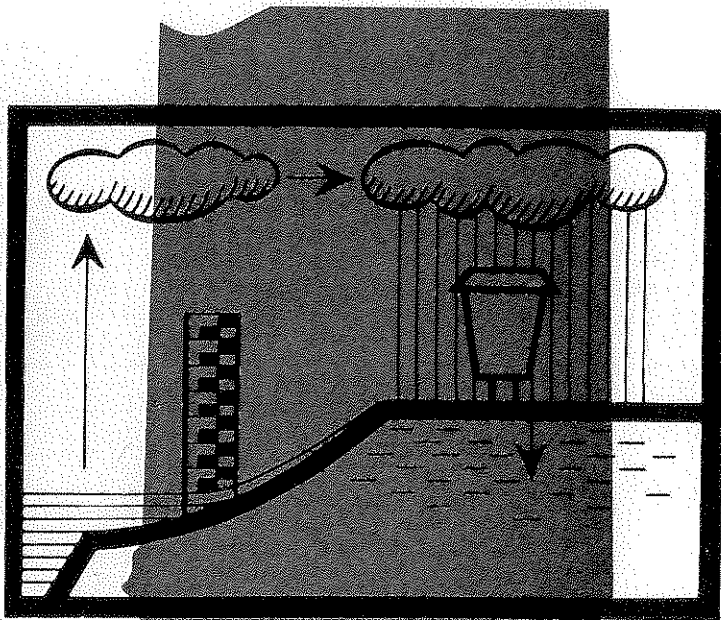


# EFFECT OF WET-AIR OXIDATION ON THE CHEMICAL COMPOSITION OF SEWAGE SLUDGES



by  
**L. E. Sommers**  
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**August 1976**



**PURDUE UNIVERSITY**  
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**WEST LAFAYETTE, INDIANA**

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A completion report of OWRT Project No. A-045-IND  
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SUMMARY

Sewage sludge samples were obtained from waste treatment streams in two sewage treatments before and after wet-air oxidation. Wet-air oxidation resulted in a 10 - 20% reduction in volatile solids. Concurrently, total N levels in the sludges decreased by >50% while P, Cu, Zn, Ni, Cd and Pb concentrations either increased or remained unchanged. From the standpoint of applying sewage sludge to agricultural land, inclusion of a wet-air oxidation unit in a treatment plant will decrease the desirability of utilizing sewage sludge as a fertilizer material.

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The expense involved with disposing of large volumes of water associated with non-dewatered sludges and the problems encountered in dewatering various sewage plant sludges has stimulated interest in evaluating methods to facilitate dewatering of sewage sludges. One of the processes being considered to aid sludge dewatering is pretreatment by wet-air oxidation. During this "low pressure" process, significant amounts of energy are consumed in maintaining the sludge at pressures of 240-260 psi and temperatures of 350-400<sup>o</sup>F (180-200<sup>o</sup>C) for a time period sufficient to cause partial oxidation of organic matter and alteration of sludge physical characteristics. In addition to consuming considerable amounts of energy, wet-air oxidation may alter the chemical properties of sewage sludge resulting in decreased amounts of plant nutrients. This will be of particular importance if application on agricultural land is being considered as an alternative for sludge disposal. Due to the lack of data concerning the chemical composition of sewage sludges before and after wet-air oxidation, this work was initiated to obtain information on the forms of N, P and metals (Cu, Zn, Ni, Cd and Pb) in sludges processed by wet-air oxidation at two waste treatment plants.

MATERIALS AND METHODS

Originally, it was anticipated that periodic samples would be obtained from the Terre Haute and Speedway, Indiana, treatment plants and subjected to chemical analysis; however, maintenance problems encountered during operation of the wet-air oxidation units precluded a systematic sampling approach. Thus, samples were obtained whenever the wet-air oxidation units were operating functionally. Samples of sludge and decant liquors were obtained before and after wet-air oxidation. All samples were placed in plastic



containers and maintained frozen during transport and storage. Immediately after the samples were thawed, they were subjected to the chemical analyses described below.

