

## ENZYMIC HYDROLYSIS TECHNOLOGY DEMONSTRATION - PRODUCTION OF ENHANCED TREATED BIOSOLIDS FOR AGRICULTURAL RECYCLING



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### ABSTRACT

The Sixth Community Environment Action Programme re-instates Europe's commitment to a sound use of sludge on land and the LIFE Instrument is one of the Measures aimed to achieve the long-term goal of 75% of urban sludge as being suitable for land spreading in the whole of the EU. The Blackburn Sludge Treatment Scheme was chosen by LIFE to demonstrate best practice. Thermophilic pre-treatments were the preferred bolt-on upgrades for the Blackburn Digestion facility to guarantee an enhanced treated sludge for agricultural recycling. As conventional thermal pasteurisation techniques with their short treatment times and high temperatures do not allow sufficient biological activity to develop, the United Utilities Enhanced Enzymic Hydrolysis (EEH) process was selected for its combined advantage of simple yet robust operation and optimum enzyme activity at 55°C. Operation at 55°C allows the plant to guarantee the minimum 6-log *E. coli* reduction using only low-grade heat. This is important, as it would enable any standard CHP scheme to use a greater proportion of the biogas for electricity production. To date the first full scale EEH plant at Blackburn has shown excellent pathogen control capability with indicative VS destruction rate of 55%, even under highly overloaded hydraulic conditions. The enhanced treated cake has been used as a seedbed fertiliser for maize and cereal crops. It has proved to be of low odour and has provided the opportunity for surface spreading on grassland, even in close proximity to residential areas. Increasingly the ability to produce a low odour product will help to make sludge recycling more sustainable where the land bank is limited and more acceptable to the public.

### KEYWORDS

Anaerobic digestion; enzymic hydrolysis; pathogen; sewage sludge; agricultural recycling

### INTRODUCTION

The key principle of the UK Government's vision for sustainable waste management is based on recognising that waste has value and can be moved up the adopted Waste Hierarchy. In this context the agricultural recycling of biosolids (sludge) is regarded as Best Practical Environmental Option. This is also reflected in Europe's Sixth Community Environment Action Programme, which includes the sustainable use of natural resources and management of wastes (Decision No 1600/2002/EC). To this end the Commission is putting forward a proposal to re-instate its commitment to a sound use of sludge on land and expects to see an increase in the quantity of sludge destined for land-spreading across the EU in the next five to ten years. Measures are also proposed to achieve the long-term goal of 75% of urban sludge suitable for land spreading in the whole of the EU. One of the spearheads of Community environment policy is LIFE, the Financial Instrument for the Environment (LIFE website 2006). LIFE works towards the implementation of Community policy defined by the Sixth Action Programme for the Environment based on a practical approach.

The specific objective of LIFE-Environment is to contribute to the development of innovative techniques and methods by co-financing demonstration projects. LIFE contributes to the implementation, development and enhancement of the Community environmental policy and legislation as well as the integration of the environment into other EU policies. LIFE also supports the development of new solutions to environmental problems facing the EU. Enzymic Hydrolysis is seen as an innovative sustainable solution for the treatment of sewage sludge for agricultural recycling and as such it qualified for LIFE support. The Blackburn project was selected for Technology Demonstration, which set out to show the technical capability of the demonstration plant as well as the economic and environmental impact of its operation. This paper constitutes a major output of the

Monitoring programme.

## **SLUDGE DIGESTION AND ENZYMIC HYDROLYSIS**

Sludge digestion has been known for over a century, but it was not widely practised until more recently. For example, not so long ago treatment simply meant keeping sludge in lagoons to achieve odour, pathogen and volume reduction. The advent of digesters allowed the process to be speeded up and to make biogas for heating. The start of the cessation of sludge disposal at sea in 1998 was met with concerns about the safety of the agricultural recycling of sludge. Thereafter, the BSE crisis also heightened public disquiet in relation to food safety. The “safe sludge matrix” was an agreement between the British Retail Consortium and the Water Industry, brokered by DEFRA in response to public concerns. In recent years, escalating energy prices and growing interest in renewable energy sources have also increased interest in new methods to improve the performance of sludge digestion.

Clearly, the nature of digestion requirements has not changed, but the stakes are now much higher and performance demand has become more exacting. In order to operate the process successfully and be able to achieve the ever increasing performance target one needs to have a greater understanding of the process mechanisms, its potential and limitations. This is not a trivial task as sludge is not a well-defined substrate and the interplays between the digester microbiology and hydrodynamics are so complex that attempts to elucidate key process aspects like mixing and foaming have met with little success so far. Many operators have established empirical relationships based on their knowledge and experience with the process in order to design and operate their own digestion assets. For example, United Utilities Asset Standards require primary digesters to achieve an average hydraulic retention time of 16 days (based on active volume & average feed volume). This assumes sludge feed dry solids of 4% to 8%, and an operating temperature of 35°C. The process is to be completed with 14 days retention minimum (batch) in a secondary digester to achieve adequate pathogen reduction.

For United Utilities the principal motivation in seeking process improvement during AMP3 was HACCP compliance to provide confidence to the various stakeholders in a sustainable way. The Enzymic Hydrolysis process was developed against this backdrop. It has allowed a step change in pathogen reduction performance without any major changes in digestion practice. The process has relatively low energy demand and its simplicity of operation and ability to be retrofitted to existing plants has been widely recognised.

