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EXPERIMENTAL ANALYSIS OF BIOLOGICAL OXIDATION CHARACTERISTICS OF HYGAS COAL GASIFICATION WASTEWATER

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PROLOGUE

In July of 1976, the Department of Energy initiated a comprehensive program for environmental assessment of its high-BTU coal gasification pilot plant installations. The overall objective of the program is to develop the methodology and data base necessary for meaningful assessment of the environmental impact of the coal gasification processes. The environmental characterization efforts at each pilot plant are focused on scalable process units, with the goal of establishing rules and strategy for scaleup to commercial-size installations.

Carnegie-Mellon University, in its role as assistance, coordination and evaluation contractor for the DOE environmental assessment program, has prepared a series of technical documents in support of program objectives and activities within and across the coal gasification facilities. This report represents one in that series. Reports are also available describing the unique C.M.U. role and summarizing program activities.

ABSTRACT

An eight month experimental study was performed to assess biological treatability characteristics of Hygas coal gasification process pilot plant wastewater comprised of cyclone and quench condensates. The study evaluated treatability characteristics of ammonia stripped and unstripped wastewater at full strength and at 1:1 dilution. It was determined that minimum pretreatment required for biological oxidation consisted of reducing wastewater alkalinity, and decreasing raw ammonia concentration by dilution or by stripping. Kinetic studies with stripped Hygas wastewater showed the waste could be processed at mean cell residence times varying from 10 to 40 days with hydraulic residence times of 2 to 3 days; the bacteriological yield coefficient was 0.11 (COD basis), and the decay coefficient was 0.02/day. The study also determined correlations for dissolved oxygen uptake, the effect of COD removal rate on zone settling velocity and sludge volume index, removal efficiencies for various constituents, and adsorption isotherms for biological effluent COD on activated carbon and process char. Nitrification was completely inhibited in this study; it was suspected that this was due to high wastewater boron concentration (80-110 mg/l), however additional testing at lower loadings is required to confirm this observation. Hygas wastewater seemed to inhibit bacteriological growth because mean cell residence times less than 10 days do not appear feasible and because apparently higher yield coefficients were observed with diluted wastewater.

Introduction

This report summarizes essential elements and significant findings of an eight month, bench scale biological oxidation treatability study performed on Hygas coal gasification process effluent. This paper is abstracted from a detailed report on the treatability study; the detailed report will be submitted in July, 1978.

Biological Treatment of Coal Conversion Effluents

A thorough review was performed on all available published literature pertaining to field or experimental investigations on biological treatment of coal conversion effluents. A discussion of biological treatment of coke plant wastewater was included because it was shown that there are some similarities between coke plant ammonia liquor and Hygas wastewater, and because a much larger body of literature is available pertaining to biological treatment of ammonia liquors than treatment of coal conversion effluents. Reports describing biological treatment of Lurgi process effluent, Synthane condensate, and coal liquefaction effluents were reviewed also.

A general conclusion from the literature review was that there are significant deficiencies in state of the art knowledge pertaining to design and operation of biological oxidation facilities for processing coke plant ammonia liquor and for treatment of conversion effluents. It was found that the available data base for design of coke plant biological oxidation facilities was lacking, and that answers to important design and operation questions remain largely unknown. Given available information on processing of ammonia liquors, it would be a challenging task to design a coke plant wastewater treatment facility, let along to attempt to extrapolate that information to design of a coal conversion process wastewater treatment facility.

Contradictory information exists with regard to such basic parameters as dilution requirements, acceptable ammonia levels, suitable pH conditions and organic loadings.

Optimal operating criteria for removal of specific compounds such as SCN^- remain unknown. Very little data is available which clearly define permissible levels of organic loading; there is insufficient data for evaluating the relationship between organic (ie. BOD or COD) removal, sludge residence time, hydraulic residence time, and sludge concentration. Most researchers have not obtained biological performance parameters under test conditions in which sludge residence time was carefully controlled and in which biological reactors had been given sufficient time to pass through an appropriate balance period.

Objectives of the Treatability Study

The purpose of the biological treatability study was to define activated sludge performance characteristics with Hygas wastewater and to address some of the basic questions indentified with regard to biological oxidation of coal coking and coal conversion effluents. Specific objectives of the study were:

1. To identify minimum dilution requirements for Hygas wastewater.
2. To determine the effect of ammonia stripping on biological treatment.
3. To evaluate the effects of coal gasification wastewater acid-base chemistry on the need for pretreatment.
4. To determine Hygas wastewater biological oxidation kinetic growth constants.
5. To determine removal efficiency of primary wastewater constituents through pretreatment and biological oxidation.
6. To evaluate physical properties of Hygas wastewater activated sludge.
7. To determine the adsorption capacity of Hygas process solid waste (char) and activated carbon for biological effluent residual COD, and
8. To identify and to assess removal of wastewater trace organics by pretreatment and biological oxidation.

Objectives one through seven are described in the detailed report, and will be reviewed here briefly. Results of identification and removal of trace organic compounds

will be presented in later reports.

Experimental Design and Protocol

Scalable process effluent from the Hygas pilot plant was envisioned to be comprised of product gas cyclone slurry water and product gas quench condensate. These two streams were blended in proportion to their respective flow rates to form the waterwater used in the treatability study. This strategy ensured that all constituents which may appear in full-scale process condensates were present in the wastewater used in this study; however, because of makeup water employed in the cyclone slurry, the concentration of the wastewater used in this study is likely to be somewhat less than observed in full-scale process condesnates. Experiments are now in progress to evaluate biological treatment of the raw gas quench water without blending. Since this stream has higher concentrations of constituents than the blend used in this study, it should serve to identify any additional processing difficulties not evaluated in this study.

Special provisions were made to handle certain problems resulting from the variable and intermittent nature of Hygas pilot plant operation. To accomodate the intermittent Hygas operating schedule, large quantities of wastewater from two pilot plant runs were collected and shipped to C.M.U. Solids were removed from the cyclone water; cyclone and quench waters were homogenized separately, and then preserved by freezing. Wastewater was withdrawn from freezer storage, composited and prepared as necessary during the course of the investigation. This procedure ensured an adequate supply of wastewater of relatively uniform composition. There was no concern about loss of sample homogeneity because the wastewater was frozen and thawed in 5-gallon containers and because the wastewater prior to freezing contained essentially no solid or emulsified constituents which may have undergone phase separation during freezing.

Wastewater was obtained from Hygas pilot plant runs No. 62 (June 9, 1977) and

No. 64 (August 24, 1977); both samples represent gasification of Illinois No. 6 bituminous coal at approximately 70% carbon conversion. Typical analysis of the quench and cyclone condensates from these two runs are shown on Table I, typical analysis of the raw Hygas wastewater blend used in the treatability study is shown in Table II.

A special investigation was performed to assess potential effects of freezing on wastewater quality. Results of this study are presented in Table III which shows no significant changes through as many as four freeze-thaw cycles. There was approximately a three day period from the time of sample collection at the Hygas plant until the time samples were stored by freezing. Comparison of the data presented in Table II with results of analysis of properly preserved samples (data not shown) suggests perhaps only CN^- suffered losses during the three day interval between sample collection and freezer storage. A more appropriate value for cyanide concentration may be several mg/l; however, this should not have any significant effect on biological treatability characteristics.

The basic experimental design for the treatability study is presented in Figure 1. This experimental design permitted evaluation of the study objectives as outlined above. Rationale for selection of the specific operating conditions shown in Figure 1 are explained in the detailed report. Selection of appropriate pH conditions reflected concern for minimizing potential HNO_2 and NH_3 toxicity to nitrifying bacteria while encompassing the pH range recommended as being suitable for SCN^- oxidizing organisms. Sludge age values were selected on the basis of literature review recommendations and on the basis of a lowest value of cell residence time found to be permissible in Hygas wastewater, ie. ten days. Hydraulic loadings were selected on the basis of conservative estimates of biological kinetic growth constants.

The biological oxidation units consisted of continuous flow stirred tank reactors. Care was taken to ensure that the bacteriological cultures were properly acclimatized

TABLE 1

TYPICAL ANALYSIS OF HYGAS WASTEWATER

Specification	Parameter mg/l										
	COD	Phenol	NH_3-N	CN^-	SCN^-	$S^=$	$SO_4^=$	$PO_4^=$-P	Alkalinity as $CaCO_3$	Conductivity µmhos/cm	pH (units)
Quench Condensate											
Run 62	6740	1100	8650	0.23	41	305	41	-	22,500	-	8.4
Run 64	8600	1800	8750	0.34	107	295	120	0.5	18,300	36,000	8.3
Cyclone Condensate											
Run 62	1800	520	340	0.01	3.6	11	93	-	440	-	7.5
Run 64	2000	380	280	0.01	1.7	8.1	210	0.8	1,000	2,200	8.1

Note: Wastewater used in treatability study consisted of a 1:1 mixture of Hygas quench and cyclone condensates.

TABLE II

ANALYSIS OF VARIOUS SAMPLES OF RAW HYGAS WASTEWATER

Date	Parameter, mg/l								
	COD	Phenol	NH_3-N	CN^-	SCN^-	$S^=$	$SO_4^=$	Alkalinity as $CaCO_3$	pH (units)
6-16-77	3500	775	3500	0.42	-	155	-	-	7.9
6-24-77	4200	775	3500	0.42	-	155	-	-	7.9
6-29-77	4000	560	3500	0.22	22	95	-	14,000	7.8
7-07-77	3300	590	2600	0.16	45	221	-	12,500	8.0
7-13-77	5000	660	3900	0.10	-	159	-	9.180	7.8
7-20-77	3500	600	4000	0.16	30	168	-	12,700	7.8
8-04-77	3800	690	3300	0.35	27	71	-	12,920	7.9
8-16-77	3400	680	3300	0.25	20	104	-	11,000	7.9
9-08-77	3680	840	3740	-	33	-	-	14,800	7.8
9-21-77	4700	840	3780	0.70	34	-	-	-	7.8
9-28-77	5140	620	4300	0.10	21	60	-	9.800	7.7
10-13-77	3020	570	3070	0.10	17	-	-	-	7.8
11-10-77	5300	800	4620	-	20	155	-	13,600	7.8
1-06-78	4200	900	4550	0.85	37	172	184	15,500	8.0
Average	4050	710	3690	0.32	28	138	184	12,600	7.8

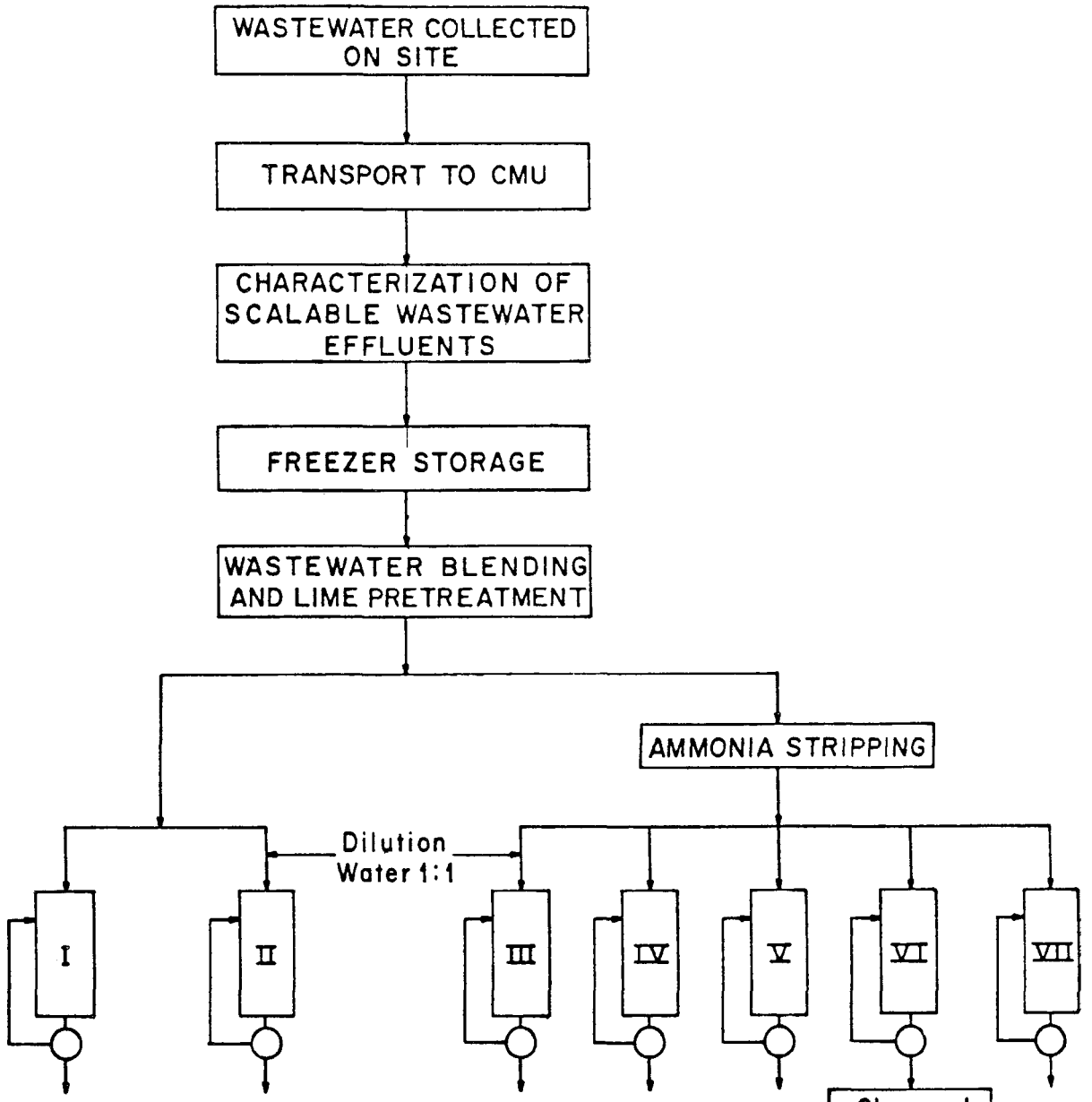
Note: Analysis made after blending quench and cyclone wastewaters prior to pretreatment.

