TRANSIENT EFFECTS OF RAPID TEMPERATURE INCREASE IN THERMOPHILIC ANAEROBIC DIGESTION: BIOCHEMICAL STABILITY AND PRODUCTION OF VOLATILE SULFUR COMPOUNDS

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ABSTRACT

The City of Los Angeles, Bureau of Sanitation, has converted its digestion processes at Hyperion and Terminal Island Treatment Plants (HTP and TITP) to thermophilic operation. A two-stage continuous-batch process was established at HTP, while a single-stage sequencing batch process was established at TITP. This was to evaluate compliance with the Class A pathogen reduction requirements of U.S. EPA 40 CFR Part 503. The first part of this contribution includes full-scale data on the production of volatile sulfur compounds (VSCs), biochemical stability of the processes, and digester performance during short-term episodes of rapid changes in the digester temperature in the range of about 55 to 65 ^{0}C. The objective to increase the temperature at HTP was to comply with Alternative 1 of 40 CFR 503.32, which required a relatively high temperature because of the limited batch digester capacity (minimum temperature of 56.3 ^{0}C at 16 hours holding). TITP operations were already complying with the Alternative 1 time-temperature relation (minimum temperature of 55^{0}C at 24 hours), but the digester temperature was increased to evaluate whether this could help preventing fecal coliform recurrence in post-digestion biosolids. Rapid increases in the digester temperature at TITP (up to 65.5 ^{0}C) and HTP (up to 58 ^{0}C) caused biochemical instability, declining digester performance, and increased production of methyl mercaptan and, to a lesser extent, hydrogen sulfide. It is likely that these effects observed at full-scale were transient responses to rapid changes in temperature. The second part of this contribution includes a steady-state temperature-stress study conducted as part of the Hyperion Advanced Digestion Pilot Program. These pilot-scale studies indicated stable, steady-state operation at temperatures as high as 56 – 58 ^{0}C with minimum impact on digestion performance. A full-scale study is currently being conducted at HTP to further evaluate the effect of high temperature on the biochemical stability and production of VSCs, but under steady-state conditions with relatively small increases of the thermophilic digester temperature.

KEYWORDS

Class A biosolids; thermophilic anaerobic digestion; volatile sulfur compounds; biochemical stability; transient effects

INTRODUCTION

Many activities on thermophilic anaerobic digestion are currently driven in the U.S. by bans of Class B biosolids land application and requirements of Class A biosolids disinfection standards (U.S. EPA 40 CFR Part 503). Class A biosolids are disinfected (U.S. EPA, 1994) and can be applied to land without site restrictions by U.S. EPA 40 CFR Part 503 (U.S. EPA, 1993). Exceptional Quality (EQ) biosolids are sometimes required by stricter local regulations (e.g., Kern County, California) to continue biosolids land application at these locations.

Early studies on thermophilic anaerobic digestion of wastewater treatment sludges mostly focused on process stability (Fair and Moore, 1932; Pohland and Bloodgood, 1963; Buhr and Andrews, 1977). More recent implementations of thermophilic anaerobic digestion have focused on meeting the Class A standards for pathogen destruction (Volpe et al., 1993; Watanabe et al., 1997), including two-stage processes with a thermophilic stage (Ghosh, 1998; Huyard et al., 1998). In full-scale applications, thermophilic digester temperatures are usually in the lower end of the thermophilic temperature range (50 to 55 ^{0}C). Anaerobic digestion above 60 ^{0}C has only been investigated in lab-scale studies, focusing on process performance (Varel et al., 1980; Nozhevnikova et al., 1999), pathogen removal (Gabb et al., 2000) and microbial population dynamics (Ahring et al., 2001). Based on common practice criteria, it is usually recommended to keep the temperature below 60 ^{0}C (Ahring, 1994; van Lier, 1996).

The City of Los Angeles Bureau of Sanitation has conducted a program of producing EQ biosolids at Hyperion and Terminal Island Treatment Plants (HTP and TITP) (Iranpour et al., 2002a, b, c, d, e; 2004 a, b). A two-stage continuous-batch process has been implemented at HTP, while a single stage sequencing batch process has been implemented at TITP. This was to comply with the Class A pathogen reduction requirements of U.S. EPA 40 CFR Part 503. During thermophilic operation, the digesters were operated for short periods of time with increasing temperatures in the range of about 55 to 65 ^{0}C. As the limited batch digester capacity at HTP allowed for a maximum holding time of 16 hours in the second stage, the objective to increase the temperature was to comply with the time-temperature relation of Alternative 1 in 40 CFR 503 by raising the temperature to a minimum of 56.3 ^{0}C. The Alternative 1 time-temperature relation is met by TITP during standard operation of the single-stage sequencing batch process (24 hours holding, minimum temperature of 55^{0}C). The objective to increase the digester temperature at this plant was to prevent the recurrence of fecal coliforms in the post-digestion biosolids that was observed during standard operation.

The first part of this contribution includes full-scale results obtained during short-term episodes of rapidly increasing and decreasing digester temperatures at HTP and TITP. The second part of this contribution includes a steady-state temperature-stress study conducted as part of the Hyperion Advanced Digestion Pilot Program. The results focus on the production of volatile sulfur compounds (VSCs), biochemical stability of the processes, and digester performance.

MATERIALS AND METHODS

Plant descriptions and experimental setup

Terminal Island Treatment Plant: TITP is located in San Pedro, California, and has four egg-shaped digesters (Figure 1a) with each digester having a volume of 5,000 m^3. The digesters were fed with a mixture of primary sludge and thickened waste activated sludge (TWAS) at a total feed rate of 570 m^3/day. Digesters 1, 3, and 4 were operated in a single stage sequencing batch process in order to meet the time-temperature requirement of U.S. EPA 40 CFR Part 503 (Alternative 1) for Class A biosolids (24 hours holding, requiring a temperature of at least 55 ^{0}C). Each digester was operated on a 3-day cycle of sludge feeding, holding, and withdrawal (Figure 1b). Approximately 11% of the digester volume was withdrawn/fed in each cycle, resulting in an average hydraulic retention time (HRT) of approximately 26 days. In order to evaluate the prevention of fecal coliform recurrence in the post-digestion train, the temperature of Digester 1 was rapidly increased from 57.5 to 65.5 ^{0}C and held constant for approximately one and a half weeks. The digester temperature was then reduced to about 58 ^{0}C.

Hyperion Treatment Plant: HTP is located in Playa del Rey, California. It has three batteries with 6 (batteries D1 and D2) or 8 (battery E) egg-shaped digesters (Figure 2a). Each digester has a capacity of 9,500 m^3. The plant's feed consisted of primary sludge and TWAS at average rates of 11,300 and 3,000 m^3/day, respectively. The digesters were operated in a two-stage process. The first stage contained 16 digesters operated in a continuous mode at an average HRT of 9.9 to 10.9 days. The second stage contained 4 digesters operated in a batch mode in a 32-hour cycle of sludge feeding, holding and withdrawal (Figure 2b). The holding time was 16 hours. This would require a minimum temperature of 56.3 ^{0}C if following the time-temperature requirement of Alternative 1. Each batch digester was fed/withdrawn up to 60 to 70% of its total capacity during each cycle. In order to operate the batch digesters at a holding time of 16 hours, temperatures of continuous and batch stages were rapidly increased to about 58 ^{0}C and more than 56.3 ^{0}C, respectively. Then, the temperatures of both stages were reduced to about 53 ^{0}C.

HTP Advanced Digestion Pilot Plant Set-up: A long-term pilot-scale temperature stress study was conducted at the Hyperion Advanced Digestion Pilot Plant Facility. The pilot-plant has three 5.3 m^3 digesters and four 2.65 m^3 mixing tanks (Figure 3). One digester was dedicated for the temperature stress study. The feedstock to this digester was a blend of 70% primary sludge and 30% TWAS by dry weight. The HRT was approximately 10 days.

Sampling and analytical procedures

Sampling: Digester gas samples were collected in Tedlar bags and digester biosolids samples were collected in 1-liter plastic bottles. At TITP, digester biosolids samples were obtained from sampling ports in the discharge of Digester 1. Digester gas samples were obtained from a dedicated sampling port for Digester 1. At HTP, biosolids samples were taken from the individual first-stage digesters for analysis of volatile fatty acids (VFA) and total alkalinity. Total solids and volatile solids destruction (TSD and VSD, respectively) were determined in the combined outflow from the first stage. Digester gas samples were collected from the combined outflow of the first and second stage. Sampling from the dedicated digester of the Hyperion Advanced Digestion Pilot Program was identical to sampling from full-scale operations.

Analytical: The Environmental Monitoring Division at HTP and TITP performed all analyses according to procedures summarized in Table 1.

Microbial analysis: The pilot-scale and full-scale tests described herein achieved disinfection that in general met the Class A limits for indicators and pathogens. Those results are not presented as they have previously been reported (Haug et al., 2002; Hernandez et al., 2002; Iranpour et al., 2002d, e; 2004a, b).

RESULTS

Temperature profiles

Terminal Island Treatment Plant: From October 20 to November 7, 2002, the average temperature of Digester 1 was rapidly increased to 65.5 ^{0}C and kept constant for about one and a half weeks (Figure 4a). The digester temperature was then decreased to about 58 ^{0}C.

Hyperion Treatment Plant: Stability parameters, process performance, and VSC production at HTP were correlated with the average first-stage digester temperature, because most of the digestion process occurred in the first-stage digesters (HRT of about 10 days) rather than in the second stage (holding time of 16 hours). In the last week of September 2002, the average temperature of the first-stage digesters was rapidly increased from 54.4 ^{0}C to around 58 ^{0}C and held constant for a period of three weeks in October 2002 (Figure 4b). In November 2002, the average first-stage digester temperature was reduced to about 53 ^{0}C.

HTP Advanced Digestion Pilot Program: The temperature was increased from 56.6 ^{0}C to approximately 61 ^{0}C in two steps of approximately 2.2 ^{0}C in August and September 2001 (Figure 4c). The performance was monitored for several months, after which the temperature was further increased to 65 ^{0}C (January – April 2002). After each increase of temperature, the digester stayed at the new temperature for several weeks in order to attain steady-state operation. After April 2002, the digester was operated at a temperature in general between 55 – 56 ^{0}C.