Suspended and volatile solids were determined gravimetrically by drying at 105°C for 16 hours and 650°C for 4 hours, respectively. Total N was determined by a modified semi-micro Kjeldahl procedure on both filtered and non-filtered samples<sup>1</sup>. Soluble and exchangeable ammonium was estimated by steam distillation of MgO-KCl treated samples followed by acidimetric titration of NH<sub>3</sub> as described by Bremner<sup>2</sup>. Total P was estimated after HNO<sub>3</sub>-HClO<sub>4</sub> digestion<sup>3</sup> by colorimetric determination of orthophosphate<sup>4</sup>. Metal analyses (Cd, Pb, Ni, Cu, and Zn) were performed by atomic absorption spectrophotometry (Varian A.A.-6) after digestion of sludge samples with HNO<sub>3</sub>-HClO<sub>4</sub><sup>3</sup>.

## RESULTS AND DISCUSSION

Operational considerations of wet-air oxidation units. The sewage treatment plants located in Speedway and Terre Haute, Indiana, have recently installed nearly identical wet-air oxidation units. The 1970 installation cost of the 39 gpm unit in Terre Haute was \$684,000. The Terre Haute Wastewater Treatment Plant has a 20 mgd primary treatment unit and a 24 mgd secondary activated sludge treatment unit. The plant currently receives an average daily flow of approximately 9 mgd. The Speedway Wastewater Treatment Plant, which utilizes an oxygen activated sludge process for secondary treatment, was designed for 7.5 mgd and receives a current average daily flow of approximately 4.5 mgd. The internal flow streams for these plants are shown in Figures 1 and 2.

The Terre Haute feed sludge has been anaerobically digested in excess of 21 days while the Speedway feed sludge is a mixture of primary sludge and waste oxygen activated sludge which has been detained a maximum of 7 days in a holding tank. Consequently, bicarbonate and sulfide levels in the two sludges are dissimilar, resulting in differences in operational problems encountered in wet-air oxidation. In a normally functioning anaerobic

