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Conversion of Municipal Solid Waste Into Carboxylic Acids by Anaerobic Countercurrent Fermentation

Effect of Using Intermediate Lime Treatment

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Abstract

Municipal solid waste (MSW) and sewage sludge (SS) were combined and anaerobically converted into carboxylate salts by using a mixed culture of acid-forming microorganisms. MSW is an energy source and SS is a source of nutrients. In this study, MSW and SS were combined, so they complemented each other. Four fermentors were arranged in series for a countercurrent fermentation process. In this process, the solids and liquid were transferred in opposite directions, with the addition of fresh biomass to fermentor 1 and fresh liquid media to fermentor 4. An intermediate lime treatment of solids exiting fermentor 3 before entering fermentor 4 was applied to improve the product acid concentration from the untreated MSW/SS fermentations. All fermentations were performed under anaerobic conditions at 40°C. Calcium carbonate was added to neutralize the carboxylic acids and to control the pH. Iodoform was used as a methanogen inhibitor. Carboxylic acid concentration and gas composition were determined by gas chromatography. Substrate conversion was measured by volatile solids loss, and carboxylic acid productivity was calculated as the function of the total carboxylic acids produced, the amount of liquid in all fermentors, and time. The addition of intermediate lime treatment increased product concentration and conversion by approx 30 and 15%, respectively. The highest carboxylic acid

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concentrations for untreated MSW/SS fermentations with and without intermediate lime treatment were 22.2 and 17.7 g of carboxylic acid/L of liquid, respectively. These results confirm that adding a treatment step between fermentor 3 and fermentor 4 will increase the digestibility and acid productivity of the fermentation.

Index Entries: MixAlco process; municipal solid waste; sewage sludge; fermentation; carboxylic acids.

Introduction

In recent years, oil prices have been rising, generating renewed interest in biomass energy. In addition, Middle East conflicts have raised concerns about the reliability of oil. Wastes such as industrial byproducts, agricultural and forestry residues, animal manure, municipal biosolids, and municipal solid wastes (MSW) represent a major environmental problem for all countries. Accumulations of wastes, limited disposal alternatives, and increasing environmental regulations have become a serious problem, especially in areas with high population densities. Using waste biomass reduces environmental impacts from waste disposal and air pollution and provides a source of liquid fuels that does not result in increased CO_2 in the atmosphere *(1,2)*. Waste biomass represents an ideal feedstock for conversion into chemicals and fuels and can be utilized to reduce both the amount of fossil fuels burned and the emission of greenhouse gases.

MSW and sewage sludge (SS) are potential substrates for conversion to chemicals and fuels. For the purpose of the present research, we refer to MSW as the biodegradable organic fraction, which consists mainly of paper-based products, lignocellulosic materials, food wastes, and yard waste. SS is the byproduct of wastewater treatment and consists of residual solids from conventional aerobic or anaerobic sewage treatment. In the United States, approx 236 million tons of MSW were generated in 2003 *(3)* and about 6.9 million dry tons of SS in 1998 *(4)*.

MSW is an excellent energy source but lacks nutrients necessary to maintain microorganisms during the fermentation. SS is a good source of nutrients but lacks energy-yielding carbohydrates. In this study, MSW and SS are combined, so they complement each other, making the process an attractive alternative for managing two different streams that are produced in every community.

During the last 30 yr, many approaches have emerged for using biomass to obtain useful chemicals. Sterzinger *(5)* discusses biomass gasification to produce gases that are burned in a gas turbine to generate electricity. Methane production by anaerobic fermentation from agricultural residues *(5)*, MSW *(6)*, and SS *(7)* has been studied. However, the low cost of natural gas has not allowed large-scale commercialization of these alternative fuel technologies. The most common method of biomass-to-energy conversion is simultaneous saccharification and fermentation. This process involves the enzymatic hydrolysis of lignocellulosic biomass to sugars and the fer-

