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Laboratory Investigation into Factors Affecting Performance of Anaerobic Contact Process for Pear Processing Wastewater

Joon Moo Hur, Bu Soon Son*, Bong Ki Jang*, Jong An Park*, Jong Whoa Lee* and Joon Hyun Kim**

New Environmental Research Engineering Co.
**Department of Environmental Health Science, Soonchunhyang University*
***Sam Sung Corporation, Construction Group*

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Results obtained from this research showed that the anaerobic contact process was applicable to pear waste with COD removal efficiencies of up to 95% depending on conditions, provided ammonium and phosphate salts were added as well as other nutrients, present in the commercial fertilizer, Milorganite or in yeast extract. These latter materials were required in minimum concentrations of 5 and 1.5 g/L, respectively, in the feed independent of HRT and volatile solids loading rate, with part of the effect due to the mineral fraction.

Digestion was satisfactory over the whole range of volatile solids loading rates and liquid retention time of 30 to 0.5 days tested, although treatment efficiency dropped off noticeably between 1 and 0.5 day liquid retention time because of poorer flocculation and separation of anaerobic bacteria.

Settling of anaerobic bacteria including methane producing bacteria was related to settling of mixed liquor suspended solids only at 1 to 5 days liquid retention times, at other liquid retention times anaerobic microorganism settled markedly less efficiently than mixed liquor suspended solids.

Further studies are being made to provide information of practical and basic interest. Data on the composition of the active fraction of yeast extract might solve many practical nutrient problems encountered with the anaerobic contact process and improve its economics. Further improvement in the flocculation and settling of anaerobic bacteria as well as other bacteria would improve overall performance and allow the use of shorter liquid retention times with dilute waste. Knowledge about the numbers of methane formers present would allow a degree of understanding and control of the process not presently attainable.

Key words : Anaerobic contact process, Pear processing wastewater

I. Introduction

This article presents results obtained from part of a continuing study of the anaerobic treatment of food plant wastes using the anaerobic contact process. For many food processing plants, factors such as location, climate, and seasonal operation, as well as high strength and nutritionally unbalanced wastes may make it difficult or uneconomic to use conventional methods such as activated sludge process, biological filtration, and lagooning.

The anaerobic contact process, however, with its advantages of relatively simple equipment,

limited space requirements, low accumulation of biological solids, possible high loading rates, and the production of a useful by-product of methane, would appear to offer a solution for some food plant waste treatment problems.

The anaerobic contact process, developed only uses complete mixing of the fermenter contents to overcome the inefficient use of fermenter volume and low reaction rates characteristics of conventional anaerobic digestion(McCarty, 1968; Torien, 1969; Hertjes, 1979; Frostell, 1981; Schlegel, 1981; Nahle, 1991). Long solids retention times with short liquid retention times are achieved by recirculating fermenter liquid through a solid-liquid separation or concentration unit which minimizes waste of solids in the effluent from the process(Dague, 1970; Frostell, 1985; Pohland, 1992).

The main objective of the study of the this process is to determine the effect of operating variables such as loading rate, liquid retention time, pH, alkalinity, temperature, and addition of nutrients, on the process performances. Study of factors affecting adaptation of the fermentation to different wastes, solids separation and sludge return, and new methods of analysis are also planned. Actual rather than synthetic waste was chosen as fermentation substrate because it was felt that the more direct approach would better serve the objectives of the study. Pear processing waste was selected for the initial part of the work because it was typical of many fruit processing wastes(high carbohydrate content, nutritionally unbalanced), and could be readily obtained and stored as a frozen concentrate.

Promising results were obtained in the initial phase of the study(van den Berg, 1971). Treatment efficiencies, in terms of reduction in chemical oxygen demand(COD), varied between 75% and 95%, depending on volatile solids loading rate and liquid retention time of 10-86 days. Addition of yeast extract, in addition to ammonium and phosphate salts, was necessary.

However, separation of solids from the process effluent for return to the fermenter was a problem, and solids return did not seem to be directly related to return of bacterial floc. Determination of changes in the anaerobic bacterial population was also a problem.

This article presents results obtained over a long range of liquid retention time of 0.5 to 30 days, and a higher range of volatile solids loading rate than were tested initially, and also includes the results of a study of nutrient requirements. Further data on solids and bacterial floc separation are also given and an improved system fro sedimentation and sludge return is described.

