

MEMBRATEK (Pty) Ltd

Report to the
WATER RESEARCH COMMISSION

on

**AN INVESTIGATION INTO THE APPLICATION OF THE
ANAEROBIC DIGESTION/ULTRAFILTRATION PROCESS FOR
THE TREATMENT OF METAL-CUTTING-FLUID WASTE WATER**

by

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WRC Report No. 593/1/95
ISBN 1 86845 159 3

**PRETORIA
March 1995**

EXECUTIVE SUMMARY

AN INVESTIGATION INTO THE APPLICATION OF THE ANAEROBIC DIGESTION/ULTRAFILTRATION (ADUF) PROCESS FOR THE TREATMENT OF METAL-CUTTING-FLUID WASTE WATER

PROJECT BACKGROUND

The disposal of metal-cutting-fluid wastewater, generated by machining operations in the automotive industry, is becoming a concern due to environmental considerations. A manufacturer of diesel engines and components currently employs a waste treatment company to collect this wastewater from its manufacturing facility. The effluent is subsequently disposed to a toxic waste dump. This means of disposal may not be tolerated by the authorities in the near future. As such, other treatment methods must be found.

The ADUF process was seen as a potential treatment method since biological processes have been shown to decompose such effluents. Since no previous operational experience with the ADUF process on metal-cutting-fluid wastewater was available, experimental work concerning the biodegradability of this effluent had to be performed.

PROJECT OBJECTIVES

The investigation into the application of the ADUF process to metal-cutting-fluid wastewater, on a laboratory scale, was carried out to determine the following :

- i) biodegradability of the factory effluent by means of mesophilic anaerobic digestion;
- ii) flux values for ultrafiltration at stabilized digester conditions;
- iii) maximum digester load rate and limits of general operating parameters;
- iv) quality of final treated effluent at stabilized digester conditions.

RESULTS AND CONCLUSIONS

COD reduction percentages of more than 85% could be obtained. Final treated effluent COD values were dependant on influent COD level. The ultrafiltration membranes were expected to contribute to this reduction since the organics in metal-

cutting-fluid waste are reported to be 65% anaerobically biodegradable.

The lack of nutrients and alkalinity in the wastewater resulted in poor digester buffer capacity and low load rates. A maximum sludge load rate of $1,05 \text{ gCOD.gVSS}^{-1}.\text{d}^{-1}$ could be achieved.

Long term stability of membrane flux was demonstrated and no cleaning was done throughout the 3 000 h trial period.

PRESENT STATE OF THE ART

The project objectives were satisfied in the sense that the biodegradability of the effluent by anaerobic digestion was established and that stable membrane flux could be demonstrated for the duration of the experiment without resorting to chemical cleaning. A negative aspect proved to be the low digester load rates which were achieved. The experimental results presented in this project report should be seen as an initial phase in the optimization of the ADUF process for this particular application. It may be required to consider additional treatment or a combination of processes in order to address the fraction of the effluent which is not biodegradable.

RECOMMENDATIONS FOR FURTHER RESEARCH

Further work should be performed to investigate the effect of mineral and trace elements additions to the digesters, as well as the inoculation with strains of bacteria which are designed to decompose the specific compounds present in this effluent. This should enhance the anaerobic digestion rate and improve digester load rates to more economic levels.

ABSTRACT

The treatability of metal-cutting-fluid waste water, originating from machining operations in the automotive industry, by the ADUF (Anaerobic Digestion-Ultrafiltration) process was investigated during an initial laboratory scale study.

The lack of nutrients and alkalinity in the waste water resulted in poor digester buffer capacity and low load rates. A maximum sludge load rate of $1,05 \text{ gCOD.gVSS}^{-1}.\text{d}^{-1}$ could be achieved.

COD reductions in excess of 85% were achieved when based on influent and permeate COD. The ultrafiltration membranes are expected to contribute to this reduction since the organics in metal-cutting-fluid waste are reported to be 65% anaerobically biodegradable.

Long term stability of membrane flux was demonstrated and no cleaning was done throughout the 3000h trial period.

ACKNOWLEDGEMENTS

The research results presented in this report emanated from a project funded by the Water Research Commission, entitled:

An investigation into the application of the aduf process for the treatment of metal-cutting-fluid waste water

The financing of the project by the Water Research Commission and the cooperation of Atlantis Diesel Engines are gratefully acknowledged.

