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TREATMENT OF BIOMASS GASIFICATION WASTEWATERS USING WET AIR OXIDATION, SOLVENT EXTRACTION, AND REVERSE OSMOSIS

S.E. Petty
N.E. Bell
C.J. English, Jr.

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Pacific Northwest Laboratory
Richland, Washington 99352

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N. E. Bell
C. J. English, Jr.

Pacific Northwest Laboratory,
Battelle Memorial Institute
Battelle Boulevard
Richland, Washington 99352

ABSTRACT

Biomass gasification wastewaters (BGW) from Texas Tech University's biomass gasifier were treated using wet air oxidation (WAO), solvent extraction, and reverse osmosis (RO) technologies. These technologies were reviewed to determine if they could be competitive or complimentary to innovative biological technologies studied earlier.

This paper reviews results obtained from experimental studies completed on these three technologies during the past year. Wastewaters treated by WAO exhibited chemical oxygen demand (COD) removal percentages ranging from 0% at 5.1 MPa (750 psi) and 150°C to 85% removal at 13.8 MPa (2000 psi) and 300°C. Oxidation times varied from 20 to 180 minutes, although it was determined that the removal of COD became less dependent on reaction time as the severity of treatment conditions was increased. Color removal using WAO ranged from 56 to 99%. Solvent extraction studies indicate that none of the conventional solvents (i.e., methyl isobutyl ketone, n-butyl acetate, n-butanol, kerosene, toluene, tri-butyl phosphate, tri-n-octyl phosphine oxide, and mixtures of these compounds) are effective in the removal of organics; the best distribution coefficient of 1.3 was given by n-butanol. Hence, solvent extraction is probably not an effective treatment technology for BGW. RO studies on biologically treated BGW indicate 99.7 to 99.8% of the initial COD (~7000 mg/l) can be removed from these wastewaters. Essentially total removal of color was also observed.

Our results indicate that BGW should be treatable to an acceptable discharge level by using WAO, biological, and RO technologies in a

three-step series. This conclusion may be helpful to industry in deciding how to best treat BGW to acceptable discharge levels.

INTRODUCTION

Conversion of biomass to a useful source of energy, through gasification, generates wastewaters which must be treated prior to discharge to the environment. Demonstration of cost-effective wastewater treatment technologies is necessary for many reasons(1): 1) to factor wastewater treatment costs into estimates of costs for commercial scale production facilities; 2) to assess the environmental impacts and health risks of discharged effluents; and 3) to assess the impact of recycled effluents on commercial scale process performance (if applicable).

The principal task of this U.S. Department of Energy (DOE) sponsored research effort has been to evaluate existing and promising treatment technologies for the treatment of BGW by conducting bench-scale experimental studies.

This paper presents results obtained from physical-chemical experiments conducted on BGW from January to September 1981. Treatment technologies evaluated include WAO, solvent extraction, and RO. Earlier investigations by Maxham and co-workers(1-4) emphasized treatment of BGW using innovative biological technologies. Comparison of the effectiveness of physical-chemical technologies with biological technologies has resulted in suggestions for potentially effective treatment schematics for BGW.

WASTEWATER CHARACTERISTICS

Wastewater samples for these studies were obtained from the Synthesis Gas From Manure (SGFM) gasifier at Texas Tech University in Lubbock, Texas. Once received, these samples were Whatman No. 1 filtered (to remove coarse suspended solids) and stored at 4°C. Six different BGW samples have been used for treatability studies at Pacific Northwest Laboratory (PNL). Analyses of these samples are presented in Table 1. COD values ranged from 30,000 to 70,000 mg/l for the six samples. BGW6, which was used in this study, had a pH of 7.4 and a COD of 30,000 mg/l. The major organic contaminants contributing to these high COD values have been reported to be carboxylic acids(5). Testing at PNL has confirmed the presence of acetic and propionic acids in BGW6.

TABLE 1. Analyses of Biomass Gasification Wastewaters

	BGW 1[1]	BGW 2[1]	BGW 3[1]	BGW 4[1]	BGW 5[1]	BGW 6[2]	Kao[3]
pH, standard units	3.9	3.5	3.7	5.5	4.0	7.4	2.3
Chemical Oxygen Demand, mg/l	45,800	43,600	47,600	67,900	32,800	30,800	43,960
Total Organic Carbon, mg/l	15,200	15,300	16,700	22,900	10,900	12,730[4]	
Total Kjeldahl (Organic) Nitrogen, mg/l	-	200	95	5,462	157	5,690[4]	
Total NH_3-Nitrogen, mg/l	194	18	16	2,330	32	3,300[4]	

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[1] Maxham 1981.

[2] This work.

[3] Kao 1980.

[4] English 1981.

WET AIR OXIDATION(6)

The application of WAO for degradation of wastewater organics has been addressed by many investigators(7-10). In general, it is felt that degradation of organics by WAO proceeds in three steps: 1) thermal decomposition, 2) partial oxidation, and 3) complete oxidation. Carboxylic acids, formed as intermediates from partial oxidation of larger molecules, are somewhat resistant to further oxidation. As complex organics are converted to carboxylic acid intermediates, the concentration of acids in the wastewater may increase in less severely treated wastewaters. This often results in a greater decrease in COD than in biochemical oxygen demand (BOD) for the treated wastewaters. Use of higher operating temperatures and pressures (increased severity in operating conditions) will oxidize carboxylic acids to CO_2 and water, but operating at more severe conditions is often economically unattractive.

