THREE PHASE SEPARATION OF THERMAL HYDROLYSED WASTE ACTIVATED SLUDGE

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Abstract

Thermal hydrolysis process (THP) has proven to be a valuable process to optimize anaerobic digestion by increasing the biogas production from waste activated sludge (WAS) and by substantially increasing the capacity of digester plants. Sustec BV (part of DMT) is continuously developing its continuous THP (cTHP) technology TurboTec®. Within these developments, Sustec BV discovered the capability of WAS to be separated into three different phases with direct dewatering at high temperatures (>100 °C) after cTHP. The produced (gelatinous) intermediate, formed during this thermal treatment without chemical additions, mainly consists of polysaccharides and proteins. The intermediate composition seems to have similarities with the produced alginate out of Nereda® sludge. However, in contrast to the formation of alginate out of Nereda® sludge, this recovery of potential valuable materials can be applied on all thermal hydrolysed WAS. At this moment, further research is being done on the exact composition of the produced intermediate and its potential applications

Keywords

Circular economy, direct dewatering, resource recovery, thermal hydrolysis, three phase separation.

Introduction

Waste water can be considered as a significant potential source for renewable energy and raw materials. Water authorities such as the Dutch Waterboards are committed to striving for the transformation of waste water treatment plants (WWTP's) into "energy and resource factories". This objective is also reflected in the European project: "full scale demonstration of energy positive sewage treatment plant concepts towards market penetration", (Powerstep). Next to improvements in the water line, specifically the sludge line has great potential for recovery of renewable energy, nutrients, valuable resources and las but not least cost reduction.

Mesophilic anaerobic digestion (MAD) is applied at WWTP's to convert surplus sludge into stabilised biosolids and biogas. The digester performance can be strongly enhanced by applying a thermal hydrolysis process (THP) as a pre-treatment prior to digestion. In a THP pre-treatment the sludge is treated at a high temperature (140 – 160 °C) and high pressure (4 – 6 bar). The advantages of applying THP are more biogas production, increased volatile solids (VS) reduction, shorter digestion retention times and an increased total solids (TS) content of the final sludge cake. By THP it is also possible to apply higher loadings to the digester, resulting in more capacity in the same volume. These effects result in substantial lower costs for the sludge treatment, while at the same time more renewable energy is produced.

The potential of the THP-process was recognised almost a decade ago by the Dutch company Sustec BV, when it decided to develop a practical process of its own. The activities led from lab experiments to determine overall process parameters, to pilot plant development and operation and finally to full-scale design, construction, and operation.

Developing the basic ideas into a working process has led to the typical set-up of the TurboTec® process as illustrated in Figure 1. In the Netherlands two full-scale references at WWTP Venlo and Apeldoorn are nowadays operational and working according to this principle, while two additional plants are in the design stage.

