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Characterization of Particulate Emissions from Municipal Wastewater Sludge Incinerators

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■ Particulate emissions from a group of municipal sludge incinerators, three with multiple-hearth furnaces and one with a fluidized-bed furnace, were characterized. Three plants operated at or near autogenous burning conditions. Chemical element composition was determined for total and sized emission samples by X-ray fluorescence analysis. Objectives of the investigation were to obtain specific elemental emission factors and to provide source inventories and source signatures, especially in terms of particle size, to assist the development and evaluation of source apportionment models. Considerable enrichment of several elements (S, V, Cu, Zn, Cd, Sn, Pb) in the particulate emissions compared to their content in the sludge feed was observed. The largest average enrichment ratios were found with cadmium (31), zinc (13), lead (9), and sulfur (8).

Introduction

With the trend in the production of municipal wastewater sludge on a definite increase, an attendant increase in the use of incineration in the management of sludge disposal is expected. A characterization study has been conducted on the particulate emissions from the stacks of a group of municipal sludge incinerators, three with multiple-hearth furnaces and one with a fluidized-bed furnace. One purpose of the investigation was to provide information on the concentration of chemical elements, especially heavy metals such as cadmium and lead, in the emissions. A related objective was to obtain source emission factors and source signatures to assist in the development and validation of source apportionment models.

The chemical compositions of municipal sludges vary with location and time because the sludges result from a diversity of manufacturing activities as well as human excreta and food residues. Thus, as demonstrated in the analyses of Furr et al. (*1*) on the municipal sludge from 16 cities, there is no typical sludge. Enrichment of some elements, notably Cd and Pb, in the emissions from incineration compared to the concentration in the sludge feed can occur. Therefore, predictions of the stack emissions based on the content of the sludge being burned are likely to be erroneous.

These investigations involved the collection of representative emission samples for particulate mass emission determination, for chemical characterization, and for particle size determinations of total mass emissions and individual chemical element emissions. Sulfur dioxide and sulfuric acid emissions were determined. The gaseous concentration of hydrocarbons and nitrogen oxides were monitored, and a few samples were collected to determine the emissions of high molecular weight organic compounds. At each plant process, samples of the feed sludge were collected and analyzed. Information on the four incinerator facilities during the test periods are shown in Table I.

The sludge was preconditioned thermally with the hot exhaust gases from the furnaces to produce a higher solids sludge cake for better burning. At the multiple-hearth incinerator O, this process was carried to the point of positive heat balance, autogenous burning. Some supplemental fuel was burned at all of the sites. At incinerator O only a total of 16 gal of fuel oil was burned, and that only during the last two of the 29-h sampling periods for shutdown purposes. The remainder of the time the sludge was autogenously combusted. At incinerator P an average of 40 ft^3/h of natural gas was burned. The supplemental fuel oil at incinerator Q ranged from 0 to 64 gal/h and averaged 17 gal/h over the 3-day test period. Incinerator R was not designed for autogenous combustion and never reached this condition. An average of 40 gal/h of fuel oil was burned.

Experimental Section

Sampling Procedures. A standard U.S. Environmental Protection Agency (EPA) Method 5 sampling train (*2*) was used to collect particulate samples for mass emission rate determinations. The impingers were adapted to determine gaseous sulfur oxides by EPA Method 8 (*3*). The first impinger contained 150 mL of 80% isopropanol and was followed by a high purity glass wool plug in the U-tube between the first and second impinger. The second and third impingers contained 100 mL each of 3% hydrogen peroxide solution and the fourth impinger was filled with approximately 400 g of indicating silica gel. Prior to sampling, a velocity profile of the duct at the sampling location was determined through transversing. All Method 5 and Method 8 samples were collected with a 5-ft heated Pyrex-lined probe at a single point having the average velocity of the flue gas within the duct.