TABLE III

WASTEWATER QUALITY CHANGES

AS A RESULT OF STORAGE BY FREEZING

Parameter	Raw Wastewater	Freeze-Thaw Cycles			
		First	Second	Third	Fourth
COD, mg/l	3150	3100	3030	3010	3010
Phenol, mg/l	620	600	600	610	-
NH_3-N, mg/l	3150	2980	2890	2930	2900
$S^=$, mg/l	250	230	230	220	220
CN^-, mg/l	0.61	0.54	0.49	0.41	0.28
SCN^-, mg/l	38	40	41	45	45
pH	7.8	7.8	7.85	7.8	-



Nominal Biox Hydraulic Residence Time, θ, Days	3	3	3	3	2	2	3
Mean Cell Residence Time, θ_c, Days	20	20	20	20	15	10	4

For all Reactors pH = 7.0–7.5, Temperature 22–25°C; Dissolved Oxygen > 3 mg/l

Figure 1 Experimental Design for Biological Oxidation Studies on Hygas Wastewater.

to Hygas wastewater. Once this was achieved, the reactors were managed on the basis of mean cell residence time. The attainment of steady state operation was defined as that interval (balance period) necessary to achieve three complete solid wasting cycles. Thus, steady state operation occurred from 30 to 120 days after the reactors were acclimatized. The reactors were then run for an additional eight to fourteen weeks to obtain sufficient steady state performance data.

Wastewater Pretreatment

An important finding of the study was that excessively high values of wastewater alkalinity caused biological reactor pH to fluctuate upward controllably. This was a result of CO_2 being stripped from solution during aeration. Hence alkalinity removal is one requirement for minimum recommended form of wastewater pretreatment prior to biological oxidation. Alkalinity removal was achieved in this study by adding lime to the wastewater and precipitating alkalinity as a calcium carbonate sludge. Wastewater treated in this fashion served as feed to reactors numbers I and II (Figure 1). The feed to the remaining reactors (Nos. III to VII) was pretreated by lime addition to remove alkalinity and then processed for ammonia removal by air stripping at pH levels between 9.8 and 10.1. This reduced wastewater ammonia concentration to 100 - 150 mg/l. Lime addition was carefully controlled by experimental design so as to leave approximately 1000 mg/l residual alkalinity in the wastewater feed as measured from a pH value of 7.3. It was found that this value of alkalinity provided sufficient buffering capacity to prevent downward pH changes as a result of the acid producing reactions accompanying biological oxidation.

Note that the majority of the ammonia present in Hygas wastewater is compensated by an equivalent fraction of dissolved acid gas which is largely CO_2. Thus the ammonia exists primarily as so-called free ammonia, and both CO_2 and NH_3 may be liberated by high temperature stripping operations.

Wastewater characteristics following lime pretreatment and ammonia stripping are shown on Table IV; average raw, lime treated, and ammonia stripped wastewater characteristics, and percentage change through ammonia stripping are shown on Table V. COD and phenol concentration decreased about 9% and 7% respectively, primarily as a result of chemical precipitation. Averages of 92% and 96% of alkalinity and ammonia respectively were removed as a result of pretreatment.

Biological Oxidation Studies

Figure 2 illustrates the type of biological reactor used in this investigation; the general layout of the experimental apparatus is shown in Figure 3. The reactors were made of glass with internal clarifiers. Electric stirrers were employed to prevent biological floc clumping. Foaming was a continual problem; it was remedied to a certain extent by the use of electric stirrers; however through most of the investigation antifoam agent was applied daily to the surface of the reactor to prevent biological solids accumulation in the froth. It was observed that foaming was more of a problem in the reactors receiving higher loadings. Cell wastage was accomplished daily by syphoning the appropriate volume of mixed liquor, filtering, and returning the filtrate to the aeration basin. Cell wastage calculations took into account the amount of volatile suspended solids carried out in the cell effluent.

Table VI presents the general sampling and analysis schedule observed during this investigation. Phenol and COD removal efficiencies were used during acclimatization and startup to help assess sludge viability. Microbial activity, sludge settling rate, O_2 uptake, and effluent SCN^- concentration were also used as indicators of process stability during startup.

Estimate of Nutrient Requirements

Table VII presents an analysis of sludge nutrient requirements to determine if Hygas wastewater would pose any nutrient deficiencies. The calculations are based

TABLE IV

WASTEWATER CHARACTERISTICS DURING PRETREATMENT

Date	Parameter, mg/l							Residual Alkalinity* as $CaCO_3$	pH units	Lime Dose g/l
	COD	Phenol	NH_3-N	CN^-	SCN^-	$S^=$	$SO_4^=$			
						LIME PRETREATMENT				
6-29-77	3700	590	3300	0.43	33	84	--	650	10.0	--
7-07-77	3800	660	2000	0.29	16	45	--	558	10.0	24
7-13-77	3800	580	3300	--	39	--	--	1680	9.8	19
7-20-77	3600	690	3000	0.07	17	156	--	560	9.9	19
8-04-77	3800	670	2600	0.19	25	119	--	770	10.0	20
8-16-77	3250	670	3000	0.25	45	150	--	800	9.9	17
9-08-77	3430	--	3370	--	42	234	--	1100	11.4	--
9-21-77	3230	530	3290	0.56	30	49	--	595	10.8	20
9-28-77	5100	620	3550	0.20	19	17	--	2850	9.9	20
10-13-77	3170	570	2830	--	18	145	--	2200	9.9	20
11-10-77	5100	800	4280	--	27	11	277	1160	9.8	20
1-06-78	4000	900	2940	0.90	--	46	205	1650	9.8	20
Average	3830	660	3200	0.36	28	96	241	1210	10.1	20
						AMMONIA STRIPPED				
6-16-77	3800	775	220	0.36	--	36	--	--	9.8	
6-24-77	2500	750	87	0.09	20	5	--	--	10.0	
7-07-77	--	690	110	0.11	37	29	--	390	10.0	
7-13-77	3700	610	200	0.08	36	8	--	1450	10.0	
7-20-77	3500	460	87	--	33	12	--	525	10.1	
8-04-77	3700	760	110	0.12	15	14	--	480	10.4	
8-16-77	3450	640	190	0.13	21	13	--	640	9.8	
9-08-77	4000	640	97	0.10	14	--	--	900	10.0	
9-21-77	3500	570	137	1.20	15	15	--	760	10.3	
9-28-77	5100	720	152	1.00	22	13	--	1200	9.8	
10-13-77	2900	570	127	--	--	20	--	2600	10.2	
11-10-77	4400	800	71	--	27	7	190	1100	10.1	
1-06-78	3850	580	144	1.3	--	5	282	1600	10.1	
Average	3700	660	133	0.45	24	14	236	1060	10.0	

Note: *Alkalinity measured after initial adjustment to pH=7.3.

TABLE V

AVERAGE WASTEWATER QUALITY CHARACTERISTICS FOLLOWING PRETREATMENT

Specification	Parameter, mg/l								
	COD	Phenol	NH_3-N	CN^-	SCN^-	$S^=$	$SO_4^=$	Alkalinity as $CaCO_3$	pH (units)
Raw Wastewater	4050	710	3690	0.32	28	138	184	12,600	7.8
Lime Pretreatment	3830	660	3200	0.36	28	96	241	1,210	10.1(1)
Ammonia Stripped	3700	660	133	0.45	24	15	236	1,060	10.0(1)
Net change (%) between raw and stripped wastewater	-8.6	-7.0	-96	-	-14	-89	+28	-92	-

Note: (1) Alkalinity measured after pH adjustment to 7.3.

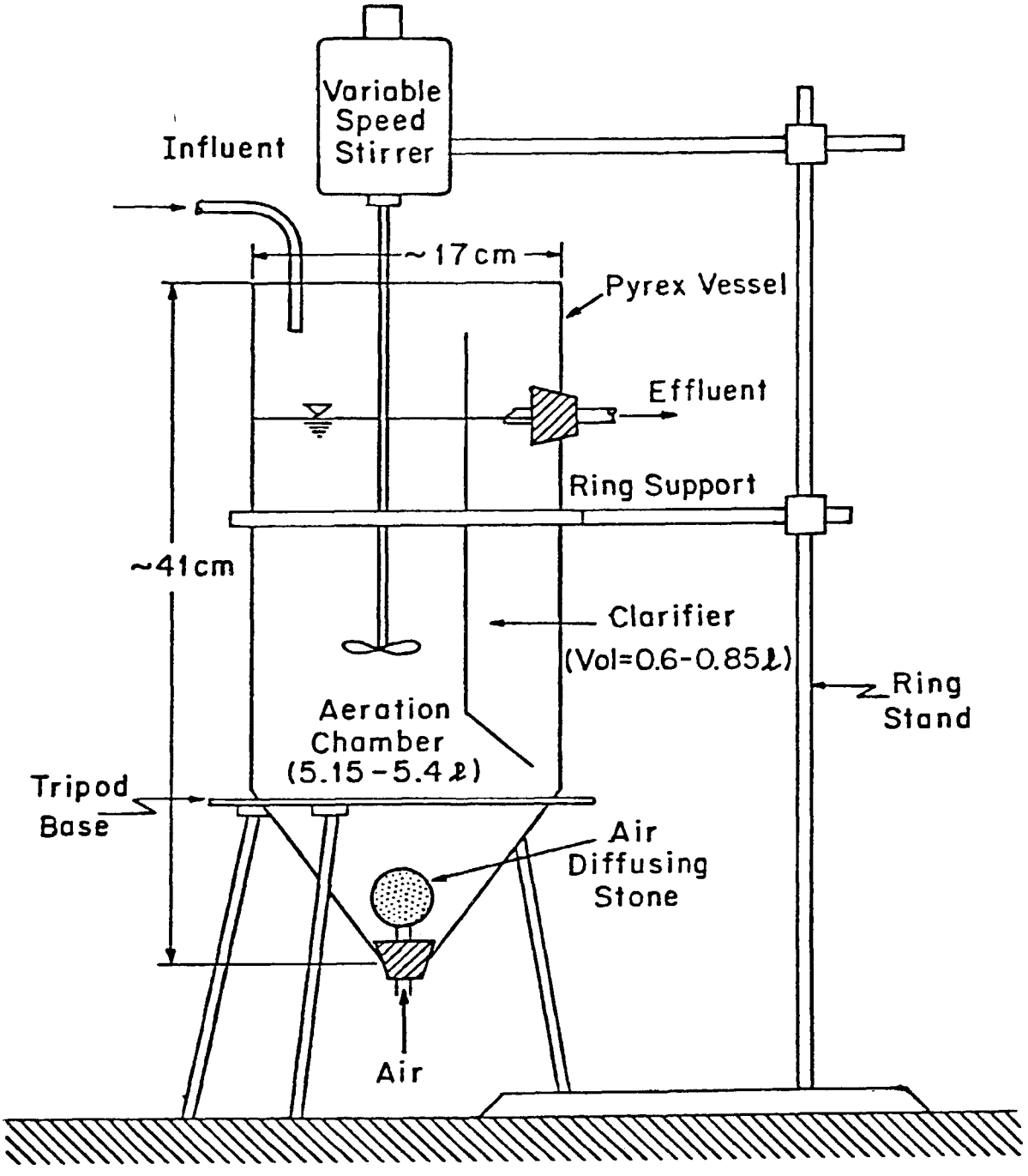


Figure 2 General view of continuous stirred tank reactor for biological oxidation kinetic studies.

