

*Full Length Research Paper*

# Physico-chemical characteristics of urban sediments and factors influencing ultimate methane yields

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The main objective of this study was to analyze sediments in 14 urban streams in the city of Busan, South Korea, and to evaluate their biochemical methane potentials (BMP) by use of conventional BMP tests. Then chemical oxygen demand (COD), proximate analysis, volatile solids (VS), organic carbon content and elemental analysis were conducted to determine sediment characteristics. The results showed that COD, VS and organic carbon contents were 15.2 ~ 75.1 mg/g, 2.3 ~ 11.5% and 1.3 ~ 34.2%, respectively. From BMP tests, the ultimate methane and carbon dioxide yield (mL/g VS) and biodegradability (%) were determined to be 10.1 ~ 179.4, 10.3 ~ 34.4, and 4.0 ~ 30.1, respectively. To determine the correlations between ultimate methane yield, C/N ratio, COD, and VS and organic carbon content, a linear model was fitted to the data using a least-squares algorithm. Except for COD ( $r^2 = 0.7586$ ) and VS ( $r^2 = 0.7876$ ), the linear model was well fitted to each data point with good correlation coefficient values ( $r^2 = 0.9795 \sim 0.9858$ ). A multivariate regression analysis was also performed to find multiple effects of factors on the ultimate methane yield. From these findings, empirical equations are provided that contain C/N or VS ratios for the prediction of ultimate methane yields from urban stream sediments in Busan.

**Key words:** Sediments, urban streams, C/N ratio, chemical oxygen demand (COD), biochemical methane potentials (BMP) test, volatile solids.

## INTRODUCTION

Beginning in the 1970s, South Korean urban areas started to become densely populated in parallel with industrial development, and urban streams were polluted by the illegal discharge of used waste water. The organic compounds in waste water settled as sediments where the organic compounds degraded under anaerobic conditions before being released into the atmosphere as such greenhouse gases as methane and carbon dioxide (Persson, 1982; Smith et al., 1995; Dawson, 1998; Pouria et al., 1998). At present, Korean water quality management focuses on the treatment of domestic and industrial wastewater before discharge into rivers and streams, and there have been few studies on the relationship between stream sediments, water quality and the release of greenhouse gases from stream sediments (Yoon et al., 2006). Methane is considered to be the

second most important greenhouse gas following carbon dioxide. In general, 20% of methane released into the atmosphere is believed to originate in natural wetlands and rivers (Khalil et al., 1994). Organic compounds in river sediments are degraded into low-molecular-weight compounds by methanogens (Rees, 1980; Gujier et al., 1983; Christophersen et al., 1996). Factors affecting anaerobic degradation of organic compounds are pH, temperature, nutrients, and presence or absence of toxic compounds.

In general, methanogens play a role in the anaerobic degradation of organic compounds at pH between 6.0 and 8.3, but efficiency of anaerobic degradation lessens at low pH due to the increase of heavy metals in solution (Angelidaki et al., 1994). Similarly, higher temperatures (>55°C) may inhibit metabolism of methanogens due to the degeneration of enzyme. Methanogens are more sensitive to nutrients than other microorganisms, and typically produce maximum methane in anaerobic digestion at a carbon-nitrogen (C/N) ratio between 12 and 16 (Song et al., 2004; Chi et al., 2011; Forster-Carneiro

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**Table 1.** Characteristics of 14 streams selected for study in Busan, South Korea.

Stream site	Length (km)	Area (km <sup>2</sup> )	Annual water resource (10 <sup>6</sup> m <sup>3</sup> )	Run-off (%)	Low flow	Drought flow Q355	Minimum flow Q275	Normal flow Q185	Flood flow Q95
A: Chun	6.30	16.37	27.07	60.0	0.012	0.064	0.153	0.305	0.521
B: Oncheon	14.13	56.28	86.67	57.7	0.039	0.203	0.489	0.978	1.669
C: Woodong	1.00	3.7	5.66	56.7	0.002	0.040	0.050	0.070	0.130
D: Bujeon	4.19	7.66	9.04	55.8	0.016	0.021	0.046	0.098	0.211
E: Bosu	3.80	8.17	11.19	55.7	0.020	0.026	0.057	0.121	0.261
F: Gudeok	0.6	1.8	2.74	55.7	0.004	0.006	0.013	0.027	0.058
G: Seokdae	7.75	22.55	30.97	61.7	0.187	0.200	0.240	0.310	0.500
H: Songjeong	4.70	17.14	23.22	55.9	0.027	0.044	0.096	0.149	0.274
I: Hakjang	5.40	19.42	31.29	59.2	0.040	0.050	0.090	0.170	0.360
J: Daeri	1.60	4.30	6.24	62.0	0.094	0.100	0.110	0.124	0.166
K: Manhwa	2.50	8.15	12.73	58.9	0.006	0.030	0.072	0.144	0.245
L: Jukseong	4.50	17.03	25.56	59.0	0.012	0.063	0.150	0.301	0.513
M: Seobu	3.00	4.42	6.90	58.9	0.003	0.016	0.039	0.078	0.133
N: Ilgwang	6.20	19.24	29.44	63.1	0.019	0.044	0.133	0.156	0.303

