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NITROGEN IN THE PROCESS OF WASTE ACTIVATED SLUDGE
ANAEROBIC DIGESTION

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Abstract: Primary or secondary sewage sludge in medium and large WWTP are most often processed by anaerobic digestion, as a method of conditioning, sludge quantity minimization and biogas production. With the aim to achieve the best results of sludge processing several modifications of technologies were suggested, investigated and introduced in the full technical scale. Various sludge pretreatment technologies before anaerobic treatment have been widely investigated and partially introduced. Obviously, there are always some limitations and some negative side effects. Selected aspects have been presented and discussed. The problem of nitrogen has been highlighted on the basis of the carried out investigations. The single and two step – mesophilic and thermophilic – anaerobic waste activated sludge digestion processes, preceded by preliminary hydrolysis were investigated. The aim of lab-scale experiments was pre-treatment of the sludge by means of low intensive alkaline and hydrodynamic disintegration. Depending on the pretreatment technologies and the digestion temperature large ammonia concentrations, up to 1800 mg NH₄/dm³ have been measured. Return of the sludge liquor to the main sewage treatment line means additional nitrogen removal costs. Possible solutions are discussed.

INTRODUCTION

The anaerobic sewage sludge digestion process is a well-established and widely applied technology at medium and large wastewater treatment plants. It is an attractive environmental friendly and economic technology with possible to achieve energy recovery. The main advantage is changing the visual and physical characteristic properties of the raw sewage sludge retained in primary settling tanks. The digested raw sludge as well as waste activated sludge have a dark black colour due to the presence of ferrous sulfide (FeS) and an acceptable odour. In the past, digested sludge was dried at ambient conditions for several months, and was turned to a consistence similar to ordinary garden earth. Also it was considered that the sludge does not contain pathogenic microorganisms. Primary mechanical treatment, followed by biological sewage treatment, in the case of medium and large treatment plants is rather seldom at present. Most often biological enhanced nutrients removal technologies based on modified activated sludge process are applied. In effect only surplus activated sludge has to be rejected, which is defined as Waste Activated Sludge (WAS). A large part of WAS are microorganism cells, which are

more difficult to biodegrade under anaerobic conditions. Nevertheless, it is expected to reduce about 40% of the overall load of waste sludge to be disposed. Also degradation of organic material results in the production of biogas containing about 55–70% CH₄, which can be used as a valuable source of energy. Other beneficial features include the stabilization of sludge, the improvement of sludge dewater ability, and the potential to inactivate and reduce pathogenic microorganisms.

Anaerobic sludge digestion has also some limitations, such as the length of time it takes for the sludge to be digested and the ineffectiveness of totally breaking down the solids [12]. The rate limiting step, mainly in the case of WAS, is agreed to be the hydrolysis of organic matter. In order to overcome the slower (much more difficult) WAS biological hydrolysis, pre-hydrolysis technologies (in front of the anaerobic digester) are to be applied. The pre-hydrolysis technologies have been widely investigated in the last two decades. They include chemical, mechanical, ultrasonic, thermal, thermo-chemical and microbiological methods. Some of the technologies have already been implemented on the full technical scale.

Pretreatment processes as well as the sludge digestion conditions can influence the quality of sludge and the sludge supernatant. Each of the pre-hydrolysis technologies, also defined as sludge disintegration technologies, causes a desired increase of soluble chemical oxygen demand (SCOD) or proteins in the liquid. It is assumed that organic matter solubilisation permits easy transformation to methane in the methanogenesis step of anaerobic digestion. Solubilisation of organic matter is also effected by the increased temperature in mesophilic (about 36°C) and thermophilic (about 55°C) conditions. Especially the increase of the temperature of about 20°C to the level of thermophilic conditions results in about fivefold increase of the level of dissolved COD (SCOD). According to Sørensen et al. [24] there is a shift in the kinetics of different processes in the anaerobic degradation, and a certain inhibition of the methane producing bacteria. Nevertheless, anaerobic thermophilic WAS digestion has gained much attention in recent years. The advantages claimed include enhanced hydrolysis of complex organic materials, better volatile solids destruction, higher biogas production and high pathogen destruction [15]. The thermophilic anaerobic digestion in practice is used in combination with mesophilic digestion as the first or secondary step. The two stages of anaerobic digestion are substantiated by a higher sensibility of thermophilic anaerobic digestion to inhibitory substances or such factors as temperature changes. The thermophilic digestion process is not always fully justified. Bhattacharya et al. [2] reported a relatively low volatile organic compounds removal rate of only 9.7%, what they considered to be not sufficient to justify the use of thermophilic sludge digestion.