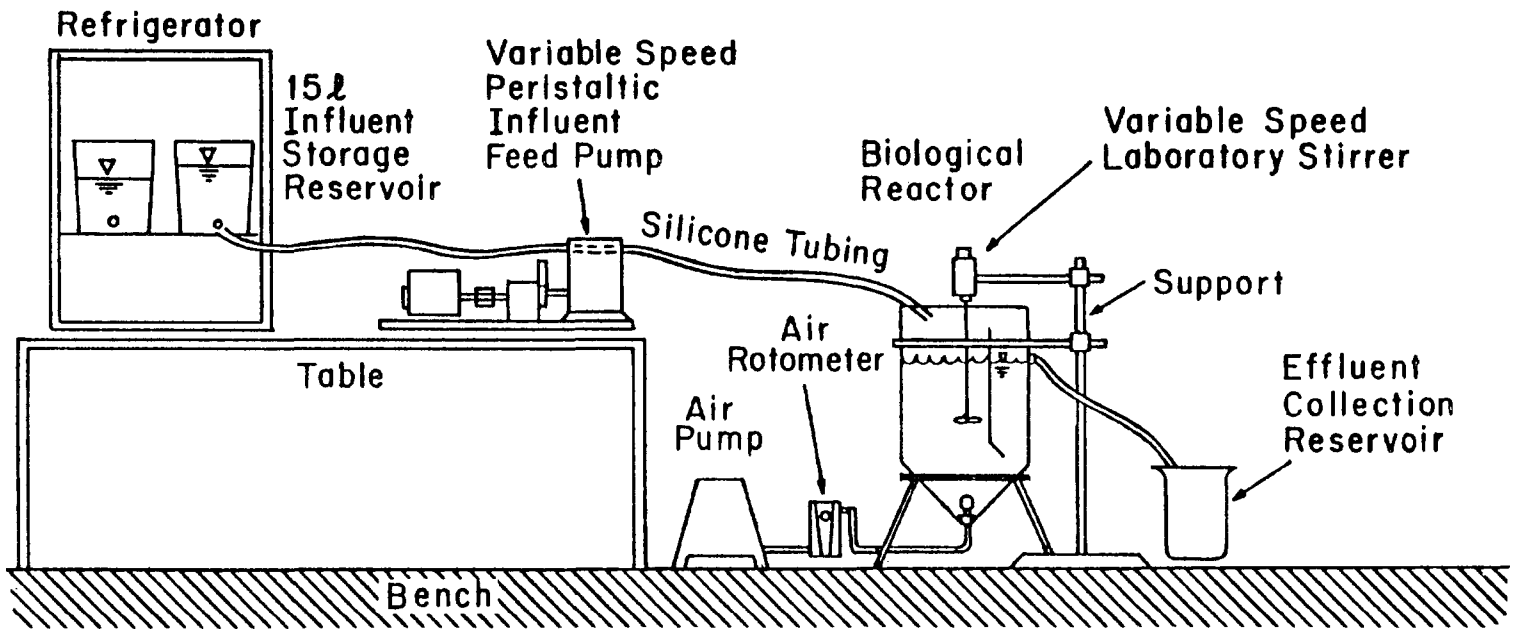


Figure 3 General layout of biological oxidation experimental apparatus.

TABLE VI

BIOLOGICAL OXIDATION STUDIES SAMPLING AND ANALYSIS SCHEDULE

Frequency	Sampling Location		
	Reactor	Influent	Effluent
Daily	Feed preparation pH and adjustment Volumetric throughput Sludge Wasting MLSS General housekeeping	pH and adjustment	TSS
Three times per week	MLVSS	COD	COD VSS
Approximately two times per week		SCN^- NH_3-N	SCN^- NH_3-N
Approximately once per week	Settling rate/SVI Dissolved oxygen O_2 uptake rate	CN^- NO_3-N Phenols Alkalinity Conductivity $S^=$	CN^- NO_3-N Phenols Alkalinity Conductivity $S^=$
Occasionally, or as required on composite samples	Microbial activity	$PO_4^{\equiv}$ B $SO_4^=$ BOD Kjeldhal-N/TKN Trace metals Trace organics	$PO_4^{\equiv}$ B $SO_4^=$ BOD Kjeldhal-N/TKN Trace metals Trace organics

TABLE VII

ESTIMATION OF NUTRIENT REQUIREMENTS FOR

BIOLOGICAL TREATMENT OF HYGAS WASTEWATER

Element	Cell Composition, Percentage of Dry Weight[1]	Estimate of Wastewater Concentration Requirements[2], mg/l	Measured Wastewater Composition After Lime Pretreatment
C	50	--	--
O	20	--	--
N	14	84	133
H	8	--	--
P	3	18	1.8
S	1	6	110
K	1	6	4.6, 4.7
Ca	0.5	3	12, 16
Mg	0.5	3	2.1, 2.5
Cl	0.5	3	25
Fe	0.2	1.2	0.66, 0.68
Co			0.4
Cu			<0.01
Mo			0.04
Mn			0.04
Na			40, 60
Zn			0.4
Other			

Notes: 1. Data source: Luria, 1960.

2. Based on assumed values of MLVSS=3000 mg/l, θ_c=10 days, reactor volume=5.5 l, and θ=2 days.

on approximate values of cell composition and assumed maximum values of cell wastage rate. Estimated wastewater nutrient composition is compared against measured lime pretreated wastewater elemental concentration and no serious deficiencies are observed except for phosphorous. Hence, phosphorous was added routinely to lime pretreated wastewater at concentrations greater than 18 mg/l P.

Survey Analysis for Toxic/Inhibitory Elements

Survey analysis was made for those elements which can inhibit microbiological activity. Table VIII shows analysis for elements known to inhibit nitrifying organisms, cause upset in biological treatment, and/or impair growth of microorganisms. The elemental concentrations shown in Table VIII were compared with review articles on the effect of heavy metals and elements on bacteriological organisms. It appeared that none of the elements presented a serious problem except for boron. Boron is not present at concentrations which would impair the activity of many classes of organisms, however it is suspected of being present at sufficient concentrations to inhibit nitrifying bacteria.

Biological Kinetic Model

The biological kinetic model used in this study is based on substrate and cell mass balances, and on growth rate of microorganisms and concentration of rate limiting substrate.

Sludge wastage rate is equal to net growth as long as the unit is not operated near cell washout conditions:

$$\frac{1}{\theta_c} = \mu - k_d \qquad (1)$$

where θ_c = mean cell residence time, day $^{-1}$

μ = specific growth rate, day $^{-1}$

k_d = decay coefficient, day $^{-1}$

TABLE VIII

SURVEY ANALYSIS OF HYGAS WASTEWATER FOR POTENTIAL

TOXIC/INHIBITORY ELEMENTS TO BIOLOGICAL OXIDATION

Element	Concentration mg/l
Ag	<0.01
As	0.005
B	80 - 110
Cd	0.01
Co	0.04
Cr	0.04
Cu	0.39
Fe	0.25
Hg	<0.002
Mn	0.04
Mo	<0.1
Ni	<0.03
Pb	0.10
Se	0.37
Zn	0.40

Note: Concentration measured after pretreatment by lime precipitation except for As and Se which were measured on raw Hygas wastewater.

Cell growth μ is equal to the substrate removal velocity, q, and a biological yield coefficient, Y.

$$\mu = Yq \tag{2}$$

where μ = mg cells produced per day per mg cells in reactor

Y = mg cells produced per mg substrate (COD) removed

q = mg substrate (COD) removed per day per mg cells in reactor

Thus

$$\frac{1}{\theta_c} = Yq - k_d \tag{3}$$

Substrate removal velocity is given by

$$q = \frac{F\ (S_o - S_1)}{V\ \ X_1} \tag{4}$$

where F = flow, l/day

V = reactor volume, l

X_1 = cell concentration, ie. MLVSS, mg/l

S_o = influent substrate concentration, COD, mg/l

S_1 = effluent substrate concentration, COD, mg/l

Cell growth may be described as

$$\mu = \frac{\hat{\mu}\ S_1}{K_s + S_1} \tag{5}$$

where $\hat{\mu}$ = maximum cell growth rate, day $^{-1}$

K_s = substrate concentration to give cell growth equal to one-half $\hat{\mu}$

Under normal loading conditions in activated sludge processes, $S_1 \ll K_s$, hence

$$\hat{\mu} \simeq K\ S_1 \tag{6}$$

where K = rate coefficient, 1/mg/day

Thus

$$\frac{F\ (S_o - S_1)}{V\ \ X_1} = \frac{\mu}{Y} \simeq \frac{KS_1}{Y} = kS_1 \tag{7}$$

where k = substrate removal rate coefficient, 1/mg/day.

Dependent variables S_1 and X_1 are measured for independent variables θ_c, F, V, and S_o. A plot of $1/\theta_c$ versus q is used to compute Y and k_d. A plot of q versus S_1 yields k. These constants can be used for functional design of prototype units.

This type of design model has been used successfully by various authors (Jenkins and Garrison, 1968; Pearson, 1966; Lawrence and McCarty, 1970; Adams, 1974; and Adams, Eckenfelder, and Hovious, 1975; Lawrence and Brown, 1976, etc.). The model is suitable for design of activated sludge units for removal of substrate material.

Biological Oxidation of Undiluted Stripped Wastewater

The main series of biological oxidation kinetic experiments are represented by Reactors IV, V, VI, and VII on Figure 1. In these experiments θ_c, θ (hydraulic residence time), and S_o were maintained as experimental constants, while S_1 and X_1 were system variables. There was no variability in θ_c, as this was imposed by the solids wasting schedule. Influent COD was held reasonably stable by the wastewater storage procedure; influent COD had a coefficient of variation of 11-16% during the time that the reactors were at steady state operation; θ was maintained as constant as was feasible by use of peristaltic feed pumps.

Figures 4 and 5 show representative performance data for reactors VI and VII respectively. These figures show values of influent and effluent COD, MLVSS, hydraulic residence time, and COD removal rate. Data similar to that shown in Figure 4 and 5 were obtained for all reactors in this study. Steady state performance data for reactors IV - VII were averaged and plotted according to Equation 3 on Figure 6. These data illustrate that influent COD, effluent COD, mean cell residence time, hydraulic residence time, and sludge concentration are correlated well by the kinetic model. The data show a predicted yield coefficient, Y, of 0.11 and a decay coefficient of 0.02/day. The value of decay coefficient is somewhat reasonable for the waste

