

ACTIVATED-SLUDGE TREATMENT AND ORGANIC CHARACTERIZATION
OF OIL SHALE RETORT WATER

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ABSTRACT

A sample of Oxy-6 retort water was analyzed for its organic composition and evaluated for response to treatment by combinations of ammonia stripping and ozonation. This was followed by activated sludge treatment with and without powdered activated carbon (PAC). Biological treatment of both raw and ammonia-stripped influents without the addition of PAC resulted in dispersed and poorly settleable biological flocs. These systems exhibited very little biological growth and poor process stability. It was concluded that biological treatment of full-strength raw or ammonia-stripped oil shale retort water was not practical. Stable biological oxidation could be established when preceded by ammonia stripping and ozonation. However, problems of dispersed and low biological growth also persisted in this process.

Use of activated sludge with PAC to treat ammonia-stripped retort water alleviated many of the problems related to toxicity and low biological growth. This process was shown to remove 89% of the chemical oxygen demand and produce an easily settleable floc. This type of reactor was operated with a hydraulic residence time of six days and a solid residence time of forty days. Pretreatment of ammonia-stripped retort water by ozonation prior to activated sludge-PAC processing provided some benefits to the treatment system. The PAC is apparently essential to the formation of a settleable floc and may also remove material inhibitory to biological treatment. In all cases it was necessary to adjust the pH of the influent to a range of 5.0 to 5.5 in order to maintain the pH of the reactor mixed liquor at about 7.0.

The organic materials in the retort water that are refractory to biological oxidation belong to the base/neutral fraction and consist of oxygenated, unsaturated nitrogen heterocyclics. Their disposition in biological treatment varies, but in general the relative proportions of the compound classes are maintained with treatment. The piperidinones, pyrrolidinones, and cyclopentanopiperidinones are the dominant groups identified in the base/neutral fraction of the influent, and were refractory in biological treatment. The acid fraction consisted primarily of carboxylic acids as large as C₁₁, and these compounds were removed almost completely by activated sludge treatment.

INTRODUCTION

The nature of organic constituents in oil shale retort water such as that from Occidental Oil Shale Company's modified in-situ retort 6 (Oxy-6) is considered to be extremely varied and complex (1). Moreover, these constituents are difficult to treat efficiently in a biological oxidation process (2-6). It is desirable to achieve efficient biological treatment for oil shale retort water, because this would provide industry with an option for reducing

relatively high concentrations of organic material in retort water. This may be significant considering the quantity of retort water which must be handled. Effective reduction in retort water organic concentrations could facilitate water reuse and conservation, as well as reduce the potential for environmental contamination. For these reasons, this study was conducted to: (a) provide a comprehensive analysis of the organic material in treated retort water, (b) evaluate the performance efficiency of variations in activated sludge treatment for processing retort water, and (c) follow the disposition of certain organic compound classes in the treatment processes.

EXPERIMENTAL APPROACH

The modified in-situ retort water used in this study was a sample collected from the separator tank immediately downstream from Oxy-6 retort. The sample was collected, filtered, and held in cold storage at Laramie Energy Technology Center for subsequent distribution.¹

Characterization of the organic material consisted of analyses of the ammonia-stripped and biologically treated samples using (a) methylene-chloride solvent extraction to fractionate and concentrate the dissolved organic material into three fractions; and (b) analysis by gas chromatography (GC), mass spectrometry (MS), and high-resolution MS. The gas chromatograph used was a Hewlett-Packard (HP) 5840 fitted with various accessories including an HP-50M fused-silica carbowax 20M capillary-column. Previous work (1) with fused silica OV-101 columns provided less distinct peaks in separations and in peak shape. A limited analysis with a high-resolution MS (VG 7070) fitted with a SLAM magnet and a Varian 3700 series GC was also used to verify the existence of certain compounds detected by the GC/MS analysis.