II. Materials and Methods

2.1. Reactor set-up and feed preparation

A laboratory anaerobic contact process system used in this study is illustrated in Figure 1. Consideration in designing the reactor was adapted from the earlier study(van den Berg, 1971). Completely-mixed reactor(fermenter) consisted of 12″×24″ outer diameter plexiglass with ground shape, 1/2″ plexiglass cove, and had a working volume of 30 liters. Baffles were installed to improve the mixing efficiency and avoid the vortex mixing. Mechanical mixing system was used, and rotating speed was variable from 50 to 150 rpm. Sedimentation unit for solid-liquid separation consisted of sedimentation zone, effluent withdrawal port and venting unit for digester gas.

Agitation was provided by a single-bladed rubber impeller, rotating at 10-15 pm depending on the suspended solids content to prevent the

rising gas bubbles in the sedimentation unit. Fermenter mixed liquor inlet tube was vented to avoid disturbing of effluent layer by gas bubbles.

The level in the reactor was controlled within narrow limits by the position of the inlet tubes to the pump transferring fermenter mixed liquor to the sedimentation unit. The level in the sedimentation unit was controlled by the overflow outlet. At very short retention times, a clear supernatant was obtained by operating the sedimentation unit on a one-hour draw and fill cycle rather than continuously, while maintaining continuous agitation. During the cycle, using suitable pumps operating on a timer, the flask was filled and allowed to settle for about 45minutes. The required amount of effluent was then withdrawn and the remainder of the liquid in the flask returned to the fermenter. Complete set-up, except for refrigerator waster bath was kept in temperature-controlled chamber maintained at 35℃.

Pear waste from a mechanical pear peeling line a cannery was stored frozen until required and then prepared as described previously (Lenz, 1972). In brief, after thawing, the waste was diluted with distilled water to about 9% solids, homogenized in a ultrasonic processor for three minutes, and screened to remove fibers that might plug the tubes in the peristaltic pumps. Water, salts and nutrients were added as required. For most tests, constant concentrations of ammonium and phosphate salts(4.8g NH_4HCO_3, 0.4g KH_2PO_4; 0.42g Na_2HPO_4 per liter of distilled water feed) were maintained to ensure an alkalinity of about 3,000 mg/L as $CaCO_3$ in the fermenter liquid as well as an adequate supply of nitrogen and phosphate for the microorganisms. A typical composition of concentrated feed without added salts or nutrients is given in Table 1.

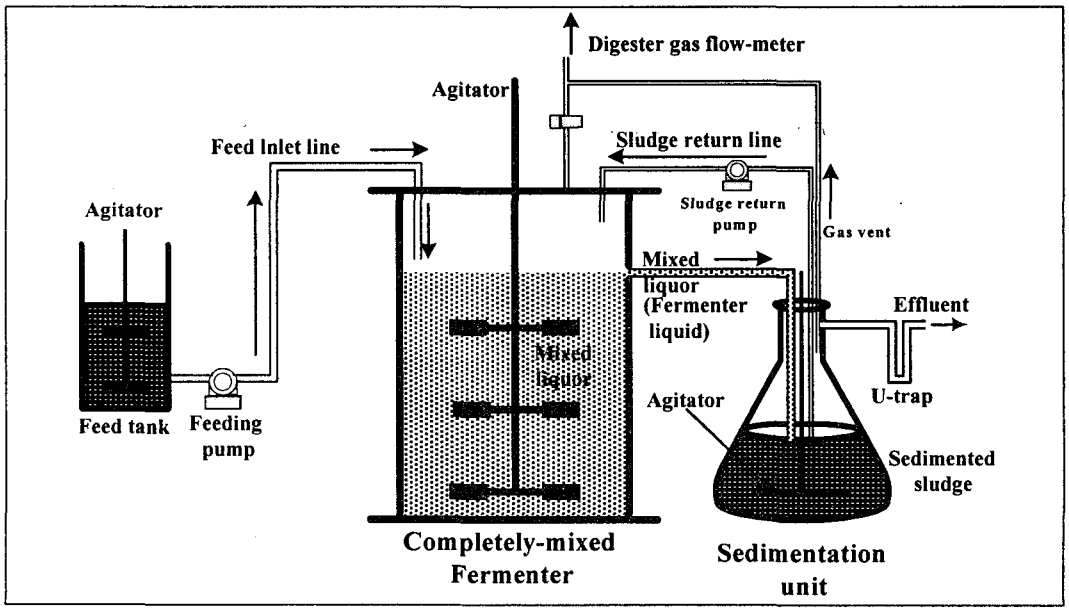

Fig. 1. Schematic diagram of ananerobic contact process system used in this study.