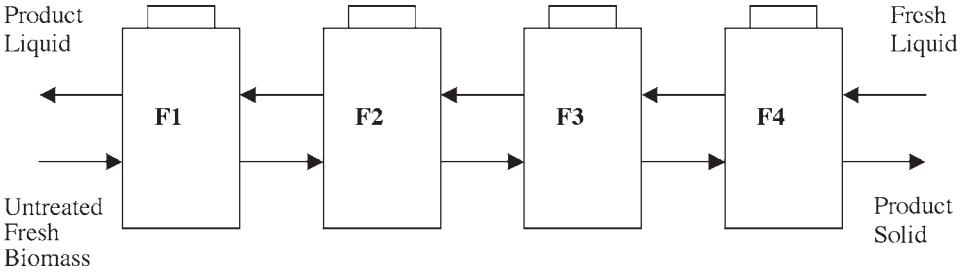

Fig. 1. Normal countercurrent fermentation procedure.

mentation of these sugars to ethanol. The high cost of enzymes and the need for sterile operating conditions are the primary drawbacks of this process.

Holtzapple et al. *(1,8)* developed the MixAlco process, another route to obtain fuels from biomass, which uses low-value materials and has low operating costs. In this process, the biomass is first pretreated with lime to increase digestibility. Then, using anaerobic countercurrent fermentation with a mixed culture of acid-forming microorganisms, carboxylate salts are produced. These salts are subsequently concentrated, thermally converted into mixed ketones, and finally hydrogenated to mixed alcohols.

Countercurrent fermentation allows the least reactive biomass to contact the lowest carboxylic acid concentration, which in batch fermentations could not be digested because of the accumulation of carboxylic acid (Fig. 1). As the solids are transferred from one fermentor to the next upstream fermentor (i.e., from F1 to F2, F2 to F3, and F3 to F4; see Fig. 1), the biomass becomes less reactive and the carboxylate salt concentration becomes lower. This flow arrangement reduces the inhibitory effect of high product concentrations by adding fresh liquid to the least-reactive biomass. Both high conversions and high product concentrations are possible by using countercurrent operation.

Aiello-Mazzarri *(9)* reported that biomass digestion in experiments with MSW/SS countercurrent fermentations was <50%. Rapier *(10)* reported that *in situ* rumen digestibility of MSW was >70%. Ross *(11)* hypothesized that over time the microorganisms populating the surface of the substrate become less active, creating a barrier to further digestion by more-active microorganisms. Groleau and Frosberg *(12)*, using electron microscopy, observed that microorganisms coat the substrate surface in mixed-acid fermentations. In the present study, the normal countercurrent process was modified by adding an intermediate lime-treatment step between F3 and F4, as shown in Fig. 2. Because raw MSW already contains many digestible components (e.g., office paper), it is not necessary to treat the raw MSW fed to F1; many components are already digestible without pretreatment. Further, treating the solids exiting F3 with lime removes dead or less-active microorganisms from the substrate surface and increases the digestibility of the less-reactive solids to improve the product concentrations.

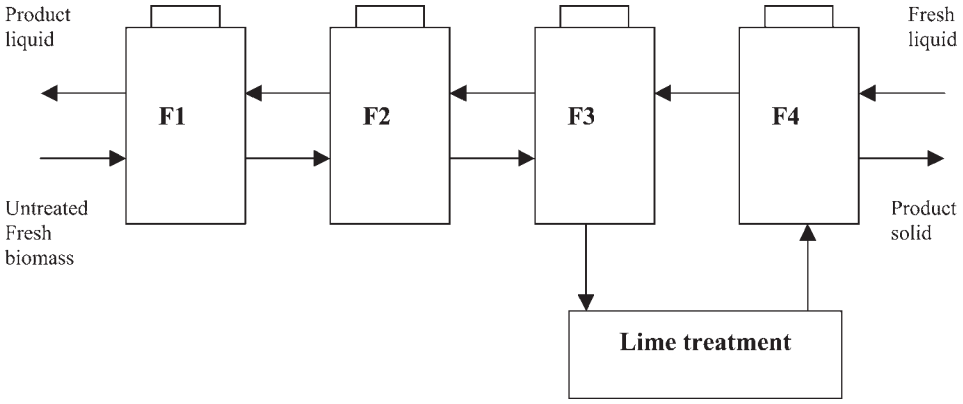

Fig. 2. Countercurrent fermentation procedure with intermediate lime treatment.

