&EPA Project Summary

Removal of Metals in Combined Treatment Systems

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This project assessed the variables influencing the removal of metals through combined industrial-municipal treatment plants. The metals investigated were: aluminum, cadmium, chromium, copper, iron, lead, nickel, and zinc. The metals were studied at sub-toxic influent concentrations, and the interrelationships which influence metal removal were assessed.

The research was performed in two phases. Phase I involved batch studies on raw sewage and activated sludge, to identify and define the impact of individual chemical and physical parameters on metals removal. These batch studies consisted of three parts. In Part A, metal solubility in filtered raw sewage and secondary effluent was determined as a function of pH. Part B investigated the equilibrium sorption of the test metals onto primary sewage solids and onto activated sludge solids. In Part C, the effect of sewage variables such as detergent and ammonia concentration on metal sorption was evaluated. In Phase II, eight pilot treatment plants, each consisting of primary clarifier, aeration basin, and secondary clarifier, were operated at varying influent metal levels to study the effect of significant variables indicated from the Phase I results.

The results of this project indicate that the removal of metals in combined industrial-municipal treatment systems is influenced by a number of wastewater and treatment plant operation characteristics. The principal parameters controlling metal partitioning for each metal investigated were the total metal and suspended solids concentrations. The segregation of influent metals between the sludge (primary and secondary) phases and the plant effluent can be predicted, based upon the relationships identified in this study.

This Project Summary was developed by EPA's Robert S. Kerr Environmental Research Laboratory, Ada, OK, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

In recent years, increasing attention has been focused on the chemistry, biological effects, treatment, fate, and control of heavy metals in the environment. Findings include the discovery of heavy metals at high concentrations in surface waters receiving municipal and industrial waste discharges containing such metals, coupled with the recognition of potential health hazards and adverse environmental impacts associated with major disposal methods for metal-laden municipal and combined sludges. While the management of metals originating directly from industrial discharges has been implemented under effluent limitations guidelines and National Pollutant Discharge Elimination System (NPDES) permits, the control of industrial plus non-industrial metals entering combined municipal-industrial public-owned treatment works (POTWs) has been found to be much more difficult. As a result, heavy metals discharge into the municipal sewage treatment systems and their fate during the sewage treatment processes have become subjects of considerable interest.

Most studies to date concerning heavy metals in sewage treatment processes have involved attempts to perform mass balances of metals around a POTW and determine percentage of removal of each

metal of concern across that POTW. However, there is a relative lack of information on the actual mechanisms affecting the distribution of heavy metals between liquid and solid phases through a municipal sewage treatment plant. Such an understanding is essential for developing criteria that can be used to predict the distribution of heavy metals through combined sewage treatment systems.

Methods and Procedures

As indicated above, this investigation was divided into four parts, and a brief description of each part of the project is given here:

IA. Batch studies on tap water, filtered raw sewage, and filtered conventional activated sludge mixed liquor, to determine the solubility limits of the eight metals.

IB. Batch studies on raw sewage and activated sludge mixed liquor, to develop sorption data for selected metals.

IC. Batch studies on raw sewage and conventional activated sludge mixed liquor to investigate the influence of both domestic and industrial waste constituents on metals distribution between the soluble and solid phases.

II. Continuous-flow pilot-scale conventional activated sludge studies to evaluate the effect of variables such as total metal concentration, total volatile suspended solids (TVSS), soluble organic carbon (SOC), and major inorganic ligands, on heavy metals distribution in different process liquids.

In Part IA of the study, solubility of metals at different pH levels was determined for tap water, raw sewage, and activated sludge mixed liquor.

Tap water used in this study came from Chicago's city water distribution system, while the raw sewage and activated sludge mixed liquor were obtained from the West-Southwest Wastewater Treatment Plant operated by the Metropolitan Sanitary District of Greater Chicago. Batch experiments were performed for each test liquid (tap water, raw sewage, and mixed liquor).