Table 1. Typical composition of high-strength feed before addition of salts and nutrients.

Total solids(%)	8.8
Total volatile solids(%)	8.6
Suspended solids(%)	3.8
Volatile suspended solids(%)	3.7
COD(mg/L)	120,000
Acid-indigestible, non-protein solids(%)	3.2

2.2. Operation and treatment test

For each test, anaerobic contact systems were operated until steady-state conditions had been obtained for at least a week or until it became evident that steady-state conditions were unattainable under the operating conditions. The latter situation prevailed when the concentration of volatile acids continued to increase and the pH to decrease even after frequent interruption of feed addition to allow the anaerobic bacterias to catch up.

Steady-state was usually obtained 4 to 6 weeks after a change in test conditions had been made. Feed addition was basically continuous where cycling was used, it was on a 20 minutes basis, as was effluent removal, except as 0.5 and 1 day liquid hydraulic retention time as noted above.

The more concentrated feed used at 10 to 30 days liquid hydraulic retention times was pumped as such into the fermenter(reactor). The more dilute feed used at 0.5 to 5 days liquid hydraulic retention times was prepared by continuously mixing concentrated feed(usually at 120,000 mg/L as COD) with salt solution(4.8 g NH_4HCO_3, 0.4 g KH_2PO_4, 0.42 g Na_2HPO_4 per liter of distilled water feed) at the fermenter inlet. Undigestible material and excess biological solids accumulating in the fermenter were removed at the beginning of each test. Special precautions to exclude air from contact with the fermenter liquid were not necessary except when the sedimentation unit was operated on a batch basis(0.5 and 1 days HRT), then in gas spaces in the fermenter and sedimentation unit were continuously flushed with a nitrogen gas.

2.2.1. Nutrient test

Tests were conducted to determine the minimum quantity of yeast extract required for various combinations of liquid hydraulic retention times and volatile solids loading rate(Difco yeast extract was used) as well as to pinpoint the active component in yeast extract and to find cheaper substitutes. The latter tests were all made using a liquid retention time of 10 days and a volatile solids loading rate of 2.4 $kg/m^3/d$. These values were selected because they allowed the composition of the fermenter liquid to change reasonably quickly without washing out the methane formers and without overloading the fermenter.

Yeast extract components tested in this study and in combination included the minerals in yeast extract, mineral salts, amino acids and vitamins. A solution of the minerals in yeast extract was obtained by ashing yeast extract at 600℃ and dissolving the residue in hydrochloric acid. The amount of the resulting solution to be added to the feed was calculated on the basis of the yeast extract normally added to the feed(1.5 g/L). Minerals usually required by microorganisms and possibly present in pear waste in insufficient quantities, were added in the form of a concentrated salt solution as shown in Table 2. Amino acids were added in the form of easily hydrolyzed casein(up to 5 mL of a 10% suspension per liter feed). Vitamins added up to 10 mL of the solution specified in Table 3)

included folic acid and cyanocobalamin, of direct concern in methane fermentations, as well as vitamins ordinary required by many micro organisms.

Table 2. Composition of salt solution used in nutrient study.

Components	Content (g/L)
$FeCl_2 \cdot 4H_2O$	10
$MnCl_2 \cdot 4H_2O$	10
$CoCl_2 \cdot 6H_2O$	10
$MgCl_2 \cdot 6H_2O$	10
$CaCl_2$	10
H_3BO_3	5
$(NH_4)_6MO_7O_{24} \cdot 4H_2O$	10

Table 3. Composition of vitamin solution used in nutrient study.

Component of vitamin	Content (mg/L)
Nicotinic acid	1,130
Riboflavin	110
Pyridoxine hydrochloride	85
Folic acid	50
Thiamin hydrochloride	15
Vitamin B_{12}(cyanocobalamin)	2

Yeast extract substitutes tested included basker's yeast, yeast grown directly in the feed containing the salt solution specified in Table 2 (1 mL/L) and extract made from this yeast as well as Milorganite, the commercial fertilizer composed of sterilized, dried digested sewage sludge. Commercial basker's yeast was added in amounts up to 20 g/L.