Materials and Methods

Substrates

Municipal Solid Waste

MSW was prepared to simulate the organic fraction of the landfill waste reported by Holtzapple et al. *(13)*. The components of MSW were collected, sun-dried, ground, and passed through a 10-mm screen in a hammer mill (Forest Science Research Laboratory, Texas A&M University). Then the components were combined, ground, and passed through a 6-mm screen to ensure a uniform mixture. Fats and oils were not added to the MSW to prevent spoiling during storage.

Sewage Sludge

Aerobically treated SS was obtained from Bryan Wastewater Treatment Plant Number 3 (Bryan, TX), dried, and ground in a hammer mill fitted with a 3-mm screen.

Lime Treatment

The solids from F3 were treated with lime ($Ca(OH)_2$) at 100°C for 1 h with 0.1 g of lime/g of dry biomass and 10 mL of distilled water, which was enough to form the slurry. After treatment, CO_2 was bubbled through the biomass slurry to neutralize the lime and the slurry was dried at 105°C. Detailed procedures of the lime treatment are available in ref. *9*.

Countercurrent Fermentation Experiments

In countercurrent operation, liquid and solids flow in opposite directions in four-fermentor trains. At the laboratory scale, the fermentors operate in a semicontinuous manner. In an industrial scale, the fermentation would operate in a continuous countercurrent manner.

Countercurrent fermentations (A1, A2) were initiated as batch cultures under anaerobic conditions by adding 80 g of MSW, 20 g of SS, 2 g of calcium carbonate, 0.15 g of urea, 0.20 g of nutrients, and 50 mL of terrestrial inocula to 200 mL of deoxygenated water medium in each fermentor. The fermentors were operated in batch mode until the culture was established (7–10 d). Then the countercurrent operation was initiated with the transfer of liquid and solids occurring every 1, 2, or 3 d. All the fermentations were conducted at 40°C.

The fermentors were operated in a four-stage countercurrent system as described in Fig. 1. The liquid produced in one reactor was fed to the next reactor upstream, and the solids from a reactor were moved to the next reactor downstream. This allowed the less-reactive biomass to contact the lowest carboxylic acid concentrations and the most-reactive biomass to contact the highest carboxylic acid concentrations. A constant wet cake of predetermined weight was maintained in each fermentor to achieve steady-state conditions, which were evidenced when a consistent acid concentration was produced for at least 2 wk consecutively.

At each transfer session, the fermentors were taken from the incubator and the gases produced were released and measured. The fermentors were opened, purged with nitrogen, capped with a centrifuge bottle cap, and centrifuged for 25 min to separate the solids and the liquid. The solid and liquid were subsequently transferred in opposite directions.

A 3-mL sample of the liquid from F1 was taken for carboxylic acid analysis, and the rest was decanted into a collection bottle for later VS analysis. Solids from F4 were collected in a centrifuge bottle for VS analysis. Fresh biomass was added to F1, and fresh liquid medium was added to F4. The entire transfer process was made under continuous nitrogen purge. Once the transfer was completed, the fermentors were closed and placed back in the incubator.

An intermediate lime treatment of solids exiting F3 before entering F4 was studied. Fermentation solids from F3 were treated with lime before they were added to F4, as shown in Fig. 2. Initially, solids from F3 were collected for 4 to 5 wk, treated with lime, and dried. The dry matter of untreated and treated solids was determined. During the collection time, fresh biomass was added once a week to maintain the culture and the reactor weight. Details of the procedures are presented in ref. *9*.

Reaction Conditions

The fermentations were performed under anaerobic conditions at 40°C. Every 3 d, after each liquid/solid transfer, 2.0 g of calcium carbonate was added to each fermentor to neutralize the carboxylic acids and to control the pH. Urea was added as a nitrogen source. To maintain anaerobic conditions, nitrogen from a high-pressure liquid nitrogen cylinder (Praxair, Bryan, TX) was flushed whenever the fermentors were open to the atmosphere. The solid and liquid transfer procedures are detailed in ref. *9*.

Media and Nutrients

The fermentation media consisted of deoxygenated distilled water, 0.28 g of sodium sulfide/L of distilled water, and 0.28 g of cysteine hydrochloride/L of distilled water. The dry nutrient mixture added to all fermentors was the modified Caldwell and Bryant medium *(14)*.

