

# ENHANCEMENT OF THE ANAEROBIC DIGESTION PROCESS OF PRIMARY AND SECONDARY SLUDGE BY THERMAL AND CHEMICAL PRE-TREATMENT

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## ABSTRACT

Anaerobic digestion is a technique for activated sludge stabilisation which is applied worldwide. The anaerobic digestion process follows four major steps: hydrolysis, acidogenesis, acetogenesis and methanogenesis with hydrolysis as the rate-limiting step of the overall process during sludge digestion. In the present work, chemical pre-treatment using NaOH has been applied to primary and secondary sludge from a municipal waste water treatment plant in order to improve the anaerobic digestion process. Thermal pre-treatment has also been studied applied to secondary sludge. Contact time (30-480 minutes) and NaOH concentration (0.05-0.3 mol/l) were the factors considered during the study of chemical treatment. Thermal treatment was applied to secondary sludge at different temperatures (110-134°C) and times (20-90 min) in an autoclave. The effectiveness of the two processes was evaluated by means of three parameters: the degree of disintegration using the chemical oxygen demand (% DD<sub>CO<sub>D</sub></sub>), the increment in soluble protein concentration (SPC, in percentage) and the increment of the relationship between filterable volatile solids and the total volatile solids (FVS/TVS, in percentage). Highest NaOH concentration (0.3 mol/l) and temperature (134°C) tested were found as the best conditions respectively for chemical and thermal treatment in the range of values studied. The time during which the treatment is applied also influences the process but in a less extend.

**Keywords:** *Anaerobic digestion, chemical pre-treatment, thermal pre-treatment, sludge hydrolysis.*

## INTRODUCTION

Anaerobic digestion is a sludge treatment used in a number of municipal wastewater treatment plants (MWWTP) to stabilize organic matter. Mass reduction, methane production and improved dewatering properties of the treated sludge are the main features of this process (Tiehm et al., 1997). The slow degradation of sewage sludge is a disadvantage of the anaerobic digestion process with residence times of about 20 days in conventional digesters. This fact implies the construction of large equipment and significant space requirements in MWWTP. The anaerobic digestion process follows four major steps: hydrolysis, acidogenesis, acetogenesis and methanogenesis with hydrolysis as the rate limiting step of the overall process (Gavala et al., 2003). During the hydrolysis step both solubilization of insoluble particulate matter and biological decomposition of organic polymers to monomers or dimers take place. Thermal, mechanic and chemical treatment have been investigated as a possible pre-treatment step to accelerate sludge hydrolysis. Different combinations of these treatments have been also studied (Chiu et al., 1997). The aim of the pre-treatment is disintegrate sludge solids to facilitate the release of cell components and other organic matter. This previous disintegration will result in an improvement of the overall process velocity and the degree of sludge degradation (Müller, 2000).

In the present work, chemical treatment using NaOH has been applied to primary and secondary sludge from a MWWTP in an attempt to reduce the mean hydraulic retention time of the existing digesters and to improve their production of methane. A thermal treatment has also been studied for secondary sludge. The experimental design technique has been applied in all cases.

## MATERIALS AND METHODS

Primary and secondary sludge from a biological MWWTP sited in La Llagosta (Barcelona, Spain) were used in the study. After collection, sludge was stored at 4°C until its utilization. Chemical treatment using different NaOH concentrations (ranging from 0.05 to 0.3 mol/l) and contact times (from 30 to 480 min) was applied to sludge in 1L beaker where 500 ml of sludge were mixed with 50 ml of NaOH solution at different concentrations. The mixture was kept under agitation during all the selected contact time for each concentration. An autoclave (P Selecta Autester - E) was used for thermal pre-treatment. Sludge was treated applying different combinations of temperature (from 110 to 134°C) and time (from 20 to 90 min). Temperature and time ranges were limited by the autoclave used. Volume of heated sludge was 500 ml.