The biological treatment study consisted of bench-scale experiments in which continuous-stirred reactors were used to evaluate variations in activated-sludge applications. With the exception of one experiment (see point 2 in Figure 1), all treatment units received retort water influent that had been:

1. Ammonia-stripped by heating the water in a sealed tank at about 90°C and purging with nitrogen gas. The outlet gas was condensed on a cooled glass surface to maintain the volume. The retort water was stripped to a residual ammonia concentration of about 50 mg/L NH₃-N.
2. Nutrient-enriched with phosphorus.

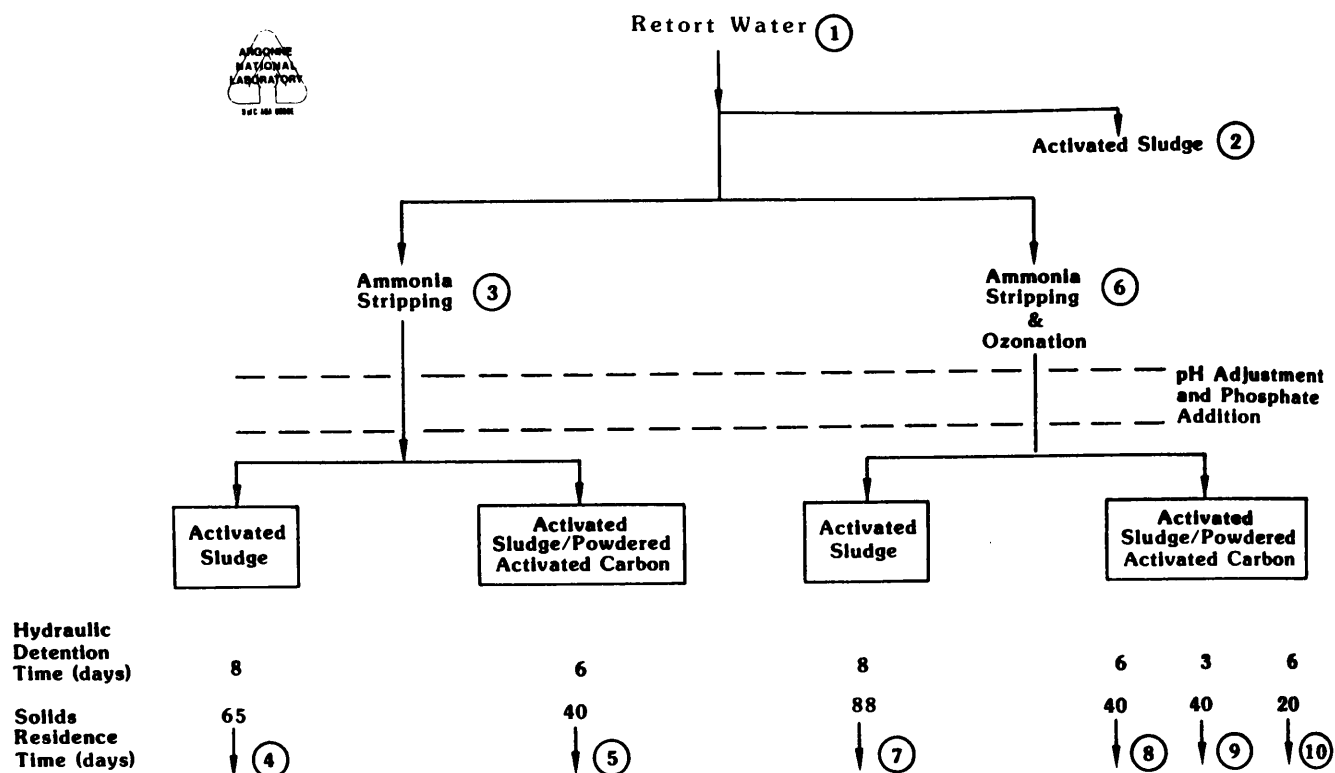


Fig. 1 Flow Diagram of Oxy-6 Retort Water Treatment Study

3. Treated with sulfuric acid prior to biological oxidation in order to lower reactor influent pH from a value of about 10 after ammonia stripping, to the range of 5.0-5.5. This was necessary to maintain a reactor-liquor pH of about 7.