Inocula

The inoculum used in all the experiments included rumen fluid from a fistulated steer (University Nutrition and Field Laboratory, Texas A&M University), swamp material from Bee Creek Park (College Station, TX), and compost material from domestic and commercial piles. To minimize exposure to oxygen, the swamp material and compost were collected in bottles filled with deoxygenated distilled water.

Methanogen Inhibitor

Iodoform (CHI_3) was used as methanogen inhibitor in all the fermentations. A CHI_3 solution (20 g of CHI_3/L of ethanol) was added individually to each reactor continuously throughout the fermentations. The CHI_3 solution was kept in amber-colored glass bottles, and special care was taken to replace the cap immediately after use.

Analytical Methods

Gases produced during fermentation were accumulated within the reactor. Every sampling day, the volume of gas produced since the last transfer session was measured. The total gas volume from each fermentor was measured by displacing water in an inverted, glass graduated cylinder apparatus filled with 30% $CaCl_2$ solution. The $CaCl_2$ minimized microbial growth in the water tank and reduced water evaporation. The $CaCl_2$ solution had an acidic pH (5.6), which prevented CO_2 adsorption.

By gas chromatography (GC), the gas was analyzed for CH_4 (every 2 or 3 d) to check inhibition of the methanogen. A 5-mL sample was taken through the reactor septum and analyzed using an Agilent 6890 series gas chromatograph equipped with a thermal conductivity detector. A 4.6-m stainless steel packed column with a 2.1-mm id (60/80 Carboxen 1000, Supelco® 1-2390 U) was used. The inlet temperature was fixed at 230°C, the detector temperature was set at 250°C, and the oven temperature was maintained at 225°C for 5 min. Helium was used as carrier gas. The total elution time for a sample was 5 min.

It was assumed that only CH_4 and CO_2 were produced from the microbial digestion. Knowledge of the total amount of gas and the amount of CH_4 allowed CO_2 production to be calculated. The CO_2 produced was differentiated into biotic and abiotic CO_2. The biotic CO_2 was produced directly from the fermentation, whereas the abiotic CO_2 was produced when the

carboxylic acids were neutralized with calcium carbonate. It was assumed that 1 mol of abiotic CO_2 was produced for every 2 mol of acid produced. In the mass balance calculations, only the biotic CO_2 should be included; therefore, the abiotic CO_2 was subtracted from the total CO_2 produced.

The fermentor broth was analyzed by GC to measure the concentration of carboxylic acids. The fermentation broth consisted of a mixture of carboxylate salts and carboxylic acids. A broth sample was mixed with equal parts of an internal standard (4-methyl-*n*-valeric acid) and 3 *M* H_3PO_4. All salts were converted into their corresponding acids, so product concentrations are reported as grams of carboxylic acid/liter. The analysis was performed using an Agilent 6890 series gas chromatograph (Palo Alto, CA; www.agilent.com) equipped with a flame ionization detector and a 7683 series injector. A 30-m fused-silica capillary column (model no. 123-3232 CX; J & W Scientific) was used. The column head pressure was maintained at 90–103 kPa (13–15 psig). At every sample injection, the gas chromatograph temperature program allowed the temperature to rise from 50 to 200°C at a rate of 20°C/min. Then the temperature was held at 200°C for 10 min. Helium was used as carrier gas, and the total run time per sample was 17 min.

Volatile solids (VS) in the initial substrates and solid fermentation residues were determined by first drying the material at 105°C and then ashing the material at 550°C for at least 3 h. VS are defined as the fraction of a dry sample that volatilizes after ashing. Determination of VS in the liquid fermentation broth followed the same heating procedure as just described except that prior to drying the liquid was mixed with lime to ensure that the carboxylic acids would not volatilize and alter the measurement.

For all the countercurrent fermentation experiments, a complete mass balance was obtained on the entire train over a steady-state period. The closure represents the difference between the mass entering and exiting the fermentation system. The mass balance closure is as follows:

$$\text{closure} = \frac{\text{mass out}}{\text{mass in + water of hydrolysis}} \tag{1}$$

$$= \frac{\text{undigested VS + dissolved VS + carboxylic acids produced + biotic } CO_2 + CH_4}{\text{mass in + water of hydrolysis}} \tag{2}$$