Samples used for chemical characterization by X-ray fluorescence analysis (XRF) were collected with a modified Method 5 train in which the conventional sample box, filter holder, and glass impingers were replaced by an EPA-designed heated sample box that housed a stainless steel

Table I. Furnace Types, Control Systems, and Sludge Loadings of Tested Incinerators

site designation	furnace type	control equipment	av load of unit tested, kg/h (dry)	sludge moisture, %
O	seven-chambered multiple-hearth	wet, tray-type scrubber	1940	63.5
P	eight-chambered multiple-hearth	wet, tray-type scrubber	1740	49.3
Q	fluidized-bed	wet, tray-type scrubber	839	66.2
R	seven-chambered multiple-hearth	single-pass cyclone scrubber	1890	71.3

Table II. Elemental Composition (%) of Municipal Wastewater Sludge

	site O		site P		site Q		site R		
element	av	sd	av	sd	av	sd	av	sd	av content 16 cities[a]
Na	1.0	0.09	0.7	0.11	<0.6		<0.2		0.44
Mg	1.8	0.08	1.0	0.17	0.87	0.13	0.92	0.03	0.60
Al	3.4	0.28	3.6	0.47	4.0	0.23	5.2	0.14	1.83
Si	10.1	0.46	13.0	3.3	17.0	2.7	12.0	0.34	
P	6.4	0.44	4.4	0.22	4.1	0.13	3.7	0.12	1.56
S	0.93	0.15	1.3	0.41	0.82	0.46	0.47	0.06	
Cl	0.45	0.01	0.08	0.18	<0.5		0.68	0.19	0.38
K	1.3	0.09	1.1	0.24	1.2	0.15	1.1	0.02	1.22
Ca	17.0	0.62	6.2	1.2	7.5	1.0	9.8	0.15	3.62
Ti	0.84	0.06	0.76	0.05	0.77	0.04	1.18	0.04	0.23
V	0.03	0.01	0.04	0.01	0.03	0.01	<0.01		0.004
Cr	0.22	0.02	0.50	0.27	0.22	0.24	0.38	0.02	0.14
Mn	0.70	0.11	0.23	0.11	0.34	0.12	0.11	0.008	0.019
Fe	6.7	1.0	17.0	7.9	7.6	6.4	5.4	0.10	3.06
Co	0.01	0.003	0.03	0.02	0.01	0.02	0.01	0.002	0.001
Ni	0.03	0.01	0.14	0.08	0.06	0.07	0.06	0.008	
Cu	0.12	0.02	0.75	0.43	0.27	0.37	0.44	0.02	0.13
Zn	0.30	0.04	1.1	0.08	1.0	0.07	1.4	0.03	0.21
As	<0.3		<0.3		<0.03		<0.03		0.0014
Se	<0.04		<0.04		<0.04		<0.04		0.0003
Br	<0.05		<0.05		<0.05		<0.05		0.005
Cd	0.02	0.005	0.04	0.05	0.08	0.04	0.03	0.005	0.010
Sn	0.30	0.02	0.25	0.03	0.25	0.03	0.19	0.006	0.022
Sb	0.03		0.02	0.004	0.01	0.001	0.01	0.001	0.001
Ba	0.25	0.02	0.41	0.11	0.45	0.04	0.27	0.01	0.06
Pb	0.34	0.13	0.48	0.18	0.59	0.25	0.23	0.03	0.18

[a] Reference 1.

filter holder for 47-mm filters. The particulate characterization samples were collected in sets consisting of two Gelman A glass fiber filters and two Millipore AA, one Nuclepore 0.8 μm, and six Teflon 0.2 μm filters. Sampling periods ranged from 15 s to 10 min to provide a variety of loading on the filters. In all cases, an attempt was made to sample isokinetically. The Millipore and Teflon filters were analyzed by XRF.

University of Washington Mark III cascade impactors were used to collect samples for particle size distribution measurements of total mass and individual chemical element. The impactor samples were taken in-stack at the sampling point of average gas velocity used for the characterization sampling. Samples were collected isokinetically with a sampling rate between 0.5 and 0.75 ft^3/min through the impactor.

Composite preburn sludge samples and postburn ash were collected at all incinerators except the fluidized-bed unit, where only sludge samples were taken. The sludge samples were heated to 600 °C for 30 m to remove the volatile content before their chemical analysis.