LIST OF CONTENTS

1.	INTRODUCTION	1
1.1	ANAEROBIC DIGESTION	1
1.2	ADUF PROCESS	1
1.3	METAL-CUTTING-FLUID WASTE WATER	1
2.	OBJECTIVES	2
3.	MATERIAL AND METHODS	2
3.1	EXPERIMENTAL APPARATUS	2
3.2	SAMPLE ANALYSES	3
3.3	OPERATING CONDITIONS	3
3.4	ANAEROBIC SLUDGE CONDITIONING	4
3.5	ADDITIONAL CONSIDERATIONS	4
4.	RESULTS AND DISCUSSION	5
4.1	METAL-CUTTING-FLUID WASTE WATER	5
4.1.1	Treatment Routes	5
4.1.2	Waste water Characteristics	5
4.2	DIGESTER PERFORMANCE	6
4.2.1	Sludge Concentration and Load Rate	6
4.2.2	COD Reduction	9
4.3	MEMBRANE PERFORMANCE	9
4.3.1	Membrane Flux	9
4.3.2	Fouling	10
5.	CONCLUSIONS AND RECOMMENDATIONS	12
5.1	CONCLUSIONS	12
5.2	RECOMMENDATIONS	12
	BIBLIOGRAPHY	13

LIST OF TABLES

Table 4.1	Cutting oil Feed COD, TKN and P values	6
Table 4.2	Solids content of Anaerobic Sludge	8
Table 4.3	Feed and Permeate COD values	9

LIST OF FIGURES

Figure 4.1	Plot of sludge load rate against operating time	7
Figure 4.2	Plot of pH against operating time	7
Figure 4.3	Plot of volatile acid/alkalinity ratio against operating time	8
Figure 4.4	Plot of membrane flux and temperature against operating time	11
Figure 4.5	Plot of average membrane feed pressure against operating time	11

LIST OF ABBREVIATIONS

ADE	Atlantis Diesel Engines (Pty) Ltd
ADUF	Anaerobic Digestion-Ultrafiltration
BOD	Biological oxygen demand
COD	Chemical oxygen demand
LMH	Membrane productivity expressed in litres per square metre membrane area per hour
MENTUF	Trade name for low-cost ultrafiltration system
MMCO	Molecular mass cut-off
SS	Suspended solids
TKN	Total Kjeldahl nitrogen
UF	Ultrafiltration
VSS	Volatile suspended solids

1. INTRODUCTION

1.1 ANAEROBIC DIGESTION

Anaerobic digestion has become a well established and expanding waste treatment technology which continues to sustain confidence world-wide. Success has been reported for treatment of a very wide range of industrial effluents. Various anaerobic designs are advocated for maintaining high biomass levels, longer sludge retention times and shorter hydraulic retention times which are the key economic factors.

However, none of the current digester designs consistently prevents wash-out of biomass which is a serious technological problem. This problem may be addressed by membrane assisted bioreactors (*Ross et al., 1989*).

1.2 ADUF PROCESS

ADUF is a South African developed membrane-assisted process for the treatment of industrial effluents which eliminates the sludge concentration and retention problems associated with conventional systems. The process utilizes locally manufactured tubular UF membranes instead of imported technology (*Strohwalder, 1991a*).

To date successful laboratory and pilot-scale studies have been carried out on wine distillery, malting, egg, brewery, chemical, fruit and maize-processing effluents. These investigations have culminated in the commissioning and operation of a full-scale ADUF plant for the treatment of a maize-processing effluent at Meyerton (*Ross et al., 1992a*).

The ADUF process is a new technology in South Africa and does not have a long track record on full-scale application. For this reason, there are many questions which cannot be answered at this early stage of its development. The main problems needing further research relate to maintaining high membrane permeate quality and flux, irrespective of the diversity in consistency and composition of feed, as well as long membrane system life as these factors will have a direct bearing on process costs.

Various other research tasks need to be carried out which may confidently be expected to lead to the development of rational quantitative design criteria, more favourable economics and improved reliability. For these reasons experimental work must be conducted on any new feedstock which is under investigation.

1.3 METAL-CUTTING-FLUID WASTE WATER

A manufacturer of diesel engines is facing the problem of treating the metal-cutting-fluid waste water which is generated by machining operations. At present the waste water is removed by a waste disposal company (*Atkins, 1993*). The subsequent treatment involves pH-adjustment to a value between 7 and 9, followed by co-disposal with other waste to a toxic waste dump. It is doubtful whether co-disposal will be allowed in the near future

due to increasingly stringent environmental regulations. There is concern that the present permit, granted to ADE for co-disposal of this effluent, may not be renewed by Water Affairs. Alternative treatment methods for this waste must, therefore, be found.

