



Alkaline thermal sludge hydrolysis

E. Neyens*, J. Baeyens, C. Creemers

*Department of Chemical Engineering, Katholieke Universiteit Leuven, de Croylaan 46,
3001 Heverlee, Belgium*

Received 27 June 2002; accepted 5 October 2002

Abstract

The waste activated sludge (WAS) treatment of wastewater produces excess sludge which needs further treatment prior to disposal or incineration. A reduction in the amount of excess sludge produced, and the increased dewaterability of the sludge are, therefore, subject of renewed attention and research. A lot of research covers the nature of the sludge solids and associated water. An improved dewaterability requires the disruption of the sludge cell structure. Previous investigations are reviewed in the paper. Thermal hydrolysis is recognized as having the best potential to meet the objectives and acid thermal hydrolysis is most frequently used, despite its serious drawbacks (corrosion, required post-neutralization, solubilization of heavy metals and phosphates, etc.). Alkaline thermal hydrolysis has been studied to a lesser extent, and is the subject of the detailed laboratory-scale research reported in this paper. After assessing the effect of monovalent/divalent cations (respectively, K^+/Na^+ and Ca^{2+}/Mg^{2+}) on the sludge dewaterability, only the use of Ca^{2+} appears to offer the best solution. The lesser effects of K^+ , Na^+ and Mg^{2+} confirm previous experimental findings.

As a result of the experimental investigations, it can be concluded that alkaline thermal hydrolysis using $Ca(OH)_2$ is efficient in reducing the residual sludge amounts and in improving the dewaterability. The objectives are fully met at a temperature of 100 °C; at a pH \approx 10 and for a 60-min reaction time, where all pathogens are moreover killed. Under these optimum conditions, the rate of mechanical dewatering increases (the capillary suction time (CST) value is decreased from approximately 34 s for the initial untreated sample to approximately 22 s for the hydrolyzed sludge sample) and the amount of DS to be dewatered is reduced to approximately 60% of the initial untreated amount. The DS-content of the dewatered cake will be increased from 28 (untreated) to 46%.

Finally, the mass and energy balances of a wastewater treatment plant with/without advanced sludge treatment (AST) are compared. The data clearly illustrate the benefits of using an alkaline AST-step in the system.

© 2002 Elsevier Science B.V. All rights reserved.

Keywords: Sludge; NaOH; KOH; $Ca(OH)_2$; $Mg(OH)_2$; Thermal hydrolysis; Reduction; Dewaterability

* Corresponding author. Tel.: +32-16-32366; fax: +32-16-322991.
E-mail address: elisabeth.neyens@cit.kuleuven.ac.be (E. Neyens).

Nomenclature

IE	equivalent-inhabitant
COD	chemical oxygen demand (mg/l)
CST	capillary suction time (s)
DS	dry solids content (%)
MDS	mineral part of the DS (%)
ODS	organic part of the DS (%)
T	temperature (°C)
P	pressure (Pa)
PE	polyelectrolyte
blanco	untreated, reference sample
ECP	extracellular polymers
AST	advanced sludge treatment
SRF	specific resistance to filtration
WAS	waste activated sludge

1. Introduction

As a result of the wide application and utilization of the WAS-process, excess sludge presents a serious disposal problem: this excess sludge is an inevitable drawback inherent to the process, despite sludge minimization in e.g. extended aeration processes. Many efforts have been devoted to reduce the excess sludge by treatments such as digestion and dewatering.

Today the most challenging aspect of the sludge treatment is to remove as much as possible of its water portion so that sludge behaves as a solid. It is well-known that activated sludge is very difficult to dewater. A lot of work has been done on understanding the nature of water in sludge [1–3]. Biological sludge contains various fractions of water, including free water, interstitial water, vicinal water and water of hydration, associated with its solids [3]. These water fractions affect the liquid–solid separation of sludge. The big portion of sludge solids are organic in nature and a complicated mixture of bacteria, viruses, protozoa and other micro-organisms which exist in either unicellular or in floc forms. A considerable amount of sludge water is trapped either inside these sludge micro-organisms or within the floc structure. It is impossible to extract cell-associated water by conventional dewatering processes, which can only remove free water that is not attached to the sludge solids and a part of interstitial water that is trapped in the crevices and interstitial spaces of the flocs. Release of interstitial water held inside the cell structure involves disruption of sludge cells and this does not occur during conventional dewatering.

Several disintegration methods have been investigated [4]:

- heat treatment, in the temperature range from 40 to 180 °C;
- chemical treatment using ozone, acids or alkali;
- mechanical disintegration using ultrasounds, mills, homogenisers and others;

Table 1
Literature data on the results of several thermochemical pre-treatment processes

Reference	Main conclusions			
	Reagent	Temperature (°C)	Time	Results
[5,6]	H ₂ SO ₄	121	5 h	75–80% TSS solubilized
[7]	H ₂ SO ₄	120	5 h	60–70% TSS solubilized
[8]	H ₂ SO ₄	150–200	15–40 min	Enhanced conditioning
	KOH	150–200	15–40 min	Hampered conditioning
[9,10]	HCl	175–200	1 h	52–54% COD solubilized
	NaOH	175–200	1 h	54–55% COD solubilized
	Ca(OH) ₂	175	1 h	40% COD solubilized
[11]	Base, H ₂ SO ₄	60–90	1–20 min	Significantly improved dewaterability
[12]	NaOH	20–40	0.5–24 h	45% of COD solubilized
[13]				Gas production increased by 112% over control levels
[14]	H ₂ SO ₄ , base	165	75 min	Filter cakes >65% DS
[15]	H ₂ SO ₄	150–160	1 h	Significantly improved dewaterability
[16]	H ₂ SO ₄	90	1 h	50–60% TSS solubilized Filter cakes >50% DS
[17]	NaOH	95	1 h	55–65% Dry organic matter solubilized Filter cakes >43% DS
[18]	NaOH		45 min	Significantly improved dewaterability
[19]	H ₂ SO ₄	120–160	1 h	Significantly improved dewaterability Filter cakes >60% DS

- freezing and thawing;
- biological hydrolysis with or without enzyme addition.

The influence of the thermochemical pre-treatment on several sludge characteristics (e.g. dewaterability, COD-solubilization, percentage DS in filter cake, . . . , etc.) has been extensively studied experimentally and is reviewed as shown in Table 1.

The reduction of the amount of sludge and/or its improved dewaterability by alkaline hydrolysis is less documented than acid hydrolysis and literature references are scarce.

Alkali treatment is a harsh method. At extremely high pH values of medium, the cell loses its viability, it cannot maintain an appropriate turgor pressure and disrupts. Alkali added to the cell suspension react with the cell walls in several ways, including the saponification of lipids in the cell walls, which leads to solubilization of membrane. The high alkali concentrations cause much degradation. Disruption of sludge cells leads to leakage of intracellular material out of the cell. On the other hand, as Katsiris and Kouzeli-Katsiri [1] stated when the pH of sludge samples increased, the bacterial surfaces become increasingly negatively charged. This creates high electrostatic repulsion which causes desorption of some part of extracellular polymers.

Cell disruption affects the liquid–solid separation properties of sludge in different ways, not only by the release of interstitial water trapped inside the sludge cells but also by

promotion of the flocculation. Vallom and McLoughlin [20] demonstrated that the lysis of cells normally causes the release of polymers into the medium. These polymers include proteins, RNA, DNA, carbohydrate, etc., which are all high molecular weight polymeric materials that possibly act as polyelectrolytes and thereby promote flocculation.