Each site was monitored during the testing period on a continuous basis for nitrogen oxides and hydrocarbon emissions. A heated Teflon line was used to deliver the samples to a Thermo Electron Model 44 duel Chamber single detector monitor for nitrogen oxides and to a Scott Model 166 total hydrocarbon analyzer with a single ionization detector.

Analytical Methods. Elemental contents of the emission characterization, sludge, and ash samples were determined with a Siemens MRS-3 multichannel wavelength dispersive X-ray fluorescence spectrometer with 15 fixed-wavelength monochromators and a scanning channel to analyze for 11 additional elements. Requirements of this system, which has been optimized for the analysis of aerosol samples (*4*), are light, uniform deposits on low-mass substrates. The characterization samples collected on Teflon, Millipore, and Nuclepore filters were analyzed directly. Samples that were collected with the cascade impactors resulted in small piles on the impactor stages that could not be analyzed directly. The material on each stage was suspended in liquid and subsequently redeposited on polycarbonate films (poreless Nuclepore) (*5*). Bulk samples such as the ashed process feed sludge and the thick deposits on glass fiber filters were formed into thin carbowax films suitable for XRF analysis. The samples were ground, mixed with carbowax and solvent in an agate mortar and pestle until the solvent dried, and then transferred to a hydraulic press where a flat specimen was formed under 10 tons of force. Solutions such as those obtained from the backup impinger of Method 5 were analyzed by procedures recently developed in our laboratory (*6*). A Collision nebulizer was used to generate liquid aerosols from the analytic solution. Adequate dilution was added to dry the particles. A filter in the air stream after a small mixing chamber was used to collect thin, uniform deposits suitable for analysis.

Results and Discussion

Sludge Content. The composition of the ashed sludge

Table III. Change of Elemental Composition (%) of Sludge from Incinerator O with Heating

element	temp, °C						normalized enrichment[a]
	100	200	400	600	800	1000	
Mg	0.53	0.60	1.5	1.6	1.8	1.9	0.98
Al	0.86	0.86	2.4	2.4	2.8	2.9	0.89
Si	2.5	2.4	7.7	7.4	8.8	9.2	0.94
P	1.6	1.6	4.5	4.5	4.9	5.3	0.89
S	0.54	0.59	0.88	0.86	0.91	0.64	0.50
K	0.33	0.32	1.0	1.0	1.1	1.2	1.00
Ca	4.1	4.1	12.6	12.2	13.7	14.8	0.93
Ti	0.21	0.19	0.60	0.60	0.64	0.68	0.91
Cr	0.07	0.11	0.23	0.23	0.24	0.20	1.04
Mn	0.22	0.18	0.60	0.58	0.66	0.66	0.84
Fe	1.9	1.8	6.1	6.4	6.7	7.2	1.07
Co				0.01	0.01	0.01	
Ni			0.03	0.04	0.04	0.04	
Cu	0.04		0.11	0.13	0.13	0.13	1.09
Zn	0.09	0.08	0.29	0.31	0.33	0.32	0.97
Cd			0.01	0.02	0.02	0.02	
Sn	0.09	0.09	0.24	0.24	0.28	0.27	0.85
Ba	0.06	0.05	0.19	0.18	0.21	0.20	0.95
Pb			0.24	0.24	0.29	0.15	

[a] Enrichment at 600 °C relative to potassium (% element/% potassium) at 600 °C divided by (% element/% potassium) at 100 °C.

feed material is shown in Table II for the elements determined by XRF. The values are the averages for four to six composite samples taken during the 2–3-day testing periods at each incinerator. The relative standard deviation was generally less than 10% for the more abundant elements (concentration greater than 1%). For comparison, the average content of sludge from 16 cities as determined by Furr et al. (*1*) is also listed in Table II. To determine whether there were significant volatilization losses of the analyzed elements during the ashing of sludge samples at 600 °C, we heated a group of samples for 45 min at the temperatures indicated in Table III and analyzed them by XRF. The concentration of each element at 600 °C was compared to its concentration in the unwashed (100 °C dried) sludge sample. The concentrations were normalized to that of a nonvolatile element (potassium) at the two temperatures. The results from samples taken at incinerator O, shown in the last column of Table III, indicate that only sulfur was significantly changed relative to potassium. Similar results were found with samples from incinerator P.