More recently, there has been a surge in interest in Enzymic Hydrolysis as a means for improved volatile solid (VS) destruction and concomitant increase in biogas yield. It is widely known that surplus activated sludge (SAS) contains more water and lower proportion of fermentable organic matter per mass unit than Primary sludge. González et al (2006) stated that the destruction of volatile and organic matter achieved through anaerobic digestion of this type of sludge is about 30%, a value that is significantly lower than the 50% commonly obtained with other types such as Primary sludge. In the UK, it is common practice to combine SAS and Primary sludge before digestion and this typically produces a VS destruction rate of about 45%, as reported by Panter (2006). Traditionally, it was held that increasing digester retention time tended to increase the level of VS destruction; however, in practice a comprehensive Performance Audit by United Utilities of all its digesters in AMP3 showed that there was little correlation in digester retention time and VS destruction performance (Figure 1). There is a general expectation and it is almost taken for granted that any hydrolytic pre-treatment would be accompanied by improved VS destruction. This stemmed from the early work of Ghosh and Klass (1977) that showed operations with separated acid (hydrolytic) and methane phases resulted in increased biogas production and volatile solids destruction. The first full-scale sewage sludge treatment plant with separated acid gas phases (commonly known as two-phase digestion) in DuPage County (Illinois) has been operating successfully since 1991. A recent review by Wilson *et al* (2005) showed that by 2004 there were 8 known two-phase digestion plants in operation plus 13 others either being built or at advanced design stage. Interestingly, they showed that in some trials 80-90% VS destruction was obtained. In the same review these authors showed that on average two-phase digestion plants were achieving 55% VS destruction compared to 45% for single-phase digesters under comparable retention times (16 days average). Table 1 shows a performance summary of the first three United Utilities digestion plants with Enzymic Hydrolysis. Average VS destruction of the plants ranged from 54 - 63% for the stated periods (Note that the HRT of the Crewe digesters varied considerably in the period). The VS destruction rates seen so far represent a considerable improvement in performance comparing to previous levels (Figure 1).

The solid to gas conversion rate and gas quality are other aspects of two-phase digestion that have been a subject of interest. The Manuals of Water Pollution Control (1979) suggested that 0.62 to 1.18 m<sup>3</sup> gas /kg VS destroyed should be expected. The wide range reflects the large variability in the constituents of the sludge and also the difficulty in obtaining comparable gas data using different types of gas meters under different temperature and pressure condition. The cause of the low apparent gas yield for Bromborough (Table 1) requires further investigation. In a two-phase digestion plant, up to 25% of the volatile solids may be converted to soluble substances or gases in the acid phase. Ghosh *et al* (1995) suggested that the biogas from the acid phase was typically 67% CO<sub>2</sub> and 30% methane and accounted for 16% of the overall biogas production. Shana *et al* (2006) reported similar results for Thames acid phase digester, but also indicated that the overall biogas quality was virtually unchanged in comparison with biogas from a single-phase digester. These findings are in line with United Utilities expectation and highlight the need for good biogas blending to ensure consistent performance of all gas burning equipment.

## **THE BLACKBURN SLUDGE TREATMENT SCHEME**

Blackburn is a major sludge centre for the Northern region. Every year the centre processes 13,500 tonnes sludge (tDS) by Mesophilic Anaerobic Digestion (MAD) for 0.5M people from the Blackburn and South Lancashire area and safely recycle it to agriculture as a valuable fertiliser for farmers. However, the introduction of the Safe Sludge Matrix at the end of 1998 required more stringent control of pathogens in sludge. As most of the accessible land bank within 25 miles of the works is grassland, this called for the highest standard of treatment. Thermophilic pre-treatments are the preferred upgrades for MAD facilities that need to guarantee an enhanced treated product for agricultural recycling. While conventional thermal pasteurisation techniques can achieve the required pathogen standard, their short treatment times and the high temperature do not allow sufficient biological activity to develop. On the other hand, the Enzymic Hydrolysis process originally developed by United Utilities to improve MAD performance proved to be an excellent method for generating hydrolytic activities. By combining the advantage of proven operation of the original process at 42°C with optimum enzyme activity at 55°C United Utilities created EEH, a hybrid process that aimed to improve VS reduction and biogas yield whilst achieving excellent pathogen control capability at the same time. The technology is particularly appealing because of its low operating temperature enabling it to work successfully with low-grade heat such as waste heat from CHP plants that maximise the Renewables Obligation Certificates. Also, in the future the process could be run to provide a valuable source of VFA for biological nutrient removal applications.