Variations in treatment of the ammonia stripped retort water, as described in Fig. 1, included:

1. Activated sludge (NH₃/AS) -- reference point 4.
2. Activated sludge with powdered activated carbon (NH₃-AS/PAC) -- reference point 5.
3. Ozone pretreatment and activated sludge (NH₃/O₃-AS) -- reference point 7.
4. Ozone pretreatment and activated sludge with powdered activated carbon (NH₃/O₃-AS/PAC) -- reference points 8, 9, and 10.

The PAC concentration in the mixed liquor of the reactors was maintained at 5000 mg/L, and the ozone dose applied to biological reactor influent was estimated at 70 to 75 mg/L. Dissolved oxygen in the reactor was maintained at greater than 3 mg/L; reactors were operated at room temperature. All biological reactors were allowed a period of acclimation followed by a period to achieve steady-state operating conditions. The reactors were then operated for six to seven weeks or more to obtain sufficient effluent characterization data.

TREATMENT

The Oxy-6 retort water characteristics are shown in Table 1. Ammonia stripping achieved significant reductions in ammonia without the need for addition of lime or caustic. Stripping resulted in an increase in pH and slight losses in SOC and COD and a decrease in alkalinity by about 50%.

The pH of the ammonia stripped water was lowered to pH 7 before ozonation. The effects of ozonation, as reported in Table 1, were a significant reduction in color and lowered levels of phenol, COD, SCN, and pH. An increase in organic nitrogen was also measured.

Activated-sludge treatment of raw unstripped retort water with pH adjustment and phosphorus enrichment was tested (see point 2 in Fig. 1). The initial microbiological seed was a mix of activated-sludge solids from units treating coal-coking and coal-gasification condensate water. During this portion of the study, feed strength was gradually increased to 100%. It was found that stable performance conditions could not be achieved at the high feed strengths, that biological solids settled poorly, and that concentrations of mixed-liquor volatile suspended solids declined steadily. For these reasons, it was concluded after several months of unsuccessful acclimation, that it is not feasible to treat unstripped retort water at high feed strength with the microbiological culture used.

Table 1 Typical Characteristics of Oxy-6 Retort Water

Parameter	Average Concentration		
	Raw	Ammonia-Stripped	Ozonated ^a
	(mg/L except as noted)		
COD	8400 - 9100	7000 - 7200	6500
SOC ^b	2800 - 3300	2200 - 2300	2000
Phenol	45	18 - 24	0.4
NH ₃ -N	1100 - 1250	7 - 40	33
Organic Nitrogen	34	21	53
NO ₃ -N	<1.0	<1.0	<1.0
pH (units)	8.7	9.9 - 10.3	6.4
Alkalinity (as CaCO ₃)	6300 - 7800	3300 - 4100	900 - 1800
Color (Pt-Co units)	2500	2500	300
SCN	55 - 60	45 - 52	4
TDS	9000 - 9500	9000 - 10,000	10,000

^aO₃ dose = 70 mg/L at pH of 7.0.

^bSoluble organic carbon.

Biological treatment of ammonia-stripped retort water (NH₃-AS) achieved a 67% reduction of COD across the biological reactor. However, the mixed liquor consisted of pinpoint, dispersed flocs with poor settling characteristics. This resulted in a slow decrease of mixed liquor volatile suspended solids. Despite an operating period of about 100 days, it is probable that the reactor had not sustained steady-state operation because the reactor mixed-liquor suspended solids were declining and because effluent suspended solids were high. Hence, it was concluded that biological treatment of full-strength, ammonia-stripped retort water was not a promising treatment alternative.