The nonvolatile elemental content of the postburn bottom ash samples from the three multiple-hearth furnaces was essentially the same as found in ashed sludge samples from those sites. No bottom ash was available at the fluidized-bed incinerator as the ash, which was less dense than the sand of the bed, was continuously swept out at the top of the reactor. Greenberg et al. (*7*) have recently reported an investigation on another fluidized-bed incinerator in which they examined the composition of the ash collected at the bottom of the scrubber. For most elements, they found a ratio of concentration in the scrubber bottom ash to concentration in sludge of less than 1.0. This was attributed to loss by vaporizing out of the sludge or dissolving in the scrubber solution.

Elemental Emissions. The mean concentrations of the elements determined by XRF in the emissions from the four plants are shown in Table IV. The relative standard deviation of the samples analyzed (between 45 and 95 samples per site) to obtain each of these concentrations averaged 33%, 72%, 32%, and 68% for O, P, Q, and R, respectively. The greater variability of P and R reflect the wider range of operating conditions, i.e., the rates of feed and stack gas flow during the testing periods. It is immediately apparent that some of the elements such as zinc, cadmium, and lead were higher in the emissions than would have been expected based solely on their concentration in the sludge. Table V lists the average composition of the emission sample from each incinerator in per cent of the total mass. The concentrations of lead in the emissions have increased to about 3% from a mean concentration of 0.5% in the sludges. Enrichment factors, calculated by dividing the concentration in the sludge, are listed in Table VI. Conditions causing enrichment are volatility and association of the volatile elements with the extremely fine particles that are not efficiently removed by scrubbers.

Total Particulate Mass and Sulfur Oxides. The emission rates and concentrations of particulate mass, sulfur dioxide, and sulfuric acid were obtained from the Method 5 and Method 8 tests run after the scrubber outlets. The emissions shown in Table VII are the mean values obtained from running six to eight tests at each incinerator. Lowest particulate emissions were observed at the fluidized-bed incinerator, Q. The higher sulfur oxide emissions from incinerator R were presumably attributable to the supplementary fuel oil that was burned to maintain combustion.

Nitrogen Oxides and Hydrocarbons. The results of monitoring oxides of nitrogen and total hydrocarbons are shown in Table VIII. The sample values are 20-min averages of strip chart recordings; the averages represent the mean of all the 20-min averages. The average concentrations are similar at incinerator O, P, and Q, but the nitrogen oxides are much lower at R. No measurement of hydrocarbons was made at R.

Particle Size Distribution. The average mass median diameters obtained from the particle size distribution measurements were 0.28, 0.30, 1.1, and 0.85 μm at incinerators O, P, Q, and R, respectively. Individual size distributions for several elements as well as the total mass distribution are shown on Figure 1. For convenience, the largest and smallest size fractions have been assigned finite values. The zinc distribution (1A), predominantly submicrometer particles, was characteristic of the sulfur, cadmium, lead, and other elements which exhibited enrichment in the emission probably because of their volatility in the furnace. Phosphorus (1B) exhibited a group of midrange particles around 2 μm. Iron was distributed through all sizes with a significant fraction of large particles

