

Mechanical sludge disintegration for the production of carbon source for biological nutrient removal

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ABSTRACT

The primary driver for a successful biological nutrient removal (BNR) is the availability of suitable carbon source, mainly in the form of volatile fatty acids (VFA). Several methods have been examined to increase the amount of VFAs in wastewater. This study investigates the mechanism of mechanical disintegration of thickened surplus activated sludge (SAS) by a deflaker technology for the production of organic matter. This equipment was able to increase the soluble carbon in terms of VFA and soluble chemical oxygen demand (SCOD) with the maximum concentration to be around 850 and 6530 mg l⁻¹, for VFA and SCOD, respectively. The particle size was reduced from 65.5 µm to 9.3 µm after 15 minutes of disintegration with the simultaneous release of proteins (1550 mg l⁻¹) and carbohydrates (307 mg l⁻¹) indicating floc disruption and breakage. High performance size exclusion chromatography (HPSEC) investigated the disintegrated sludge and confirmed that the deflaker was able to destroy the flocs releasing polymeric substances that are typically found outside of cells. When long disintegration times were applied (≥10 minutes or ≥9000 kJ kg⁻¹ TS of specific energy) smaller molecular size materials were released to the liquid phase, which are considered to be found inside the cells indicating cell lysis.

Keywords activated sludge, biological nutrient removal, carbon source, cell lysis, mechanical disintegration, particle size, polymers

1. Introduction

Biological nutrient removal (BNR) is widely used in modern municipal and industrial wastewater treatment plants. A significant factor in the success of the BNR process is the availability of a suitable carbon source. Many authors have reported a specific ratio of carbon to phosphorus (P) and carbon to nitrogen (N), which can indicate the suitability of the wastewater for BNR treatment (Johnsson *et al.*, 1996; Randall *et al.*, 1992; Grady *et al.*, 1999 and Curto 2001). The most common methods for increasing these ratios and hence make the wastewater treatable by a BNR process, are the pre-treatment of the wastewater (McCue *et al.*, 2003) or the addition of extra carbon. The extra carbon can either be organic carbon in the form of methanol, ethanol, acetic acid and glucose, hydrolysed primary and secondary sludge or industrial wastes rich in readily biodegradable organic matter and volatile fatty acids (VFAs) (Charlton 1994 and Llabres *et al.*, 1999).

The online fermentation of wastewater is able to increase the concentration of VFAs (by 25 %) and improve the performance of the process (McCue *et al.*, 2003). Likewise sludge fermentation can increase the available carbon and improve nutrient removal (Hatziconstantinou *et al.*, 1996 and Charlton 1994). Unfortunately, the application of these methods is not a straightforward procedure, since site enlargement is required and usually the fermenters are prepared for permanent use without being flexible to any modifications (flows, retention times etc.) (Munch and Koch, 1999). In addition, nutrients are also released from sludge during the fermentation process, increasing the overall amount of nutrients that have to be removed. Industrial wastes, such as the fermented organic fraction of municipal solid waste, fermented leachate of food waste and nightsoil have been shown to contain rich organic substrate in terms of VFA and soluble chemical

oxygen demand (SCOD) (Llabres *et al.*, 1999; Bolzonella *et al.*, 2001 and Choi *et al.*, 1996). The addition of the above wastes in BNR process has been reported to have a positive impact increasing the removal of nitrogen (~ 28-109 % increase) and phosphorus (~ 97-410 % increase) (Lim *et al.*, 2000 and Lee *et al.*, 1997). For instance, in the UK the wastewater treatment plants in Derby and Reading in Severn Trent Water and Thames Water areas, respectively, are successful BNR sites with domestic sewage being mixed with industrial effluent. When the additional carbon is in the form of methanol or acetic acid, there is an improvement in BNR performances (Tam *et al.*, 1992; and Fass *et al.*, 1994). The only and important disadvantage of the addition of direct organic carbon is the high operational cost. Similarly, the industrial wastes are not always applicable to a given wastewater treatment plant since transportation requirements may result in extra costs. Moreover, the content of nutrients in some industrial wastes is high, increasing the load to the process.

Researchers investigating alternative carbon sources, which can easily be used without high operational cost, observed that sludge disintegration used in sludge treatment processes is able to increase the SCOD and VFA content. The ultimate goals of these methods are foam control, increase in biogas production and sludge mass reduction (Muller 2000b). However, there is little information about the application of the disintegrated sludge to BNR in order to improve P and N removal. Muller (2000a and 2000b), made a comparison of mechanical, thermal and chemical sludge treatment. The highest degree of disintegration was reached by the chemical (ozone) treatment (52%) with the mechanical methods reaching medium degrees of disintegration (20-37%) with a relatively low energy input (1000-10000 kJ kg⁻¹). Stirred ball mills and high pressure homogenizers were used for sludge disintegration by Muller (2000), to produce a carbon

source for denitrification. According to their experiments the maximum denitrification rate was up to 15 mg NO₃-N g⁻¹ VSS h⁻¹. Chiu *et al.*, (1997) reported that the combination of alkaline treatment with ultrasound vibration brought an increase in SCOD and VFA at 41.5 and 28 times, respectively. The maximum increase in SCOD was reported by Wang *et al.*, (1999) at 50 times with ultrasound equipment of 400 W treating 100 ml of surplus activated sludge (SAS) for 40 minutes.

