

**SEWAGE SLUDGE PRE-TREATMENTS FOR ENHANCING ITS ANAEROBIC BIODEGRADABILITY**

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**1. ABSTRACT**

*Several tests at laboratory scale were carried out to study different pre-treatments for centrifuged sewage sludge (SS) prior to its anaerobic fermentation. Results show that digestability is improved by means of these pre-treatments. The concentrations achieved show a considerable improvement, although it is not very high due to the dilute nature of the sewage sludge. Concentrations of VFA of more than 6300 mg/L can be obtained with an aerobic thermophilic pre-treatment of 24 h, followed by a fermentation of 5 days at mesophilic temperatures, which represents an improvement of 21.5% with respect to the control fermenter without pre-treatment.*

*Concentrations of VFA of around 7500 mg/L can be obtained with an alkaline pre-treatment of 24 h, followed by a fermentation of 5 days at mesophilic temperatures, which represents an improvement of around 45 % with respect to the control fermenter without pre-treatment. This pre-treatment introduces a foreign anion into the system.*

*A review of some pretreatments is also carried out.*

**2. INTRODUCTION**

Organic solids are present in very large quantities as products or waste from the agriculture, food industry, household and many other fields. Biological treatments of organic waste such as anaerobic digestion or aerobic composting have become very usual. The anaerobic digestion has advantages in comparison to composting such as a better handling of wet waste, the production of useful digester gas and the attenuation of smell and the possibility of obtaining valuable by-products such as volatile fatty acids.

Significant effort has been dedicated in recent years to find ways of improving the performance of anaerobic digestion different, especially when treating solid wastes, characterised by a high degree of particulate material. With these substrates, both accessibility of hydrolytic microorganisms to the solid matter and hydrolysis of complex polymeric components constitute the rate-limiting step (Eastman and Fergusson, 1981; Noike *et al.*, 1985; Pavlostathis *et al.*, 1988).

Several treatments have been tested to improve biodegradability. These treatments can be biological, mechanical or physico-chemical.

**1.1 Biological pre-treatments**

Among other physico-chemical parameters, the rate of anaerobic hydrolysis is a function of the microbial population and especially the source of hydrolytic enzymes. In accordance with Delgenes *et al.* (2001) hydrolysis of proteins require protease and peptidase which break proteins into peptides and aminoacids. Hydrolysis of lipids leads to the production of glycerol and long - chain fatty acids via the action of lipases. Cellulose, the most abundant carbohydrate in waste, is hydrolysed by a complex mixture of enzymes including endoglucanase, cellobiohydrolase and Beta glucosidase, which act in concert to produce glucose. Following these basic principles, the classic addition of complexes of enzymes has been carried out recently by Rademacher *et al.* (1999) to improve the efficiency of anaerobic sewage sludge digestion, and by Scheidat *et al.* (1999) who added to thickened municipal primary sludge a mixture of peptidases, carbohydrolases and lipases (from 0 to 10% on TS) which significantly improved hydrolysis at 39° and 51°C.

However they did not study the technical and economic feasibility of this addition, which are key points in the application of these methods. Economic feasibility of this pretreatment was reported by Rademacher *et al.* (1999) reporting the results of the wastewater treatment plant of Aachen-Soers (430000 IE), The anaerobic digestion systems was composed by two sludge digestion units of 10000 m<sup>3</sup> each. As a way to improve digestion, an enzyme complex including mainly cellulase was added continuously to one of the digestion units, at enzyme doses of 500-700 mg/kg. The authors concluded that enzyme addition results in an additional solid removal of 2 tons/day and a higher biogas production of 840 m<sup>3</sup>/day. This procedure led to a significant reduction of the sludge treatment costs. Based on sludge

disposal costs of 280,19 EU/t dry solid, and a 9245 EU/m<sup>3</sup> biogas benefit for the Aachen-Soers plant, a net annual cost reduction of approximately 175100 EU/year was estimated by the authors.

Among other biological methods of improvement, Capela et al. (1999) reported the influence of a pre-composting treatment on the start-up and performance of dry anaerobic digestion of pulp mill sludge. The effect was clearly visible through methane yields and consequent solids reduction which were greater than with the digestion of untreated sludge. In the same line of pre-treatment, Hasegawa and Katsura (1999) reported a 50% improvement in yields when sewage sludge was solubilised under slightly thermophilic aerobic conditions prior to anaerobic digestion. They suggest that thermophilic aerobic bacteria secrete external enzymes which dissolve sludge more actively than commercial proteinase. A similar study has also been carried out in a pilot plant in which there is an aerobic step before a leaching operation takes the lixiviates to an anaerobic reactor (Wellinger et al., 1999).