TITP full-scale operations

Digester 1 was operated at 57 ^{0}C for several months in a biochemically stable condition before increasing the temperature. No observable changes in digester performance were observed when the temperature was raised to 60 ^{0}C and held constant for one week. Changes in VSC production and digester performance were observed, however, when the temperature was further increased to 65.5 ^{0}C.

Production of VSCs. Hydrogen sulfide and methyl mercaptan were identified as the most important VSCs in the digester gas. Carbonyl sulfide, ethyl mercaptan, 2-propyl mercaptan, and 1-propyl mercaptan were not detected at TITP or at HTP. The concentrations of hydrogen sulfide (450 ppm_v) and methyl mercaptan (500 ppm_v) in digester gas were the highest at a digester temperature of 65.5 ^{0}C (Figure 5). Reduction of the digester temperature to 60 ^{0}C caused lower concentrations of both compounds. The concentration of dimethyl sulfide was lower than those of hydrogen sulfide and methyl mercaptan and was not significantly affected by the digester temperature.

Biochemical stability: The VFA concentration in digester biosolids samples peaked at a maximum of approximately 1,000 mg/l shortly after the digester temperature increase to 65.5 ^{0}C (Figure 6a). VFA concentrations before the temperature increase (October 2002) were in the range of 63 – 252 mg/l with an average of 154 mg/l. The total alkalinity was not significantly affected by the digester temperature and remained relatively constant in the range of 1,650 to 1,850 mg/l (results not shown). Consequently, the trend of the VFA to total alkalinity ratio versus the digester temperature, shown in Figure 6b, was similar to the one observed for the VFA concentration. The VFA to total alkalinity ratio increased from 0.1 to a maximum of 0.55 shortly after increasing the digester temperature to 65.5 ^{0}C.

Digester performance: The methane concentration in digester gas decreased to 42% at a digester temperature of 65.5 ^{0}C, however, it returned to a normal concentration of about 60% when the digester temperature was reduced to 58-60 ^{0}C (Figure 7). VSD also decreased at higher digester temperatures. VSD was 56% at 60 ^{0}C but decreased to 51% at 65.5 ^{0}C. Higher digester temperatures did not affect the ammonia concentration or the pH of digester biosolids, which were constant during the entire period of testing in the ranges of 557 to 647 mg/l and of 6.9 to 7.1, respectively.

HTP full-scale operations

First-stage continuous digesters were biochemically stable at an average temperature of approximately 54.4 ^{0}C during August and the first part of September 2002. Odor complaints from residential areas close to HTP rose in frequency, however, while increasing the temperature to 58 ^{0}C in October 2002.

Production of VSCs. A large increase of the methyl mercaptan concentration (up to 375 ppm_v) and an increase of the dimethyl sulfide concentration (up to 40 $ppm_{v)}$ were observed at a temperature of 58 ^{0}C (Figure 8). Both concentrations sharply dropped when the temperature of the digesters was reduced to approximately 53 ^{0}C. The hydrogen sulfide concentration in the digester gas was relatively constant in the range of 100 – 200 ppm_v at digester temperatures of 54.4 – 58 ^{0}C. The composition shown in Figure 8 is that of digester gas in the combined outflow from the first and second-stage digesters.

Biochemical stability: A close correlation but with a delayed response of about one week was observed between the average VFA concentration and the temperature in first-stage digesters (Figure 9a). When the temperature was increased from 54.4 to 58 ^{0}C, the VFA concentration rose from approximately 400 mg/l to over 1,000 mg/l. When the digester temperature was lowered, the VFA concentration declined as well. The total alkalinity ranged from 3,000 mg/l to 3,700 mg/l and was not significantly influenced by the digester temperature (results not shown). The VFA to total alkalinity ratio increased to a maximum of about 0.33 at a digester temperature of 58 ^{0}C, but was smaller at lower digester temperatures (Figure 9b).

Digester performance: Methane and carbon dioxide concentrations in the digester gas (Figure 10), VSD (Figure 11), and the ammonia concentration (data not shown) remained about the same during the entire testing period. Although the pH increased from approximately 7.2 to 7.7, there was no apparent correlation with the digester temperature.

HTP Advanced Digestion Pilot Study

Production of VSCs: VSCs in digester gas were not measured.

Biochemical stability: A close correlation between the VFA concentration in digester biosolids and the digester temperature was observed (Figure 12a). The maximum VFA concentration was approximately 3,000 mg/L at 65 ^{0}C. When the digester temperature was decreased to 55-56 ^{0}C, the VFA concentration dropped to 500 – 1,000 mg/L. Similarly, a close correlation between total alkalinity and the digester temperature was observed (Figure 12a). The total alkalinity declined to approximately 1,600 mg/l at 65 ^{0}C and rose to more than 2,500 mg/l at 55-56 ^{0}C. The VFA to total alkalinity ratio consequently rose to approximately 1.5 and then declined to approximately 0.4 in the same temperature range (Figure 12 b).

Digester performance: VSD was approximately 50% at digester temperatures of 55 – 56 ^{0}C but it decreased at higher temperatures (Figure 13). Increasing the digester temperature also caused a decrease in the amount of gas produced (Figure 14a), while the methane concentration in digester gas decreased (Figure 14b).

DISCUSSION

Transient and steady-state operations

The short-term episodes of higher temperatures during full-scale digester operations at TITP and HTP were attained by a rapid increase of the temperature. As a result, the processes became biochemically unstable and probably there was not sufficient time for the microbial populations to adapt to the higher temperatures. Therefore, the observed impacts during the full-scale operations may have been due to a transient response to the higher digester temperatures. In contrast, the HTP Advanced Digestion Pilot study was conducted with small increments of the digester temperature over a long period of time, which would have allowed stabilization of the digester. Consequently, the pilot-scale results probably reflected a steady-state performance and the observed impacts were probably mainly due to the higher digester temperatures tested.

Odor compounds

This study provides a quantitative evaluation of VSC production during transient and rapid increases of the temperature of full-scale thermophilic anaerobic digesters. The results indicate that methyl mercaptan concentrations in digester gas increased during operation at HTP and TITP with increasing digester temperatures. Hydrogen sulfide concentrations also increased at TITP but not at HTP. Possible causes for this apparent discrepancy are:

a) The maximum digester temperature at HTP was significantly lower than at TITP.
b) HTP and TITP add $FeCl_2$ to the digesters to reduce the emission of hydrogen sulfide by precipitation. The hydrogen sulfide concentration in digester gas therefore does not reflect the actual production, which may be plant specific.

Hydrogen sulfide and methyl mercaptan are compounds with a low odor threshold (in the lower ppb range) and, therefore, are important components in odor emissions from thermophilic operations. A small increase of the dimethyl sulfide concentration was also observed during operation with increasing digester temperatures. VFA production could be another factor contributing the observed increase in odor emissions at HTP. Although VFA were only analyzed in biosolids, it is likely that the elevated production of VFA by increasing temperatures and the volatilization of VFA caused higher concentrations in air emissions.

Biochemical stability and digester performance

The increase in the VFA to total alkalinity ratio during the TITP and HTP full-scale operations indicated that the rapid and short-term increase in temperature caused biochemical instability of the digesters. However, this instability was reflected only in a small decrease in VS destruction during full-scale operations at TITP and no change in digester performance was observed during full-scale operations at HTP. This discrepancy may have been a result of the short period of operation of the TITP and HTP digesters at higher temperatures, which perhaps prevented a larger deterioration of the digester performance. Additional studies are currently being conducted at HTP using a dedicated first-stage digester in order to study the long-term effect of high temperature on performance and biochemical stability, as well as on VSC production under steady-state operation. The temperature of this digester is increased by approximately 0.5 ^{0}C (1 ^{0}F) per month from 53 to 58 ^{0}C allowing the digester to reach steady-state conditions at each temperature.

Biochemical instability and low performance occurred at temperatures of 61 ^{0}C and higher during the study conducted at the HTP Advanced Digestion Pilot facility. This is consistent with recent laboratory studies on the anaerobic digestion of manures that have also demonstrated biochemical instability and poor digester performance at temperatures over 60 ^{0}C (Ahring et al., 2001; Gabb et al., 2000; Varel et al., 1980).

Microbiological issues

Recent investigations suggested a complex mechanism of the formation and degradation of VSCs by microbial populations in anaerobic digesters, in which methanogens are suspected to catalyze the demethylation of VSCs (Higgins et al., 2003). Thus, it may be postulated that the elevated VSC production at increasing digester temperatures is a consequence of biochemical instability and an imbalance between VSC production and degradation processes. A reduction of the methanogenic activity during episodes of increasing temperature could potentially cause two effects:

1. Less production of methane from VFA, thereby causing accumulation of VFA in the digester.

2. Reduced demethylation of VSCs, thereby causing accumulation of methyl mercaptan and dimethyl sulfide in digester gas.

The observation that stable digester performance was observed soon after reducing the digester temperature may indicate that the microbial imbalances observed during the full-scale operations were reversible. Understanding the mechanism of the biochemical instability and production of VSCs may provide a basis for developing strategies to reduce or control these problems during thermophilic operations. Therefore, additional studies to define the effect of the temperature on the activities, diversity and densities of microbial communities in full-scale digesters (e.g. acetotrophic and hydrogenotrophic methanogens, sulfate-reducing bacteria and others) are needed.

VSCs as indicators of toxicity in anaerobic digestion processes

Several authors have proposed the use of VSCs as indicators of toxicity in anaerobic processes (Speece, 1996; Yerkes et al., 2002). Yerkes et al. (2002) detected elevated concentrations of methyl mercaptan and dimethyl sulfide when toxic compounds such as chloroform or tetrachloro ethylene were added to an anaerobic sludge digester. Since methanogenic bacteria seem to play an important role in the biodegradation of methylated sulfur compounds, an increase in the concentration of these compounds can be expected when the methanogenic activity is inhibited by the presence of toxic compounds or other stress factors. The present results of full-scale operation at HTP and TITP provide additional support to this idea, because elevated concentrations of methyl mercaptan and dimethyl sulfide may perhaps be a result of a microbial imbalance caused by rapidly increasing the digester temperature.