The decomposition of organic matter results also in nitrogen compounds release. Proteins, aminoacids and urea are the main carriers of nitrogen. Nitrogen in proteins is bonded either as amino groups -NH₂ or as “peptic bonds” -NH-CO-.

Ammonium (NH₄⁺) and free ammonia (NH₃) are the two most predominant forms of inorganic nitrogen present in the fermenter. Chen et al. [5] reported that free ammonia is the most toxic of both due to the fact that it can pass through the cell membrane into the cell causing proton imbalance and potassium deficiency. The part of ammonia concentration present as NH₄⁺ or free ammonia NH₃ depends on the temperature and pH. Experiments [25], with treatment of protein-rich substrates (10% wt. of nitrogen) have shown that ammonium nitrogen concentrations can achieve the range of 5 to 10 g N/dm³

and the observed inhibition effect on production of methane starts with concentration of ammonium nitrogen of about 1 200 mg N-NH₄⁺/dm³ and rise of ammonia up to 3 000 mg N-NH₄⁺/dm³. Also rise of pH value to the range 8.5–9.0 [25] due to the shift to a higher ratio of free to ionized ammonia could bring the decrease in methane production to only 15–20% of original value. With increase of pH, the NH₄/NH₃ ratio decreases dramatically.

Although rise of temperature has a positive effect on the microbial growth rate it also results in a higher free ammonia concentration. Anaerobic thermophilic digestion of WAS leads to release of around 30 g N-NH₄⁺/kg TS, what is equal to 55% of the nitrogen content in the sludge [24]. It is well known that thermophilic digestion is more easily inhibited due to the presence of free ammonia than mesophilic digestion. Eventual increase in the amount of VFA at thermophilic conditions can result in pH decrease and consequently to a lower free ammonia concentration. As given by Liu and Sung [18] and summarized by Appels et al. [1], ammonia concentrations below 200 mg/dm³ are beneficial to the process of anaerobic digestion because nitrogen is an essential nutrient for the micro-organisms. Free ammonia of 560–568 mg N-NH₃/dm³ can cause a 50% inhibition of methanogenesis at pH 7.6 under thermophilic conditions [22]. The acetogenic population is more tolerant than the methanogens. When the concentration of ammonia was increased to 4051–5734 mg N-NH₃/dm³, the acidogens were hardly affected whereas the methanogens lost 56.1% of their activity [5]. However, the methanogenic bacteria can be acclimated to ammonia inhibition as a result of a shift in the methanogenic population or because of internal changes in the predominant methanogenic species. Chen et al. (2008) and Sung and Liu (2003) showed that the acclimated methanogens bacteria could tolerate concentrations up to 2 g N/dm³ in thermophilic conditions without inhibition, albeit with total inhibition of the methanogenic activity when a concentration of 10 g N/dm³ was reached.

In general, however, it is assumed that ammonia-nitrogen concentration in the range of 1500 to 3000 mg/dm³ is suspected to have an inhibitory effect on anaerobic digestion.

Minimization of ammonia concentration in the supernatant is a critical problem to be solved or at least mitigated. This is especially important in the case of reject water return to the biologically treated main wastewater stream. In this study, therefore, effects of selected pre-treatment technologies (pre-hydrolysis) as well as conditions of excess activated sludge anaerobic digestion have been discussed and investigated.

MATERIALS AND METHODS

Sludge pre-treatment and anaerobic digestion

Excess sludge denominated as Waste Activated Sludge (WAS) was taken from a local municipal wastewater treatment plant in the south of Poland. The plant was working according to the Enhanced Biological Nutrient Removal (EBNR) processes, without primary settling tanks. The plant was designed for a flow of 120 000 m³/d. At present the amount of treated wastewater is about 90 000 m³/d. Solid retention time (SRT) is about 14 days and the concentration of mixed liquid suspended solids (MLSS) is 4.3–4.7 g/dm³. Sludge samples (WAS) were collected from the secondary settling tanks. The average concentration of suspended solids (SS) was about 9.5 g/dm³.

The anaerobic digestion experiments were performed in glass fermenters (3.0 dm³ volume). The reactors were located in thermostatic conditions, with a constant temperatures 35±1°C and 55±1°C, what means under mesophilic and thermophilic

conditions respectively. In addition to batch separate anaerobic fermentation experiments at mesophilic or thermophilic conditions for a period of 25 days, two stage experiments, mesophilic process followed by thermophilic process were also carried out. In the case of the two stage digestion experiments, the duration of the first stage, the mesophilic stage was 10 days, and the thermophilic process was continued for a period of 15 days.