In the past, a lot of research has studied the influence of cations on the settling and dewatering of activated sludge [21–23]. An important part of this research is the relation between concentrations and ratios of monovalent:divalent cations and settling and dewaterability of activated sludge.

The present study is inspired by these findings but considers other perspectives. First of all, adding cations is still an important factor but these cations are added as hydroxybases to increase the pH of the sludge. The influence of pH (and also temperature) plays the dominant role in this study, rather than the concentrations of cations. Secondly, in the previous research, cations were added to the feed of reactors in which the activated sludge process was simulated, while in our study hydroxybases are batch added to the thickened sludge.

The paper, hence, describes the experimental results obtained for thermal alkaline hydrolysis of thickened sludge (6% DS content). NaOH, KOH, Ca(OH)₂ and Mg(OH)₂ are compared with respect to their efficiency to hydrolyze and dewater the sludge samples. The effect of temperature and pH are examined.

2. Materials and methods

2.1. Materials

Sludge samples were obtained from the thickener underflow (approximately 6% DS) at a municipal sewage treatment plant in Kessel-Lo (Belgium). The location of the hydrolysis pre-treatment in the wastewater treatment process is illustrated in Fig. 1.

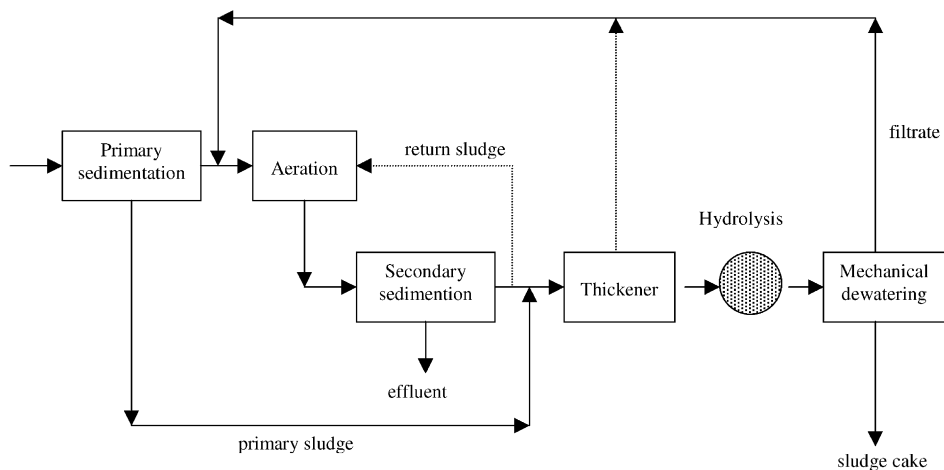


Fig. 1. Location of the hydrolysis treatment in the wastewater treatment process.

The ODS-content varied between 50 and 60%. The density of the untreated (6% DS) sludge was 1.048 kg/l.

2.2. Experimental set-up and procedure

Alkaline hydrolysis of sludge implies a treatment at moderate to high temperature and at high value of pH. The experiments were carried out in a reactor, constructed as a pressure vessel with a heating shell. The temperature of the sludge–alkaline mix was kept constant by setting the temperature of the thermal fluid, circulating through the outer heating shell of the reactor. Fig. 2 illustrates the experimental set-up.

Samples of 200–300 ml of sludge were batch-treated at different but fixed operating conditions.

The samples, prior to any treatment, will be referred to as ‘blanco’-sample.

Each experiment was repeated three times and average values of the results are given in the text. The error on the individual experiments was within 10%.

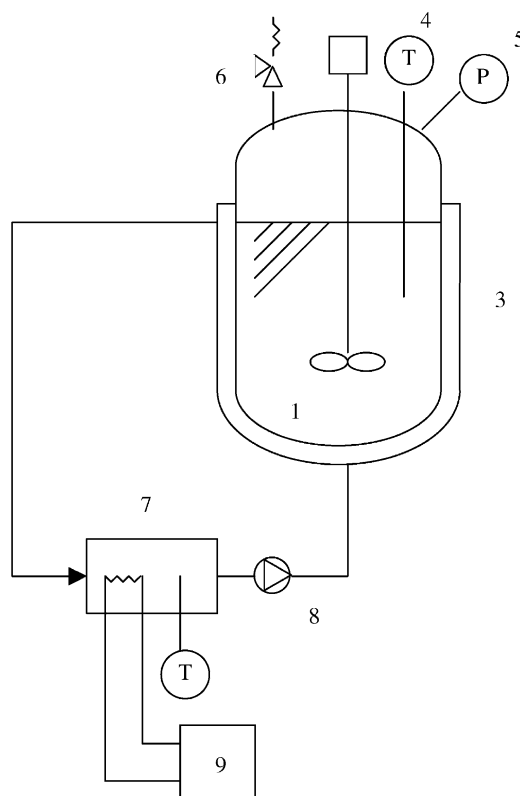


Fig. 2. Experimental set-up: reactor vessel (1); with mixer (2); heating shell (3); and temperature (4); and pressure probe (5); pressure relief valve (6); thermal fluid circuit with thermal fluid vessel (7); circulating pump (8); and voltage regulator (9), the applied voltage was controlled by T(4).

2.3. Conditioning

Cationic polymer (Ciba® ZETAG 7878 FS40) was used for conditioning and dewatering of thickened sludge from the reactor. Polymer solutions were made to a final concentration of 0.1% by mixing concentrated polymer with distilled water for 30 min. During conditioning the polymer was added to a 200 ml sludge sample and mixed for 30 s at a mixing speed of 200 rpm. After mixing, the CST (using the apparatus and procedure described in [24]) was measured and the optimum dosage was considered as the dose which resulted in the minimum CST. A CST-value of approximately 20 s is recognized as corresponding to a sludge of good dewatering characteristics [25].

2.4. Dewatering

Sludge was also dewatered by using a vacuum-assisted Buchner filtration at a vacuum pressure of 38 cm mercury, and assessed for a 100 ml sample during a set time of 10 min. The resulting filter cake was used to determine both the approximate cake volume and the ODS-content of the cake.

The same PE-dosage was used as for the CST-tests.

2.5. Relevant properties

The ‘blanco’ reference sludge sample contains suspended and dissolved matter of organic and mineral nature.

Total dry solids are measured after evaporation of H₂O (105 °C) [26]. Further calcination at 605 °C drives off the organic matter (ODS) and the mineral content (MDS) is weighed as residual ash [26]. The same procedure applies to determine the DS/ODS/MDS-content of mechanically dewatered sludge (filter cake).

3. Results

To fully determine the most appropriate operating conditions when using alkaline hydrolysis, major governing parameters were combined in experiments to assess the influence of: the kind of base used, the time of reaction, the temperature and the pH. All results are related to the equivalent property for the ‘blanco’-untreated sludge sample.

The results will be separately described and discussed, although the overall results will lead to general conclusions and recommendations.

3.1. Comparison of NaOH, KOH, Ca(OH)₂ and Mg(OH)₂ with respect to their efficiency to reduce the residual amounts of sludge

In order to investigate the effect of the kind of base used, the residual volume of the sludge phase (filter cake phase) as measured from the 10-min vacuum-filtration is determined.