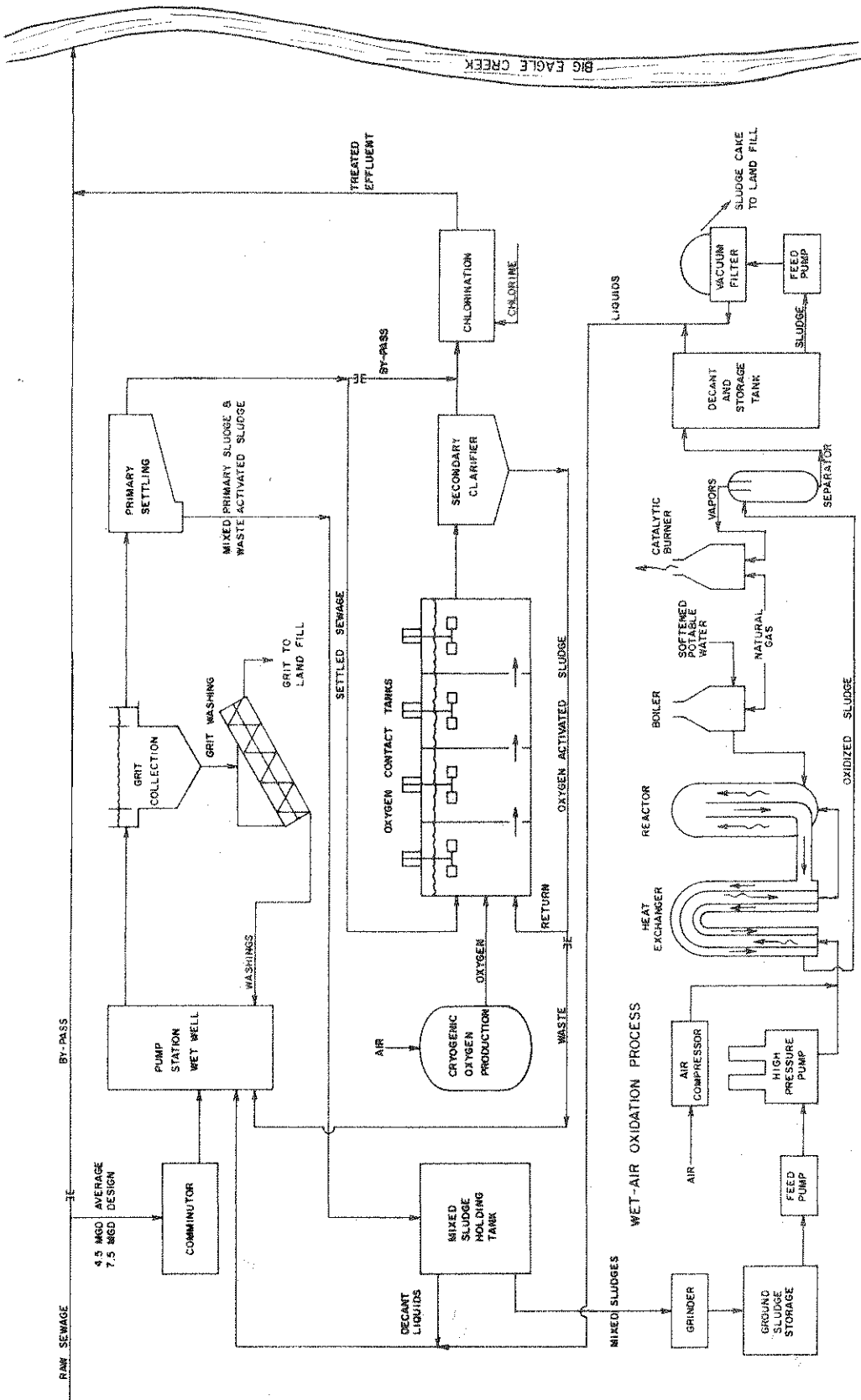


FIGURE 1. SPEEDWAY, INDIANA WASTEWATER TREATMENT PLANT FLOW DIAGRAM.