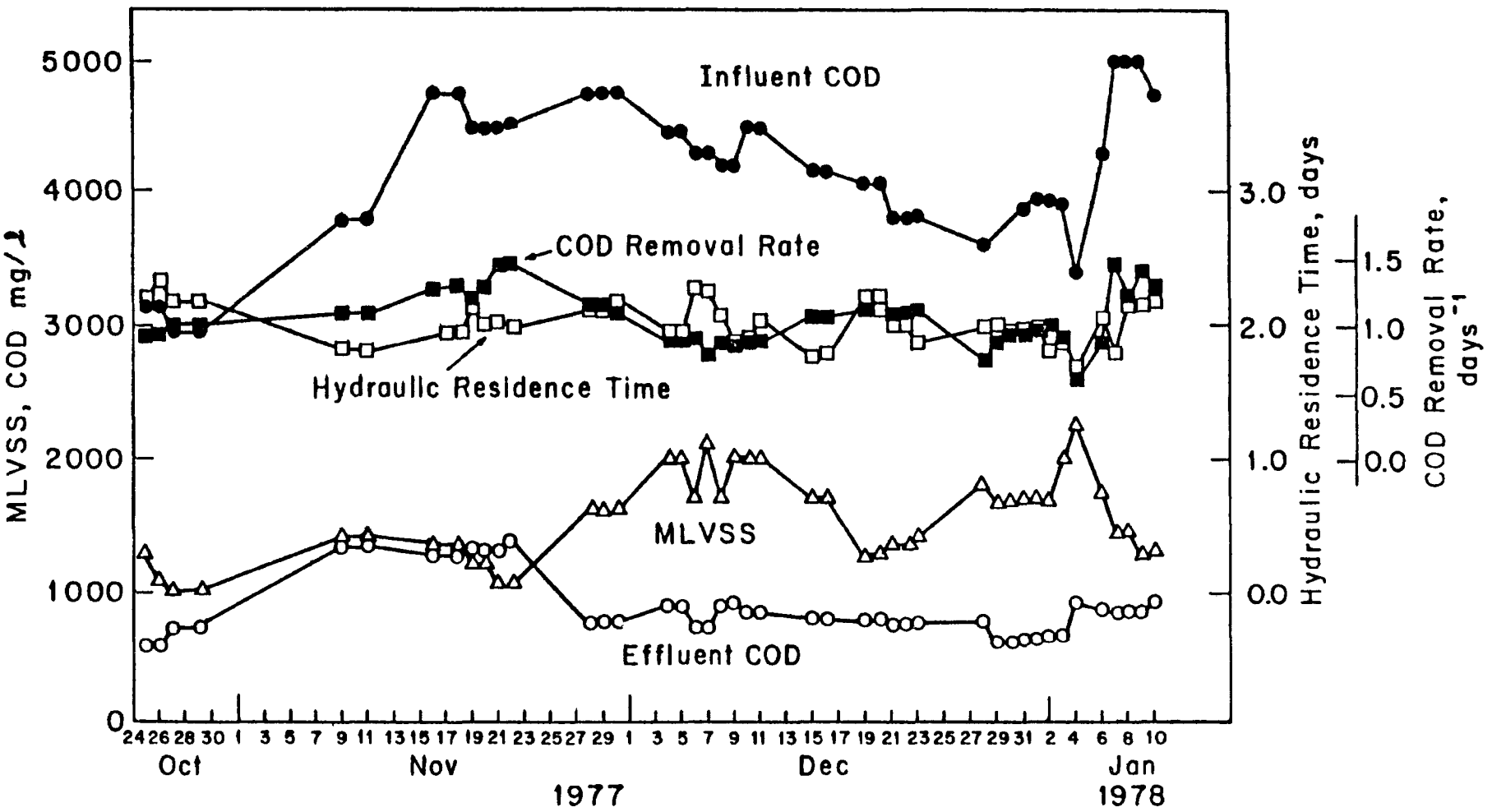


Figure 4: Representative Performance Data for Biological Reactor Number VI, θ_c=10 days

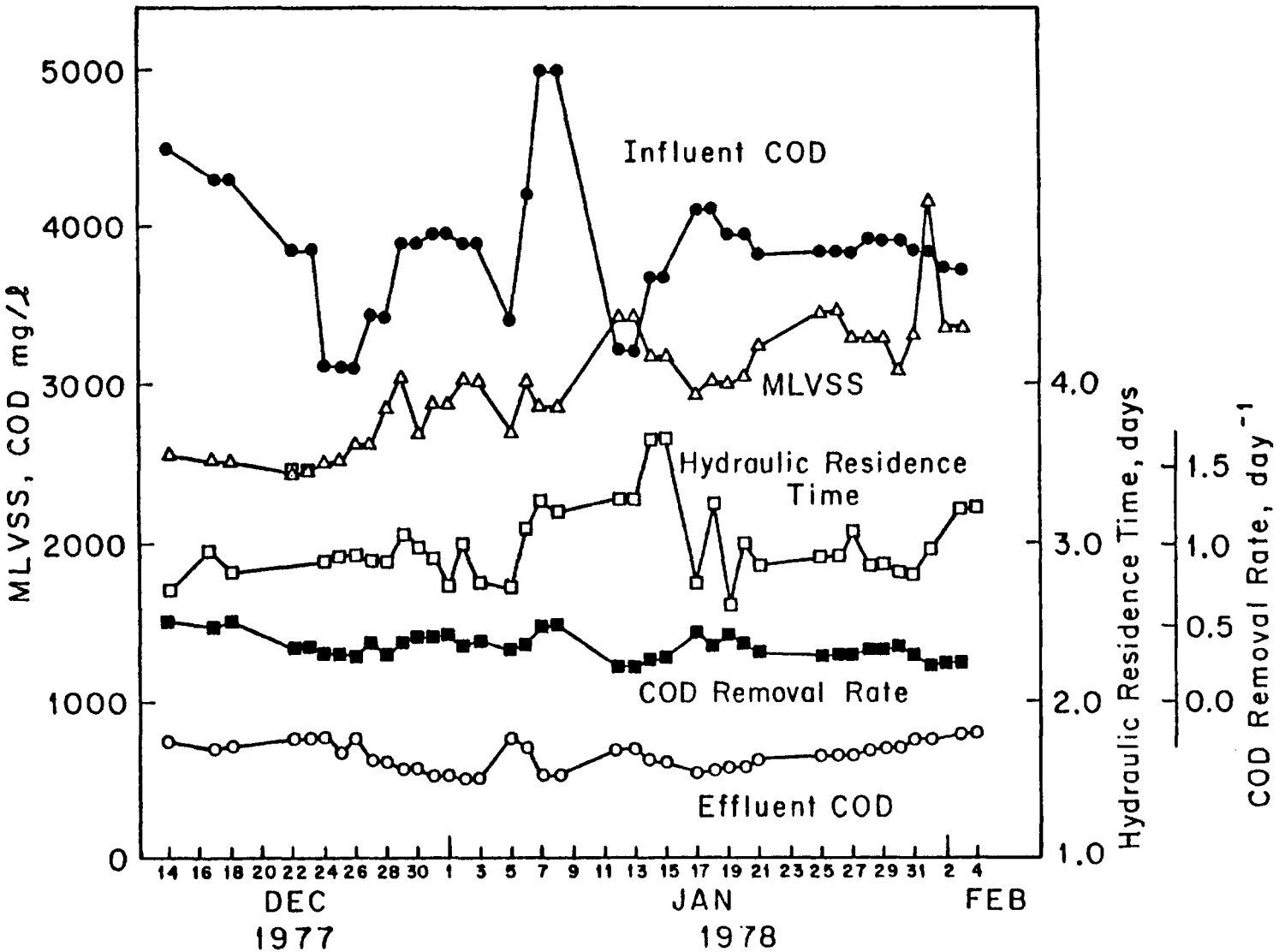


Figure 5: Representative Performance Data for Biological Reactor Number VII, θ_c=40 days

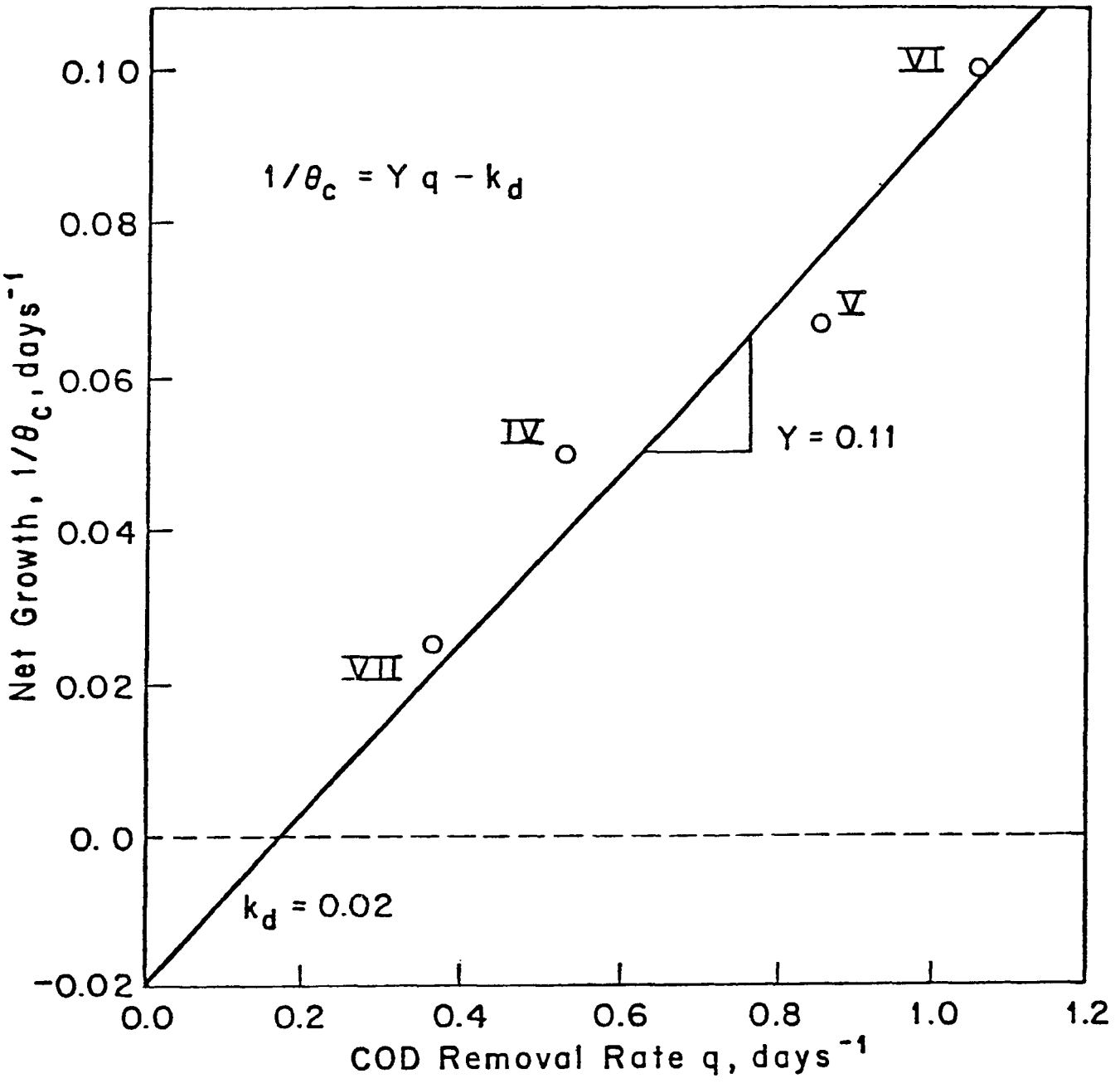


Figure 6: Correlation Between COD Removal Rate and Net Growth for the Main Series of Biological Oxidation Experiments to Give Yield Coefficient Y and Organism Maintenance Coefficient k_d.

based on limited literature data. However, the yield coefficient is less than that observed in a study on treatment of coal liquefaction wastewater, and it is also less than that which is predicted from theoretical considerations using phenol as a model substrate and calculating cell yield based on substrate thermodynamics and bacteriological energetic pathways. It is noted that the yield coefficient is similar to an estimated value computed from phenol loading data at one coke plant.

Figure 7 shows effluent COD values plotted against COD removal rate. The data show that lower values of effluent COD may be obtained with lower loadings, but a considerable amount of effluent COD appears to be relatively non-biodegradable. This finding is substantiated in part by measurement of COD and BOD for influent and effluents for reactors VI and VII. These data are presented in Figure 8 and regression analysis gives:

$$BOD = 0.80\ COD - 260 \qquad r^2 = 0.988$$

This shows that treated wastewater could contain approximately 325 mg/l COD and yet have a negligible value of BOD. For comparison purposes a COD and BOD correlation was made for influent and effluent from another biological reactor processing a synthetic Hygas wastewater mixture containing phenol, SCN^-, CN^-, NH_3, and nutrients. The synthetic feed showed:

$$BOD = 0.66\ COD - 34 \qquad r^2 = 0.978$$

Evidently the synthetic mixture possesses a greater potential for oxidation to very low COD values. Effluent BOD for the synthetic wastewater was often less than 30 mg/l while COD values were less than 80 mg/l, these values correspond to 98% and 97% removal of BOD and COD respectively. The synthetic mixture was much more efficiently processed than the more complex Hygas wastewater.