This study examines mechanical sludge disintegration using a deflaker, a technology designed for processing the pulp in paper industries. The main goal of this study was to examine whether this equipment is capable of producing extra carbon from SAS and to investigate the mechanisms of disintegration. The suitability of the carbon produced for improving biological nutrient removal will be investigated in further research.

2. Material and methods

The SAS used in this study was collected immediately after the belt thickener with the total solid (TS) concentration in the range of 4-7 %, from wastewater treatment plants (WWTPs) that operate in BNR (BNR SAS) or standard activated sludge mode (non BNR SAS) (Table 1).

2.1 Disintegration

The equipment used for sludge disintegration was a 10" Pilao DTD Spider Deflaker with a 30 kW motor fitted with 230 mm discs with 3 active cell layers (Figure 1). The gap distance between stator and rotor was 0.6-0.9 mm and the rotation speed 3000-3600 rpm, (Withey 2003). The disintegration process was conducted as a batch with five litres of thickened SAS to be treated each time at four different retention times, 2, 5, 10 and 15

minutes. The maximum monitored temperature of disintegrated sludge was 35 °C and considered to have negligible effect on disintegration (Wang *et al.*, 2005). To quantify maximum carbon release in SAS a thermal extraction method was established, which was a modification of a method reported by Zhang *et al.*, (1999) for the extraction of extracellular polymers. This method was used only for comparative reasons as was assumed that thermal extraction could indicate the absolute carbon release causing cell lysis. During that method thickened SAS was left for an hour under the conditions of 1bar and 105°C.

2.2 Analytical methods

Particle size analysis was carried out using a Malvern Mastersizer 2000 (Malvern, UK). The Mastersizer uses an optical unit to detect the light scattering pattern of sludge particles dispersed in deionised water. High performance size exclusion chromatography (HPSEC) was carried out using high performance liquid chromatography (HPLC) (Shimadzu VP Series, Shimadzu, UK) with UV detection set at 254 nm and a BIOSEP-SEC-S300 column (Phenomenex, UK). For each sample a chromatogram of absorbance against time was produced. Larger molecular size compounds were eluted from the column first and smaller molecules later.

Apart from the total solids all the other analysis were carried out in the filtered (0.45 µm) supernatant of the untreated and treated sample after centrifugation (10500 g, 7°C). The concentration of Total Solids (TS), soluble COD, VFA, NH_4^+ and P was measured according to APHA Standard Methods (1998). The soluble protein content was determined according to standard protein method by Branford (1976) with a protein diagnostic kit (Sigma, England). The concentration of soluble proteins was determined from a standard curve constructed using bovine serum albumin (P0914 Sigma – Aldrich,

Gillingham, UK) in deionised water, as the standard. Soluble carbohydrate concentrations were determined using the phenol sulphuric method introduced by Dubois *et al.*, (1956). Carbohydrate concentration was calculated from a calibration curve constructed using a glucose standard.

3. Results-Discussion

The mechanical disintegration of thickened SAS by the deflaker was evaluated in terms of soluble carbon release, and its correlation with energy input. In addition, the mechanism of the process was investigated in order to identify whether the deflaker is able to cause floc disruption, floc breakage or cell lysis.

3.1 Carbon and phosphorus release and energy input

The initial concentration of VFA and SCOD in thickened SAS was always dependant on the WWTP source (BNR or non BNR). In BNR thickened SAS was ranged from 0 to 19 mg l⁻¹ and 120 to 342mg l⁻¹ for VFA and SCOD, respectively and in non BNR from 186 to 311 mg l⁻¹ and 752 to 1180 mg l⁻¹ for VFA and SCOD, respectively. The results from mechanical disintegration of BNR thickened SAS at four different retention times in the deflaker are summarized in Table 2. As expected, the increase in VFA and SCOD was related to the disintegration time. The maximum concentration of VFA and SCOD measured were 530mg l⁻¹ and 6180 mg l⁻¹ for 10 and 15 minutes retention time, respectively. Additionally, phosphorus release also occurred, again increasing with the retention time in the equipment with the maximum concentration measured to be 480 mg l⁻¹ for 15 minutes of disintegration. The ammonium was not frequently measured and only a few measurements have been taken in the disintegrated sludge by deflaker for 15 minutes, which showed that there is an increase reaching the concentration of ~60 mg l⁻¹.

The results from disintegration of non BNR SAS followed the same trend as the BNR sludge, although here the initial concentrations of VFA and SCOD were higher (Table 3). This is expected as in the non BNR WWTP the soluble available carbon is not totally consumed and can still be found in surplus sludge. Phosphorus release was much lower with the maximum concentration to be 135 mg l^{-1} , as in non BNR WWTP the biologically stored phosphorus content in sludge is lower than in sludge from BNR WWTP. The maximum concentration of the released P was 135 mg l^{-1} for 15 minutes of disintegration, much lower than the respective concentration after disintegration of BNR sludge (480 mg l^{-1}).