## 1.2 Mechanical pre-treatments

Pretreatment of the substrate by mechanical disintegration (size reduction of particles) should have positive effects on the anaerobic biodegradability of the substrate. The obvious reason is the increase of the available specific surface to the medium. Two effects have been reported: first, if the substrate has a high fibre content and low degradability, their comminution leads to improved gas production; and second, size reduction can lead to more rapid digestion (Palmowsky and Müller, 1999, 1999a).

Engelhart et al. (1999) studied the effects of mechanical disintegration (by a high- pressure homogenizer) on anaerobic biodegradability of sewage sludge. A 25% increase in volatile solids reduction was achieved. Investigations of degradation of soluble proteins and carbohydrates showed that a slowly degradable fraction of carbohydrates was released *via* disintegration.

In another study, Hartmann et al. (1999) found an increase of up to 25% in biogas from fibres in manure feedstock, after pre-treatment of the whole feed in a macerator before digestion. The authors recommend this method because of its low operational cost for a fuller degradation of particulate organic matter. Furthermore, looking at the size distribution, they found that the change in biogas potential did not correlate with a smaller size of fibre. Results from the maceration indicate that the biodegradability of the fibres is rather enhanced by shearing, which is not necessarily reflected by a change in size distribution. Confirming these results, Angelidaki and Ahring (1999) found an average increase of 17% biogas potential after mechanical maceration of biofibres contained in manure. In general the smaller the fibres, the higher the biogas potential. The best results showed an about 20% increase with fibres smaller than 0.35mm. The chemical treatment of the fibres with NaOH, NH<sub>4</sub>OH or a combination also led to increased methane potential. Combination of both treatments, chemical and mechanical, did not lead to any further increase. No significant difference in the biogas potential was found from fibres in the 5-20 mm range. They also studied the above- mentioned addition of hemicellulolytic or cellulolytic enzymes without any improvement in biogas potential. However, with the hemicellulose degrading bacteria B4, a 30% increase was recorded.

As stated, anaerobic digestion of solid wastes is rate-limited by the hydrolysis step, and so physico-chemical treatments are often used to promote solubilisation of organic matter. However, the substrate solubilisation step limited the anaerobic digestion of an industrial microbial biomass (Delgenes et al., 1999). A thermochemical pre-treatment based on sodium hydroxide addition was used to enhance COD solubilization at the following optimal conditions: pH = 12, T = 140°C for 30 minutes. 70% solubilization was achieved. However, anaerobic biodegradability of the pre-treated substrate did not improve, remaining near 40%. The poor anaerobic biodegradability performances were attributed to the soluble molecules generated being refractory and/or inhibitory to anaerobic microorganisms. Fractionation of the soluble pre-treated microbial biomass demonstrated that high molecular weight compounds are involved in the poor biodegradability and biotoxicity observed. Contrary to these findings, Schieder et al. (1999) stated that, with increasing pressure and temperature, the organic part of the waste is split up into short-chain fragments that are biologically well-suited for micro-organisms. After testing with food scraps and canteen waste in a pilot plant for 1800 t raw material per year, they claim that the thermal hydrolysis process gives complete energy recovery, i.e. more energy is produced than is needed for running the plant.

Table 3 shows a summary of different thermochemical studies extracted from Delgenes et al. (2001).

Reference	Substrate	Optimal conditions defined	Effect on solubilization	Effect on biodegradability
Stuckey and Mc Carty., 1978	Waste activated sludge	175°C, 30meqNaOH/l, 1h	55% COD solubilization	78% conversion of COD to CH <sub>4</sub>
Haug et al., 1978	Organic sludge	175°C pH=12 30mn	68% COD solubilization	Increase of 57% of CH <sub>4</sub> production
Patel et al., 1993	Water hyacinth	121°C pH=11 1h	58,48% COD solubilization	Increase in CH <sub>4</sub> production
Tanaka et al., 1997	Combined sludge	130°C 0.3gNaOH/gVSS 5mn	45% VSS solubilization	Increase of 220% of CH <sub>4</sub> production
Penaud et al., 1998	Industrial sludge	140°C pH=12 30mn	75% COD solubilization	40% biodegradability

Table 3: effect of thermochemical pretreatment on solubilization and anaerobic biodegradability of various wastes. (extracted from Delgenes et al. 2001)

In this paper, some pretreatments are carried out based on the experiences reported in the literature. The objective is to obtain a mixture of volatile fatty acids as concentrated as possible from diluted and concentrated sewage sludge.

## 2. MATERIALS AND METHODS

A series of experiments were carried out with sewage sludge coming from the S. Feliu wastewater treatment plant, with a VS concentration of 3,897 mg/L. VFA production from sewage sludge has been studied both in mesophilic and in thermophilic conditions in four continuous 3 and 5-liter stirred digesters. Temperature was controlled by means of hot water recirculation at 55°C or 35 °C. Four tests are carried out at an HRT of 2, 3, 4 and 5 days (mesophilic conditions). Again, four HRT were tested for the aerobic pre-treatment: 5, 12, 18 and 24 hours (in this case, thermophilic conditions).

