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F I N A L R E P O R T

STUDY OF A NON CONVENTIONAL SYSTEM OF INDUSTRIAL WASTEWATER TREATMENT: HIGH RATE ANAEROBIC DIGESTION AND HIGH RATE OXIDATION PONDS

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1. INTRODUCTION

The Agency for International Development of the United States of America contracted ICAITI to carry on a study of a non-conventional system of infustrial wastewater treatment, under the terms and conditions of Grant PDC-5544-G-SS-5100-00, amended october 29, and november 4, 1987. ICAITI subcontracted the Algal Biotechnology Laboratory of the Jacob Blaustein Institute for Desert Research, Ben Gurion University of the Negev (Israel), to obtain technical services regarding the high rate algal cultivation. a second subcontract was established with the "Centro de Investigación y Estudios Avanzadados" (Center for Research and Advanced Studies), of the "Instituto Politécnico Nacional" (National Polytechnical Institute), of México. The second contract was the result of the need to obtain enough amount of the kraft pulp mill wastewaters, since the plant in Guatemala has not yet started operations.

The original objectives of the program were:

1. Characterization of the various effluents from a nearly built kraft pulping mill.

2. Study of the high rate anaerobic digestion of a selected wastewater from the kraft mill.

3. Study of a photosynthetic system that can treat the effluent from the digester in a high-rate oxidation pond.

4. Operation of a bench scale system for the treatment of the selected mill wastewater, incorporating the results of the two technologies explored.

This present version of the final report is a late one, made to include presentations submitted in 1991. The research work was finished within the specified time terms, although some delay in reporting advances was caused by the counterpart from CINVESTAV moving to Canada.

2. SUMMARY OF ACTIVITIES

2.1 Training Activities with the Algal Biotechnology Laboratory, Ben Gurion University of the Negev (Israel).

The major line of collaboration with the Israeli group was in training of the ICAITI personnel. A Guatemalan researcher, Liliana de Barrios, stayed in Sade Boger for a training on basic aspects of algal growth. Professor in charge in Israel: Dr. Avigad Vonshak.

A second training stay by Lic. Liliana de Barrios was from March 26 to April 11, 1987. Stress was on the cultivation procedures for algal.

2.2 Research at the algal Biotechnology Laboratory, Israel.

The research work was mostly centeres in the search for strains that could grow adecquately in the effluents. These strains were transfered to ICAITI after prelimminary growth experiments.

2.2 Joint Research with the Center for Research and Advanced Studies (CINVESTAV) of the National Polytechnic Institute of Mexico.

Work on Karft condensates and Bleaching Effluents has been included in this effort. Please refer to the technical sections of this report for results. Researcher in charge at CINVESTAV: Ing. Héctor Poggi V.

2.3 Research Activities at ICAITI

Activities were limited because of the difficulties in obtaining the industrial effluents. Work was done in the two phase system of the simulated Kraft condensate and bleaching effluents. Please refer to the technical sections of the report for details. Researcher in charge at ICAITI: Ing. José Francisco Calzada M.

Three students from two local Universities worked in the research project. Two thesis works were also presented. See the thesis section of this report for cover pages.

2.4 Course on Algal Biotechnology

A course on Algal Biotechnology was offered by ICAITI in Antigua Guatemala on August 29-31, 1989. A copy of the program is included. Proffessors Avigad Vonshak and Amos Richmond participated as main instructors. It must be noted that two works from ICAITI were presented as case studies. One of them described the treatment of the Kraft pulp mill effluents and resulted from the project.

2.5 Coordination Meetings

The First Coordination Meeting was held at the Laboratory of Algal Biotechnology, The Jacob Blustein Institute for Desert Research of the Ben Gurion University of the Negev (Sade-Boger). A complementary meeting was also held at the main campus of the University (Beer-Sheva). A presentation of some research works currently being carried on al ICAITI was done. These meetings took place in April 7/10, 1980, and they were coordinated by Dr. Avigad Vonshak.

The second coordination meeting was held at ICAITI, during the week of July 21-25. The participants were Avigad Vonshak (BGU), José Francisco Calzada, Carlos Rolz and Liliana de Barrios (ICAITI). Dr. Vonshak indicated that the initial samples (untreated effluents) were received in Israel in May 28 and the group had already started some screening work with algal strains. He also presented a first progress report from his group.

During the stay of Dr. Vonshak, he was able to participate in the tests of a gas-lift fermentor using algal cultures. He presented a lecture on some of the research activities of his group and accompanied the ICAITI scientist for a visit to the Embassy of Israel, to describe the US-Israel CDR programs to the Ambassador, Dr. Moshe Dayan and the Secretary, Mr. Shmooel Livne.

A coordination meeting was held in Mexico with Ing. Héctor Poggi on March 9-11, 1987. Activities in the CINVESTAV were then planned.

A second coordination meeting with Ing Poggi was held in Guatemala on May 8-11, 1988, to review the initiation of the experiments and the original difficulties with the reactors.

A coordination meeting (final) was held with all the participants in Guatemala, during the Algal Biotechnology Course above described.

Travel of Dr. Poggi to Canada, to continue studies delayed some of the reporting and an informal meeting on the results was held during the Symposium on Anaerobic Digestion for the Treatment of Wastewaters in Latin America (Mexico, November 8 and 9, 1990).

3. TWO STAGE ANAEROBIC DIGESTION OF CONDENSATE FROM THE KRAFT PULP PROCESS

4. SUPPLEMENTED KRAFT CONDENSATE TREATMENT IN HIGH RATE ANAEROBIC PROCESSES

RESEARCH NOTE

TWO-STAGE ANEROBIC DIGESTION OF CONDENSATES FROM THE KRAFT PULP PROCESS

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ABSTRACT

The treatment of black liquor condensates from the kraft pulp process was studied using a two stage anerobic digestor in which the upper section contained surfaces covered with unreticulated polyurethane foam. COD reductions in the range of 70-91% were obtained with COD loads of 0.77-0.86 g/l.d using one-day hydraulic retention times.

KEY WORDS

Anerobic Digestion, Treatment, Kraft Process, Pulp,

Condensate, Reactors, Methane, Wastewaters

INTRODUCTION

The treatment of pulp and paper effluents, and more specifically, through anerobic processes, has been reviewed by several authors (Cocci et al., 1982; Gillespie, 1984; Salkinoja-Salonen et al., 1983; Webb, 1984 being some examples). One of the disposal problems is generated by the condensates from the black liquors in the sulfate and sulfite processes.

Norrman (1983) treated the condensate from the evaporation of kraft black liquor using three types of anerobic reactors: a) packed bed, b) expanded bed and c) fluidized bed. The last two units required recirculation and the third one also some dilution. A COD reduction of more than 80% was obtained with volumetric loads of 2 kg COD per cubic meter per day in a), 10 in b) and 13 in c). In another report, loading rates 1.5-2.0 kg COD/m^3.day gave reductions of 30-90%, with gas productions of approximately 0.5 m^3/m^3.d and specific methane production of 0.35 m^3/kg COD degraded (Norrman and Narbuvold, 1984).

In a recent work, Minami et al. (1986) found that the condenste stream from a kraft mill could not ferment continously under anaerobic conditions. The effect was attributed to the presence of an inhibitor, an oily material. Once this material had been removed through the use of concentrated sulfuric acid and diatomite filter aid, the

residual liquid could be fermented easily, with minimun hydraulic retention time of 0.64 days, maximun BOD removal rate of 11.47 kg/m^3.d and biogas production eqivalent to 82-87 per cent of the theoretical volume. The anerobic reactor was filled with activated anaerobic sludge, pumice stone and either model wastewater or raw condensates with mineral additives. Total BOD loads ranged from 0.77 to 5.54 kg/m^3.d. The objective of the present study was the evaluation of the capacity of anaerobic reactors using sponge surfaces to treat the raw condensates from kraft pulping. The sponges were made of unreticulated polyurethane and they have proved to be useful, as support materials for bacterial growth, in the treatment of other polluting effluents (Calzada et al.,1984a).

MATERIALS AND METHODS

The condensates were generated in a kraft pilot plant. The black liquors from the digestion were evaporated to obtain the condensate, the usual effluent in the industry.

The anerobic reactors were Allhin condensers in which the internal chambers contained small cubes of unreticulated polyurethane foam sponge (0.02mx0.02mx0.02m). The reactors were fed twice a day through the bottom and discharged through the top. The colonization by anaerobic bacteria was done using effluents from an active methanogenic reactor mixed with the condensate.

The hydraulic residence times in these semicontinous reactors were calcultaed from the daily liquid input. Analysis of the feed and effluent were done as described elsewhere (Calzada et al., 1984b). The gas production was determined through water displacement in glass cylinders.

RESULTS AND DISCUSSION

The results of the COD reductions are shown in Table 1. It can be seen that, without recirculation, maximun loads for the semicontinous system were close to 1.0 kg COD per cubic meter of digestor per day. The COD reductions for those loads were in the range of 78-89%, leaving COD levels usually below 200 mg/l, which can be discharged directly into the disposal systems or further purified using algal ponds.

The COD reduction values agreed with those of Norrman and Narbuvold (1984) for high rate systems without recirculation. They were much lower than the removal data of Minami et al. using condensates previosly treated to eliminate the inhibition.

The gas production rates and the pH values of the effluents are presented in Table 2. The gas production rates were lower than those reported for the nutrient-enriched feeds used in the work of Norrman and Narbuvold (1984). The results showed the technical feasibility of treating the kraft mill condensates using anaerobic reactors in which the microflora

is adhered to unreticulated polyurethane foam surfaces within the reactor. Hydraulic retention times of one day were found to be relative minumun values for semicontinous systems.

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TABLE 1 ANAEROBIC TREATMENT OF KRAFT CONDENSATES
(REDUCTION OF CHEMICAL OXYGEN DEMAND)

H R T (days)	COD Load (g/l/day)	COD reduction in Col. A (%)	COD reduction in Col. B (%)
2	0.37	82.2	84.2
2	0.37	82.2	84.7
2	0.37	83.6	84.2
2	0.55	94.1	94.7
2	0.55	84.9	84.9
2	0.99	83.2	81.3
1	0.86	77.9	84.0
1	0.86	89.4	88.5
1	0.86	89.4	89.4
1	0.77	90.8	88.4
1	0.77	70.2	74.7
0.5	2.08	33.6	37.0
0.5	1.61	13.3	17.9
0.5	1.90	39.8	44.3

TABLE 2 ANAEROBIC TREATMENT OF KRAFT CONDENSATES
(GAS PRODUCTION RATES AND pH VALUES)

H R T	Column A		Column B	
	Gas Prod. (v/v/d)	pH	Gas Prod. (v/v/d)	pH
2	0.044	6.80	0.044	6.80
2	0.031	6.85	0.019	6.80
2	0.044	6.85	0.062	6.95
2	0.079	6.40	0.081	6.40
2	0.071	6.40	0.078	6.40
2	0.052	6.80	0.064	6.75
1	0.025	6.75	0.018	6.80
1	0.010	6.90	0.010	6.90
1	0.025	7.00	0.032	7.05
1	0.035	6.90	0.028	6.80
1	0.032	6.80	0.025	6.70
0.5	0.012	6.30	0.012	6.70
0.5	0.012	6.10	0.000	6.20
0.5	0.038	6.40	0.012	6.40

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SUPPLEMENTED KRAFT CONDENSATE TREATMENT IN HIGH RATE ANAEROBIC PROCESSES

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ABSTRACT

Kraft condensate from black liquor recovery (KC) with nutrient enrichment was treated in three processes: Anaerobic Fluidized Bed (ANFBED) containing granular activated carbon as the medium support, a Downflow Anaerobic Filter (DANFIL) filled with corrugated polyvinyl chloridate (PVC) modules and volcanic stone, and a hybrid Upflow Anaerobic Sludge Blanket Reactor (HUASB) packed at its top with non reticulated polyurethane; in parallel and in the mesophilic range.

The reactors exhibited a Chemical Oxygen Demand (COD) removal efficiency in the range 56 to 82%. The loading rate (LR) varied between 0.83 to 2Kg COD/m^3-d and a Hydraulic Retention Time (HRT) range of 2 to 0.75 day for both the DANFIL and the HUASB, and 1.2 to 3.8 Kg COD/m^3-d and 1 to 0.4 day respectively for the ANFBED. The biogas production was 50% of the theoretical maximum for all three processes.

It can be concluded that it is feasible to treat supplemented KC in high rate anaerobic processes, and that the nutrient supplementation to the KC allows further process intensification

(reaching lower HRTs) and higher biogas production compared to results with non-supplemented KC.

Among the three processes treating supplemented KC, the ANFBED appears to be the most intensive and efficient. This could be presumably a consequence of the adsorption characteristics of its granular medium support and its larger surface area for biomass adhesion and growth.

INTRODUCTION

In Mexico, four big industrial activities account for nearly 90% of the industrial wastewater discharges, as shown in Table 1. They are the sugar industry, the pulp and paper manufacture in second place, the steel industry and the oil production and refining.[1]

The Mexican pulp and paper industry generates about 12% of the volume of the industrial wastewaters, and it is known that many of the substances contained in these wastewaters are toxic or at least refractory to biological degradation.[2,3]

Almost 75% of the Mexican pulp is manufactured using the Kraft process with pine wood as feedstock. The Kraft process involves the digestion of wood chips with an alkaline-sulfidized liquor, and the subsequent bleaching of the raw pulp with chlorine and other oxidant compounds. The spent liquor is further concentrated by evaporation in multi-effect evaporator trains, and burned for chemicals recovery and reuse. This concentration stage is the source of the so named Kraft condensate (KC).

At present, the Mexican pulp and paper industry is facing strong pressures dealing with new discharges permits and limits, according to new regulations issued by the Mexican Ministry of Environment and Urban Development. In this context, a research project was started, aimed at assessing the anaerobic treatability of Kraft condensate, Kraft bleaching effluent and whitewater from paper-making machines.

High rate anaerobic treatment processes were selected for this study, because they offer many advantages over the aerobic and physico-chemical treatment, with none of the drawbacks of the conventional one-through anaerobic digesters:

- these processes have been shown to handle dilute effluents,
- can cope with recalcitrant and toxic substances, provided a right acclimation is done,
- are suitable for soluble wastes,
- are faster than conventional digestion is, and they may operate at Hydraulic Retention Times (HRT) comparable to ones of the aerobic treatment,
- they require less nutrients than aerobic processes do,
- for medium and high strength organic load wastewaters, biogas recovery and use as fuel is possible,
- savings in aeration investment and operating costs are an additional appeal when compared with aerobic treatment.[4,5]

Furthermore, encouraging applications of high rate anaerobic processes to pulp and paper wastewaters have been reported since the early eighties.[6,7,8]

This work presents the results concerning the KC treatment, and its main objectives were:

- to determine the technical feasibility of a dilute Kraft condensate treatment in high rate anaerobic processes.
- to assess the effect of nutrient supplementation of the KC on the anaerobic process performance.
- to compare the performance of three selected processes fed with nutrient enriched KC:
    - Downflow Anaerobic Filter (DANFLOW)
    - Hybrid Upflow anaerobic Sludge Blanket (HUASB)
    - Anaerobic Fluidized Bed reactor (ANFBED)

MATERIALS AND METHODS

The characteristics of the reactors fed supplemented KC follow:

HUASB: glass column 4 cm diameter × 1.4 m height, expansion chamber 10 cm diam. at the top. It contained non-reticulated polyurethane modules in the superior third part of the reactor.