Table IV presents average influent and effluent concentration data for COD, phenol, ammonia, thiocyanate, alkalinity, cyanide, and sulfide. The reactors removed on the average 80% of the COD and better than 90% of the phenol. High phenol removal is not uncommon in treatment of phenolic wastes; an average effluent phenol concentration

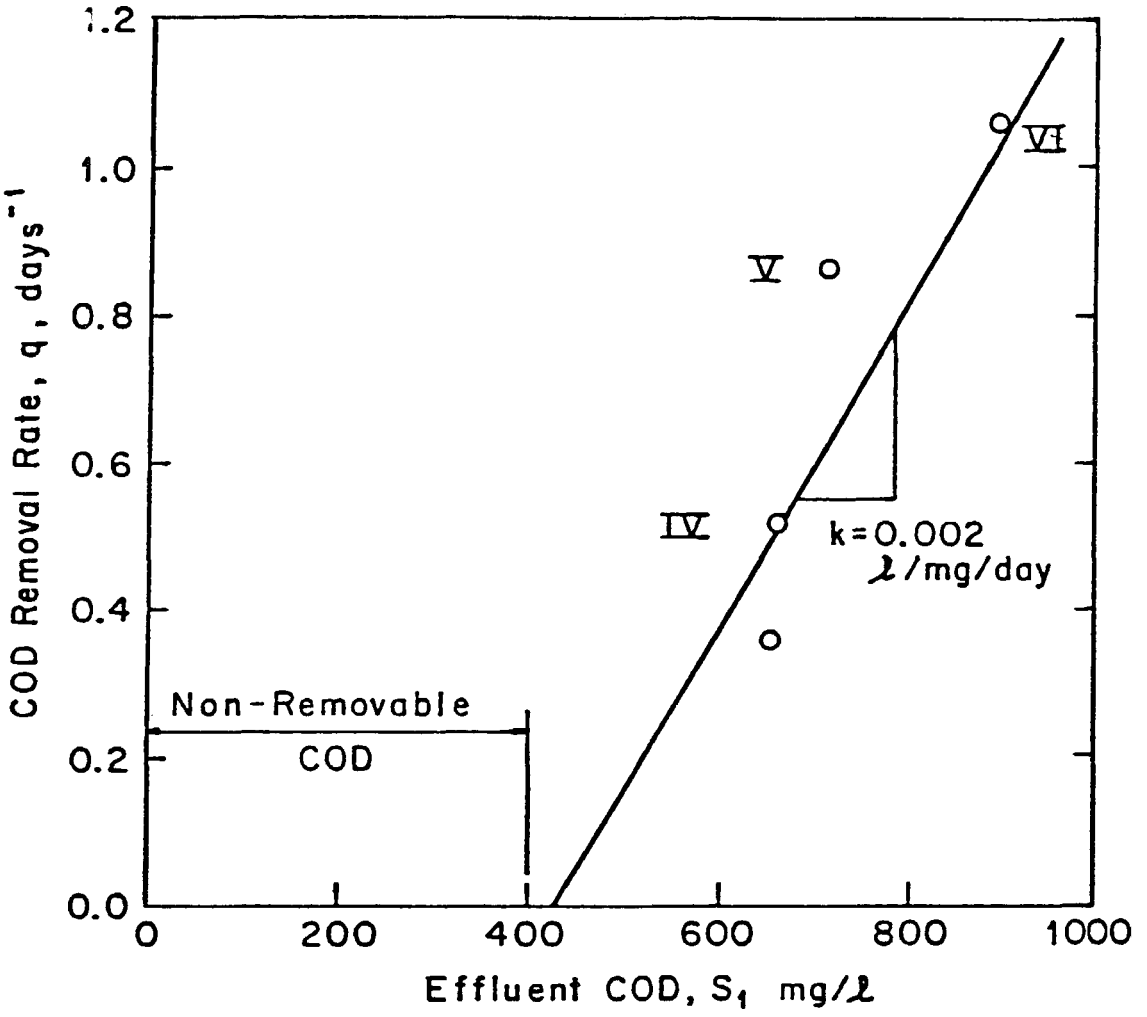


Figure 7: Correlation of Effluent COD with COD Removal Rate

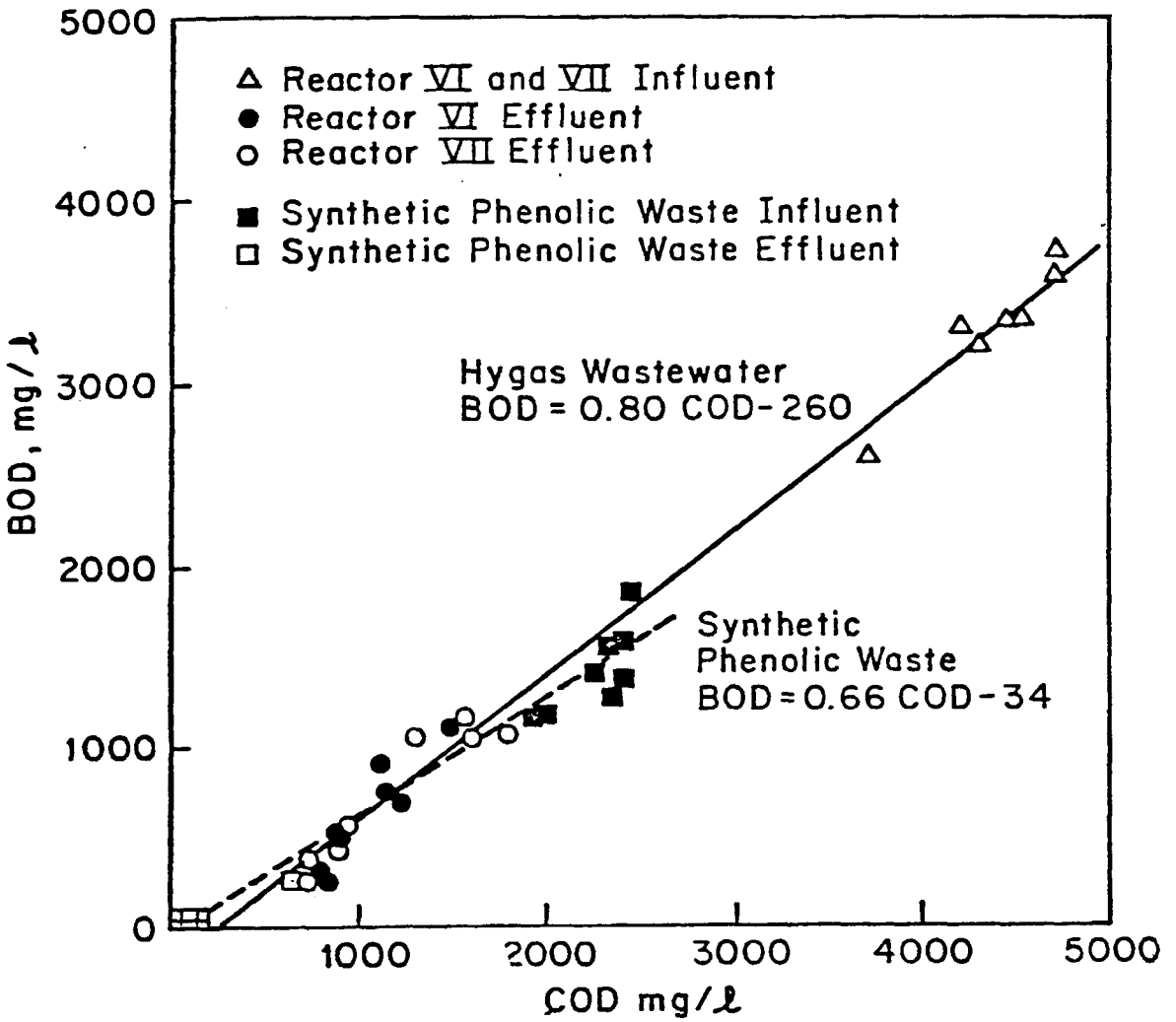


Figure 8: Correlation of BOD and COD Values for Hygas Wastewater and for a Synthetic Phenolic Wastewater.

TABLE IX

AVERAGE INFLUENT AND EFFLUENT CHARACTERISTICS AND PERFORMANCE FACTORS FOR BIOLOGICAL OXIDATION STUDIES ON FULL STRENGTH STRIPPED WASTEWATERS [1]

Reactor	Constituent[1], mg/l																			COD Removal Rate[2], day^{-1}	MLVSS mg/l	Hydraulic Residence Times, days	Mean Cell Residence Time, days
	COD			Phenol			NH_3-N			SCN^-			Alkalinity (as $CaCO_3$)			CN^-		$S^=$					
	Inf.	Eff.	% Rem.	Inf.	Eff.	% Rem.	Inf.	Eff.	% Rem.	Inf.	Eff.	% Rem.	Inf.	Eff.	Diff.	Inf.	Eff.	Inf.	Eff.				
IV	3540	660	81	620	0.1	>99	136	87	36	12	2	83	660	280	380	0.5	0.3	<9	<5	0.52	1900	2.97	20
V	3710	700	81	625	0.3	>99	148	101	32	12	2	83	710	260	450	0.4	0.4	<10	<6	0.86	1820	2.05	15
VI	4190	890	79	855	1.9	>99	136	103	24	24	4	83	1700	930	770	1.3	1.1	<2	<3	1.06	1580	2.05	10
VII	3880	660	83	940	0.5	>99	137	122	11	23	2	91	1590	710	880	1.4	1.3	<3	<3	0.37	3000	2.98	40

Notes: 1. See Appendices A-4 through A-7 for analysis of various individual and composite samples for biological oxidation kinetic study data, and Appendices A-11 through A-14 for constituent concentration data.

2. Values reported here represent mean of daily values; q was computed as the mean of individual daily removal rates rather than as a single removal rate based on mean data.

of 0.5 mg/l or less is obtained with COD removal rates less than about 0.8 per day. An increase in effluent phenol concentration at high loadings was observed in this study; this has also been reported to occur in treatment of coke plant wastewater. The data in Table IX show 11-36% removal of NH_3-N; as discussed later, this represents removals resulting largely from cell wastage and not nitrification. The biological oxidation process consumed on the order of 400-800 mg/l of alkalinity. There was essentially no removal of cyanide; this indicates that the cyanide present in the wastewater was complexed and was therefore not amenable to biological treatment.

Figure 9 shows typical measurements of O_2 uptake. Oxygen uptake data may be correlated by recognizing that the oxygen requirements in the activated sludge process are related to the oxygen consumed to supply energy for synthesis and oxygen consumed for endogenous respiration.

$$\frac{R_r}{x_1} = a' \frac{S_o - S_1}{x_1 \theta} + b' \qquad (8)$$

$$= a'q + b' \qquad (9)$$

where R_r is oxygen utilization per day, mg/day; a' is the fraction of COD used for oxidation; and b' is the fraction of MLVSS oxidized (oxygen basis). Figure 10 shows correlation of oxygen utilization with COD removal rate and it is seen a'=0.27 g O_2/ g COD removed and b'=0.07 g O_2/g MLVSS per day; these values lie within the range of values reported for treatment of phenolic and coal liquefaction effluents.

Figure 11 shows typical data for sludge zone settling velocity and sludge volume index. Figure 12 shows the relationships between zone settling velocity, sludge volume index, and COD removal rate. Optimal values of zone settling velocity and sludge volume index are associated with mean cell residence times on the order of 15 days with comparable COD removal rates of about 0.5 to 0.8 per day.

In the execution of these studies sufficient data was taken to establish correlations between reactor MLVSS and MLSS. Examples of such correlations are shown in Figure 13.

