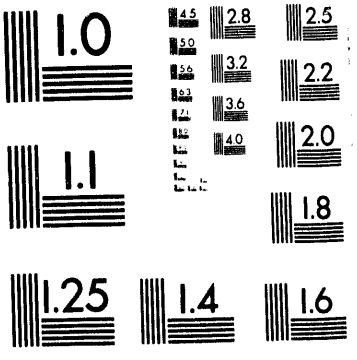
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DOE/MC/27226-3568

DEVELOPMENT OF BIOLOGICAL COAL GASIFICATION (MicGAS PROCESS)

12th Quarterly Report

DOE-METC Contract No. DE-AC21-90MC27226

Submitted to:

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DISCLAIMER

DEVELOPMENT OF BIOLOGICAL COAL GASIFICATION
(MicGAS PROCESS)

1. CONTRACT: For the DOE contract # DE-AC21-90MC27226 MOD A000 following tasks were completed:

Task 1. NEPA Compliance and Updated Test Plan
This task has been completed.

Task 2. Enhance Methane Production

Subtask 2.1 Bacterial Strain Improvement. Several experiments were conducted to study the efficiency of granulated sludge consortium (GSC) on the biomethanation of Texas lignite (TxL). With an aim of obtaining a better culture than Mic-1, GSC was used as inoculum at different concentrations (0%, 1% and 10%). The experiments were carried out in 60-mL vials containing 40 mL 0.1% SNTM + TxL + GSC.

1. The first experiment was conducted under anaerobic conditions (N_2:CO_2) in 60-mL vials containing 40 mL 0.01% SNTM + 1% TxL + 10% GSC. All vials (control and experimental) were incubated under static conditions at 37°C. Methane production was measured periodically in the vial headspace in all experimental vials. After 20 days of incubation, methane was found to be up to 67 mole%. VFA analysis shows insignificant quantities of acetic, propionic, butyric, isobutyric, valeric and isocaproic acids. Caproic acid was at 32-34 ppm concentration which is unusual. As this was the first experiment with the GSC, the occurrence of caproic acid will be further investigated.

2. The second experiment was conducted to determine whether CH_4 production was from biogasification of coal or from substrates used for growing the GSC. The GSC was maintained in acetate, butyrate and propionate medium. Some "residual" quantities of these substrates might affect CH_4 production. That Is why this experiment was conducted as the previous one, but 1% or 10% "washed or unwashed" GSC was used. The GSC was "washed" three times with sterile distilled H_2O (40-45 mL). Methane accumulation was negligible (8 cc) in the vials that contained only 1% GSC ("washed or unwashed") during the cultivation period. There was no significant difference in CH_4 production in the vials that contained 10% "washed or unwashed" GSC. The average CH_4 produced for this experiment was 61 cc in each vial.

3. The effect of two different anaerobic conditions (N_2:CO_2 and H_2:N_2) on biomethanation of Texas lignite was also studied. The logic for this experiment was to test whether H_2 in H_2:N_2

mixture can serve as an H_2 donor to the Ch_4 formation during biogasification of coal at 1% and 10% solids loadings. The experiment was conducted in 60-mL vials that contained 0.1% SNTM + 1% or 10% TxL + 10% GSC in two sets. The medium in each set of control and experimental vials was purged with a mixture of deoxygenated H_2:N_2 (60:40) or N_2:CO_2 (80:20).

There was no significant difference in the total biogas production between the control and experimental vials. However, in the "N_2:CO_2" set total gas production was approximately 15% higher. The cumulative methane production decreased with an increase in the solids loadings from 1% to 10%. While CO_2 concentrations increased in both experimental sets (Figure 1). These results confirm our previous observations on the inhibitory effect of increased solids loadings on biomethanation of TxL. This phenomenon might be due to the excessive liberation of CO_2 at higher TxL loadings and will be further investigated.