The NH₃-AS/PAC unit treated the same quality of influent retort water as did the NH₃-AS unit, but in this case Amoco PX-21 powdered activated carbon (PAC) was added to the reactor mixed liquor to maintain a PAC concentration of 5,000 mg/L. The reactor was operated at a hydraulic detention (θ_H) time of 6 days and a solids residence time (θ_c) of 40 days. These operating conditions produced an easily settled biological floc. The NH₃-AS/PAC reactor gave an effluent COD of 950 mg/L, which corresponded to reactor COD removal efficiency of 86% and a system COD removal efficiency (including ammonia stripping) of 89%. The soluble organic carbon concentration was reduced by 90% as a result of stripping and AS/PAC treatment.

The performances of reactors treating ammonia-stripped and ozonated influent retort water was somewhat similar to that observed for the NH₃-AS reactor, except in this case reactor mixed-liquor volatile suspended solids remained at a comparatively steady level. The reactor was operated at an hydraulic detention time of eight days. A long value a sludge age was necessary because the unit demonstrated low biological growth. For this reason sludge was not intentionally wasted from the aeration basin. A sludge age of 88 days was calculated from the solids removed with the reactor effluent. The COD removal efficiency was 79% across

the biological reactor, and effluent color was 500 Pt-Co units. It was concluded that full-strength retort water could be treated biologically by employing pretreatment consisting of ammonia stripping and ozonation. The system demonstrated dispersed biological growth, however, as well as very little yield in the mixed liquor. For these reasons, AS treatment of ozonated-stripped retort water does not seem practical. During the first few minutes of ozonation in batch tests, foaming was excessive; this problem, however, would probably not be significant in a continuous-operation mode.

Three biological reactors were operated with addition of PAC to evaluate the combined effect of pretreatment consisting of stripping and ozonation followed by activated sludge-PAC treatment. These reactors were operated at different combinations of hydraulic detention times and solids retention times to evaluate the effects of changing these parameters on performance. One of these reactors was operated at θ_H equal to 6 days and θ_c of 40 days to provide comparison data with the AS/PAC reactor which received stripped retort water without ozonation. It appeared that ozone pretreatment at a dose of 70 mg/L rendered the retort water slightly more amenable to treatment in the AS/PAC reactors. This can be seen in Table 2 by comparing the biological COD removal of the NH₃-AS/PAC reactor with that of the NH₃/O₃-AS/PAC reactor operated at θ_H of 6 days and θ_c of 40 days. The latter reactor achieved 90% COD removal efficiency, a slight increase over the NH₃-AS/PAC reactor. The total efficiency of the NH₃/O₃-AS/PAC system, including that of ozonation, was only 4% more efficient than the reactor without ozonation.

Table 2 summarizes average operating conditions for the various reactors in which food-to-microorganism ratio and specific utilization rate have been calculated from an estimated value of the biomass concentration in the mixed liquor for the AS/PAC reactors. It was assumed that the PAC was

Table 2 Average Operating Conditions for Retort Water Treatment

Activated Sludge Reactor	θ_H (day)	θ_C (day)	S_0 Influent COD ^a	MLSS (MLSS-PAC) ^b	MLVSS (MLVSS-PAC) ^c	S Effluent ODD	F/M^d $\frac{F/M^d}{S_0/(9 H^k)}$	U^e $\frac{S_0 - S_1}{\theta_H X}$	O_2 Consumption Rate ^f (day ⁻¹)	Biological COD Removal (%)	Overall COD Removal Efficiency From Raw Water (%)
AS(g)	10	248	8,300	1,910	1,670	2,830	0.50	0.33	0.28	66	68
NH ₃ -AS(g)	8	65	8,400	2,300	1,590	2,780	0.66	0.44	0.30	67	71
NH ₃ -AS/PAC	6	40	8,370	11,900 (6,900)	10,600 (5,600)	950	0.25	0.22	0.08	88	89
NH ₃ /O ₂ -AS	8	88	7,900	4,190	2,830	1,690	0.35	0.27	0.31	79	81
NH ₃ /O ₂ -AS/PAC	6	40	6,500	10,500 (5,500)	9,800 (4,800)	670	0.23	0.20	0.12	90	93
NH ₃ /O ₂ -AS/PAC	3	40	6,500	14,400 (9,400)	13,000 (8,000)	980	0.27	0.23	0.13	85	89
NH ₃ /O ₂ -AS/PAC	6	20	6,500	8,400 (3,400)	7,900 (2,900)	580	0.37	0.34	0.18	91	94