Experiments were conducted at $T = 120\text{ °C}$ ($P = 1.2\text{ bar}$) and $\text{pH} \approx 10$. The amounts of base added to obtain pH 10 are indicated in Fig. 3.

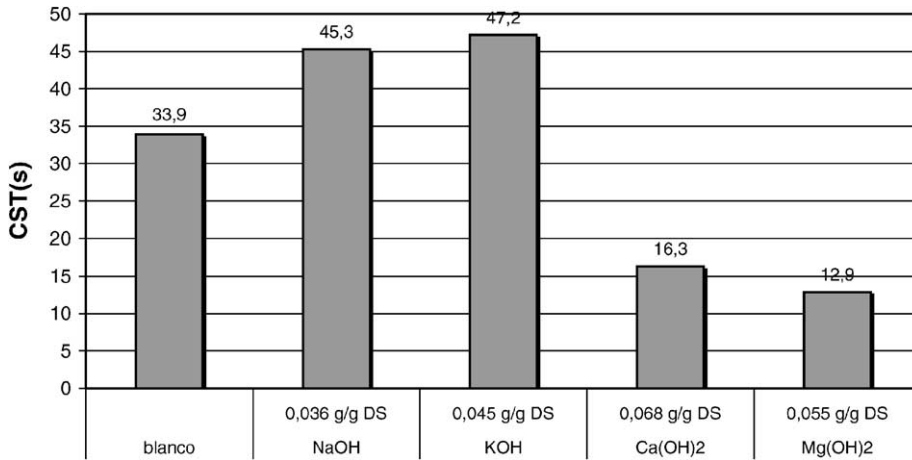


Fig. 3. CST: comparison of NaOH, KOH, Ca(OH)₂ and Mg(OH)₂ (reaction conditions: pH 10, 120 °C).

Figs. 3 and 4 show that only alkaline thermal hydrolysis using Ca(OH)₂ and Mg(OH)₂ is efficient in reducing the CST-value and the residual sludge amounts (as measured from the 10-min vacuum-filtration), indicating a better dewaterability.

3.1.1. Effect of adding divalent cations

Higgins and Novak [21] showed that the settling and dewatering properties of the activated sludge systems were dependent on both the concentrations and ratios of cations added. High divalent:monovalent cation concentrations ratios improved settling and dewatering. Therefore, as shown in Figs. 3 and 4, adding Ca(OH)₂ or Mg(OH)₂ results in

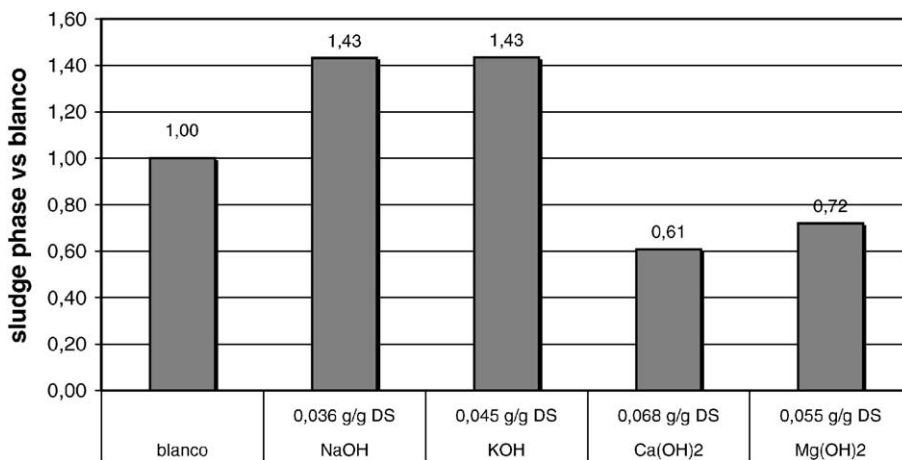


Fig. 4. Sludge phase: comparison of NaOH, KOH, Ca(OH)₂ and Mg(OH)₂ (reaction conditions: pH 10, 120 °C).

increased dewaterability. The residual volume of the cake phase as measured from the 10-min vacuum-filtration (Fig. 4) shows that adding $\text{Ca}(\text{OH})_2$ reduces the sludge volume by 40%. The filtration rate, as determined from CST-measurements, follows the same trend (Fig. 3). The lower CST-values correspond with a higher filtration rate.

Several researchers have suggested cations aid in flocculation by bridging negative sites on exocellular biopolymers which promotes an increase in the floc size, floc density and floc resistance to shear [27–29]. This model proposes that divalent cations act as a bridge between negatively charged sites on exocellular biopolymer. Therefore, improvements in settling and dewatering due to increased particle sizes should correlate with an increase in the bound exocellular biopolymer content involved in the aggregation process. Higgins and Novak [21] showed that divalent cations act to bind protein and not polysaccharide within the floc in these systems. As a result, the improvements in settling and dewatering are well correlated to the bound protein content in addition to the increase in divalent cation concentration. These results suggest that protein is an important biopolymer component in the flocculation process.

Adding $\text{Ca}(\text{OH})_2$ gives better results than adding $\text{Mg}(\text{OH})_2$ (Figs. 3 and 4). This finding can also be explained by previous research. Forster and Lewin [30] revealed that Ca^{2+} had a higher binding capacity than Mg^{2+} on ECP in activated sludge. Eriksson and Alm [29] confirmed this result and showed that Ca^{2+} is measured in higher amounts than Mg^{2+} , which is related to its higher affinity for ECP.

3.1.2. Effect of adding monovalent cations

- The use of NaOH increases the CST-value and the volume of the cake (Figs. 3 and 4). This is in-line with the literature observations. Bruus et al. [28] and Novak and Randall [31] revealed that excess sodium in activated sludge systems has been shown to result in deterioration in the settling and dewatering properties of activated sludge. Higgins and Novak [21] showed that addition of sodium resulted in poor settling and dewatering properties when the monovalent:divalent cation ratio exceeded approximately 2 to 1, expressed on an equivalent basis. It was thought that the poor settling and dewatering that occurred with high sodium concentrations was a result of ion-exchange processes in which divalent cations were displaced from within the floc by the sodium. This theory explains the fact that settling and dewatering should be improved by a reduction in the monovalent:divalent cation ratio.

Monovalent cations reduce the strength of the bonds, and this leads to a loose structure, often decreasing floc density and floc resistance to shear [21,30,32].

Novak et al. [33] showed another effect of Na^+ addition, i.e. a marked deterioration in floc strength. The consequence of this is that flocs can be expected to disintegrate when mixed liquor and waste solids are pumped, mixed or dewatered. This may explain some of the problems with solids penetrating belts during industrial dewatering, as a result of sodium hydroxide addition for pH control.

- Adding KOH to the thickened sludge also results in higher CST-values and an increased volume of sludge cake (Figs. 3 and 4).

Murthy and Novak [23] showed that an increase in potassium ions was correlated with an increase in soluble protein in the activated sludge and an increase in soluble polymers is

associated with a deterioration in dewatering properties. Their study revealed that potassium was the only ion that was positively correlated with slime protein and polysaccharide. Sodium ions were not positively correlated with slime and soluble proteins, although this cation was also associated with a deterioration in dewatering properties. Following this observation, it was anticipated that potassium would not physico-chemically interact with activated sludge flocs in the same manner as does sodium. The interaction of potassium (unlike sodium) with activated sludge flocs is not completely explained by simple charge competition in the divalent charge bridging model. In general, excess sodium always produced poorly settling sludges, poor dewatering and weak flocs. Excess potassium produced poor dewatering but flocs that were resistant to shear and settled well. The effect the ion has on improvements in settling property and floc strength needs further investigation, as it is anomalous to the trend that would be explained by the divalent charge bridging theory and as expressed by sodium ion concentration.