The energy consumption of the deflaker was 5.7 kW for each 5 L of sludge. After 15 min of disintegration at 1.14 W ml^{-1} the SCOD was increased by 20 times. This compares well to the increase in SCOD by 7.7 times reported by Mao *et al.*, (2004) with a sonication of 20 kHz frequency and at 4 W ml^{-1} . The performance of disintegration by the deflaker can also be compared with other disintegration techniques by using specific energy, which can be defined as the amount of mechanical energy that stresses a certain amount of sludge (Muller 2000a). The effect of specific energy from the deflaker on SCOD shows as expected, that greater energy input gave higher release in SCOD (Figure 2). The rate of SCOD release is higher for the first $2500 \text{ kJ kg}^{-1} \text{ TS}$, than when more energy is applied. This rate (specific energy $<2500 \text{ kJ kg}^{-1} \text{ TS}$) is the same for the two sludges with different solids concentration, but with higher energy input the more thickened sludge proved to be more efficient as with the same energy gives higher SCOD release. The degree of disintegration for COD (DDCOD) was also taken into account, which was calculated by the equation below:

$$\text{DDCOD} = [(\text{COD}_d - \text{COD}_u) / (\text{COD}_{th} - \text{COD}_u)] \cdot 100 \quad [\%],$$

Where COD_d , COD_u , and COD_{th} are the SCOD values for the disintegrated (COD_d), untreated (COD_u) and thermal extracted (COD_{th}) sample (Muller 2000a). The maximum degree of disintegration was taken as being released by a thermal extraction method, which gives similar results with other methods used in literature for the same purpose (Lehne *et al.*, 2001, Muller 2000b, Tiehm *et al.*, 2001).

The degree of disintegration by the deflaker as a function of specific energy is shown below (Figure 3). Stirred ball mills (SBM), which are a similar type of mechanical equipment to the deflaker and an ultrasonic homogenizer (UH) were tested by Muller (2000a). A comparison of deflaker to the other equipment showed that to achieve a 25% degree of disintegration SBM, deflaker and UH required 3000, 4000 and 10400 kJ kg⁻¹ TS of specific energy, respectively.

3.2 Mechanisms of disintegration

The results from the particle size distribution from both sludges showed that the deflaker was able to decrease the size of the particles (Figures 4 and 5). The median size of the raw BNR sludge was around 65.5 μm and was reduced to 9.3 μm after 15 minutes of disintegration. Similarly, the size of particles in raw non BNR sludge was 45.6 μm and after 10 minutes of disintegration with the deflaker was 9.3 μm . The formation model of floc consists of primary particles (~2.5 μm), microflocs (~13 μm) and porous flocs (~100 μm) (Jorand *et al.*, 1995). Particle size distribution shows that after disintegration not only most of the porous flocs are destroyed, but also some of the microflocs with the simultaneous release of polymeric compounds which link the microflocs to each other

(Jorand *et al.*, 1995). That is in agreement with Chu *et al.*, (2001) who reported a porous floc destruction at an ultrasonic treatment of 20 kHz frequency at 0.44 W ml^{-1} and can be confirmed by the fact that the reduction in particle size takes place with the increase in organic matter (SCOD, VFA).

According to Lehne *et al.*, (2001) 3000 kJ kg^{-1} TS are required for floc size reduction and decrease the median size of the particles down to 10-15 μm . They reported that further disintegration caused a smaller decrease in particle size, from 10-15 μm to 3 μm with high amount of specific energy (from 3000 to 100000 kJ kg^{-1} TS). Here the deflaker was able to cause porous floc disruption as the median particle size is decreased to approximately 10 μm and for similar specific energies ($\sim 2500 \text{ kJ kg}^{-1}$ TS) (Figure 6). In addition, as mentioned above the SCOD release follows a different rate between the first 2500 kJ kg^{-1} TS applied and the rest specific energy added. The release in SCOD is faster for the first 2500 kJ kg^{-1} TS of specific energy and around $0.96 \text{ mg SCOD per kJ kg}^{-1}$ TS of specific energy added, when also particle size is significantly reduced and then the rate is decreased to $0.36 \text{ mg SCOD per kJ kg}^{-1}$ TS of specific energy added with almost no reduction in the particle size. That means that during the floc breakage, a big amount of organic matter is released in the liquid phase ($1500\text{-}1900 \text{ mg l}^{-1}$ in terms of SCOD) and for further disintegration and energy input organic matter is also released, but without any difference in particle size indicating that is not coming from the disruption or breakage of flocs.

It is still not certain though, whether cell lysis takes place as the released organic matter after 2500 kJ kg^{-1} TS of specific energy could be not due to cell lysis, but due to further floc breakage. The concentrations of soluble proteins and carbohydrates in the

supernatant before and after disintegration were also examined in order to evaluate disintegration. Protein analysis has been reported as another method for assessing disintegration showing that can be a relatively quick and effective method (Schmitz *et al.*, 2000). The concentrations of proteins, carbohydrates, SCOD and VFA after disintegration were related to the retention time (Figures 7 and 8). Here, disintegration was compared with the thermal extraction method, as an estimation of maximum release. The predominant component of the released material is SCOD, which includes the proteins, carbohydrates and volatile acids. The protein concentration indicates that a part of the extracellular polymeric substances (EPS), which link the microflocs to each other, have been destroyed (Jorand *et al.*, 1995, Wang *et al.*, 2005). The concentration of proteins increased approximately 27 and 5 fold for BNR and non BNR sludge, respectively. This increase is significantly higher than the one reported by Nah *et al.*, (2000) from mechanical treatment using a high pressure pump and a nozzle (2.5 times), but lower than the increase reported for ultrasound treatment (200 W and 9 kHz) for 40 minutes (~48 times) (Wang *et al.*, 1999). The results agree with Wang *et al.*, (2005) who reported an increase in SCOD, proteins, polysaccharide and DNA after disintegration by ultrasound (300-1200 W, 20 kHz and 0-30 minutes retention time).