Experimental Equipment and Operating Conditions

Experimental apparatus for WAO experiments centers around the autoclave (Figure 1) which is a closed vessel in which samples may be subjected to high temperatures and pressures. The design of this autoclave was based on designs of autoclaves by other researchers(11-14). Based on these earlier studies, it was determined that a stirred (1000 rpm) autoclave would be suitable for laboratory scale WAO experiments. The autoclave (3.78 l) was constructed of Hastelloy C, to resist corrosion, and equipped with a magnetically coupled stirrer. The autoclave was also equipped with a thermocouple for temperature control, a high pressure regulator for pressure control, and a cooling coil for quenching the reaction.

Operating conditions were chosen based on actual WAO treatment operations and similar experimental studies by other researchers(10,11,13,14). Operating temperatures and pressures were selected to minimize sample volatilization; in most experiments no more than 7% of the sample volume was evaporated. Experimental operating conditions are summarized in Table 2.

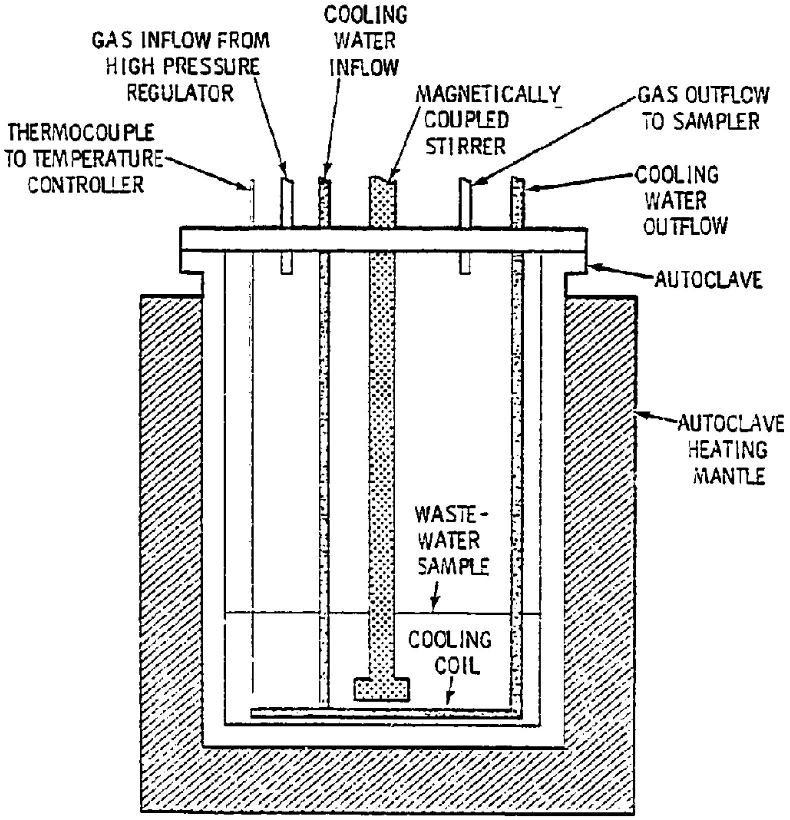

FIGURE 1. Laboratory Scale Wet Air Oxidation Apparatus

TABLE 2. Operating Conditions for WAO Experiments

Run No.	Temperature (°C)	Pressure, MPa (psi)	Reaction Time, min	Sample Vol, ml
1	150	5.2 (750)	20	690
2	150	5.2 (750)	40	690
3	150	5.2 (750)	60	690
4	150	5.2 (750)	120	690
5	150	5.2 (750)	180	690
6	200	6.9 (1000)	20	780
7	200	6.9 (1000)	40	780
8	200	6.9 (1000)	60	780
9	200	6.9 (1000)	120	780
10	200	6.9 (1000)	180	780
11	250	10.3 (1500)	20	980
12	250	10.3 (1500)	40	980
13	250	10.3 (1500)	60	980
14	250	10.3 (1500)	120	980
15	250	10.3 (1500)	180	980
16	300	13.8 (2000)	20	1130
17	300	13.8 (2000)	40	1130
18	300	13.8 (2000)	60	1130
19	300	13.8 (2000)	120	1130
20	300	13.8 (2000)	180	1130

For the apparatus used in these experiments, 13.8 MPa (2000 psi) was the maximum pressure suggested for use. Consequently, operating pressures used ranged from 5.2 MPa (750 psi) to 13.8 MPa (2000 psi), while temperatures used ranged from 150°C to 300°C. Reaction times selected spanned the range of times reported for WAO applications -- 20 to 180 minutes.