Novak et al. [33] believed that the distinctly different results in changes in floc stability and settling resulting from Na^+ and K^+ addition reflect fundamental differences in the role of each of these ions, and this could result from the physiologically different roles each plays in the bacterial metabolism. Potassium is a more critical cation for bacteria than Na^+ because it is a key element required by some enzymes involved in protein synthesis. Therefore, it might be expected that differences will exist in the quality and character of the proteins associated with the bioflocs.

This fact has an important implication. The ion-bridging model, as stated before, assumes an indifferent role for cations, with only the charge and ionic volume being of importance. The present results confirm that Na^+ and K^+ have different physiological roles within activated sludge systems. Consequently, using the monovalent:divalent cations concentration ratio alone without consideration of these other factors, could lead to serious errors in predicting solids dewatering performances.

The previous experimental findings indicate that adding $\text{Ca}(\text{OH})_2$ seems most appropriate to achieve the objectives. The alkaline thermal hydrolysis using $\text{Ca}(\text{OH})_2$ is, therefore, studied in more detail in the following experiments.

3.2. Time of reaction

The required time of reaction was determined at various operating temperatures and different pH-values. The effect of the pH will be discussed further in the text. Results are illustrated in Table 2 for hydrolysis at 120°C (1.2 bar) and $\text{pH} \approx 10$, i.e. after addition of $0.068 \text{ g Ca}(\text{OH})_2/\text{g DS}$.

Table 2
Illustration of the results concerning the required time of reaction

Time (min)	CST (s)	Residual sludge volume after filtration (ml/100 ml) vs. blanco	Percentage DS in sludge phase
30	44.7	0.75	42.36
60	16.3	0.61	48.92
90	22.5	0.63	46.25

Table 3
Grams of $\text{Ca}(\text{OH})_2$ per gram of DS needed to obtain various pH-values

pH	$\text{Ca}(\text{OH})_2$ (g)/DS (g)
8	0.036
10	0.068
12	0.090

A reaction time of 60 min seems most appropriate: vacuum-filtration proceeds to the lowest residual sludge volume, the CST-value is the lowest and the DS-content of the sludge phase is the highest. The slightly higher value of CST for longer reaction times is not understood and currently subject to additional tests.

These findings were confirmed for all combinations of T and pH. A reaction time of 60 min was, therefore, used as standard in all the further investigations.

3.3. Effect of pH

In order to investigate the effect of the pH, experiments were conducted at a temperature of 120 °C during 60 min. Table 3 indicates the amounts of $\text{Ca}(\text{OH})_2$ used.

The results can be summarized as follows:

- (i) The total amount of DS and ODS hardly changes, although there is a slight increase in DS-content with increasing pH due to an increase in the amount of $\text{Ca}(\text{OH})_2$ added. At a pH of 10–12, this increase is still limited to 5%.
- (ii) Conditioning and dewatering experiments were performed to determine the optimum polymer dose required for the different pH conditions. In general, as the pH increased, the optimal polymer dose for conditioning decreased, as shown in Fig. 5. Novak et al.

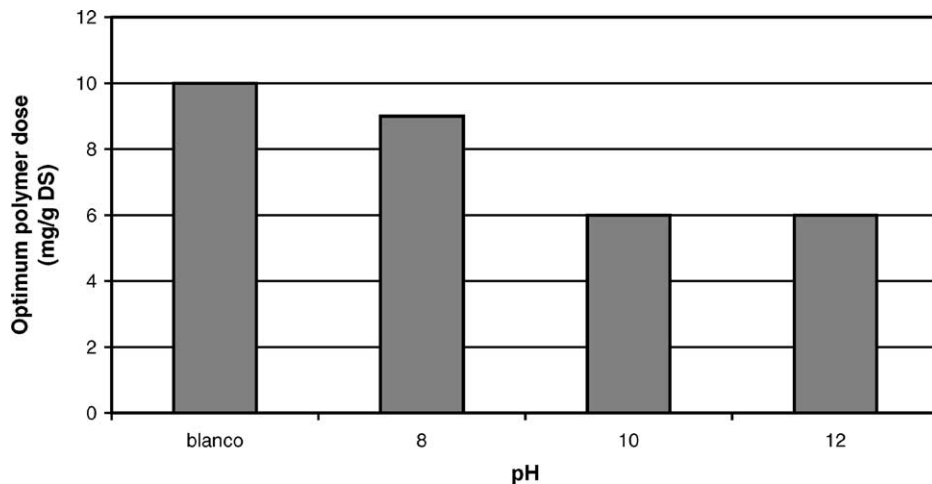


Fig. 5. Optimum polymer dose at different pH.

[34] reported that the polymer dose required for conditioning was a function of the concentration of colloidal particles in the supernatant of a biological suspension. Also, Karr and Keinath [35] reported that ‘supracolloidal’ particles in the size range of 1–100 μm had the greatest effect on the dewaterability of sludges and as the concentration of the particles in this size range increased, dewaterability decreased. In addition to a decrease in the number of small particles, the increase in calcium ions also decreased the charge of the particles, measured by the zeta potential, which could decrease the polymer dose for conditioning. Novak et al. [34] reported that charge neutralization was an important mechanism in polymer conditioning of activated sludge.

Murthy and Novak [36] showed that an increase in the required polymer conditioning implies a greater number of negative charged sites available in the floc and an increase in negative biological colloids (biocolloids) in solution. Greater soluble effluent COD should result from the greater amount of anionic biocolloids in solution.

Therefore, the decrease in polymer demand is thought to be associated with a change in particle size distribution and a decrease in the particle charge resulting from cation addition.

- (iii) The residual volume of the cake phase as measured from the 10-min vacuum-filtration, decreases with increasing pH indicating a better dewaterability, as illustrated in Fig. 6.

A pH of 10 reduces the sludge volume by 40%, indicating a high DS-concentration. A further increase in pH does not significantly improve the results.

These results are in-line with previous research done by Higgins and Novak [21] who stated that increasing amounts of divalent cations, increased the dewaterability of the sludge.

- (iv) The filtration rate, as determined from CST-measurements, follows the same trend (Fig. 7). The lower CST-values correspond with a higher filtration rate. Higgins and

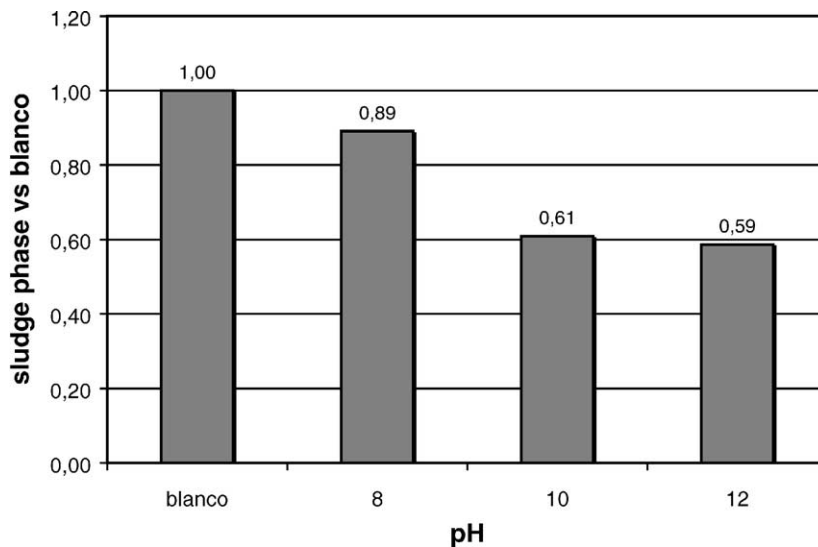


Fig. 6. Residual volume of the sludge phase at different pH.