CONCLUSIONS

1. In general, a rapid and short-term increase of the temperature in full-scale operations to 65.5^{0}C at TITP and 58 ^{0}C at HTP was associated with biochemical instability, increased VSC production and a minor decline of digester performance. These effects were probably transient responses to rapid temperature changes.
2. Increasing the digester temperature caused transient increases of the production of methyl mercaptan (TITP and HTP) and hydrogen sulfide (TITP), while production of other sulfides only slightly increased or was not detected.
3. The increase of odor complaints at HTP during a short-term episode of relatively high temperature may be associated with elevated production of methyl mercaptan.
4. Increasing the digester temperature generally caused an increase in VFA production in full-scale digesters at HTP and TITP and the pilot-scale digester at the HTP Advance Digestion Pilot facility.
5. A minor declining of the digester performance was observed in full scale operations at TITP. No decline in performance was observed in full-scale operations at HTP.
6. The maximum temperature for steady-state operation of pilot-scale digesters without an effect on performance was approximately 56 – 58 ^{0}C.

A full-scale study with one digester at HTP is currently being conducted to evaluate the production of volatile sulfur compounds with small increases in temperature. This is to further investigate the biochemical stability under conditions that allow the microbial populations to acclimate to higher temperatures.

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Figure 1a. TITP- Schematic of single stage sequencing batch thermophilic reactors

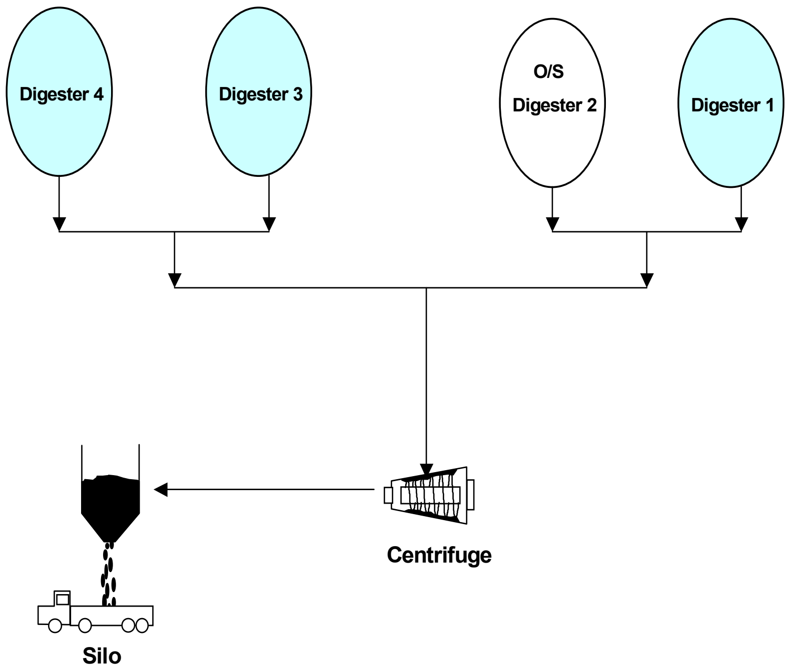


Figure 1b. TITP-Digester cycles for batch operation

Day	Day 1	Day 2	Day 3
Hours 0	24	48	72
Digester 1	F	H	W
Digester 3	W	F	H
Digester 4	H	W	F
F = feed, H = hold, W = withdraw			

Figure 2a. HTP- Schematic of two-stage continuous/batch thermophilic digestion

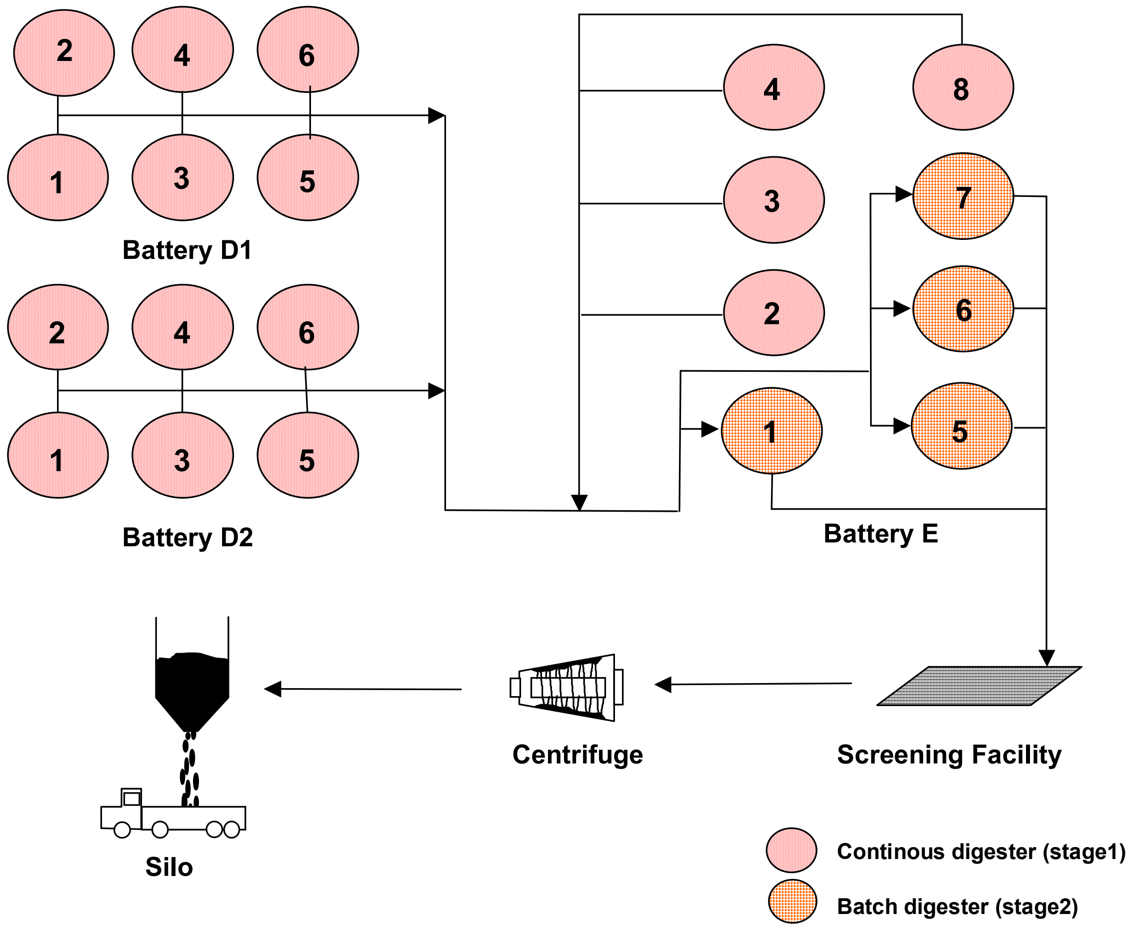
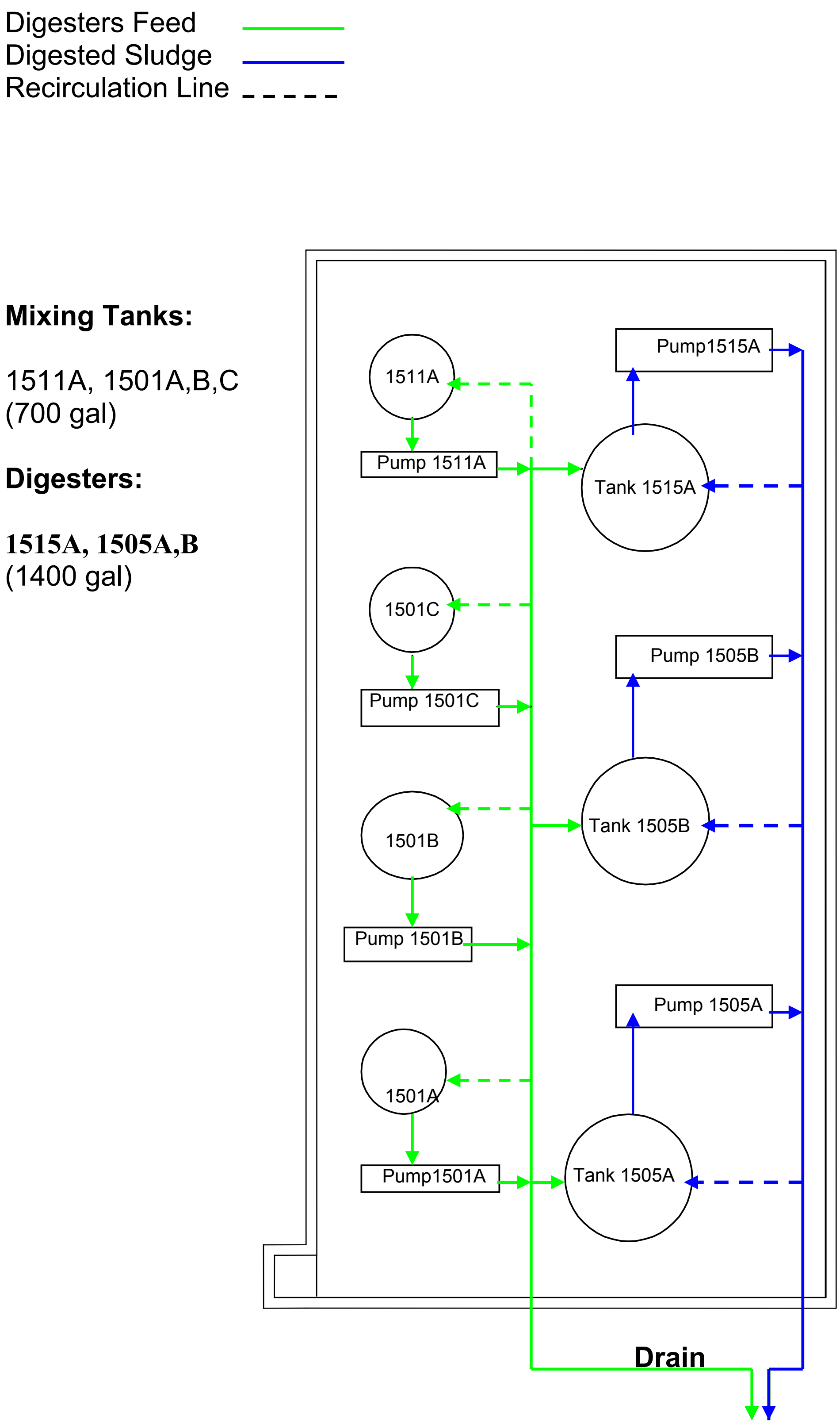


Figure 2b. HTP- Digester cycles for batch operation

Digester	Sequence #1	Sequence #2	Sequence #3	Sequence #4
Hours 0	8	16	24	32
1E	F	H	H	W
5E	W	F	H	H
6E	H	W	F	H
7E	H	H	W	F
F = feed, H = hold, W = withdraw				

Figure 3. HTP-Schematic Advanced Digestion Pilot Plant



Mixing Tanks:

1511A, 1501A,B,C
(700 gal)

Digesters:

1515A, 1505A,B
(1400 gal)

Figure 4. Digester temperature profiles: a) TITP – Digester temperature (2002); b) HTP – First stage digesters average temperature (2002); c) HTP – Pilot Digester Temperature (2001-2003).