Since this waste water is very specific to the automotive and metal machining industries, and also differs substantially from domestic waste water, it would be preferable to treat the water at the point of origin. It has been proven that water based metal-cutting-fluids can be treated successfully by anaerobic digestion (*Kim et al., 1992a*). The expectation was that anaerobic digestion combined with membrane separation should result in a final treated effluent of acceptable quality.

A laboratory study to investigate the application of the ADUF process to this type of effluent was, therefore, conducted to investigate the process. Additional treatment to improve final effluent quality did not form part of this investigation.

2. OBJECTIVES

The major aim of this project was to determine the applicability of the ADUF process to the treatment of metal-cutting-fluid waste water from the automotive industry, in particular:

- To investigate the requirements of an ADUF process for the treatment of metal-cutting-fluid waste water.
- To determine the process parameters and limits of the ADUF process when applied to the treatment of metal-cutting-fluid waste water.
- To generate additional design data for scale-up to process plant level.

3. MATERIAL AND METHODS

Experimental work was mainly concerned with the gathering of operational data for process optimization. Efforts were made to note the effect of nutrient addition in instances where there was thought to be a deficiency. Suspended material *e.g.* grit, sand and metal shavings were removed before anaerobic treatment by screening.

3.1 EXPERIMENTAL APPARATUS

Anaerobic digestion was carried out in a stainless steel reactor of 150 l capacity, having an active sludge volume of 75 l determined by the setting of the mechanical float valve. The initial VSS concentration should be between 10 and 15 g/l. Sludge circulation was achieved by means of a positive, diaphragm type pump which could be fitted with appropriate pulley sets for the variation of delivery rate, if desired.

Biomass separation was effected with a MEMTUF ultrafiltration unit of 0,44 m² membrane area (4x10 tube configuration), fitted with

polyethersulphone membranes of 40 000 MMCO. The ultrafiltration permeate was recycled to the digester in order to maintain a constant level inside the digester. A float-operated return valve and gooseneck combination serve to direct excess permeate to drain while maintaining a gas seal.

Biogas was collected at the top of the reactor and the wet gas flow rate was totalized with the aid of a mechanical gas meter. The concentrated biomass was returned to the digester after passing through a heat exchanger. Feed was dosed into the sludge return line, from the feed buffer tank, by means of an adjustable dosing pump.

3.2 SAMPLE ANALYSES

Collection of operating data and chemical sample analyses were performed daily. The performance of the digester was monitored by means of the following parameters (*Ross and Louw, 1987*).

- Volatile acid/alkalinity ratio by Ripley titration (*Ross et al., 1992b*)
- Influent and permeate COD
- Digester and permeate pH
- Digester temperature
- Daily influent volume
- Membrane inlet and outlet pressure
- Membrane flux by time/volume measurements

A periodic nitrogen and phosphate balance was carried out, as well as a determination of SS and VSS to check sludge concentration.

The volatile acid and alkalinity content of the UF permeate were taken as being representative of the digester content since the MMCO of the UF membranes, which were used in the experiment, was too high to result in any retention of low molecular weight organic acids or mineral salts.

The feed rate of metal-cutting-fluid waste water to the digester was varied according to the specific COD content of the effluent to effect a steadily increasing load rate of total COD per day. The ratio of volatile acids to alkalinity, coupled with the COD of the UF permeate, were used as primary indicators of digester performance.

3.3 OPERATING CONDITIONS

Typical operating conditions for the unit were:

- Membrane inlet pressure 150kPa, outlet pressure 50kPa. Adjusted by setting the bypass valve on the pump.
- Digester temperature 37-39°C.
- Digester pH 6,8 to 7,5. Feed to the digester was terminated at pH 6,6.

3.4 ANAEROBIC SLUDGE CONDITIONING

The digester was started with an inoculum from a full-scale anaerobic reactor treating wine distillery waste. The full-scale anaerobic reactor had been inactive when the inoculum was collected in the off season of wine distillery operations. This necessitated the digester of the ADUF unit to be activated with a 8,5 g/l glucose substrate for the first week, after heating the sludge to 35°C. Following initial acclimatization the digester was fed with a 10% metal-cutting-fluid waste and water mixture to which the major nutrients were added. The mixture comprised the following constituents:

■ metal-cutting-fluid waste	100 g/l
■ glucose	1,2 g/l
■ sodium carbonate	1,0 g/l
■ urea as N	0,3 g/l
■ superphosphate as PO ₄	0,1 g/l
■ water	remainder

The decision that this mixture be fed to the digester for the remainder of the investigation, was taken because of the following:

- The metal-cutting-fluid waste water separated, upon standing, into a water and oil fraction. The mixture had an extremely high COD value, tentatively reported as 2123 g/l and caused problems by fouling analytical equipment (*Louw, 1994*). The feedstock for the digester was subsequently derived from the water fraction.
- The water fraction of the waste water contained insufficient nitrogen and phosphorus for bacterial growth.
- The COD of the water fraction was approximately 10% that of the mixture. This, however, was still too high to allow operation at practical hydraulic load rates while maintaining acceptable sludge load rates. The water fraction was therefore diluted.