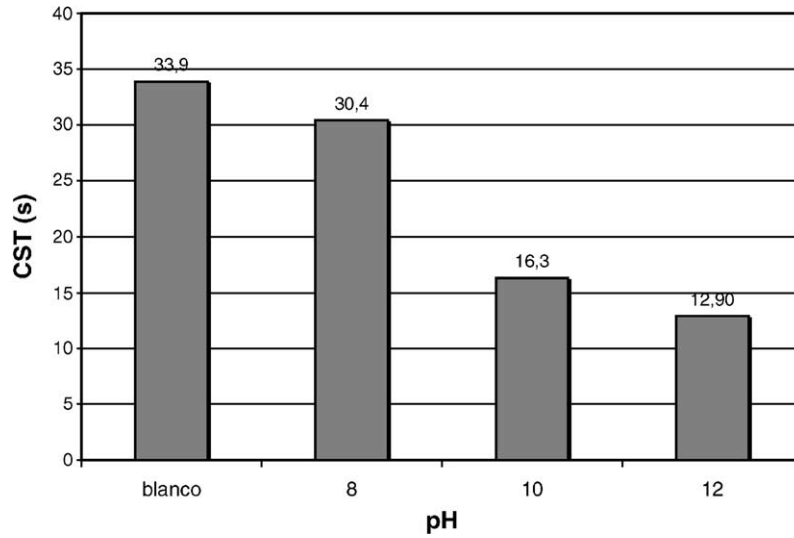


Fig. 7. CST-values of hydrolyzed samples at different pH.

Novak [21] also showed better dewatering properties, measured by specific resistance to filtration (SRF) and CST with increasing amounts of divalent cations.

The filtration and subsequent analysis of the hydrolyzed samples reveal that the DS-content in the cake is almost 50% at pHs of 10 and 12, against 28% DS for the 'blanco' (Fig. 8).

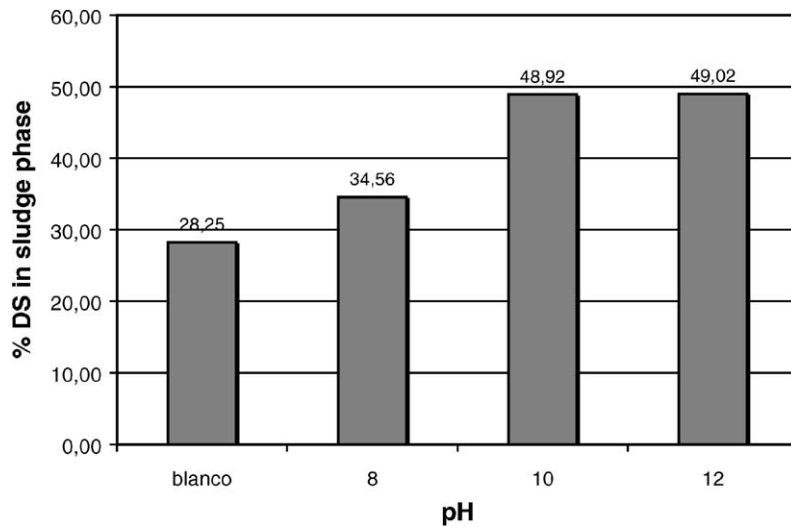


Fig. 8. Percentage DS in the sludge cake at different pH.

Previous research done by Higgins and Novak [21] revealed an increased cake solids content as the calcium concentration was increased. Forster and Lewin [30] reported that calcium ions added to activated sludge decreased the bound water content of the sludge, which would increase floc density and cake solids. In addition, increasing the divalent cation concentration increased the bound biopolymer concentration and the floc strength. This suggests that the divalent cations create a tighter bound network of biopolymer that was more resistant to shear. The tighter bound network of exocellular polymers would decrease the amount of bound and/or inter-floc water, forming a denser floc with increased cake solids.

Erdinçler and Vesilind [18] gave another explanation for the increased compactibility of alkali treated samples. When alkali treatment was applied to sludge samples, a cell disruption was observed leading to a considerable increase in the protein levels of the samples. This increase can be explained by desorption of extracellular polymers by high electrostatic repulsion (caused by the increased pH [1]), release of proteins due to detergent solubilization and leakage of proteinaceous intracellular material. These dissolved extracellular polymers and intracellular material are all high molecular weight polymeric materials that possibly act as polyelectrolytes and thereby promote flocculation [20] and floc compactibility.

- (v) The effect of the alkaline hydrolysis on the properties of the water phase is reflected in the release of DS and ODS into the water phase.

DS and ODS are released into the water phase to an increasing extent with increasing pH (Fig. 9): at pH 10–12, DS and ODS concentrations are a three–four-folds of the concentrations in their ‘blanco’ water phase.

The present findings partly contradict previous results by Murthy and Novak [36] who noticed an increase in the concentration of solution biological polymers

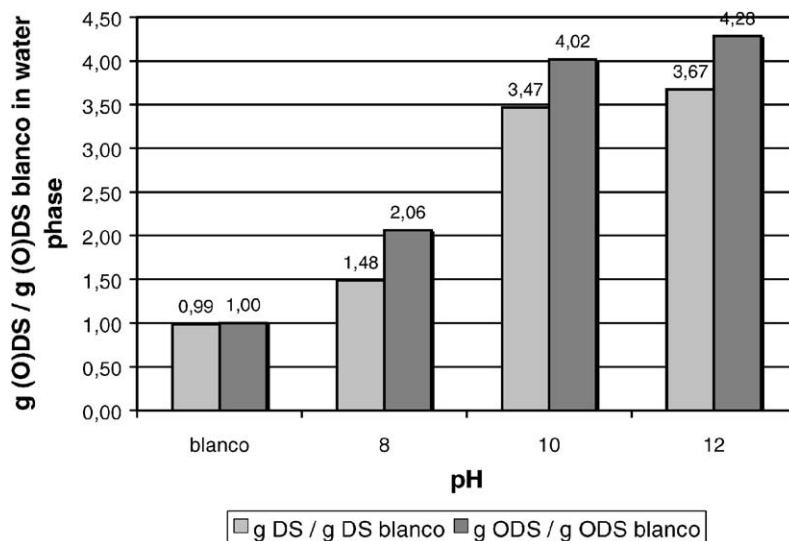


Fig. 9. DS and ODS in the water phase at different pH.

(biopolymers) in the effluent for monovalent cations only, whereas divalent cations tend to retain the biopolymers in the floc. $\text{Ca}(\text{OH})_2$ clearly releases biopolymers which affect the COD-value.

As a result of the previous experimental findings, it can be concluded that the objectives are already achieved at a pH of 10. An additional increase of the pH-value does not significantly improve the results obtained. The following sensitivity analysis with respect to temperature was, hence, performed at a pH = 10.