## **MATERIALS AND METHODS**

### **Analytical methods and procedures**

- 1) *Sludge samples*: All sludge samples were spot samples. Hydrolyser samples were taken from dedicated sampling ports from individual reactor tanks. Digested samples were fresh samples taken directly from the discharge points of the digesters. Analysis followed within 6 hours of sampling or within 24 hours if kept at 4°C.
- 2) *Filtered samples*: Sludge samples were filtered through Munktell MGC 150 mm filters. Filtrate was analyzed directly for COD, total VFA (TVFA), and ammonium using Dr. Lange cuvette test kits, LCK 114, 365, and 303 respectively, with a Dr. Lange LT200 incubation block and Lasa50 spectrophotometer.
- 3) *Speciated analysis*: Components of volatile fatty acids, were analyzed with a Varian 3400 gas chromatograph equipped with a Chromosorb 101 (80/100 mesh) column (Length: 2.5 m, diameter: 2.3 mm) and a flame ionization detector (FID).
- 4) *Other analyses*: All other analyses were done by United Utilities Lingley Mere Laboratories, using accredited methods. *E. coli* enumerations were carried out with either the membrane filter method (conventionally treated samples) or the Most Probable Number Method (enhanced treated samples).

### **Enhanced Enzymic Hydrolysis Plant**

Whilst 70°C is the popular temperature for thermal pasteurisation systems (Cumiskey 2005), it presents a number of difficulties. High sludge temperatures require high water temperatures (typically +80°C) and this in turn leads to scaling and potential sludge baking problems. The standard Enzymic Hydrolysis plant, with 2 days retention at 42°C comes very close to achieving the required 6 log pathogen reduction required for Enhanced Treated (Mayhew *et al*, 2004) and all that was required was a polishing stage, rather than a bolt-on pasteurisation plant. Operation at 55°C allows the plant to guarantee the minimum 6-log *E. coli* reduction using only low-grade heat. This is important, as it

would enable any standard CHP scheme to use a greater proportion of the biogas for electricity production.

The initial concept for the plant was to introduce a batch hold time into the thermophilic stage to ensure that all the sludge was held at 55°C for a fixed period of time. This prevents short-circuiting and allows a simplified HACCP procedure for the plant. However to convert a plug flow reactor with 6 tanks in series (Figure 2) to a half plug-flow, half batch system required some ingenuity. The solution was to use reactor 4 as a balancing tank. Reactors 5 and 6 alternate between being the digester feed tank and the batch hold tank for the thermophilic sludge. This system allows the plant to operate the mesophilic stage in plug flow and the thermophilic stage as a batch hold, whilst maintaining a continuous feed to the digesters. To minimise drives and simplify the process the sludge recirculation pumps for the heat exchanger are also used to transfer sludge from reactor 4 to reactors 5 & 6.

The EEH plant at Blackburn STW was designed to be compliant with the prevailing United Utilities Asset Standards at the time. The aim was to pre-treat 540m<sup>3</sup>/d (average flow) of sludge to achieve at least 6-log reduction of *E. coli* before digestion. The feed sludge was pre-thickened by gravity belt thickeners and fed to the plant from an air-mixed buffer tank. Following 2 days retention (at the design peak flow of 741m<sup>3</sup>/d) in the Hydrolyser the sludge is fed to the 4 existing anaerobic digesters, each with a nominal working volume of 2,200m<sup>3</sup> (assuming an active volume of 95%). Each digester was equipped with 3 external Heat-a-Mix units powered by a single piston-type gas compressor.

## RESULTS AND DISCUSSION

### Hydrolyser and Digester performance

Commissioning of the Blackburn Hydrolyser started in November 2005 and the plant was put into beneficial use in January 2006. Comprehensive monitoring of the plant as part of the EC LIFE project evaluation programme was initiated in early January. A full report of the plant performance will be available at the end of 2006.

The Blackburn Hydrolyser has been fully operational for 9 months by the time of writing (September 2006). Apart from several brief interruptions due to faulty control valves, the plant has seen loading from 200m<sup>3</sup>/d up to the maximum design throughput without any difficulties. However, operational experience downstream of the Hydrolyser has been mixed. Although there are 4 digesters at Blackburn, digester 1 has been out of service for the first 4 months due to a mixer problem. Figure 3 provides a summary of the throughput regimes for the 4 individual digesters. Since mid April 2006 all digesters have experienced intermittent mixing problems. At the time of writing all 4 digesters are in service but with only 50% of the mixing power (2h on 2h off). This compromise allows the digesters to achieve full throughput at a reduced VS destruction rate while awaiting the arrival of new mixers.

Following is a summary of the digestion plant performance data for the first four months of the monitoring period. The 3 working digesters had to cope with an average daily load of 600m<sup>3</sup>/d during this period, which was well in excess of United Utilities Asset Standards. It is interesting to note that despite the mixing problems and low hydraulic retention times (HRT of between 10 and 11 days) the digesters have been stable throughout as indicated by the low VFA level (generally < 100mg/L) and no significant foaming episodes. This is consistent with findings from the other 3 sites with Enzymic Hydrolysis, which have operated for several years.

Table 2 provides a summary of the Blackburn process streams analysis from mid January to the end of April. The average VS destruction rate during this period was 55.1%. This value should be taken as indicative since it was based on very limited data which was collected during a period during which the digesters have suffered mixer failures. On the other hand, the Enzymic Hydrolyser performance has been consistent throughout the monitoring period. Figure 4 shows that the total amount of VS converted in Hydrolyser fall mostly in the 10 - 30% band (average 18%). The conversion rate is based on the VS balance between the input and output of the Hydrolyser. It is thought that some of the converted materials would appear as biogas (mainly CO<sub>2</sub>) and the remainder as soluble substances (ammonia and soluble COD components). Although Hydrolyser conversion rate is a good indicator of Hydrolyser performance, at present there is no obvious way of correlating this parameter to digester performance. This is an interesting area for further work.