3.5 ADDITIONAL CONSIDERATIONS

The unit was operated on a continuous basis since the anaerobic process is a biological one. Similarly, shocks in load rates, temperature and pH were prevented where possible, especially during start-up, as the bacteria cannot adapt rapidly to changes in environment.

A rapid drop in temperature, for example, will result in digester overload since the activity of the bacteria halves with each 10°C reduction in temperature. Similarly, the membrane flux will be adversely affected, resulting in an approximate 2% flux drop per 1°C decline in temperature (*Ross, 1990*).

4. RESULTS AND DISCUSSION

4.1 METAL-CUTTING-FLUID WASTE

4.1.1 TREATMENT ROUTES

The automotive industry is facing increasingly stringent regulations with regard to the disposal of effluent from manufacturing operations. Metal-cutting-fluid waste water, in particular, poses a problem in the sense that the recent shift from the use of oil-based towards water-based formulas creates an effluent which is difficult to treat. The reason for this is that the water-based formulations form stable emulsions from which the water is not easily separated.

Three major routes for treating this waste are identified, *viz.* concentration and incineration, biological treatment and disposal to toxic waste dumps.

The concentration and incineration approach requires a separation of water and oil. The oil fraction may be separated from the water by means of ultrafiltration, allowing it to be incinerated while the water fraction is of sufficient quality for disposal or suitable for upgrading and reuse (*Bhattacharyya et al., 1979*).

Disposal of metal-cutting-fluid waste to toxic dumps is becoming unacceptable due to environmental considerations and other treatment methods must be found. This investigation into the treatment of metal-cutting-fluid waste was prompted by this very consideration.

The biological treatment of metal-cutting-fluid waste, however, is receiving increased attention since this treatment route offers the advantages of disposing of the effluent while resulting in a final treated effluent suitable for discharge or reuse. Results reported in the literature indicate that this effluent may be treated aerobically with an activated sludge process (*Baker et al., 1983*) or anaerobically (*Kim et al., 1992a*). Alternatively a combination of aerobic and anaerobic processes can be considered, although the COD reduction by anaerobic/aerobic treatment and straight aerobic treatment were shown to be similar. The different treatment schemes were all able to reduce COD, but a substantial amount of residual organics remained in the treated effluents (*Kim et al., 1992b*). These residual organics appeared to be either non-biodegradable or difficult to degrade.

4.1.2 WASTE WATER CHARACTERISTICS

Water-based cutting oil formulations which are used in machining operations typically contain ethanalamines, polyglycols, chlorinated or sulphonated paraffins, mineral oils and similar compounds (*Baker et al., 1983*). The exact composition is generally proprietary and depends on the particular formulation. As such no effort was made to identify and quantify the various components of the metal-cutting-fluid waste which was treated in the experiment. The waste water was nevertheless analyzed for COD, as well as total nitrogen and phosphorous content which are important parameters relating to load rate and nutrient levels.

The results of these analyses are presented in Table 4.1. The sample of 17/02/94 reflects the actual levels of the parameters which were analyzed. The remainder of the samples, however, reflect constituent levels after dilution with water and the addition of urea and phosphate as described previously. It was attempted to arrive at a COD:N:P ratio of 100:1,6:0,2 as suggested in the literature in order to create suitable conditions for bacterial growth (Ross, 1989).

TABLE 4.1 : CUTTING OIL FEED COD, TKN & P VALUES

DATE	HOUR (h)	COD (g/l)	TKN (g/l)	P (mg/l)	COD:N:P ratio
17/02/94	0	138	1,55	9	100:1,1:0,007
16/05/94	1850	11,5	0,21	65	100:1,8:0,57
15/06/94	2560	14,2	0,29	88	100:2,0:0,62
05/07/94	2990	92	1,19	60	100:1,3:0,07

4.2 DIGESTER PERFORMANCE

The digester was operated continuously for a period of 3000 hours, except for a mechanical breakdown of the metal-cutting-fluid feed pump at 905h which necessitated a two day stoppage. The feed rate to the digester was varied according to the COD content of the waste water and the sludge concentration in the digester in an effort to control the sludge load rate.