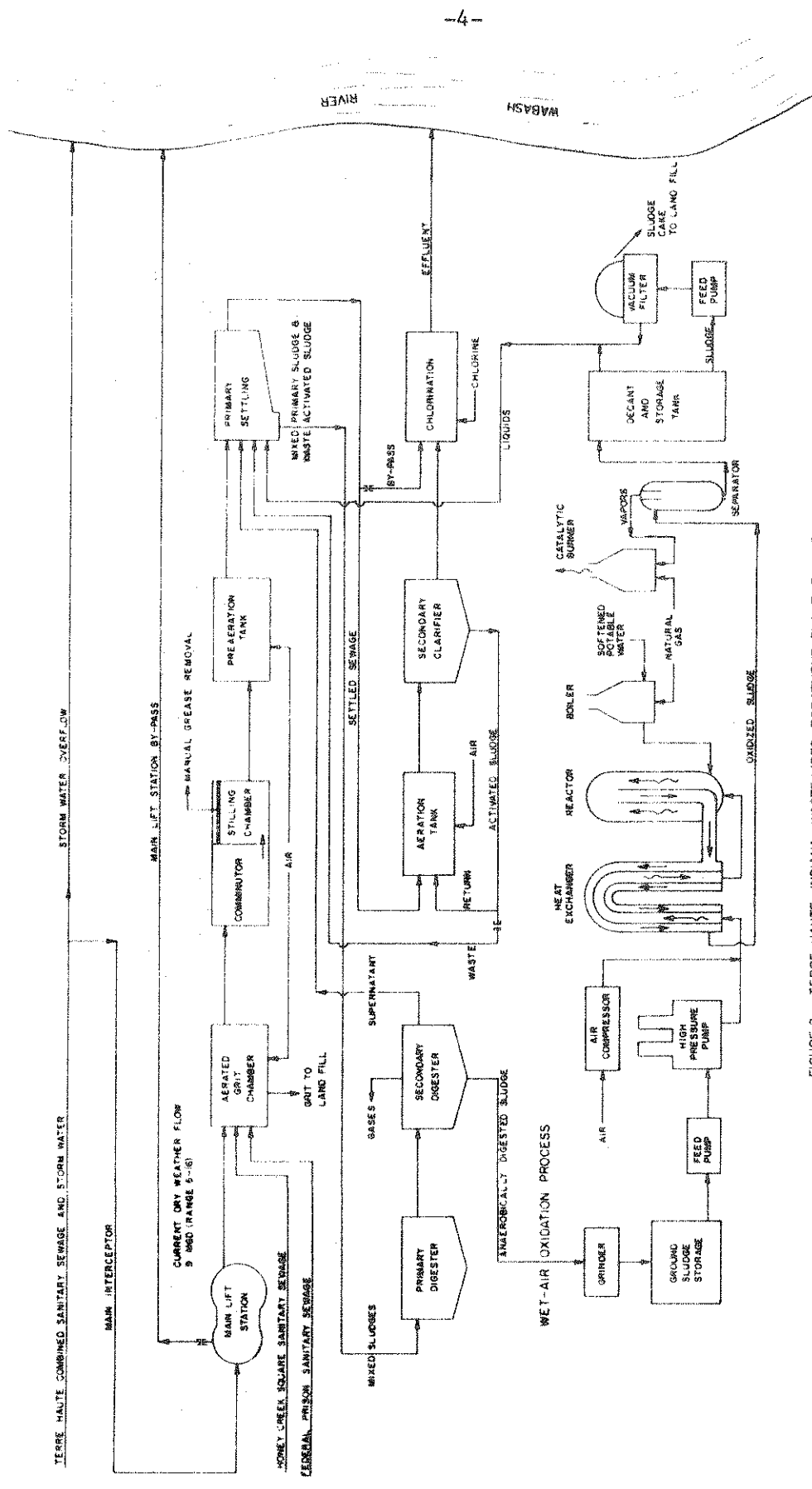


FIGURE 2. TERRE HAUTE, INDIANA WASTEWATER TREATMENT PLANT FLOW DIAGRAM

digester, bicarbonates can approach 5,000 mg/l and numerous sulfur compounds are converted to sulfides<sup>5</sup>. Bicarbonate levels in the Terre Haute digested sludge averages 3,500 mg/l (unpublished data). The bicarbonate and sulfide content of the Terre Haute anaerobic sludge caused rapid formation of a one inch thick scale in the heat exchangers (carbonate scale) and in the reactor vessel (sulfate scale). Selective formation of carbonate and sulfate scales results from the differential effect of temperature on the solubility of carbonate and sulfate salts, and from the oxidation of sulfides to sulfates in the process. Continual operational difficulties are encountered at the Terre Haute installation as a thick scale covers all internal surfaces including valves and sensor probes. The hot nitric acid wash used in cleaning has negligible effect on the sulfate scale, and thus, removal must be accomplished with a chipping hammer.

In contrast, the Speedway plant has not encountered severe operational difficulties because the sludge does not undergo complete anaerobic digestion in the preceding sludge storage tank. Therefore, elevated concentrations of bicarbonate and sulfide are not present in the feed sludge. Operationally, this is reflected by the formation of a one-eighth inch thick scale which is removed by an acid wash every four to five weeks.

Nitrogen and phosphorus content of sewage sludges. In general, the wet-air oxidation process results in increased settleability of sewage sludge solids since the majority of samples analyzed prior to wet-air oxidation contained solids contents ranging from 4-7% whereas post wet-air oxidation samples contained approximately 20% solids (Table 1). Thus, physicochemical properties of the sludge were altered by the high temperature-pressure treatment encountered during the wet-air oxidation process. The increased settleability is probably not a result of increased density since only 10-20% reductions in volatile solids were obtained during the wet-air oxidation process (Table 1). This data also indicates that the reduction in volatile solids differs for the two treatment plants. This may be due to the differences in the type of feed sludge and the efficiency of heat transfer for the two wet-air oxidation units. Variation in the feed sludge within each

Table 1. Effect of wet-air oxidation on suspended and volatile solids and forms of P in sewage sludge\*