### Production of Volatile Fatty Acids

Evidence of the working of hydrolytic enzymes and acidogenic microbial activity in the EEH process can be seen in the SCOD and TVFA profiles of the plant (Figure 5). The VFA composition did not change over the process (Figure 6). Both the soluble COD and the VFA levels increased at a constant rate during the mesophilic phase. However, while the rate of VFA production seemed to plateau out after reactor tank 3 where the process temperature was raised to 55°C, the soluble COD level continued to rise. This observation was consistent with the results of Roberts (1998, see Table 3) which indicated that short reactor retention times at 55°C were insufficient for the development of thermophilic acidogenic activity. Shifting the process temperature from 42°C to 55°C inactivates the mesophilic bacteria responsible for acid fermentation but still allows hydrolytic activity to operate.

Trends in the soluble COD production did not indicate an influence of the change in process temperature on the hydrolysis rate. One might expect that hydrolytic enzymes could increase in activity with temperature until denaturation impairs enzyme function. With no observable temperature effect, it is possible that a balance between denaturation and increased activity resulted. Alternatively, other factors such as mass transport kinetics may be rate limiting hydrolytic function.

The observed rates for hydrolysis and acidogenesis for the process at the nominal loading conditions of 5.0 % dry solids with 400 m<sup>3</sup>/d sludge throughput were not necessarily characteristic of the process. Similar effluent SCOD and VFA levels were achieved over a range of sludge flow rates (200 to 740m<sup>3</sup>/d) with otherwise relatively constant influent dry solids content. The influence of loading and temperature on hydrolytic and acidogenic activity requires further investigation. Strategies to gain greater benefit from the retention time under thermophilic conditions with respect to hydrolysis and acidogenesis are under consideration.

### **Pathogen reduction performance**

The main purpose of the current project was to improve the pathogen destruction capability of the digestion process to enable the sludge to be safely recycled to agriculture. Good and reliable pathogen destruction performance was clearly vital for the success of the project.

Table 2 shows the analytical results of samples from the various process streams. It was found that in the conventional mode of operation the EEH process was able to destroy 99.95% of *E. coli* and 99% *Salmonella*. In the enhanced mode of operation the EEH process was able to destroy 100% *Salmonella*. The destruction of *E. coli* was beyond the level of detection (LOD) for this indicator micro-organism. It should be noted that currently there is no reliable method for the enumeration of *E. coli* below 100cfu/gDS. This is generally regarded as the Limit of Detection (LOD) for *E. coli*. The transition from operation in the conventional mode to enhanced mode cause a marked change in the *E. coli* content of the digested sludge can be seen from Figure 7. It is clear that despite the erratic performance of the digester mixers and the highly hydraulic overload conditions in the digesters, the EEH plant at Blackburn has been able to provide an excellent pathogen control capability consistently achieving the enhanced treated standard for sludge agricultural recycling.

### **Recycling of digested cake from Blackburn works**

The market for spreading liquid sludge on grassland has become constrained by concerns over nitrate leaching and the high costs of storing liquid sludge during closed periods. Consequently, the use of digested cake will help to maintain the land bank for sludge recycling in grassland-dominated North West. Apart from the pathogen risk, the agronomic properties of the products are also of interest to farmers. Table 5 shows that nutrient and micro-nutrient contents of the Blackburn cake are very typical of biosolids. The levels of heavy metals are also well within the more stringent limits currently being proposed for a new EU Directive.

Cake from Blackburn is recycled mainly within the Fylde area of Lancashire. There are large arisings of animal manures in the area, which is also under the new Catchment Sensitive Farming initiative, and parts are within a nitrate vulnerable zone. Farmers have previously been supplied with conventionally treated sludge cake and limed cake. So far in 2006 the enhanced treated cake has been used as a seedbed fertiliser for maize and cereal crops. Many farmers do not perceive the new product as different from what they have received previously. Yield response to the two products tends to be similar. However, the advanced product has made a significant improvement in odour performance. It has proved to be of low odour and has provided the opportunity for surface spreading on grassland, even in close proximity to residential areas. Increasingly the ability to produce a low

odour product will help to make sludge recycling more sustainable where the land bank is limited and more acceptable to the public.

## CONCLUSION

The Sixth Community Environment Action Programme re-instates Europe's commitment to a sound use of sludge on land and the LIFE Instrument is one of the Measures aim to achieve the long-term goal of 75% of urban sludge suitable for land spreading in the whole of the EU. The Blackburn Sludge Treatment Scheme was chosen by LIFE to demonstrate best practice. Thermophilic pre-treatments were the preferred bolt-on upgrades for the Blackburn Digestion facility to guarantee an enhanced treated sludge for agricultural recycling. As conventional thermal pasteurisation techniques with their short treatment times and high temperatures do not allow sufficient biological activity to develop, the United Utilities EEH process was selected for its combined advantage of robust operation and optimum enzyme activity at 55°C. To date the first full scale EEH plant at Blackburn has shown excellent pathogen control capability with indicative VS destruction rate of 55%, even under very high hydraulic overload conditions. The enhanced treated cake from Blackburn has proved to be of low odour and has provided the opportunity for surface spreading on grassland. Increasingly the ability to produce a low odour product will help to make sludge recycling more sustainable where the land bank is limited and more acceptable to the public.