To summarize, the particle size distribution showed that disintegration caused a decrease in particle size with the simultaneous release of organic matter in the form of proteins and carbohydrates. The breakage of flocs released the polymeric substances used for the formation of flocs in the aqueous phase. But, to determine whether cell lysis takes place further information was required.

The high performance size exclusion chromatography (HPSEC) was used as a “fingerprint” technique for the organic materials that are produced during disintegration. The results showed that during short retention time (<10 minutes) large molecular size materials (first peaks in the graph) are released, which are thought to be polymeric substances and typically found outside of the cells (extracellular) (Figure 9) (Jorand *et al.*, 1995). At longer retention time (≥ 10 minutes) the release not only of the extracellular, but also of smaller molecular size materials (last peak in the graph) can be observed. These materials can either be disrupted extracellular polymers or intracellular material after cell lysis. On the other hand the thermal extraction method seems to have released small molecular size materials without destroying the polymeric substances (Figure 9c). Taking also into account that the destruction of polymers would not have caused an overall increase of the soluble organic matter as was observed (Figures 7 and 8), we consider that the appearance of smaller compounds in BNR SAS disintegration are mainly material that have been released due to cell lysis. Moreover, after the destruction of the flocs and the removal of the polymeric substances from the cell walls the shear forces from the deflaker would be able to break the cell walls (Wang *et al.*, 2005). Results from HPSEC confirmed the data from particle size distribution proving that during mechanical disintegration with the deflaker, firstly flocs are disrupted decreasing the particle size and releasing extracellular material and then cell lysis starts taking place releasing some extra organic matter, for longer periods of disintegration (≥ 10 minutes or ≥ 9000 kJ kg⁻¹ TS of specific energy).

After the particle size distribution it was suspected that the increase in organic matter after 2500 kJ kg⁻¹ TS of specific energy added, with the simultaneous steady size of the particles is due to cell lysis. The HPSEC results showed that cell lysis appears when more

energy is added ($9000 \text{ kJ kg}^{-1} \text{ TS}$), proving that the extra SCOD released for energies between 2500 and $9000 \text{ kJ kg}^{-1} \text{ TS}$ is because of further breakage of the flocs. Using the $9000 \text{ kJ kg}^{-1} \text{ TS}$ of specific energy as a threshold for cell lysis we can estimate the proportion of SCOD that comes from floc breakage and cell lysis (Figure 2). For a total energy input of $14250 \text{ kJ kg}^{-1} \text{ TS}$ and 4900 mg l^{-1} of SCOD released the 73 % or 3900 mg l^{-1} SCOD comes from floc disruption or breakage and the remaining 27 % from cell lysis. The above could be verified by confirming the threshold of cell lysis by doing an assay for DNA, where the significant increase in the concentration means cell lysis.

The lack of available soluble carbon is a major problem in the BNR process. This study shows that the deflaker process can be used to disintegrate the sludge and release trapped organic material to the liquid phase. Unfortunately, there is little information in the literature about the application of disintegrated sludge in BNR, as an extra carbon source. Most of the applications of sludge disintegration to date have consisted of improving anaerobic digestion and reducing sludge production. Muller (2000b) has reported some exemplary results of how denitrification rate can be improved by different disintegration methods. As reported by the same author mechanical disintegration on adapted biomass gave a higher denitrification rate compared with thermal disintegration (no relation with thermal extraction method mentioned above) and biological hydrolysis.

A comparison has been made between the available carbon produced and phosphorus released by disintegration in the form of SCOD to P ratio (Table 4). The ratios are low (7-13 for BNR sludge) compared to the ratios found in the literature for successful BNR plants (40-60) (Randall *et al.*, 1992; Carlson *et al.*, 1996), but with longer disintegration giving higher ratio. That means that mechanical disintegration releases more phosphorus

than the produced carbon is theoretically able to remove. The ratios for non BNR sludge are higher than the ones produced from BNR sludge (77 and 13.6 for non BNR and BNR sludge, respectively), but are expected to decrease, when the WWTP be converted to BNR and the phosphorus being removed biologically. This is an indication that the addition of mechanically disintegrated sludge in a BNR process can cause deterioration instead of improvement. The sidestream removal of the released phosphorus (i.e. struvite or calcium phosphate precipitation) before being added to the process could make disintegration by deflaker to have a positive impact on BNR. On the other hand, the increase of ammonium was not significant enough to affect negatively the SCOD:NH_4^+ and this ratio was increased (250 %) to ~ 70 which is much higher than the theoretical requirements for nitrogen removal (8-14 for TCOD:TKN) (Carlson *et al.*, 1996, Grady *et al.*, 1999). Whether the extra soluble carbon produced by the deflaker can be utilised by the bacteria and improve both denitrification and phosphorus removal needs to be examined experimentally in further research.