Data are from the Busan Development Institute (2006).

et al., 2010). The biochemical methane potential (BMP) test is a batch incubation test for the measurement of anaerobic biodegradability of organic compounds under an anaerobic environment where temperature and pH are maintained at optimum conditions, and microorganisms and nutrients are provided (Owen et al., 1979; Shelton et al., 1984; Chen et al., 1995). In this study, 14 urban streams in the city of Busan, Korea was selected. Busan, in the southeastern part of the country is Korea's second most populous city with 3.6 million residents. Sediment and water samples were collected and analyzed for chemical oxygen demand (COD), ignition loss, total organic carbon and heavy metals.

In addition, five sediments that showed wide range of organic content underwent biochemical methane potential (BMP) testing to determine possible methane production. Relationships

between potential methane production and other factors were also analyzed.

## MATERIALS AND METHODS

### Stream studies

The city of Busan has 44 streams with a total length of 192 km. Of these, 23 are located in the Kangseo and Kijang districts. In all, 14 streams were selected for study. Characteristics and locations of the 14 are summarized in Table 1 and shown in Figure 1. Sediment samples were collected from three sites of each stream at the upper, middle and lower sections. Samples were collected three times at each point and mixed homogeneously. Then, all samples were stored in a refrigerator at 4°C before analyses.

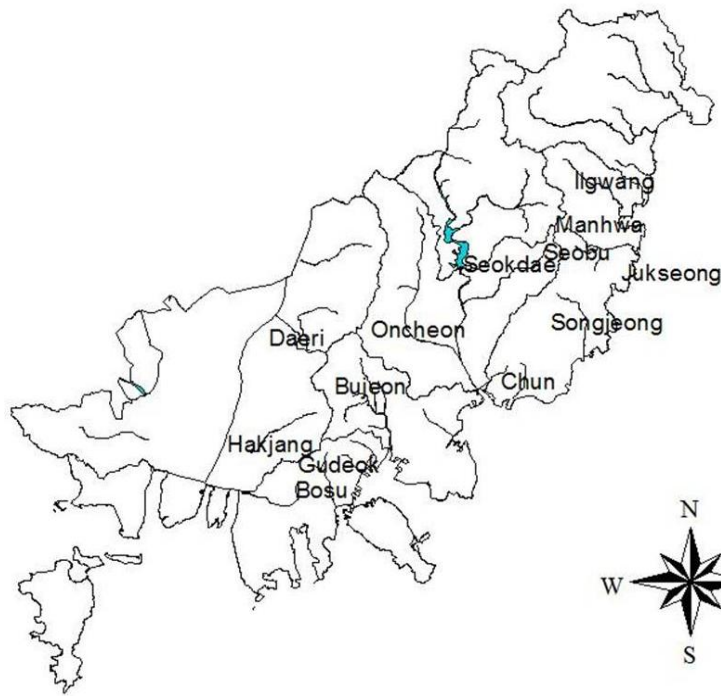
### Sediment analyses

Sediment pH was measured according to the method

described by the Soil Environment Conservation Act of the Korea Ministry of Environment. Samples were dried in a drying machine at temperatures of 105 to 110°C for 24 h, and then 5 g of sample was mixed with 25 ml of distilled water. Next, pH was measured with a Corning® pH electrode. The pH meter was calibrated with pH 4.0, 7.0 and 10.0 standards. Ignition loss of sediments was measured by determining differences in weights before and after heating of 5 g of sample at 550°C for 2 h. COD of sediments was measured according to the method described by the soil environment conservation act using 0.1 N K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. Elemental analyses and organic carbon contents of sediments were conducted on samples, which had been dried and crushed, using a Macro and Micro Elemental Analyzer (Elementar, Germany) and total organic carbon analyzer (Shimadzu, Japan).