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## TABLES AND FIGURES

**Table 1** Performance Summary for United Utilities digestion plants with Enzymic Hydrolysis

	Macclesfield Jan 04 – Aug 05	Bromborough (Jan 04 - Aug 06)	Crewe (Oct 05 - May 06)
Average feed (m <sup>3</sup> /d)	173.4	235.0	See main text
Estimated digester HRT (days)	16 (75% active volume)	16 (75% active volume)	See main text
Feed DS% (VS%)	4.86 (74.4)	6.23 (75.6)	7.00 (72.0)
Digested DS% (VS%)	2.53 (59.3)	2.86 (61.0)	3.79 (60.7)
VS destruction %	59.0	62.9	54.3
Apparent gas yield m <sup>3</sup> / kg VS destroyed	1.08	0.68	1.10

**Table 2** Summary of the Blackburn EEH process streams analysis

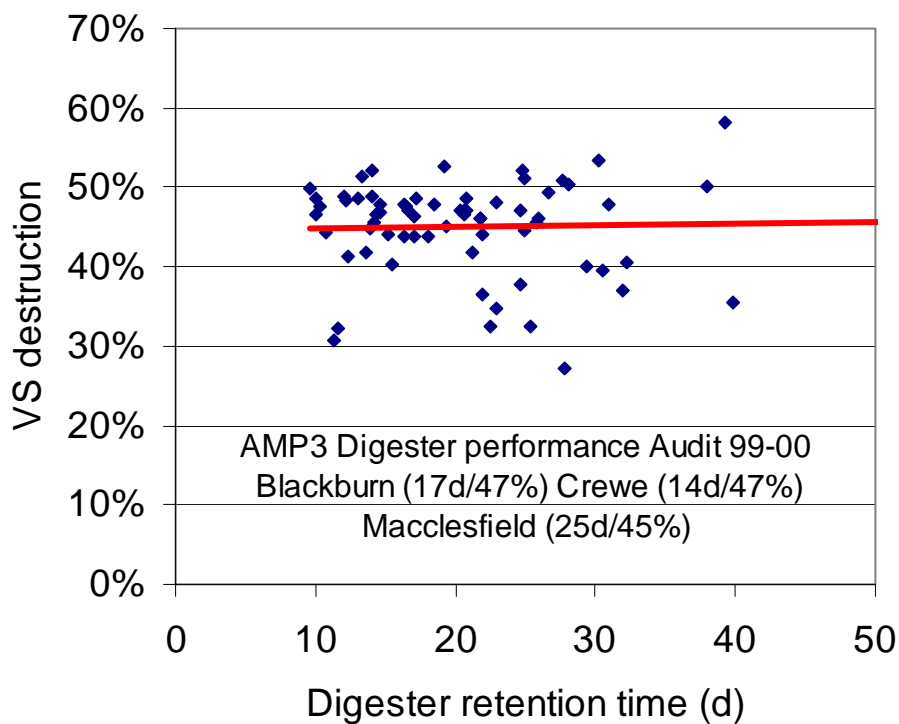
	<b>Alkalinity</b>	<b>Ammonia</b>	<b>pH</b>	<b>DS</b>	<b>VS</b>	<b><i>E. coli</i></b>	<b><i>Salm.</i></b>
	mg/L	mg/L		%	%	cfu/gDS	No./2gDS
<b>Raw sludge feed</b>							
	1,655	240	5.89	4.83	71.81	6,276,477	1,545
<b>Treated sludge (conventional mode operation, Jan - Feb 06)</b>							
Dig2	2,512	657	7.24	2.64	60.76	3,432	14
Dig3	2,403	736	7.10	2.65	59.88	4,110	16
Dig4	2,848	762	7.33	2.87	57.02	3,140	13
<b>Treated sludge (enhanced mode operation, Feb - April 06)</b>							
Dig2	3,049		7.21	2.41	59.84	<LOD	0
Dig3	2,878		7.22	2.40	59.63	<LOD	0
Dig4	3,014		7.18	2.80	58.46	<LOD	0

**Table 3** Summary of Thermophilic pre-treatment performance from Roberts (1998).

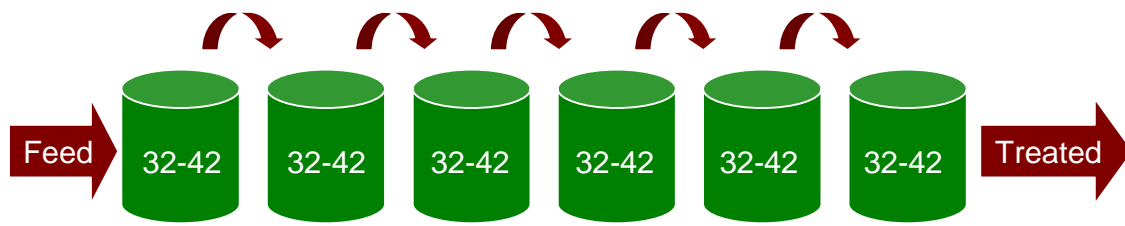
<b>Mean values (over 61 days)</b>	<b>DS</b>	<b>VS</b>	<b>pH</b>	<b>Alkalinity</b>	<b>tVFA</b>
	g/L	g/L		mg/L	mg/L
Raw feed	27.71	18.97	6.51	1,915	2,336
TAnD 4h HRT @55°C	25.80	17.22	6.61	2,103	2,543
TAnD 8h HRT @55°C	26.45	17.42	6.83	2,285	2,385
TAnD 12h HRT @55°C	25.46	16.86	6.86	2,390	2,271