To calculate the water of hydrolysis, it was assumed that the biomass could be represented as cellulose, which has a monomer weight of 162 g/mol. When cellulose is hydrolyzed, it gains 1 mol of water/monomer; therefore, the water of hydrolysis is calculated as follows:

$$\text{water of hydrolysis} = \text{VS digested} \times \frac{18}{162} \tag{3}$$

Operational Parameters

The liquid residence time (LRT) determines how long the liquid remains in the system and also affects the final product concentration. LRT is calculated as follows:

$$\text{LRT} = \frac{\text{TLV}}{Q} \tag{4}$$

in which LRT is measured in days (d); Q is the flow rate of liquid out of the fermentor set (L/d); and TLV is the total liquid volume, calculated as

$$\text{TLV} = \sum_i (\overline{K}_i \cdot w + \overline{F}_i) \tag{5}$$

in which $\overline{K}_i$ is the average wet mass of solid cake in Fermentor i (g), w is the average liquid fraction of solid cake in Fermentor i (L of liquid/g of wet cake), and $\overline{F}_i$ is the average volume of free liquid in Fermentor i (L).

The VS loading rate (VSLR) is calculated as follows:

$$\text{VSLR} = \frac{\text{VS fed/d}}{\text{TLV}} \tag{6}$$

Biomass is composed of VS and ash, and except for the lignin most of the VS are reactive. The digestion process converts part of the VS into gas and liquid products, with some solids remaining undigested. In the liquid products, VS consist of carboxylic acids, extracellular proteins, and energy storage polysaccharides *(11)*. The following terms are used throughout the present article:

$$\text{conversion} = \frac{\text{VS digested}}{\text{VS fed}} \tag{7}$$

$$\text{yield} = \frac{\text{total carboxylic acids produced}}{\text{VS fed}} \tag{8}$$

$$\text{total acid selectivity} = \frac{\text{total carboxylic acids produced}}{\text{VS digested}} \tag{9}$$

$$\text{total acid productivity} = \frac{\text{total carboxylic acids produced}}{\text{L of liquid in all reactors} \times \text{time}} \tag{10}$$

VSLR [g VS/L liquid in all fermentors·d]

Conversion [g VS digested/g VS fed]

Yield [g total acids/g VS fed]

Total acid selectivity [g total acids/g VS digested]

Total acid productivity [g total acids/(L liquid·d)]

Results and Discussion

MSW/SS countercurrent fermentations were conducted with and without intermediate lime treatment at different LRTs and VSLRs. Table 1 provides the operating parameters for each fermentation.

Table 2 presents the results of the countercurrent fermentations with and without intermediate lime treatment. Fermentations A1 and A2 were initiated using the same substrate (80% untreated MSW/20% SS) and the same inocula mixture. Both fermentations were started on the same day and operated identically for the first 30 d. On d 32, the procedures for an intermediate lime treatment were started on fermentation A2. The solids from F3 were removed for treatment, and fresh solids were periodically added to F4 to maintain the culture. The solids collected over a certain period were treated with lime, neutralized to pH 7.0 by bubbling with CO_2, and dried. The amount of dry-treated solids was divided by the number of transfers during the collection period. This amount was added to F4 over the same period as it was collected to maintain consistency and ensure that all the solids removed from F3 were added to F4. Details for the transfer procedure are presented in ref. *9*. The addition of lime-treated F3 solids to F4 started on d 50.

Steady-state conditions on fermentation A1 were evidenced between d 61 and 253, whereas fermentation A2 reached steady state on d 94 and was operated until d 253. Figure 3 shows the total acid concentrations obtained during fermentations A1 and A2. The total carboxylic acid concentration, acid productivity, selectivity, yield, and conversion of fermentation A2, with the intermediate lime treatment between F3 and F4, were higher than those of fermentation A1, without the intermediate treatment. A *t*-test (two-sample assuming unequal variances; $\alpha = 0.05$) performed on the steady-state total carboxylic acid concentration of fermentation A1 compared with fermentation A2 showed that there were significant differences between the two fermentations. These results indicate that the additional treatment step between F3 and F4 can increase the digestibility and acid productivity of the fermentation. Mass balance closure and conversion were determined as 97 and 43.0%, and 92 and 47.8%, for fermentation A1 and A2, respectively (Fig. 4).