ANFBED: glass column 4 cm. diam.× 1.6 m height, an expansion chamber at the top 10 cm diam.; filled with 1 litre of granular activated carbon (GAC) 500 micrometer average diam. as medium support.

DANFIL: glass column 4 cm diam. × 1.4 m height, no expansion chamber, filled with PVC packing modules (96% void volume) at the upper and lower parts of the reactor, and 65% porosity volcanic stone in the middle.

Feeding was intermittent, with a timer and peristaltic pumps. Continuous recycling was provided for the ANFBED, while the DANFIL and HUASB operated with recycling only during the start-up and acclimation periods. The whole setup was inside a controlled temperature room at 32°C. The reactors fed non-supplemented KC (UANFIL) were made up of Allhin condensers filled with nonreticulated polyurethane modules. They were operated with intermittent feeding in the upflow mode and no recycling, in the mesophilic range.

Start-up and acclimation procedures were conservative, as described elsewhere.[9,10] Analyses of the liquid streams were done following the Standard Methods[11], and the alkalinities ratio, alfa was determined as recommended by Ripley et al.[12]

The gas production was measured by saline water displacement method, and the corresponding pressure and temperature corrections were made. Its composition was analyzed using gas chromatography as a control, and a mini-Orsat syringe method for carbon dioxide determination on a routine basis.[13]

The experimental design was simple. The reactors were operated in parallel, and a succession of pseudosteady states at decreasing HRT was followed (or equivalent, increasing Loading Rates, LR).

The nutrient and trace elements supplementation added to the KC is shown in Table 2.

RESULTS AND DISCUSSION

Table 3 contains the raw KC main characteristics. It was a low-medium strength effluent, and its organic matter content was mainly soluble. The biodegradability of the organic load is fair to good, as reflected by the Biochemical Oxygen Demand (BOD) value and the BOD/COD ratio.

The pH was alkaline, but the buffering capacity was poor or nonexistent. Nutrient contents (nitrogen, phosphorus) were lower than the required values according to the recommended ratios COD:N:P for anaerobic treatment. Nutrient contents (nitrogen, phosphorus) were lower than the required values according to the recommended ratios COD:N:P for anaerobic treatment.[5] The COD/S ratio was much higher than 15 to 20, therefore no serious sulfide toxicity problems were anticipated.

Figure 1 shows the COD evolution in the feed and effluents of the three reactors fed supplemented KC, for the steady states at different hydraulic retention times. The ANFBED and the DANFIL had a consistent low effluent COD, despite the increasing load at 0.75 day HRT.

On the other hand, the HUASB suffered a severe loss of active sludge during the 1 day - HRT run. The hydraulic overloading associated to the intermittent feeding, together with the flocculent characteristics of the sludge, promoted the wash-out of the active biomass. In consequence, the effluent COD increased, but the reactor could remove a marginal amount of the organic load, suggesting that the microorganisms attached to the polyurethane packing were helpful in alleviating the elutriation upset.

The COD removal efficiencies for the reactors treating supplemented KC are plotted versus HRT in Figure 2.

The ANFBED exhibits a dependable organic load removal between 70 to 82%. The DANFIL COD efficiency increases with an increase in LR, perhaps due to a 'maturation' process reported for anaerobic fixed-film treatment, and suggesting that biomass concentration inside the reactor and/or biomass activity increased with time.[5,14]

The same effect, but less marked, can be seen for the ANFBED.

The drop in removal efficiency for the HUASB in the 1 day HRT run (from 75% down to 42%) is evident, due to the washout commented above. The reactor was recovered from this collapse following a strategy that included reseeding and changing the feed pattern. In the last run at 0.75 day HRT, the HUASB again reached an organic load removal efficiency of about 75%.

The biogas productivity for all the three processes treating supplemented KC was low (Figure 3), as it was expected to be because the low organic content of the KC.[14]

The ANFBED displayed the most intensive biogas productivity on the basis of working reactor volume, and this parameter showed a direct correlation with the LR.

The biogas yield (not shown here) was half the maximum for all the three reactors. Methane content was between 40 to 60%, with carbon dioxide less than 10%. Unusually high contents of nitrogen (20% to 40%) were found. This is probably due to the combined effects of slightly alkaline pH in the reactor liquors, dissolved N_2 flashing, and a postulated microaerophilic nitrification followed by denitrification in the lowest parts of the reactors. The gas composition is similar to that reported by Yoda et al. during the anaerobic treatment of municipal wastewater (another diluted effluent) in an ANFBED.[15]

Routine biogas flammability tests were always positive for the three reactors during the three tried HRT runs.

The pH and alfa parameter, of the reactor effluents, are plotted in Figures 4 and 5 respectively. pH values in the neutral and slightly alkaline range, and alfa values less than 0.5 are highly correlated with good methanogenic regimes in anaerobic treatment.[4,5,12]

The effluent pHs ranged between 6.9 and 7.7, including the HUASB washout episode. The alfa values fluctuated between 0.20 and 0.45, with a logical trend to increase with the first rise in LR (run at 1.5 day HRT to run at 1 day HRT), and remaining in steady values during the last run.

The slight increase in COD removal efficiencies at higher LR, the sustained or increasing biogas productivities with increasing LR, together with the pH and alfa values evidencing well

developed methanogenic regimes, strongly indicate that the reactors could be accelerated far beyond the minimum HRT 0.75 day without risk of process failure.

Figure 6 presents the average results for the UANFIL reactors fed with non-supplemented KC. The main features are:

- the process fails between 1 day HRT and 0.5 day HRT.
- the pH is acidic for all the runs.
- the biogas productivity is poor.
- the COD removal efficiency is good to excellent at the lowest LR.

The results of this experiment suggest a poor or unstable methanogenic regime, and perhaps a COD reduction linked to biomass growth instead of biogas generation.

A short comparison between the ANFBED in the present work (ANFBED-CIEA) and an ANFBED experiment run by Norrman[16] (ANFBED-Norrman), both fed supplemented KC, is displayed in Table 4.

The ANFBED-Norrman performance is superior to the ANFBED-CIEA in biogas productivity and methane content.

The COD removal efficiencies of both reactors are of the same order, while the ANFBED-CIEA shows advantages in attaining significant lower recycling ratios (lower upward linear velocity for fluidization) and a healthier pH, while the ANFBED-Norrman shows a quasi-acidogenic pH value.

The differences in pH could be explained by the psychrophilic operation of the ANFBED-Norrman and its lower pH may account for the disparity in biogas productivities (inducing bicarbonate passage to the gas phase).

The inferior and beneficial low value of fluidizing velocity in case of ANFBED-CIEA is certainly related to the low density of the activated carbon used, as compared with the sand used in the ANFBED-Norrman.

Norrman reported acidogenic failure for LR above 4 Kg COD/m^3-d, expressed on fluidized bed working volume, while further work is needed to demonstrate the performance of the ANFBED-CIEA at LR above 3.8.

A similar comparison among the performances of the DANFIL and three UANFIL from Norrman and co-workers treating supplemented KC, is shown in Table 5.[16,17,18]

The COD removal efficiencies and overall performances are similar, with Norrman's filters exhibiting acidic pH and higher biogas productivities.

CONCLUSION

The results of this work confirm the feasibility of treating diluted KC from pulp manufacture with high rate anaerobic processes.

The nutrient and trace elements enrichment of the feed seems to have a beneficial effect on the process performance, leading to highest biogas productivities, reasonable methane contents in biogas, and better stability of the methanogenic regime.

Regarding the performance comparison among the three reactors treating supplemented KC, they ranked as follows:

- Organic load removal	ANFBED > DANFIL > HUASB
- Biogas productivity	ANFBED > DANFIL > HUASB
- Process intensity	ANFBED > HUASB = DANFIL
- Ease of operation	DANFIL > ANFBED > HUASB
- Methanogenic regime stability	Reasonable and similar

From this ranking, it is concluded that the Anaerobic Fluidized Bed reactor is the most promising configuration.

The superior performance of the ANFBED could be related to the use of granular activated carbon as medium support. The GAC could provide an additional pollutant removal (via adsorption and bioregeneration), and a larger surface area for biomass adhesion and growth.

Cost analysis is required to determine the economic advantages and feasibility of each configuration.

No conclusive inferences can be drawn from the comparison with Norrman's experiments with supplemented diluted KC and the processes studied in this work. Further research is needed to determine the ANFBED-CIEA potential to surpass the barrier LR of 4 Kg COD/mg^3-d, and for the DANFIL and the HUASB to operate a LR higher than 2 Kg COD/m^3-d.

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LIST OF ABBREVIATIONS

ANFBED	Anaerobic Fluidized Bed reactor
BOD	Biochemical Oxygen Demand
COD	Chemical Oxygen Demand
DANFIL	Downflow Anaerobic Filter
GAC	Granular Activated Carbon
HRT	Hydraulic Retention Time
HUASB	Hybrid Upflow Anaerobic Sludge Blanket
KC	Kraft Condensate effluent
LR	Loading Rate
N	Nitrogen
P	Phosphorus
PVC	Polyvinylchoride
S	Sulfur
UANFIL	Upflow Anaerobic Filter

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Table 1. Industrial Wastewater Discharges in Mexico

INDUSTRY	DISCHARGES M m^3/year
Sugar	620
Pulp and Paper	100
Steel	38
Oil	31
Chemical	21
Textile	21
Tannery	12
Food	11
Coffee	5

Table 2. Kraft Condensate Nutrient and Trace Element Supplementation

Ammonium Phosphate, mg/L	170
$ZnSO_4 \cdot 7H_2O$, mg/L	0.1
$MnC_{12} \cdot 4H_2O$, mg/L	0.03
$CuC_{12} \cdot 2H_2O$, mg/L	0.01
$NiC_{12} \cdot 6H_2O$, mg/L	0.02
$Na_2MoO_4 \cdot 2H_2O$, mg/L	0.3
H_3BO_3, mg/L	0.3
$FeCl_3$, m/L	3

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Table 3. Kraft Condensate Characteristics

pH	9.3
Total Alkalinity, mg $CaCO_3$/L	64
Conductivity, microS/cm	180
COD, mg 02/L	1240
BOD, mg 02/L	880
BOD/COD ratio	0.71
N Kjeldahl, mg N/L	14
Total P, mg P/L	2.6
Sulfate, mg SO_4/L	17
Sulfide, mg S/L	3.4
COD/Total S ratio	120

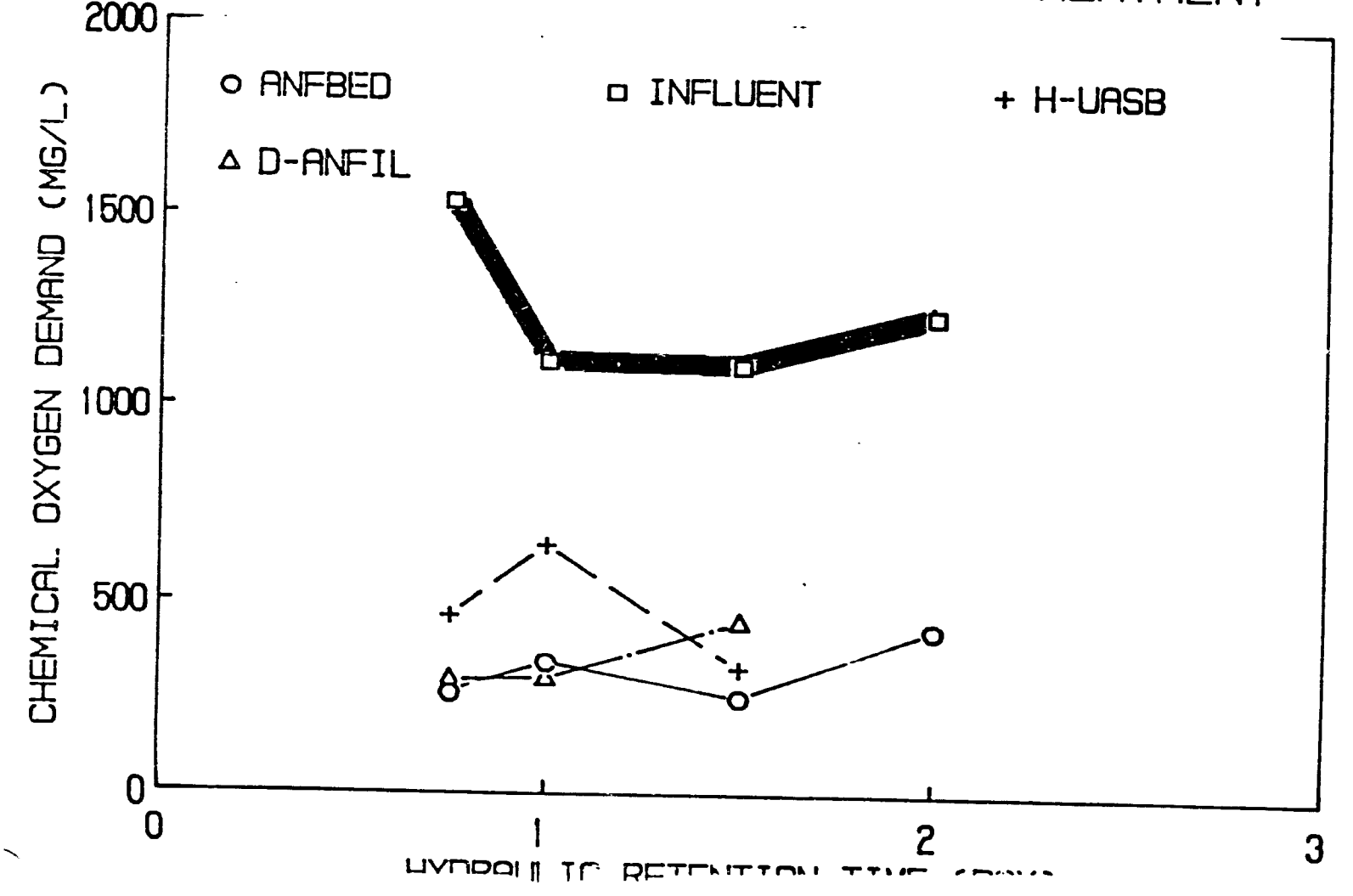
Table 4. ANFBED-Norrman and ANFBED-CIEA Summarized Performances