### Biochemical methane potential test

The anaerobic mineral medium developed by Shelton and Tiedje (1984) was used for the BMP test. The medium consisted (per liter) of the following components:



Sites	Latitude	Longitude
Chun stream	35°09'27.67" N	129°09'11.27" E
Oncheon stream	35°12'23.64" N	129°04'41.58" E
Woodong stream	35°10'17.62" N	129°08'22.98" E
Bujeon stream	35°11'14.03" N	129°02'29.53" E
Bosu stream	35°07'26.45" N	129°00'31.82" E
Gudeok stream	35°07'22.52" N	129°01'06.38" E
Seokdae stream	35°13'38.35" N	129°08'50.58" E
Songjeong stream	35°11'49.25" N	129°12'25.78" E
Hakjang stream	35°08'39.33" N	128°59'25.08" E
Daeri stream	35°12'05.94" N	129°00'14.95" E
Manhwa stream	35°14'49.86" N	129°12'56.00" E
Jukseong stream	35°14'43.68" N	129°13'23.98" E
Seobu stream	35°14'47.26" N	129°13'09.82" E
Ilgwang stream	35°15'58.93" N	129°14'04.20" E

Figure 1. Map showing the studying area in Busan, South Korea.

phosphate buffer, 0.27 g of  $KH_2PO_4$  and 0.35 g of  $K_2HPO_4$  (adjusted to pH 7.0); mineral salts, 0.53 g of  $NH_4Cl$ , 75 mg of  $CaCl_2 \cdot 2H_2O$ , 100 mg of  $MgCl_2 \cdot 6H_2O$ , and 20 mg of  $FeCl_2 \cdot 4H_2O$ ; and trace metals, 0.5 mg of  $MnCl_2 \cdot 4H_2O$ , 0.05 mg of  $H_3BO_3$ , 0.05 mg of  $ZnCl_2$ , 0.03 mg of  $CuCl_2$ , 0.01 mg of  $NaMo_4 \cdot 2H_2O$ , 0.5 mg of  $CoCl_2 \cdot 6H_2O$ , 0.05 mg of  $NiCl_2 \cdot 6H_2O$ , and 0.05 mg of  $Na_2SeO_3$ . The medium was autoclaved for 10 min to drive off  $O_2$  and cooled to 35°C. Sludge samples were obtained from secondary anaerobic digesters in 2-L jars. The sludge samples were from waste water treatment plants in Busan. A 10% diluted sludge sample was prepared by adding 1 part of filtered sludge to 9 parts of mineral medium. Sediment samples were inserted in 500-ml capacity serum bottles containing a 10% diluted sludge sample, and then  $N_2$  gas was sparged into the bottle to remove  $O_2$ . Total gas production ( $CH_4 + CO_2$ ) was measured by manometer (Figure 2). Methane production was measured by injecting 0.1 ml of headspace gas from serum bottles into a gas chromatograph (GC).

An HP 5890 GC equipped with a thermal conductivity detector and a POCAPAK Q column (2.4 m x 3.2 mm) was used to measure the gas concentrations of methane and carbon dioxide. A 0.1-ml of sample was manually injected into the GC. Temperatures of the injector and detector were 100 and 200°C, respectively. The temperature of column was held at 40°C.

Methane production ( $V_{CH_4}$ ) was corrected by the following equation:

$$V_{CH_4}(35^\circ C) = C_1(V_1 + V_0) - C_0V_0 \quad (1)$$

Where  $V_{CH_4}$  indicates produced methane volume (ml),  $C_1$  = methane content (%) at sampling time,  $C_0$  = methane content (%) at previous sampling time,  $V_1$  = biogas volume measured by syringe (ml) and  $V_0$  = gas phase volume of the reactor (ml).

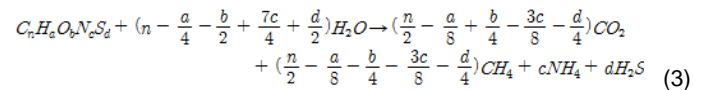
Ultimate methane yield and kinetic constant were obtained using

the following equation:

$$B = B_u(1 - e^{-kt}) \quad (2)$$

Where  $B_u$  = ultimate methane yield (ml  $CH_4$ /g VS),  $B$  = accumulated methane yield (ml  $CH_4$ /g VS) and  $k$  = kinetic constant ( $d^{-1}$ ).

