2008, 2010; Strąpoć et al., 2011). Data presented here fall in a similar range. However, in many studies in the literature and in patented methods the nutrients and other materials added to the test systems are themselves a significant source of the methane increases observed. In other words, in many other studies, additional methane production observed may have come from the conversion of added nutrients. In this study, zero to only a small percentage (1–3%) of the gases were generated from samples amended with 50% nutrients from a direct conversion of nutrients (Table 4).

In this study, regardless of the nutrient amendment level, methane generated from bituminous coal waste materials was at the same order of magnitude as that produced from the analogous bituminous coal. With no nutrient amendment, bituminous coal waste materials generated up to 93% of the methane produced by bituminous coal samples. At 10% and 50% nutrient amendments, this proportion dropped to 69% and 60%, respectively. Moreover, with no additional nutrients, carbon dioxide concentrations detected from the bituminous coal waste samples were over twice as high as those produced from the bituminous coal itself. These bituminous coal waste materials represented materials that have probably undergone partial surface degradation, both chemically and biologically, and were therefore potentially more susceptible to subsequent initial microbial degradation. No conclusion as to whether the observed rates of methane and/or CO_2 production in any of the samples can be maintained for an extended period can be drawn from this study. Nevertheless, these results suggest that coal waste products, such as culm and gob, and potentially other waste hydrocarbon materials can be economically converted to useful fuel. It is likely that methane can be efficiently produced in a variety of application scenarios including typical bioreactors, above-ground, large-scale, waste pile reactor systems, and potentially underground in-situ reactors. The data suggest a significant economic impact in several areas, such as savings in coal mining, tar sands, and oil shale production fuel costs can possibly be obtained.

Table 4
The best CH_4 and CO_2 production from test results for lignite, bituminous coal, and coal waste, compared to the maximum theoretical gas generation from nutrient amendments.

		Nutrient	Bituminous coal	Lignite	Coal waste
0% Nutrient	CH_4 [ppm]	0	290	440	270
	CO_2 [ppm]	0	6100	130,000	14,000
10% Nutrient	CH_4 [ppm]	428	580	2000	400
	CO_2 [ppm]	286	29,000	95,000	33,000
50% Nutrient	CH_4 [ppm]	2143	200,000	110,000	120,000
	CO_2 [ppm]	1429	50,000	120,000	60,000

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