FINAL REPORT

ANAEROBIC DIGESTION OF BLEACH EFFLUENT A PILOT PLANT- AND BENCH SCALE STUDIES

by

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SAPPI CSIR UHDE

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TITLE:

ANAEROBIC DIGESTION OF BLEACH EFFLUENT A PILOT PLANT- AND BENCH SCALE STUDIES

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ABSTRACT: Anaerobic digestion was considered for the treatment of bleach effluent as an organic and sulphate removal step. This investigation formed part of an overall project, the bleach chemicals recovery process.

> A pilot plant investigation using an ADUF (Anaerobic Digestion Ultra Filtration) pilot plant was performed. The principle of the ADUF process is to combine ultrafiltration with anaerobic digestion to retain the sludge. In order to cover different aspects of anaerobic digestion bench scale studies were also performed by UHDE (Pty) Ltd and the CSIR.

Good sulphate and chlorate removal was possible but COD (Chemical Oxygen Demand) reduction was low. Packed bed digestion performed better than sludge blanket digestion (Bench scale study). Results from the completely mixed digester (pilot plant) were better than those from the other reactor types (bench scale). No significant difference was observed between high temperature digestion (thermophylic) and low temperature digestion (mesophylic) - bench scale study. Significant biogas production was observed with the pilot plant operating in the completely mixed mode. (Very little biogas was produced in the other studies).

KEYWORDS:

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EFFLUENT TREATMENT, BLEACH PLANT, BIOLOGICAL PROCESSES, ANAEROBIC DIGESTION

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SINOPSIS

ANAEROBIC DIGESTION OF BLEACH EFFLUENT - A PILOT PLANT- AND BENCH SCALE STUDIES

Anaerobiese behandeling van die bleikuitvloeisel met die oog op vermindering van die sulfaat- en organiese inhoud was deel van 'n oorhoofse projek ~ Die bleikchemikalieë herwinningskonsep.

Bankskaalstudies deur die WNNR en UHDE (Mpy) Bpk asook 'n loodsaanlegstudie deur SAPPI is onderneem. 'n ADUF (Anaerobic digestion ultrafiltration) loodsaanleg wat van ultrafiltrasie gebruik maak om die slyk te behou is vir die loodsaanlegstudie gebruik.

Goeie afname in sulfaat- en chloraatkonsentrasies kan bereik work maar CSB (Chemiese suurstofbehoefte) reduksie was minder suksesvol. Die gepakte bed verteerder is met die slykkombers verteerder vergelyk en eersgenoemde het beter presteer (bankskaalstudie). Die resultate van die volledig gemengde verteerder (loodsaanleg) was beter as die van die ander tipes verteerders (bankskaal). Geen noemenswaardige verskil tussen hoë temperatuur vertering (termofilies) en lae temperatuur vertering (mesofilies) is waargeneem nie (bankskaalstudie). Beduidende biogas produksie is waargeneem met die loodsaanleg in die volledig gemengde modus - maar baie min in die ander studies.

1. INTRODUCTION

Aerobic digestion was considered as an organic and sulphate removal step, as part of an overall process - bleach chemicals recovery process. Due to the following advantages over other biological processes:

- (a) Energy recovery via biogas production
- (b) Sulphur recovery via hydrogen sulphide production
- (c) Low energy requirements
- (d) Low nutrient requirements
- (e) Low sludge production

it was considered as an economical favourable process.

A pilot plant study was undertaken at the Ngodwana mill. The pilot plant was operating on a side stream of the bleach effluent (Initially a mixture of 4:1 D/C (bleaching stage): E (extraction stage) effluent and later straight D/C effluent). Subsequently it was also decided to contract the CSIR to compare the mesophylic (low temperature) with the thermophylic (high temperature), and the sludge blanket with the packed bed digestion processes. Lastly a company UHDE (Pty) Ltd was also contracted to evaluate the packed bed anaerobic reactor, and especially the applicability of the French patented, S.G.N. plastic packing material.