	Temp. °C	Max. LR Kg COD/ M^3-Day	HRT Day	pH	COD Eff. %	Biogas Product. M^3/M^3-D	Recycle Ratio
ANFBED Norrman	22	4.6	0.3	5.2-6.4	80-90	0.78	450
ANFBED CIEA	32	3.8	0.4	7.3	82	0.51	50

Table 5. Norrman's Filters and DANFIL Summarized Performances

	Temp. °C	Max. LR Kg COD/ M^3-Day	HRT Day	pH	COD Eff. %	Biogas Productivity m^3/m^3-Day
UANFIL Norrman Ref. 16	37	1-2	0.7-1.4	6.1-6.6	90	0.4
UANFIL Norrman Ref. 18	28	2	NA	NA	70-90	0.5
UANFIL Norrman Ref. 17	38	0.1-0.3	1.7-8.6	< 6.4	> 80	NA
DANFIL	32	2	0.75	7.1	81	0.23

CHEMICAL OXYGEN DEMAND:
INFLUENT AND REACTOR EFFLUENTS.
KRAFT CONDENSATE ANAEROBIC TREATMENT



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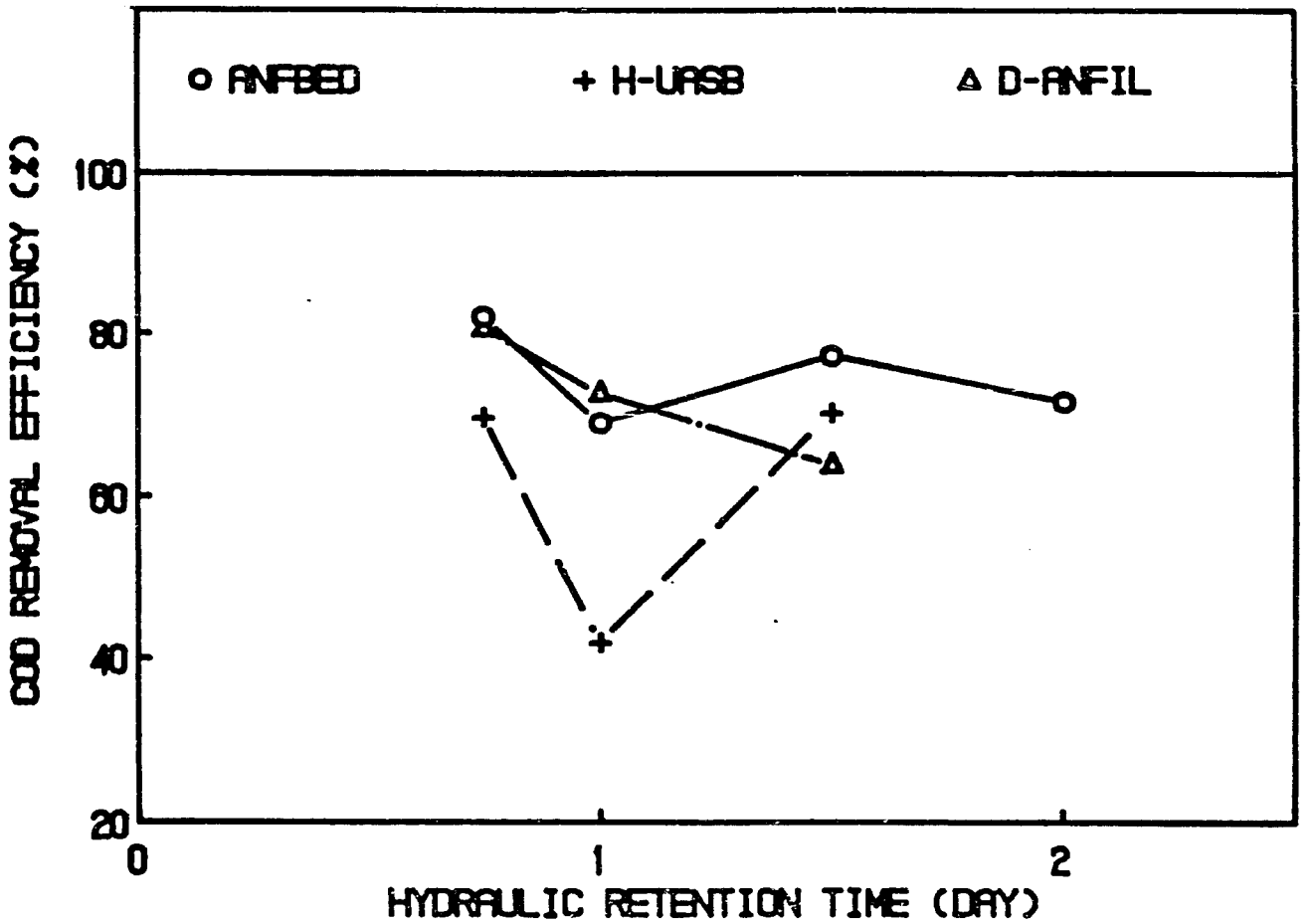
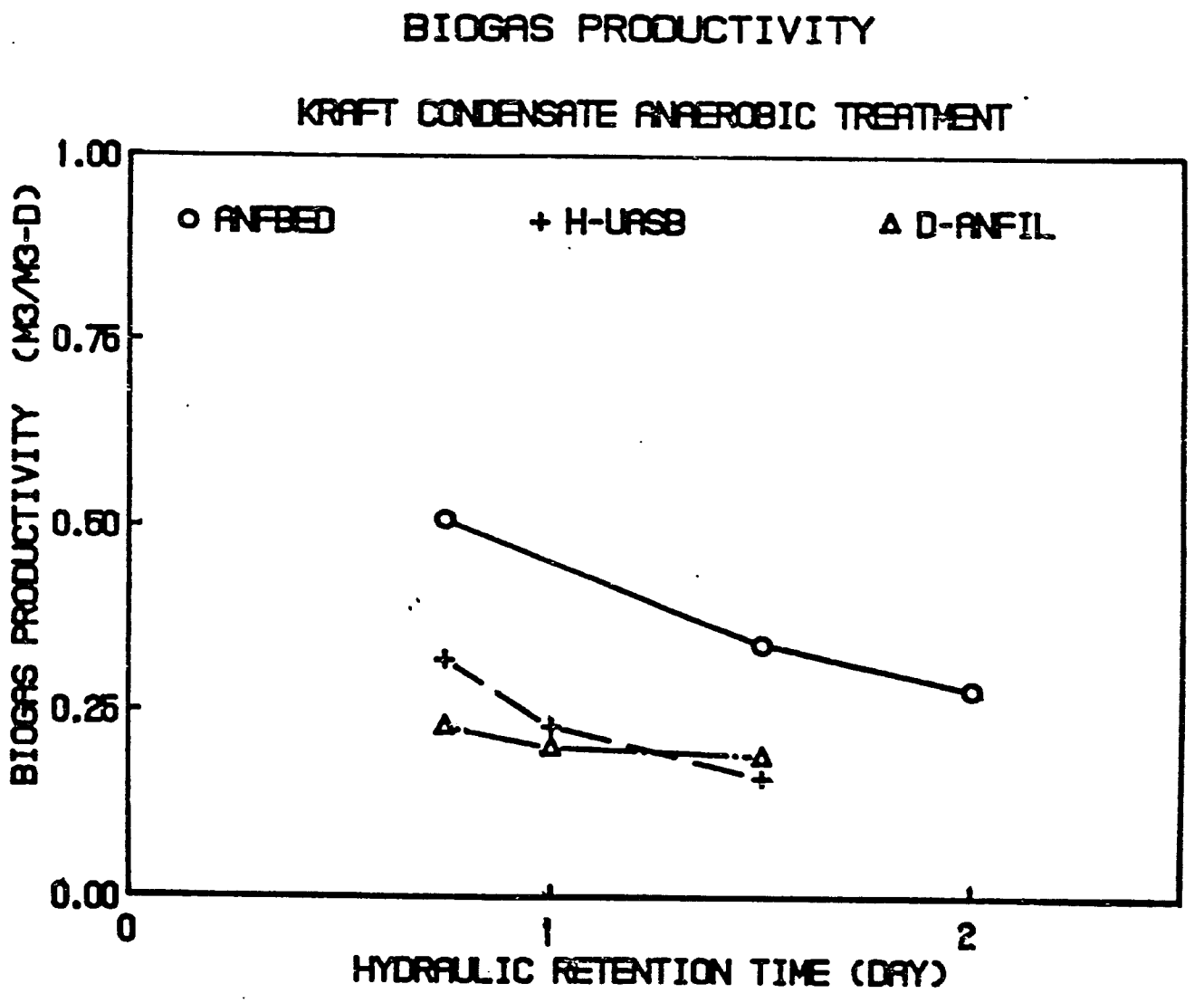
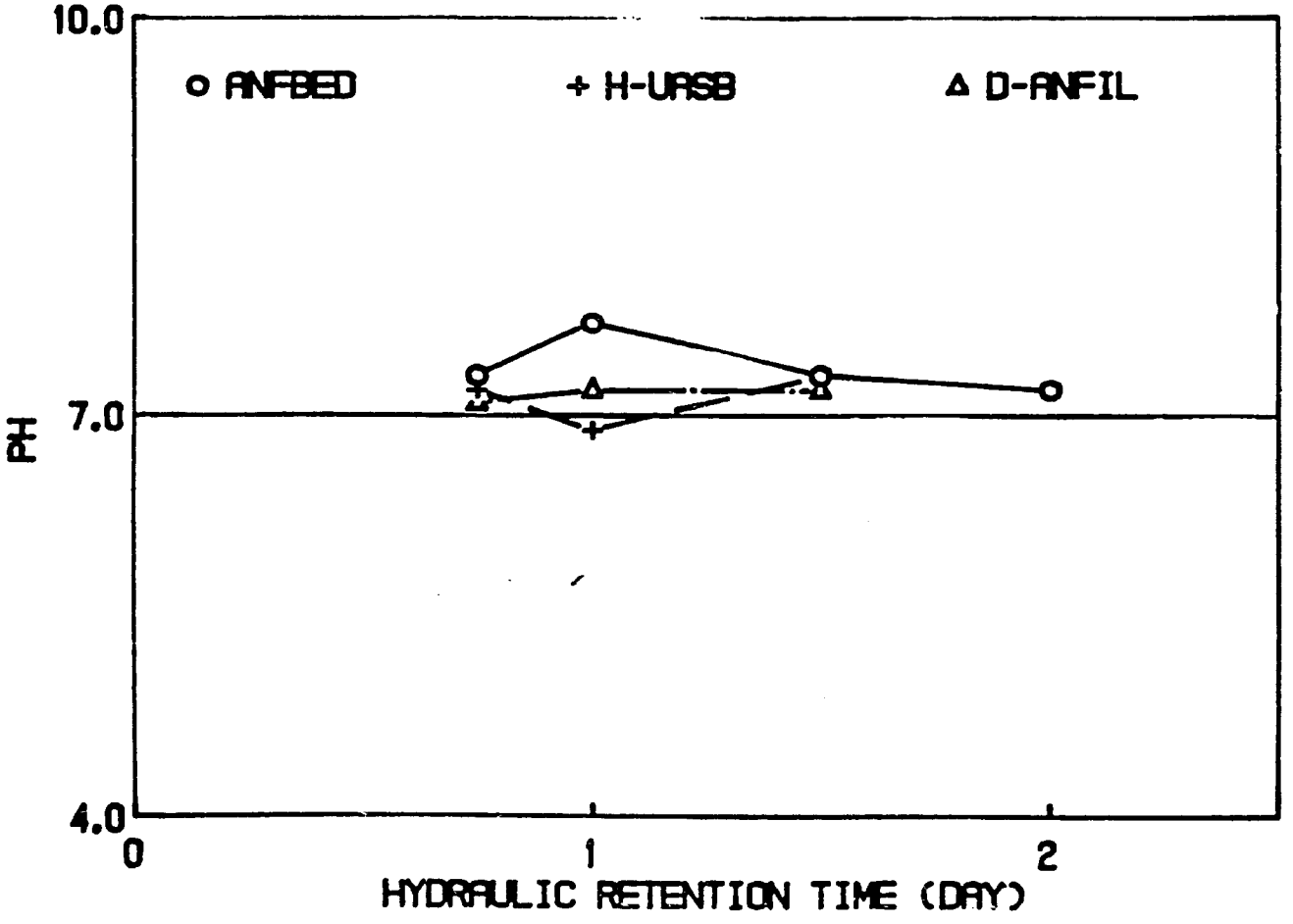
CHEMICAL OXYGEN DEMAND
REMOVAL EFFICIENCY
KRAFT CONDENSATE ANAEROBIC TREATMENT




BIOGAS PRODUCTIVITY
KRAFT CONDENSATE ANAEROBIC TREATMENT
o ANFBED
+ H-UASB
Δ D-ANFIL
BIOGAS PRODUCTIVITY (M3/M3-D)
1.00
0.75
0.50
0.25
0.00
0
1
2
HYDRAULIC RETENTION TIME (DAY)