The feed to the experimental reactors consisted of a mixture of 20% inoculum and various parts of pre-treated (WASD) or not pre-treated WAS. In the pre-treatment processes chemical and physical destruction methods were applied. The compositions of the sludge feed are elucidated along presented appropriate experimental data. The experimental set up is shown in Fig. 1.

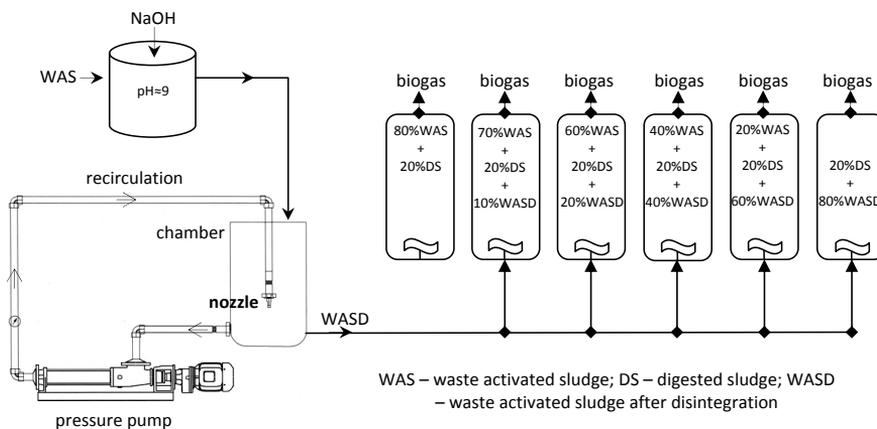


Fig. 1. Experimental set up of disintegration and fermentation processes

For chemical WAS disintegration (lysis of microorganisms cells) sodium hydroxide (NaOH) 2M was used. NaOH was added to samples of activated sludge in amounts sufficient to maintain a given pH value (8, 9, 10 and 11) for 30 minutes. Approximately from 0.8 to 6 mL NaOH (2M) per dm^3 of WAS.

For physical destruction of WAS the process of hydrodynamic disintegration was applied. The experimental set up for hydrodynamic disintegration execution consisted of a 12 bar pressure pump, rating 0,54 kWh, output 500 dm^3/h , which recirculated sludge from a 25 litre volume container, through a 1.2 mm nozzle. To force 25 litres of WAS through the nozzle 3 minutes were required. Disintegration was carried out for 15, 30, 60 and 90 minutes.

The chemical disintegration pre-treatment process (mainly at pH 9), followed by hydrodynamic disintegration for 30 minutes was also used. That process could be understood as a hybrid disintegration technology (WASD).

Analytical methods

All chemical analyses were performed for samples before and after each stage of disintegration and during anaerobic digestion. All chemical and physical parameters were

determined according to the procedures given in the Standard Methods for Examination of Water and Wastewater (19th ed.) [8]. The proteins concentration was determined by the Lowry method [11]. For determination of the concentration of soluble chemical oxygen demand (SCOD), proteins, ammonium nitrogen, nitrate nitrogen, nitrite nitrogen and phosphates a spectrophotometer HACH DR5000 was applied. Samples were centrifuged in all cases by 10 min at 30 000 g and then filtrated on paper filter.

For colorimetric determinations a spectrophotometer HACH DR/5000 was used. pH was measured with a WTW inoLab Level2 meter, equipped with a SenTix K1 electrode for pH.

RESULTS AND DISCUSSION

The effects of alkalization and hydrodynamic disintegration on organic matter release

According to the described procedure of waste activated sludge (WAS) solubilization, NaOH was added before hydrodynamic disintegration. Different doses of NaOH were added, what resulted in different values of pH in the range of 6.5 up to 12. The value of pH measured after 30 min of the selected dose addition was given as real alkalization value. The duration of 30 min was selected because in that period the solubilization quantity was 60–71% of total solubilized organic matter. Similar solubilization time can also be found in other papers [4, 17, 26]. However, the effectiveness of anaerobic digestion in terms of solids removal, gas production and nutrients release here presented were carried out at pH 9. High pH values above 10 causes protein to lose their natural shape, saponification of lipid and hydrolysis of RNA.

Destruction of bacteria cells and release of enzymes results in hydrolytic lysis of polypeptides. The WAS microorganisms destruction rate was measured as changes of soluble chemical oxygen demand value (SCOD) Fig. 2. Also the dissolution of proteins was determined (see Fig. 3).