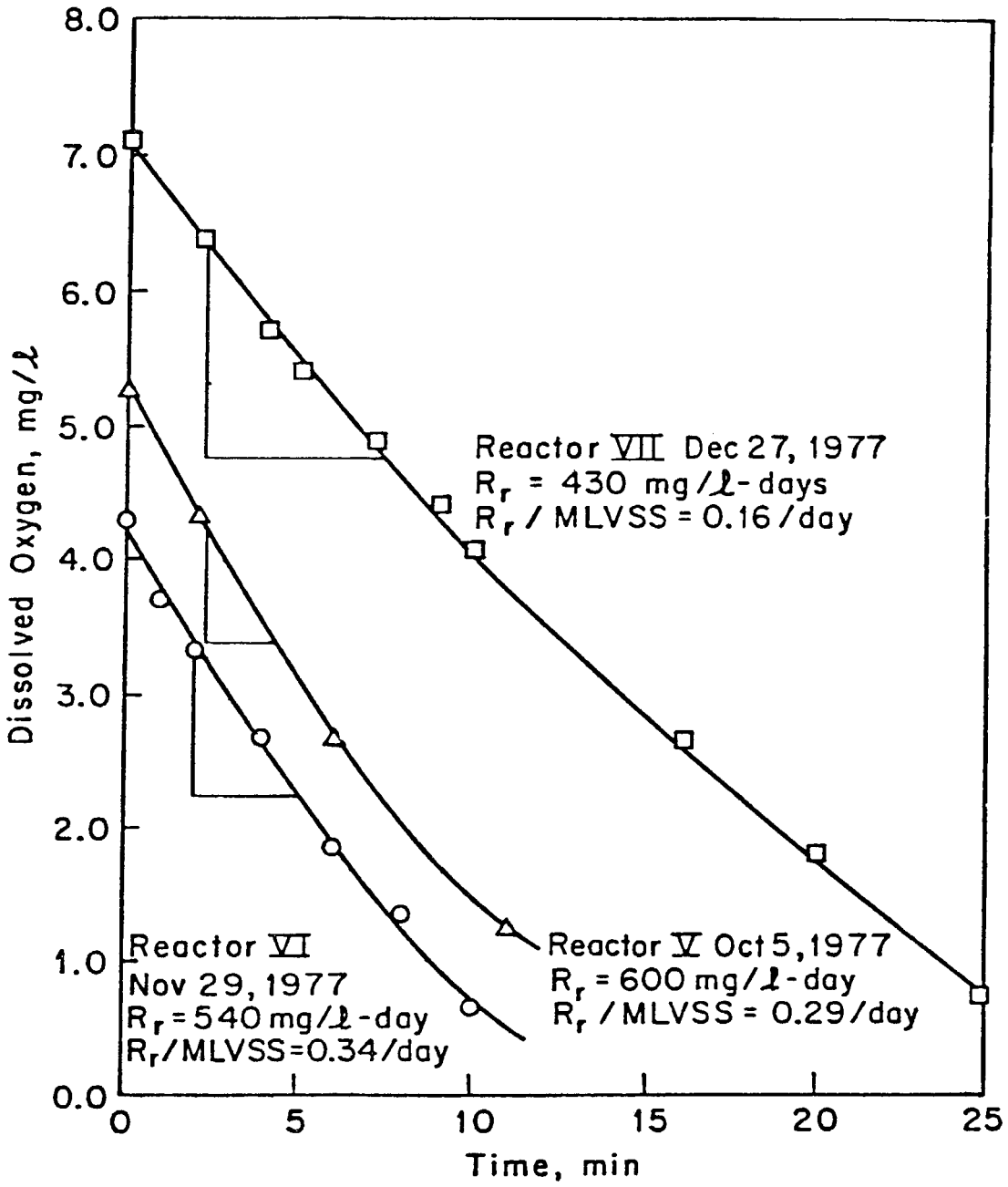


Figure 9: Typical Measurements of Oxygen Uptake Rate

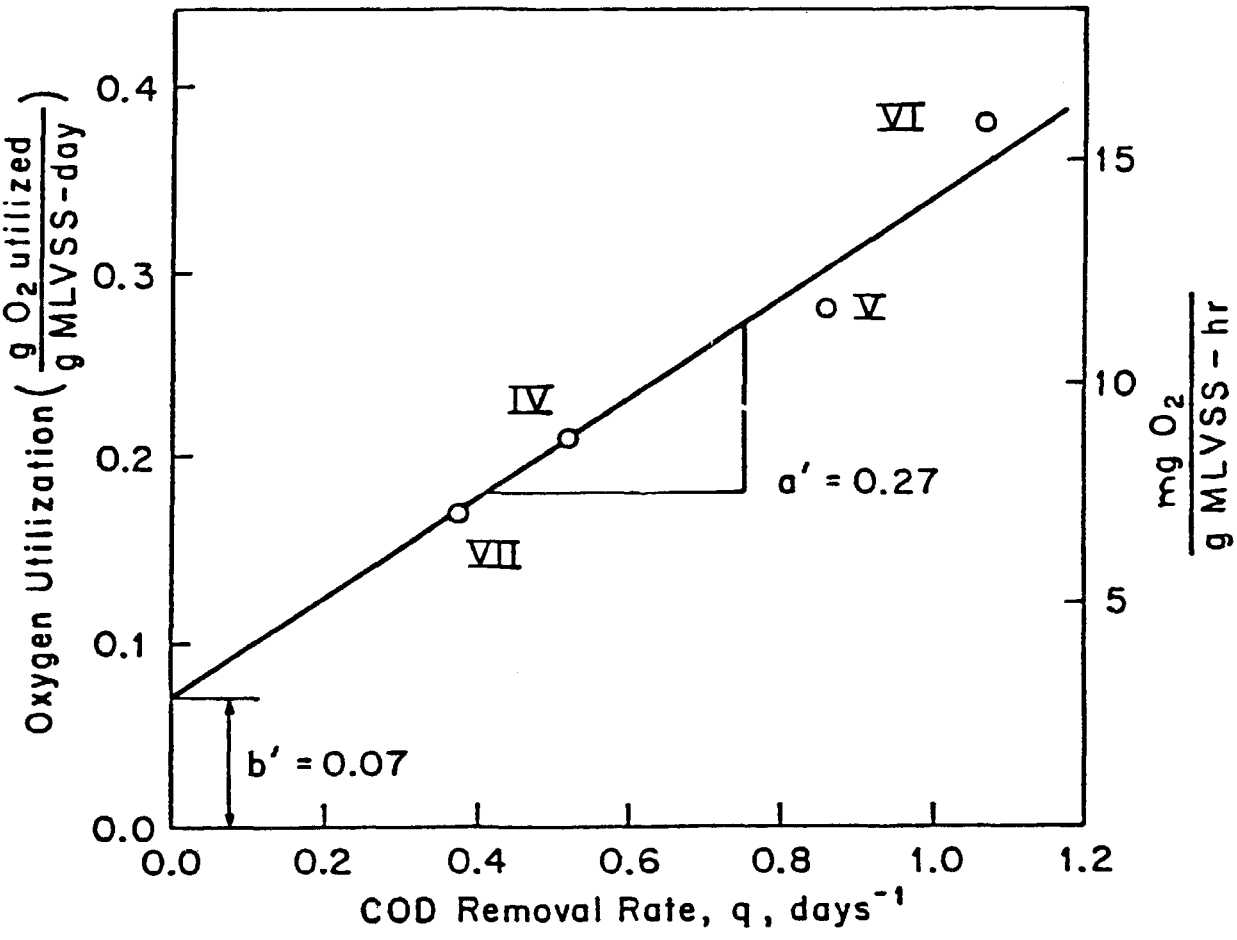


Figure 10: Correlation of Oxygen Utilization with COD Removal Rate. Mean Values of O_2 Uptake Rate and COD Removal Rate Obtained during Steady State Operation were used in this correlation.

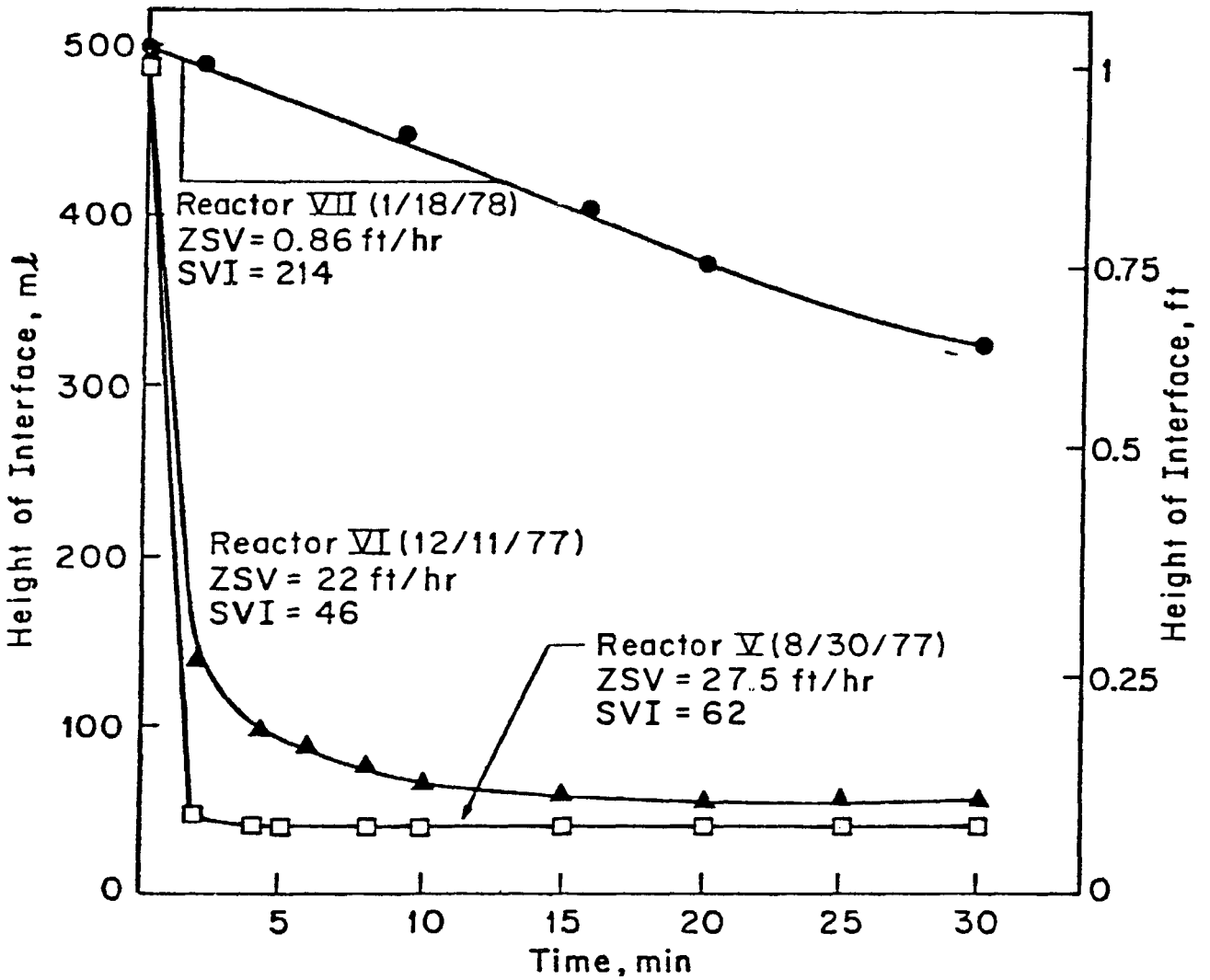


Figure 11: Typical Measurements of Zone Settling Velocity and Sludge Volume Index.

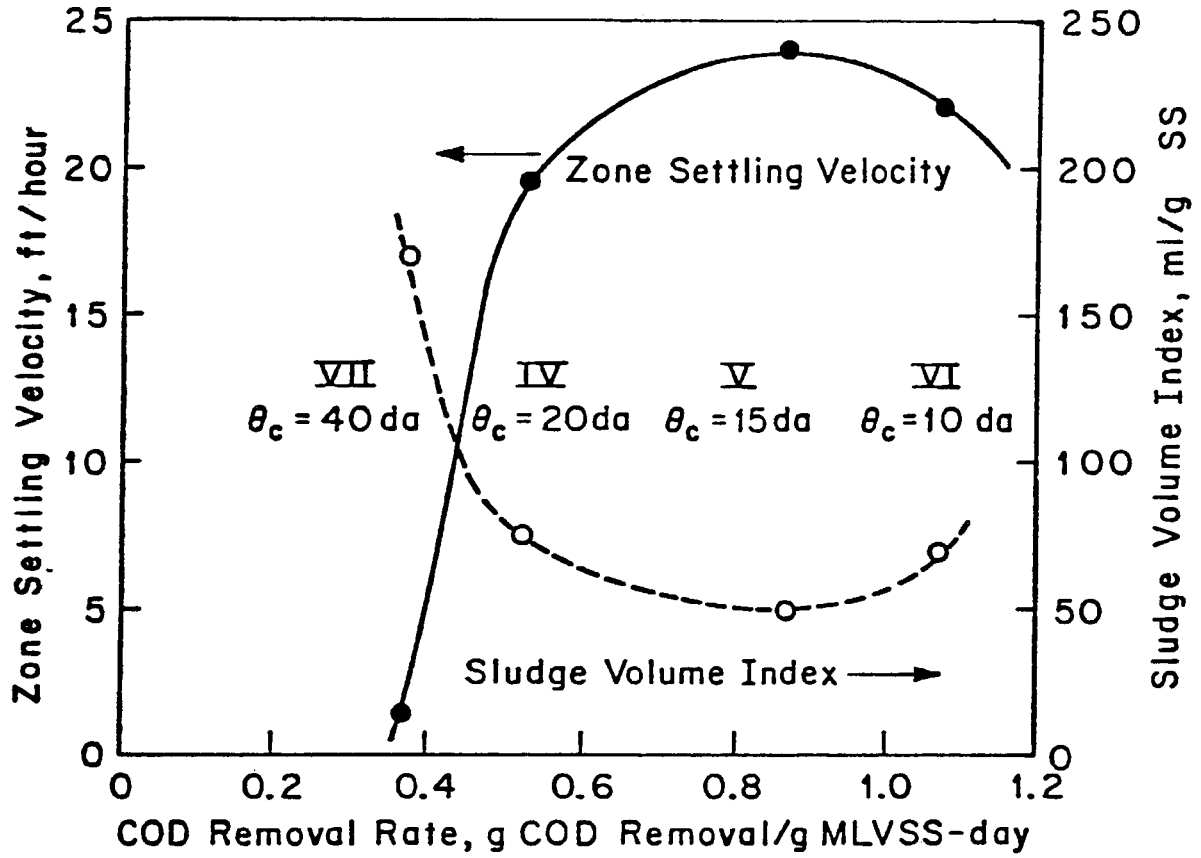


Figure 12: Correlation of Zone Settling Velocity and Sludge Volume Index with COD Removal Rate.