4. Conclusions

- The examined disintegration technique, the deflaker proved to be able to increase the soluble carbon in the form of VFA and SCOD.
- The increase in concentration of VFA, SCOD, proteins and carbohydrates was related to the time of disintegration and the source of the sludge, with non BNR SAS resulting in higher values, with the maximum concentration of VFA and SCOD at 852 mg l^{-1} and 6530 mg l^{-1} , respectively.
- The increase in the concentration of phosphorus during the disintegration of BNR SAS reaching 480 mg l^{-1} for 15 minutes of retention time led to a relatively low ratio of SCOD:P (~ 13), which indicated that the addition of disintegrated sludge

would deteriorate the process. The sidestream stripping of phosphorus immediately after the disintegration is considered essential in order to have an improvement of BNR performances.

- Particle size distribution showed that the deflaker decreased and destroyed the porous flocs and microflocs releasing the organic matter that links particles together.
- High performance size exclusion chromatography (HPSEC) confirmed that at short disintegration time (2-10 minutes or 2000-8000 kJ kg⁻¹ TS) the deflaker was able to release the extracellular material, while disintegration for longer period (≥ 10 minutes or 9000 kJ kg⁻¹ TS of specific energy) caused cell lysis. No cell lysis was observed for the non BNR sludge.
- The addition of 15000 kJ kg⁻¹ TS of specific energy increased SCOD significantly, from which 73 % probably came from floc disruption and breakage and the rest from cell lysis.
- Further research will examine the application of disintegrated sludge on BNR, as extra carbon source. Firstly, lab scale tests will investigate the suitability of the released carbon on BNR enhancement and then tests on a pilot plant will examine the conditions under which this internal carbon can be applied.

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Table 1 Summary of the samples collected from different WWTPs with different solid content

	Solid concentration (%) for different samples of thickened SAS						
BNR WWTP	5.8	5.9	4.3	6.3	5.2	7.2	6.1
Non BNR WWTP	4.3	6.2	5.6	5.2			

Table 2 VFA, SCOD and P release from thickened BNR SAS (4.3-7.2 % T.S.) treated with deflaker for different retention times

	Raw		2 minutes		5 minutes		10 minutes		15 minutes	
	Ave	Max	Ave	Max	Ave	Max	Ave	Max	Ave	Max
VFA (mg l⁻¹)	3	19	113	113	185	263	205	299	403	530
SCOD (mg l⁻¹)	176	342	1525	1845	2383	2780	3060	4175	4440	6180
Sol. P (mg l⁻¹)	107	159	216	216	232	242	253	300	326	480
NH₄⁺ (mg l⁻¹)	8-10								50-60	

Table 3 VFA, SCOD and P from thickened non-BNR SAS (4.3-6.2% T.S.) treated with deflaker for different retention times

	Raw		2 minutes		5 minutes		10 minutes		15 minutes	
	Ave	Max	Ave	Max	Ave	Max	Ave	Max	Ave	Max
VFA (mg l⁻¹)	236	311	486	486	541	772	611	611	852	852
SCOD (mg l⁻¹)	1004	1180	3745	3745	4110	5500	6040	6410	5810	6530
Sol. P (mg l⁻¹)	46	69	59	59	62	77	78	78	100	135

Table 4 SCOD:P and SCOD:NH₄⁺ ratios produced in sludge by disintegration for different retention times

	Raw	2 minutes	5 minutes	10 minutes	15 minutes
	BNR SAS				
SCOD:P	1.6	7	10	12	13.6
SCOD:NH₄⁺	20				70
	Non BNR SAS				
SCOD:P	21	63	66	77	58.1



Figure 1 Pilao 10" spider deflaker

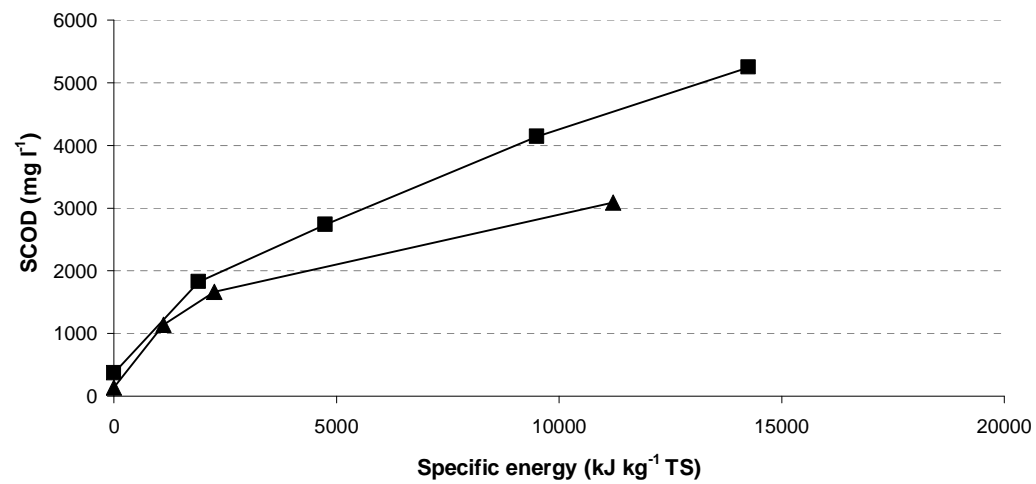


Figure 2 Effect of specific energy on SCOD for BNR sludge with two different solid concentrations (■ 7.2 % TS, ▲ 6.1% TS)