There were slight differences in the protein and COD concentrations in the experimental vials with 1% and 10% TxL. Maximum protein content was observed in the vials that contained 10% TxL and head space of N_2:CO_2. On the other hand, maximum COD concentration was not affected by the composition of head space gas mixture. The two anaerobic conditions used (H_2:N_2 and N_2:CO_2) had insignificant influence on COD (39.6 and 34.1 g O_2/L, respectively). These COD values in the experimental vials were 5-6 times higher compared to those in control vials.

The concentration of VFAs varied during the course of the experiments (Figures 2-4). In both experimental sets (with 1% and 10% TxL) less propionate and isobutyrate was observed than in the control vials. Nonetheless, in all cases propionate accumulation was the highest among the VFAs monitored. This result indicates a limiting factor for overall CH_4 production from the TxL. Maximum isovalerate concentration (60.75 ppm) was obtained on day 21 in experimental vials that contained 10% GSC and 10% TxL under N_2:CO_2. Heptanoic acid was observed in the experimental vials after 10-14 days of cultivation. Maximum production of this acid (26.45 ppm) was observed on day 21 in the experimental vials containing 1% TxL. The appearance of isovaleric acid peak coincided with maximum concentration of heptanoic acid. This phenomenon was not observed in any of the previous experiments with TxL and Mic-1 consortium. The appearance of heptanoic and isocaproic acids seems to be due to the metabolism of GSC on the TxL. Further experiments to confirm the accumulation ofpropionate (Figure 4), and higher production of isovalerate and heptanoate (Figure 3) are planned.

Subtask 2.2 Addition of Co-Substrate. Inoculum for the experiment on the addition of co-substrates (such as methanol, citrate, oxalate, succinate, etc.) for enhancement of TxL biomethanation has been prepared. The inoculum was obtained by centrifuging the liquid contents from the vials, that contained 1% TxL and 10 mM citrate. The experiment is planned for the next reporting period.

Subtask 2.3 Low-cost Nutrient Amendment. For this experiment, several samples of sewage sludge have been taken from a local wastewater treatment plant. The plan is to solubilize the sludge with NaOH and then use it as a nitrogen supplement to evaluate its effectiveness in comparison with Hoffmann's Dried blood and Sheftone T™. Cost analysis will be done in order to evaluate the cost effectiveness of the medium for biogasification of Texas lignite.

Conclusions:

- Granulated sludge consortium (GSC) could be used for biomethanation of TxL.
- "Washed or unwashed" GSC did not influence CH_4 production.
- Anaerobic conditions containing deoxygenated N_2:CO_2 anaerobic conditions provide better biomethanation of TxL.
- The appearance of heptanoic and isocaproic acids seems to be due to the metabolism of GSC on TxL.

Planned Future Work:

- Study the effect of pH of the culture medium on biogasification of TxL.
- Investigate the effect of co-substrates on biomethanation of TxL.
- Confirm the effect of methanol as an additional hydrogen donor for enhancement of biogasification of TxL.