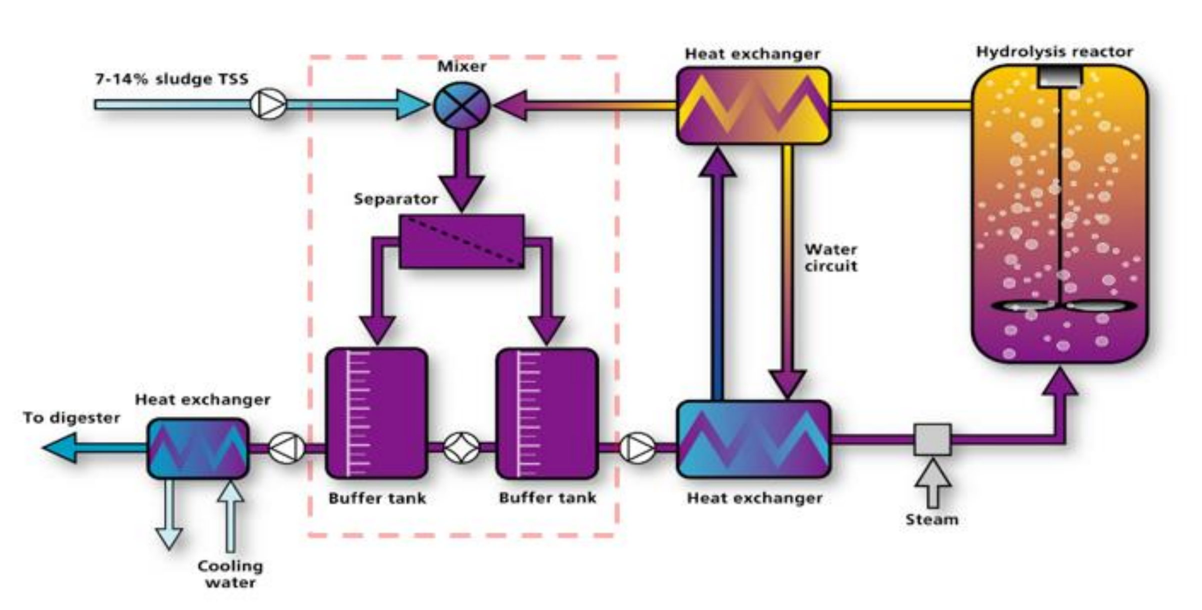

Figure 1: TurboTec® process

Benefits of thermal hydrolysis are not limited to the improvement of the anaerobic digestion process. The main financial benefits with cTHP can be achieved by increasing the total solids content of the sludge cake with limited polymer (PE) use (sludge flocculant to improve dewatering).

In the framework of its development of the TurboTec® cTHP, Sustec has paid considerable attention to the effect of thermal hydrolysis on the dewatering of biosolids on laboratory and full scale. The main findings of these experiments were:

- The degree of dewatering that can be achieved is significantly higher for hydrolysed sludge than for hydrolysed digested sludge. Apparently, part of the effect of the thermal hydrolysis treatment is lost in the anaerobic digestion process.
- An increase in the centrifugation temperature of the sludge showed a positive effect on the final total solids content of the sludge cake (pellet).
- This increase in the total solids content of the pellet also caused an increase of the total Chemical Oxygen Demand (COD) content in the liquid part (supernatant) of the sludge sample.

With the experimental results above, Sustec developed the concept of dewatering on a high temperature directly downstream of the thermal hydrolysis process. The process diagram of this next generation of cTHP principle is illustrated in Figure 2.

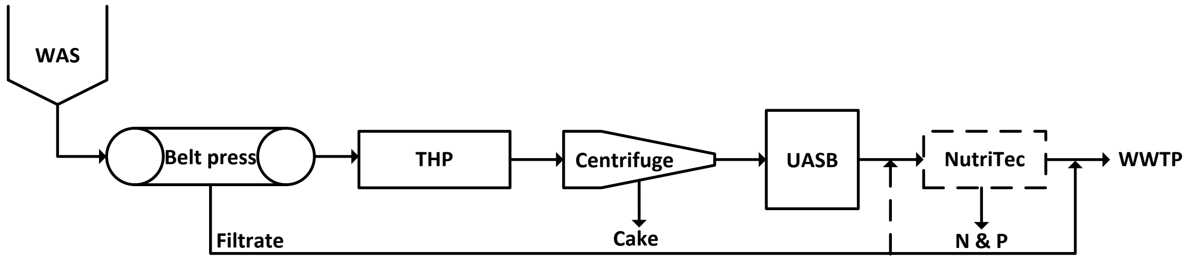

Figure 2: **Process diagram of the next generation of cTHP**

The concept in Figure 2, with the inclusion of the dewatering in the process, allows for a further optimization in the design of the anaerobic conversion process. Considering the potential for this concept Sustec has performed a demonstration test at the WWTP Venlo making use of the existing cTHP at this site. Findings of that research project have been presented at recent conferences dedicated to sludge treatment e.g. at WEFTEC 2017 (van de Ven, M. 2017).

As part of these development and research activities, Sustec BV discovered the capability of WAS to be separated into three different phases with direct dewatering at high temperatures (>100 °C) after cTHP. The produced (gelatinous) intermediate, see figure 3, formed during this thermal treatment, without chemical additions has been the subject of additional research of which the results are discussed in this paper.