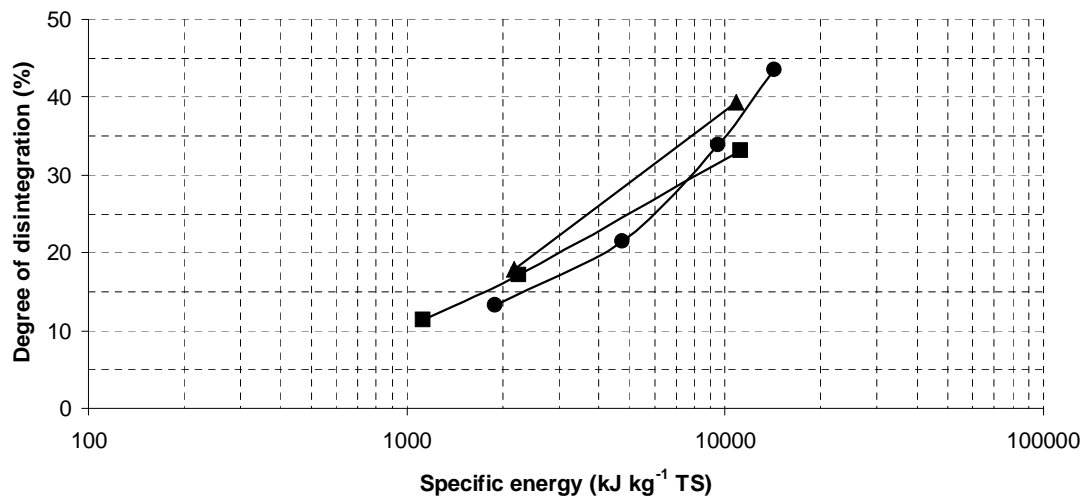


Figure 3 Degree of disintegration by the deflaker as a function of specific energy for three different sludge samples (● BNR SAS 7.2 % TS, ■ BNR SAS TS 6.1% TS, ▲ non BNR SAS 6.2% TS)

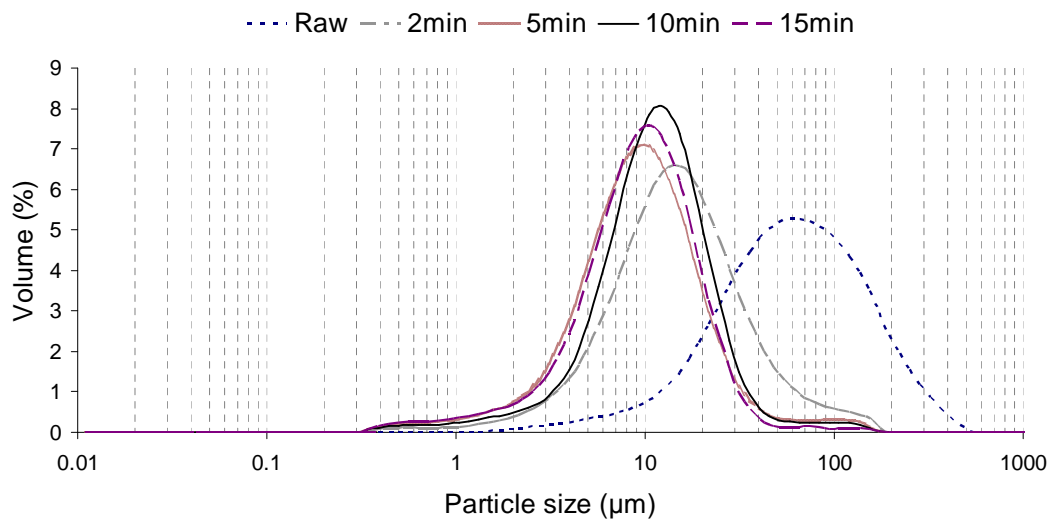


Figure 4 Particle size distributions for raw and disintegrated thickened BNR SAS

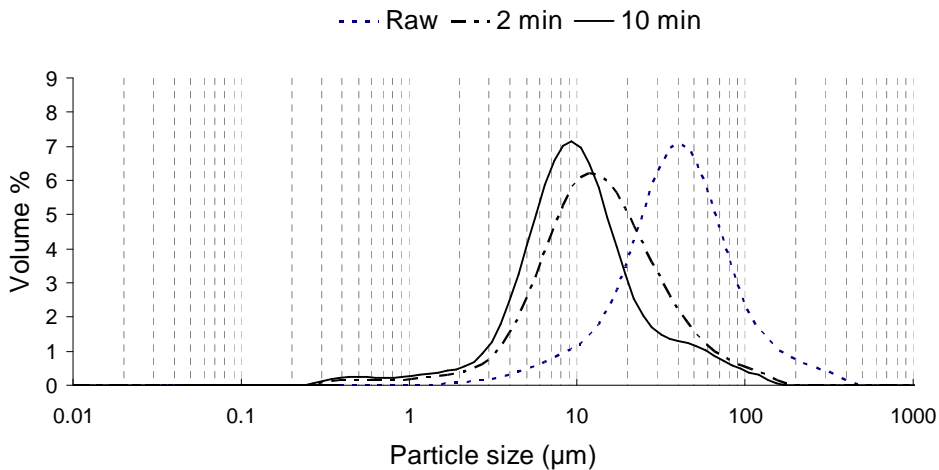


Figure 5 Particle size distributions for raw and disintegrated thickened non BNR SAS