**Table 4** - Heavy metals and nutrients in biosolids in comparison with proposed EU Limits

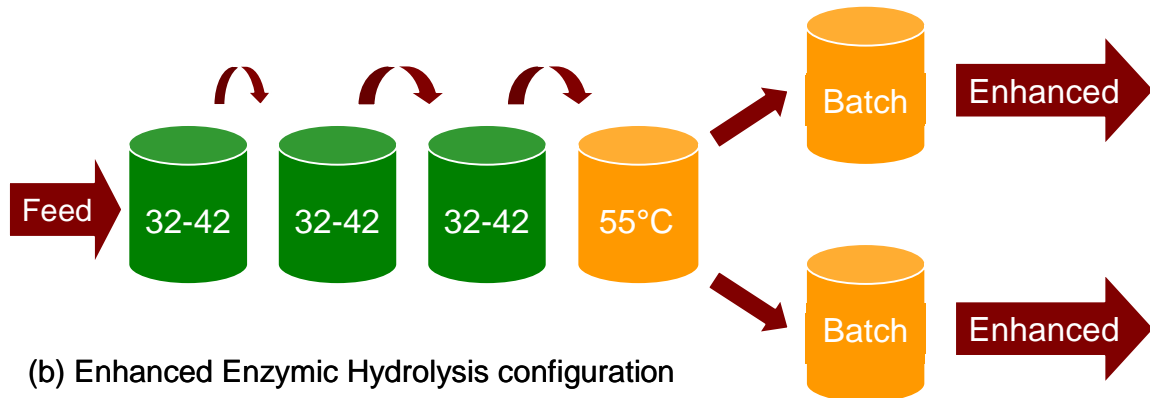
Elements	Blackburn	EU Limit values	Blackburn	EU Limit values
	(mg/kg dry solid)	(mg/kg dry solid)	(mg/kg P)	(mg/kg P)
Cd	1.50	10	62	400
Cr	70.93	1,000	2,956	40,000
Cu	287.50	1,000	11,979	40,000
Hg	1.10	10	46	400
Ni	34.37	300	1,432	12,000
Pb	163.50	750	6,813	30,000
Zn	781.75	2,500	32,573	100,000
N	38,800			
P	24,000			
S	6,900			
Mo	9.10			
Se	2.11			