Experimental Results and Discussion

Extent of oxidation was measured by reduction of COD and color with reaction time. Figures 2 and 3 present COD and color removal, respectively, for various operating temperatures and pressures. Results show that the degree of oxidation was a strong function of temperature. No COD removal was observed during any of the low temperature runs (150°C, 5.2 MPa). As the temperature of reaction was increased, COD removal also increased. This trend is supported by results from previous studies(7,9,14-16). At the most severe operating conditions (300°C, 13.8 MPa) COD removal percentages of 80% were observed.

The effect of reaction time on COD removal also appeared to be strongly dependent on temperature. At an operating temperature of 300°C, COD removal was essentially independent of reaction time. However, as the operating temperature dropped below 300°C, reaction time began to influence COD removal. At these lower temperatures, for a fixed operating temperature, increased reaction time increased COD removal.

Color removal data (Figure 3) followed the same trends as observed in the COD removal versus reaction time curves; more color was removed as the severity in operating conditions increased. Since color in wastewaters is normally caused by long chained aliphatic or large aromatic organic molecules, color removal offered a qualitative means for evaluating the mechanisms by which the organics were destroyed. For each temperature and pressure, more color removal than COD removal was obtained for a given reaction time, particularly at the low temperatures. In general, these results indicate a stepwise degradation was occurring. For example, greater than 50% color removal was obtained with the low temperature runs while no COD removal was obtained. This would imply that hydrolysis of large, highly colored organics was occurring without any oxidation. For the 300°C temperature run, 98% color removal was