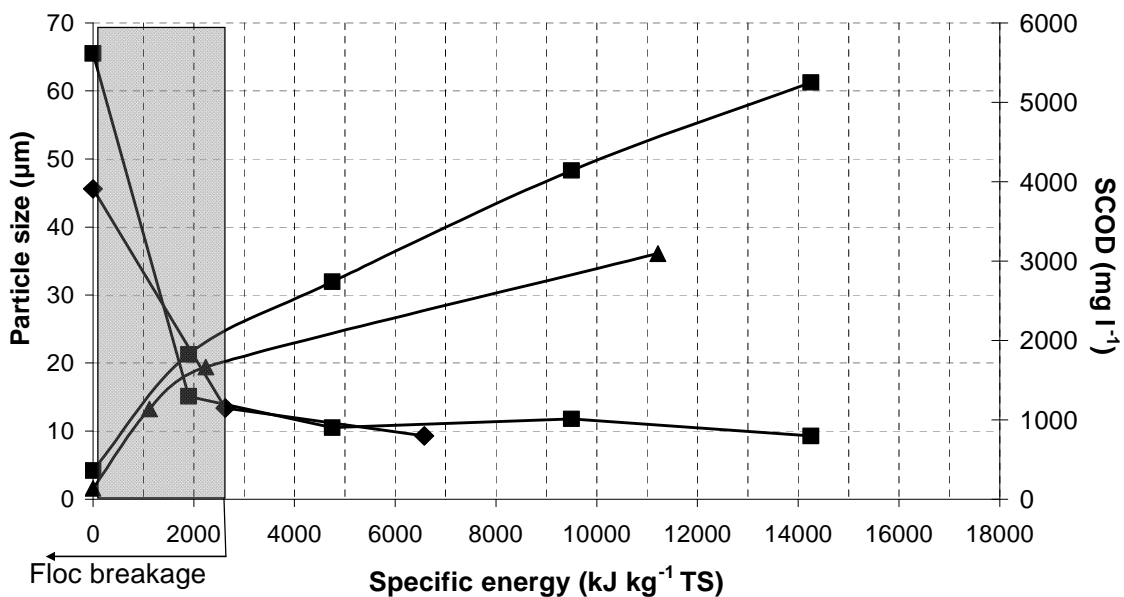


Figure 6 Effect of specific energy of disintegration on particle size and SCOD by deflaker for three sludge samples (■ BNR SAS 7.2 % TS, ▲ BNR SAS 6.1 % TS and ◆ non BNR SAS 5.2 % TS)

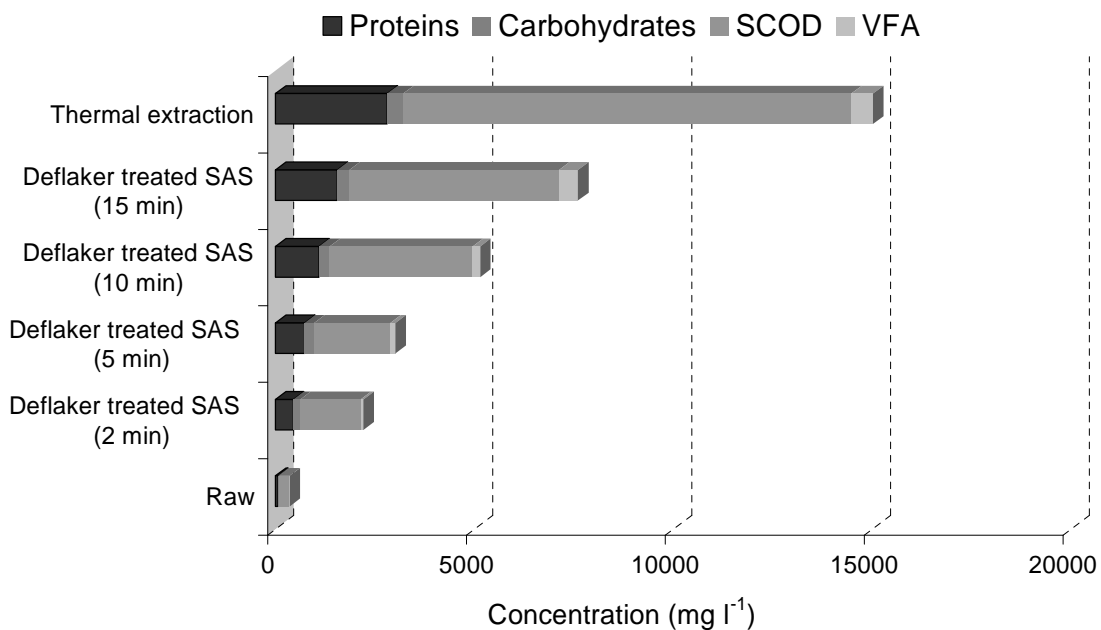


Figure 7 Disintegration results for BNR thickened SAS (7.1 % TS)