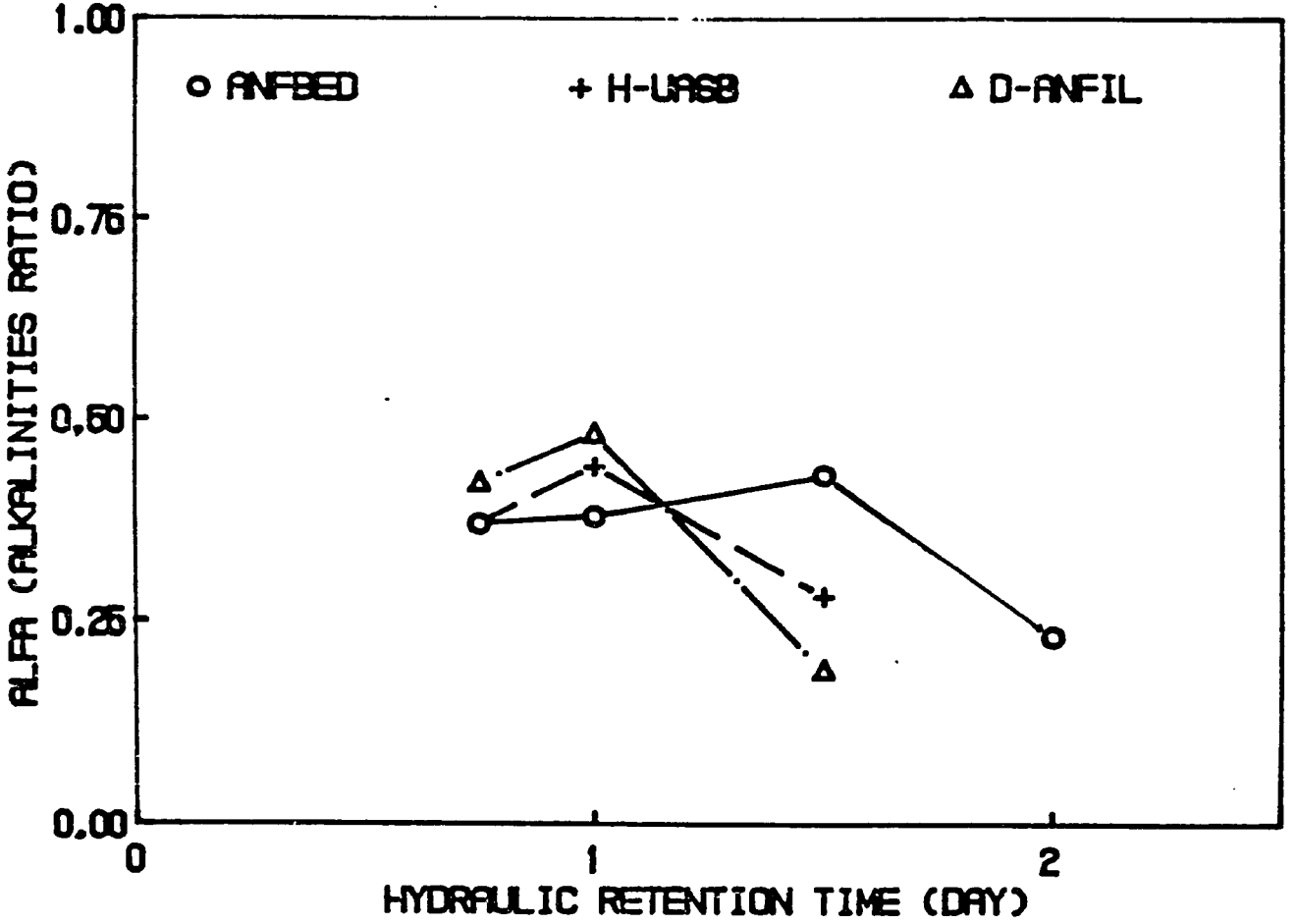
PH IN REACTOR EFFLUENTS

KRAFT CONDENSATE ANAEROBIC TREATMENT



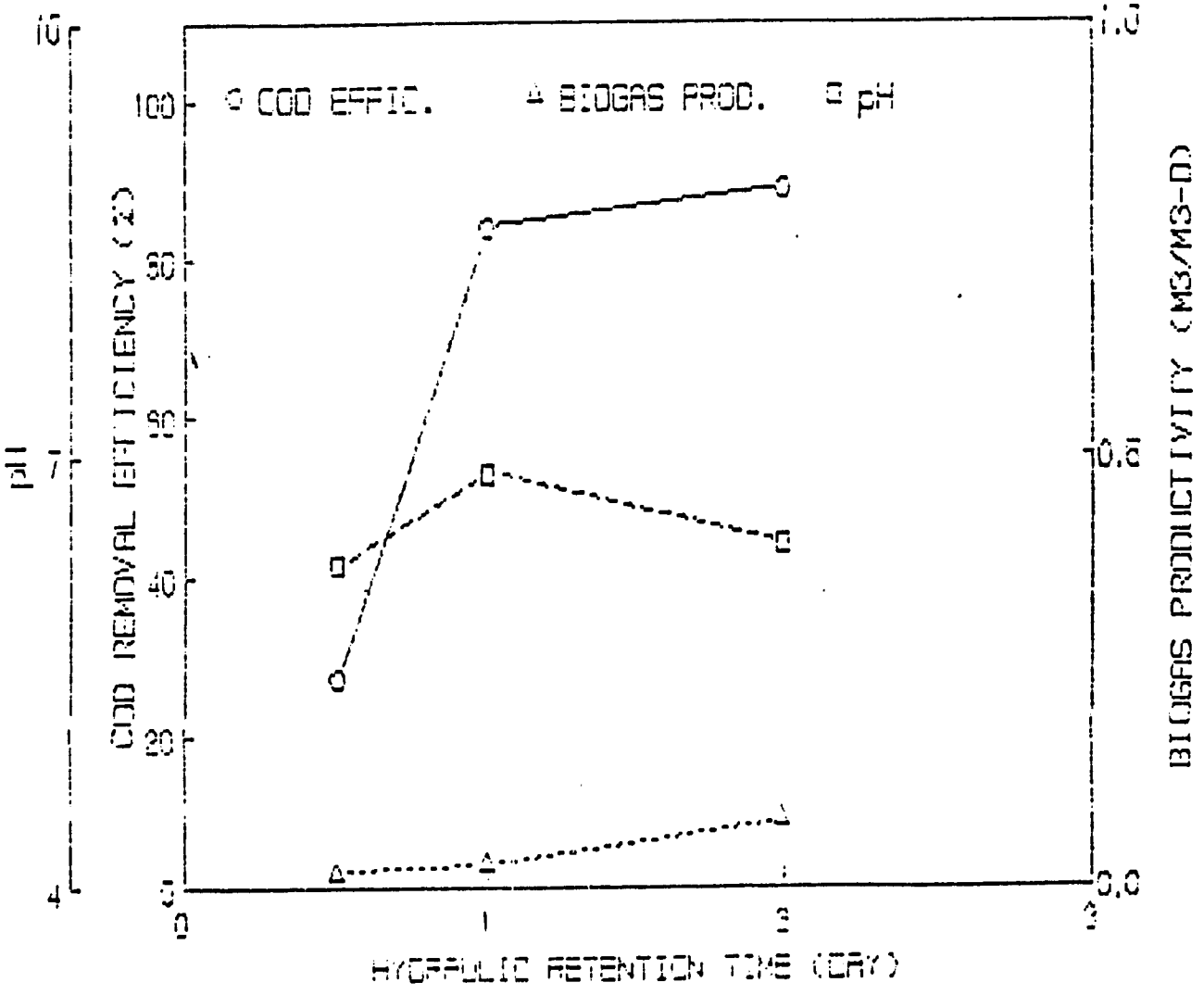
ALFA EVOLUTION IN REACTOR EFFLUENTS

KRAFT CONDENSATE ANAEROBIC TREATMENT



NON-SUPPLEMENTED

KRAFT CONDENSATE ANAEROBIC TREATMENT



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5. ANAEROBIC TREATMENT PULP AND PAPER MILL WASTEWATERS IN MEXICO

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EXPERIENCIAS EN EL TRATAMIENTO ANAEROBICO DE EFLUENTES DE FABRICAS DE PAPEL Y CELULOSA EN MEXICO

ANAEROBIC TREATMENTOF PULP AND PAPER MILL WASTEWATERS IN MEXICO

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THIS PAPER PRESENTS AN OVERVIEW OF THE TREATMENT OF KRAFT PULP MILL EFFLUENTS (KRAFT BLEACHED WASTEWATER AND KRAFT CONDENSATE) AND PAPER MILL WASTEWATER (WHITEWATER) IN ANAEROBIC HIGH RATE ADVANCED PROCESSES IN MEXICO. KRAFT BLEACHED PULP WASTEWATER (KB) WAS FED IN PARALLEL TO AN ANAEROBIC FLUIDIZED BED REACTOR (ANFBED, ACTIVATED CARBON AS MEDIUM SUPPORT) AND A DOWNFLOW ANAERÓBIC FILTER (DANFIL), BENCH SCALE AND MESOPHILIC CONDITIONS. IT WAS FOUND THAT THE REMOVAL OF ORGANIC LOAD WAS FAIR TO POOR, UP TO 30% ON CHEMICAL OXYGEN DEMAND (COD) BASIS. INTERESTINGLY, TOXICITY OF EFFLUENT WAS REDUCED IN A FACTOR OF 6 AS MEASURED BY THE DETOXIFICATION FACTOR BASED ON DAPHNIA MAGNA BIOASSAYS. THERE WAS NO SIGNIFICANT DIFFERENCE BETWEEN REACTOR PERFORMANCES IN THE RANGE OF LOADING RATES EXPERIMENTED.

RAW WHITEWATER WAS TREATED IN AN ANFBED (GROUND COCONUT SHELL AS MEDIUM SUPPORT). ORGANIC MATTER DEPURATION WAS IN THE RANGE BETWEEN 56 TO 74% COD, PROVIDING A GOOD DEGREE OF PRETREATMENT. RETENTION OF FIBROUS SOLIDS INSIDE THE REACTOR WAS OBSERVED, AND THEIR SETTLING CHARACTERISTICS IMPROVED. THE PROCESS WAS INTENSIVE (HYDRAULIC RETENTION TIMES AS SHORT AS 0.4 DAYS) AND BIOGAS PRODUCTION WAS ATTRACTIVE (UP TO 0.6 M3/M3/D).

KRAFT CONDENSATE (KC) SUPPLEMENTED WITH NUTRIENTS AND ALKALINTY WAS TREATED IN AN ANFBED, DANFIL AND HYBRID UPFLOW ANAEROBIC SLUDGE BLANKET REACTOR (HUASB). ORGANIC MATTER REMOVAL UP TO 82% COULD BE ACHIEVED (COD) AT HYDRALIC RETENTION TIMES AS SHORT AS 0.4 DAYS IN THE ANFBED. THE HUASB SHOWED NO GRANULATION OF THE ACTIVE SLUDGE AND HYDRAULIC INSTABILITY. IT WAS CONCLUDED IN GENERAL THAT KC DEPURATION WAS SATISFACTORY, AND THAT THE ANFBED OUTPERFORMED THE OTHER TWO REACTOR PEFORMANCES.

IN OVERALL, HIGH RATE ADVANCED ANAEROBIC PROCESSES SHOWED TO BE A FEASIBLE ALTERNATIVE FOR PULP AND PAPER MILL WASTEWATER PRETREAT-

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MENT. THEY COULD ACCOMPLISH A BETTER DETOXIFICATION OF KEY EFFLUENTS THAN CONVENTIONAL AEROBIC PROCESSES ALONE AND SUBSTANTIAL SAVINGS IN AERATION COSTS (INVESTMENT AND OPERATION) CAN BE MADE.

INTRODUCTION

Four industrial activities account for nearly 90% of the industrial wastewater discharges in Mexico: sugar industry, pulp and paper manufacture (PPI), steel industry and the oil production and refining-Fig. 1. The Mexican PPI generates nearly 12% of the volume of the industrial wastewater discharges, and it is known that several chemical species contained in these wastewaters are toxic or refractory to biological degradation (Poggi et al., 1989).

Almost 75% of the Mexican pulp is manufactured using the Kraft process with pine wood as feedstock. The Kraft process involves the chemical digestion of wood chips with an alkline-sulfidized liquor, and the subsequent bleaching of the raw pulp with chlorine or other oxidant compounds. The bleaching is accomplished alternating alkaline extraction and oxidation (chlorine has been the standard choice for this), generating a wastewater that is known as Kraft bleaching effluent (KB). The KB contains chlorinated-organics (CO) of a wide range of families and molecular weights. The low molecular CO belonging to the family of chlorophenols, chloroguaiacols and chloroveratrols have been associated with toxicity to aquatic life and biological treatment. The spent liquor from chemical digestion is further concentrated by evaporation in multi-effect evaporator trains and burned for chemicals recovery and reuse. This concentration stage is the source of the so named Kraft condensate (KC). The bleached pulp is sent to Fourdrinier machines to form the paper. In Mexico, there is little integration between pulp and paper mills; that is, there are many paper mills that only manufacture the paper and no pulp. Their discharges are unique; they consist only of the purge of the Fourdrinier machine water circuit. The wastewater generated is known as whitewater (WW), high in suspended solids (from cellulose and fillers/texturizers) and low in dissolved organic matter.

The standard treatment provided by PPI to its effluents in developed countries has been aerated lagoons, in the best case (Oleszkiewicz, 1991). However, concerns regarding this approach have arisen due to lower than expected removal efficiencies, and reports of acute and sub-acute toxicity effects of treated effluents on aquatic life. Thus, development of treatment alternatives of PPI wastewater is necessary.

Pioneering work and encouraging results on the application of high-rate advanced anaerobic processes to PPI wastewaters have been reported since the early 80's (Norrman, 1982; Salkinoja-Salonen et al., 1983; Norrman & Narbuvold, 1984; Gillespie, 1984 to 1987; Tirsch, 1988). This work will review in a summarized way the results of research done in the CENTRE OF ADVANCED STUDIES AND RESEARCH (CINVESTAV DEL I.P.N.), MEXICO, on the anaerobic treatment of pulp and paper mill effluents, during a span of 3 years.

MATERIALS AND METHODS

KB effluent was treated in parallel in bench scale ANFBED and DANFIL, at 32 C. The ANFBED contained granular activated carbon as carrier medium (GAC). The whitewater was fed to an ANFBED with ground coco-nut shell acting as medium support. The KC (supplemented with nutrients and non-supplemented) was treated in and ANFBED (GAC), DANFIL and HUASB. The HUASB had non-reticulated polyurethane modules in the top part of the reactor. The experimental design involved a conservative start-up and acclimation and the determination of reactor performances at several steady states of decreasing hydraulic retention time (HRT). Bioassays with Daphnia magna (water flea, crustacean) were performed in raw and treated KB (Martínez-Jerónimo, 1990, private communication).
More detailed information on the experimental design, set-up, and analyses/monitoring can be found elsewhere (Poggi et al., 1987; Poggi et al.,1989a; Poggi et al., 1989b).

RESULTS AND DISCUSSION

Kraft bleaching effluent treatment

Table 1 shows the raw KB characteristics. It was a medium strength wastewater, acidic. It was neutralized up to pH 7.5 before feeding it to the reactors. The ratio BOD/COD was low (BOD-Biochemical oxygen demand), indicating a low aerobic biodegradability; the major part of the organic matter was recalcitrant in aerobic conditions.

The organic matter removal in the anerobic reactors was poor or marginal, up to 30% - Fig.2, slightly decreasing when the HRT decreased. However, this removal seemed to account for the organic toxic material, because the toxicity of the treated effluent was significantly less than the toxicity of the raw KB - Fig. 3. A detoxification factor (DF) was defined, as the ratio of the lethal concentration of raw KB to lethal concentration of treated effluent, where the lethal concentration was the proportion of wastewater in the mixture that would kill 50% of the Daphnia magna juveniles in the assay. A high DF means a good toxicity removal in the wastewater. DF up to 6 were achieved, interestingly at the shortest HRT, when the methanogenic regime in the reactors was not optimum. This suggests that detoxification in the reactors was performed by anaerobic non-methanogenic or facultative microorganisms. There were no significant differences between the performances of the ANFBED and the DANFIL, except perhaps for a somewhat higher DF in the ANFBED at shorter HRT.

It seemed that the anaerobic treatment of KB was beneficial from the toxicity removal standpoint. However, only 30% of aeration costs would be saved, and a subsequent large aerobic stage would be needed for post-treatment. It is plausible to believe that the

removal efficiency of organic matter in the aerobic stage could be boosted by the key toxicity decrease in the anaerobic stage. Further research is necessary to confirm this hypothesis.