3.4. The temperature of hydrolysis

Heating of sludge causes hydration of proteins, polysaccharides, lipids and other intracellular macromolecules secreted from disrupted sludge cells [18]. Consequently the vicinal water content of sludge increased while the overall free water content increased by release of some interstitial water originally trapped inside sludge cells. It seems that although some extra water binding surfaces are created, leading to an increase in vicinal water content of sludge samples, the release of interstitial water increased the overall easily removable water content of the sludge samples.

To assess the effect of the reaction temperature, a series of 60 min-tests was carried out at pH \approx 10.

Again results are compared with the findings for the ‘blanco’ untreated sample.

- (i) The total amount of DS and ODS slightly increases to a maximum by approximately 5% when compared with the DS- and ODS-concentrations in the ‘blanco’ sludge samples. This increase in DS-content is due to adding $\text{Ca}(\text{OH})_2$.
- (ii) The residual volume of the sludge phase decreases with increasing temperature (Fig. 10) and, so does the CST-value (Fig. 11). Low residual volumes and low CST-values

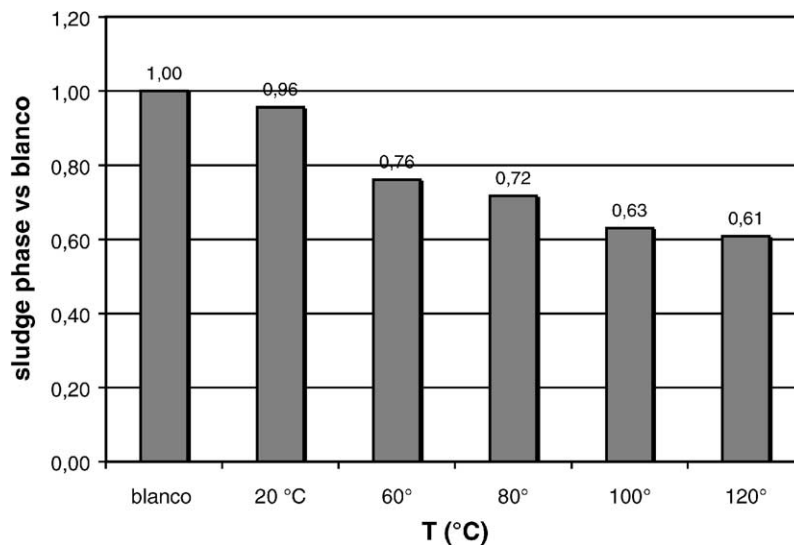


Fig. 10. Residual volume of the sludge phase at different temperature.

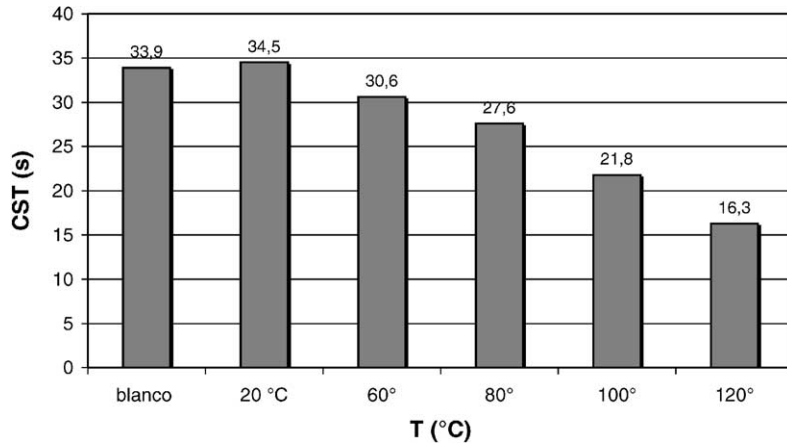


Fig. 11. CST-values of hydrolyzed samples at different temperature.

indicate high dewaterability and high dewatering rate. The amount of sludge to be dewatered at a temperature of 100 °C is reduced to approximately 60% of the initial untreated amount and the CST-value is decreased from approximately 34 s (initial untreated) to approximately 22 s. Both findings stress the importance of working at temperatures around 100 °C.

Worth mentioning is the fact that alkaline hydrolysis without heating the sludge already results in a reduction of the residual sludge volume by approximately 10%.

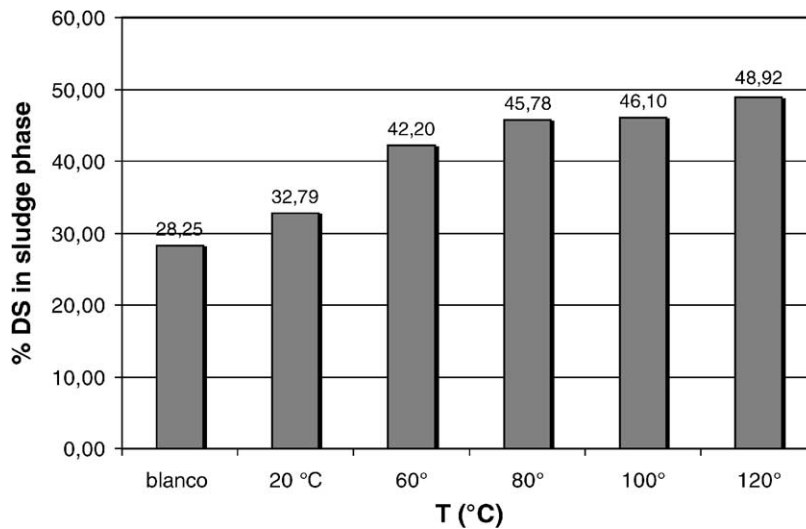


Fig. 12. Percentage DS in the sludge filtration cake at different temperature.

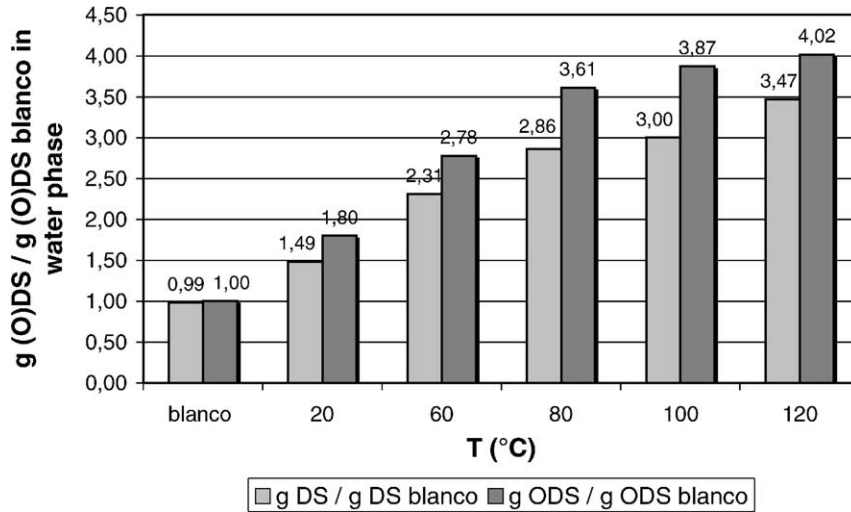


Fig. 13. DS and ODS in the water phase at different temperature.

The improvement in compactibility of the samples with increasing temperature can be explained by the hydrolyzation of exocellular and intracellular materials leading to destruction of colloidal properties of these macromolecules.