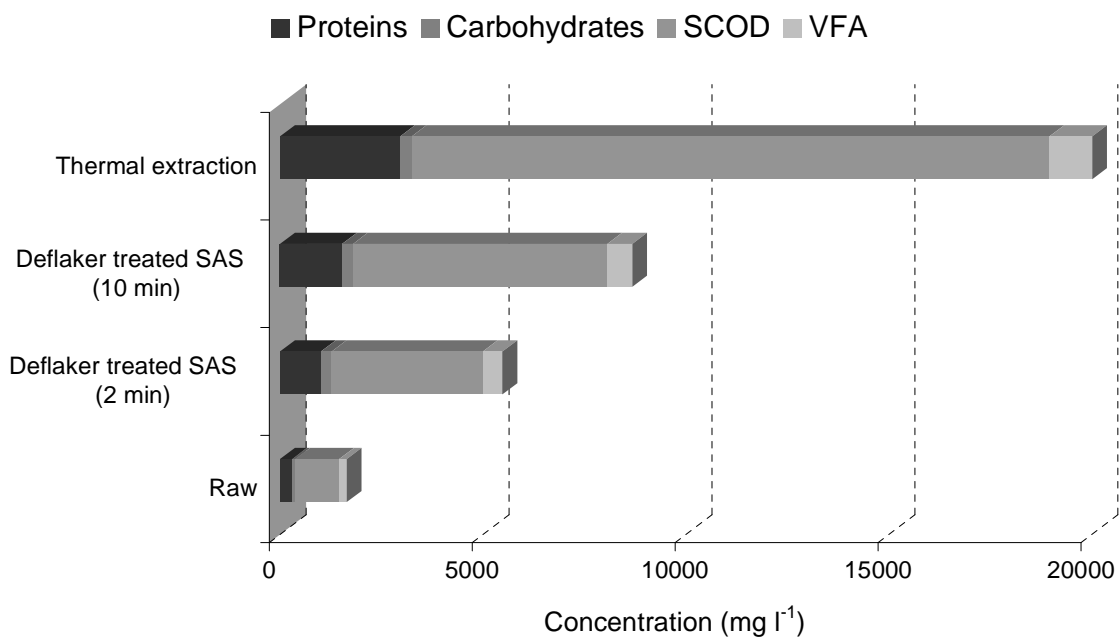


Figure 8 Disintegration results for non BNR thickened SAS (6.2 % TS)

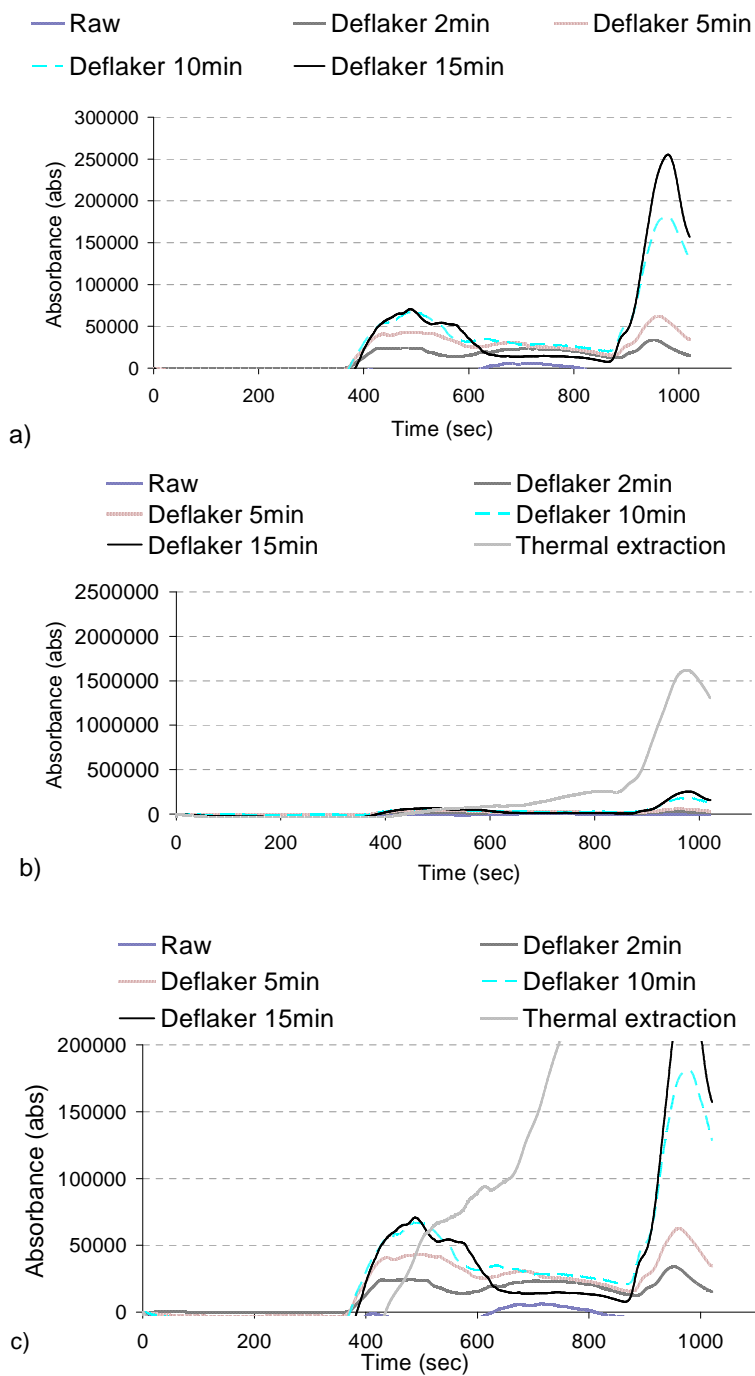


Figure 9 HPSEC results for BNR thickened SAS a) disintegration from deflaker, b) disintegration from deflaker compared with thermal extraction and c) the same as b) in different scale observing the absorbance of the large molecular size material.