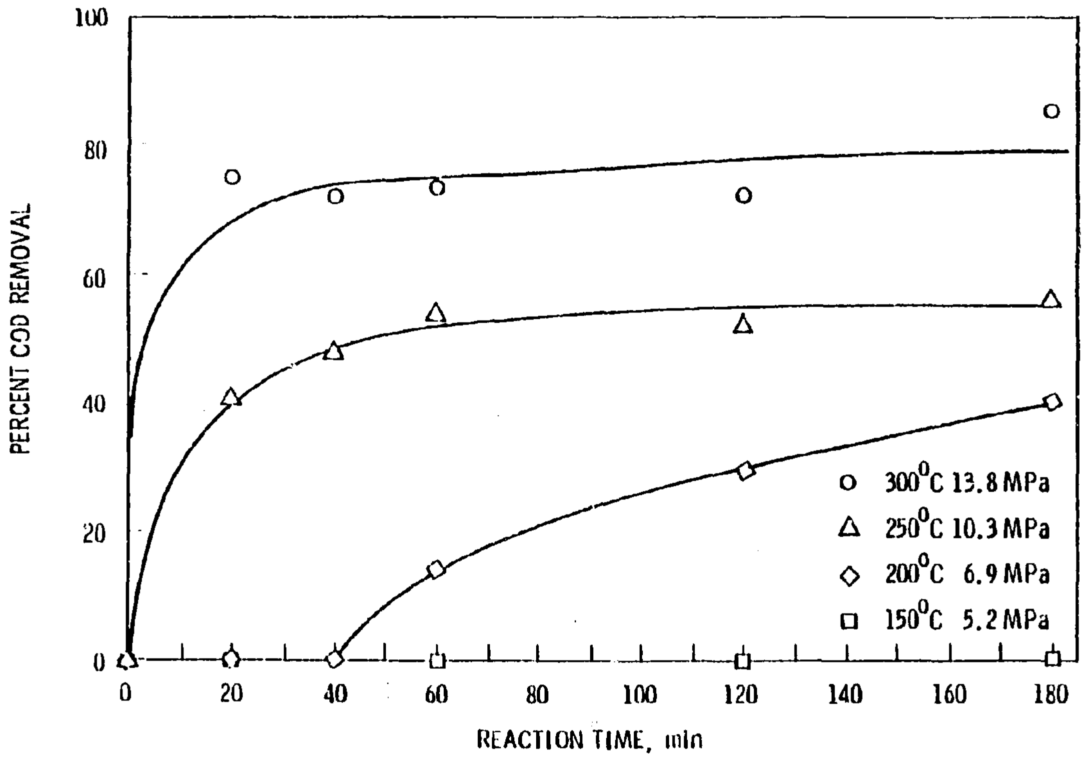

FIGURE 2. COD Removal vs Reaction Time

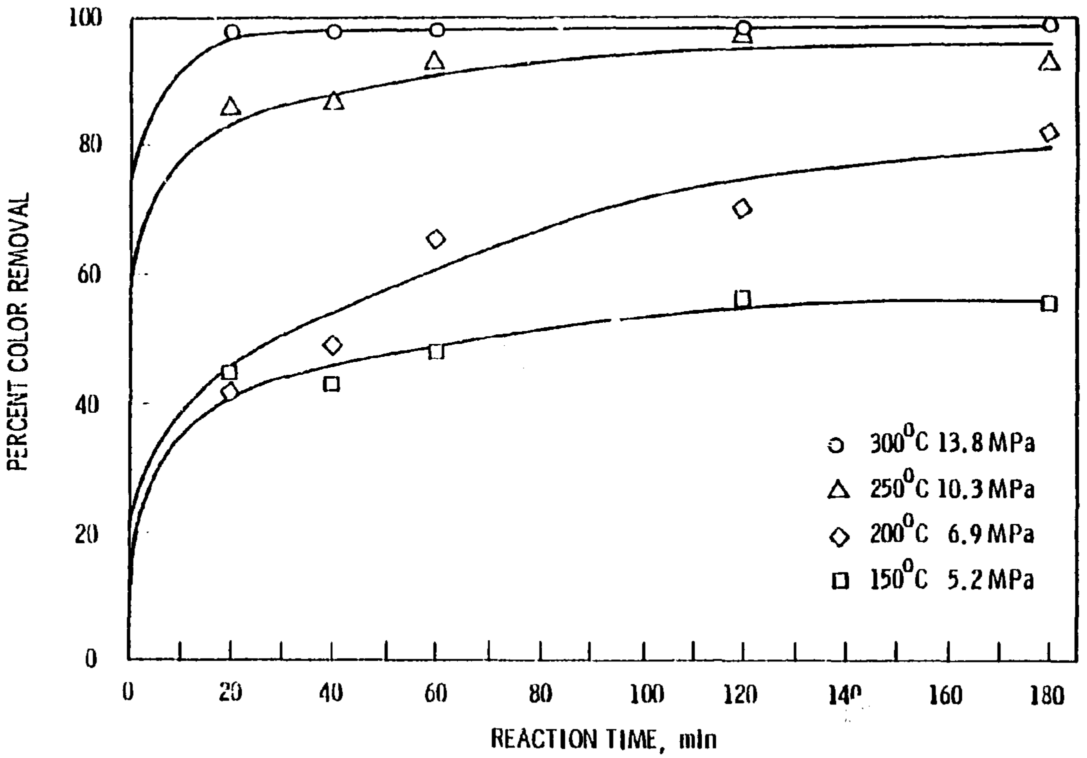

FIGURE 3. Color Removal vs Reaction Time

observed at all reaction times, supporting the lower temperature observation that rapid degradation and partial oxidation of large organic molecules occurred before total oxidation.

SOLVENT EXTRACTION(17)

Solvent (liquid-liquid) extraction is a process in which contaminants can be separated from the aqueous phase by the addition of an immiscible solvent which preferentially dissolves the contaminants. After the organic and aqueous phases have been mixed, they are separated by gravity or mechanical action. A typical solvent extraction process is shown in Figure 4.

Solvent extraction was proposed as a method for removing organics from BGW for several reasons: 1) the process is insensitive to fluctuations in contaminant concentrations; 2) the process is not affected by toxic compounds, unlike biological treatment systems; 3) a wide variety of solvents are available which implies the solvents used can be selected to extract particular contaminants in the wastewater; and 4) contaminants can often be recovered and sold, offsetting the cost of treatment. Solvent extraction costs tend to be proportional to the volume of water treated (regardless of contaminant concentration), unlike biological and carbon adsorption processes whose costs are proportional to the amount of contaminants present. Thus, for wastewaters with high contaminant levels, solvent extraction can be more cost effective than other treatment techniques.

Solvent Selection

Selection of a proper solvent depends on what constituents are present in the wastewater being treated. Kao(5) reported that the major constituents of BGW were acetic and propionic acids. Based on this information, a review of the literature was conducted to determine those solvents which were effective in extracting acetic and propionic acids from aqueous solutions. Based on the literature review(18-23) and previous experience, it was determined that methyl isobutyl ketone (MIBK), n-butyl acetate, n-butanol, MIBK/n-butyl acetate (1:1 vol), MIBK/n-butanol (1:1 vol), tri-n-butyl phosphate, tri-n-octyl phosphine oxide (TOPO)/kerosene (1:9 wt), TOPO/MIBK (1:9 wt), kerosene, and toluene were the best solvents available for extraction of BGW.