Fermentation B1 was started by altering the feed conditions from fermentation A1. The fermentors were placed back in the incubator on d 270, and the countercurrent transfers were initiated on d 274. The fermentors were reinoculated with 50 mL of rumen fluid on d 279. Fermentation B2 was started by changing the feed condition from fermentation A2. As in fermentation A2, an intermediate lime treatment was applied to the solids exiting F3. The solids from F3 were collected over a certain period, treated with lime, neutralized to pH 7.0, and dried. The treated solids were added to F4 over the same period, as they were collected.

The steady-state conditions on fermentation B1 were evidenced between d 369 and 594, whereas on fermentation B2 they were evidenced

Table 1
Operating Parameters for Untreated MSW Countercurrent Fermentations

Parameter	Fermentation train			
	A1	A2	B1	B2
Intermediate lime treatment between F3 and F4	No	Yes	No	Yes
LRT (d)	20.5	20.5	24.2	24.0
VSLR (g of VS/[L of liquid in all fermentors·d])	4.2	4.4	7.2	7.1
VS feed at each transfer (g of VS)	11.8	11.8	19.8	19.8
Solid feed at each transfer (g of dry)	13.8	13.8	23.0	23.0
Liquid feed to F4 at each transfer (L)	0.20	0.20	0.20	0.20
Frequency of transfer	Every 3 d	Every 3 d	Every 3 d	Every 3 d
Centrifuge procedure	Double	Double	Double	Double
CHI_3 addition rate (mg of CHI_3 added/L of liquid fed to F4)	4	4	4	4
Nutrients addition rate (g of dry nutrients added/L of liquid fed to F4)	1.00	1.00	1.00	1.00
Urea addition rate (g of urea added/L of liquid feed to F4)	0.75	0.75	0.75	0.75

Table 2
Results for Untreated MSW/SS Countercurrent Fermentations[a]

Parameter	Fermentation train			
	A1	A2	B1	B2
Intermediate lime treatment between F3 and F4	No	Yes	No	Yes
LRT (d)	20.5	20.5	24.2	24.0
VSLR (g of VS/[L of liquid in all fermentors·d])	4.2	4.4	7.2	7.1
Average pH in all fermentors	6.02 ± 0.39	6.03 ± 0.34	5.94 ± 0.53	5.89 ± 0.11
Total carboxylic acid concentration (g/L)	13.57 ± 0.65	15.79 ± 0.63	17.74 ± 1.21	22.17 ± 0.75
Acetic acid (wt%)	37.74 ± 5.54	45.81 ± 2.26	44.29 ± 4.95	49.78 ± 2.67
Propionic acid (wt%)	20.44 ± 3.38	16.84 ± 2.52	13.17 ± 2.28	14.77 ± 2.10
Butyric acid (wt%)	20.20 ± 2.81	18.52 ± 2.40	20.27 ± 3.30	18.73 ± 2.62
Valeric acid (wt%)	9.69 ± 1.57	7.45 ± 1.18	8.53 ± 1.19	6.33 ± 1.44
Caproic acid (wt%)	8.44 ± 1.35	7.33 ± 2.21	9.81 ± 2.47	7.11 ± 1.65
Heptanoic acid (wt%)	1.16 ± 0.47	1.99 ± 1.06	2.02 ± 1.27	1.29 ± 1.08
Conversion (g of VS digested/g of VS fed)	0.419	0.478	0.302	0.310
Yield (g of total acids/g of VS fed)	0.171	0.197	0.095	0.125
Selectivity (g of total acids/g of VS digested)	0.408	0.413	0.314	0.403
Total carboxylic acid productivity (g of total acids/[L of liquid·d])	0.718	0.876	0.868	0.886
Biotic CO_2 productivity (g of CO_2/[L of liquid·d])	0.620	0.690	0.984	0.785
CH_4 productivity (g of CH_4/[L of liquid·d])	0.003	0.002	0.004	0.001
Mass balance closure (g of VS digested/g of VS in)	0.97	0.92	0.92	0.90

[a]All errors are ± 1 SD.