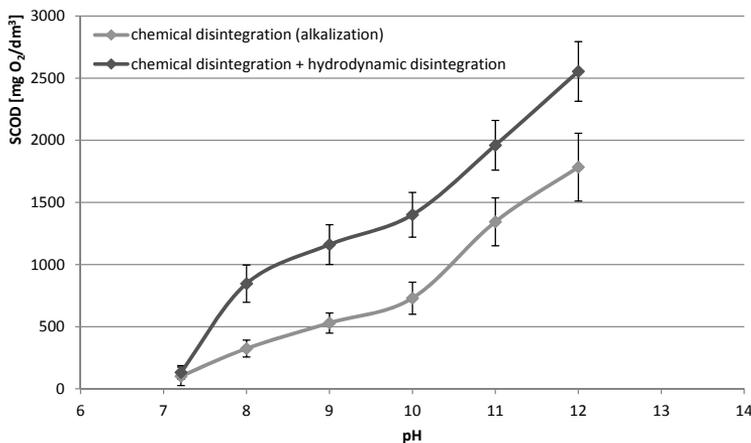


Fig. 2. Release of SCOD after alkalization only and alkalization + hydrodynamic disintegration processes

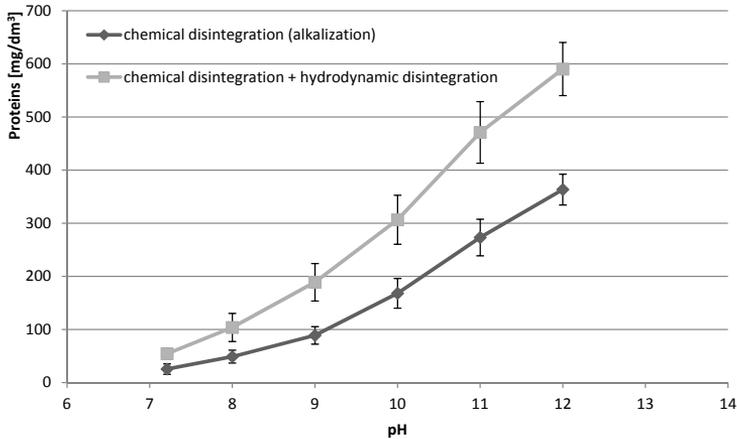


Fig. 3. Proteins dissolution after alkalization only and alkalization + hydrodynamic disintegration processes

Nutrients in the process of anaerobic digestion

Liquids associated to WAS in general contain similar concentrations of nitrogen compounds to the treated biologically wastewater. Although the WAS is partially thickened before feeding to the anaerobic digesters, the concentration of nitrate nitrogen reaches most often the highest values, like in the presented example below in Fig. 4.

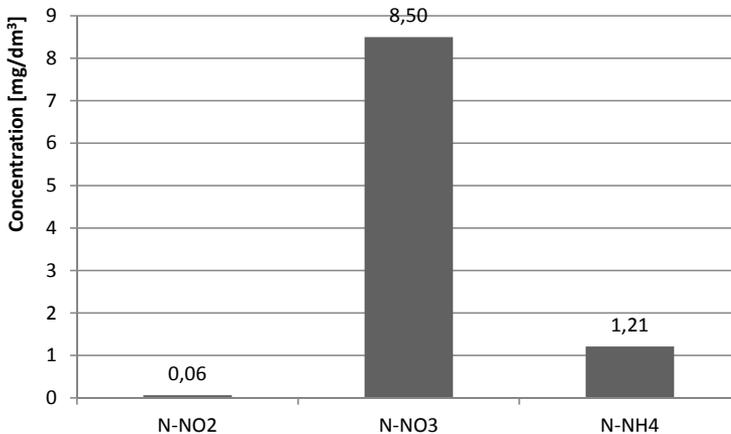


Fig. 4. Concentration of nitrogen forms in WAS liquid

Disintegration of WAS has very limited effect on the nitrogen compounds concentration. The concentration of all the investigated nitrogen forms – nitrites, nitrates and ammonia nitrogen, increases due to disruption of microorganisms and

external organic matter. A most pronounced increase happened in the case of nitrate nitrogen. The highest release of nitrate nitrogen can be explained by aerobic condition in the process of hydrodynamic disintegration, and also oxidation by hydroxyl radicals generated in the cavitation process. The obtained results are totally different from the results of nitrogen compounds solubilization given by Feng et al. [10]. Ultrasound WAS disintegration in the range of 500 to 26 000 kJ/kg TS resulted in ammonia nitrogen increase from 10 to 43 mg N-NH₄⁺/dm³, with almost no increase of nitrates at the level of about 6 mg N-NO₃/dm³ [10].

Assuming that the concentration of nitrogen is about 16–19% in proteins, then the total amount of nitrogen forms is not released in proportion to proteins dissolution.