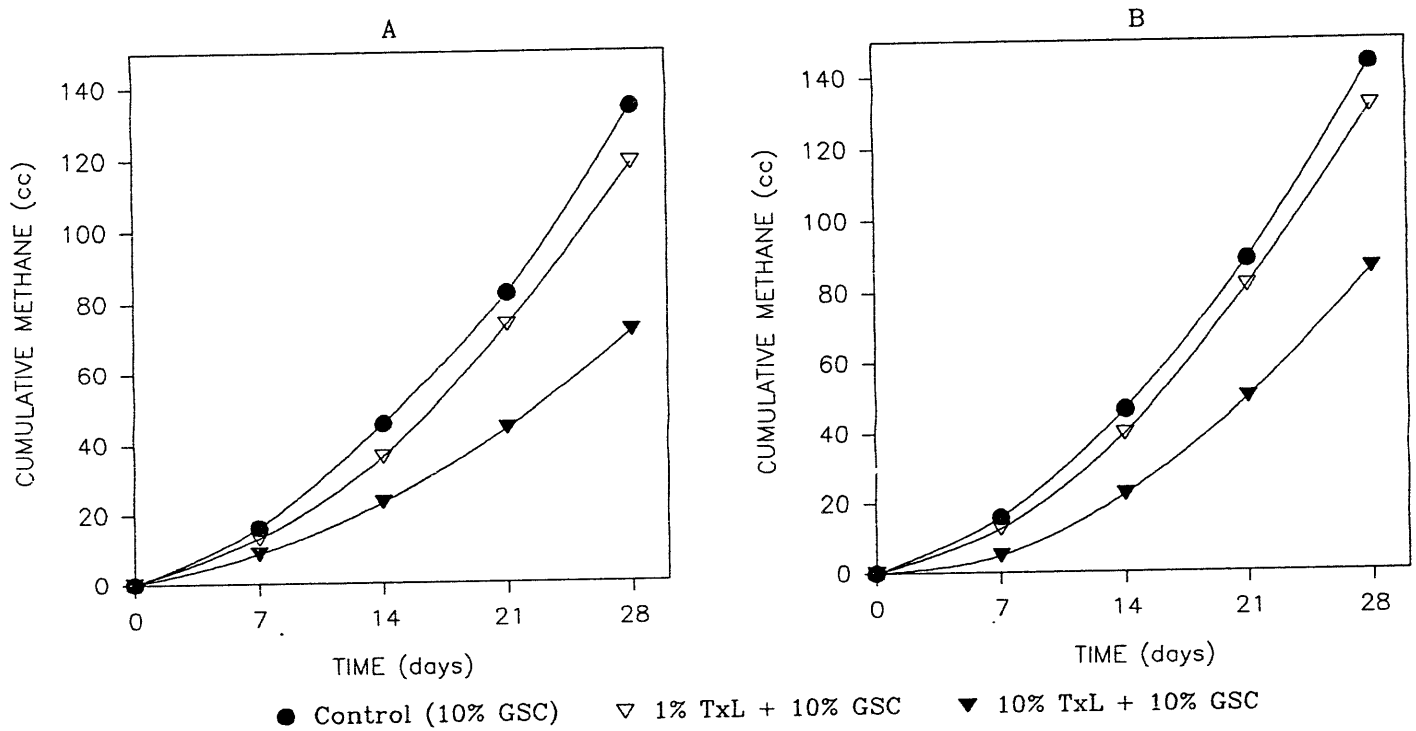

Figure 1. Influence of Different Anaerobic Conditions H_2:N_2 (A) and N_2:CO_2 (B) on Cumulative Methane Production by Granulated Sludge Consortium (GSC).