Sample No.	Sample Type	Solids		Total P	Total P†		Soluble P†	
		Susp.	Volatile		Soluble	Particulate	Inorg.	Org.
				%		mg/l		
1	Pre	4.6	47.6	1.226	0.102(8)	1.125(92)	30.62(5)	16.18(3)
	Post	21.6	40.3	1.240	0.005(1)	1.235(99)	37.45(1)	<0.01(<1)
2	Pre	4.4	49.8	1.160	0.106(9)	1.054(91)	27.30(5)	19.45(4)
	Post	21.2	39.3	1.223	0.004(1)	1.219(99)	10.05(<1)	6.00(<1)
3	Pre	7.3	46.2	1.147	0.092(8)	1.055(92)	29.44(4)	37.06(5)
	Post	23.4	35.4	1.258	0.002(1)	1.257(99)	5.90(<1)	<0.01(<1)
4	Pre	6.2	44.6	1.088	0.028(3)	1.060(97)	3.78(<1)	13.88(2)
	Post	25.8	31.8	1.170	0.003(1)	1.166(99)	6.50(<1)	1.90(<1)
Avg.	Pre	5.6	47.1	1.155	0.082(7)	1.074(93)	22.78(4)	21.64(3) †
	Post	23.0	36.7	1.223	0.004(1)	1.219(99)	14.98(<1)	<0.01(<1)
				Speedway				
1	Pre	4.0	53.6	1.475	0.155(10)	1.320(90)	57.05(10)	4.75(<1)
	Post	17.8	39.5	1.892	0.004(1)	1.888(99)	8.85(<1)	1.50(<1)
2	Pre	2.7	59.0	1.645	0.187(10)	1.458(90)	42.74(10)	20.58(5)
	Post	16.2	43.2	2.628	0.018(1)	2.610(99)	66.15(2)	<0.01(<1)
3	Pre	3.8	59.0	1.543	0.118(8)	1.425(92)	20.20(4)	23.65(4)
	Post	29.8	26.3	2.456	0.009(1)	2.447(99)	14.60(<1)	12.50(<1)
Avg.	Pre	3.5	57.2	1.554	0.153(10)	1.401(90)	40.00(8)	15.48(3)
	Post	21.3	36.3	2.325	0.010(1)	2.315(99)	29.85(<1)	8.83(<1)

\*Data expressed as percentage of oven-dry solids or mg/l sludge liquid.  
 †Numbers in parenthesis are concentrations expressed as a percentage of total P.

wastewater treatment plant would reflect how well the preceding treatment units were functioning. Due to the difference in solids content of the original samples, it is essential that all data on the chemical composition of sludges before and after wet-air oxidation be expressed on an oven-dry solids basis. Soluble constituents were obtained by analysis of filtered sludge samples (i.e., all particulate matter had been removed) but are expressed on an oven-dry solids basis, in some cases, to facilitate comparisons.

The wet-air oxidation process resulted in decreased concentrations of soluble total P (Table 1). Soluble total P, on the average, decreased from 0.082 to 0.004% and from 0.153 to 0.010% for the Speedway and Terre Haute sludges, respectively, as a result of wet-air oxidation. The proportion of P present in particulate forms increased from approximately 90 to >99% after wet-air oxidation. The decrease in soluble organic P suggests that the soluble forms of P are more susceptible to oxidation at high temperature and high pressure than the particulate forms of P present in the sludge solids. Both increases and decreases in the concentration of soluble inorganic P resulted from wet-air oxidation. Concurrent with the decrease in soluble total P, sludges treated by wet-air oxidation were enriched in particulate forms of P. This occurred for samples obtained from both the Speedway and Terre Haute treatment plants. For the Terre Haute sludge, almost a 2-fold increase in particulate P occurred. The correlation between reductions in volatile solids, soluble total P, and soluble organic P indicates that soluble forms of P are more susceptible to alteration by wet-air oxidation. Even though particulate organic P may be oxidized by the process, the inorganic released may be retained by the sludge solids and therefore is not recovered as soluble inorganic P. The majority of P present in sludges is bound to particles (i.e., precipitates, sorption reaction products, etc.), and will be influenced to a lesser extent by the wet-air oxidation process. Organic matter in the original sludge tends to dilute the concentration of P and thus, particulate P will be enriched following wet-air oxidation.