Table IV. Mean Concentrations of Elemental Emission ($\mu g/m^3$)[a] from Municipal Sludge Incinerators

	site O		site P		site Q		site R	
element	av	sd	av	sd	av	sd	av	sd
Na	350	110	290	230	100	50	240	170
Mg	90	32	440	550	51	18	21	6
Al	128	47	1570	1980	160	48	37	20
Si	590	100	4180	4826	270	80	620	290
P	590	120	1730	1890	210	69	570	350
S	660	300	930	410	730	270	2610	910
Cl	230	90	130	110	47	65	1990	2280
K	210	90	460	500	54	22	120	110
Ca	780	170	2620	2660	440	150	160	88
Ti	28	12	450	460	33	10	16	21
V	~35		38	14	9	2	<6	
Cr	97	39	480	330	14	8	230	106
Mn	27	13	82	30	23	8	10	1
Fe	370	80	7070	8170	228	71	230	145
Co	<6		25	8	<6		<6	
Ni	<22		125	121	<22		<22	
Cu	85	29	810	690	14	4	520	310
Zn	810	220	1840	1240	87	28	1830	1650
As	<29		<29		<29		<29	
Se	~45		20	3	18	17	26	1
Br	~49		57	19	39	17	170	140
Cd	42	15	34	15	7	2	1890	1410
Sn	180	51	1230	450	30	9	790	640
Sb	6	2	23	7	<2		43	40
Ba	10	4	290	280	20	7	14	15
Pb	510	190	1170	530	114	65	2140	1880

[a] Corrected to normal temperature and pressure (20 °C, 1 atm).

Table V. Mean Composition (% of Total Mass) of Elements in Particulate Emissions from Municipal Sludge Incinerators

	site			
element	O	P	Q	R
Na	2.76	0.57	1.96	0.34
Mg	0.71	0.86	1.01	0.03
Al	1.02	3.05	3.07	0.05
Si	4.69	8.13	5.33	0.91
P	4.69	3.36	4.14	0.84
S	5.25	1.80	14.42	3.83
Cl	1.80	0.26	0.93	2.91
K	1.71	0.89	1.07	0.18
Ca	6.20	5.09	8.76	0.23
Ti	0.22	0.88	0.65	0.02
V	0.28	0.07	0.18	
Cr	0.77	0.94	0.28	0.33
Mn	0.21	0.16	0.46	0.01
Fe	2.91	13.75	4.52	0.34
Co		0.05		
Ni		0.24		
Cu		1.58	0.28	0.77
Zn	6.44	3.58	1.72	2.69
Se		0.04	0.36	0.04
Br		0.11	0.77	0.26
Cd	0.33	0.07	0.13	2.77
Sn	1.42	2.39	0.59	1.16
Sb	0.05	0.05		0.06
Ba	0.08	0.56	0.40	0.02
Pb	4.07	2.27	2.26	3.14

evident even in the controlled (post scrubber) emissions. Only a very small fraction of the total mass (1D) was greater than 2 μm.

At incinerator R particle size measurements were also made before the scrubber. Figure 2 shows the distribution at the scrubber inlet (2C) where large particles were preponderant, but a second mode occurred near 2 μm and at the scrubber outlet (2D), where the mass concentration of the smaller particles predominated because of more efficient removal of the larger particles by the scrubber. The

Table VI. Enrichment Ratios of Elements in Emissions Relative to Content in Sludge

	incinerator				
element	O	P	Q	R	av enrichment
Na	2.76	0.81			
Mg	0.42	0.86	1.16	0.03	0.38
Al	0.30	0.85	0.78	0.01	0.49
Si	0.46	0.63	0.31	0.08	0.37
P	0.73	0.76	1.01	0.23	0.68
S	5.64	1.38	17.6	8.14	8.19
Cl	4.00	0.63		4.28	2.97
K	1.32	0.81	0.89	0.16	0.80
Ca	0.36	0.82	1.16	0.023	0.59
Ti	0.26	1.16	0.84	0.02	0.62
V	9.33	1.75	6.0		5.69
Cr	3.50	1.88	1.27	0.87	1.88
Mn	0.30	0.70	1.35	0.10	0.61
Fe	0.43	0.81	0.59	0.06	0.54
Co		1.67			
Ni		1.71			
Cu	5.67	2.11	7.0	1.75	4.13
Zn	21.47	3.25	28.7	1.89	13.83
Br			7.7		
Cd	16.50	7.0	1.6	98.9	31.
Sn	4.73	9.56	2.36	6.10	5.68
Sb	1.67	2.50		4.6	2.92
Ba	0.32	1.36	0.89	0.07	0.66
Pb	11.97	4.70	3.83	13.65	8.53