Figure 3: **Intermediate product obtained from direct dewatering after THP**

Background

One of the recent developments in the treatment of wastewater is the Nereda® system, with the typical characteristic that it makes use of aerobic granular sludge as opposed to the common floc-structure of waste activated sludge. Recent research by Delft University of Technology has shown that the substance responsible for the granular growth is a polymer termed alginate-like exopolysaccharides (ALE). The properties established for ALE, make ALE a valuable raw material with many potential applications.

Upon discovering the possibility of separating a gelatinous intermediate in the treatment course by cTHP from common WAS, interest arose into the nature of this intermediate and its apparent similarity with ALE from granular sludge. Taking common WAS as the starting point could potentially increase the possibilities for re-use of this material significantly.

Another motivation for further research is the recognition of the negative role of extracellular polymeric substances (EPS) in the final dewatering of WAS. Removing part of the EPS selectively prior to dewatering could improve the performance and reduce operational costs, both for flocculant use and for final disposal.

Material and method

Materials

The tests were performed on WAS from two different locations:

- WWTP Venlo, a carrousel type WWTP without primary clarification, around 300.000 PE;
- WWTP Tilburg, a WWTP with primary and secondary clarification, around 340.000 PE.

In both cases, the WAS was sampled after mechanical thickening by a belt thickener. For Tilburg this resulted in samples with a TS-content of around 5%. In Venlo, where the thickened WAS is sent to cTHP treatment, TS content is considerably higher at values around 11%.

For the various experiments, 10 g samples of the WAS as received were used. In one series of experiments, the Venlo WAS was diluted to reach the same TS-content as the Tilburg WAS.

Method

The various samples were subjected to thermal hydrolysis at different temperatures by placement during 90 minutes in a thermostat (Hach Lange, LT 200) contained in an aluminium sealed tube. The tube construction can resist the forces induced by the pressure increase during hydrolysis.

Directly after removal from the thermostat, the tubes were placed in the centrifuge (Thermo Scientific, Hereaus Megafuge 16). Different gravity forces were applied to investigate the influence on the separation and to determine the optimum conditions.

After centrifugation, the tubes are left to cool to ambient temperatures and then the sample is divided into three fractions:

- Supernatant, liquid gathered in the top-part of the tube;
- Intermediate, gel like material, if present, non-soluble material gathered on top of:
- Pellet, compressed fraction of suspended solid materials resting at the bottom of the tube.

These different fractions could be removed separately from the tubes and form the basis for the rest of the investigations. Samples were analysed for Total Solids (TS), Volatile Solids (VS), Total Nitrogen (N), ammonia (NH3), Total Phosphate (P) and Chemical Oxygen Demand (COD).

The intermediate fraction is further characterised for Protein, Carbohydrate, and Fatty Acid content.

The protein content in the intermediate was determined by a modified version of the Kjeldahl-N method (Hogendoorn, 2013). The soluble ammonium and nitrate were withdrawn from the total nitrogen value. The insoluble N can be converted to protein by assuming all insoluble N creates from protein, and the protein contains 16% nitrogen (Raunkjær et al., 1994). The total nitrogen, nitrate, and ammonium concentrations were measured by making use of Hach Lange cuvette tests. The formula below was used to determine the protein content of the intermediate:

$$Protein = (Total\ nitrogen - NO_3 - NH_4) * 6{,}25$$

Carbohydrate content was determined by a phenol-sulfuric acid assay with D-glucose used as standard (Michel Dubois 1956). For the accuracy of carbohydrate determination in intermediate, the tests were executed in triplicate.

The fatty acid content in intermediate was measured by the modified method from Smolders et al (1995).

Results

Characterisation of the sludge samples

In table 1 the overall parameters of the sludge samples from Venlo and Tilburg are presented.

Table 1: Macro parameters of the two types of sludge

Sample Point	Total solids (%)	Volatile Solids (% of TS)	Total COD (g/l)
Venlo	10.0	75.4	117
Tilburg	5.2	69.0	50.2

Determination of optimal temperature

The first set of experiments was done to determine the optimum temperature for the hydrolysis to create a maximum amount of intermediate. This was done separately for both types of sludge. The experiments were performed with in total around 10 g of sample and the overall parameters indicated in table 1 above. The separation was performed at 10,000 G-force for this set of experiments. The figures below show the results for Venlo and Tilburg.