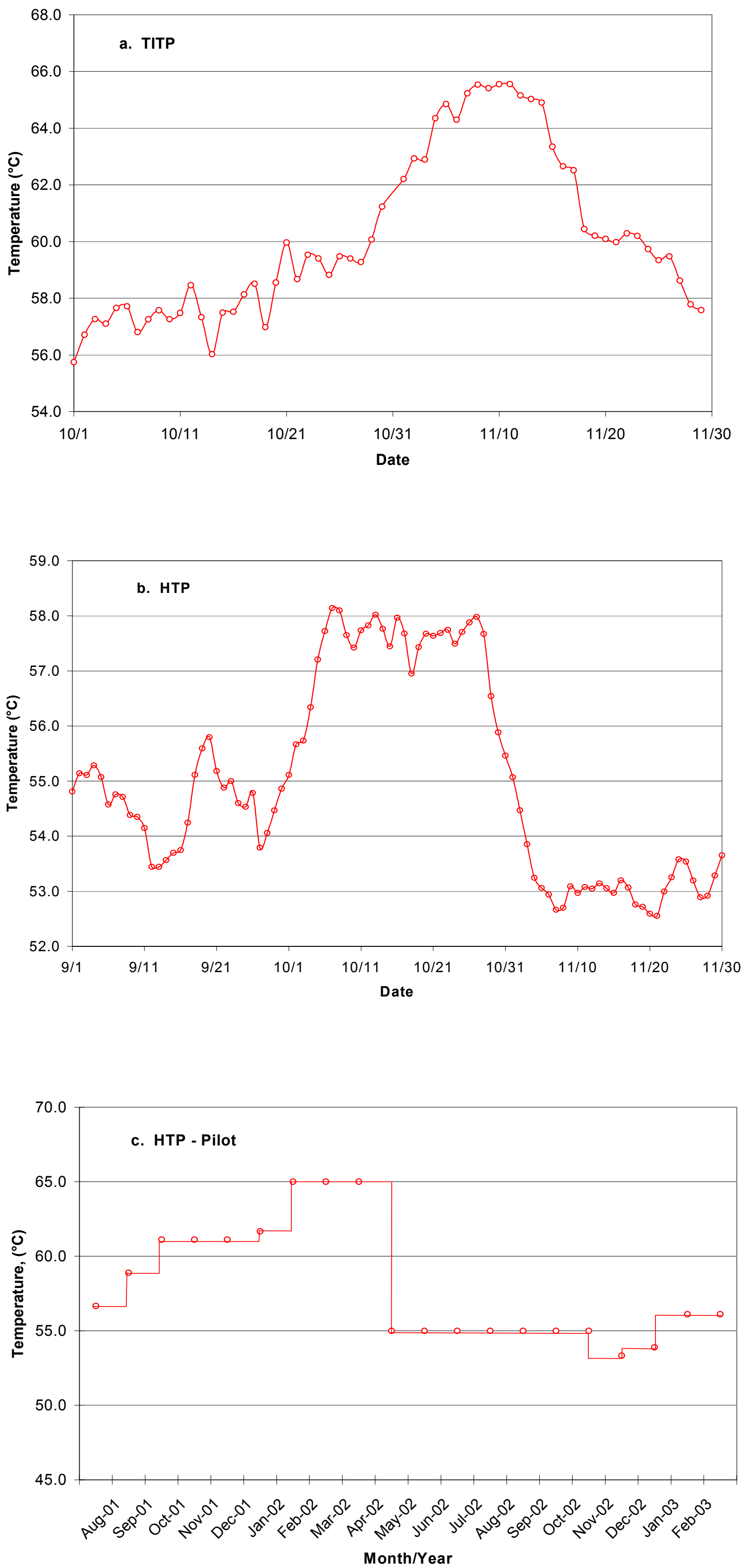


Figure 5. TITP - Volatile sulfur compounds and temperature

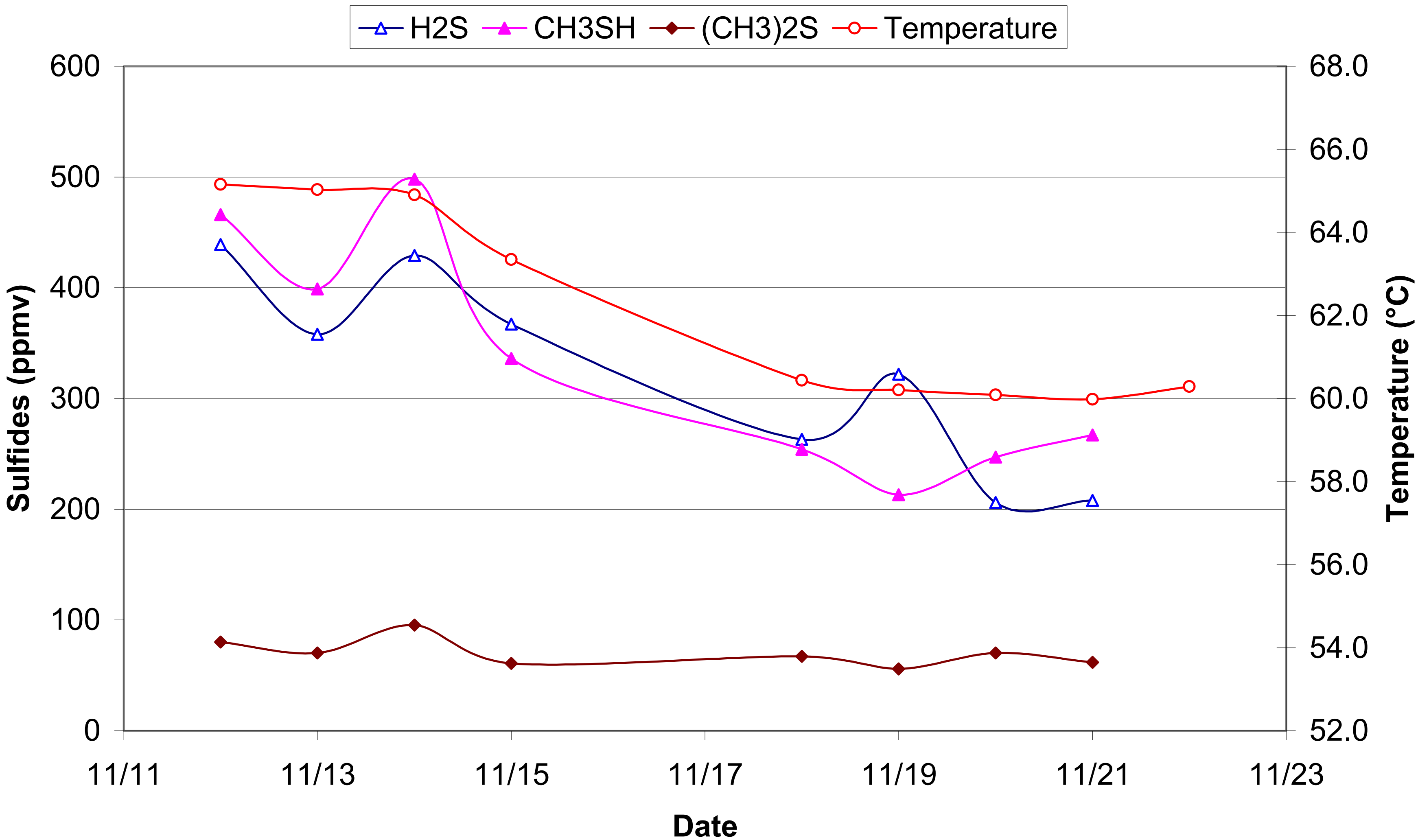
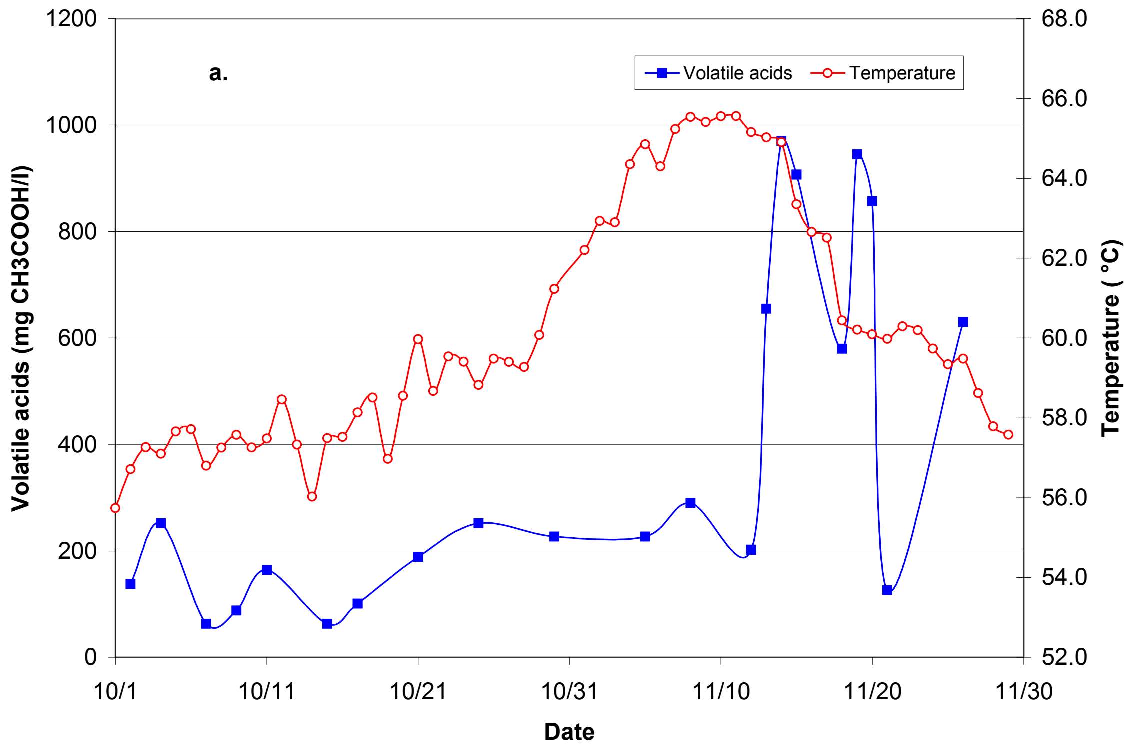