Initially, the test liquid was filtered using a 0.45-micron membrane filter. Raw sewage and activated sludge mixed liquor were settled prior to membrane filtration, to enhance membrane filtration efficiency. Initial pH levels of 6, 7, 8, and 9 (± 0.3 units) were established in units of each group by pipetting sodium hydroxide or nitric acid into the test liquid, as required, with constant stirring. Following pH adjustment, the appropriate concentrated metal solution was pipetted into the test liquid. Simultaneously, pH adjustment was made to maintain the target test pH

level. Metal solution was added until a visible precipitate formed and remained after one minute of continuous stirring. The sample was continuously stirred during the metal addition step, and the pH was monitored.

The test vessels were sealed with parafilm and placed on a shaker with continuous shaking at ambient temperature. After two hours, all units were readjusted to correct for any pH change. Aliquots of the test liquids were taken at 6, 12, and 24 hours for measurement of pH, soluble metal, and SOC. Background analyses on the test liquids included pH, total dissolved solids (TDS), total volatile dissolved solids (TVDS), initial SOC, background metals, sulfide, sulfate, total phosphorus, orthophosphate, ammonia, hardness, and alkalinity.

In Part IB, sorption of metals onto sludge was studied by measuring the amount of metal associated with the sludge fraction after the metal is added to the test liquid, at a level below its solubility limit as determined in Part IA.

Batch experiments were set up in a similar fashion to that described in Part IA. In this component of the project, unfiltered samples were taken, their pH adjusted to the desired levels, and the selected metals added. The amount of metal added was below its solubility limit, to avoid precipitation. After the metal addition, the samples were constantly stirred, and aliquots of samples were taken at 0.25, 0.50, 1, 3, 6, and 24-hour time intervals, to measure pH and soluble metal concentration. The samples from the 24-hour test period were also analyzed for total organic carbon (TOC), SOC, inorganic carbon, total suspended solids (TSS), TVSS, TDS, and TVDS, total phosphorus, orthophosphate, and alkalinity.

Part IC was designed to investigate, in depth, the influence of domestic and industrial waste constituents on the distribution of heavy metals between the soluble and solid phases of raw sewage and activated sludge mixed liquor. This objective was accomplished by spiking aliquots of test liquids with each selected waste constituent and determining how the phase distribution of metals was affected.

Seven different domestic/industrial waste parameters, at three levels for each parameter, were tested. For each parameter tested, a series of seven different metals combinations were evaluated. Metal Combinations 1 through 4 consisted of mixtures of eight metals at low (Combination 1) to high (Combination 4) relative concentrations. Metal Combinations

5 and 6 were replicates of Combination 3, providing a statistical basis for the evaluation of experimental results. In Metal Combinations 7 and 8, the metal levels were varied randomly (i.e., some metals were at high and others at low concentrations). Random metal combinations were incorporated in the studies in order to determine whether interactive effects upon metal removal result from preferential removal of specific metals by the sludge phase. There was a control group to which no metal was added. All metals concentrations fell within the range of typical influent values reported for POTWs.

Each set of raw sewage test samples was mixed on a shaker table for four hours at ambient temperature. At the termination of the mixing period, an aliquot of the whole fraction of each sample was taken for analyses. An additional aliquot was filtered through a 0.45-micron filter to obtain soluble samples. Since metals influent to activated sludge units have had extended contact periods with raw sewage, the settled supernatant resulting from the raw sewage experiments was utilized as the media for introduction of metals to the activated sludge samples.

Part II of this investigation was designed to study the distribution of metals in different process liquids of continuous-flow pilot-scale conventional activated sludge systems receiving raw sewage, spiked with heavy metals at different concentrations.

The continuous-flow studies of Part II were divided into several runs, each run involving eight separate parallel pilot-scale activated sludge treatment systems. There were 39 different activated sludge treatments contained in this phase of the project. Table 1 presents the concentrations of different heavy metals in the raw sewage fed during the 39 different activated sludge runs. These individual metals concentrations and combinations were selected on a random basis, to simulate low, high, and mixed levels of metals in raw sewage.