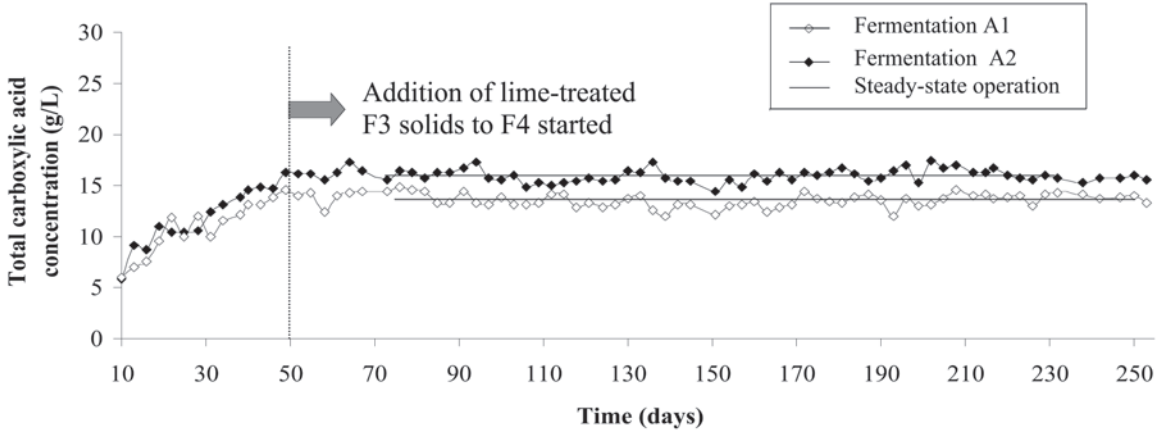

Fig. 3. Total acid concentration for untreated MSW/SS fermentation A1 (LRT = 20.5 d and VSLR = 4.2 g/[L·d]) and fermentation A2 (intermediate lime treatment; LRT = 20.5 d and VSLR = 4.4 g/[L·d]).

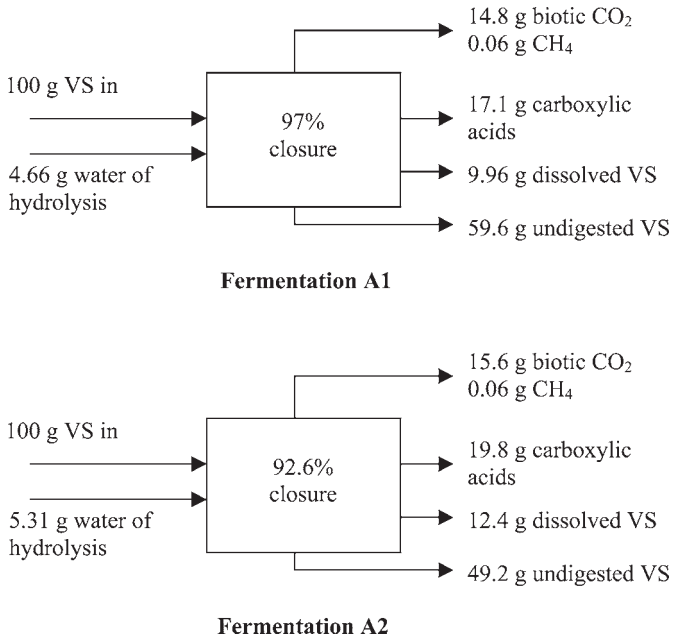

Fig. 4. Mass balances for MSW/SS fermentations A1 and A2.

between d 421 and 609. Low concentrations of CH_4, <0.5 mol%, were detected during steady-state operation. Figure 5 presents the average total carboxylic acid concentration of the product liquid from F1 as a function of time. A complete mass balance was performed on both systems, and the mass balance closure was found to be 91.1 and 90.3%, for fermentation B1 and B2, respectively (Fig. 6). Conversion was determined to be 34.1% for fermentation B1 and 31.0% for fermentation B2.

The total carboxylic acid concentration, acid productivity, selectivity, yield, and conversion of fermentation B2, with intermediate lime treatment between F3 and F4, were slightly higher than those of fermentation B1, without the intermediate treatment. Based on a *t*-test (two-sample assuming unequal variances; $\alpha = 0.05$) performed on the steady-state total carboxylic acid concentration of fermentation B1 compared with fermentation B2, there were significant differences between the two fermentations.