2. CONCLUSIONS

- 1. It is possible to achieve the following reduction in concentration, of components in the D/C effluent, with the anaerobic digestion process.
 - 1.1 Chlorate 100% reduction
 - 1.2 Sulphate 50-80% reduction
 - 1.3 Chemical oxygen demand (COD) 30-35% reduction

- 2. The bench scale studies indicated that the packed bed digestion process performs better than the sludge blanket process.
- 3. Very good performance was achieved with the pilot plant in the completely mixed mode. The results were better than any of the other studies.
- 4. No significant difference was observed in the results from the mesophylic (low temperature) and the thermophylic (high temperature) digestion processes.
- 5. Very low methane production was observed in all the studies except for the completely mixed digester (pilot plant) and it was concluded that the activity of the methane producing bacteria was inhibited by compounds in the effluent, or by competition with sulphate reducing bacteria for energy sources.
- 6. The low COD reduction can be attributed to the low anaerobic biodegradability of the toxic fraction, which was found to be more aerobically biodegradable (3).

3. RECOMMENDATIONS

- 3.1 Based on the results obtained anaerobic digestion can be recommended for chlorate and sulphate removal. Further work must however still be done to maximize COD removal.
- 3.2 The ADUF process completely mixed digester combined with ultrafiltration is recommended for another study. The opinion is held that knowledge gained on anaerobic digestion and ultrafiltration can make another anaerobic digestion study on bleach effluent very valuable.
- 3.3 It is recommended that the following be considered for future pilot

plant work:

- 1. The need for the addition of nutrients
- 2. The possible advantage of treating a mixture of D/C and E effluent
- Dilution of the raw feed with a recycle stream to reduce shocks at the point of contact between sludge and feed.

4. LITERATURE REVIEW

4.1 Definitions

Bacteria can be classified as aerobic - those that can live only when oxygen is present, anaerobic - which require that oxygen not be present, and facultative - which can live either in the presence or absence of oxygen.

Aerobic digestion (9) can be defined as a bacterial fermentation, by which organic matter is broken down in the absence of dissolved oxygen, to produce a mixture of carbon dioxide and methane gases.

Bacteria can also be classified in 3 groups according to the temperature range in which they can exist. The psychrophiles exist in the psychropilic (10) temperature range $-5 \rightarrow 20^{\circ}$ C. The mesophiles exist in the mesophilic range $-25 \rightarrow 40^{\circ}$ C - with optimum system performance (7) at $35 \pm 3^{\circ}$ C. The thermophiles exist in the thermophilic range - $40 \rightarrow 60^{\circ}$ C - with the optimum temperature = $55 \pm 2^{\circ}$ C. System performance can falter near 42° C as this represents the transition from mesophilic to thermophilic organisms.

The normal bacterial population in waste water treatment can survive in a range of pH from about 5 - 9, but optimum operation will occur if the pH is around neutral (about $6,5 \longrightarrow 7,5$).

4.2 Advantages and Disadvantages of anaerobic digestion

Advantages

- 1. Energy recovery via production of biogas
- Sulphur recovery via production of hydrogen sulphide (if sulfates are present)
- 3. Lower energy requirements
- 4. Lower nutrient requirements
- 5. Lower sludge production
- Sludge can be stored during relatively long shut-down periods and re activated in a short period of time

Disadvantages

- 1. Difficulty in treating diluted or cold effluents
- 2. The methane producing step is relative sensitive to pH changes, shock loadings and sulphur concentrations
- 3. Relatively long start-up periods during which the process is relative sensitive
- 4. Post treatment is necessary due to odorous compounds

4.3 Reactor types

Different anaerobic reactor types (5) were developed for full scale treatment of effluents. The <u>anaerobic lagoon</u> is a low rate reactor and mixing is only by the evolution of the biogas. The <u>contact reactor</u> a high rate reactor - is a closed tank with an agitator followed by a settling tank. In the <u>UASB (Upflow anaerobic sludge blanket)</u> system a high rate reactor - the feed is introduced through a distribution system at the bottom. It flows upward through a layer of micro organisms (sludge blanket). A separation system is installed at the top of the digester to separate sludge particles, gas and treated effluent. In the fluidized bed reactor - a high rate process - the effluent flows through a fluidized bed of micro organisms attached to a carrier material. A certain minimum flowrate is needed to keep the bed fluidized. In a <u>fixed bed digester</u> - a high rate process - the organisms grow either on the surface or in the void space of a packing material (usually a plastic material with a large specific area). The effluent may be passed downward or upward through the bed.