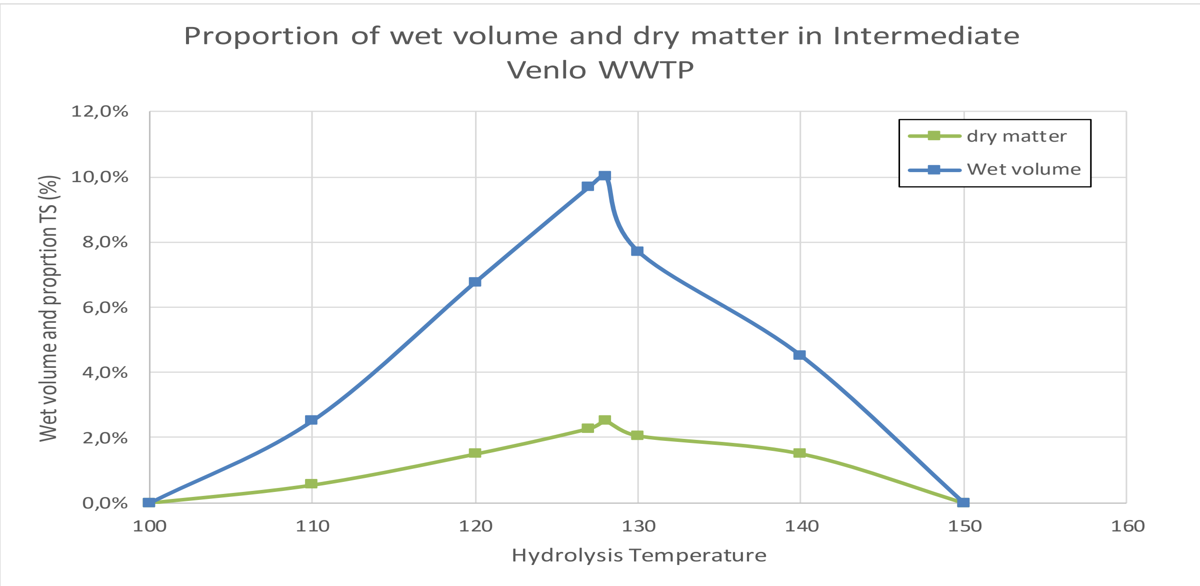

Figure 4: Wet volume and dry matter production of intermediate for Venlo sludge at different temperatures