Finally another smooth pretreatment was studied using sodium hydroxide at a diluted concentration of 1500 mg/L. Predigestion at alkaline conditions was carried out during a period of 24 h previous to the fermentation period.

A second series of similar experiments were carried out using centrifuged sewage sludge with an initial VS concentration of 3,897 mg/L and VS final concentration of 18.230 mg/L. achieved after centrifugation.

VFA production from concentrated sewage sludge has been studied in this period in mesophilic conditions in four 5-liter stirred digesters. Temperature was controlled by means of hot water recirculation at 55°C (aerobic pre-treatment) and 35°C (fermentation).

## 3. RESULTS AND DISCUSSION

### 3.1 Raw Sewage Sludge

A fermenter was used as a control to compare the effect of the pretreatment. This was tested at different retention times as shown in Table 1. Temperature of this digestion was 35°C.

Results were as presented in Table 10:

HRT (Days)	Acetic acid (mg/L)	Propionic acid (mg/L)	Butyric acid (mg/L)	Valeric acid (mg/L)	Total VFA (mg/L)
2	1225	96	25	1	1321
3	1371	39	23	0	1410
4	1812	75	18	2	1887
5	1742	66	12	1	1808

Table 1. Results of the control fermenter.

Digestion at HRT 4 days gave the best results and was selected as a control for the subsequent experiments.

### Aerobic Thermophilic Pretreatment

Aerobic thermophilic processes have been found of interest in the treatment of some solid wastes, and particularly of municipal sludges, because they are effective in the reduction of pathogenic organisms, the bio-oxidation of sorbed pollutants removed with solids during primary treatment which are largely recalcitrant to anaerobic degradation, and the solubilization of particulate biodegradable matter. Aerobic digestion was carried out in a similar digester but operated with oxygen and at a thermophilic temperature of 55°C. Digestion time was set at different durations as mentioned before (from 6 to 24 hours). Transfer to the anaerobic digester was carried out by means of a Master Flex peristaltic pump. The enhanced digestability was checked by means of the anaerobic fermentation:

HRT (hours)	Acetic acid (mg/L)	Propionic acid (mg/L)	Butyric acid (mg/L)	Valeric acid (mg/L)	Total VFA (mg/L)
6	1923	96	30	5	2019
12	2126	41	25	6	1853
18	2335	95	12	3	2430
24	2402	87	12	4	2489

Table 2. Fermentation after the thermophilic digestion pre-treatment

As can be seen some improvement in VFA concentration was achieved, which can be situated around 30%. Table 3 details these improvement as percentage respect to the fermentation at 4 days (Table 1).

HRT (hours)	Acetic acid (%)	Propionic acid (%)	Butyric acid (%)	Valeric acid (%)	Total VFA (%)
6	6	28	67	150	7
12	17	-45	39	200	-2
18	29	27	-33	50	29
24	33	16	-33	100	32

Table 3. Percentage of improvement after aerobic thermophilic pre-treatment.

As can be seen, a significant increase in valeric acid is produced. However, from the practical point of view, the percentage of improvement for TVFA is obtained when the pre-treatment is carried out for a period longer than 18 hours

### Alkaline pre-treatment

Finally a set of experiments was carried out using an alkaline pre-treatment. This time experiments were carried out at the different fermentation retention times. Results are shown in Table 4:

HRT (hours)	Acetic acid (mg/L)	Propionic acid (mg/L)	Butyric acid (mg/L)	Valeric acid (mg/L)	Total VFA (mg/L)
6	1845	98	30	0	1943
12	2152	47	28	0	2199
18	2756	90	20	1	2846
24	2633	45	15	2	2678

Table 4. Fermentation after the alkaline digestion pre-treatment

As can be seen some improvement in VFA concentration was achieved, which can be situated around 50%. Table 5 details these improvements as percentage respect to the control fermentation (Table 1).

HRT (hours)	Acetic acid (%)	Propionic acid (%)	Butyric acid (%)	Valeric acid (%)	Total VFA (%)
6	51	2	20	-	47
12	57	21	22	-	56
18	52	20	11	-50	51
24	51	-32	25	100	48

Table 5. Percentage of improvement after the alkaline pre-treatment

Results show that digestability is improved by means of these pre-treatments. However, the concentrations achieved are not very high due to the dilute nature of the sewage sludge. Alkaline pretreatment produces a higher concentration of VFA, but introduces a strange ion in the medium. Aerobic pretreatment does not have this problem but the concentrations achieved are lower. A possible strategy to improve the obtention of a solution richer in VFA would be to use dewatered sludge (by means of a filter press or a centrifuge). This strategy was studied in the next stage of the experimentation.