4.2.1 SLUDGE CONCENTRATION AND LOAD RATE

The digester was started with a sludge inoculum which had a volatile suspended solids (VSS) content of 10,3 g/l as presented in Table 4.2. The initial sludge load rate (SLR) of 0,12 gCOD.gVSS⁻¹.d⁻¹ could be increased to 0,28 gCOD.gVSS⁻¹.d⁻¹ during the first week of operation while on a glucose feed. A plot of sludge load rate against time is given Figure 4.1.

The introduction of metal-cutting-fluid waste water had no serious effect on the volatile acid/alkalinity ratio, although digester pH dropped from 7,5 to 7,0 as may be seen in Figure 4.2. A VSS reduction from 10,3 g/l to 7,6 g/l was noticed, however, and pig effluent was added to the digester to provide nutrients and minerals. This achieved the desired effect by increasing the VSS to 27,8 g/l at 435h as reflected in Table 4.2.

An effort to increase the SLR resulted in a sharp increase in the volatile acid/alkalinity ratio from 0,25 to 0,68 as shown in Figure 4.3 at 560h. A corresponding decrease in pH was noticed and the feed rate to the digester was reduced to correspond with a SLR of about 0,1 gCOD.gVSS⁻¹.d⁻¹. A second attempt to increase the SLR to 0,7 gCOD.gVSS⁻¹.d⁻¹ at 1000h caused a digester overload as reflected in a corresponding drop in digester pH to 6,5 and a sharp rise in the volatile acid/alkalinity ratio to 0,98.

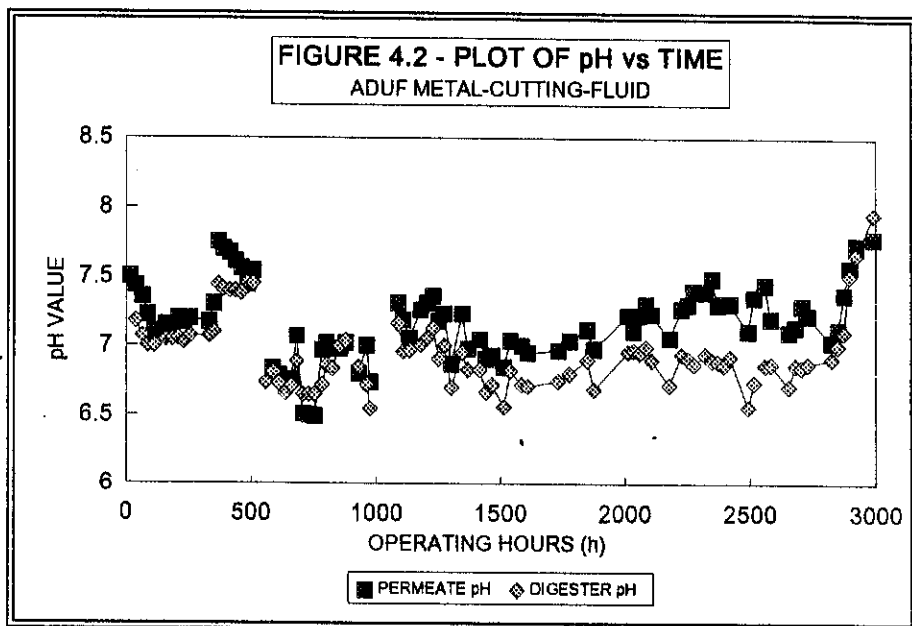
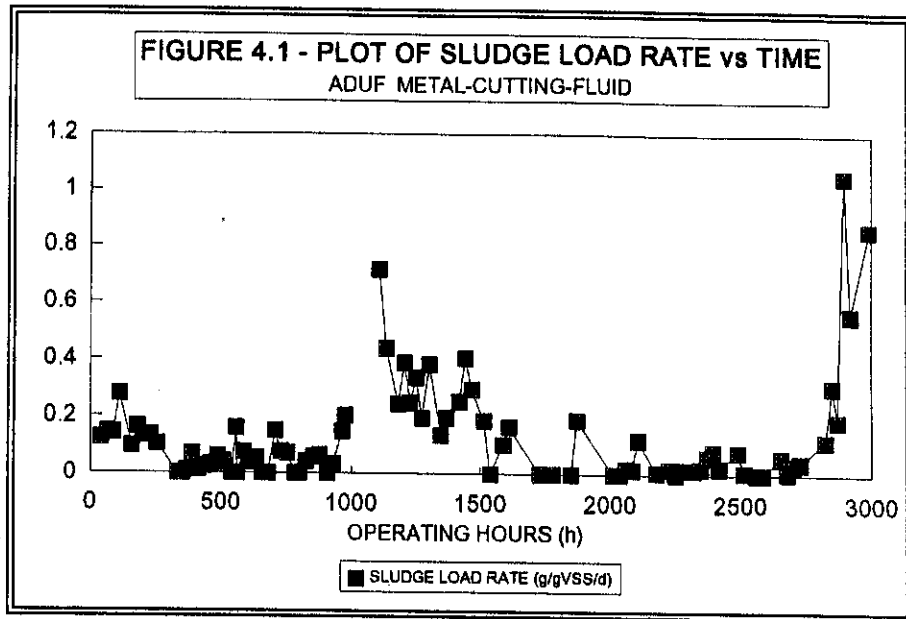
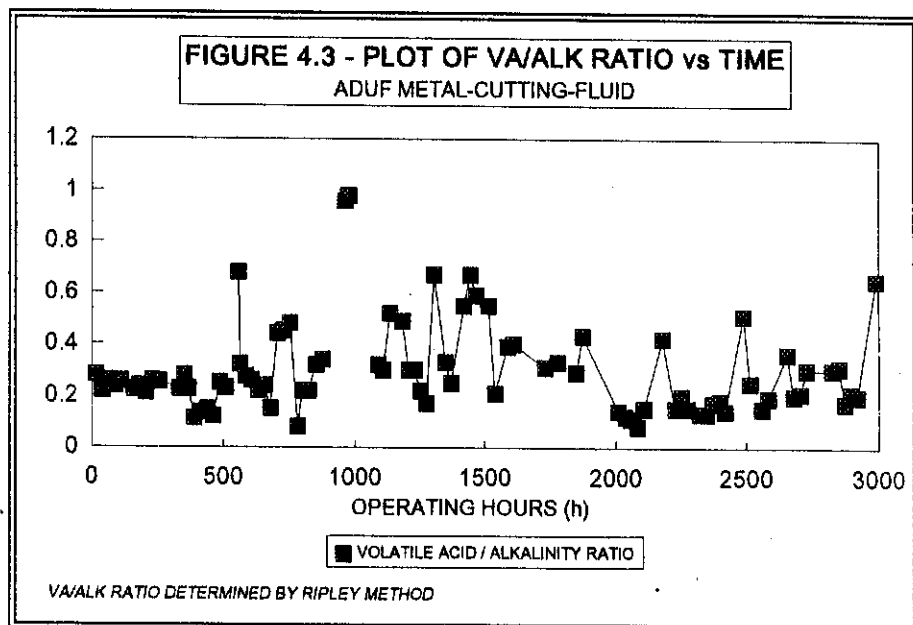