The change of nitrogen compounds with the time of disintegration is shown in Fig. 5.

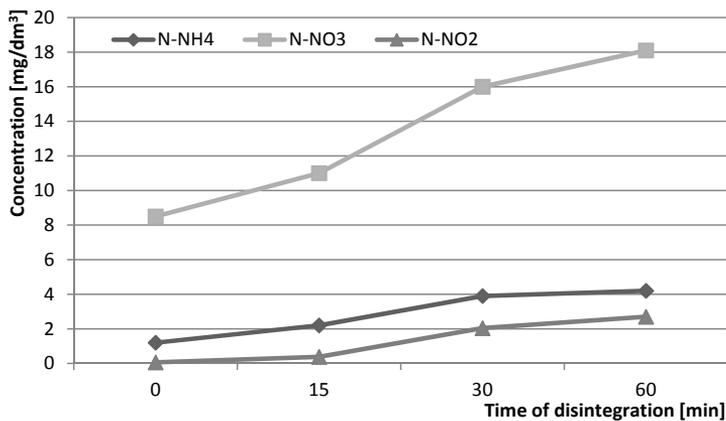


Fig. 5. Increase of nitrogen forms dissolution with time of disintegration

Comparing the amount of proteins released (Fig. 3) to the amount of nitrogen compounds dissolved (Fig. 5) it becomes evident that the proteins are not decomposed in the process of hydrodynamic disintegration. Decomposition of proteins occurs later under anaerobic biodegradation processes enhanced by the increased temperature.

There is a different situation in the case of phosphates. The activated sludge flocs are an agglomeration of bacteria maintained together due to the presence of extracellular exopolymers (EPS). Cloete and Oosthuizen [6] have examined the composition of extracellular exopolymers and found that they contained on average between 27% and 30% phosphorous. Disintegration of activated sludge flocs results therefore in mechanical destruction of EPS and bacteria dispersion as well as partial or complete bacteria cells destruction itself. Anaerobic sludge digestion under mesophilic or thermophilic conditions leads to further dissolution of phosphates [13–14].

In the process of anaerobic digestion a drastic increase of ammonia nitrogen is observed. Degradation of proteins and other nitrogenous organic compounds are the key nitrogen release substances. The proteins denaturation under increased temperature is

however very important. The carried out investigations of classic mesophilic digestion conditions, at the temperature of $35\pm 1^\circ\text{C}$ for 21 days, led to the release of ammonia-nitrogen between 700 to $750\text{ mg N-NH}_4^+/\text{dm}^3$ (Fig. 6), in the liquid associated to discarded anaerobic digested sludge.

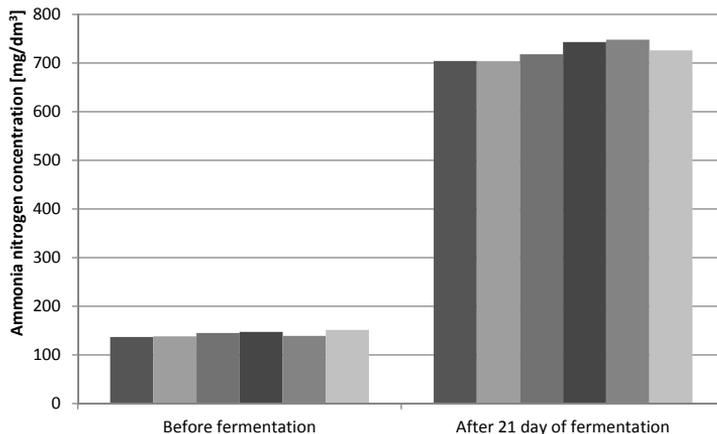


Fig. 6. The effects of ammonia nitrogen release at mesophilic anaerobic digestion (six different series, consecutively with disintegrated activated sludge participation of 0, 10, 20, 40, 60 and 80%, plus 20% inoculum)

High ammonia nitrogen concentrations in the sludge liquor are very common. For example, Borowski and Szopa [3], have presented ammonia concentrations in the range of 900 to $1000\text{ NH}_4^+/\text{dm}^3$. There are exceptions however, Sung and Santha [21] measured average ammonia concentrations as high as $2330\text{ mg NH}_4^+/\text{dm}^3$. The concentration of ammonia nitrogen in reject waters depends very much on the feed sludge biomass concentration. The activated sludge biomass contains nitrogen in the range between 70 to 90 g TKN/kg TS. The higher concentration of the biomass, the higher the concentration of ammonia nitrogen in the reject water can be. As already mentioned before, the pre-treatment processes contributed to the drastic increase of ammonia nitrogen concentration before sludge anaerobic digestion. The impact of the pre-treatment processes used in this study, i.e. alkalization and hydrodynamic disintegration, on the concentration of ammonia nitrogen is minor (Fig. 4). The amount of disintegrated activated sludge in the feed, being 10 up to 80% (Fig. 7), had a very limited influence on the concentration of ammonia nitrogen in the digested sludge slurry.