It has to be remarked that the temperatures indicated as shown in Figs. 10 and 11 are reaction temperatures, not dewatering temperatures: the CST-test and filtration were performed after sludge cooling to ambient temperature. The effect of the dewatering temperature on the dewaterability is not studied.

- (iii) The DS-content in the cake increases with increasing temperature, as illustrated in Fig. 12. At 120 °C, the DS-content of the sludge phase is 20% higher than that of the 'blanco' sludge phase.
- (iv) The effect of alkaline thermal hydrolysis on the properties of the water phase is reflected in the release of DS and ODS into the water phase. At elevated temperatures, DS and ODS are released into the water phase, where their concentrations are considerably higher than the concentrations in the 'blanco' water phase (Fig. 13).

Ca(OH)₂ also efficiently precipitates heavy metals and phosphates from the water phase.

4. Discussion and recommendations

As a result of the experimental investigations, it can be concluded that alkaline thermal hydrolysis using Ca(OH)₂ is efficient in reducing the residual sludge amounts and in improving the dewaterability. The objectives are fully met at a temperature of 100 °C; at a pH ≈ 10 and for a 60-min reaction time, where all pathogens are moreover killed.

Under these optimum conditions, the rate of mechanical dewatering increases (the CST-value is decreased from approximately 34 s for the initial untreated sample to approximately

22 s for the hydrolyzed sludge sample) and the amount of DS to be dewatered is reduced to approximately 60% of the initial untreated amount. The DS-content of the dewatered cake will be increased from 28 (untreated) to 46%.

5. The economy of using alkaline thermal hydrolysis

Based upon these findings, the initial balance of a traditional wastewater treatment facility can be adapted to integrate the results of the experiments. Mass and energy balances of a wastewater treatment implementing the technique described previously are studied for the case of a daily sludge production of 60 g DS per equivalent-inhabitant (IE). Concluding results are illustrated in Fig. 14 and Table 4 for alkaline thermal hydrolysis at the proposed optimum conditions. The data clearly illustrate the benefits of implementing the AST.

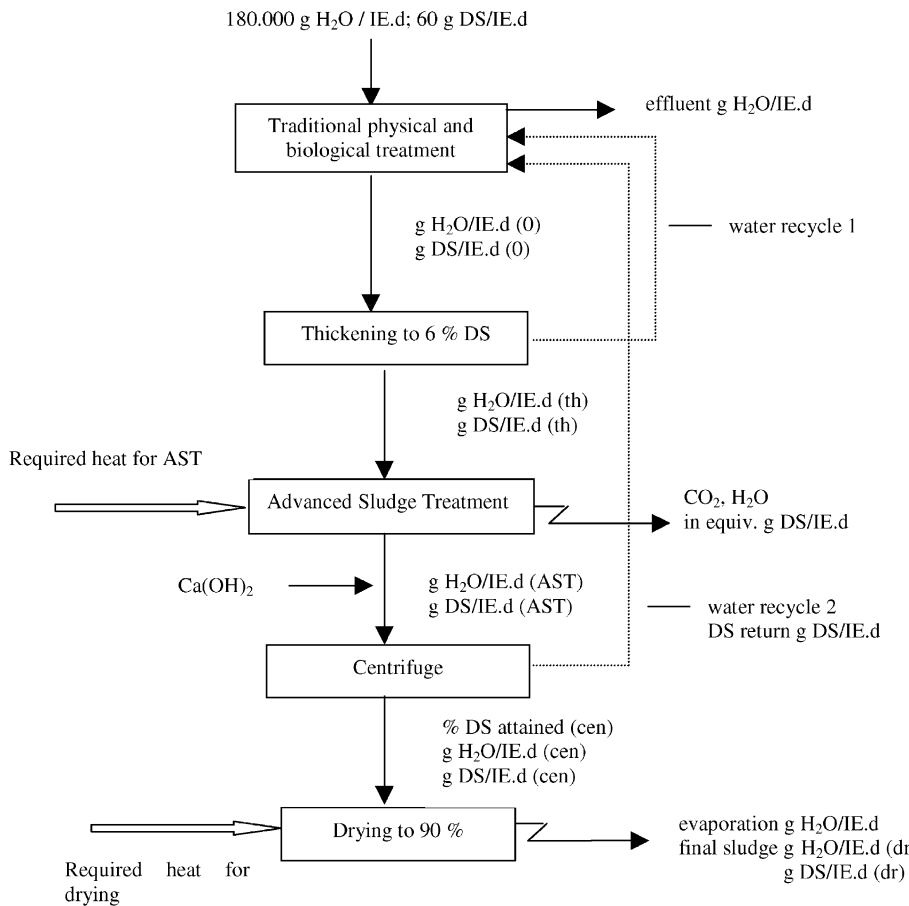


Fig. 14. Mass and energy balances of a wastewater treatment implementing advanced sludge treatment (coefficients are given in Table 4) prior to mechanical dewatering and sludge drying.

Table 4
Coefficients of mass and energy balances of Fig. 14

	Traditional	Alkaline hydrolysis
Effluent (H ₂ O (g)/IE per day)	179.820	179.820
H ₂ O (g)/IE per day (0)	3.940	3.940
DS (g)/IE per day (0)	60	60
Water recycle 1 (H ₂ O (g)/IE per day)	3.000	3.000
H ₂ O (g)/IE per day (th)	940	940
DS (g)/IE per day (th)	60	60
H ₂ O (g)/IE per day (AST)	–	940
DS (g)/IE per day (AST)	–	60
Required heat for AST (kJ/IE per day)	–	79
Ca(OH) ₂ (g/IE per day)	–	4
Water recycle 2 (H ₂ O (g)/IE per day)	760	873
DS return (g DS/IE per day)	–	3
Percentage DS attained in filterpress	28	46
H ₂ O (g)/IE per day (cen)	154	67
DS (g)/IE per day (cen)	60	61
Required heat for drying (kJ/IE per day)	388	135
Evaporation (H ₂ O (g)/IE per day)	147.3	51
Final sludge (H ₂ O (g)/IE per day)	6.7	6
Final sludge (DS (g)/IE per day)	60	61

Alkaline thermal hydrolysis does not reduce the amount of DS/IE per day (from 60 to 61 g DS/IE per day) but the hydrolyzed sludge can be mechanically dewatered to higher percentage of DS-values. The percentage DS of the sludge cake is 46%, which is very high when compared to the traditional 28%, resulting in a significantly reduced energy for subsequent drying as compared with the traditional treatment (135 kJ/IE per day compared to 388 kJ/IE per day). The heat required for the alkaline thermal hydrolysis (with partial recovery of the heat to the influent of the subsequent batch) is 79 kJ/IE per day. There is still a net heat saving of over 50%.

Contrary to acid hydrolysis, an alkaline AST avoids acid-corrosion effects, thus allowing the use of common construction steel.

6. Conclusions

The costs related to sludge treatment are constantly increasing. The reduction of the amount of sludge produced and an increased dewaterability are hence of paramount importance. The use of advanced treatment technologies is hence subject of numerous investigations. The current paper described the results of detailed investigations with respect to alkaline hydrolysis of thickened sludge (6% DS content). Experiments were carried out to determine the effect of hydrolysis. Results confirm that a substantial reduction of DS is achieved with better dewaterability when sludge is hydrolyzed using Ca(OH)₂. The percentage DS of the sludge cake is 46%, compared to the traditional 28%, resulting in a significantly reduced energy for subsequent drying as compared with the traditional treatment

(135 kJ/IE per day compared to 388 kJ/IE per day). An alkaline AST avoids acid-corrosion effects, thus allowing the use of common construction steel.