Whitewater treatment

The raw whitewater was low-medium strength wastewater, high in suspended solids of poor settleability and very low aerobic biodegradability (Table 1). The ANFBED removed between 56-74% of the incoming organic load (COD basis) in the range of 2.7 to 0.4 days HRT - Fig. 4. Biogas yields were 75% of the maximum and biogas productivities reached a peak of 0.6 m3/m3/d The relationship between organic load removal rate and organic load was linear in the range of the HRT studied, suggesting a stable methanogenic regime The reactor was able to entrap/degrade a fraction of the incoming suspended solids; a blanket of sludge accumulated above the fluidized bed in the reactor. This sludge had a Sludge volumetric index (SVI) less than 100, and contained 0.02% Nitrogen (on dry matter basis). Whether this sludge could contribute to the overall degradation activity inside the reactor was not known and further research is needed to assess it.

The ANFBED performance compared favourably well with results of other researchers working with different reactor configurations (Table 2). The ANFBED exhibited a good organic matter removal efficiency, considering that it was fed a low strength, high in suspended solids, low biodegradability index (BOD/COD ratio) and low nutrient levels whitewater.

Kraft condensate treatment

The Kraft condensate was a low-strength wastewater, with little suspended solids, high pH but small buffer capacity, and almost none nutrient concentration. The biodegradability ratio BOD/COD was fair to good - Table 1.

The first set of experiments were run with supplemented KC (alkalinity, Nitrogen and Phosphorus, trace metals - Poggi et al., 1989a). Fig. 5 shows the organic matter removal efficiency of the three reactors. The HUASB lost part of its active biomass (in the form of light density, non-granular sludge) as consequence of hydraulic upset in the middle of the second HRT run (namely 1 day HRT). After reseeding it recovered reasonably well. The biogas productivity is shown in Fig. 6. In general, the ANFBED outperformed the other two reactor configurations when treating supplemented KC. It achieved removal efficiencies in the 70 to 82% range (COD basis) and the highest biogas productivities consistently.

The performance of upflow anaerobic filters (UANFIL) fed with non-supplemented KC is shown in Fig. 7. The anaerobic regime became unstable (acidogenic), and the COD removal efficiency fell down at the shortest HRT. These results suggested that the supplementation

of the KC was beneficial and could help to accelerate further the anaerobic processes (e.g., to achieve stable operation at shorter HRT).

CONCLUSIONS

The results of the work developed in the CINVESTAV del IPN lead to the following conclusions:

-the anaerobic treatment of pulp and paper mill effluents with high rate advanced processes is a feasible alternative,

-it could offer substantial savings in volume (investment) and aeration (operational) costs of the aerobic stages, and in some cases could eliminate the need of subsequent aerobic treatment,

-anaerobic reactors provided extra removal of toxicity in selected pulp-mill wastewaters,

-in particular, the Anaerobic Fluidized Bed reactor (ANFBED, or RANLEF in Spanish) is a very intensive and effective reactor configuration for industrial wastewater treatment.

ACKNOWLEDGMENTS

The authors are indebted to Mr. E. Lemus (Rohm & Haas), Ms. María Luisa Torrecillas and Mr. R. Corona de la Vega (ECOTEC S.A. DE C.V.) for making this presentation possible. Also, the authors wish to thank Mr. Alfredo Hernández-González, Mr. Pablo Nava, Ms. Elvira Ríos (CINVESTAV DEL I.P.N., Dept. Biotechnol. and Bioeng.) for laboratory analytical work; Prof. E. Martínez-Jerónimo (National School of Biological Science, I.P.N.) for his cooperation with bioassays, Prof. C. Durán de Bazúa (Faculty of Chemistry, U.N.A.M.) for her input in whitewater treatability studies, and Mr. F. Calzada-Mejicano (ICAITI, Guatemala) for funding the experiments with pulp-mill effluents. Supply of wastewaters by four major Mexican pulp and paper companies are gratefully acknowledged.

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Table 1: Characteristics of raw PPI wastewaters in this work

Parameter	Kraft bleaching	Whitewater	Kraft condensate
pH	2.4	7.0	9.3
Total alkalinity, mg $CaCO_3$/L	0	312	64
Conductivity, µSiemen/cm	---	---	180
COD, mg O_2/L	2430	2064	1240
BOD7, mg O_2/L	490	596	880
BOD/COD	0.2	0.3	0.7
Total suspended solids, mg/L	270	366	5
N Kjeldahl, mg N/L	---	---	14
Phosphate, mg PO_4/L	3.5	1.7	2.6
Sulfate, mg SO_4/L	35.8	146	17
Sulfide, mg S/L	0	0	3.4
Chloride, mg Cl/L	2400	---	---

Table 2: Comparison of performances of several reactors treating whitewater

Influent	Reactor Configuration	Supplementation d	e	f	Loading Rates[g]	COD Removal Efficiency
TMP-mill wh.w. clarified BOD/COD=0.44	ANFIL[a]	Yes	No	No	0.2-4.0[h]	30–60%
bleachery effluent plus paper-mill wh.w. plus WAS raw BOD/COD=0.60	ANCON[b]	No	No	No	1.3–1.6	69–78%
Idem except that BOD/COD=0.40	ANCON[c]	No	No	No	0.4–0.8	72–87%
TMP-mill wh.w. clarified BOD/COD=0.59	UASB[a]	Yes	Yes	Yes	5–8	60–70%
paper-mill wh.w. raw BOD/COD=0.25	ANFBED[a]	No	No	No	0.8–6.6	56–74%

a: bench scale

b: pilot scale

c: full scale

d: Alkalinity

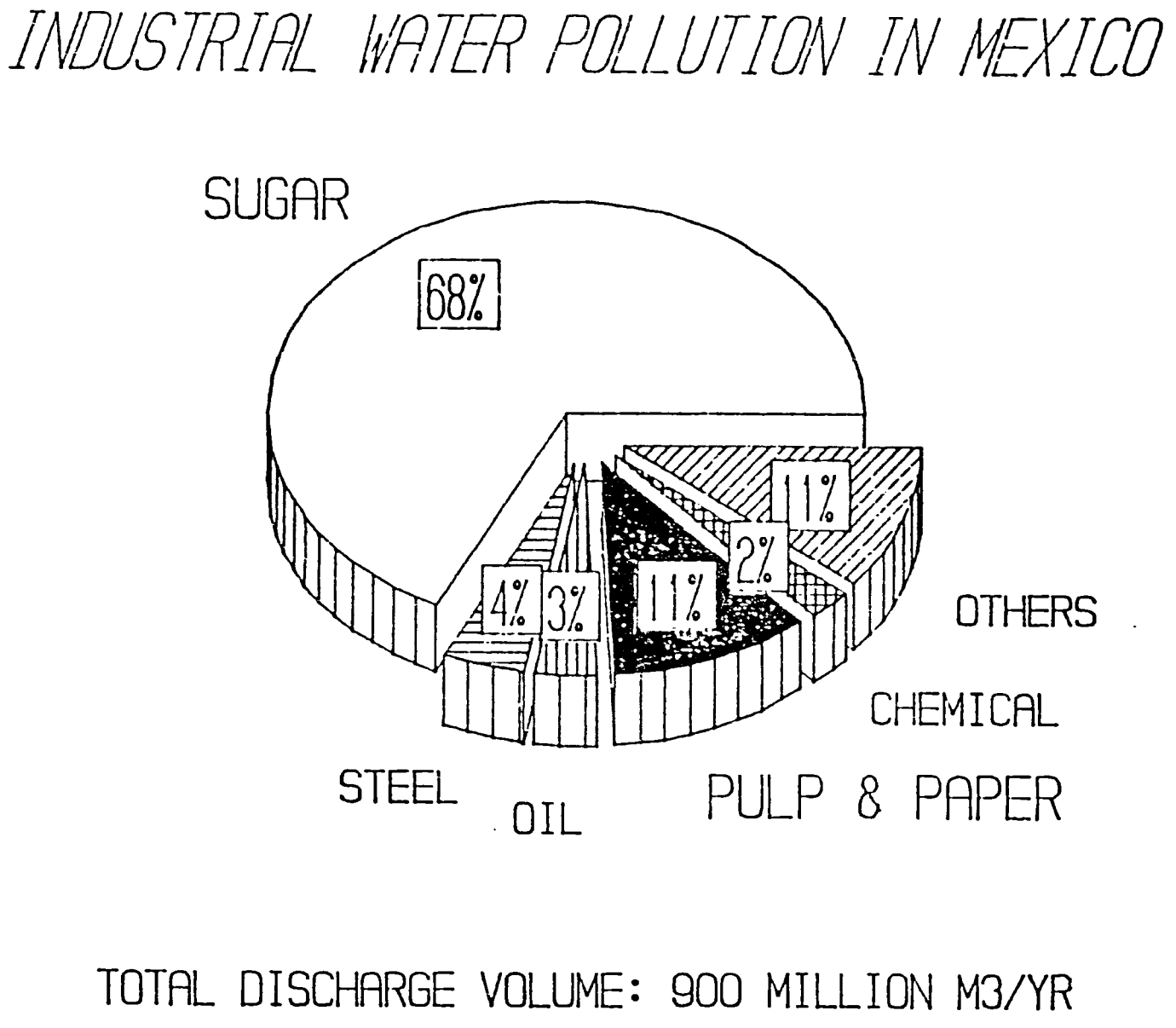
e: Nutrients (Nitrogen and Phosphorus)

f: trace elements

g: kg COD/m^3-d

h: kg BOD_5/m^{3-d}

Fig. 1: Industrial wastewater discharges in Mexico



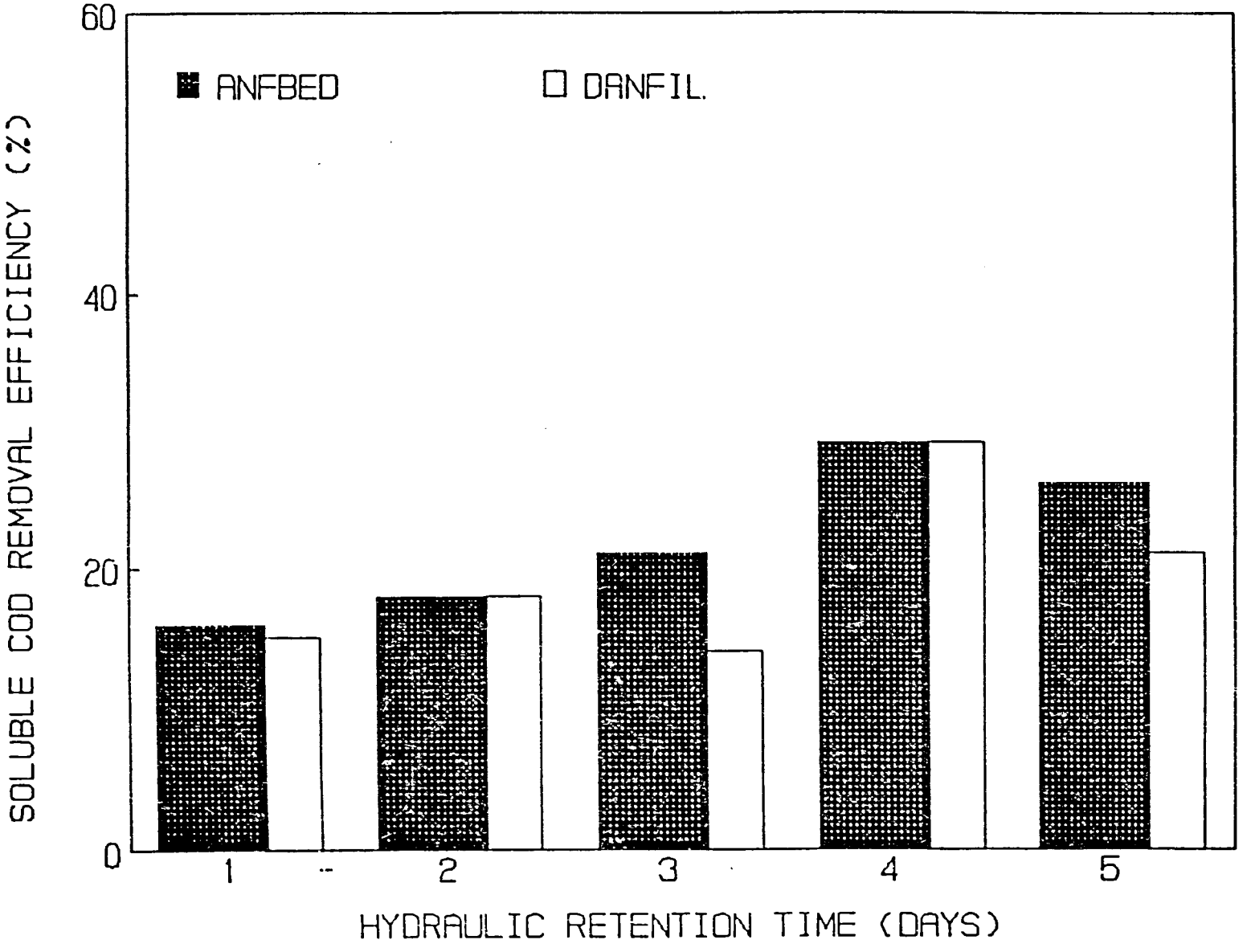


Fig. 2: Kraft bleaching effluent treatment-removal efficiency of COD as function of the hydraulic retention time.
ANFBED, Anaerobic fluidized bed reactor
DANFIL, Downflow anaerobic filter reactor