^aConcentrations in mg/L except as noted

^bMLSS concentration excluding 5000 mg/L of PAC

^cMLVSS concentration minus 5000 mg/L of PAC, which is assumed 100% volatile.

^dFood to Microorganism Ratio (d⁻¹) based on ODD, and MLSS minus PAC concentration in mixed liquor

^eSpecific substrate utilization rate (d⁻¹) based on ODD, and MLVSS minus PAC concentration in mixed liquor

^fUnits in mg/L O₂ per mg/L MLVSS·day

^gThese reactors are not considered viable treatment options. Non-steady state data are included here for comparison purposes only. See text for discussion.

100% volatile, and thus 5000 mg/L was subtracted from the measured value of reactor MLVSS to estimate biomass concentration. Previous tests with this PAC have shown that this is a reasonable assumption. Table 2 also compares COD removal efficiencies across the biological reactors, as well as COD removal efficiencies from raw water to reactor effluent. Note that the data for the AS and NH₃-AS systems represent non-steady state conditions. Treatment of raw retort water by AS or treatment of full-strength ammonia stripped retort water by AS are not considered feasible options; performance data for these reactors are presented for purposes of providing information on operating conditions which resulted in unsuccessful treatment.

Best overall COD removal efficiency and reactor COD removal efficiency was achieved in the NH₃/O₃-AS/PAC system operated at θ_H of 6 days and θ_C of 20 days. Pretreatment by NH₃/O₃ followed by AS/PAC treatment at θ_H of 6 days gave somewhat lower effluent COD concentrations and somewhat higher overall COD removal efficiencies than the comparable NH₃-AS/PAC treatment train.

Table 3 compares the effluent quality of the four AS/PAC reactors. Ozonation prior to AS/PAC treatment provided lower effluent color, ammonia-nitrogen, and organic-nitrogen. Data in Tables 2 and 3 indicate that the following is essential for effective activated sludge treatment: (i) ammonia stripping, (ii) influent pH adjustment to the pH range of 5.0 to 5.5, and (iii) inclusion of PAC. Phosphorus addition may also be essential, although it was not tested directly. Inclusion of ozonation as a pretreatment step can be considered marginally beneficial for promoting more efficient removal of COD, color, ammonia, and organic-nitrogen.

In practice it is desirable to operate AS/PAC reactors at high values of θ_C to minimize PAC makeup and regeneration requirements. It is also desirable to operate with θ_H as low as practicable to minimize aeration tank size and associated capital and operating costs. It is noted that a θ_C of 100 days with AS/PAC systems have been reported for refinery waste treatment (8); however, it is not known if such long values of θ_C may be acceptable for treatment of retort water. Minimum practical values of θ_H are also not known for treatment of retort water, although data presented here suggest that successful treatment may be achieved with θ_H in the range of three to six days.

Ozonation was shown to remove color, and it also may have oxidized some of the nitrogen species associated with nitrogen heterocyclic compounds in oil shale retort water. Partial evidence of this was observed by an increase concentration of organic (Kjeldhal) nitrogen after ozonation. Nitrogen heterocyclic compounds are not usually reported by the Kjeldhal digestion technique, and ozonation probably rendered some of these materials amenable to the digestion procedure.