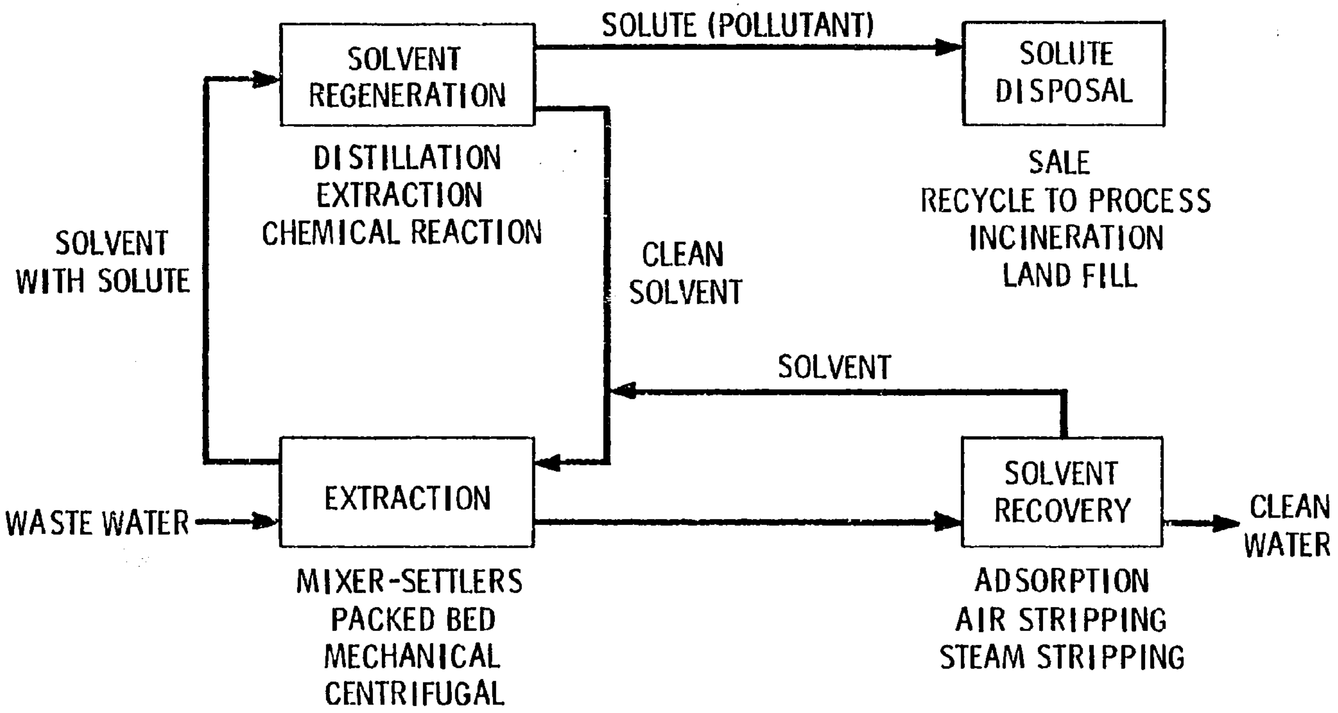

FIGURE 4. Typical Solvent Extraction Process

Experimental Equipment and Operating Conditions

Each extraction was performed by combining 50 ml of solvent or solvent mixture with 25 ml of BGW in a separatory funnel which was shaken for 5 minutes. The two phases were allowed to gravity settle for at least 2 hours. Samples of the organic and aqueous phases were taken and then stored in glass bottles at 4°C until COD analysis could be performed.

Experimental Results and Discussion

Distribution coefficients (K_D≡ ratio of organic phase concentration to aqueous phase concentration at equilibrium) were calculated assuming a complete material balance closure. The equilibrium aqueous phase COD value (the denominator) was determined by subtracting the solvent contribution to COD from the total COD after extraction. The equilibrium organic phase COD value (the numerator) was calculated by subtracting the equilibrium aqueous phase COD (after extraction) from the COD value of the aqueous feed. Results from these calculations are presented in Table 3.

The only solvent with a distribution coefficient greater than unity was n-butanol with a K_D of 1.28. A K_D greater than unity is normally desired in order to keep solvent to feed ratios low and reduce solvent regeneration costs. The MIBK/n-butanol mixture was next best with a K_D of 0.46. After the MIBK/n-butanol mixture, the phosphoryl solvents were best; TOPO/MIBK had a K_D of 0.27, and TBP had a K_D of 0.16. All other solvents gave distribution coefficients less than 0.1. The low K_D values indicate that large quantities of organics other than acetic and propionic acids were present in BGW6. Once a more detailed chemical analysis of BGW6 is completed, it may be possible to identify more effective solvents.

REVERSE OSMOSIS(24)

RO is a membrane separation process typically used for the removal of solutes (at low concentrations) from aqueous solutions. In RO, the osmotic pressure exerted by a solution is overcome by applying an external pressure which, in the presence of a semipermeable membrane, causes

TABLE 3. Extraction of COD by Various Solvents

Solvent	Distribution Coefficient $\left(\frac{mg/l}{mg/l}\right)$
Methyl Isobutyl Ketone (MIBK)	0.0097
n-Butyl Acetate	0.095
n-Butanol	1.28
MIBK/n-Butyl Acetate (1:1 Vol)	0.035
MIBK/n-Butanol (1:1 Vol)	0.46
Tri-n-Butyl Phosphate	0.16
Tri-n-Octyl Phosphine Oxide (TOPO)/kerosene (1:9 wt)	0.092
TOPO/MIBK (1:9 wt)	0.27
Kerosene	0.088
Toluene	0.057

separation of the solvent from the solute. The membrane itself may also enhance separation through chemical and/or steric effects, but these effects are not well understood.

Experimental Equipment and Operating Conditions

One important parameter in the design of an effective RO process is membrane selection. The membrane used in these experiments was the TFC-85, formerly the NS-100, which has been reported to be very effective for the separation of organics from solution(25,26). A schematic of the experimental setup in shown in Figure 5. Feed was continuously recycled through the apparatus during an experiment. Operating pressures ranged from 4.85 to 5.55 MPa (700-800 psi) for all experiments. A heat exchanger was placed in the feed accumulator to remove heat which was generated by viscous dissipation during operation of the RO unit; the heat exchanger maintained a feed temperature of 20 to 25°C. Permeate was collected by an automatic sampler, sealed in glass bottles, and stored at 4°C until sample analysis were performed.