Table VII. Emission Rates and Concentrations of Total Particulate Mass and Sulfur Oxide Gases

incinerator	particulate mass		SO_2		H_2SO_4	
	kg/h	mg/m^3	kg/h	ppm	kg/h	ppm
O	0.89	13.7	0.07	0.4	0.10	0.4
P	2.98	103.2	0.16	2.1	0.15	1.3
Q	0.094	9.4	0.30	11.1	0.022	0.6
R	2.13	199.6	3.34	92.4	0.31	7.0

[a] Concentrations corrected to normal temperature and pressure (20 °C, 1 atm).

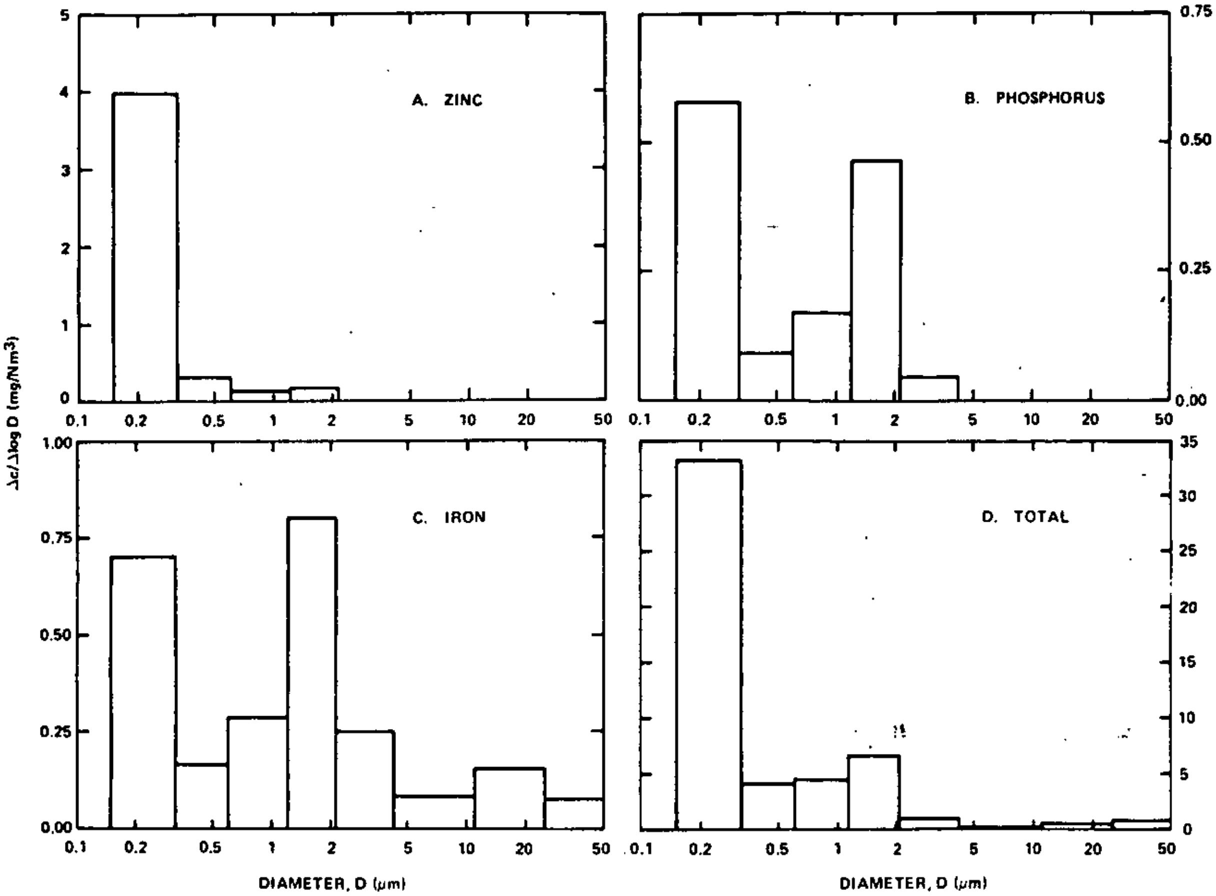

Figure 1. Particle size distribution of zinc, phosphorus, iron, and total particulate emissions from incinerator O.