Pilot-scale experiments at 100–120 °C and pH = 10 are currently being performed to confirm the laboratory results and to take into consideration some process characteristics so that the alkaline AST method can be further technically and economically assessed.

Acknowledgements

The authors thank Aquafin (Aartselaar, Belgium) for making sludge samples available and for sponsoring the research on Advanced Sludge Techniques.

References

- [1] N. Katsiris, A. Kouzeli-Katsiri, Bound water content of biological sludges in relation to filtration and dewatering, *Water Res.* 21 (11) (1987) 1319–1327.
- [2] K.R. Tsang, P.A. Vesilind, Moisture distribution in sludges, *Water Sci. Technol.* 22 (12) (1990) 135–142.
- [3] P.A. Vesilind, The role of water in sludge dewatering, *Water Environ. Res.* 66 (1) (1994) 4–10.
- [4] J.A. Müller, Prospects and problems of sludge pre-treatment processes, *Water Sci. Technol.* 44 (10) (2001) 121–128.
- [5] P.I. Yang, A.F. Gaudy, Control of biological solids concentration in extended aeration, *J. WPCF* 46 (1974) 543–553.
- [6] P.I. Yang, A.F. Gaudy, Nitrogen metabolism in extended aeration processes operated with and without hydrolytic pre-treatment of portions of the sludge, *Biotechnol. Bioeng.* 16 (1974) 1–20.
- [7] T. Singh, D.W. Patterson, Improvement of the aerobic sludge digestion process efficiency, *J. WPCF* 46 (1974) 102–112.
- [8] J.G. Everett, The effect of heat treatment on the solubilization of heavy metals, *Water Pollut. Contr.* 73 (1974) 207–209.
- [9] D.C. Stuckey, P.L. McCarty, Thermochemical pre-treatment of nitrogenous materials to increase methane yield, *Biotechnol. Bioeng. Symp.* 8 (1979) 219–233.
- [10] D.C. Stuckey, P.L. McCarty, The effect of thermal pre-treatment on the anaerobic biodegradability and toxicity of waste activated sludge, *Water Res.* 18 (1984) 1343–1353.
- [11] G. Alsop, R.A. Conway, Improved thermal sludge conditioning by treatment with acids and bases, *J. WPCF* 54 (1982) 146–152.
- [12] R.V. Rajan, J. Lin, B.T. Ray, Low-level chemical pre-treatment for enhanced sludge solubilization, *J. WPCF* 61 (1989) 1678–1683.
- [13] B.T. Ray, J. Lin, R.V. Rajan, Low-level alkaline solubilization for enhanced anaerobic digestion, *J. WPCF* 62 (1990) 81–87.
- [14] G.L. Kovacs, U.S. Patent 5 087 378 (11 February 1992).
- [15] G. Smith, J. Göransson, Generation of an effective internal carbon source for de-nitrification through thermal hydrolysis of pre-precipitated sludge, *Water Sci. Technol.* 25 (4–5) (1992) 211–218.
- [16] S.E. Woodard, R.F. Wukasz, A Hydrolysis/Thickening/Filtration Process for the Treatment of Waste Activated Sludge, *Water Quality International '94*, Book 5.
- [17] R. Burghardt, R. Krull, D.C. Hempel, R.H. Braunschweig, H. Pelousek, H. Bahnmüller, Alkalische Hydrolyse von Klärschlamm am Beispiel der Kläranlage Dormagen der Bayer AG, *Korrespondenz Abwasser* 44 (10) (1972) 1806–1811.
- [18] A. Erdinler, P.A. Vesilind, Effect of sludge cell disruption on compactibility of biological sludges, *Water Sci. Technol.* 42 (9) (2000) 119–126.
- [19] E. Neyens, J. Baeyens, M. Weemaes, B. De heyder, Hot acid hydrolysis as a potential treatment of thickened sewage sludge, *J. Hazard. Mater.*, in press.

- [20] J.K. Vallom, A.J. McLoughlin, Lysis as a factor in sludge flocculation, *Water Res.* 18 (12) (1984) 1523–1528.
- [21] M.J. Higgins, J.T. Novak, The effect of cations on the settling and dewatering of activated sludges: laboratory results, *Water Environ. Res.* 69 (2) (1997) 215–224.
- [22] M.J. Higgins, J.T. Novak, Dewatering and settling of activated sludges: the case for using cation analysis, *Water Environ. Res.* 69 (2) (1997) 225–232.
- [23] S.N. Murthy, J.T. Novak, Effects of potassium ion on sludge settling dewatering and effluent properties, *Water Sci. Technol.* 37 (4–5) (1998) 317–324.
- [24] P.A. Vesilind, A.H. Davis, Using the capillary suction time device for characterizing sludge dewaterability, *Water Sci. Technol.* 20 (1) (1988) 203–205.
- [25] H.W. Campbell, P.J. Creculo, Assessment of sludge conditionability using rheological properties, in: T.J. Casey, P. L'hermite, P.J. Newman (Ed.), *Methods of Characterization of Sewage Sludge*, D. Reidel Publishing Company, Holland, 1984.
- [26] J. Baeyens, L. Hosten, E. Van Vaerenbergh, *Wastewater Treatment*, Kluwer Academic Publishers, The Netherlands 1997, pp. 876 (in Dutch).
- [27] Y. Tezuka, Cation-dependent flocculation in a *Flavobacterium* species predominant in activated sludge, *Appl. Microbiol.* 17 (1969) 222.
- [28] J.H. Bruus, P.H. Nielsen, K. Keiding, On the stability of activated sludge flocs with implications to dewatering, *Water Res.* 16 (1992) 1597.
- [29] L. Eriksson, B. Alm, Study of flocculation mechanisms by observing effects of a complexing agent on activated sludge properties, *Water Sci. Technol.* 24 (1991) 21–28.
- [30] C.F. Forster, D.C. Lewin, Polymer interactions at activated sludge surfaces, *J. Effluent Water Treatment* 12 (1972) 520.
- [31] J.T. Novak, C.W. Randall, Sludge flocculation at an industrial waste facility, in: *Proceedings of the 18th Mid-Atlantic Industrial Waste Conference on Toxic and Hazardous Wastes*, Technomic Publishing Company Inc., Lancaster, 1986.
- [32] S.N. Murthy, J.T. Novak, R. de Haas, Monitoring cations to predict and improve activated sludge settling and dewatering properties of an industrial wastewater, *Water Sci. Technol.* 38 (1998) 119.
- [33] J.T. Novak, N.G. Love, M.L. Smith, E.R. Wheeler, The effect of cationic salt addition on the settling and dewatering properties of an industrial activated sludge, *Water Environ. Res.* 70 (5) (1998) 984–996.
- [34] J.T. Novak, H. Becker, A. Zurow, Factors influencing activated sludge properties, *J. Environ. Eng.* 103 (1977) 815.
- [35] P.R. Karr, T.M. Keinath, Influence of particle size on sludge dewaterability, *J. Water Pollut. Control Fed.* 50 (1978) 1911.
- [36] S.N. Murthy, J.T. Novak, Influence of cations on activated-sludge effluent quality, *Water Environ. Res.* 73 (1) (2001) 30–36.