Theoretical methane yield was estimated using Buswell's equation:



Where  $n$  = carbon content (%) / 12,  $a$  = hydrogen content (%) / 1,  $b$  = oxygen content (%) / 16,  $c$  = nitrogen content (%) / 14 and  $d$  = sulfur content (%) / 32.

Biodegradability of sample was calculated using the following equation where biodegradability is defined as the ratio of ultimate methane yield obtained by BMP test to theoretical methane yield estimated using Buswell's equation:

$$Bio\ deg\ radability = \frac{Ultimate\ methane\ yield}{Theoretical\ methane\ yield} \times 100 \quad (4)$$

## RESULTS

### Physico-chemical characteristics of sediments

Results for the sediment analyses are summarized in

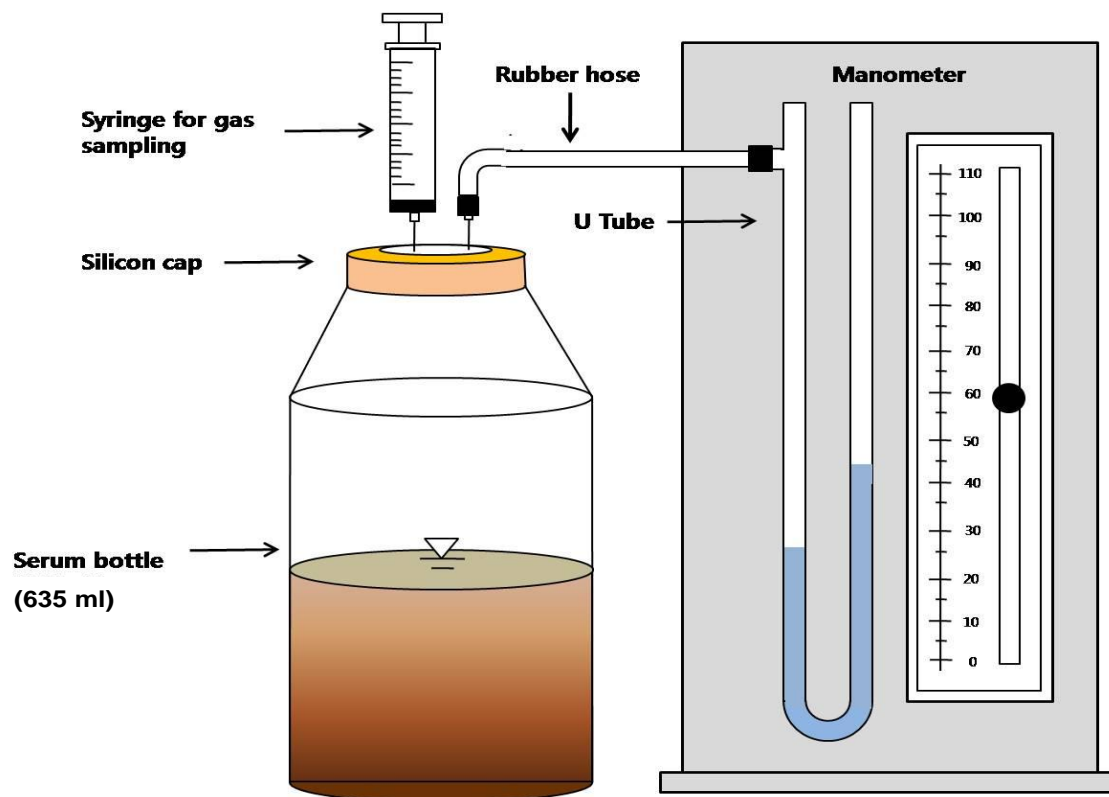


Figure 2. Schematic of BMP test set-up.

Table 2. Water content ranged from 26.8 to 91.0%. Ash content (non-flammable mineral content) ranged from 3.2 to 71.2%. Parameters indicating organic matter contents are COD, ignition loss and organic carbon contents. Values for these parameters ranged from 1.20 to 75.07 mg/g, 0.19 to 11.54%, and 0.17 to 34.21% for COD, ignition loss and organic carbon, respectively.