Total solids (TS) and total volatile solids (TVS) from a sludge sample were determined before and after treatment following standard procedures (Standard Methods). Other sludge samples (before and after treatment) were centrifuged and filtered. The filtrate obtained was stored at 4°C until soluble chemical oxygen demand (SCOD) (Standard Methods, Closed Reflux, Colorimetric Method), Soluble Protein Concentration (SPC) (Bradford Method, Coomassie Protein Assay Reagent kit), total filterable solids (TFS) and filterable volatile solids (FVS) (Standard Methods) were determined. A spectrophotometer (Ultraspec 2100 pro UV/visible) was used to measure SCOD and SPC. Maximum SCOD was determined following the method proposed by Müller (2000).

## RESULTS AND DISCUSSION

The effectiveness of a chemical treatment using NaOH and a thermal treatment in an autoclave to disintegrate sludge solids has been studied by means of the experimental design technique. Contact time and NaOH concentration were the factors considered during the study of the chemical treatment. This treatment was investigated for primary and secondary sludge. Thermal treatment was only applied to secondary sludge. The factors considered in this case were time and temperature. The response variables were three: the increment in soluble protein concentration (SPC, expressed as a percentage respect to the concentration in the original sludge), the increment of the relationship between filterable volatile solids and total volatile solids (FVS/TVS, percentage respect to original sludge, too) and the degree of disintegration using the chemical oxygen demand (% DD<sub>COD</sub>). %DD<sub>COD</sub> was calculated as

$$\left( \text{SCOD}_{\text{after treatment}} - \text{SCOD}_{\text{before treatment}} \right) / \left( \text{SCOD}_{\text{maximum}} - \text{SCOD}_{\text{before treatment}} \right)$$

Nine experiments were undertaken for each treatment one of them in triplicate. The best operation conditions for each of the proposed treatments and the results obtained for the response variables under these conditions are summarized in table 1. As can be seen in table 1, highest NaOH concentration (0.3 mol/l) tested was found as the best for the chemical treatment of both

**Table 1.** Effectiveness of chemical (NaOH) and thermal treatment of primary and secondary sludge under the best conditions found for each of them.

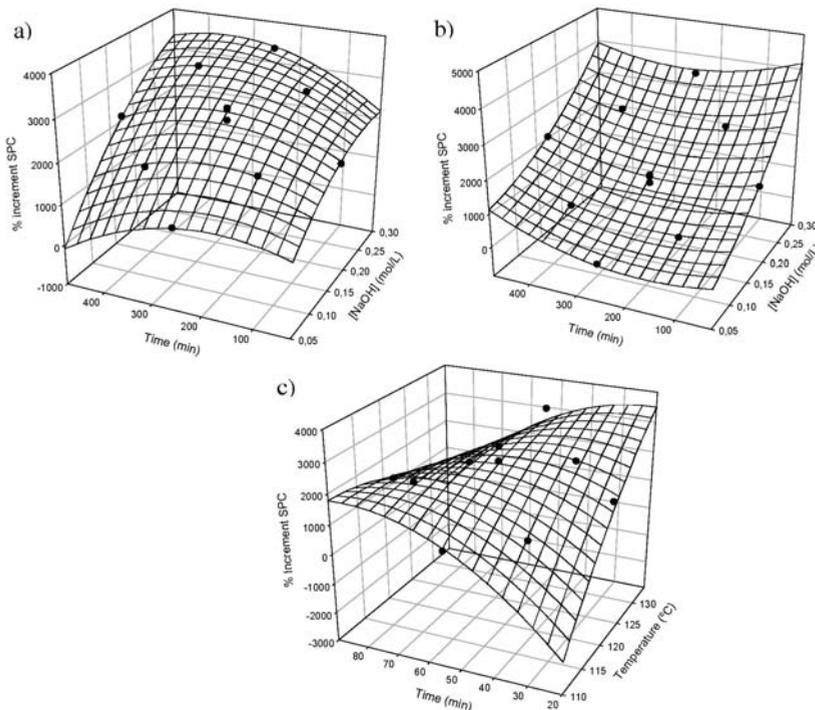
Pre-treatment	Type of sludge	Time (min)	[NaOH] (mol/l)	Temperature (°C)	FVS/TVS % incr.	SPC % incr.	DD <sub>COD</sub> %
Chemical	Primary	255	0.3	-	509	3365	52
Chemical	Secondary	255	0.3	-	686	3437	84
Thermal	Secondary	55	-	134	1104	2978	50