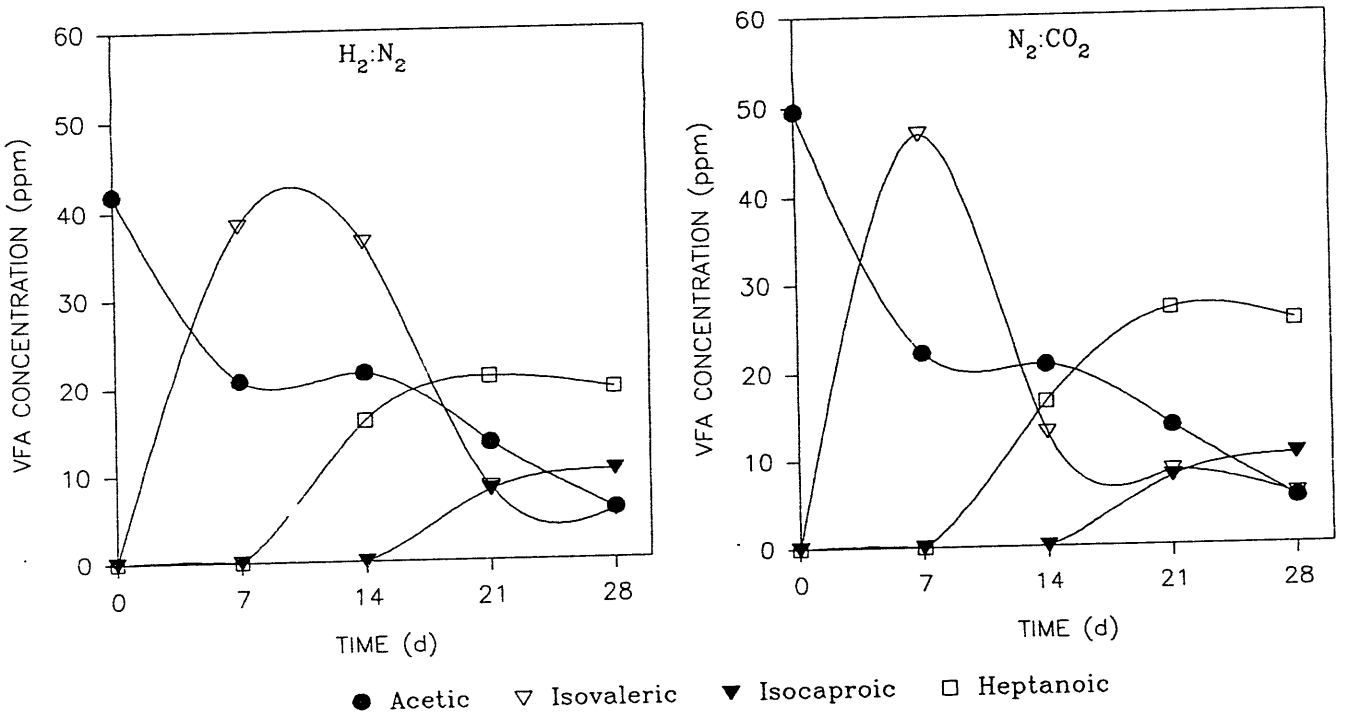

Figure 2. Time Course of VFA Concentrations During Biomethanation of 1% Texas Lignite by the 10% Granulated Sludge Consortium