Flow through the pressure vessel is shown in Figure 6. Feed flowed across the face of the membrane, passing small amounts of water (solvent) through the membrane. This solvent stream was labeled permeate. Remaining fluid passed through the pressure vessel as reject. The porous backing plate supports the membrane itself while allowing permeate to move away from the surface of the membrane.

Unlike WAO and solvent extraction experiments, which used only BGW6 feed, RO experiments used several feeds (Table 4). The first five feeds were effluents from biological experiments conducted by Maxham(1-4). Biologically pretreated feeds were used because RO was intended to be the second stage in a two-stage (biological-RO) treatment schematic. Two additional feeds were used, unpretreated BGW6 and WAO effluent (WAO). Throughout the following results and discussion section reference to these feeds is made by use of the abbreviations listed in Table 4.

Experimental Results and Disussion

The performance of the RO treatment unit was evaluated by determining COD removal. The percentage of COD removal, by feed, is presented in Table 5. With the exception of the 50:1 feed, those feeds undergoing

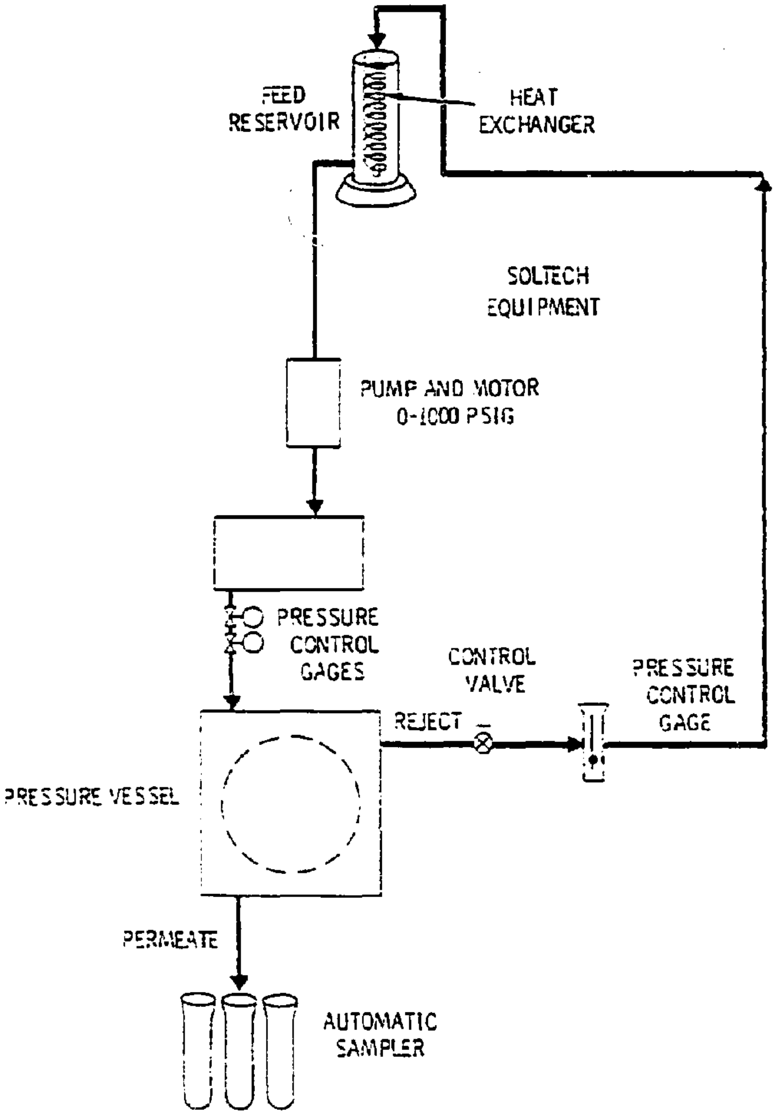

FIGURE 5. Reverse Osmosis Flow Schematic

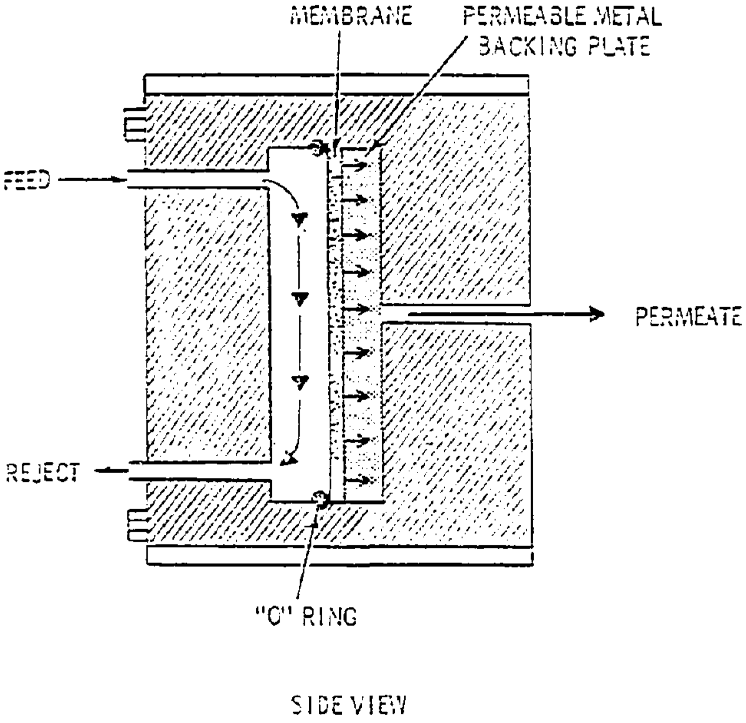

FIGURE 6. Reverse Osmosis Pressure Vessel Flow Pattern

TABLE 4. Feeds Used During RO Experiments

Feed Set No.	Abbreviation for Feed	Description of Feed	Initial COD (mg/l)
I	50:1	Third set of BGW (Maxham 1981) pretreated in anaerobic biological units 1, 2, or 3 at 50:1 dilution including the addition of 2 g/l milk solids.	98
II	25:1	Fourth set of BGW pretreated in anaerobic biological units 1, 2, or 3 at 25:1 dilution.	373
III	10:1	Fourth set of BGW pretreated in anaerobic biological units 1 or 2 at 10:1 dilution.	1,749
IV	AN-4	Fourth and fifth sets of BGW pretreated with high rate fixed film (HRFF) and anaerobic (fourth reactor) biological processes.	8,236
V	AER 1-3	Second, third, fourth, and fifth sets of BGW pretreated in aerobic biological process units 1, 2, or 3.	6,476
VI	BGW6	Sixth set of BGW without pretreatment.	18,020