**Figure 6. TITP – a) VFA in digester sludge versus digester temperature;
b) VFA to total alkalinity ratio versus digester temperature**



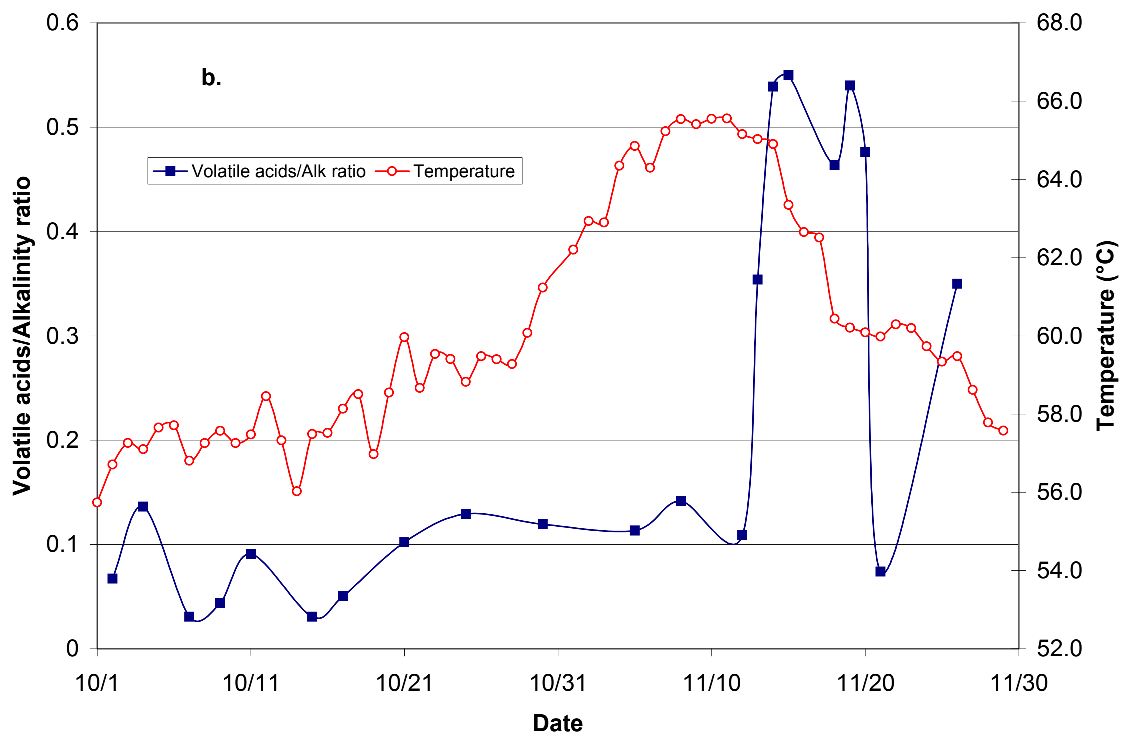


Figure 7. TITP - Methane in digester gas versus digester temperature

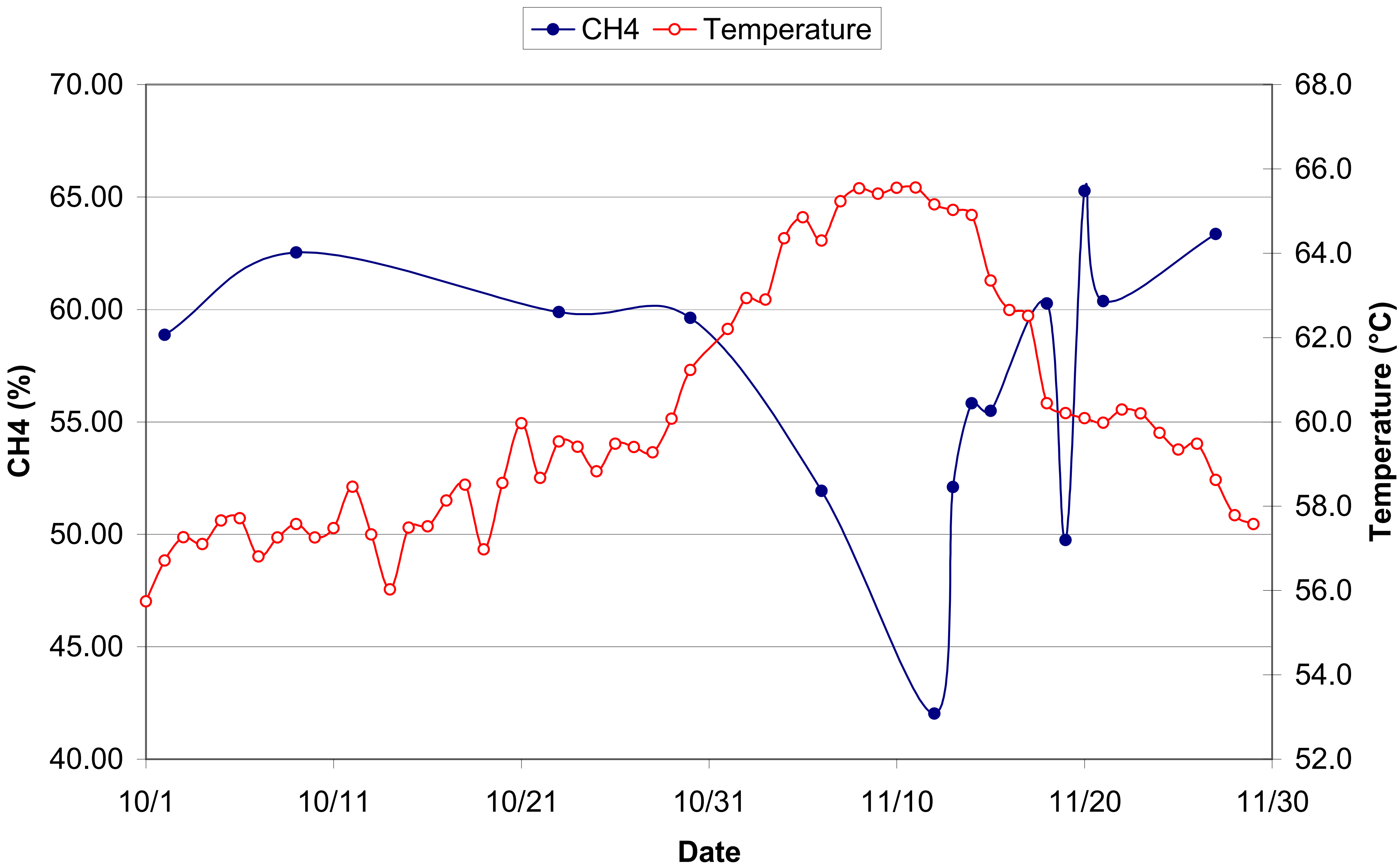