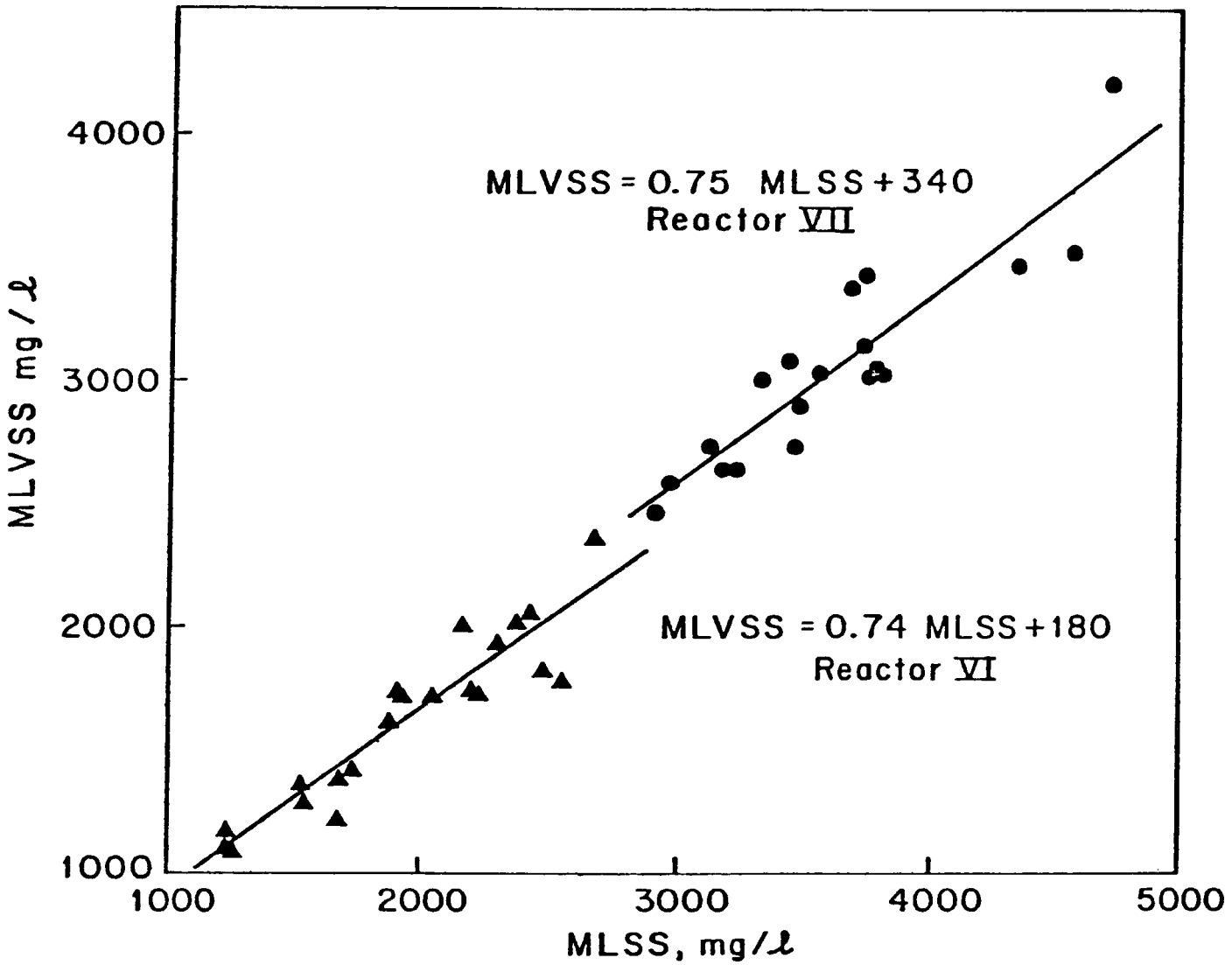


Figure 13: Correlation of Biological Reactor MLSS and MLVSS

These correlations were used in analysis of reactor kinetics when on occasion it was desired to compute a value of MLVSS based on MLSS data.

Biological Oxidation of Diluted and/or Unstripped Wastewater

Biological oxidation studies were performed with full strength and one-half strength stripped wastewater and with one-half strength stripped wastewater. The purpose was to assess the effects of ammonia concentration and dilution on biological treatment. Performance data for these tests are reported in Table X. Reactor I was acclimated slowly to full strength unstripped wastewater containing an ammonia concentration of 3200 mg/l. This reactor sustained operation at this ammonia level for about three weeks, after which it began to go into failure; resulting in high concentrations of VSS, phenol, and SCN^- in the effluent, and declining reactor MLVSS concentration. The reactor was kept on stream for about one additional month with no intentional cell wastage, after which it became evident that the reactor was not going to recover. The data reported in Table X for reactor I are from the three week period prior to onset of failure. Reactor II showed very stable performance with half-strength unstripped wastewater, NH_3-N = 1600 mg/l. This reactor was operated under steady state conditions for 3½ months; no adverse effects of the relatively high ammonia concentration were observed. This suggests that an upper limit for ammonia concentration for a biological reactor processing Hygas wastewater is less than 3200 mg/l, with 1600 mg/l being an acceptable value.

Reactor III processed half strength stripped wastewater. Reactor kinetics for this reactor and for reactors I and II are shown on Figure 14 along with the data obtained from reactors IV through VII. The data show a rather surprising result; reactors processing diluted wastewater, stripped and unstripped, appear to follow different kinetics than the reactors processing undiluted wastewater. A possible correlation for 1:1 diluted wastewater is shown on Figure 14, and an estimated yield coefficient is on the order of 0.22 assuming that the decay coefficient has not changed significantly. This is about twice that obtained with undiluted wastewater. It appears that there may be some constituents in the full strength wastewater.

TABLE X

AVERAGE INFLUENT AND EFFLUENT CHARACTERISTICS AND PERFORMANCE FACTORS FOR

FOR BIOLOGICAL OXIDATION STUDIES ON DILUTED AND/OR UNSTRIPPED WASTEWATER[1]

Reactor	Constituent[1], mg/l																											
	COD			Phenol			NH_3-N			SCN^-			Alkalinity (as $CaCO_3$)			CN^-		$S^=$		COD Removal Rate[2], day-1	MLVSS mg/l	Hydraulic Residence Times, days	Mean Cell Residence Time, days	D.O. Uptake g O_2/g MLVSS per day	Zone Settling Velocity, ft/hr
	Inf.	Eff.	% Rem.	Inf.	Eff.	% Rem.	Inf.	Eff.	% Rem.	Inf.	Eff.	% Rem.	Inf.	Eff.	Diff.	Inf.	Eff.	Inf.	Eff.						
I (full-strength unstripped)	3720	570	85	700	0.8	>99	3200	3200	-	37	11	70	910	210	700	0.13	0.15	91	11	0.50	2010	3.25	20	0.18	18
II (half-strength unstripped)	2370	275	88	340	<0.05	>99	1600	1600	-	11	1	91	500	190	310	0.3	0.2	4	9	0.22	2000	3.25	20	0.12	23
III (half-strength stripped)	1630	350	79	280	0.09	>99	67	56	16	7	2	71	370	190	180	0.3	0.4	5	6	0.31	1550	3.01	20	0.11	22

Notes: 1. See Appendices A-1 through A-3 for analysis of various individual and composite samples for biological oxidation kinetic study data, and Appendices A-8 through A-10 for constituent concentration data.

2. Values reported here represent mean of daily values; q_b was computed as the mean of individual daily removal rates rather than as a single removal rate based on mean data.

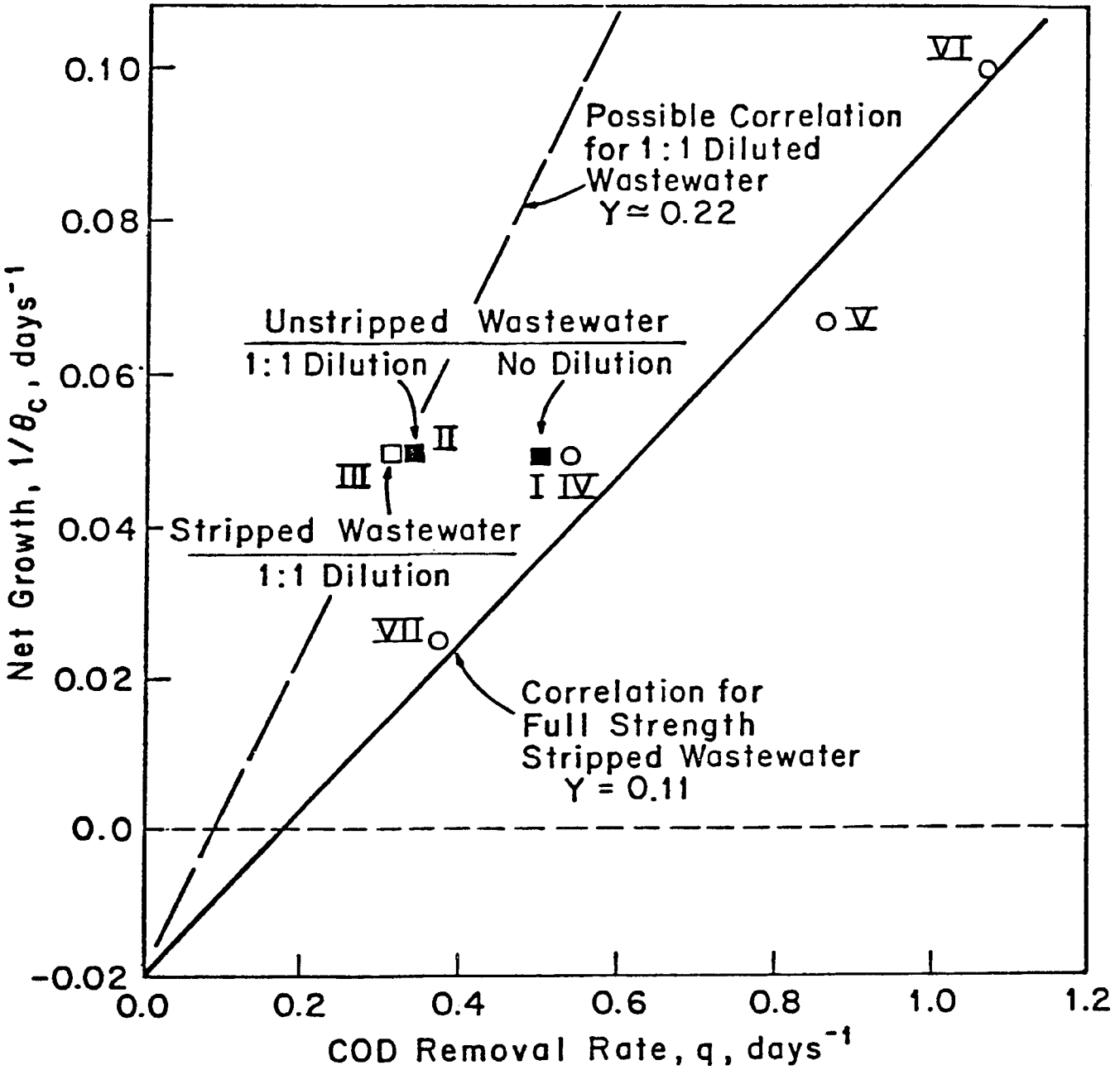


Figure 14: Correlation Between COD Removal Rate and Net Growth for Unstripped and Diluted Wastewater Feeds Compared to that Obtained for the Main Series of Biological Oxidation Experiments with Full Strength Stripped Wastewater.

which inhibited growth of the microorganisms. The nature of the inhibiting substance(s) is not known; however, boron is suspect.