**Figure 1** Relationship between digester retention time and VS destruction



(a) Conventional Enzymic Hydrolysis configuration



(b) Enhanced Enzymic Hydrolysis configuration

Figure 2 Process Flow Diagram of the Blackburn EEH Plant

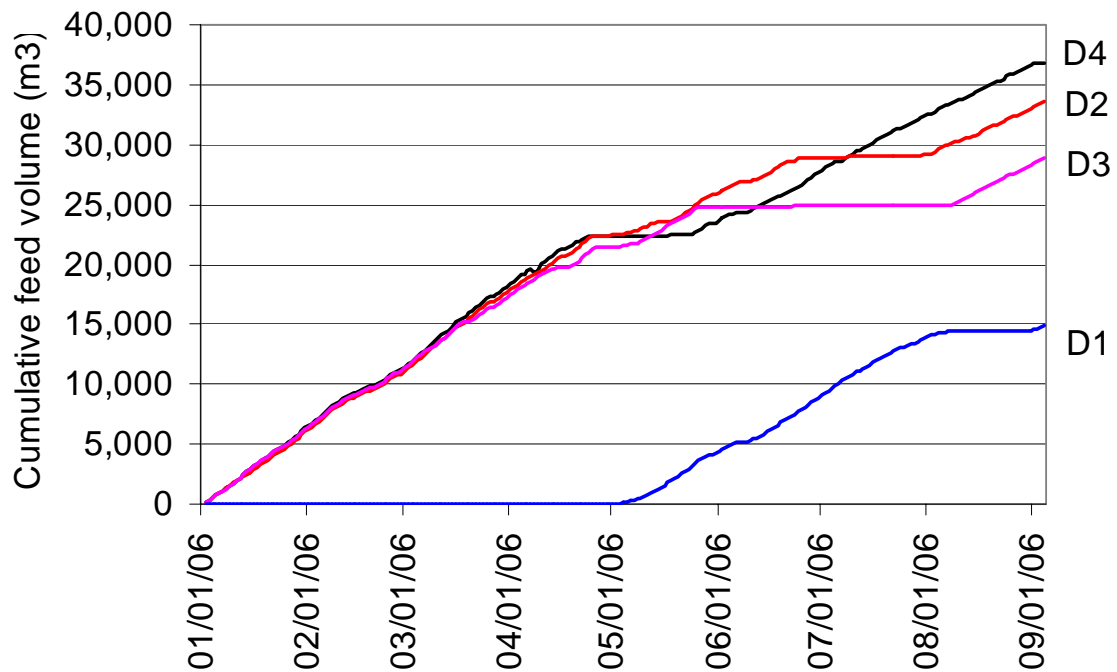


Figure 3 Throughput regimes for individual digesters at Blackburn

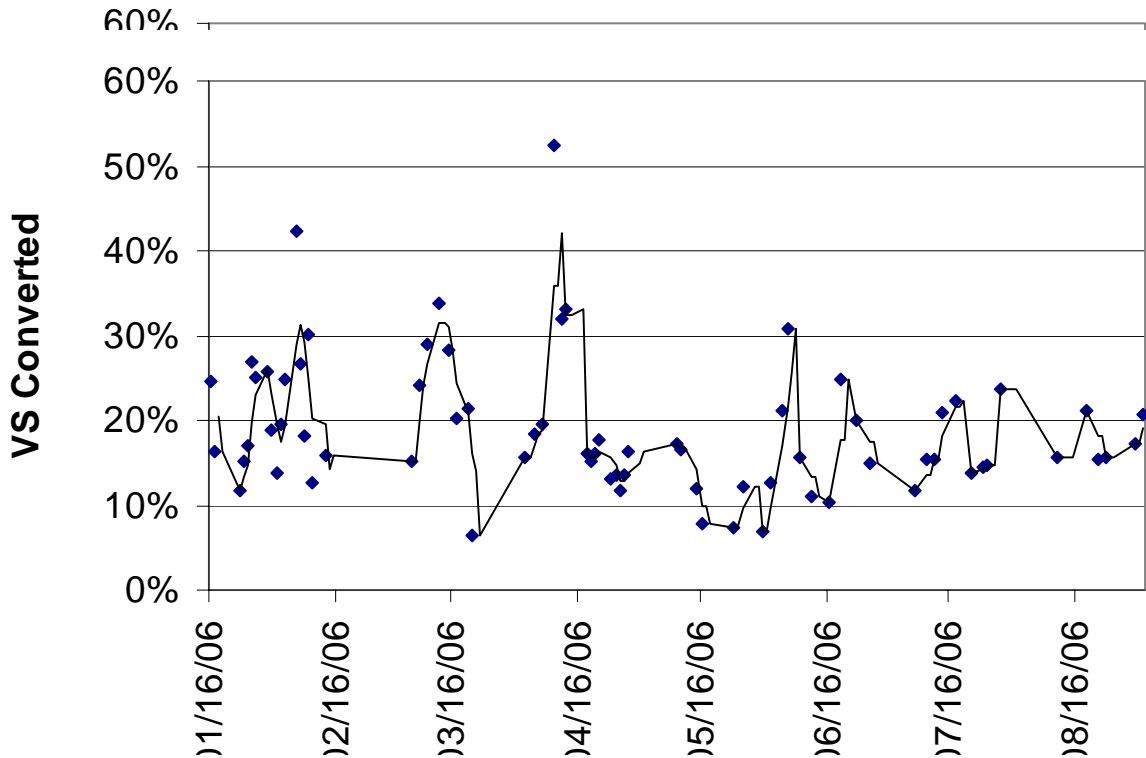


Figure 4 Total amount of VS converted in the Blackburn EEH plant

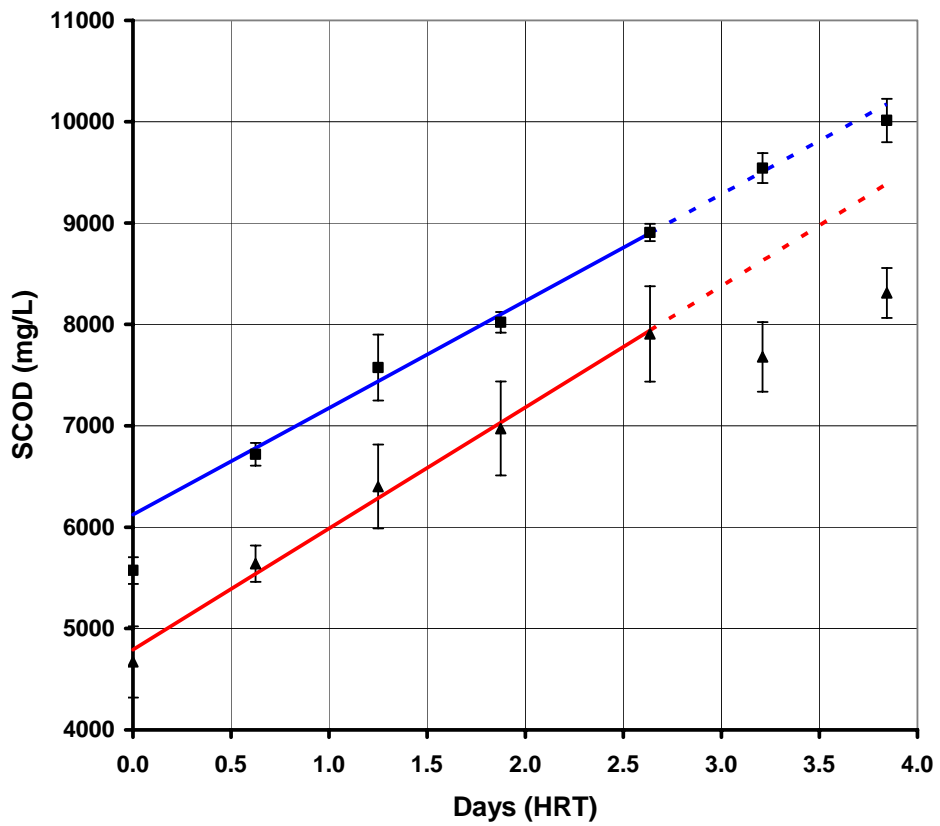


Figure 5 Typical trends in SCOD release (■) and TVFA (as COD) production (▲) in the EEH process