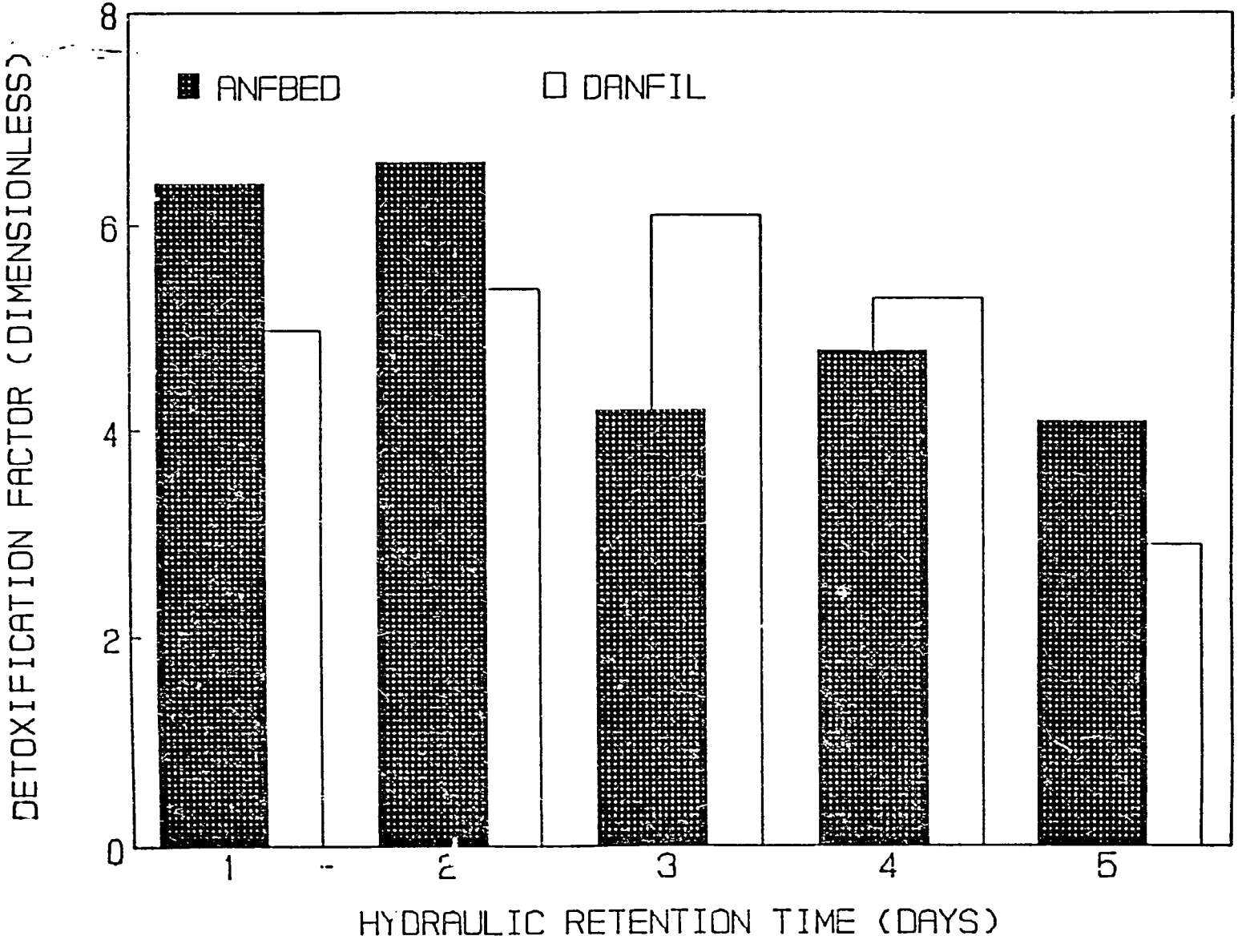


Fig. 3: Kraft bleaching effluent treatment-detoxification as function of the hydraulic retention time.
ANFBED, Anaerobic fluidized bed reactor
DANFIL, Downflow anaerobic filter reactor

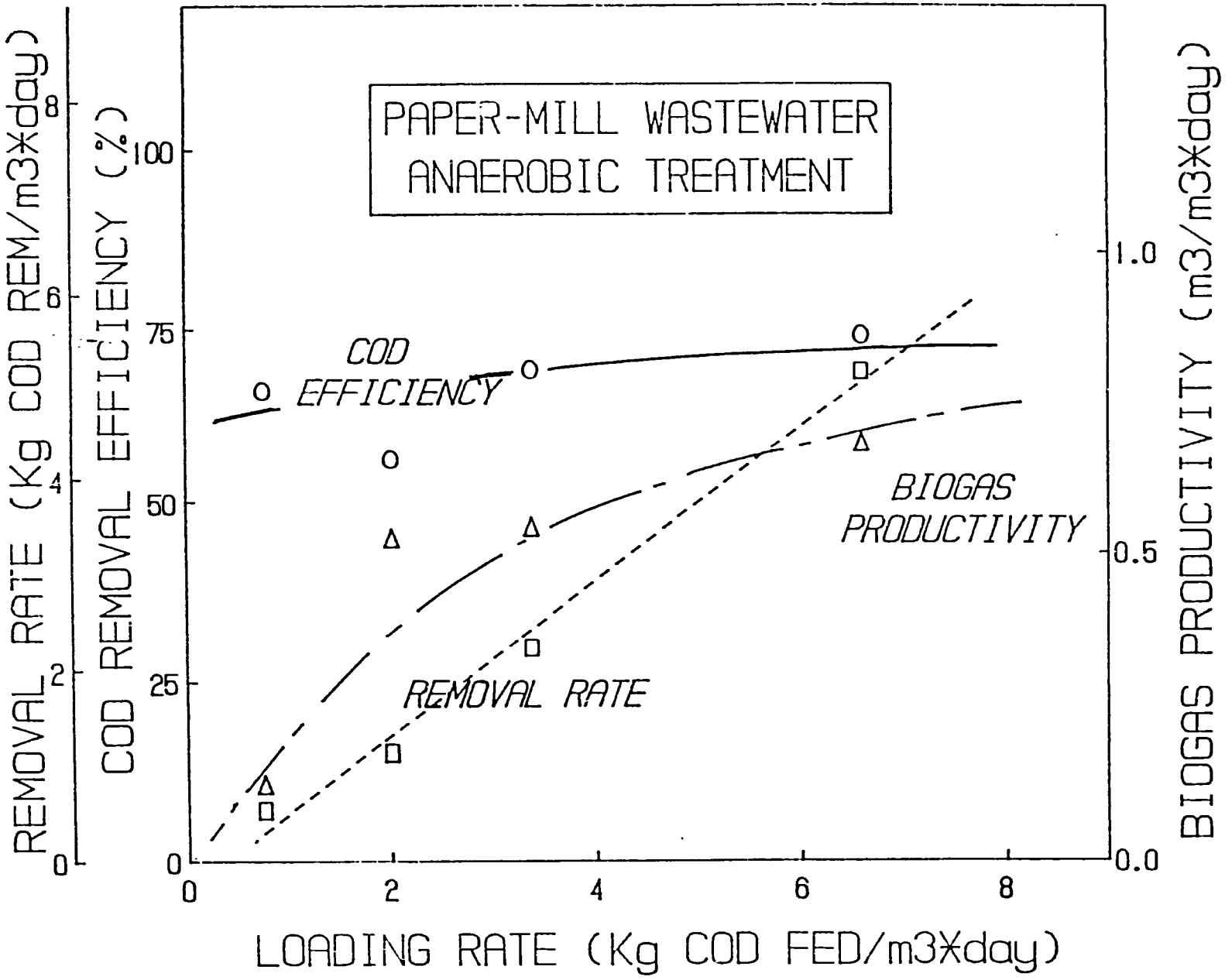
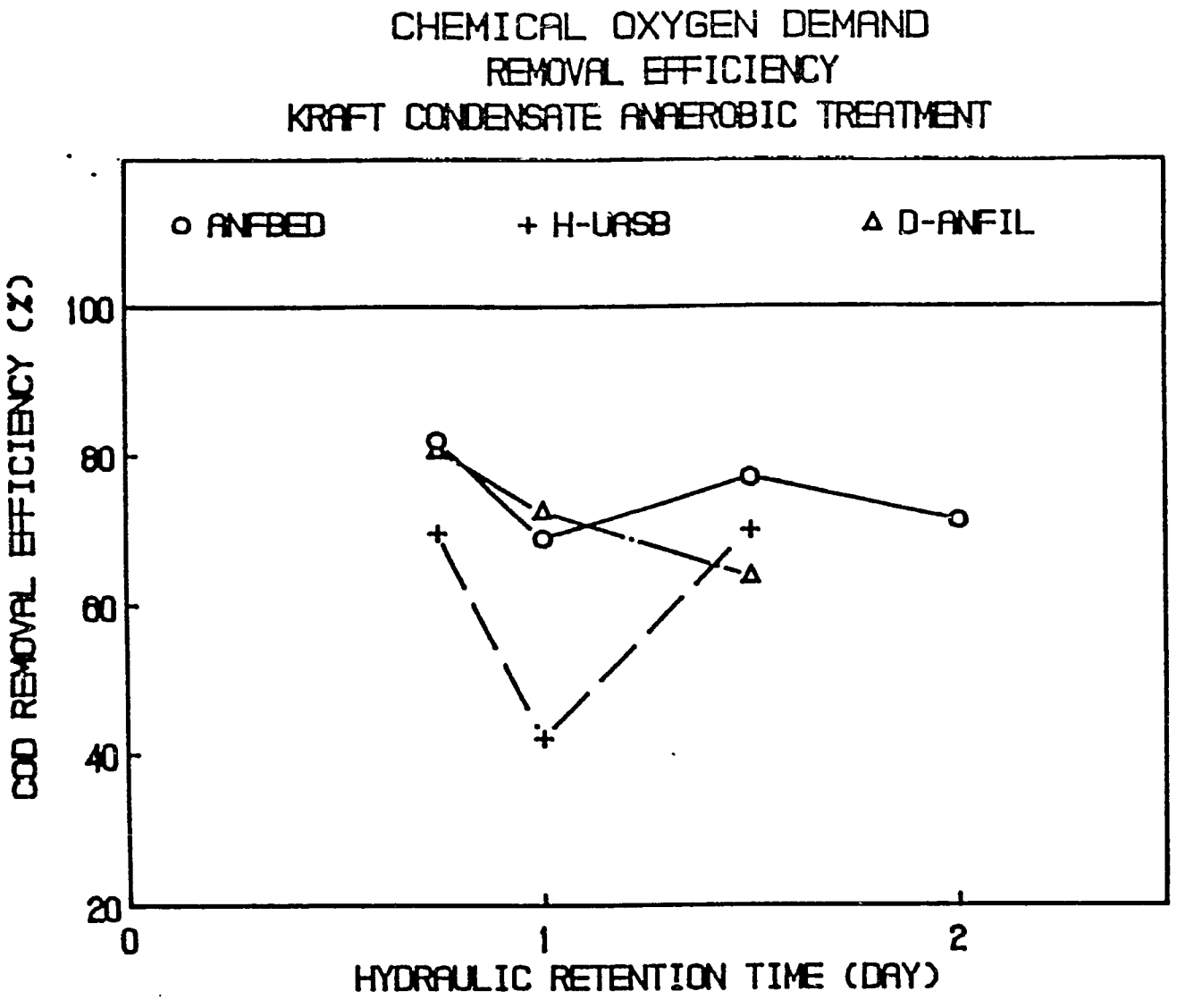


Fig. 4: Whitewater treatment in an Anaerobic fluidized bed reactor.

Fig. 5: Kraft condensate treatment-removal efficiency of COD as function of the hydraulic retention time.
ANFBED, Anaerobic fluidized bed reactor
HUASB, Hybrid upflow anaerobic sludge blanket
DANFIL, Downflow anaerobic filter reactor