Conclusion

The addition of intermediate lime treatment increased product concentration and conversion by approx 30 and 15%, respectively. The highest carboxylic acid concentrations for untreated MSW/SS fermentations with and without intermediate lime treatment were 22.2 and 17.7 g of carboxylic acid/L of liquid, respectively. These results confirm that the additional treatment step between F3 and F4 increases the digestibility and acid productivity of the fermentation. The advantage of this approach is that less lime is consumed. Raw MSW contains many digestible components (e.g.,

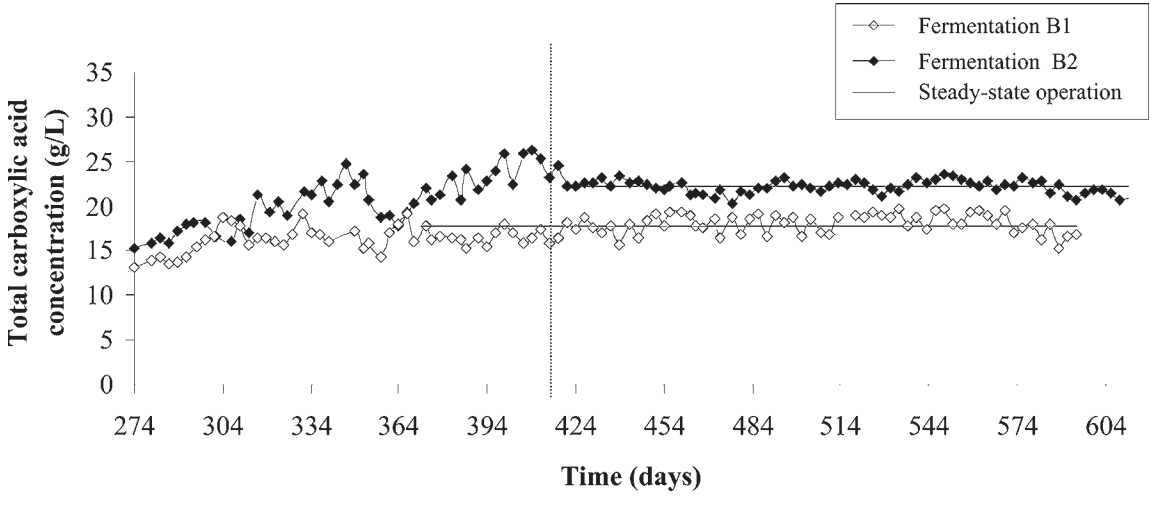

Fig. 5. Total acid concentration for untreated MSW/SS fermentation B1 (LRT = 24.2 d and VSLR = 7.2 g/[L·d]) and fermentation B2 (intermediate lime treatment; LRT = 24 d and VSLR = 7.1 g/[L·d]).

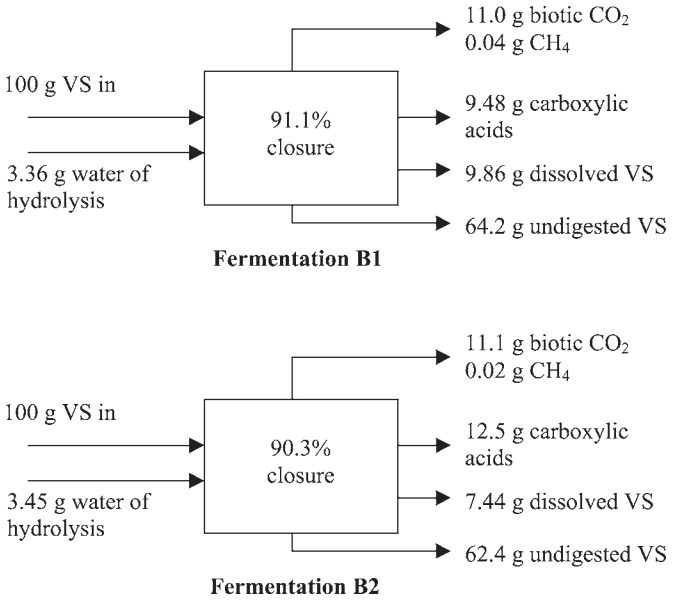

Fig. 6. Mass balance for MSW/SS fermentations B1 and B2.

food scraps) that do not need pretreatment. However, as the fermentation proceeds, the undigested residues are more refractory and do benefit from lime pretreatment. Further, any dead cells on the biomass surface would be removed, thus "freshening" the surface for additional biologic digestion.

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