Nitrification/Nitrogen Balance

Nitrate analyses were performed at various times during the study, and nitrificatio was not observed in any of the reactors including those receiving diluted feed. Typical values of influent and effluent NO_3^--N were less than 2-4 mg/l. At one point nitrifying sludge from a test reactor processing coke plant ammonia liquor was added to reactors VI and VII during their balance period. This caused effluent NO_3^--N to increase to 18 and 7 mg/l respectively for several days, but within a week effluent NO_3^--N had decreased to levels observed before seeding with nitrifying sludge. It was concluded that Hygas wastewater, whether stripped or unstripped, or full strength or half strength, was inhibitory to nitrification. It was suspected that high boron concentrations inhibited nitrification. Another reason to explain the lack of nitrification is the fact that organic loadings may have been too high. However, if nitrification was feasible, some degree of ammonia conversion should have been detected, especially for reactor III and probably for reactor VII. The COD loading for reactor II should not have inhibited nitrification based on processing data reported for biological treatment of a phenolic wastewater.

Nitrogen balances were performed on reactors IV and V (Table XI) and it was found that 70% of the removal of nitrogen could be accounted for by the nitrogen content of the cells being wasted. Significant nitrogen containing species in the influent were NH_3-N, SCN^--N, and Kjeldhal-N (10 mg/l). Kjeldhal digestion of reactor sludge revealed that approximately 14.7% of the cell biomass was nitrogen. The unaccounted nitrogen may have resulted in part from insufficient NH_3-N data to adequately describe fluctuations in stripped wastewater composition (Table II).

TABLE XI

BIOLOGICAL REACTOR NITROGEN BALANCE

Reactor	Flow l/day	Reactor Volume l	MLVSS mg/l	Mean Cell Residence Time, day	Average Removals[1], mg/l			Wastewater N Removal/day mg	Nitrogen in cells[2] wasted/day mg
					NH_3-N	Kjel-N	SCN^--N		
IV	1.76	5.2	1900	20	49	8	2.4	104	72
V	2.64	5.4	1820	15	47	3	2.4	134	96

Notes: 1. Differences between influent and effluent CN^--N and NO_3^--N were negligible.

2. Sludge contained 14.7% Kjeldhal-N.

Adsorption of Biologically Treated Effluent COD on Activated Carbon and Hygas Char

Twenty-four hour batch COD adsorption isotherm studies were performed with reactor V effluent using activated carbon and several Hygas process chars. The results are plotted on Figure 15 wherein the Freundlich adsorption isotherm has been invoked to correlate the data. These data represent preliminary observations and additional flow-through column testing must be performed to assess adsorptive capacities of activated carbon and Hygas char. It appears however that the adsorptive capacity of Hygas char is somewhat comparable to activated carbon at high equilibrium COD concentrations and that the affinity of Hygas char for biologically treated effluent COD is a stronger function of char surface area than carbon content.

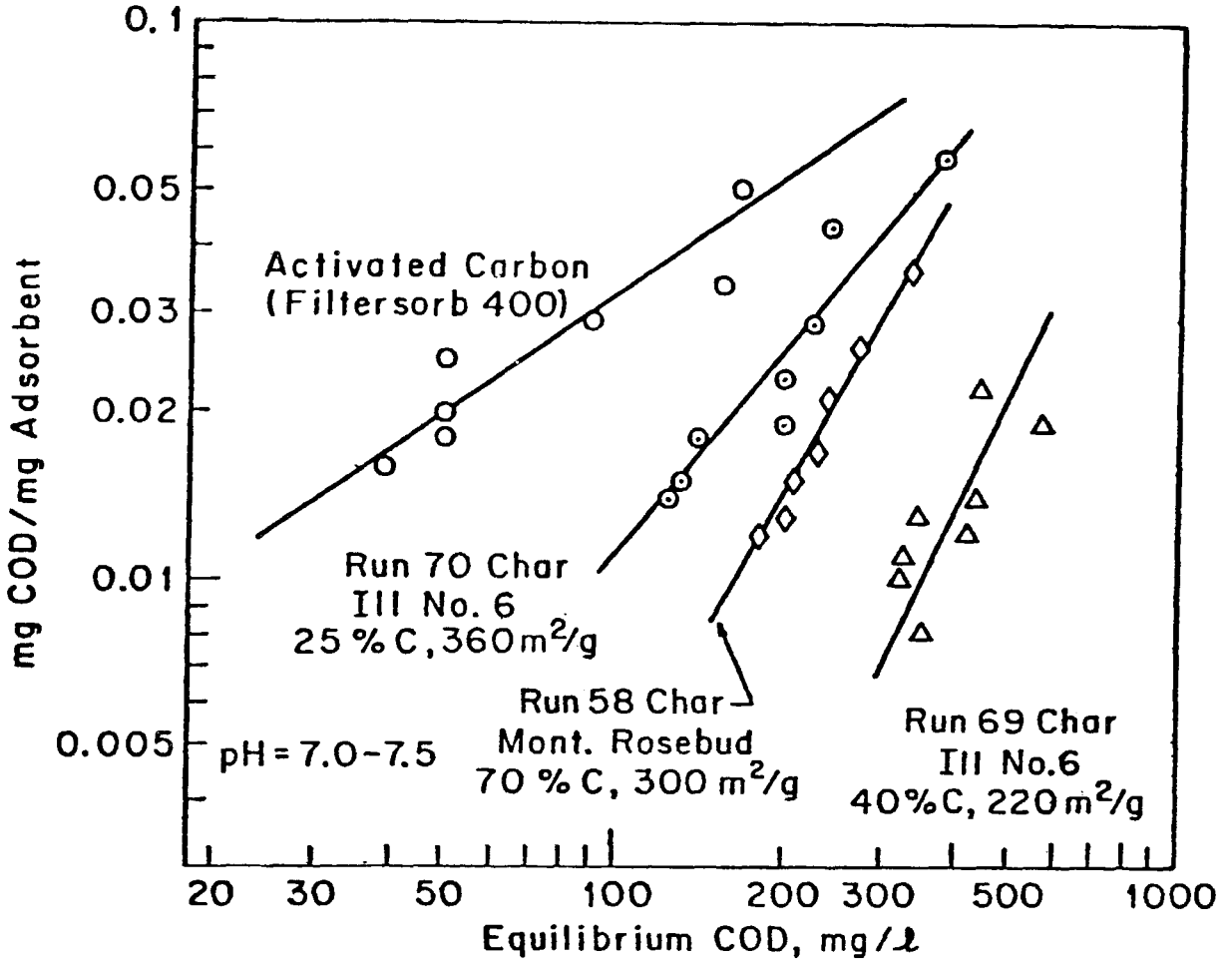


Figure 15: Adsorption Isotherms for Biologically Treated Effluent COD on Activated Carbon and on Hygas Chars

Conclusions

This study has demonstrated that Hygas process pilot plant coal gasification wastewater comprised of a 1:1 mixture of cyclone and quench effluents may be processed successfully by air activated sludge. Specific conclusions arising from the experimental investigation include the following:

1. There are many inconsistencies and inadequacies in the available published data base for biological treatment of coal conversion and coke plant wastewater. The data base for design of coke plant biological wastewater treatment facilities is inadequate to the extent that serious questions arise pertaining to design of such facilities. Because of this, it is not feasible to envision transferring other than general information pertaining to biological oxidation of coke plant effluents to processing of coal gasification wastewaters.

2. Wastewater storage by freezing was found to be an effective means for preserving the wastewater for COD, phenol, NH_3, CN^-, SCN^-, $S^=$, and pH.

3. Essential features of a biological treatability study must include appropriate concern for cell residence times as well as hydraulic residence times. One of the most serious drawbacks in the available data base is that the studies have been too short and that the attainment of steady state has been defined on the basis of hydraulic residence time rather than mean cell residence time. Biological reactors must be in operation for three mean cell residence times (balance period) prior to definable steady state operating conditions.

4. Wastewater pretreatment must include provision for removal of high wastewater alkalinity to prevent upward pH fluctuations during biological treatment. This may be accomplished by lime precipitation, or by stream or air stripping at elevated temperatures without chemical addition. Not all the alkalinity need be removed as approximately 1000 mg/l residual alkalinity (as $CaCO_3$) in biological reactor influent provided sufficient buffer capacity for pH stability during biological treatment. Another pretreatment requirement for biological oxidation is ammonia reduction. This may be achieved by either air or stream stripping

along with CO_2 removal at high temperature, or by air stripping at elevated pH, or by 1:1 wastewater dilution.

5. Phosphorous must be added as a necessary nutrient for biological treatment of Hygas wastewater.

6. Screening analyses for potentially inhibitory/toxic elements in Hygas wastewater showed no apparent problems except for boron (80 - 110 mg/l). Boron was suspected of inhibiting nitrification and may be a probable cause for observed low biological growth rates.

7. Ammonia stripped Hygas wastewater was treatable at hydraulic residence times of 2 to 3 days with mean cell residence times ranging from 10 to 40 days. It was possible to remove approximately 80% of 4000 mg/l influent COD, to reduce effluent phenol to less than about 0.5 mg/l with COD removal rates of about 0.8/day or less, and to remove about 80-90% of influent SCN^-. There was essentially no removal of CN^- (0.5 - 1.4 mg/l), which suggests that CN^- was complexed. Mean cell residence times of less than about 10 days are not likely to be sustained in Hygas wastewater. Foaming was a continual problem, and it was made worse by high loadings. A substantial fraction of biological effluent COD may be relatively refractory to biological treatment.

8. Biological kinetic analysis of reactor performance showed that influent COD, effluent COD, mean cell residence time, hydraulic residence time, and sludge concentration (MLVSS) were correlated well by the model. Treatability studies with ammonia stripped wastewater showed a yield coefficient of 0.11 (COD basis) and a decay coefficient of 0.02/day. The relatively low yield coefficient suggested that growth was inhibited. Oxygen utilization coefficient a' and b' were 0.27 g O_2/g COD removed and 0.07 g O_2/g MLVSS-day. Optimal sludge zone settling velocities (20-24 ft/hr) and sludge volume indicies (50 - 75) occurred for mean cell residence times on the order of 15 - 20 days with COD removal rates of about 0.8 - 0.5/day respectively.

9. Recommend design criteria for activated sludge treatment of stripped Hygas wastewater include: mean cell residence times of 15 to 20 days, COD removal rate of approximately 0.3-0.5/day, hydraulic residence time of about 3 days, dissolved oxygen greater than 3 mg/l, influent alkalinity of about 1000 mg/l (as $CaCO_3$), and a pH between 7.0 and 7.5.

10. Studies with unstripped Hygas wastewater demonstrated that high ammonia concentrations (3200 mg/l NH_3-N) produced unstable operating conditions and caused the reactor to go into failure after about three weeks of operation. A 1:1 dilution of unstripped wastewater (1600 mg/l NH_3-N) produced stable biological reactor performance. These data indicate approximate boundary ranges for acceptable ammonia levels in treatment of Hygas wastewater.

11. Similar reactor kinetics were observed with wastewater at 1:1 dilution for stripped and unstripped wastewater. The data suggest higher biological yield coefficient for treatment of diluted wastewater, which provides another reason for suspecting that biological growth was inhibited in full strength Hygas wastewater. However, this factor is not especially significant at recommended processing conditions.

12. Nitrification was not observed at any time during the study; it was suspected that boron inhibited nitrification. However, additional studies at lower COD removal rates need to be conducted in order to confirm this observation.

13. Preliminary adsorption isotherm data for biological reactor effluent COD on Hygas process char gave somewhat comparable capacities as activated carbon at high equilibrium COD concentrations for char with relatively high surface areas. The data indicate that char COD adsorptive capacity is more dependent upon char surface area than carbon content.

14. It is recommended that additional biological treatability studies be performed to assess pure oxygen treatment and treatment with addition of char and/or activated carbon. Additional studies need to be performed to determine under what conditions nitrification may be achieved in Hygas wastewater. It is also recommended to assess treatment of Hygas quench water (COD > 7500 mg/l) without blending with

cyclone water. Some of these recommendations are now being evaluated in subsequent screening investigations of Hygas wastewater reuse strategies.

Note:

Additional studies now in progress in our laboratory which relate to this work include:

(i) Investigation of biological treatability characteristics of Grand Forks slagging process wastewater,

(ii) Investigation of biological oxidation characteristics of a coke plant ammonia still liquor, and

(iii) Evaluation of water reuse strategies for the Hygas process.

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