In contrast to P, both soluble and particulate forms of N were decreased by wet-air oxidation (Table 2). On the average, samples obtained from both

Table 2. Effect of wet-air oxidation on forms of N in sewage sludge.\*

No.	Type	Total N		NH <sub>4</sub> -N		Organic N	
		Soluble	Particulate	Soluble	Particulate	Soluble	Particulate
				<u>Speedway</u>			
1	Pre	3.636	2.110(58)	1.121(31)	0.299(8)	0.400(11)	1.812(50)
	Post	1.458	0.916(63)	0.328(22)	<0.001(<1)	0.211(14)	0.926(64)
2	Pre	3.916	2.163(55)	1.353(34)	0.325(8)	0.394(10)	1.839(47)
	Post	1.495	0.943(63)	0.332(22)	0.001(<1)	0.218(15)	0.946(63)
3	Pre	3.279	2.148(65)	0.925(28)	0.224(7)	0.204(6)	1.924(59)
	Post	1.402	0.898(64)	0.360(26)	<0.001(<1)	0.142(10)	0.919(66)
4	Pre	3.073	2.058(67)	0.838(27)	0.284(9)	0.175(6)	1.774(58)
	Post	0.950	0.665(70)	0.163(17)	0.022(2)	0.120(13)	0.660(70)
Avg.	Pre	3.476	2.120(61)	1.059(30)	0.283(8)	0.293(8)	1.837(53)
	Post	1.326	0.856(65)	0.296(22)	0.012(<1)	0.173(13)	0.863(65)
				<u>Terre Haute</u>			
1	Pre	3.418	2.432(71)	0.919(27)	0.308(9)	0.066(2)	2.124(62)
	Post	2.086	1.700(81)	0.307(15)	0.053(3)	0.078(4)	1.692(81)
2	Pre	5.233	3.506(67)	1.533(29)	0.389(7)	0.192(4)	3.117(60)
	Post	1.997	1.489(75)	0.273(14)	0.055(3)	0.233(12)	1.434(72)
3	Pre	3.930	2.580(66)	1.211(31)	0.350(9)	0.134(3)	2.230(57)
	Post	1.242	0.855(69)	0.185(15)	<0.001(<1)	0.199(16)	0.860(69)
Avg.	Pre	4.194	2.839(68)	1.221(29)	0.349(8)	0.131(3)	2.490(59)
	Post	1.775	1.348(76)	0.255(14)	0.054(2)	0.170(10)	1.329(75)

\* Data expressed as % of oven-dry solids (for both particulate and soluble components). Numbers in parenthesis are concentrations expressed as a percentage of total N.

the Speedway and Terre Haute treatment plants exhibited greater than 50% reductions in soluble and particulate total N. However, the distribution of soluble and particulate total N was similar before and after oxidation. Even more pronounced reductions were obtained by wet-air oxidation with respect to total inorganic N (i.e.,  $\text{NH}_4^+$  and  $\text{NO}_3^-$ ). Significant amounts of both soluble and particulate  $\text{NH}_4\text{-N}$  (i.e.,  $\text{NH}_4^+$  bound to exchange sites contained in sewage sludge solids) were found in each feed sludge while following wet-air oxidation very small amounts of exchangeable  $\text{NH}_4^+$  were present in the sludges. As was found for P, significant decreases were found for soluble organic N in the Speedway treatment plant; however, increased soluble organic N was found in all of the wet-air oxidized sludge samples obtained from the Terre Haute plant. Increased soluble organic N was found for the Terre Haute samples, and it may be a reflection of the high temperature and pressure treatment converting insoluble, high molecular weight particulate organic N compounds originally present in the anaerobically digested sludge to water soluble forms of organic N. It should be noted that the actual concentration of organic N in the sludge supernatant may be increased by wet-air oxidation, resulting in an increased N load in the secondary treatment process if the decant liquors are recycled to the head of the treatment plant.

The significant reductions in total inorganic N following wet-air oxidation result from the fact that a relatively small percentage of the total inorganic N present in sewage sludge is associated with solids. Therefore, the greater settleability of the oxidized sludges causes a smaller proportion of the total inorganic N to be associated with the solid phase while the decant liquor and vacuum filter filtrate contain the majority of the inorganic N that originally entered the wet-air oxidation unit. Mass flow data were not available to determine what portion of the nitrogen compounds may have escaped as gaseous species. Inorganic nitrogen and other constituents contained in process liquids are commonly returned to the head of the treatment plant and subjected to further treatment.