Figure 8. HTP - Volatile sulfur compounds and temperature

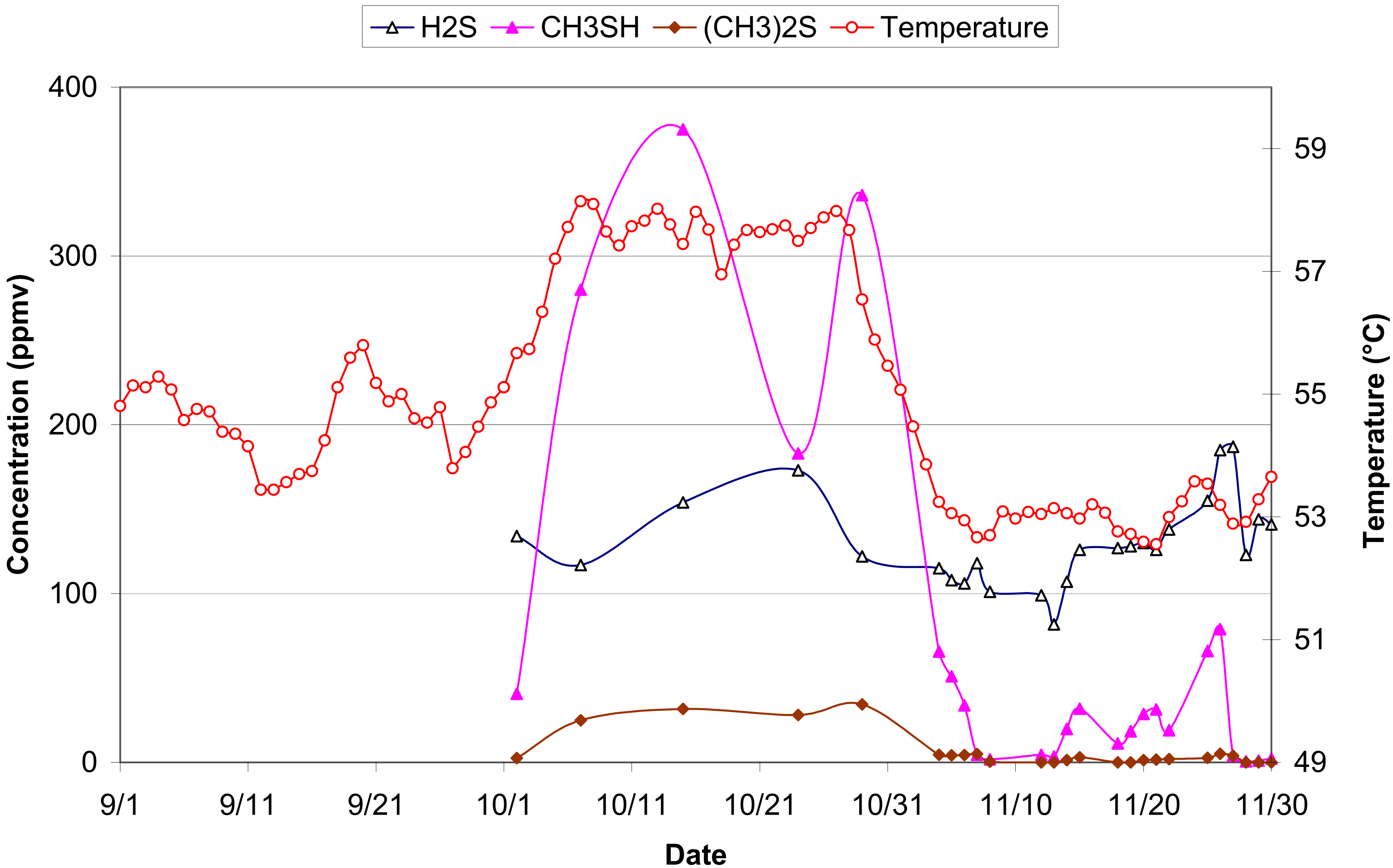
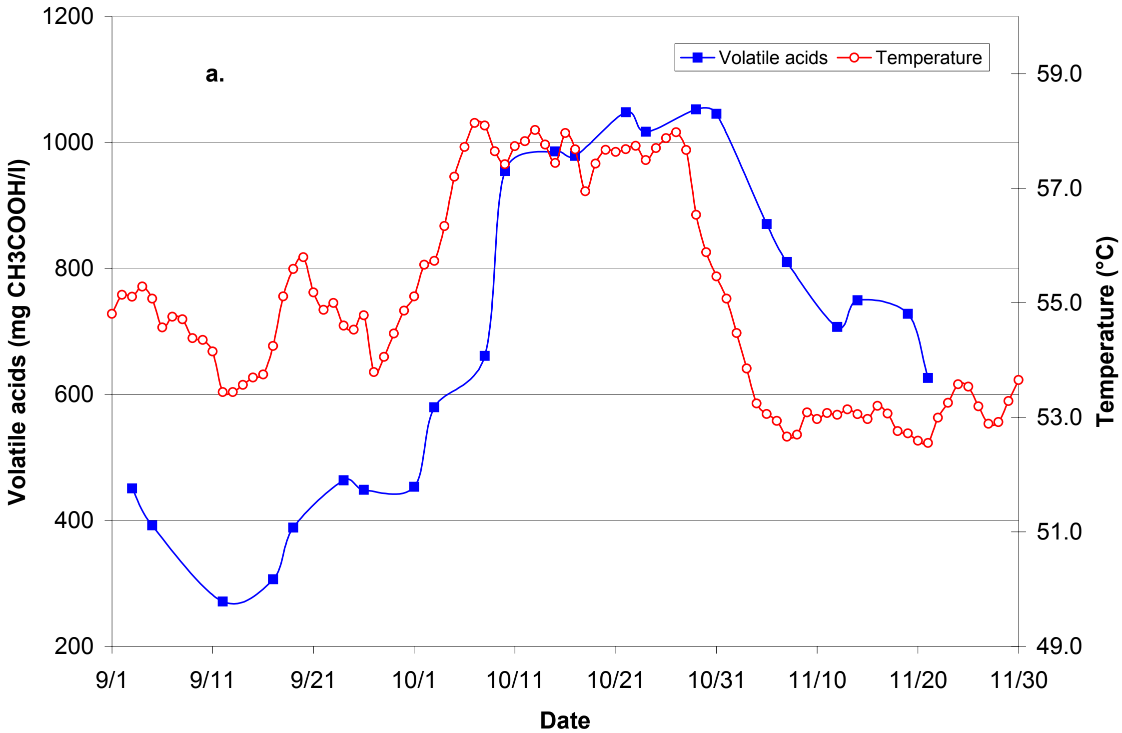


Figure 9. HTP – a) VFA in digester sludge versus digester temperature; b) VFA to total alkalinity ratio versus digester temperature