### Biochemical methane potential test

Five sediment samples obtained from Bosu, Gudeok, Manwha, Oncheon and Songjeong streams were selected for BMP tests. These samples showed broad ranges of COD, volatile solids (VS) and organic carbon content. Samples were selected to cover low, medium, and high values of COD, VS and organic carbon content. For example, samples Bosu and Gudeok showed high COD (73.07 and 75.07, mg/g respectively), while sample Oncheon showed medium COD (38.00 mg/g). Samples Manwha and Songjeong had low COD (18.93 and 15.20 mg/g, respectively).

### Variation of pH

Anaerobic digestion usually occurs between pH 6 and 8.3,

but optimum pH of methanogenes are between pH 7 and 8 (Speece, 1996). The initial pH of reactors in this study was 7.0 and the final pH of reactors after the BMP tests were 7.11 to 7.35. Thus, test conditions for pH were well maintained during the tests.

### Cumulative methane and carbon dioxide production

Results for BMP tests on samples from Bosu, Gudeok, Manwha, Oncheon and Songjeong streams are shown in Figure 3. These streams were selected for BMP tests because they showed broad range of organic matter contents. All tests were finished within 6 days and 90% of methane and carbon dioxide produced during the tests was produced within 5 days. Ultimate methane yield and kinetic constant were obtained using Equation 2. The model was fit to the data using least-squares algorithms. Results of ultimate methane yield and kinetic constants are summarized in Table 3. Ultimate methane yields for the five sediment sources shown in Table 3 were 179.4, 112.4, 24.3, 21.3 and 10.1 ml CH<sub>4</sub>/g VS for Bosu, Gudeok, Songjeong, Manwha and Oncheon, respectively. For the same sites, ultimate carbon dioxide yields were 34.4, 28.1, 12.5, 10.9 and 10.3 ml CO<sub>2</sub>/g VS. It is hard to compare my results to those for other stream sediments

**Table 2.** Summary of physico-chemical analyses of stream sediment.

Stream site	pH	COD <sub>Cr</sub> (mg/g <sup>-1</sup> )	Volatile solid (%)	Organic carbon content (%)	Elemental analysis (wt, %)					Proximate analysis (%)			
					N	C	H	S	C/N	Moisture	Volatile matter	Fixed carbon	Ash
A: Chun	7.17	16.27	0.86	1.00	0.94	0.99	0.49	0.21	1.05	39.8	2.7	0.01	57.5
B: Oncheon	7.95	38.00	5.25	2.67	1.59	2.31	0.62	0.23	1.45	55.4	3.3	0.06	41.3
C: Woodong	7.50	3.87	0.55	0.77	0.9	0.68	0.33	0.33	0.76	32.7	1.7	0	65.6
D: Bujeon	7.90	15.33	4.95	1.03	0.98	1.05	0.60	0.10	1.07	33.2	3.2	0.02	63.5
E: Bosu	6.32	73.07	11.54	34.21	3.46	35.54	5.10	1.09	10.27	91.0	5.8	0.02	3.2
F: Gudeok	6.34	75.07	11.34	23.13	2.24	16.06	3.62	0.77	7.17	88.2	6.0	0.02	5.8
G: Seokdae	7.80	11.07	0.27	0.23	1.25	0.32	0.34	0.51	0.26	26.8	2.0	0	71.2
H: Songjeong	7.66	15.20	2.34	1.28	1.30	1.36	0.63	0.22	1.05	38.5	2.8	0	58.7
I: Hakjang	8.21	1.20	0.91	0.33	1.01	0.44	0.58	0.11	0.46	31.8	3.3	0	64.9
J: Daeri	7.71	6.13	0.19	0.35	1.02	0.43	0.21	0.36	0.42	27.5	1.3	0	71.2
K: Manhwa	7.39	18.93	1.33	1.96	1.41	2.10	0.43	0.33	1.49	31.6	2.2	0	66.2
L: Jukseong	7.57	7.07	0.29	0.17	0.76	0.20	0.27	0.11	0.26	28.3	1.4	0.03	70.2
M: Seobu	7.56	30.27	4.83	1.31	0.90	1.16	0.61	0.33	1.29	48.0	3.4	0	48.6
N: Ilgwang	8.01	13.60	3.66	1.06	1.04	1.16	0.52	0.23	1.12	47.3	4.1	0.01	48.6
Mean	7.51	23.22	3.45	4.96	1.34	4.56	1.02	0.35	3.40	44.3	3.1	0.01	52.6

because most previous BMP tests were conducted on solid waste. The ultimate methane yields for the Bosu and Gudeok samples (179.4 and 112.4 ml CH<sub>4</sub>/g VS, respectively) were lower than for fish residue (277 ml CH<sub>4</sub>/g VS) but higher than for paper (144.3 ml CH<sub>4</sub>/g VS) (Cho et al., 1993).