Metal content of sewage sludges. Table 3 presents data on the metal concentrations that are found in sewage sludges before and after wet-air oxidation. In general, there was an increasing concentration of water soluble Cu, Zn, and Ni as a result of wet-air oxidation. Water soluble Cd



Table 3. Effect of wet-air oxidation on Cu, Zn, Cd, Pb and Ni in sewage sludge\*

Sample No.	Sample Type	Cu		Zn		Ni		Total Cd mg/kg	Total Pb mg/kg
		Soluble mg/l	Total mg/kg	Soluble mg/l	Total mg/kg	Soluble mg/l	Total mg/kg		
1	Pre	0.32	1262	0.31	2264	0.25	78	12.4	479
	Post	0.52	1169	0.61	2187	0.30	70	12.1	500
2	Pre	0.30	1156	0.26	2100	0.15	79	12.7	475
	Post	0.65	1088	0.71	1991	0.35	68	11.6	488
3	Pre	0.45	1096	0.36	1991	0.25	67	11.4	484
	Post	0.20	1088	0.24	2067	0.40	76	11.8	514
4	Pre	0.40	844	0.44	1630	0.10	58	9.2	368
	Post	1.30	700	0.61	1652	0.25	64	9.6	384
Avg.	Pre	0.37	1090	0.34	1996	0.19	70	11.4	451
	Post	0.67	1011	0.54	1974	0.32	70	11.3	471
Speedway									
Terre Haute									
1	Pre	0.10	774	0.04	1890	0.42	972	51.1	780
	Post	0.10	863	0.11	2319	1.85	1064	51.3	990
2	Pre	0.15	570	0.08	1666	0.75	867	65.6	712
	Post	0.10	1080	0.11	3051	5.72	1124	111.5	978
3	Pre	0.10	603	0.06	1886	6.05	894	58.4	566
	Post	0.56	612	0.51	2122	7.72	1006	68.8	966
Avg.	Pre	0.12	649	0.06	1814	2.41	911	58.4	686
	Post	0.25	852	0.24	2497	5.11	1064	77.2	978

\*Data expressed as mg/kg oven-dry solids or mg/l sludge liquid.

and Pb were non-detectable in all samples. This data also indicates that sludges from different sources will support different concentrations of soluble metals. For example, water soluble Cu and Zn are lower in the Terre Haute sludge than in the Speedway sludge, while the Terre Haute sludge supported a higher concentration of water soluble Ni. However, concentrations of water soluble metals were increased in all cases by wet-air oxidation. There was very little change in total metal concentrations as a result of wet-air oxidation in the Speedway samples. However, in samples obtained from the Terre Haute treatment plant, increased concentration of metals were normally found in the wet-air oxidized sludge, especially for Pb, Cd, Zn and Cu. As shown for P, the increased concentration of metals corresponds to a decrease in volatile solids. Another factor that should be noted when comparing total metal concentrations in sludge is the variation that occurs with time in the same treatment plant. The variability in sludge composition found in this study is supported by data obtained previously on sewage sludge composition<sup>6</sup>. With respect to the total concentrations of metals present, the Speedway sludge contains relatively low concentrations of all metals while the Terre Haute sludge is enriched with Cd, which may be a major concern when considering land application of sludges.

Composition of decant liquors. The properties of decant liquors from wet-air oxidation units are presented in Table 4. Unfortunately, an insufficient number of samples were obtained at several sampling times to obtain valid estimates of the chemical composition of decant liquors. However, the data gives an indication of the N, P and metal concentrations that are commonly found in decant materials. The data should also indicate the quality of the vacuum filter filtrate, which was not sampled in this study. The relatively high concentration of volatile solids in the decant solutions indicates the presence of low molecular weight organic compounds. Soluble organic N in decant liquors is in all cases greater than the concentrations that were found in the sludges before and after wet-air oxidation. It is also apparent that the majority of the  $\text{NH}_4\text{-N}$  present in the decant liquors is soluble rather than being bound to solids as an exchangeable ion. The

Table 4. Properties of decant liquors from wet-air oxidation units.