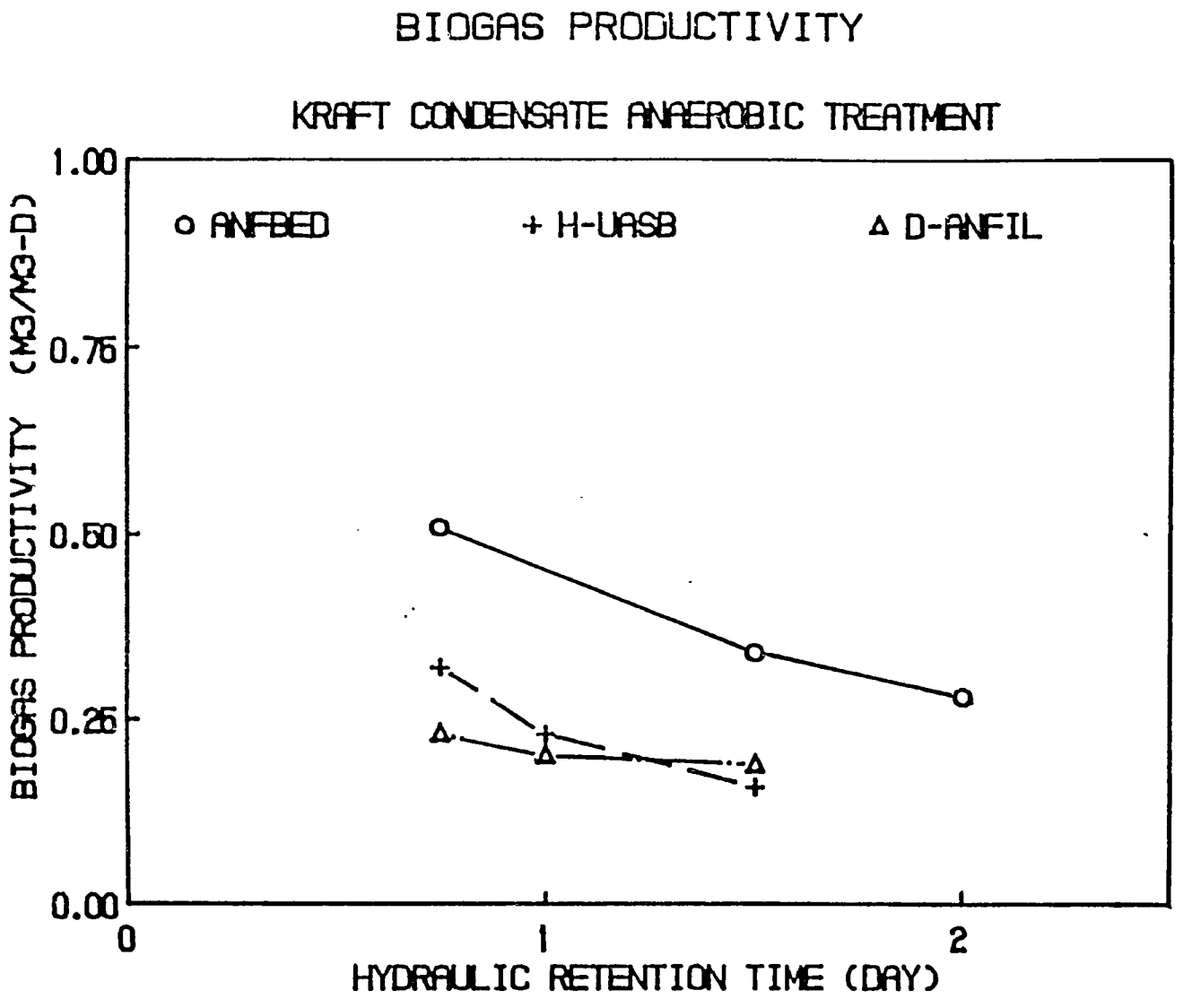


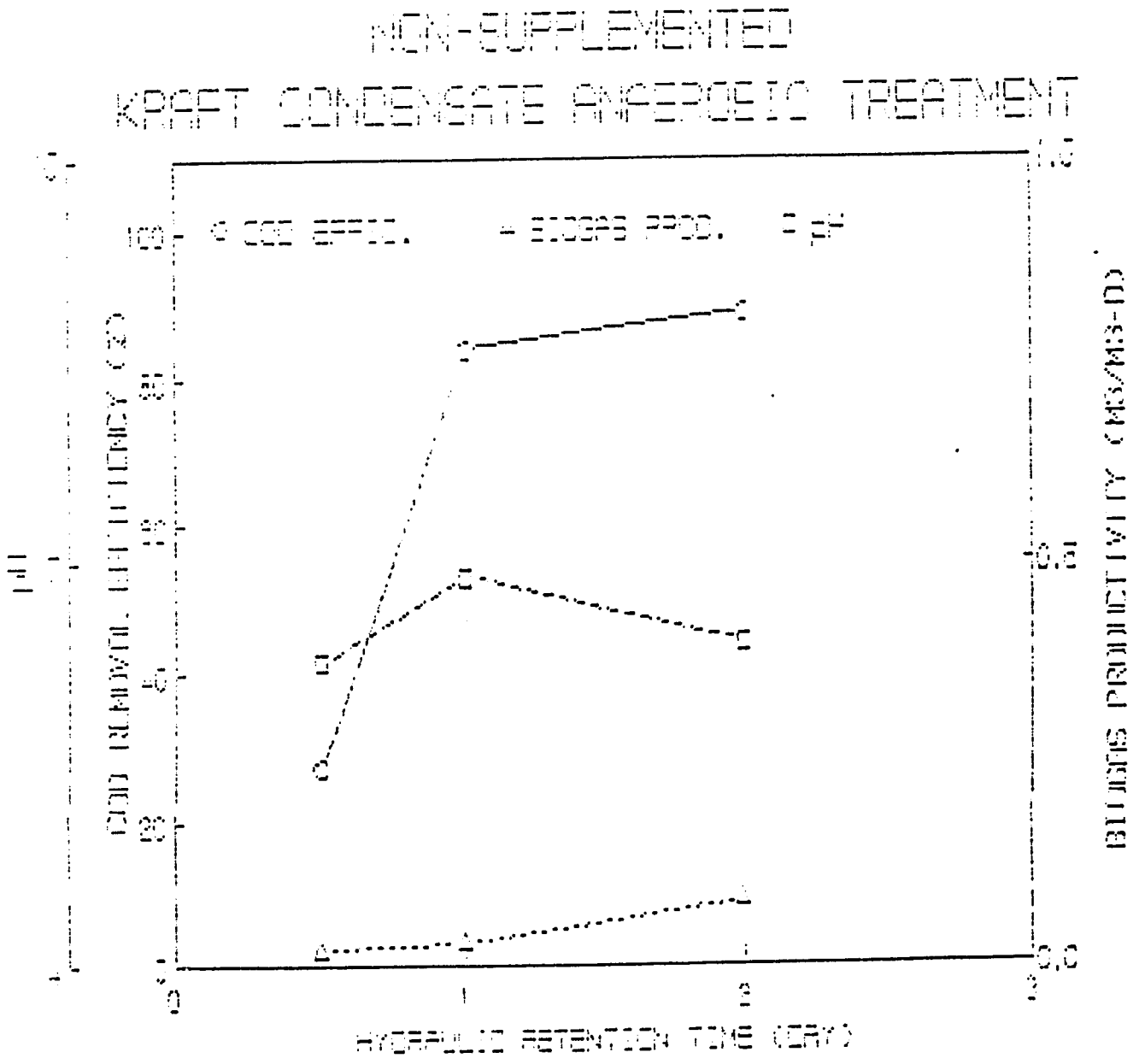
Fig. 6: Kraft condensate treatment-biogas productivity as function of the hydraulic retention time. Keys same as in Fig. 5

CURRICULUM VITAE

THE GROUP OF BIOENGINEERING OF ADVANCED ANAEROBIC PROCESSES STARTED ITS ACTIVITIES IN THE CINVESTAV (CENTRO DE INVESTIGACION Y DE ESTUDIOS AVANZADOS, MEXICO) SINCE 1984. IT WAS THE FIRST RESEARCH TEAM FOCUSING ON THIS TOPIC IN MEXICO CITY, ESPECIALLY IN THE DEVELOPMENT OF THE ANAEROBIC FLUIDIZED BED REACTOR (ANFBED OR RANLEF IN SPANISH ACRONYM). THE CINVESTAV HAS ALREADY FILED A PATENT APPLICATION FOR THIS REACTOR AND IS LOOKING FOR COOPERATIVE WORK WITH THE PRIVATE SECTOR IN ORDER TO EXPLORE FULL SCALE APPLICATIONS OF THIS TECHNOLOGY.

HECTOR POGGI-VARALDO IS URUGUAYAN BY BIRTH (HE IS AN EXTREMELY BAD FOOTBAL SOCCER PLAYER), HE EXPECTS TO BE A MEXICAN BY ADOPTION IN THE NEAR FUTURE, AND PERHAPS WILL APPLY FOR CANADIAN CITIZENSHIP IN THE LONG RUN, WHATEVER IS FIRST. HE IS A CHEMICAL ENGINEER, HOLDS A MASTER IN ENVIRONMENTAL ENGINEERING AND HOPEFULLY WILL EARN A PH.D. DEGREE IN ENVIRON. ENG. IN FEW MONTHS MORE. HE WORKS AS AN ASSOCIATE PROFESSOR IN THE CENTRE FOR ADVANCED STUDIES AND RESEARCH, CINVESTAV DEL IPN, MEXICO CITY, MEXICO, AND RECENTLY HE WON A POSITION AS ASSISTANT PROFESSOR IN THE UNIVERSITY OF MANITOBA, CANADA. HE HAS WORKED FOR THE URUGUAYAN AND MEXICAN PRIVATE SECTOR BEFORE GOING INTO THE EXCITING ACADEMIC WORLD. HE HAS CONSULTED IN WATER AND WASTEWATER TREATMENT IN URUGUAY, MEXICO AND CANADA.

Fig. 7: Non-supplemented Kraft condensate treatment in Upflow anaerobic filters



6. COMBINED BIOLOGICAL WASTEWATERS TREATMENT ANAEROBIC DIGESTION AND ALGAL GROWTH

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* Accepted for publication in the VI International Symposium on Anaerobic Digestion. Sao Paulo, Brasil, 1981.

COMBINED BIOLOGICAL WASTEWATER TREATMENT ANAEROBIC DIGESTION AND ALGAL GROWTH

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ABSTRACT

A two-phase biological process comprised of anaerobic (digestion in hybrid reactors) and aerobic (algal grwoth) stages was studied. Two industrial wastewaters were used as test substrates: 1) bleaching effluents from the Kraft pulping process and 2) vinasses (distillery spent slops) from ethanol fermentation of sugar cane molasses.

A 53% reduction of the organochlorinated compunds of kraft bleaching effluent was obtained with a 4-day HRT anaerobic digestion, with some further reduction in the algal phase (uo to a total of 78% in 2 days), using Spirulina algae. Detoxification, the major objective in this case, was effective as measured by liberation of chlorides.

Using vinasses with 64% non-volatile solids, the anaerobic digestion reached a 24% reduction of the COD under high loading rates (58-75 g/l.d). Further COD reductions (34%) were achieved through the growth of Spirulina in diluted effluents of the digestor. The filamentous algal biomass can be harvested and used for feed.

KEYWORDS

Anaerobic Digestion, Alga, Wastewaters, Spent Slops, Pulping, Kraft, Bleaching, Methane.

INTRODUCTION

The utilization of anaerobic digestion for the treatment of agroindustrial effluents has shown definite advantages, specially for some compounds tha appear recalcitrant under aerobic conditions. The trend of use increased with the development of the high-rate digestors, including the hybrid types. A further purification of the digestor effluent is frequently needed to reach environmentally acceptable levels of hazardous compounds or chemical oxygen demand (COD).

Two effluents have been selected as examples in this work. One comes from the pulp and paper production (bleaching effluent) and the other from the ethanol production from sugar cane (distillery spent slops or vinasses). These have been selected for comparison purposes, in order to have: 1) a concentrated effluent with high COD but without organchlorinated compounds (vinasses) and a concentrated effluent with those substances (bleaching wastewaters).

The anaerobic digestion of the above effluents is not new. Treatment of pulp and paper industry wastewaters can be found in papers of the last decade (Cocci et al, 1982; Salkinoja-Salonen et al, 1983 being some early examples). At the same time, the biodegradation of lignin-derived molecules under anerobic conditions were being described (Colberg and Young, 1982). Both aerobic and anaerobic treatments have been suggested for the treatment of molasses based fermentation wastes (Skogman, 1979).

In papermills effluent treatment plants applying the activated sludge principle, an increase in degradation performance can be achieved by anaerobic pretreatment of high-strength partial flows (Huster and Möbius, 1990).

Oxygen for the aerobic treatment of the wastewaters can be obtained from biogenic production by microalga during photosyntesis and from absorption of atmospheric oxygen through the air-water interphase. This is the basis for the High Rate Algal Pond systems (Grobbelaar et al, 1988) and it represents an interesting alternative to the costly mechanically aereated basins. In fact, anaerobically digested swine manure have been treated by microalgae, including cyanobacteria (de la Noüe and Basséres, 1989; Pouliot et al, 1989).

The dual system whose results are described in this artible includes the high rate anaerobic digestion in a hybrid unit using unreticulated polyurethane sponge supports and a section filled with pumice stone. The sponges have proven to be useful as bacterial supports, in the treatment of other polluting agroindustrial effluents (Calzada et al, 1984). The liquids from the anaerobic digestion are treated in glass cultivation containers with alga (Spirulina and Anabaena).

It must be stressed that the combined treatment may have several objetives: degradation or organochorinated compounds, COD reduction, biomass production for feed and biogas production when possible. In the present work, the achievment of these different objetives is emphasized.

MATERIALS AND METHODS

Microorganisms:
Mixed anaerobic bacterial cultures used as inocula were obtained from active methanogenic digestors associated to the retting of banana stems. Adaptation to the kraft effluents and to the vinasses was done gradually by partial substitution, untial a steady state was achieved (determined by constant chemical or gas production parameters). A period equivalent to more than 10 hydraulic retention times (HRT) was then allowed before the results were taken as definitive. Cyanobacteria used in subsecuent experiments were: Spirulina platensis ICAITI A993 (UTEX B629) and Spirulina sp. ICAITI A987, selection based on preliminary experiences that included other alga. Light limitation was detected when more than 20% effluent in tap water used, so this was the upper limit set for the experiments.

Wastewaters:
Bleaching effluents from Kraft pulping of Pinus chips were produced under laboratory conditions simulating those of the average pulp production (See table 1).

TABLE 1 Kraft Pulp Bleaching Wastewaters

Sample	COD (g/l)	Ocl gcl/l	Cl- gcl/l	pH
1	1.85	0.13	4.34	6.4
2	1.30	0.15	4.34	6.5
3	1.46	0.10	4.42	6.6
4	1.47	0.09	3.78	6.7
5	1.89	0.10	4.07	6.6
6	2.06	0.11	4.10	6.7

Vinasses were obtained from the DARSA distillery in Santa Lucía, Guatemala (See table 2). These vinasses originated from the slops of the distillation of ethanol fermentation broths, in which molasses, and not concentrated virgin syrups, had been used as the carbohydrate source. This is relevant to the present research, since the vinasses from molasses are more recalcitrant to biological treatments than those of virgin syrups.

TABLE 2 Distillery Spent Slops

Sample Group	Total S (%)	VS (%)	COD (g/l)	pH
1	5.6	1.7	58.0	4.6
2	5.7	2.3	56.5	4.6
3	8.5	4.6	82.2	4.3

Anaerobic Treatment:
For the anaerobic treatment of bleaching effluents, 1.5-liter glass columns packed with thin pieces of unreticulated polyurethane sponge (0.01m) in vertical position in the upper half. Upflow was obtained using peristaltic pumps controlled by timer. The temperature was kept at 35-37OC by operating the columns in a controlled temperature room. Chloride and organochlorinated compounds were determined in the feed and in the effluent, by the potentiometric method (Keith, 1981). Other chemical analysis of the feed and effluent were done as described elsewere (Calzada et al, 1984). Gas production was determined by displacement of water.

Vinasses were treated using 1.5-liter glass columns packed with thin pieces of unreticulated polyurethane sponge (o.01m wide) in vertical position in all the column (Column A) and in the upper half of the column (Column B). In this second case, the lower half acted as a sludge accumulation chamber and was provided due to the insoluble solids present in the vinasses as suspended particles. HRT was one day. The other conditions were similar to those of the experiments with bleaching effluents.

Aerobic Treatment:
To grow the inocula for the algal experiments, synthetic media Spirulina medium was used (Vonshak, 1986). Natural north light was used, in the regular daily cycle, for the preparation of the inocula.

The algal biomass was separated by centrifugation and inoculated in water dilutions of the anaerobically digested wastewaters (3mg chorophyl/liter). Of these diluted "real" media, 150 ml were placed in 250 mlErlenmeyer flasks and maintained under constant illumination (400 foot-candle) in 12-hour alternate light and dark periods. The pH was initially adjusted to 8 using sodium hydroxide. The chlorophyl a was determined by ultrasonication of the sample, followed by an extraction with methanol, and then red in the spectrophotometer at 665 nm and corrected by the pigment factor (13.9 mg/l).

RESULTS AND DISCUSSION

The anaerobic digestion of vinasses or distillery spent slops used organic loads ranging from 30 to 70 g COD/l.d, using HRT of one day. Results for COD and volatile solids (VS) reductions, pH values in the effluent, gas production and methane concentration in the gas are shown in Table 3 for column A (fully packed) and in Table 4 for column B (half packed). It can be observed that the values are similar, suggesting that the lower part of the packed column is really acting as suldge accumulation chamber. Absorption of carbon dioxide in the liquid accounts for the high methane contents in the gas.

TABLE 3 Anaerobic Digestion of Vinasses
COLUMN A: Fully Packed with U.P. Sponge

LOAD (gCOD/l.d)	LOAD (grs/l.d)	% COD Reduction	% VS Reduction	Gas Prod. (v/v.d)	Methane (%)
33	24	15	26	3.3	73
33	21	23	25	2.6	55
36	22	17	23	3.7	83
39	28	22	26	3.0	43
56	32	11	27	3.4	50
59	34	12	13	4.0	63
60	39	10	--	1.9	56
66	30	21	12	3.9	46
70	37	25	22	2.3	84

TABLE 4 Anaerobic Digestion of Vinasses
COLUMN B: Halfpacked with U.P. Sponge

LOAD (gCOD/l.d)	LOAD (gsv/l.d)	% COD Reduction	% VS Reduction	Gas Prod. (v/v.d)	Methane (%)
29	17	24	14	3.5	82
33	24	22	36	3.2	57
33	24	41	43	4.2	50
33	21	28	33	3.4	51
36	22	23	30	3.9	74
38	17	31	13	4.6	92
39	28	16	18	3.4	48
66	30	19	19	4.8	41
70	37	23	22	2.3	62

The above results have been confirmed by the operation of a pilot plant (1500-liters) in the distillery (ICAITI, 1988), in which COD reductions of 30-35% and VS reductions of 18-21 were obtained with loads of 64-68 g COD/l.d using hybrid methanogenic reactor.