The effects of the addition of yeast grown in pear waste(about 8 g/L) and of yeast extract prepared by passing the feed-grown yeast twice through a Raper press at 5,000-10,000 lb/in^2 pressure(Raper, 1963), were also tested. Finally, tests were made with feed containing up to 5 g Milorganite/L. In these latter tests, Milorganite was either added directly to the feed or an extract was prepared by autoclaving Milorganite with water. Since Milorganite contains 5% nitrogen and 3% phosphate, the amount of ammonium and phosphate salts added was reduced in one test by a factor of ten.

At the start of each test involving addition of a new yeast extract component or substitute, an amount of the new nutrient equivalent to that in 20 liters of feed was added directly to the fermenter to shorten the period required for the fermenter liquid concentration of the added nutrient to become stable.

2.2.2. Treatment efficiency test

Treatment efficiencies were determined for several combinations of liquid hydraulic retention time and volatile solids loading rate. Liquid hydraulic retention times varied from 30 days to 0.5 days, usually considered the minimum for anaerobic digestion. Volatile solids loading rates varied from 1.6 kg/m^3/d, about the minimum that would be of commercial interest, to 7.21 kg/m^3/d, the highest rate possible with longer HRTs using a already concentrated waste(over 60,000 mg/L COD). Three aspects were taken into account in considering treatment efficiency.

First, the reduction in COD, comparing feed and effluent, was considered an indication of overall reduction in pollution load. Secondly, suspended solids content and methane-forming activity of effluent were compared with those of fermenter liquid to indicate flocculation and settling efficiency, and their limiting effect on the anaerobic process. Finally, the amount of suspended solids accumulating in the fermenter were determined in relation to volatile solids loading rate, since these accumulated solids presented part of the waste disposal problem

represent a limitation in reduction of the pollution load.

2.3. Analysis

Factors included in the analyses and the methods of analysis were the same as described previously(van den Berg, 1971), with one addition, the acid-indigestible, non-protein solids content of pear waste.

This was determined as follows : the suspended solids in 20 mL of pear waste was separated from the liquid by centrifugation and washed several times with water before being suspended in about 20 mL 0.1N HCl.

The suspension was placed in boiling water for five hours to hydrolyze components such as starch and pectins, cooled and filtered. The remaining solids were washed with water and dried to a constant weight. The total Kjeldahl nitrogen content of the dry residue was then determined and the weight of the dry residue corrected for its protein content(6.25×Kjeldahl-nitrogen content).

The frequency of analysis varied with the nature of the test. The pH measurements were made daily. Volatile acids concentration and alkalinity were checked 1 to 4 times per week, depending on fermenter condition, and solids and chemical oxygen demand(COD) of fermenter liquid and effluent, as indicators of performance, once a week.

The ratio of methane producing activity of effluent to that of fermenter liquid as an indication of the efficiency with which anaerobic bacteria with the floc in the sedimentation unit was generally determined only during steady-state conditions. All other parameters were conducted according to American Standard Methods (American Public Health Association, 1992).

III. Results and Discussion

3.1. Nutrient requirement

The minimum requirement for yeast extract was found to be about 1.5 g/L of feed at all volatile solids loading rates tested. This result was unusual, in that nutrients are generally required in proportion to the mass of bacteria produced rather than in a fixed concentration. This requirement for a minimum concentration independent of loading rate may indicate that yeast extract is necessary to maintain favorable conditions for anaerobic bacteria, but that it is not directly involved in growth, at least not in the quantities added.

Attempts to identify the active fraction of yeast and to find cheaper substitutes showed that some, but not all of the activity residue in the mineral fraction, and that Milorganit could be satisfactory substituted for yeast extract, as shown in Figure 2. By adding the salt solution specified in Table 2(1 mL/L of feed), or the mineral fraction of yeast extract, the volatile acid content of the fermenter liquid leveled off at 1,500~2,000 mg/L, rather than continuing to increase at it did when no nutrients were added. Similar high volatile acid contents were obtained by adding baker's yeast, yeast grown in pear waste or extract prepared from this year. With Milorganite or commercial yeast extract on the other hand, the volatile acid content was usually only 10-50 mg/L. The minimum requirement for Milorganite was 5 g/L.

Only about 10% of this was found to be soluble under the test conditions, indicating that the essential ingredients are present in relatively small quantities. Methane-forming bacteria activity tests on fermenter liquid and effluent during the nutrient requirement studies showed gthat less than 10% of the activity was lost in

effluent at any time. The effect of yeast extract and Milorganite is therefore not to improve flocculation, but apparently to increase growth rate resulting reduction in death rate.