The ultimate methane yields of other samples were similar to vegetables and fabrics (27.0 and 19.5 ml CH<sub>4</sub>/g VS, respectively) but higher than for excavated soil (4.6 ml CH<sub>4</sub>/g VS) (Cho et al., 1993).

## DISCUSSION

### Biodegradability

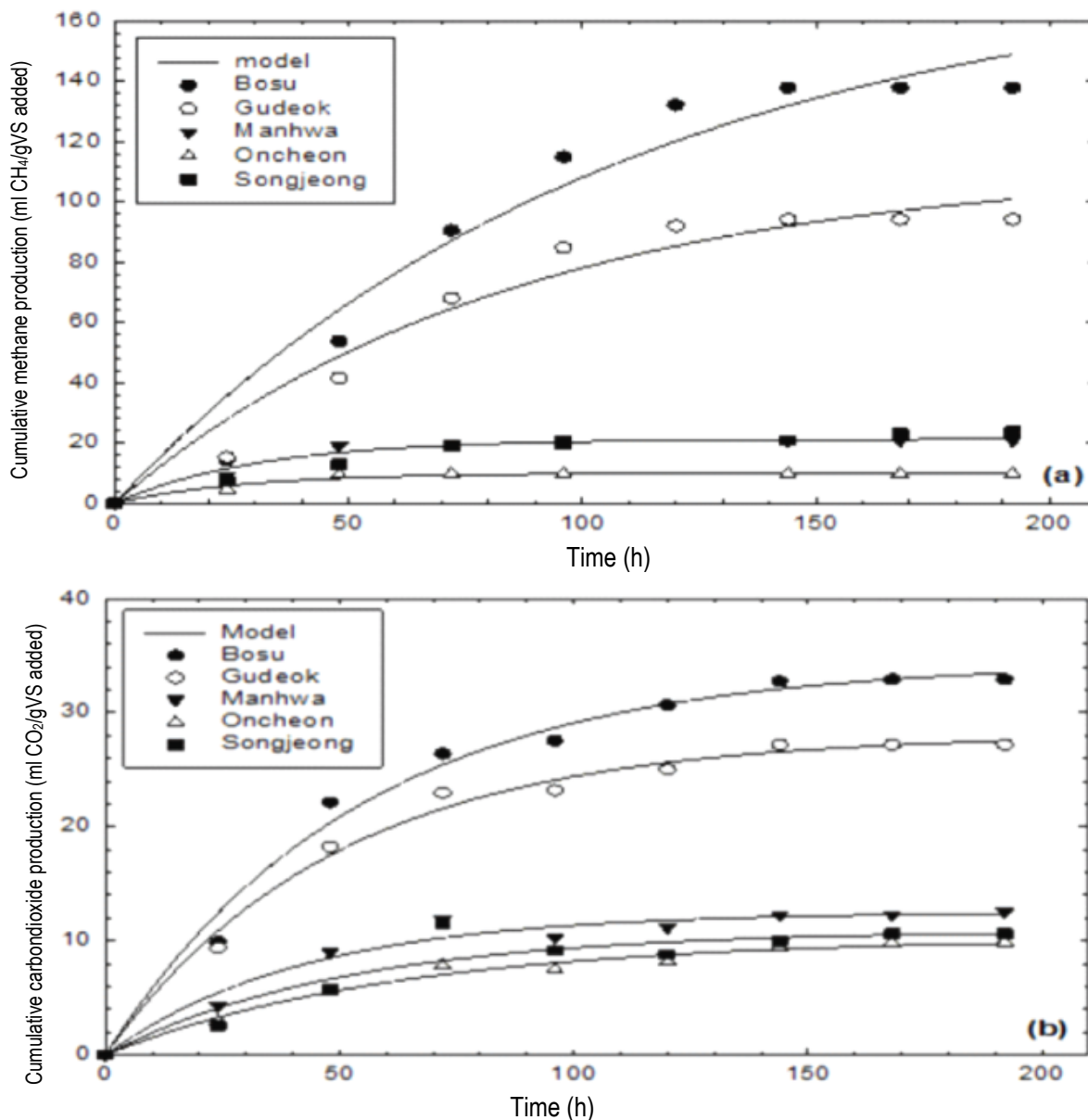
Biodegradability of sediments was estimated