Municipal sewage was pumped from a City of Chicago sewer line to a laboratory grit chamber on a continuous basis. Settled grit was discharged. Raw sewage overflowed from the grit chamber into a 300-gallon stirred holding tank, having an average six-hour detention time. The holding tank was equipped with a low level alarm, to cut off all downstream pumps and valves (except for return activated sludge pumps and excess sludge wastage valves), in the event that the raw sewage flow was interrupted. The raw sewage was pumped into a common header, and then

Table 1. Average Influent Metals Concentrations (μg/l) In Raw Sewage Fed to 39 Different Activated Sludge Systems

Treatment No.	Aluminum	Cadmium	Chromium	Copper	Iron	Lead	Nickel	Zinc
1	783	25	135	393	1265	81	672	482
2	433	42	143	359	1542	93	756	413
3	1003	140	174	274	1750	293	1629	1114
4	1310	80	630	280	1460	140	2740	826
5	678	12	113	90	1399	35	334	409
6	298	124	84	161	1247	37	369	383
7	375	63	150	177	1292	75	1780	481
8	372	143	128	530	1610	320	1220	830
10	932	60	600	150	2675	150	838	1583
11	383	28	155	429	1439	57	795	510
12	495	77	159	479	1641	88	1002	617
13	500	105	153	271	1521	158	869	643
14	295	154	122	460	2220	170	986	553
15	710	93	1062	338	3360	150	1220	1003
16	678	59	460	240	1534	90	1615	1575
17	678	12	113	90	1399	35	245	409
18	295	137	97	173	1576	154	352	450
19	677	88	183	453	636	267	2983	1114
20	520	138	144	625	1510	475	3263	694
21	661	146	500	425	3225	150	1678	1025
22	983	53	420	270	2510	140	1263	1860
23	655	24	106	308	1378	41	680	564
24	385	157	137	460	2243	75	653	477
25	785	135	109	367	2492	221	4008	514
26	240	128	124	325	1488	190	6075	766
27	834	77	513	363	3200	175	2050	1463
28	890	57	530	350	2350	180	2132	2160
29	669	11	113	90	1399	35	330	409
30	278	63	62	162	1527	100	366	440
31	567	69	90	213	936	143	490	429
32	216	98	128	180	650	120	2050	644
33	740	22	144	302	1385	66	603	520
34	778	222	253	756	2322	200	1522	536
35	1574	87	140	1071	2117	260	708	540
36	1193	102	100	210	1510	160	319	463
37	678	11	113	98	1399	35	245	409
38	337	87	84	170	1483	97	373	413
39	693	81	124	269	671	100	619	450

into eight parallel dosing tanks of two-hour detention time each. Selected metals mixtures were metered into each chemical dosing tank, in accordance with the experiment underway for that particular treatment system.

Each dosing tank overflowed to a primary clarifier of a system. The flow rate was about 130 ml/mn. Primary clarifier overflow was through a flow splitter, to control hydraulic loading to the activated sludge unit. Each activated sludge unit was constructed as a five-chamber, 100-liter total capacity unit, with removable partitions to convert from a plug to complete mixed flow mode.

Activated sludge unit mixed liquor overflowed by gravity to a secondary clarifier, where settled sludge was returned by a peristaltic pump to the activated sludge unit. The recycle ratio used for all activated sludge units in this study was 1:1. Excess sludge was wasted directly from the sec-

ondary clarifier, or by intermittent interval wasting of activated sludge unit overflow, as was most appropriate for control of sludge age. Sampling from each unit was by timer activated solenoid switch flow diverters, to yield eight-hour composite samples.

Composite samples of the raw sewage, primary effluent, activated sludge mixed liquor, secondary effluent, primary sludge, and secondary sludge were collected several times each week. Total and soluble metal analyses were performed on all process liquid samples, while the sludge samples were analyzed for total metals. In addition, pH, suspended solids, and TVSS were also measured on these samples. Soluble samples of the four process liquids were analyzed for SOC, inorganic carbon, phosphate, sulfate, chloride and ammonia nitrogen.