ORGANIC CONSTITUENT ANALYSIS

The results of organic constituent analysis of Oxy-6 ammonia-stripped retort water and of three effluents from activated-sludge treatment units are presented in Tables 4 and 5. Table 4 is a summary of the estimated concentration of extractable-chromatographable (E-C) organic material from the aqueous samples. As indicated, 80% of the E-C material in the ammonia-stripped water was in the acid fraction and only 20% was in the base and

Table 3 Effluent Quality of Activated Sludge/Powdered Activated Carbon Reactors

Parameter ^a	θ_C	Average Concentration				
		NH ₃ -AS/PAC		NH ₃ /O ₃ -AS/PAC		
		6	40	6	3	6
COD		950	670	670	980	580
SOC		270	270	270	370	220
BOD		---	61	61	81	30
Phenols		<.05	<.1	<.1	<.1	<.1
SCN		0.32	1.7	1.7	2.2	1.7
Color (Pt-Co units)		500	45	45	140	30
NH ₃ -N		20.7	9.6	9.6	10.9	9.8
NO ₃ -N		12.9	6.3	6.3	8.6	5.3
Organic N		16	9.7	9.7	11.4	9.0
CN-total		0.09	0.04	0.04	0.06	0.03
Freon Extractables		3.8	<1	<1	<1	<1
TDS		9700	9000	9000	8400	8600
Conductivity (mho/cm)		8700	8900	8900	9100	9300
Alkalinity (as CaCO ₃)		300	370	370	590	480
pH (units)		6.7	7.2	7.2	7.3	7.3

^aIn mg/L except as noted.

Table 4 Extractable/Chromatographable Fractions and Their Reduction
(Estimated and Relative Concentration)

Fraction	NH ₃ -Stripped		NH ₃ -AS		Reduction	NH ₃ /O ₃ -AS		Reduction	NH ₃ -AS/PAC		Reduction
	(mg/L)	(%)	(mg/L)	(%)	%	(mg/L)	(%)	%	(mg/L)	(%)	%
Acid	286	80	4.6	13	98	12.2	22	96	6.9	18	98
Base/Neutral	71	20	29.4	87	59	42.1	78	41	30.7	82	57
Total	357	100	34.0	100	--	54.3	100	--	37.6	100	--

neutral fractions. In contrast, the base fraction contributed about 80-90% of the residual E-C material in treated effluents. The data indicate somewhat similar removal efficiencies of the base/neutral fraction and suggest the refractory nature of this component to biological oxidation. While removal efficiency for acid fraction materials reached almost 100%, removal efficiencies of the base/neutral fraction ranged from about 30 to 60%. Minor differences in removal of base/neutral constituents among the various reactors are not considered especially noteworthy in view of uncertainties regarding the accuracy and precision of methylene chloride extraction of oil shale retort water.

The types of organic compounds in the E-C material identified by GC/MS are summarized in Table 5. The list of compound types is compiled from the identification of more than 150 compounds in the acid, base, and neutral fractions of each

sample. For purposes of presentation, the base and neutral fractions are combined because some compound types are common to both fractions.

Carboxylic acids in the C₆ to C₁₁ molecular-weight (MW) range are the predominant material in the acid fraction, and these compounds comprise almost 60% of the identified compounds in the acid fraction of the ammonia stripped influent water. The remainder of the acid fraction consists of carboxylic acids in the C₁ to C₅ MW range, phenols and cresols, and benzoic and toluic acids.

Compounds identified in the base/neutral fraction consisted primarily of unsaturated, oxygenated, nitrogen heterocyclics. The predominant group identified in the base/neutral fraction was the piperidinones (and their alkylated substitutes) which contributed almost one-half of the total. Other compounds identified in the base/neutral fraction were cyclopentanopiperidines,