TABLE 4.2 : SOLIDS CONTENT OF ANAEROBIC SLUDGE

DATE	HOUR (h)	TSS (g/l)	VSS (g/l)
17/02/94	0	15,8	10,3
24/02/94	63	8,8	7,6
14/03/94	435	35,6	27,8
14/04/94	1086	8,2	7,4
16/05/94	1850	14,2	12,2
15/06/94	2560	24,8	21,3

A reduction in SLR to $0,3 \text{ gCOD.gVSS}^{-1}.\text{d}^{-1}$ served to arrest complete metabolic digester overload, but an unfortunate electrical failure of the temperature control system at 1370h caused the digester temperature to drop to 22°C . This resulted in another digester overload as reflected in the drop of digester pH to 6,5 and rise in the volatile acid/alkalinity ratio to 0,67 as shown in Figures 4.2 and 4.3, respectively. The digester was subsequently operated at a SLR of between 0,1 and $0,2 \text{ gCOD.gVSS}^{-1}.\text{d}^{-1}$ between 1500h and 2500h as overload was imminent in this period due to low buffer capacity (alkalinity).

A final attempt to increase the SLR was made at 2750h while adding sodium carbonate to the digester in order to improve the buffer capacity. This resulted in a digester pH increase from about 7 to 8 while the volatile acid/alkalinity ratio could be maintained at an acceptable value of around 0,3. A maximum SLR of $1,05 \text{ gCOD.gVSS}^{-1}.\text{d}^{-1}$ could be reached. The investigation could, however, not be pursued because of time restraints and the unfortunate loss of biomass due to a defect valve.



4.2.2 COD REDUCTION

The reduction in COD was generally in excess of 85% as shown in Table 4.3. This is considerably higher than that reported by Kim *et al.* (1992a) who found that only 65% of the COD present in metal-cutting-fluid waste water was anaerobically biodegradable. Additional work performed by Kim *et al.* (1992b) indicates that this figure could be increased to 88% by using a combination of anaerobic and aerobic treatment.