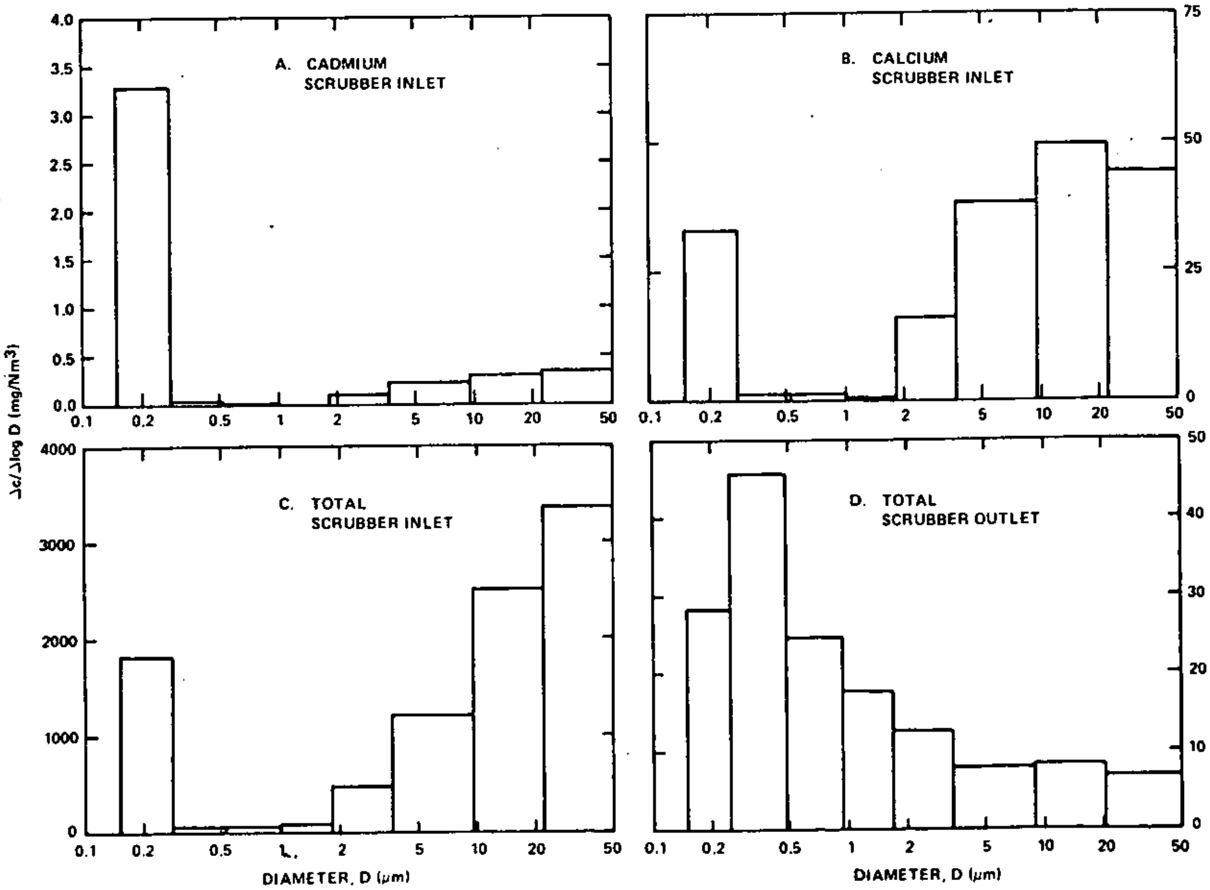

Figure 2. Particle size distribution of cadmium, calcium, and total particulate mass before the scrubber and total particulate emission after the scrubber at incinerator R.