In all experiments, as already mentioned, the sludge feed composition always included 20% inoculum (20%DS – sludge from an active digester). The inoculum had therefore a distinct influent on the ammonia nitrogen concentration in sludge quality before the anaerobic fermentation (Fig. 6).

A two stage mesophilic and thermophilic digestion process was investigated. The increase of the temperature to 55°C resulted in a further increase of ammonia nitrogen concentration to a range of $1200\text{--}1400\text{ mg N-NH}_4^+/\text{dm}^3$ (Fig. 7).

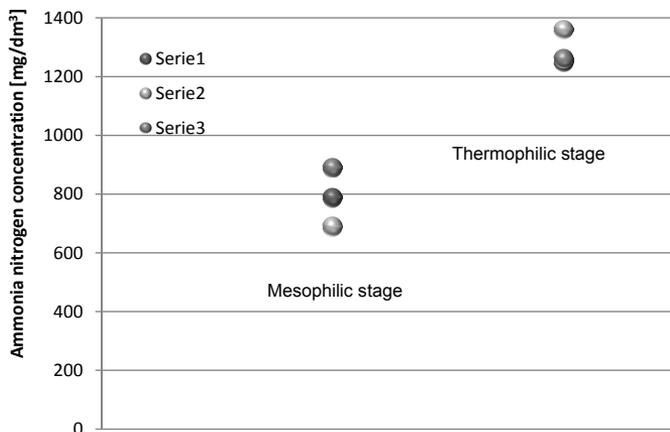


Fig. 7. Ammonia nitrogen concentration in the sludge liquor after mesophilic and thermophilic (second stage) digestion (average values for three series of experiments with different parts of pre-treated surplus sludge)

In other series of investigation of WAS samples the increase of ammonia concentration under thermophilic condition was not so drastic (Fig. 8).

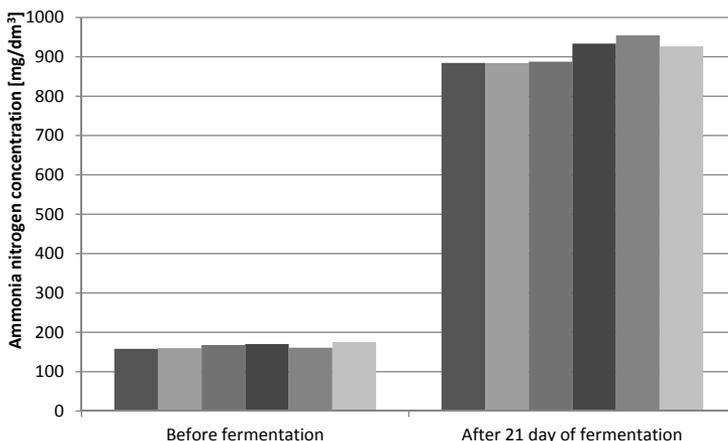


Fig. 8. Ammonia nitrogen release under thermophilic conditions (six different series, consecutively with disintegrated activated sludge participation of 0, 10, 20, 40, 60 and 80%, plus 20% inoculum)

However, based on a notable number of experiments it could be summarized that the difference between released ammonia nitrogen under mesophilic and thermophilic digestion is in the order of 25% (Fig. 9). A similar increase of phosphates (about 25%) was also observed as a result of the thermophilic conditions i.e. temperature increase to 55°C.

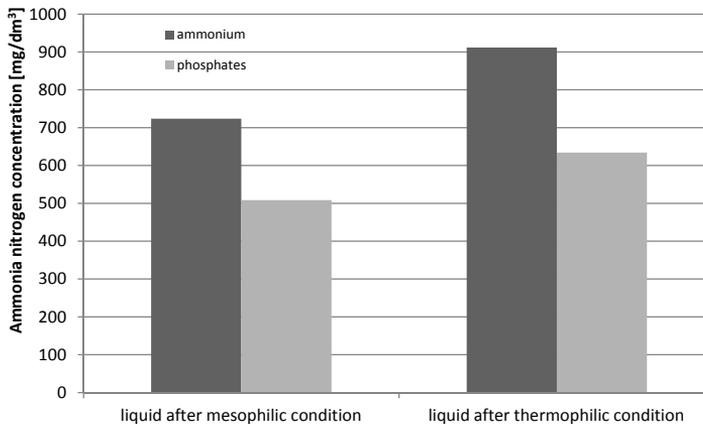


Fig. 9. Average concentrations of ammonia nitrogen and phosphates in the mesophilic and thermophilic WAS anaerobic digestion process