Metal analyses were performed by atomic absorption spectrometry. Total phos-

phorus, orthophosphate, sulfate, chloride, ammonia, calcium, hardness, and alkalinity determinations were performed according to procedures described in EPA Methods. TVSS values are reported as the weight of the dry solids per liter of sample retained by a 0.45-micron membrane filter. Total dissolved solids represented the dry solids present in the filtrate of one liter of original sample. Volatile solids are reported as the weight of residue lost upon ignition at 600°C of one liter of the original sample.

Results and Conclusions

The following conclusions were drawn from the Phase IA studies on metals solubility in filtered raw sewage and aeration basin mixed liquor.

1. At all pH values tested, equilibrium solubility conditions were achieved within six to 12 hours. Levels of metal solubility were equivalent at 24 hours to those observed at 12 hours.

2. High correlations were observed between metal solubility and process liquid pH, for all metals investigated.

3. Within each process liquid, over the 24-hour period of the solubility tests, the pH in each case shifted from the more extreme high or low initial pH values toward a final pH value of about 8. This pH shift suggests that the process liquids are well buffered, and the occurrence of more extreme pH conditions in full-scale treatment systems would indicate the presence of strong acid or basic industrial wastes which would influence metals solubility.

4. A comparison of metals solubility in filtered process liquids with that in tap water revealed that in most instances the process liquids yielded higher metals solubility than did the tap water. This response is probably due to the metal complexation effects of organic and inorganic ligands in the process liquids.

5. The pH range of minimum metals solubility, for all metals tested and in both process liquids, was in the pH range of 8 to 9, except for aluminum in mixed liquor where a pH of minimum solubility of 6.8 was observed.

The distribution of metals between the soluble and solids (sludge) phases in raw sewage and mixed liquor was studied, with metals added to the test liquids at concentrations below the metals solubility limits. The following results were observed.

1. A major portion of each added metal was removed from the soluble phase onto the solid phase in each test liquid. The distribution was essentially completed within a 15-minute contact time although

some minor additional redistribution continued for up to six hours.

2. Since the metals were added to the process liquids at concentrations below their solubility limits, removal from the liquid phase could not be by precipitation of metal salts, and therefore was due to accumulation by sorption onto the raw sewage and activated sludge solids.

3. The sorption behavior of each metal could be described by an adsorption isotherm relating μg of sludge metal sorbed per mg of TVSS, versus total metal present. Although the sorption data generally followed this isotherm, the data for most metals did not fit a standard Freundlich isotherm, based upon residual metal in solution.

4. Sorption of added metal in raw sewage ranged from 0 to 99%, with the following ranking of metals from least to most completely sorbed: iron, nickel, cadmium, copper, zinc, lead, chromium. Sorption of added metal in activated sludge mixed liquor ranged from 8 to 98%, with the following ranking of metals from least to most sorbed: iron, nickel, zinc, cadmium, chromium, copper, and lead.

It has been suggested in the literature that various waste parameters might influence the distribution of metals in raw sewage and mixed liquor between the soluble and solid phases. Constituents evaluated in Phase IC were inorganics plus hardness, detergents, SOC, pH, cyanide, and ammonia. The following conclusions were drawn, based upon statistical analysis of the experimental data.

1. Few of the waste constituents, at the levels tested, had a statistically significant effect on metals distribution between the soluble and solid phases.

2. At the 99% confidence level, SOC influenced aluminum distribution in raw sewage; pH influenced iron and nickel distribution in raw sewage; and ammonia influenced aluminum in mixed liquor.

3. At the 95% confidence level, inorganics and hardness influenced the distribution of aluminum and lead in raw sewage, and cadmium and lead in mixed liquor. Detergent strength influenced the distribution of chromium and nickel in raw sewage. In mixed liquor, chromium, iron, lead, and nickel were indicated to be influenced. At this confidence level, pH influenced the distribution of aluminum in raw sewage and mixed liquor. Ammonia was indicated to influence the distribution of cadmium in raw sewage.