4.4 Pelletization

Little is still known about all the factors contributing to the formation of a pelletized sludge with sufficient strength to resist shear, and at a sufficient rate to compensate for washout.

The following can however be presented as criteria for pelletization (11) or better said as ecological conditions under which pelletization is likely to occur.

- 1. An environment with a high partial pressure of hydrogen
- 2. A nitrogen source
- 3. A limited source of cysteine
- 4. A neutral pH

Two important conditions under which pelletization is not likely to occur seem to be:

- A system where the influent substrate does not yield hydrogen in the fermentation processes
- 2. In a completely mixed reactor

4.5 Fermentation steps

The fermentation of a carbohydrate, lipid or protein substrate to methane gas seems to take place in four stages (11) involving three groups or organisms - solubilization and acidogenesis by acidogenic

organisms, acetogenesis of short chain fatty acids by acetogenic organisms and methanogenesis by methanogenic organisms.

<u>Stage 1 - Solubilization/hydrolysis:</u> Non soluble organic compounds are hydrolysed by enzymes excreted from acidifying bacteria (extracellular process (11)). The rate of this process is relatively slow and this is often regarded as the rate controlling step (6).

Stage 2 - Acid formation: The hydrolyzed compounds are converted into organic acids such as lactic acid, butyric acid, propionic acid and acetic acid.

Stage 3 - Acetogenesis: Organics of stage 2 are converted to acetic acid, hydrogen and carbon dioxide.

Stage 4 - Methanogenesis: Methane forming bacteria convert the products from stage 3 into methane.

Sulphate is utilized by sulphate reducing bacteria. Hydrogen sulphide is the major end product, although elemental sulphur or organic sulphurous compounds may also be formed (4). Sulphate reducing bacteria use the same energy sources and thus compete with the methane producing bacteria. The sulphate reducing bacteria are also more effective and less sensitive to pH and temperature variations which cause a lowered methane production.

4.6 Treatability of bleach effluent

The practical COD limit for high rate anaerobic processes is in the range $1000 - 30\ 000\ g.m^3$ (5,8).

A screening study was done (5) on effluent streams from different paper mills. The samples which showed the greatest inhibition were predominantly bleach plant waste waters from Kraft mills. Chlorinated organic compounds were the most likely source of inhibition. The COD reduction in the samples of bleach effluent was an average 35% with the value for some samples below 20%. Only about 35% of the COD reduction could be correlated with the methane production.

5. EXPERIMENTAL WORK

5.1 Experimental work - Pilot Plant

The pilot plant investigation was divided into two stages for convenience of discussion. Stage one was the period during which completely mixed conditions were maintained in the digester. For stage two changes were made to have conditions in the digester approaching that of a sludge blanket.

5.1.1 Stage 1 - Completely mixed digester

During this period the pilot plant was operated in the ADUF (Anaerobic Digestion Ultrafiltration) mode with completely mixed conditions prevailing in the digester. ADUF mode implies incorporating ultrafiltration as a method of retaining the anaerobic sludge. The sludge is then recycled to the digester.

The pilot plant consisted of a $5m^3$ dome shaped insulated digester. Gas removal was via a water trap and gasmeter. The digester was designed to overflow (at the $3m^3$ level) into a 6001 overflow tank. The feed was introduced via a sparge ring and flow inlets - two were bent to give a circular motion and two were straight to enhance proper mixing.

As mentioned a bank of tubular ultrafiltration membranes was used to separate the sludge from the product. The concentrate from UF unit contained the sludge and was thus recycled. The permeate was the product stream (it was put to drain during the pilot plant study). The membranes were fed from the overflow tank.

The concentrate was combined with the raw feed, heated in a tubular heat exchanger and fed to the digester. The pH was read in the line feeding the membrane unit. Adjustments were made by dosing a saturated MgO solution in the overflow tank. Ammoniumcarbonate additions for alkalinity adjustment as well as diammoniumdiphosphate additions for nutrient requirements were done in the overflow tank.