Tests with Milorganite added to feed which contained only one-tenth of the ammonium and phosphate salts usually added, showed that only small amounts of these salts were really necessary for satisfactory digestion in the presence of Milorganite. In these tests the volatile acid concentration was 10 to 50 mg/l as acetic acid, the alkalinity was about 500 mg/L ac $CaCO_3$ and the pH was 6.0-6.2. It was necessary, however, to boil the Milorganite in a small amount of water or soak it overnight in a small amount of the ammonium and phosphate solution normally used. The active ingredients in Milorganite do not appear to dissolve readily in very dilute salt solution at room temperature. As a result of the low alkalinity, the fermentation was quite sensitive to temporary overloading under these operating conditions.

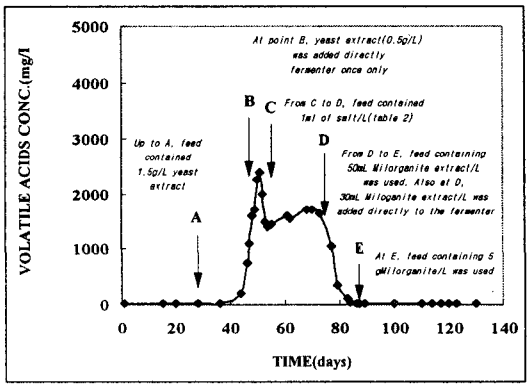

Fig. 2. Effect of withdrawal and addition of nutrients on volatile acids concentration of fermenter mixed liquor.

3.2. Treatment performances

Treatment efficiencies in terms of COD removals from feed to effluent varied from 65% to 95%, and COD removals decreased with liquid hydraulic retention time, but independent of the volatile solids loading rate as shown in Table 4.

Efficiency decreased relatively little until the liquid retention time was lowered to 0.5 days. The change then was caused by an increase in the volatile acid concentration from less than 100 mg/L as acetic acid at the HRT of 2 to 30 days to 400 to 800 mg/L as acetic acid at an HRT of 0.5 days, indicating a slower rate of conversion of volatile acid into methane gas. The higher volatile acid concentration is also reflected in the increased COD present in soluble form in the effluent at an HRT of 0.5 days, as shown in Table 5.

Table 4. Effect of liquid hydraulic retention time and volatile solids loading rate on the COD removal.

Fermenter liquid HRT(days)	COD removal (%)		
	Volatile solids loading rate ($kg/m^3/d$)		
	1.6~2.56	3.84~4.32	5.13~7.37
30	93	95	-
20	89	-	92
10	89	89	-
5	-	91	91
3	-	91	-
2	-	-	88
1	-	-	88
0.5	-	65	65

All tests were conducted with feed containing 1.5g of yeast extract/liter of dilute feed.

Figure 3 and Figure 4 illustrated suspended solids concentration of fermenter liquid and effluent, respectively. Suspended solids concentration decreased with decreasing HRT and volatile solids loading rate. The suspended solids content of effluent decreased more rapidly with decreasing liquid retention time than did that of fermenter liquid, indicating that the sedimentation efficiency increased markedly.

Table 5. Effect of liquid hydraulic retention time and volatile solids loading rate on the soluble COD in the effluent.

Fermenter liquid HRT(days)	Soluble COD percent (%)		
	Volatile solids loading rate ($kg/m^3/d$)		
	1.6~2.56	3.84~4.32	5.13~7.37
30	1	1	-
20	1	-	1
10	-	2	-
5	-	2	3
3	-	3	-
2	-	-	3
1	-	-	6
0.5	-	20	25

All tests were conducted with feed containing 1.5g of yeast extract/liter of dilute feed.

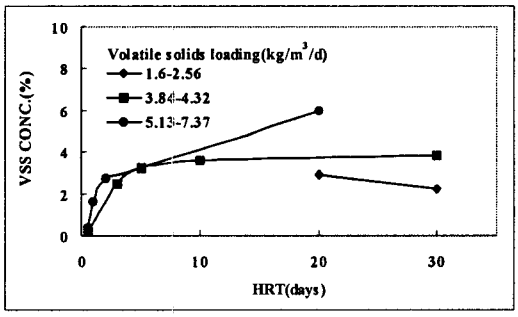

Fig. 3. Effect of HRT and volatile solids loading rate on volatile suspended solids concentration of fermenter mixed liquor.