VII	WAO	A set of feeds which underwent pretreatment by WAO.	3,936

TABLE 5. Average COD Removal for Seven BGW Feeds

Feed	Percent COD Removal ($\overline{X}$,S)	
50:1	69	8.1
25:1	94	1.8
10:1	98	0.4
BGW6	89	1.3
AN 4	∿100	0.1
AER 1-3	∿100	0.2
WAO	91	0.2

biological pretreatment had much better COD removal percentages (98% versus 90%) than those without biological pretreatment. This is probably because RO does not remove acetic acid very well but this compound is readily degraded by biological organisms. Experiments with BGW6 and WAO averaged 90% COD removal; however, these feeds eventually fouled the membrane surface, a problem not observed while using other feeds.

Normally, experiments were completed in 8-12 hours, a period too short for determining membrane degradation with time. To evaluate this phenomena, three experiments were run approximately 26 hours each. Feeds for these experiments were AN-4 and AER1-3. Results of permeate COD versus time are plotted in Figure 7. The data for each of the three experiments have been fitted to a straight line. In general, data show increasing COD values with time, an apparently negative result. However, one should recall that solute concentration was constantly increased in the feed as permeate was removed (due to recycle of feed). When a curve of percent COD removal versus time (Figure 8) was plotted from the data in Figure 6, one can observe that COD removal remained essentially constant with time, indicating negligible membrane deterioration over the 26 hour tests.

Color removal, normally considered in tertiary treatment processes, was measured for all experiments except those using 50:1 feed. Results of the color removal analysis are presented in Table 6. Nearly 100% color removal was observed for all experiments. These color removal percentages agree quite closely with the 100% removal values reported in the literature (30). Visibly, all permeate samples were colorless in contrast to the brown, opaque feed samples.

CONCLUSIONS

- Treatment of BGW by WAO can be very effective for removing both COD and color.
- Removal of COD and color by WAO increases as temperature and pressure are increased.