To improve VFA production, an aerobic pre-treatment was applied, following the procedure used with the undiluted sludge. The HRT's of the aerobic treatments are the same as in the previous period (6,12,28 and 24h). Sodium hydroxide at a diluted concentration of 1500 mg/L was used for the alkaline pre-treatment. Again, predigestion at alkaline conditions was carried out during a period of a maximum of 24 h previous to the fermentation period.

### 3.2 Centrifuged Sewage Sludge

A control fermenter was also used to compare the effect of the pretreatment on concentrated sewage sludge. This was tested at different retention times as shown in Table 6 and at a temperature of 35°C. Results were shown in Table 6:

HRT (Days)	Acetic acid (mg/L)	Propionic acid (mg/L)	Butyric acid (mg/L)	Valeric acid (mg/L)	Total VFA (mg/L)
2	3680	112	31	1	3824
3	3856	98	22	0	3976
4	4012	68	17	1	4098
5	5120	75	10	0	5205

Table 6. Results of the control fermenter with centrifuged sewage sludge

Digestion at HRT 5 days gave the best results and was selected as a control for the subsequent experiments.

**Aerobic thermophilic pretreatment**

Aerobic digestion was carried out in a similar digester but operated with oxygen and at a thermophilic temperature of 55°C. Digestion time was set at different duration as usual (from 6 to 24 hours). Transfer to the anaerobic digester was carried out manually, as there were difficulties with the use of a peristaltic pump. The enhanced digestibility was checked by means of the anaerobic fermentation (see Table 7).

HRT (hours) Aerobic pretreatment	Acetic acid (mg/L)	Propionic acid (mg/L)	Butyric acid (mg/L)	Valeric acid (mg/L)	Total VFA (mg/L)
6	5230	101	21	3	5355
12	5562	93	15	2	5672
18	5903	65	18	5	5991
24	6231	76	17	2	6326

Table 7. Results of the fermentation at HRT=5 days, after the aerobic thermophilic digestion pre-treatment

As can be seen some improvement in VFA concentration was achieved, which can be situated around 30% in the most favourable run (after 24 hours of pre-fermentation. Table 8 details these improvement as percentage respect to the fermentation at 5 days.

HRT (hours) Aerobic pretreatment	Acetic acid (%)	Propionic acid (%)	Butyric acid (%)	Valeric acid (%)	Total VFA (%)
6	2,1	34,7	110,0	-	2,9
12	8,6	24,0	50,0	-	9,0
18	15,3	-13,3	80,0	-	15,1
24	21,7	1,3	70,0	-	21,5

Table 8. Percentage of improvement of concentrated sewage sludge fermentation after aerobic thermophilic pre-treatment at HRT=5 days.

As can be seen, a significant increase in TVFA is obtained when the pre-treatment is carried out for a period longer that 18 or, better at 24 hours

*Alkaline pre-treatment*

Another set of experiments was carried out using an alkaline pre-treatment. Experiments were carried out at the same fermentation retention time (5 days). Results are presented in Table 9 as a function of the HRT of the alkaline pre-treatment.

HRT (hours) Alkaline pretreatment	Acetic acid (mg/L)	Propionic acid (mg/L)	Butyric acid (mg/L)	Valeric acid (mg/L)	Total VFA (mg/L)
6	7122	41	12	0	7175
12	7211	47	21	0	7279
18	7365	52	25	1	7443
24	7448	45	19	2	7514

Table 9. Fermentation after the alkaline digestion pre-treatment

As can be seen a more significant improvement in VFA concentration was registered as compared with the aerobic pre-treatment, which can be situated around 50%. Table 10 details these improvement as percentage respect to the control fermentation at 5 days of HRT.

HRT (h) Alkaline pretreatment	Acetic acid (%)	Propionic acid (%)	Butyric acid (%)	Valeric acid (%)	Total VFA (%)
6	39,1	-45,3	20,0	-	37,8
12	40,8	-37,3	110,0	-	39,8
18	43,8	-30,7	150,0	-	43,0
24	45,5	-40,0	90,0	-	44,4

Table 10. Percentage of improvement after the alkaline pre-treatment

Results show that digestability is improved by means of these pre-treatments. However, the concentrations achieved show a considerable improvement, although it is not very high due to the dilute nature of the sewage sludge. As in the case of undiluted sewage sludge, alkaline pretreatment produces a higher concentration of VFA, but introduces an strange ion in the medium. Aerobic pretreatment does not have this problem but the concentrations achieved are again lower.

On the positive side, it can be said that the level of VFA produced is high enough to be used for BNR in WWTP with low C/N ratios. Moreover, such pre-treatments can be used not only for SS, but also for the OFMSW with the following advantages: a) Increased biogas or VFA production; b) Reduced and more stabilized digester effluent. c) Reductions up to 50% can be easily achieved by low tech pretreatments

## 5. ACKNOWLEDGEMENTS

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