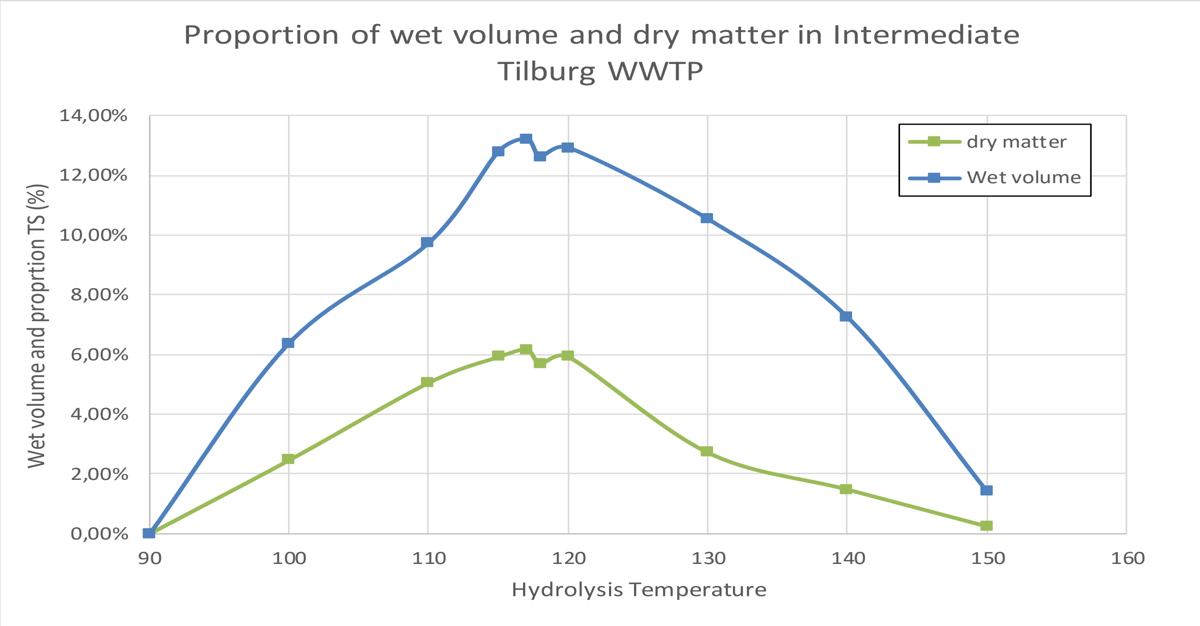

Figure 5: **Wet volume and dry matter production of intermediate for Tilburg sludge at different temperatures**

The data presented in Figure 4 and 5 show some remarkable similarities but also some specific differences:

- Both figures show that the production of the intermediate phase is dependent on temperature, below 90 – 100 °C no intermediate is formed and the same applies to temperatures above 150 °C;
- For both types of sludge, a clear optimum temperature can be indicated although the value is slightly different, for Venlo it is between 125 – 130 °C and for Tilburg the optimum lies between 115 and 120 °C;
- The maximum production of intermediate expressed as wet volume compared to the original sample is comparable, around 10 % for Venlo and around 13% for Tilburg
- The relative amount of the TS found in the intermediate is clearly higher for Tilburg at maximum values around 6 %, where for Venlo this value is around 2%;

The results described above refer to the division of volume and dry matter compared to the original sample.

With this it should be kept in mind that the TS content of the sample for Tilburg is about half of the value for Venlo (see table 1). This means that expressed in absolute quantities the difference in dry matter production of intermediate is smaller. From the Venlo sample of 10 g @ 10,0 % TS, the intermediate contains 0,03 g of TS. For Tilburg, the sample of 10 g @ 5,2 % TS, the intermediate contains 0.025 g of TS.

Determination of optimal G-force for separation by centrifuge

The tests described above were all performed by separating the treated samples at 10,000 G in the laboratory centrifuge. To investigate the effect of variation of G-forces, samples treated at the optimum temperature were subjected to a range of 3,000 – 15,000 G-forces.

Figures below show the results for Venlo and Tilburg.

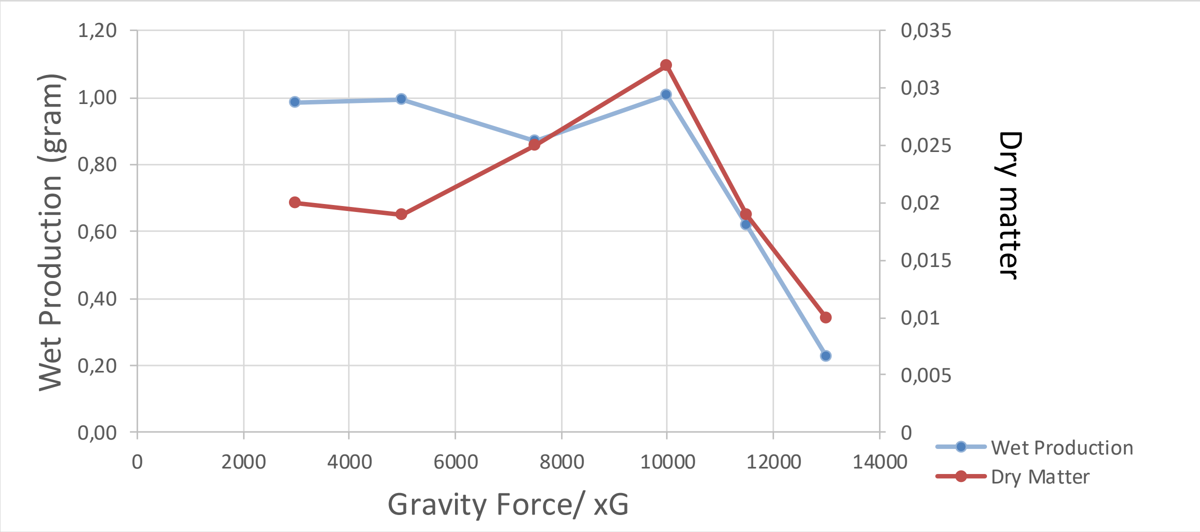

Figure 6: **Wet and dry matter production of intermediate for Venlo sludge at different G-forces**