using Equation 4 and is summarized in Table 3. Biodegradability of Bosu and Gudeok samples was much higher than for samples obtained from Manhwa, Oncheon and Songjeong. This can be explained by the high C/N values. For example, the C/N ratios of Bosu and Gudeok were 10.3 and 7.2 but those of the others were 1.5, 1.5 and 1.1, respectively. In general, degradation of organic matter accelerates when the C/N sample value is in the range of 12 to 16 (Song, 2004).

### Relationships between ultimate methane yield and physico-chemical characteristics of sediments

Organic matter included in sediments is the most important factor for the production of methane

during BMP testing because other factors such as pH, temperature and nutrients are set at constant values. Thus, relationships between the ultimate methane yield and factors related to organic matter in sediments (that is, C/N, TOC, VS and COD) were investigated and the results are shown in Figure 4, which also shows the relationship between carbon dioxide and ultimate methane yield. Because methane and carbon dioxide are final products of anaerobic degradation of BMP testing, methane production is linearly proportional to carbon dioxide production as shown in Figure 4; however, the volume of carbon dioxide produced was much lower than methane. VS and COD appeared to have a modest effect on ultimate methane production, but C/N and TOC appeared to be the most important factors. A multivariate regression



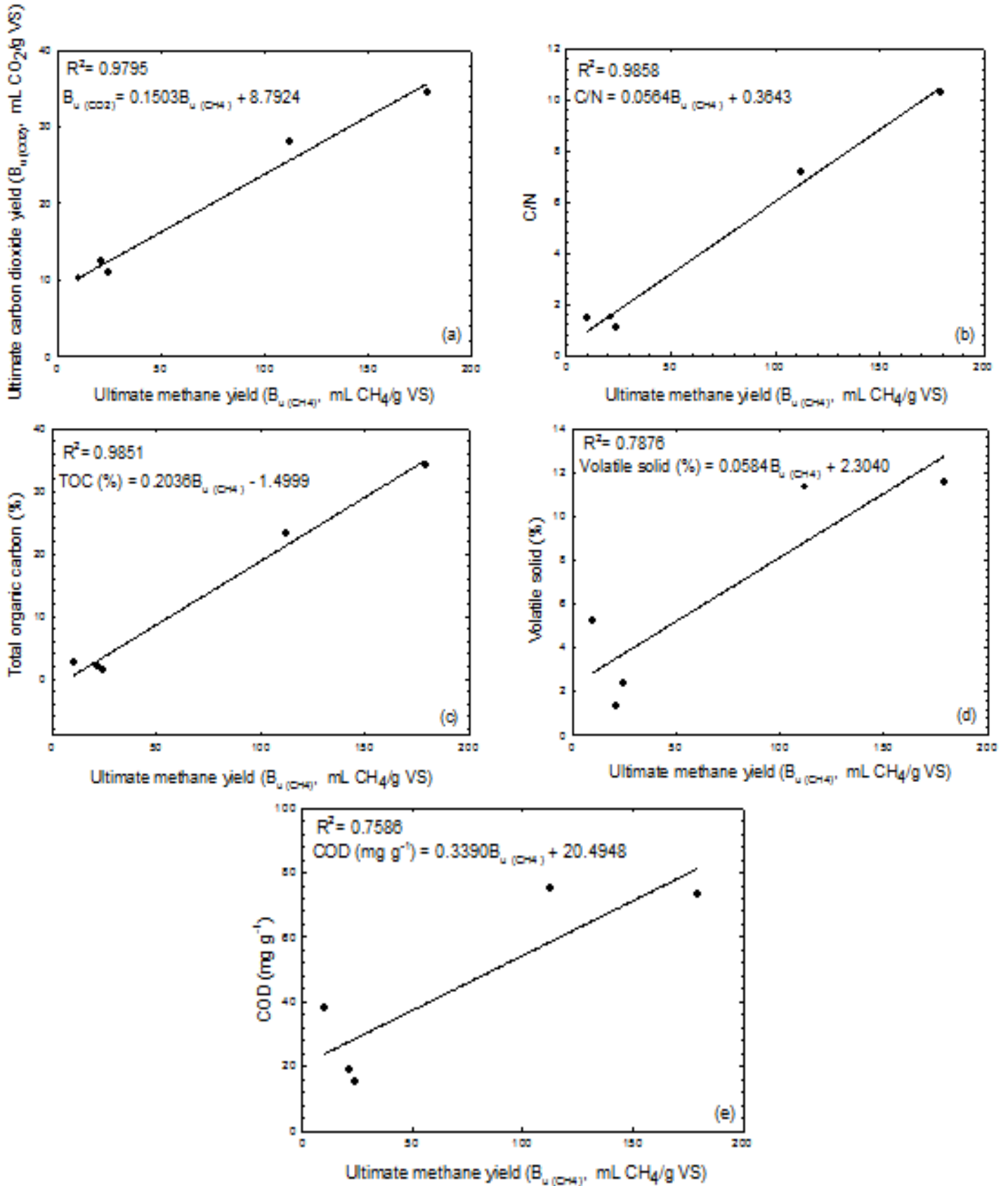
**Figure 3.** BMP test results for samples from Bosu, Gudeok, Manwha, Oncheon and Songjeong streams.