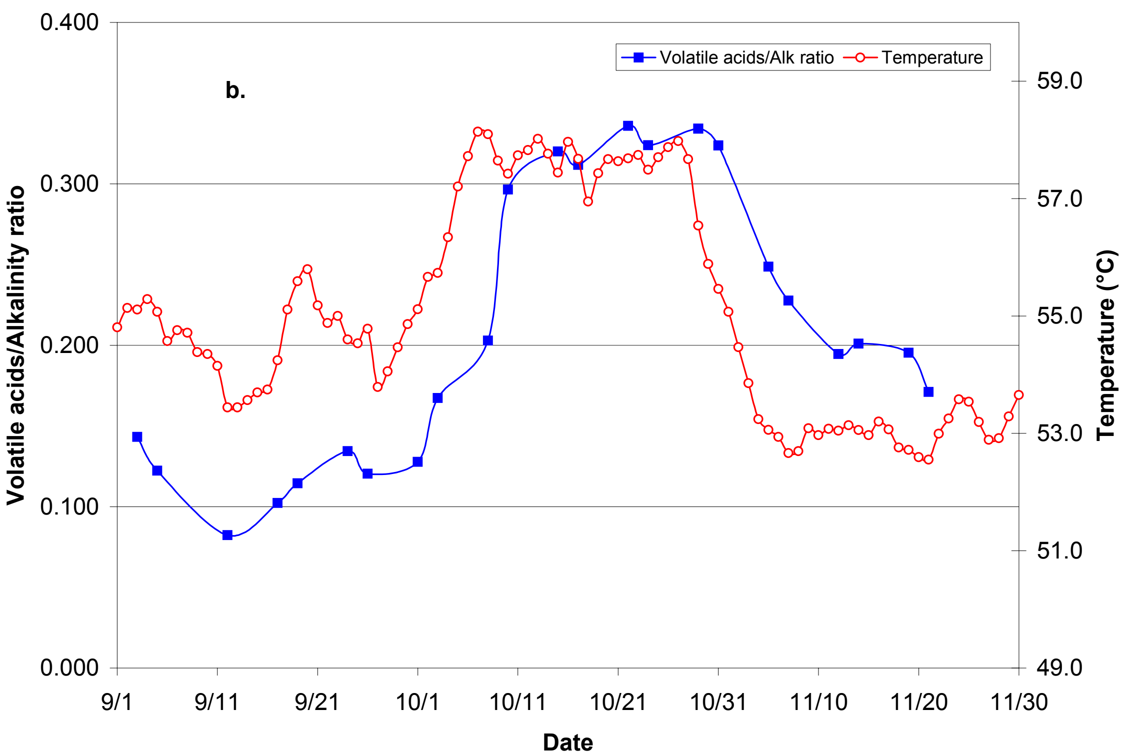


Figure 10. HTP-Composition of digester gas versus digester temperature

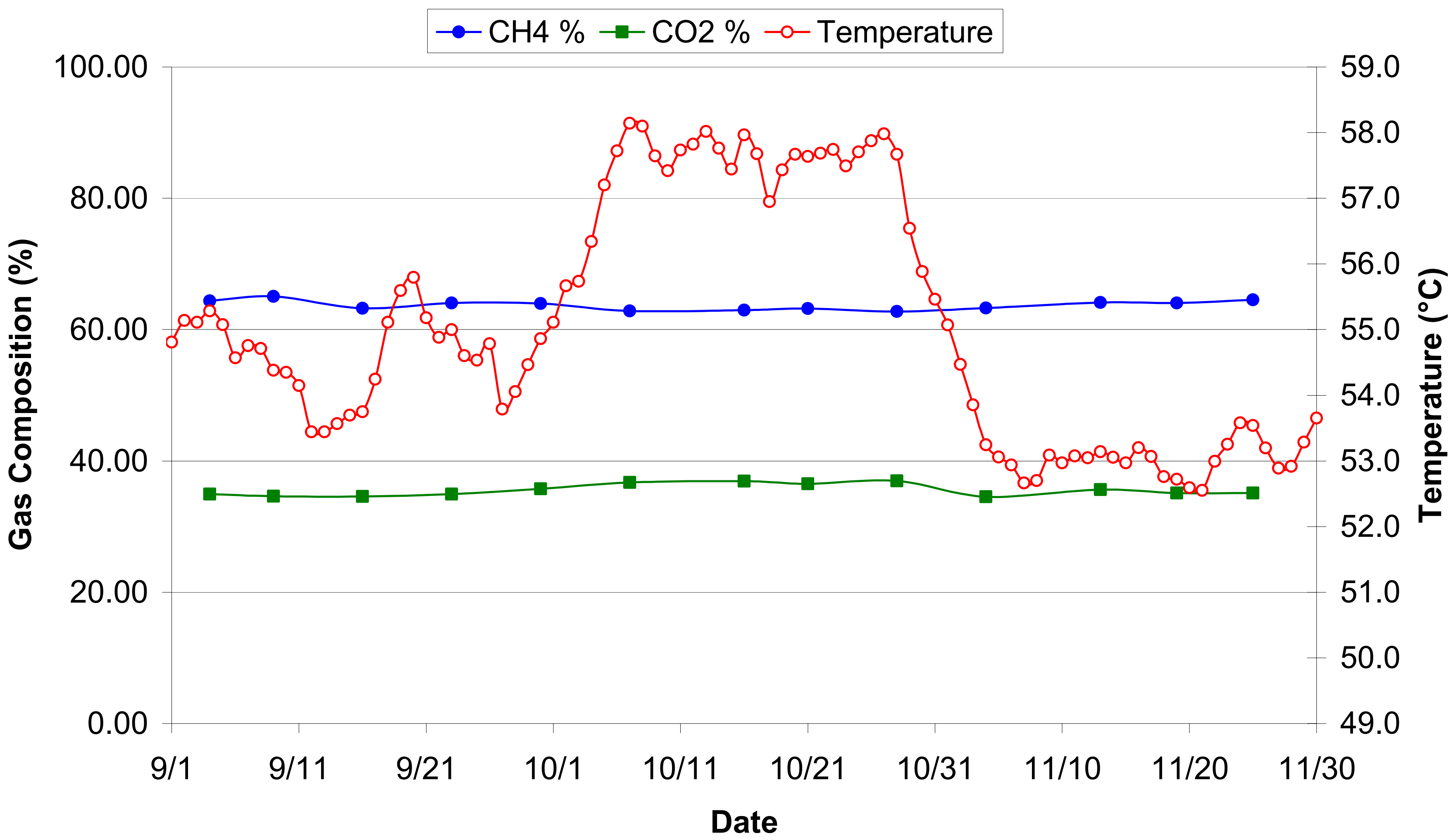


Figure 11. HTP-Volatile solids destruction versus digester temperature

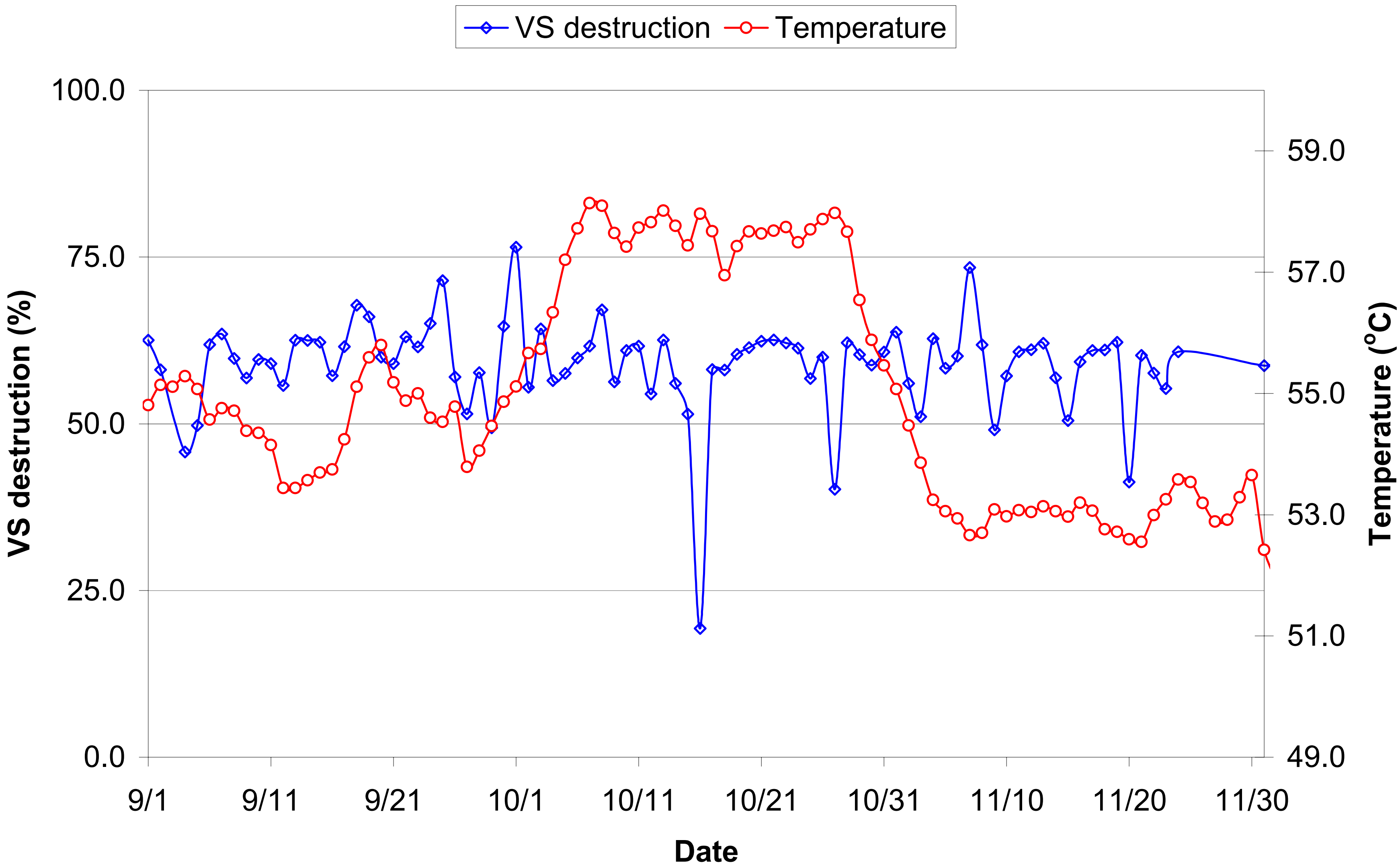
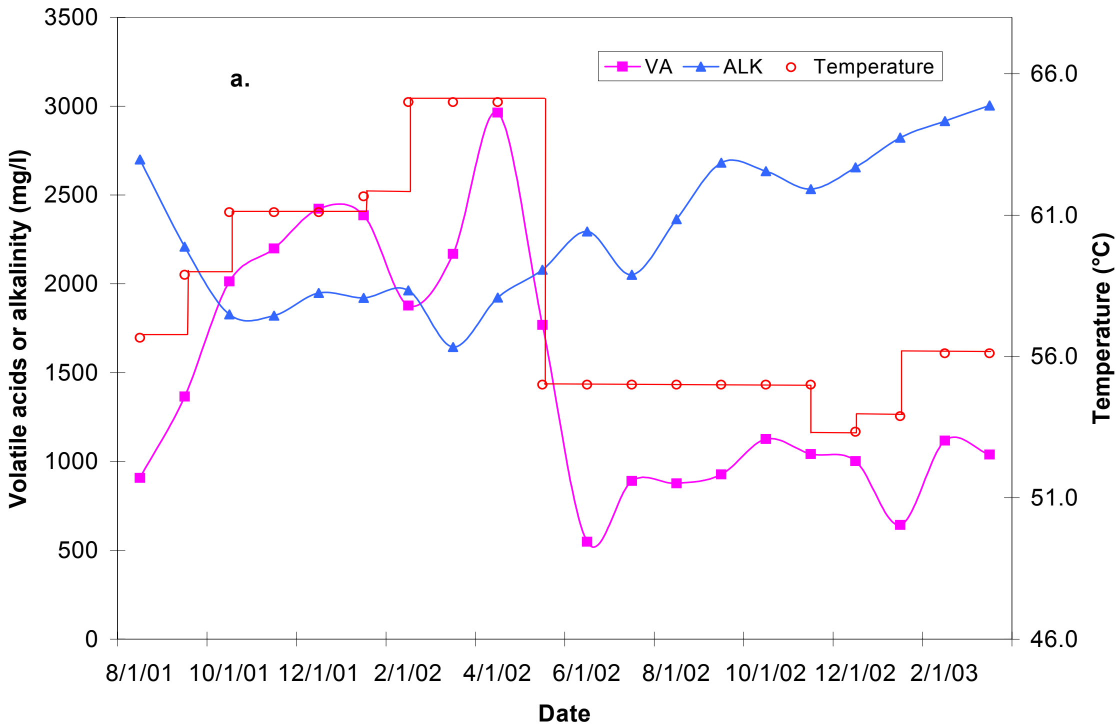


Figure 12. HTP Pilot – a) Volatile acid and alkalinity versus digester temperature; b) Volatile acid to alkalinity ratio versus digester temperature