The results of the carried out experiments based on a low intensive pretreatment processes i.e. mild chemical treatment and low disintegration pressure are characterized by a relatively low release of ammonia nitrogen and phosphates. The released concentration of ammonia nitrogen (between 700 to 1400 mg N-NH₄⁺/dm³) in the sludge supernatant is relatively low in comparison to that of aggressive methods, e.g. thermal hydrolysis where the in-digester total ammonia nitrogen concentration can be above 2500 mg N-NH₄⁺/dm³ or even above 3000 mg N-NH₄⁺/dm³.

The implication of the investigated two step pretreatment technology - alkalization in the first step, followed by hydrodynamic disintegration was also evaluated. The results confirm a low impact of the applied pretreatment technology on the ammonia nitrogen concentration in the process of anaerobic digestion (Fig. 10).

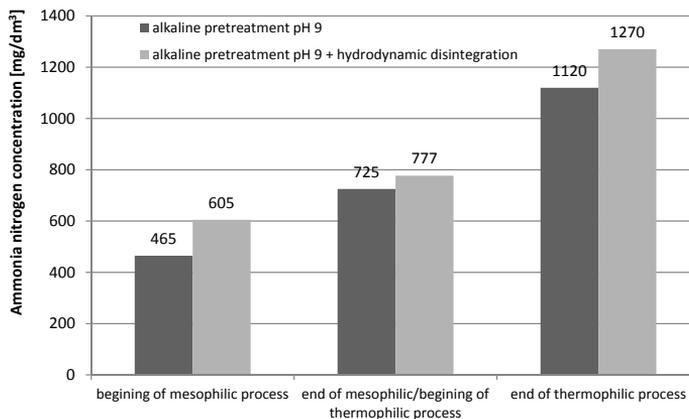


Fig. 10. The increase of ammonia nitrogen in the process of mesophilic and next thermophilic WAS digestion

The use of hybrid disintegration as a pretreatment process (alkalization + hydrodynamic cavitation) lead to an increased value of dissolution of ammonia nitrogen. However, neither the mesophilic nor the thermophilic anaerobic digestion gains from the higher disintegration rate due to additional hydrodynamic disintegration.

Ammonia nitrogen removal

The return of the digested sludge liquor to the main stream of wastewater treatment could add substantial amounts of phosphorous and nitrogen to be again removed in the biological processes. The additional load of phosphates can reach about 20% of the amount of raw sewage. Phosphates could be relatively easily precipitated in the form of struvite with an efficiency of above 95%. This is a relatively simple technology [9, 16] of moderate cost. Only magnesium has to be supplied to fulfill the struvite formula – $MgNH_4PO_4 \cdot 6H_2O$. Although the technology is well established after many investigations in the past twenty years, full scale installations are rare. Partially it is because the addition of e.g. ferrous salts for iron phosphates precipitation to the main stream of wastewater treatment is relatively cheap.

The problem of nitrogen returned to the main wastewater stream is much more difficult. First of all, the concentration of ammonia nitrogen in the reject liquid, as mentioned before, can reach high concentrations of about $2000 \text{ mg n-NH}_4^+/\text{dm}^3$, or even above. The amount of ammonia nitrogen directly removed in the process of struvite precipitation reach only about 5%. Therefore, the returned load of nitrogen can reach 100% or more of that carried by raw sewage. In the last decade different possibilities have been investigated. Most often dedicated processes such as nitrification/denitrification, Anammox, Cannon, DEAMOX, NOx processes have been tried. A process called Short Biological Nitrogen Removal (SBNR), being a modification of the Anammox technology, seems to be very interesting [7]. A new approach to anaerobic/aerobic sludge digestion treatment was presented by Novak et al. [19].

Due to the high ammonia nitrogen concentration in the reject liquor, after pH increase of up to 10–11, free ammonia can be stripped and in contact with sulfuric acid, ammonia sulfate can be produced. Vacuum distillation has been identified as a potentially valuable alternative [20].

Another approach has been suggested by Suschka and Popławski [23]. Basically, it is struvite precipitation, providing additional phosphorous salts. Adding an external source of phosphate such as superphosphate or phosphoric acid to fulfill the formula of struvite, in relation to the ammonia nitrogen concentration in the supernatant of digested sewage sludge, ammonia nitrogen can be almost completely removed. The carried out experiments have proven that the effects of ammonia nitrogen removal are independent on the chemicals used. An example of the decrease of ammonia nitrogen concentration using superphosphate is presented in Fig. 11. The residual ammonia nitrogen concentration can vary, but values as low as $25 \text{ mg N-NH}_4^+/\text{dm}^3$ can be obtained. After thermophilic sludge digestion the rejected sludge is pathogens free, and therefore the precipitated struvite can be safely used as a fertilizer in the agriculture.