**Table 3.** Ultimate methane and carbon dioxide yield and kinetic constant for each sediment sample.

Sediment source	Ultimate methane yield (B <sub>2</sub> , ml CH <sub>4</sub> /g VS)	Kinetic constant for methane (k, 1/day)	Ultimate carbon dioxide yield (B <sub>2</sub> , ml CO <sub>2</sub> /g VS)	Kinetic constant for carbon dioxide (k, 1/day)	Biodegradability (%)
Bosu	179.4	0.222	34.4	0.447	30.1
Gudeok		0.285	28.1	0.489	42.2
Manwha	21.3	0.784	12.5	0.489	4.0
Oncheon	10.1	0.864	10.3	0.386	4.0
Songjeong	24.3	0.410	10.9	0.470	5.8

analysis was conducted at the 0.05 significance level to define relationships between ultimate methane

production and C/N, TOC, volatile solid and COD. The equation used for this analysis is ( $R^2 = 0.987$ ):



**Figure 4.** Relationships between ultimate methane yield, volatile solids (VS), chemical oxygen demand (COD) and organic carbon content (TOC); and also the relationship between carbon dioxide and ultimate methane yield.



$$B_{u(CH_4), mL CH_4/g VS} = 0.96 + 21.3 C/N - 3.58 VS \quad (5)$$

Where  $B_u$  is ultimate methane yield, C/N is a ratio of carbon and nitrogen and VS is a volatile solid content (%).

## Conclusions

The objective of this study was to evaluate the potential methane production of sediments obtained from 14 urban streams in Busan, Korea. A series of elemental analyses and BMP tests were conducted to evaluate the physico-chemical characteristics and potential methane production of sediments. The primary findings and the results of the BMP tests are as follows:

- 1) pH, COD, VS, and organic carbon content ranged from 6.3 to 8.0, 15.2 to 75.1 mg/g, 1.3 to 11.5% and 0.2 to 34.2%, respectively.
- 2) Gas analyses for BMP tests indicated that more than 90% of gas was generated within 5 days and no more gas was generated after 6 days. The data obtained from BMP tests were fitted to an analytical solution. Equation 2 was used to obtain the ultimate methane and carbon dioxide yields. The ultimate methane yields ranged from 10.1 to 179.4 ml CH<sub>4</sub>/g VS and 10.3 to 34.4 ml CO<sub>2</sub>/g VS for carbon dioxide.
- 3) High values of biodegradability were observed for sediments of Bosu and Gudeok streams, indicating the presence of easily degradable organic matter in both sediments. In contrast, non-degradable organic matter is believed to be in sediments of Oncheon, Songjeong and Manhwa streams because of the low biodegradability values.
- 4) C/N and organic carbon content were the most important factors affecting ultimate methane yields. However, Equation 5, which includes C/N and VS values obtained by the multivariate regression analysis showed a slightly higher correlation coefficient (0.987) than the equations containing factors such as C/N and organic carbon content.

## ACKNOWLEDGEMENTS

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