It is, therefore, doubtful whether the COD reductions achieved in the ADUF trial are the result of anaerobic treatment only. The ability of ultrafiltration membranes, on the other hand, to reject high molecular mass organic compounds is well established (Jacobs *et al.*, 1992). It may, therefore, be reasoned that the ultrafiltration membranes contributed to the overall COD reduction. The degree to which the membranes contribute to the overall COD reduction depend on the factors which affect the reduction characteristics of ultrafiltration membranes in general, *viz.* (Strohwald, 1988):

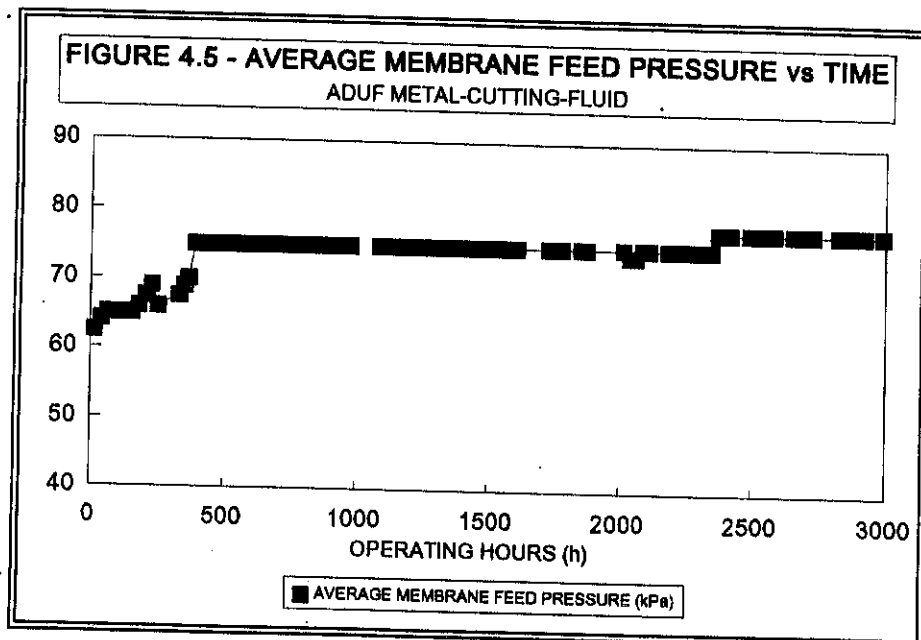
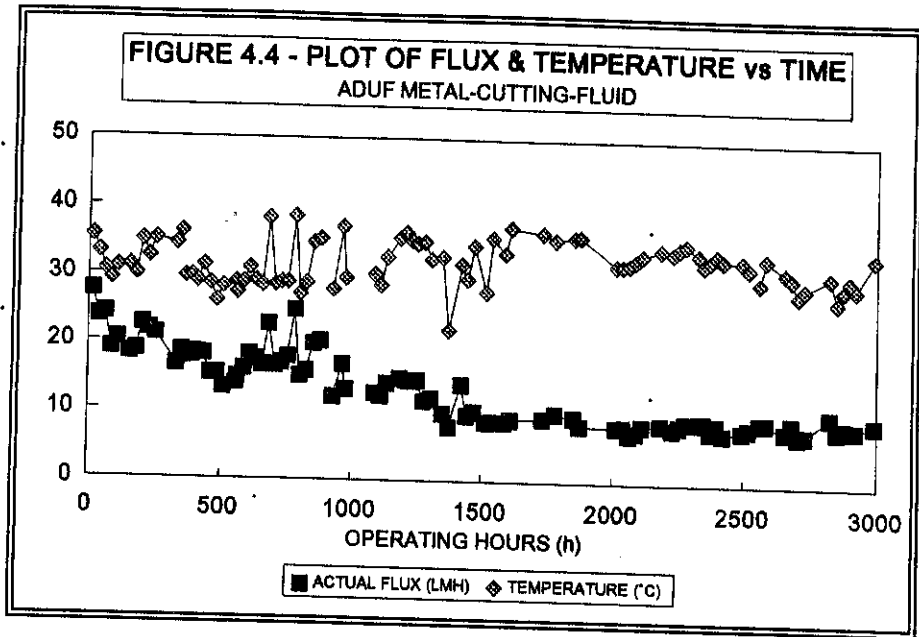
- size of the solute molecule
- shape of the solute molecule
- nature of the solute
- nature of the solvent
- presence of other solutes
- operating conditions

The contribution to COD reduction by the ultrafiltration membranes could not be investigated within the scope of this project, because of the complexity of the mechanisms involved.