The precipitation of struvite requires a pH above 8.5, preferable in the order of 9.5. In the case of phosphoric acid used as a source of phosphates, the pH of the sludge liquor decreases to about 2.4. Consecutively, the pH needs to be raised in order to enable struvite precipitation. Applying Na_3PO_4 , (super phosphate) no pH correction is needed.

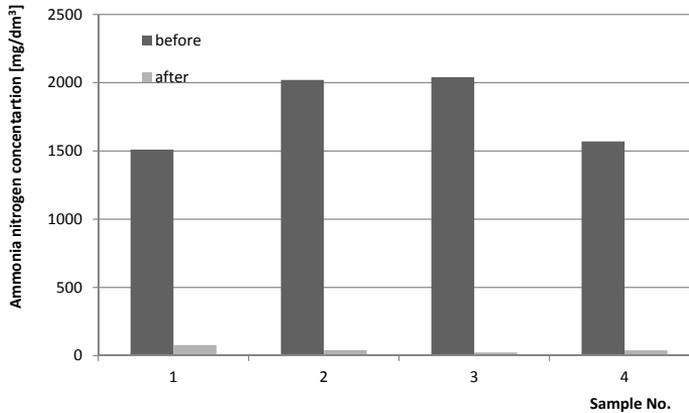


Fig. 11. The concentration of ammonia nitrogen in the centrate before and after addition of superphosphate and magnesium oxide

CONCLUSIONS

1. The surplus activated sludge biomass decomposition in anaerobic conditions is always accompanied by ammonia nitrogen release. The concentration of ammonia nitrogen in the reject water can be very high, somewhere in the order of 1000 to 3000 mg N-NH₄⁺/dm³. These high concentrations of ammonia nitrogen are considered to be a negative effect of sludge anaerobic digestion. If reject waters are returned to the treated wastewater main stream, the removal of the additional load of ammonia nitrogen will require additional energy.
2. Upgrading the anaerobic decomposition process at higher temperature, i.e. at thermophilic conditions, in comparison to mesophilic conditions results also, as presented in this study, in higher concentrations of ammonia nitrogen in the reject waters.
3. The released amount of ammonia nitrogen depends also on the applied pre-hydrolysis technology. It was demonstrated that the used low intensities pre-treatment hybrid technology, based on moderate alkalization – increase to pH 9, followed by hydrodynamic disintegration, has a negligible impact on ammonia nitrogen release.

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AZOT W PROCESIE BEZTLENOWEJ STABILIZACJI OSADÓW ŚCIEKOWYCH

Wstępne lub wtórne osady ściekowe powstające w średnich i dużych oczyszczalniach ścieków są najczęściej przetwarzane i kondycjonowane w procesie beztlenowej fermentacji, czego skutkiem jest zmniejszenie ilości osadów oraz produkcja biogazu.

W celu osiągnięcia lepszych rezultatów beztlenowej fermentacji osadów wprowadza się metody wstępnej hydrolizy osadów przed właściwym procesem fermentacji. Niektóre z tych metod znalazły zastosowanie w pełnej skali technicznej, a inne są badane i wprowadzane w sposób częściowy. Oczywiście, zawsze są pewne ograniczenia stosowania określonych metod i mogą występować pewne negatywne skutki uboczne. W artykule przedstawiono wybrane aspekty związane ze stosowaniem wstępnych procesów hydrolizy przed jedno- i dwustopniowym procesem fermentacji w warunkach mezofilowych i termofilowych. Jako wstępny proces hydrolizy zastosowano proces chemicznej i hydrodynamicznej dezintegracji. W zależności od zastosowanego procesu wstępnego oraz temperatury prowadzenia procesu fermentacji beztlenowej odnotowano znaczne stężenia amoniaku w cieczy osadowej dochodzące do $1800 \text{ mg NH}_4^+/\text{dm}^3$. Zawracanie cieczy osadowej do głównego strumienia oczyszczanych ścieków oznacza istotne zwiększenie kosztów oczyszczania ścieków. Recyrkulacja odcieków wymagałaby poniesienia dodatkowych kosztów związanych z usuwaniem azotu. W pracy, wskazano na możliwe do zastosowania procesy minimalizacji azotu amonowego w odciekach z procesu fermentacji osadów.