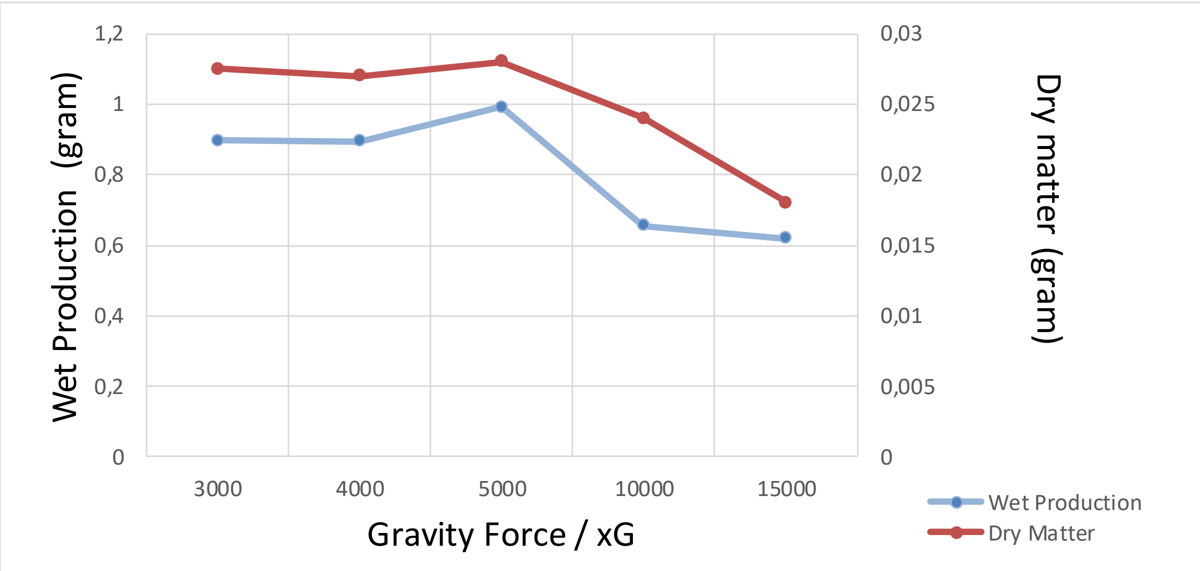

Figure 7: **Wet and dry matter production of intermediate for Tilburg sludge at different g-forces**

The values expressed in the figures relate to initial samples of around 10 gram where the intermediate production is expressed in absolute quantities.

For both types of sludge, the variation of G-forces leads to:

- A decrease of intermediate production at higher G-forces, this could be explained by the higher associated shear forces on the sludge, preventing the intermediate to agglomerate;
- At lower G-forces the production expressed as wet volume of intermediate for both sludges appears only slightly affected, whereas for the Venlo sludge a clear reduction in the dry matter production occurs.

For Venlo it appears that the optimum separation occurs at 10,000 G-force, whereas for Tilburg the optimum is reached at values around 5,000 G.

Characteristics at optimum conditions

For both locations, the samples were treated under the identified optimum conditions for temperature and separation G-forces. The different fractions produced; pellet, intermediate and supernatant were investigated further with respect to the division of COD, Nitrogen and Phosphate over these fractions.

The results are presented in Table 2.

Table 2: Division of components over the different fractions in % per component

Fraction	**Venlo**				**Tilburg**			
	TS	COD	N	P	TS	COD	N	P
Pellet	86	54	58	85	83	68	80	87
Intermediate	2.5	9	8	1	6	12	10	5
Supernatant	11.5	37	34	12	10	20	10	8

The table shows that for the components COD and Nitrogen, the relative fractions in the intermediate and in the supernatant exceed the values reached for the division of the solids (TS). The division of phosphate is more or less in line with the division of the solids.