TABLE 4.3 : FEED AND PERMEATE COD VALUES

DATE	HOUR (h)	FEED COD (g/l)	PERM COD (g/l)	COD REJ (%)
24/02/94	63	7,4	0,58	92,2
18/03/94	538	4,1	0,29	92,9
20/04/94	1250	13,5	1,47	89,1
28/04/94	1420	11,2	1,54	86,3
16/05/94	1850	11,5	0,86	92,5
15/06/94	2560	14,2	1,08	92,4
27/06/94	2824	68,3	9,65	85,9
05/07/94	2990	92,0	12,50	86,4

An analysis of the undiluted oily waste water resulted in COD and BOD values of 2120 g/l and 300 mg/l, respectively. Although some inaccuracy was suspected in the COD analysis, the difference between the COD and BOD values, comprising several orders of magnitude, point toward the possible presence of a biocide in the waste water. It is known that cutting oil formulations sometimes contain biocides to prevent their bacterial



degradation during prolonged use. It is, therefore, possible that the poor observed digester performance may have been caused by the presence of biocides in the waste water.

4.3 MEMBRANE PERFORMANCE

4.3.1 MEMBRANE FLUX

A plot of membrane flux and digester temperature is presented in Figure 4.4 for the 3000 hour duration of the experiment. It can be seen that the membrane flux declined gradually from a starting value of 29 LMH to 10 LMH over a period of 1500 hours. The flux subsequently remained stable at 10 LMH for the remainder of the test period.

It must be stressed that the operating conditions used during the experiment were not designed to optimize membrane flux. The objective was to rather observe whether long term flux stability could be maintained, which indeed proved to be the case. The absolute flux values shown in Figure 4.4 should, therefore, be viewed in this context.

The digester temperature could generally be maintained between 30 and 35°C, as shown in Figure 4.4, and it follows that the gradual decline in flux was not due to a decline in temperature, but rather due to boundary layer conditions on the membrane surface.

4.3.2 FOULING

The observed gradual decline in membrane flux corresponds to a low fouling rate as expected during operation of membranes in conjunction with an anaerobic digester. One of the main attributes of the ADUF process is that anaerobic digestion and ultrafiltration are complimentary processes. Anaerobic digestion decomposes organics which would otherwise foul the membranes, while the ultrafiltration membranes retain biomass (*Strohwald and Ross, 1992*).

No cleaning of the membranes was performed, either chemically or mechanically for the duration of the experiment. The gradual decline in flux is attributed to boundary layer effects on the membrane surface and not the blinding of pores as would be the case with severe fouling. This is collaborated by the constant average pressure of about 75 kPa which was maintained on the membranes, as illustrated in Figure 4.5.

5. CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

The precursory laboratory-scale study on the treatment of metal-cutting-fluid waste water by the ADUF process showed the following, with regards to the original objectives:

- The high specific COD of the waste water causes difficulties in achieving practical hydraulic throughputs while maintaining acceptable sludge load rates in the digester. The waste water shows a deficiency of major nutrients such as phosphate and nitrogen, as well as lacking the ability to maintain a buffer capacity in the digester.
- COD reductions in excess of 85% were possible when based on influent and permeate COD, albeit at low sludge load rates of 0,1 to 0,3 gCOD.gVSS⁻¹.d⁻¹. Using a biomass comprised of a variety of non-specific strains of bacteria resulted in a maximum load rate of 1,05 gCOD.gVSS⁻¹.d⁻¹. It is thought that the ultrafiltration membranes contribute to the overall COD reduction since the literature reports indicate that only 65% of the organics in metal-cutting-fluid waste is anaerobically biodegradable.
- Long term stability of membrane flux was demonstrated and no cleaning was done throughout the 3000h trial period.

The low sludge load rates that were obtained during this study question the economical viability of the anaerobic digestion step in terms of excessive digester volumes and capital cost. This situation could possibly be improved by using specific bacteria strains which are designed to decompose the constituents of the metal-cutting-fluid waste water. On the other hand, nutrient deficiency could be a further cause of poor digester performance. The provision of sufficient nutrients on a full-size digester is a logistical and economical problem. Blending of the metal-cutting-fluid waste with sewerage could provide a solution.

5.2 RECOMMENDATIONS

It is recommended that the following aspects be investigated, should additional investigations into the treatment of metal-cutting-fluid waste water by anaerobic digestion be considered:

- The inoculation of the laboratory-scale anaerobic digester with specific bacteria strains which are designed to decompose the particular constituents of the effluent.
- An investigation into the contribution to overall COD reduction by the ultrafiltration membranes.
- Combination of the ADUF process with other unit operations to address the COD fraction of the metal-cutting-fluid waste water which is not biodegradable.

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