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Table VIII. Concentration of Nitrogen Oxide and Hydrocarbon (ppm) in Sludge Incinerator Emissions

incinerator	NO		NO_2		hydrocarbons,
	range	av	range	av	range
O	8.8-105.8	31.4	3.2-61.3	22.7	6-7.2
P	18.9-139.9	102.5	0-63.5	26.2	3-6.4
Q	15.1-153.6	75.2	0-74.5	33.5	6.2-11.1
R	3.0-16.5	9.7	0-4.7	2.2	not monitored

contrast in the distribution of a more volatile element such as cadmium (2A) with that of calcium (2B) is evident in these prescrubber measurements.

Conclusions

The emissions from four municipal wastewater sludge incinerators, three multiple-hearth and one fluidized-bed, were characterized. The total particulate emissions, particle size distribution, sulfur oxides, elemental content, and nitrogen oxides were measured. The elemental composition of the feed stock sludge and the bottom ash was also determined. The more volatile elements, Cd, Pb, and Zn, were enriched in the emissions. Cd had the highest average enrichment ratio with a 31-fold increase in the emissions. The average mass median diameters for the emitted particles at all sites were small, ranging from 0.28 to 1.1 μm. Very few particles were larger than 2 μm. The volatile elements were found predominantly in the submicron particles.

The enrichment of Cd (especially relative to that of Zn) is much greater at incinerator R than at the other facilities. A contributing factor to this higher enrichment is the greater predominance of Cd in small particles and the lower efficiency of the control device. Incinerator R has a wet, single-pass scrubber system that is considerably less efficient for submicron particles than the scrubbers at the other incinerators. Elemental particle size measurements at incinerator R before the scrubber inlet showed that 72% of the Cd (Figure 2A) was associated with particles less than 1 μm. Only 28% of the Zn was associated with this size range.

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Removal of Trace Chlorinated Organic Compounds by Activated Carbon and Fixed-Film Bacteria

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■ A comparison was made between the removal of chlorinated benzenes and aliphatics in a granular activated carbon (GAC) column with microbial activity vs. a control column (BC) with only bacterial growth. A solution containing between 10 and 30 μg/L of each chlorinated organic compound and 1.39 mg/L sodium acetate was continuously applied to both columns under aerobic conditions at a 60-min empty-bed detention time for 2 years. The GAC column initially removed all the compounds with 95–98% efficiency. Complete breakthrough of the chlorinated aliphatics eventually occurred, and these compounds were not biodegraded in either column. Effluent concentrations of the chlorinated benzenes in the GAC column did not change significantly during the 2 years of operation. Several chlorinated benzenes were biodegraded in both GAC and BC columns after a suitable acclimation period, and carbon-14 tracer experiments confirmed removal by a biological mechanism in both columns with complete mineralization to inorganic end products. The combination of biodegradation and adsorption gave stability and reliability to the GAC performance.

Introduction

Microbial activity is reported to have beneficial effects on the performance of granular activated carbon (GAC) adsorbers in wastewater treatment (*1*). Sontheimer (*2*) proposed that service time of a GAC bed could be greatly extended through biological activity. Carbon loadings and the percentage removal of organics are increased. Other advantages of microbial activity on GAC include storage of somewhat refractory substances until microbial acclimation can result in their biodegradation and reduction in concentration fluctuations of adsorbable species in the feed (*3, 4*).

The development of microbial activity on GAC is a natural consequence of treating water containing biodegradable organics. However, the extent to which biodegradation by attached microbial growths may account for removal of trace organic contaminants in GAC is largely unknown (*5*). It is also unclear whether organic compounds may continue to be removed through biological degradation or by adsorption which is made possible by renewal of sorption sites by biological activity (*6*). McCarty et al. (*7*) found evidence for biological removals of many chlorinated benzenes and aromatic hydrocarbons on expended GAC beds at Water Factory 21, Orange County, CA. This paper reports on a laboratory study supporting this observation and conducted to help evaluate the importance

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