Table 5 Organic Compounds Identified from the Oxy-6 Retort Water

Identified E-C FRACTION	Influent NH ₃ -Stripped			NH ₃ -AS		% Reduction NH ₃ /O ₃ -AS		NH ₃ -AS/PAC	
	ug/L	% of Fraction	% Total	ug/l.	% Reduction	ug/L	% Reduction	ug/L	% Reduction
ACID									
Carboxylic Acids									
C ₁ - C ₅	40,708	21		314	99	126	100	277	99
C ₆ - C ₁₁	113,383	58		3,271	97	1,039	99	-	100
Phenols/Cresols Acids			28,268	15	- ^a	100	-	100	-
Benzoic/Toluic Acids			11,418	6	-	100	504	96	100
Total	193,777		85	3,585		1,669		277	
BASE/NEUTRAL									
Cyclohexenones			439	1	-	100	-	100	-
Pyrroles			252	1	143	43	107	58	221
Pyrrolidinones (C ₁ - C ₅)			5,541	16	4,155	25	5,262	5	6,055
Pyridines			131	<1	30	77	-	100	-
Piperidinones			15,519	45	5,667	63	8,562	44	5,956
Quinolines			455	1	382	16	-	100	36
Cyclopentanopyrrolidinones			703	2	403	43	139	80	524
Cyclohexenopyrrolidinones			2,621	8	524	80	1,223	53	762
Cyclopentanopiperidinones			8,644	25	2,729	68	8,048	7	4,011
Total			34,305	15	14,033		23,341		17,565
			Σ = 228,082		Σ = 17,618		Σ = 25,010		Σ = 17,842

a- Signifies none detected.

b+ Signifies an increase.

pyrrolidinones, and cyclohexanopyrrolidinones. Lesser groups in this fraction were cyclohexenones, pyrroles, pyrridines, quinolines, and cyclopentanopyrrolidinones.

The resistance of base/neutral fraction material to biological oxidation is also evident from the data in Table 5. The exception is the cyclohexenones which apparently are completely removed by activated sludge treatment. All the other compound groups are nitrogen heterocycles and, except for the 2% that are pyrroles, pyridines, and quinolines, the remaining compounds are unsaturated, oxygenated, nitrogen heterocyclics. The order of dominance among the four most abundant compound types in the influent is maintained in the effluents.

SUMMARY

The activated sludge process was shown effective in reducing the chemical oxygen demand and soluble organic carbon of the Oxy-6 retort water by approximately 90% when:

1. The raw water is ammonia stripped to reduce the high ammonia concentration.
2. The influent to the biological reactor has pH adjusted to about 5.5, and nutrient phosphorous is added.
3. PAC is added to the biological reactor.

Successful operation of the activated-sludge treatment requires an acclimated culture of microorganisms grown in the presence of powdered activated carbon (PAC). Although the optimal PAC concentration or type is not identified in this study, it appears from the research that the hydraulic detention time of the NH₃-AS/PAC system may be in the range of three to six days, with a mean-cell residence time of 40 days or more. Additional bench tests that optimize these parameters would be beneficial if not essential for obtaining treatment design information. The NH₃-AS/PAC system provides a settleable floc and PAC is effective in reducing the water's dark color. Inclusion of ozonation as a pretreatment step is marginally beneficial for enhancing removal of COD, color, ammonia, and organic nitrogen.

The organic compounds identified from extractable-chromatographable material are primarily carboxylic acids that are readily biodegradable. Compounds found to be refractory and dominant in the effluents from activated sludge treatment are unsaturated, oxygenated, nitrogen heterocyclics, especially pyrrolidinones, piperidinones, cyclohexenopyrrolidinones, and cyclopentanopiperidinones.

Although this analysis represents a screening organic characterization of the retort water, certain practical problems are evident with conventional GC/MS analysis of retort water. Solvent extraction by methylene chloride is not efficient in recovering polar organic material, hence compounds of some classes are probably selectively neglected in the extraction procedure. In addition, compounds larger than molecular weight of 150-200 which may be present in the sample are unable to pass through the

capillary column to the GC for analysis. Additional work, with similar objectives of characterizing and identifying the organic material in these types of complex and varied solutions should include the development of more advanced and useful analytical methods.

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