This indicates that by extracting the intermediate a relatively high proportion of the total load of COD and Nitrogen is removed from the sludge.

The material in the Intermediate has been further characterised for its composition, considering the amounts of carbohydrates, protein, and fatty acids. These results are presented in table 3.

Table 3: Division of components over the different fractions in g/kg per component

Component	**Venlo** Intermediate (g/kg)	**Tilburg** Intermediate (g/kg)
Carbohydrate	246	295
Protein	672	633
Fatty acids	Below limit	Below limit
Balance unidentified	82	72

Table 3 shows a great similarity between the composition of the intermediate of Venlo and Tilburg intermediate. In both cases about 2/3 of the material consists of protein, with around 1/4 consisting of carbohydrates. Fatty acids are practically absent, leaving a balance below 10% of yet unidentified substances.

Discussion

In this research project, the feasibility and optimum conditions of producing an intermediate phase from WAS treated by thermal hydrolysis at different temperatures was investigated.

It was shown that the evolution of the intermediate phase was strongly dependent on the hydrolysis temperature, with production starting at values around 90 °C, reaching a maximum between 120 and 130 °C and decreasing again to zero at temperatures above 150 °C.

The origin of the WAS sample (the raw material for the tests) clearly had its effect on the amount of intermediate fraction that could be produced, considering the differences between the WAS of two different WWTP's in the Netherlands. The major difference between these two WWTP's is the presence/absence of a primary clarifier. The WAS produced at the WWTP without a primary clarifier (Venlo) was a less favourable starting material considering the relative amount of TS that was retrieved in the form of the intermediate fraction.

This difference in origin of the WAS was also reflected in differences in the optimal process conditions such as treatment temperature and G-forces applied for the separation of the fractions.

In the characterisation of the intermediate fraction it became clear that this fraction is relatively rich in the amount of COD and Nitrogen compared to the division of total solids between the fractions. Further characterisation for the presence of carbohydrates, protein and fatty acids showed great similarities between the samples from the two different WWTP's. It also shows the dominance of the protein content in composition with values of around 2/3 of the total material. Carbohydrates form the major part of the remaining material making up around 1/4 of the original material. Fatty acids were below the detection limit.

With the characterisation showing a dominant proportion of protein, the composition of the intermediate fraction is different from the ALE, produced form extraction of aerobic granular Nereda® sludge (van de Roest et al, 2015). In the ALE, the polysaccharides (carbohydrates) are the dominant component. A possible explanation for this is related to the temperature at which the intermediate is produced, being at least above 100 °C. In the thermal hydrolysis treatment, the Extracellular Polymeric Substances (EPS), consisting mainly of polysaccharides are destroyed, while at the same time intracellular substances are being released as a result of cell-degradation. In the intracellular material, the protein is expected to be the dominant component (D'Abzac et al., 2010).

Conclusions

The following conclusions can be drawn:

- Thermal hydrolysis combined with direst dewatering offers new opportunities for advanced sludge treatment, resulting in resource recovery and reduction of sludge volumes for final disposal.
- In this research project it was shown feasible to produce an intermediate phase in addition to supernatant and pellet by applying a centrifuge for high temperature separation of WAS treated by thermal hydrolysis.
- The production volume of the intermediate phase is strongly dependent on the treatment temperature of the thermal hydrolysis. No intermediate phase was found below 90 °C and above 150 °C. Optimum temperature range is between 120 – 130 °C.
- Characterisation of the intermediate shows a major contribution of protein in the overall composition of around 2/3 of the volume produced. Carbohydrates form the major part of the remaining material making up around 1/4 of the intermediate.

Outlook

With the principle feasibility for production of the intermediate established and the main components identified, the logical next step would be to investigate the possible applications of this material in more detail. The considerable protein content of the intermediate provides a first indication for these applications and for the potential partners that might be interested in developing the options for the intermediate into a marketable product.

Sustec welcomes serious queries regarding this research project and is committed to bringing this example of sustainable technology to market readiness in alliance with interested third parties.

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