Parameter	Units*	Speedway <sup>+</sup> (No. 4)	Terre Haute <sup>+</sup>		
			No. 2	No. 3a	No. 3b
Solids	%	5.5	0.7	10.5	2.2
Volatile solids	%	38.2	79.4	43.6	55.8
NH <sub>4</sub> - soluble	mg/l	218.0	307.0	612	549
- particulate	mg/kg	17.0	< 0.1	114	< 0.1
Organic N - soluble	mg/l	442.0	515	710	738
Total N - soluble	mg/l	663.0	827	1330	1300
- particulate	%	0.7	1.1	1.4	1.3
Inorganic P - soluble	mg/l	16.8	-	-	-
Total P - soluble	mg/l	20.3	31.0	46.1	36.4
- particulate	%	1.1	0.6	2.6	2.1
Cu - soluble	mg/l	5.4	0.4	0.1	0.9
- total	mg/kg	755.0	297	1158	806
Zn - soluble	mg/l	0.9	0.5	0.5	0.8
- total	mg/kg	1597.0	946	3434	2621
Ni - soluble	mg/l	0.2	6.2	11.6	8.3
- total	mg/kg	91.0	1076	1291	1260
Cd - total	mg/kg	10.3	26.8	114	82.5
Pb - total	mg/kg	380.0	592	879	841

\* % and mg/kg are data expressed on an oven-dry solids basis.

+ Numbers in parenthesis refer to sample numbers used in previous tables.

solids content of decant liquors is variable and a function of the settling time after the material has entered the settling tank. Thus, considerable variation is shown for the solids content in the decant liquors.

Although this study was not designed to evaluate the effect on effluent quality, comments are appropriate on the influence of the wet-air oxidation process on the quality of effluent discharged from plants utilizing wet-air oxidation. Wet-air oxidation process liquids represent a system recycle load that was not previously present. The data collected indicates that significant quantities of nitrogenous compounds will be recycled to the head of the treatment plant while P and metals will be concentrated in the treated sludge. Unless nitrification and denitrification processes are utilized, the result will be increased ammonium and/or nitrate levels in the effluent. The heavy metals and P content of decant liquors are not greatly different from the processed sludge; however, the return of these constituents will result in increased levels of these constituents in waste treatment units and they will either pass from the plant with the effluent or be recycled back to the sludge unless the liquors are treated separately. Process data on the Speedway plant shows that on an average annual basis, the wet-air oxidation liquors constitute approximately 0.4 to 0.5 percent of the average wastewater flow. Operation of the Terre Haute unit is infrequent and thus, average data were unavailable.

#### SUMMARY

The data obtained have implications with respect to land application of sewage sludge. The annual rate of sludge application on agricultural land will be controlled by two parameters: 1) the N content of the sludge and the N requirement of the crop being grown, and 2) the annual loading rate for Cd and the total amount of metals present in the sludge. With respect to N, the wet-air oxidation process lowers the desirability of utilizing sewage sludge for the production of agricultural crops. In general, greater than 50% reductions in total N were obtained following wet-air oxidation; however,

there was essentially no change or a slight increase in the concentration of metals that are contained in the wet-air oxidized sludges. Thus, in order to apply sufficient wet-air oxidized sludge to satisfy the N requirement for a growing crop, greater amounts of sludge will be needed per annum resulting in increased annual application of metals. Thus, the life-time of the disposal site will be lessened by using wet-air oxidized sludges. In contrast to the situation with N, wet-air oxidation tended to increase the concentrations of P present in sludge and thus greater amounts of P would be added to soils in oxidized sludges. If sludge application is based on N, P in excess of crop demand will be applied to soils.

In summary, wet-air oxidation decreases N, and either does not affect or increases P and metal contents of sewage sludges. In view of the maintenance problems encountered in operating the oxidation units, judgement is required to evaluate the proper sludge conditioning process because not all sludges are suitable for wet-air oxidation. Furthermore, if land application is being considered as an alternative for disposal of sludge, the desirability of utilizing a wet-air oxidation process is doubtful.

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