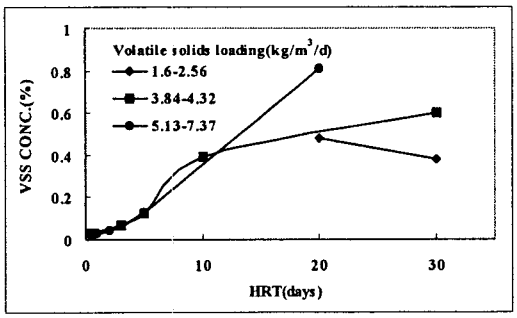

Fig. 4. Effect of HRT and volatile solids loading rate on volatile suspended solids concentration in the system effluent.

At 1 to 5 days of HRT this efficiency was over 95% as a result of an improvement in flocculation of anaerobic microorganisms(van den Berg, 1971; Velasco, 1986). Comparison of results in Figure 3 and Figure 4 shows that further improvements in flocculation are possible, leading to increasing the treatment efficiency. Sedimentation and treatment efficiencies were not affected noticeably by the increase in suspended solids concentration of fermenter liquid occurred during individual tests as a results of accumulation of indigestible solids in pear waste and excess anaerobic microorganisms.

Anaerobic microorganism including methane producing bacteria flocculated and settled readily at the HRT of 1 to 5 days, but flocculation and settling characteristics were less satisfactory at longer or shorter HRT as shown in Table 6. At the HRT of 10 to 30 days, incomplete flocculation and settling of volatile suspended solids did not affect the fermentation, presumably because the net growth rate was adequate, although slow generation time calculated from data Table 6 were 40 days or longer. At an HRT of 0.5 days, incomplete settling characteristics was associated with a high volatile acids concentration(400-800 mg/L), indicating that the number of anaerobic bacteria in the fermenter was insufficient, although the generation time must have been as short as an HRT of 2 days. Since under the latter condition, supply of volatile acids and nutrients to the anaerobic bacteria was adequate and the same or higher than at longer HRT, the incomplete flocculation must be related to factors other than nutrient supply. Possibly the condition or activity of non-methane producing bacteria, whose substrate was a function of HRT, was involved.

Comparison of results in Figure 3, Figure 4 and Table 6 show that, contrary to what is generally assumed, settling of suspended solids was not always a good indicator of the settling of

microorganisms. Although the settling of anaerobic bacteria parallelled very closely that of suspended solids at the HRT of 1 to 5 days, it differed markedly at shorter and longer liquid retention times. This indicates that solids retention time(SRT, defined as the amount of suspended solids in the fermenter per the amount of suspended solids leaving the system) a parameter used as a performance characteristics in anaerobic digestion, is not a reliable indicator of the retention time of anaerobic bacteria.

Table 6. Effect of liquid hydraulic retention time and volatile solids loading rate on separation and return of anaerobic bacteria.

Fermenter liquid HRT (days)	% of anaerobic bacteria returned to fermenter		
	Volatile solids loading rate ($kg/m^3/d$)		
	1.6~2.56	3.84~4.32	5.13~7.37
30	55	50	-
20	55	-	75
10	-	85	-
5	-	95	93
3	-	95	-
2	-	-	94
1	-	-	95
0.5	-	75	70

The amount of gas produced was proportional to the amount of volatile solids removed. The methane content of digester gas varied from between 55% and 60% depending on the alkalinity of mixed liquor.

Volatile suspended solids accumulating during tests amounted to 30 to 35% of the volatile solids present in the feed. These solids settled readily and could presumably be disposed of without great difficulty. The amount of volatile suspended solids accumulating was smaller than the amount of acid-indigestible non-protein solids added with the waste, indicating that part of the acid-indigestible solids was hydrolyzed in the fermenter.

Differences between results presented here and those obtained earlier include generally higher COD removal, which was 88 to 95% for most tests instead of 75 to 95%, lower volatile acids concentration of the fermenter liquid (less than 100 mg/L in most tests compared with 70-1,200 mg/L), better suspended solids separation (up to 95% as compared with 80%) and better separation of anaerobic bacteria (up to 95% instead of 30 to 75%), with a somewhat closer relationship between settling of methane formers and suspended solids.

Most of, if not all, of these differences can be attributed to the improved sedimentation unit and the maintenance of the minimum concentration of yeast extract required. Another difference, the accumulation of suspended solids at 30 to 35%, instead of at about 10% of the waste solids noted earlier, is in part attributable to improved solids separation, but is also a result of the increased suspended solids content of the feed used (about 43% compared with 35% in earlier study) and the shorter interval between removals of accumulated suspended solids necessitated by the higher volatile solids loading rates used in the present series of tests.

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