Table 5 shows the decrease of the COD in diluted effluent media, after 10 days of algal growth (stationary phase reached), using as a raw material for the dilutions effluent with 47 g COD/l.

TABLE 5 COD Reductions after Algal Growth (10 days) in Anaerobically Treated Vinasses

Effluent (%)	Spirulina platensis	Spirulina sp.
5	41	42
10	33	41
15	36	36
20	34	34

RAW Effluent (100%)=46.7 gCOD/l.

From the above results, the use of either Spirulina strain seems adequate for partial tertiary treatment. The light limitation problem must be overcome to increase the effectivity of the overall water purification process.

The growth of alga in the same period (10 days) is indicated in Table 6. The growth behaviour of Spirulina sp. under higher effluent concentrations is better.

TABLE 6 Algal Growth (10 days) in Anaerobically Treated Vinasses (mg Chl./l)

Effluent (%)	Spirulina platensis	Spirulina sp.
5	7.0	5.0
10	7.4	6.1
15	7.4	7.8
20	6.6	6.7

Initial Chlorophyl Conc: 3 mg/l

It must be stressed the advantage of growing algal biomass that can be used as feed, basically for poultry and fish. In fact, Spirulina grown on vinasse under the described circumstances do not present the possibility of heavy metal accumulations. The filamentous characteristics of the Spirulina cyanobacteria make it attractive for the recuperation process.

The sequential anaerobic-aerobic biological system used in the treatment of bleaching effluents from the kraft pulp production behave differently. No significant biogas production was obtained during the anaerobic stage and very small reduction (even none) in COD during the digestion. However, the organochlorinated compounds showed a decrease during the anaerobic digestion, with the corresponding liberation of cholrides. Reduction of both was observed in the algal stage. Table 7 shows the COD reductions, while Table 8 shows that of the organochlorinated compounds. Finally, Table 9 indicates the values for chlorides in the same groups of experiments, confirming the partition of the recalcitrant organochlorinated compounds.

TABLE 7 COD Reductions in Anaerobic-Aerobic Biological Treatment of Kraft Bleaching Wastewaters

Anaerobic Digestion			Aerobic Stage		
HRT (d.)	LOAD (gCOD/l.d)	% RED	% RED (2 d.)	% RED (5 d.)	Alga
5	1.46	0	17	10	S. platensis
5	1.46	0	0	0	S. sp.
4	2.41	4	24	19	S. platensis
4	2.41	4	19	22	S. sp.

TABLE 8 Reduction in Organochlorinated Compounds Anaerobic-Aerobic Biological Treatment of Kraft Bleaching Wastewaters

Anaerobic Digestion			Aerobic Stage		
HRT (d.)	LOAD (mg/l.d)	% RED	% RED (2 d.)	% RED (5 d.)	Alga
5	93	33	76	73	S. platensis
5	93	33	67	70	S. sp.
4	99	53	73	69	S. platensis
4	99	53	64	74	S. sp.

TABLE 9 Chlorides in Anaerobic-Aerobic Biological Treatment of Kraft Bleaching Effluents

HRT (d.)	Anaerobic Digestion LOAD (g/l)	Anaerobic Digestion Discharge (g/l)	Aerobic Stage Discharge 2 d.(g/l)	Aerobic Stage Discharge 5 d.(g/l)	Alga
5	4.1	4.4	2.7	2.7	S. platensis
5	4.1	4.4	4.0	4.0	S. sp.
4	4.5	4.0	3.1	3.0	S. platensis
4	4.5	4.0	3.6	3.4	S. sp.

The algal biomass for the experiments appear in Table 10. Both strains showed a similar behavior, but some late recovery seems present, unlike the above results for the reductions of COD and organochlorinated compounds.

TABLE 10 Algal Growth in Effluents from the Anaerobic Treatment of Kraft Bleaching Effluents

Alga	Chlorophyl (mg/l) 2d.	Chlorophyl (mg/l) 5d.	TRH in Ana. Reaction (d.)
Spirulina platensis	3.1	2.0	5
Spirulina sp.	3.4	4.6	5
Spirulina platensis	4.8	8.1	4
Spirulina sp.	5.1	7.0	4

CONCLUSIONS

The sequential utilization of anaerobic digestion and algal ponding for the treatment of recalcitrant effluents seems promising. Cost evaluations must be done for each particular situation in comparison with more conventional anaerobic-aerobic treatment processes in which mechanical energy is used to provide the oxygen required.

The combined biological treatment, when applied to distillery spent slops (vinasses), can provide three valuable products: partially purified water, algal biomass (feed) and biogas (fuel). In the case of an effluent with recalcitrant hazardous organochloride compounds, the purification in two stages overcomes the limitations of the aerobic systems for the degradation of those compounds and adds COD reductions. Use of the biomass obtained has not yet been fully explored.

ACKNOWLEDGMENTS

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7. ANAEROBIC TREATMENT OF KRAFT PULP MILL EFFLUENTS IN MEXICO

ANAEROBIC TREATMENT OF KRAFT PULP MILL EFFLUENTS IN MEXICO

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(2) Centre of Advanced Studies and Research, CINVESTAV del IPN, Dept. of Biotechnology and Bioengineering, P.O. Box 14-740, México D.F., 07000, México.
(3) ICAITI, Guatemala City, Guatemala, Central America.

This paper presents the results of the treatment of Kraft bleaching (KB) effluent from a Mexican pulp mill in anaerobic attached growth reactors. The KB was an acidic, medium strength wastewater on Chemical Oxygen Demand basis (COD), very poor in nutrients (Nitrogen and Phosphorus). The ratio BOD/COD was low (BOD-Biochemical oxygen demand), indicating a low aerobic biodegradability. Neutralized and non-supplemented-nutrient KB wastewater was fed in parallel to an anaerobic fluidized bed reactor (ANFBED, using activated carbon as biomass carrier) and a downflow anaerobic filter (DANFIL), both in mesophilic conditions, bench scale. Reactors were operated at low-medium organic loading rate conditions (medium to long hydraulic retention times, 1 to 4 days). Toxicity of raw KB and treated effluents were monitored with *Daphnia magna* bioassays. A detoxification factor (DF) was defined, as the ratio of the lethal concentration of raw KB to lethal concentration of treated effluent, where the lethal concentration was the proportion of wastewater in the mixture that killed 50% of the *Daphnia magna* juveniles in the bioassay.

The organic matter removal in the anaerobic reactors was fair to poor (up to 30% on COD basis) slightly decreasing when the HRT decreased. However, this removal seemed to account for the organic toxic material, because the toxicity of the treated effluent was significantly less than the toxicity of the raw KB. DF up to 6 were achieved, interestingly at the shortest HRT, when the methanogenic regime in the reactors was not optimum. This suggested that the detoxification in the reactors was performed by anaerobic non-methanogenic or facultative microorganisms. No significant differences between the performances of the ANFBED and the DANFIL were found, except perhaps for a somewhat higher DF in the ANFBED at shorter HRT.

It was concluded that the anaerobic treatment of KB was beneficial from the toxicity removal standpoint. Reactor configuration and activated carbon capability of the ANFBED did not seem to play a distinct role on reactor performance in the range of loading rates experimented. When compared with aerobic treatment, only 30% of aeration costs would be saved, and a subsequent aerobic stage would be needed for post-treatment. It is plausible to believe that the removal efficiency of organic matter in the aerobic stage could be boosted by the important toxicity removal in the anaerobic stage. Further research is necessary to confirm this hypothesis.

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* Abstract for presentation in the 46th. Purdue Industrial Waste Conference of 1992.

TREATMENT THROUGH ALGAL BIOTECHNOLOGY
(PROGRAM)
AUGUST 29-30, 1980

Biotecnologia en el manejo de algas

programa

Biotecnologia en el manejo de algas

- martes 29

8:00-9:00 inscripcion-registration

9:00-10:30 Parametros del crecimiento de algas-algal culture growth parameters (A. Vonshak)

10:30-10:45 cafe-coffee

10:45-12:00 Nutricion & medios de cultivo-Nutrition & culture media (A. Richmond)

12:00-14:00 almuerzo-lunch

14:00-15:30 Presentacion de caso Case presentations

15:30-15:45 cafe-coffee

15:45-17:00 continua-continues

programa

Biotecnologia en el manejo de algas

miercoles 30

8:00-9:45 Produccion de biomasa. Limitaciones biologicas- Biomass production. Biological limitations (A. Richmond)

9:45-10:00 cafe-coffee

10:00-12:00 biomasa. Tecnologia costos. Biomass Technology & costs (A. Vonshak)

12:00-14:00 almuerzo-lunch

14:00-15:30 Presentacion de caso Case presentations

15:30-15:45 cafe-coffee

15:45-17:00 continua-continues

programa

Biotecnologia en el manejo de algas

Jueves 31

8:00-9:45 Uso de algas.Alimento-
Algal use. Food & feed.
(A. Richmond)

9:45-10:00 cafe-coffee

10:00-12:00 Uso de algas.
Quimicos-Algal use.
Chemicals. (A. Vonshak)

12:00-14:00 almuerzo-lunch

14:00-15:30 Presentacion de caso
-Case presentations.

15:30-15:45 cafe-coffee

15:45-17:00 Discusion general-
General discussion.

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MARTES 30 AGOSTO 1989

SESION VESPERTINA

1. LA PRODUCTIVIDAD PRIMARIA Y EL NIVEL DE EUTROFIZACION DEL LAGO DE AMATITLAN

   ING. AGR. MSc. ROLANDO AGUILERA

2. TRATAMIENTO DE EFLUENTES DE BLANQUEO KRAFT

   ING. JOSE FRANCISCO CALZADA

3. ALGAS EN EFLUENTES DE LA DIGESTION ANAEROBICA DE LAS VINAZAS DE DESTILERIA DE RON

   ING. INF. ROLANDO PINEDA

4. IMPORTANCIA DEL CULTIVO DE MICROALGAS PARA LA ACUACULTURA

   LIC. CATALINA LOPEZ-GALVEZ, M. Sc.

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Institute de Investigaciones del Desierto, Boqer
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Autor de mas de doce publicaciones en revistas cientificas y de cuatro capitulos en libros sobre el tema.

-

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N.S. Ben Gurion University of the Negev
Ph.D. Ben Gurion University of the Negev

Author of more than twelve publication in referreed scientific journals and of four chapters in books on the subject

SUMARIOS DE TESIS

TRATAMIENTO DE VINAZAS MEDIANTE EL CRECIMIENTO DE MICROALGAS EN UN EFLUENTE TRATADO, PREVIAMENTE, EN ANAEROBIOSIS

TESIS

PRESENTADA A LA JUNTA DIRECTIVA DE LA
FACULTAD DE INGENIERIA
UNIVERSIDAD DE SAN CARLOS DE GUATEMALA

POR

DAVID ROLANDO PINEDA SALAZAR

PREVIO A OBTENER EL TITULO DE

INGENIERO QUIMICO

GUATEMALA, JULIO DE 1991.

16

SUMARIO

En el presente estudio se evaluó un tratamiento aerobio y el crecimiento de microalgas filamentosas en un efluente de vinazas, provenientes de una destilería de ron tratadas, previamente, por un sistema anerobio.

Las vinazas fueron diluídas para constituir un medio adecuado para el crecimiento de las algas filamentosas *Spirulima platensis* y *Spirulina sp.* Se estableció que para una carga orgánica de 9300 mg O=/l expresada como Demanda Química de Oxígeno (DQO), el tratamiento logra reducciones hasta de un 35% a una dilución de vinaza del 20% (v/v), y un periodo de diez días.

Asimismo, bajo condiciones controladas de iluminación (400 pie candela y temperatura (28°C) se evaluó el efecto de agregar diferentes substancias al medio como ayuda para el crecimiento de las microalgas. Como substancias alcalinizadoras se evaluó el hidróxido de sodio, bicarbonato de sodio y como fuente de nitrógeno, se evaluó la urea.

Se concluye que la adición de hidróxido de sodio al medio de vinazas mejora el crecimiento de alga *Spirulina platensis*, logrando una reducción de la DQO de 34.5% para una relación de dilución del 20% (v/v), siendo éste el tratamiento aerobio más eficaz entre los ensayos con bicarbonato de sodio y urea.

PINEDA SALAZAR, D.R. 1991. *TRATAMIENTO DE VINAZAS MEDIANTE EL CRECIMIENTO DE MICROALGAS EN UN EFLUENTE TRATADO, PREVIAMENTE, EN ANAEROBIOSIS.* Tésis de grado, Escuela de Ingeniería Quimica, Facultad de Ingeniería Universidad de San Carlos de Guatemala.

TRATAMIENTO DE LICORES BLANQUEO KRAFT EN DOS ETAPAS ANAEROBICA Y AEROBICA

TESIS

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RESUMEN

El presente trabajo comprende el tratamiento aneróbico y aeróbico de efluentes provenientes de blanqueo kraft para lograr reducir la cantidad de compuestos tóxicos presentes en dichos efluentes.

En el proceso aneróbico se utilizaron tres reactores de "alta productividad" que corresponden a una combinación de los diseños del reactor de flujo ascendente a través de lecho anaeróbico de lodos y del reactor de filtro anaeróbico. Los empaques utilizados en dicho tratamiento fueron poliuretano, poliuretano-piedra pómez y piedra pómez, lo que se inocularon con bacterias metanogénicas. En este tratamiento se encontró que el reactor que más se ajusta a este proceso es el empacado con piedra pómez, que al operar con tiempos de resistencia hidraúlica de cuatro días, temperatura de 37°C y pH de 7 a 7.2, se obtuvo un porcentaje de reducción de compuestos organoclorados de 62% con cargas que oscilan entre 20 y 25 mg/l/día, y además, en base a la porosidad, resistencia y costo del empaque, muestra ser el más eficiente. Los reactores metanogénicos mostraron ser poco eficientes en la reducción de demanda orgánica de oxígeno, ya que con cargas de 700 mg/l/día, el porcentaje de reducción oscila en un rango de 28 a 32%.

En el proceso aeróbico se utilizaron microalgas inoculadas en los efluentes provenientes de los reactores metanogénicos y se analizó tanto el crecimiento de microalgas como el porcentaje de reducción de compuestos tóxicos presentes en los efluentes. Este proceso se experimentó con cuatro tipos de microalgas: ***Spirulina sp, Spirulina platensis, Anabaena cylíndrica y Anabaena sp.*** La microalga que mejor operó en base a tiempo de resistencia hidraúlica (dos días), reducción de compuestos organoclorados (36%), reducción de demanda de oxígeno (14%), crecimiento y manejo durante el tratamiento fue la ***Spirulina platensis.***

GONZALEZ CABALLERO, J.G. 1991. *TRATAMIENTO DE LICORES BLANQUEO KRAFT EN DOS ETAPAS ANAEROBICA Y AEROBICA.* Tésis de grado, Facultad de Ingeniería de la Universidad Rafael Landívar. Guatemala.