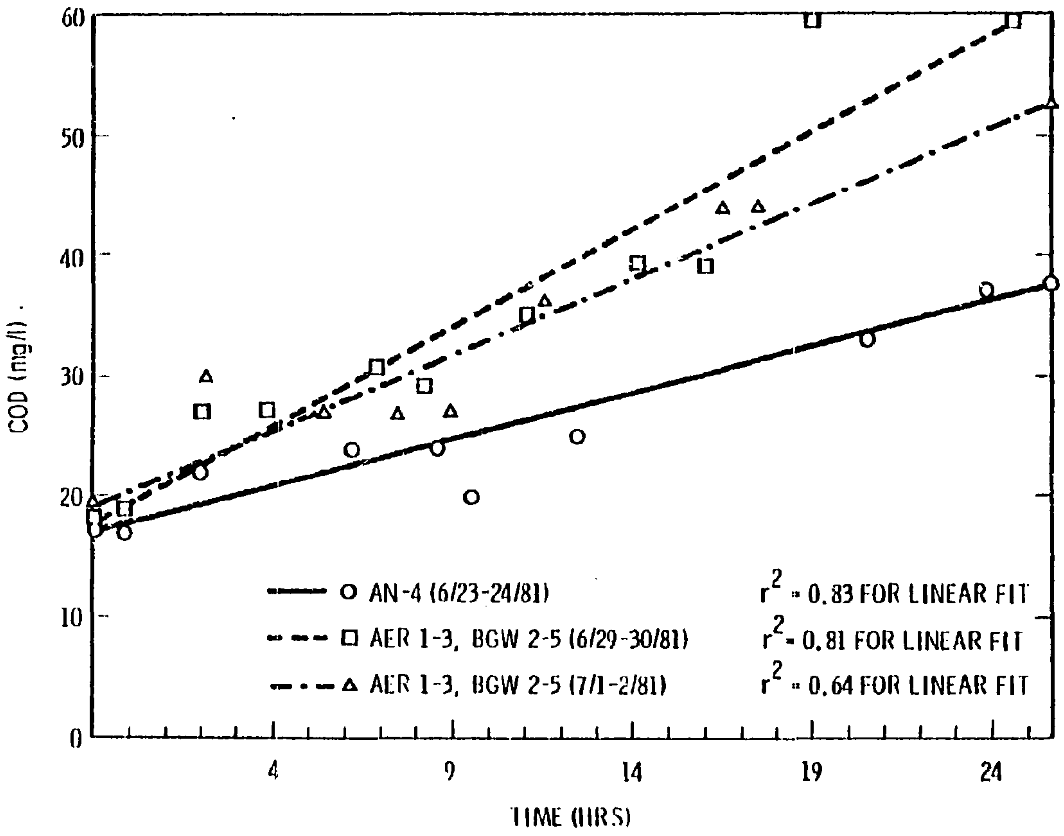

FIGURE 7. Permeate COD vs Time

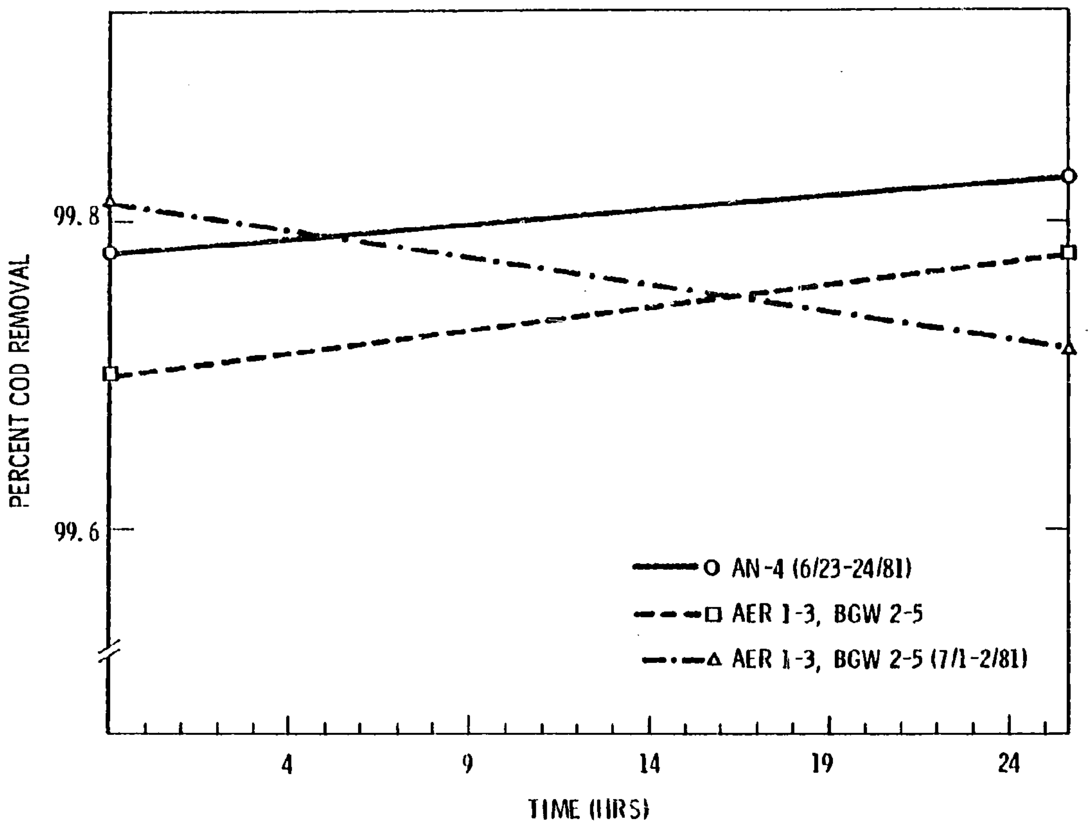

FIGURE 8. COD Removal vs Time

TABLE 6. Average Color Removal for Seven BGW Feeds

Feed	Percent COD Removal ($\overline{X}$,S)	
25:1	99	1.0
10:1	~100	0.2
BGW6	99	0.1
AN 4	~100	0.0
AER 1-3	~100	0.0
WAO	99	1.1

- Solvent extraction does not appear to be a viable technique for removing organics from BGW.
- If required, RO can be an effective treatment technology for final treatment of BGW prior to discharge.

RECOMMENDATIONS

- The treatment of BGW by WAO followed by biological processes should be investigated.
- Compounds degraded by WAO, based on operating conditions, should be identified.

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