Three methods were evaluated for membrane cleaning namely:

- 1. Sodiumhypochlorite solution
- 2. Sodiumhydroxide solution
- 3. Enzymatic detergent (Biotex)

The enzymatic detergent (Biotex) was found to be most effective.

5.1.2 Stage 2 - Sludge blanket

The pilot plant configuration during stage one, made it very difficult to adjust the effective residence of the feed in the digester. The digester was operated in the completely mixed mode and it was dependant on the concentrate recycle stream to maintain these conditions. Secondly the feedrate which could be handled in the plant depended on the rate of permeate produced by the UF unit. Based on this, it was decided to change the pilot plant configuration to make the operation of the digester independent from the UF unit. At the same time it was hoped that pelletization of the sludge would be enhanced by maintaining operating conditions in the digester favourable for a sludge blanket.

The aim was thus to keep the option open to operate the plant in

the ADUF mode but only to incorporate the UF membranes after the process has stabilized. This configuration would of course also make it possible to evaluate the process without the UF unit.

A new distribution system was installed at the bottom of the digester. This was fed from a secondary sparge ring. A piston type reciprocating pump (dosing type) was installed as feedpump. The overflow tank was incorporated as an external settling tank although it was used as is, with no additional settling aids. Provision was make to feed the membranes from this tank on a level control basis. When using the membranes the concentrate would be returned to this tank (and not directly to the digester as before). The underflow from this tank was returned to the digester via the original sparge ring with the four inlets. When the membranes were not used the overflow from this tank was the final product for the process.

The level in the digester was changed from $3m^3$ to the $5m^3$ level. A stainless steel coil was also installed in the digester for temperature adjustment of the digester contents. Provision was make for hot/cold water circulation.

5.1.3 Trials

The reader is referred to Table 1 & 2 as well as Figures 1 to 5 presented in the appendix for a summary of the trials and the results obtained.

5.1.3.1 Stage 1

During stage one various operational problems were encountered. Temperature control was very difficult, pumps gave problems and due to a free chlorine shock (7 ppm) activity of the bacteria was lost - additional sludge was loaded. Nevertheless, when analysing the data it is clear that the pilot plant performance during stage 1 was better than during stage 2. In fact the results even seem to be better than those obtained during the CSIR and UHDE studies (see discussions under 5,2 and 5,3).

As can be seen from Table 1 and Figures 2 and 3 the sulphate - and COD reductions were the highest of the whole period - over 50%. It was also during this period that most gas formation was observed. (Accurate gas readings were not possible due to level changes in the digester).

It is difficult to provide reasons for the good results obtained during this period. The following can be presented as possible contributing factors to the performance. Firstly the feed to the pilot plant was made up by mixing D/C (bleaching stage) and E (extraction stage) effluents in the ratio of 4:1 (D/C:E). It is possible that the E stream was contributing to the better performance by diluting the toxic compounds in the D/C stream to a less inhibiting level for the bacteria. Secondly and probably more important is the fact that the digester was operated in the completely mixed mode. This factor differentiates the pilot plant study from both the CSIR and UHDE studies.

It is however important to see these results against the background of the very low space loading rate 0,2 - 0,48 kg COD/m^3 day. (Space loading rate is calculated from the sludge volume in the digester, the volumetric feedrate, and the COD content of the feed e.g. 213 $1/h/3m^3 \times 1596 \text{ mg COD}/1 = 0,113 \text{ kg COD}/m^3 \text{ day}$.

5.1.3.2 Stage 2

Operational problems during stage two were less prominent. Temperature control with the coil was improved however due to the urgency of the bleach chemicals recovery project there was a great need for Therefore it was decided to increase the results. feed to the anaerobic plant dramatically. It was thought worth while to take the risk although the plant was not performing well. The feedrate was increased in the period 32-36 weeks, to a peak space loading rate of 1,4 kg COD/m^3 day in week 35. For the rest of the period the space loading rate was very low - 0,3 kg COD/m^3 day.

During the last few weeks of the trial period the anaerobic plant was operated on a very low priority basis. Due to more positive results produced in other pilot plants of the same overall project, it was decided to preferably keep them running during periods of low effluent availability.