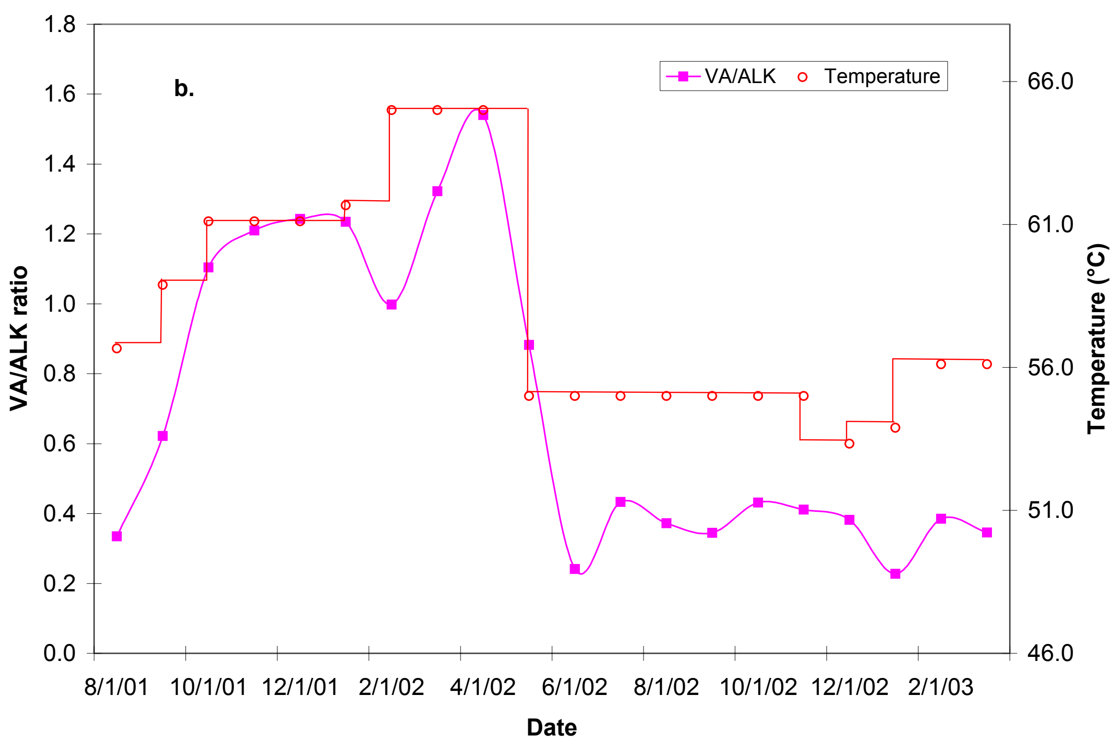


Figure 13. HTP Pilot - Volatile solid destruction versus digester temperature

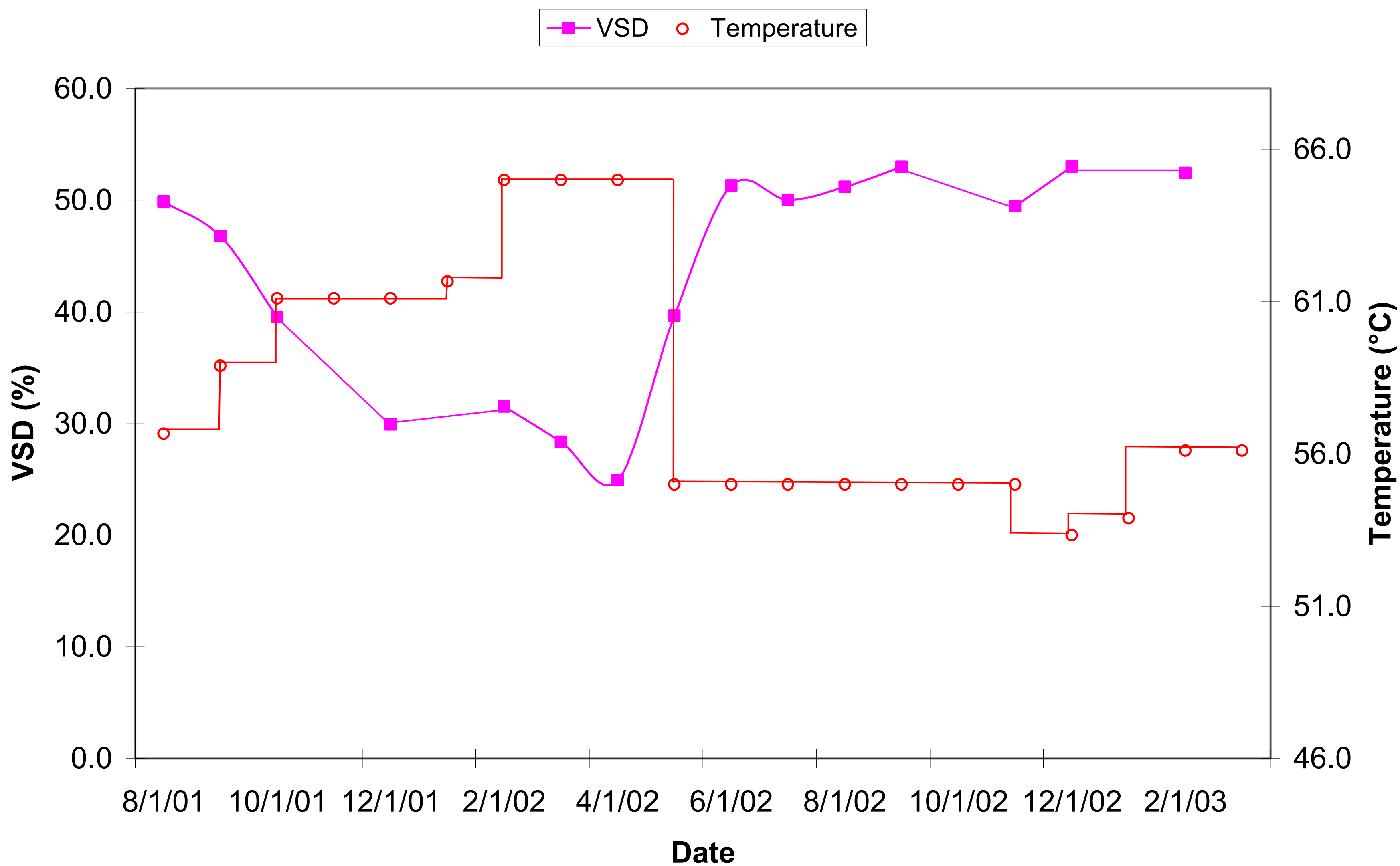
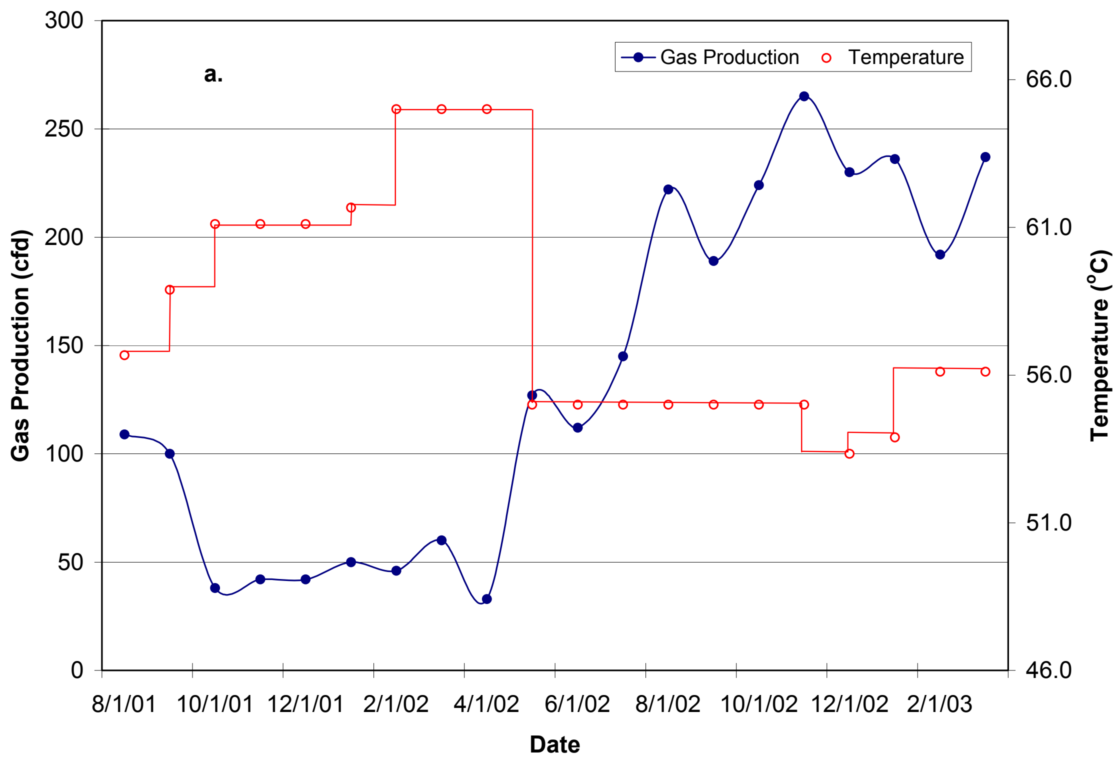


Figure 14. HTP Pilot – a) Gas production versus digester temperature; b) Methane in digester gas versus digester temperature



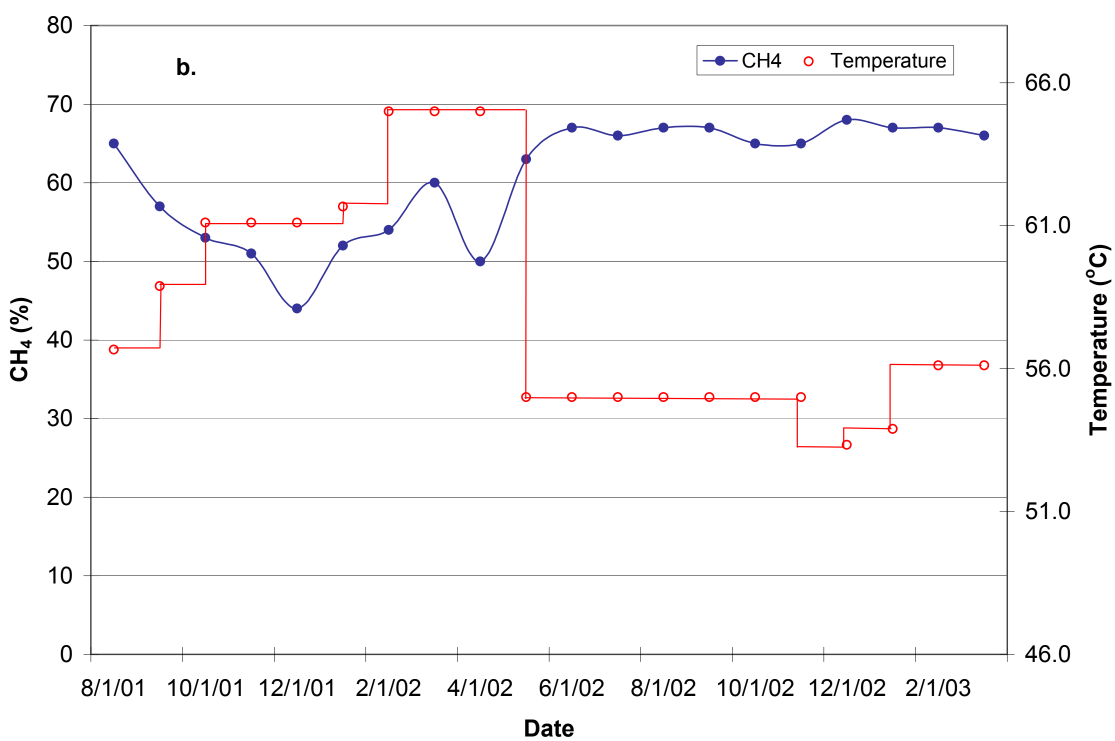


Table 1. Laboratory procedures

Parameter	Method
CH_4	U.S. EPA Method 18[1]
CO_2	U.S. EPA Method 18[1]
Alkalinity	Titration, SM 2320 B[2]
Volatile Fatty Acids	Distillation and titration, SM 5560 C[2]
Total solids	Gravimetric, 1003-105 C, SM 2540 B[2]
Volatile solids	Gravimetric, 550 C, SM 2540 E[2]
pH	Electrometric, SM 4500-H^+ B[2]
CH_3SH / $(CH_3)_2S$	SCAQMD Method 307-91[3]
H_2S	Colorimetric tube[4]

[1]U.S. EPA (1992)
[2]APHA et. al. (1992)
[3] SCAQMD (1998)
[4] SCAQMD approved Drager tube as alternate method to SCAQMD Method 307-91. No method number was assigned.