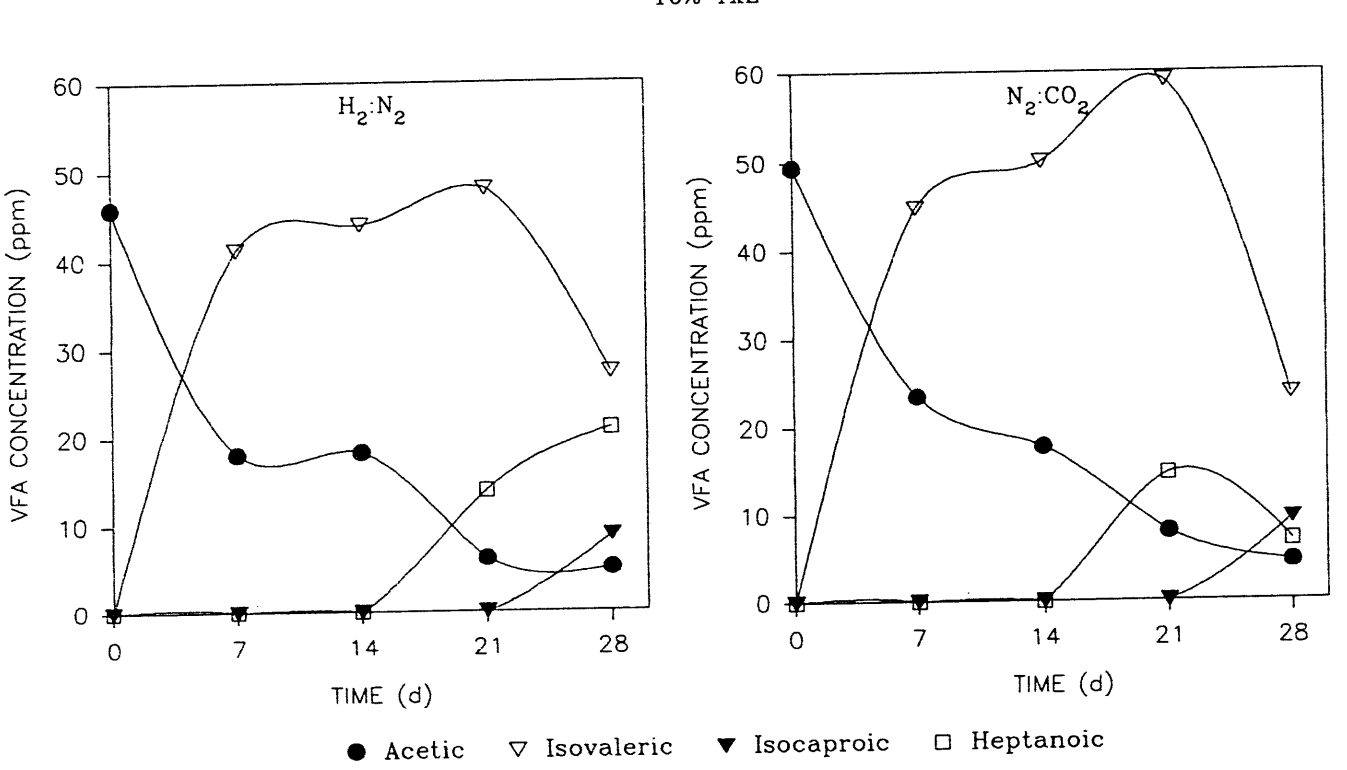

Figure 3. Time Course of VFA Concentrations During Biomethanation of 10% Texas Lignite by the 10% Granulated Sludge Consortium

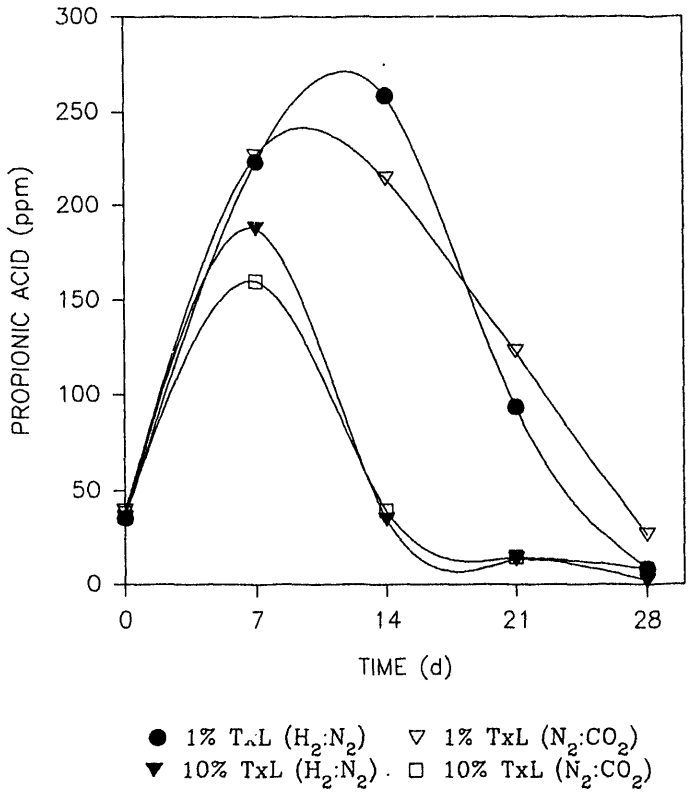

Figure 4. Time Course of Propionic Acid Concentrations During Biomethanation of 1% and 10% Texas Lignite by the 10% Granulated Sludge Consortium

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