primary and secondary sludge while highest temperature studied (134°C) lead to best results in the thermal treatment of secondary sludge. Treatment time also influences the process but in a less extend. In terms of FVS/TVS increment the best results were obtained applying the thermal treatment while the chemical treatment was found as the best if values of  $DD_{COD}$  and SPC are considered. It should be highlighted that the values of the three variables considered increased if compared with those of the original sludge in all the experiments undertaken.

To ascertain the real contribution of each of the factors considered to the final values of the response variables, the results obtained for each set of experiments were adjusted to a polynomial function:

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_1^2 + b_4x_2^2 + b_5x_1x_2$$

where:  $y$  is the response variable ( $DD_{COD}$ , SPC and FVS/TVS),  $x_1$  is the concentration of NaOH (for the chemical treatment) or the temperature (for thermal treatment) and  $x_2$  is the contact time or the time during which sludge is under the desired temperature for chemical and thermal treatment respectively. Coefficients  $b_0$ ,  $b_1$ ,  $b_2$ ,  $b_3$ ,  $b_4$  and  $b_5$  where obtained for each treatment and each response variable. Table 2 summarises the values of  $b$  coefficients obtained and the regression coefficient  $r^2$  that indicates the wellness of the adjustment. Figure 1 represents the experimental values and surface responses for chemical treatment of primary (a) and secondary (b) sludge and for thermal treatment of secondary sludge (c) when the response variable considered is the increment in SPC.



**Figure 1.** Experimental results and response surfaces obtained for (a) primary and (b) secondary sludge chemical treatment and for (c) thermal treatment of secondary sludge. Response variable considered is the increment in SPC.

If coefficients for chemical treatment in table 2 are compared, it can be noted that those corresponding to factor NaOH concentration ( $b_1$  and  $b_3$ ) are some orders of magnitude bigger than those corresponding to factor time ( $b_2$  and  $b_4$ ) and to the interaction of the two factors ( $b_5$ ). These results point to NaOH concentration as the main factor influencing the effectiveness of the treatment in terms of SPC increment. This is valid for the results obtained for the treatment of both primary and secondary sludge. On the other hand, values of the coefficients for factors temperature and time in the case of the thermal treatment are similar in magnitude revealing a similar participation of time and temperature in the SPC increment obtained. However, temperature should be pointed as the most relevant factor. Quadratic and factors interaction terms present significantly lower coefficient values.

**Table 2.** Coefficients of the polynomial function derived of the adjustment of experimental values of SPC increment obtained under different conditions of time/temperature and time/NaOH concentration

Pre-treatment	Type of sludge	$b_0$	$b_1$	$b_2$	$b_3$	$b_4$	$b_5$	$r^2$
Chemical	Primary	7.71	15254.3	3.45	-29576.1	0.012	18.48	0.9864
Chemical	Secondary	17.07	5435.7	-2.87	29962.8	0.010	-9.66	0.9908
Thermal	Secondary	-91100	1175.7	640.57	-3.52	-0.83	-4.52	0.7223

## CONCLUSIONS

Solubilization of organic matter from activated sludge can be achieved by both thermal and chemical treatment. This fact is expected to accelerate the hydrolytic step of activated sludge anaerobic digestion. NaOH concentration is the main factor that influences the effectiveness of chemical treatment while time and temperature contribute in a similar extend to the sludge disintegration by thermal treatment.

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