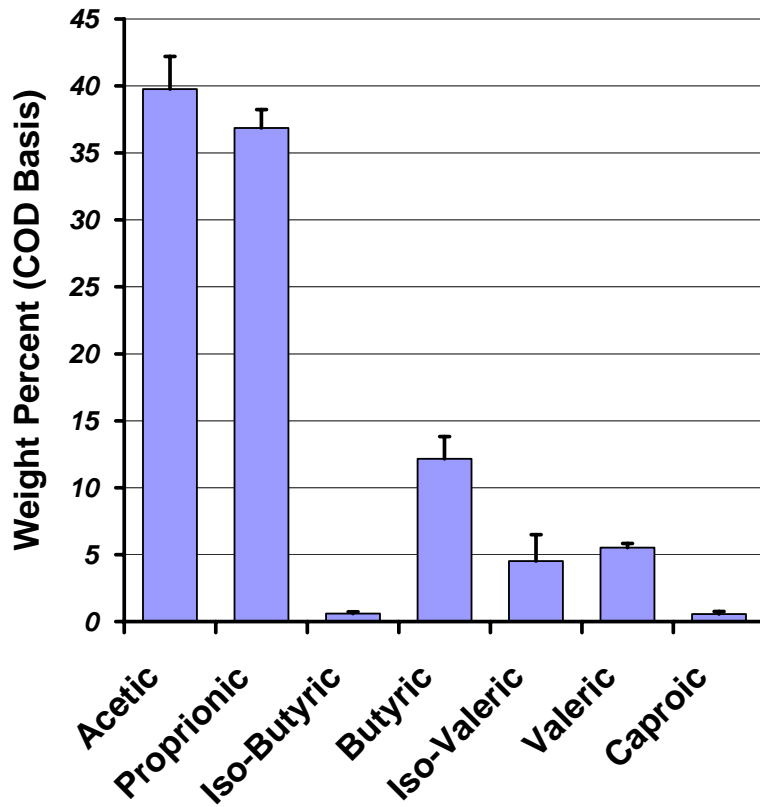


Figure 6 Average VFA composition on a COD mass basis from EEH feed to effluent

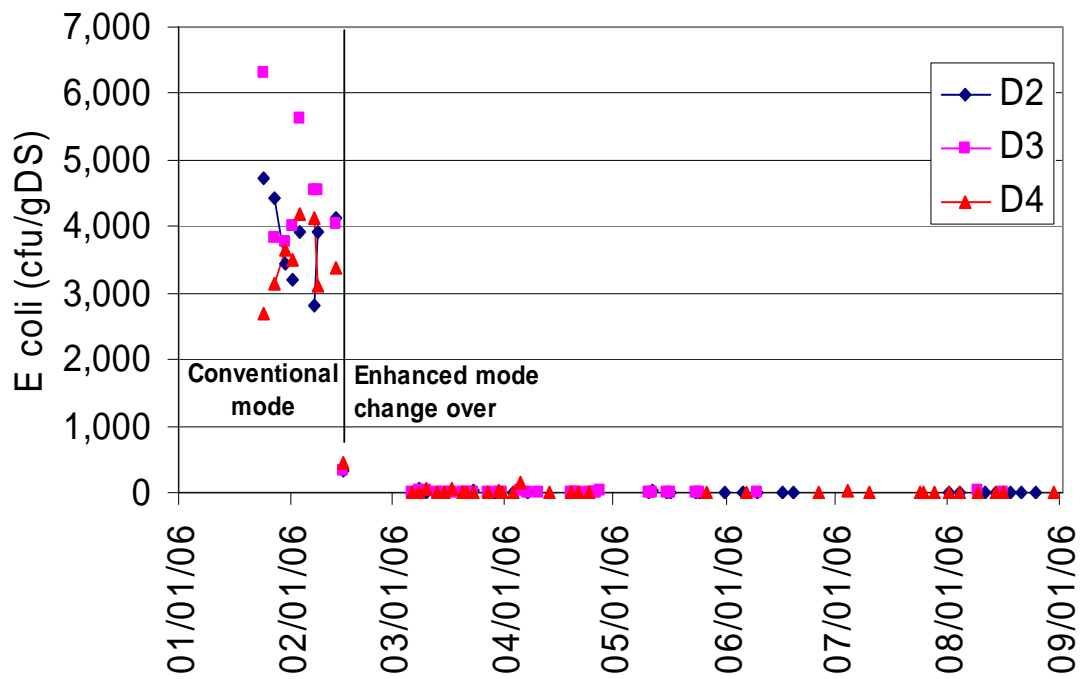


Figure 7 E. coli reduction performance of the Blackburn digestion plant