The data representing stage 2 - week 26 and onwards reflects the poor performance of the pilot plant during this period. Even during periods of very low space loading rates COD and sulphate reductions of less than 40% was experienced. It can be seen from Table 1 that very low reductions were sometimes experienced. No conclusions could be made from the performance when higher space loading rates were used. The performance of the plant was bad before the increase in space loading rates and it continued to perform badly.

5.1.4 Analytical Results

A summary of the analytical results is presented in Table 2. By viewing the results for the different components it is clear that for chlorides, silicates, sodium, calcium, magnesium and iron very little change took place over the anaerobic digester. Chlorate reductions were good and it can be assumed to be 100%.

The phosphate analysis indicates an increase on various occasions. This was due to the addition of diammoniumphosphate for nutrient requirements. The nutrient dosage was never optimized. The rule of thumb: COD:N:P in the ratio of 500:15:3 was used during the pilot plant study for calculation of quantities for nutrient dosage.

5.2 Bench Scale Studies - CSIR

The CSIR was contracted to perform a bench scale study to evaluate the following: Firstly mesophylic versus thermophylic digestion, and secondly the fixed film reactor was to be compared with the sludge blanket reactor/digester. In addition a comparative study was also done on the effect of sugar and hydrogen respectively as additional energy source.

No significant difference was observed in the results from the thermophylic and mesophylic digestion processes. When evaluating the two different reactor types - sludge blanket and fixed film - it was found that the fixed film reactor could tolerate external changes better and gave better performance on the whole. Start up performance of the sludge blanket reactors were however better.

The results obtained indicated that a COD reduction of 35%, a sulphate reduction of >80% and a chlorate reduction of 70% could be achieved by a retention time of 4 days.

Hydrogen gas was found to be a viable energy source for anaerobic digestion of the bleach effluent. The retention time was shortened to 20 hours with COD reduction the only parameter not positively enhanced by addition of the hydrogen gas.

It was concluded that anaerobic digestion is not the ideal process for COD removal from the D/C stage bleach effluent. The combination processes were suggested as alternative to only anaerobic treatment namely: a combined anaerobic - aerobic process or a combined anaerobic - activated carbon process.

5.3 Bench Scale Study - UHDE (Pty) Ltd

A three month bench scale study was undertaken to investigate the suitability of the S.G.N. (Societe Generale) fixed film anaerobic digestion process, to treat the D/C bleach effluent. The aim with the study was to evaluate the fixed film process and especially by using the S.G.N. plastic packing material.

It was however found that the biodegradability of the effluent was very low - an average COD reduction of 35% was achieved. Methane bacteria was shown to be sensitive to the toxic compounds in the effluent. Sulphate reduction was very high - 100% - at low feedrates but lower -50 to 60% - at higher feedrates. Chlorate reduction was complete. Degradation of AOX concentration was 60-85% at lower feedrates but went down to 50% at higher feedrates. Unfortunately insufficient time was available to monitor sulphate and AOX reduction over a longer period at the higher feedrates. As part of the testwork boiling and air stripping of the effluent was also done but no improvement in the biodegradability was observed. It was concluded that anaerobic digestion is not suited for biodegradation of the COD fraction of the D/C effluent because of the presence of toxic compounds. Good results in reducing sulphate and chlorate levels were however achieved.

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8. APPENDIX

FIGURE I	FEEDRATE OVER TRIAL PERIOD
FIGURE 2	COD REDUCTION
FIGURE 3	SULPHATE REDUCTION
FIGURE 4	COD - IN AND OUT
FIGURE 5	SULPHATES - IN AND OUT
TABLE 1	PLANT DATA (WEEKLY AVERAGES)
TABLE 2	ANALYTICAL DATA (WEEKLY AVERAGES)

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COD REDUCTION

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FIG. : 4 COD - IN & OUT Weight averages over trial period.



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TABLE : 1 PLANT DATA (WEEKLY AVERAGES)

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 TABLE 1
 CONTINUES :
 PLANT
 DATA
 (weekly averages)

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TABLE : 2 ANALYTICAL DATA

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TABLE 2 CONTINUES : ANALYTICAL DATA ------

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