During Phase II of the project, eight parallel continuous-flow pilot activated sludge systems were monitored around each unit process, during a total of 39 runs. Each run was approximately 30 days in length. Raw domestic sewage, spiked during each run with random levels of a mixture of test metals was treated (Table 1). Composite process liquid samples were collected several times weekly during each run, for raw sewage, primary clarifier effluent, mixed liquor, secondary clarifier effluent, and settled primary and secondary sludge analysis. Total and filtered fractions of each sample were analyzed for metals plus other constituents including SOC and TVSS. The conclusions developed from this phase of the project are comprehensive and are only briefly summarized here.

1. The removal of metals across the treatment system was directly related to the degree of distribution of each metal in the raw sewage and mixed liquor, and the efficiency of removal of the suspended solids (and associated metals) in the primary and secondary clarifiers. Thus, there, are two principal classifications of variables which influence metals removal in combined treatment systems: those associated with the metals distribution in each process liquid; and those associated with the performance of the clarifiers in solids separation.

2. In some experimental runs, negative removals of the metals were observed across the primary clarifiers, and/or the full treatment systems. These negative metals removals always resulted from negative removals of suspended solids in the primary clarifier. Intermittent negative removals of suspended solids in primary clarifier are observed in full-scale systems as well as pilot units. This negative performance of the primary clarifier in suspended solids removal suggests why some short-term mass balance studies on full-scale systems have resulted in negative full-system removals of metals.

3. Over the course of the 39 experimental runs, a wide range of concentrations of influent SOC, TVSS and metals were observed, reflecting the combination of natural fluctuations in the raw sewage composition, plus the spiking of the raw sewage with metals. Average performance of the system in solids removal was 76%, and removal of SOC averaged 61%. Ranges and averaged values are presented in Table 2.

4. Ranges of total effluent metals were also broad, although less so than the influent metals ranges. However, an evaluation of the soluble metals levels revealed that the average soluble concentration, for each metal, remained essentially constant across each unit process and the entire treatment system (Table 2).

Thus, the reduction of total metals across the unit processes was due to the sedimentation of solid-bound metal.

5. The total metal concentrations in the activated sludge aeration basin were much higher than was observed in any other process liquid. However, the soluble metals levels in all process liquids were equivalent, and the higher total metals levels in the mixed liquor resulted due to higher levels of suspended solids and their associated metals.

6. Relatively wide variation in the total metals discharged in the secondary effluent resulted from variation in effluent suspended solids; the effluent soluble level of each metal was comparable to the raw sewage soluble level of that metal.

7. The relative contribution of the soluble fraction of the effluent metals ranged from a low 2.9% for chromium up to 34.1% for nickel. Increased secondary clarifier efficiency in suspended solids removal would reduce only the non-soluble portion of the effluent metals.

8. The averaged removal of metals in the primary clarifier ranged from 14.0% for zinc to 41.1% for iron, and the metals ranked from lowest to highest removal in the primary clarifier were: zinc, copper, cadmium, aluminum, chromium, lead, iron, nickel.

9. The averaged removal of metals across the activated sludge process plus secondary clarifier ranged from 1.3% for aluminum to 38.9% for cadmium, and the metals ranked from lowest to highest as follows: aluminum, chromium, nickel, iron, zinc, copper, lead, cadmium.

10. The averaged overall removals of metal across the entire treatment system ranged from 27.6 for aluminum to 54.9% for lead, with the metals ranked from lowest to highest removal as follows: aluminum, zinc, chromium, copper, iron, nickel, cadmium, lead.

11. For the metals aluminum, chromium, iron, and nickel, the bulk of overall removal occurred in the primary clarifier. For the metals cadmium and copper, the secondary processes accounted for the majority of overall removal. Removals of lead and zinc were about equally distributed between the primary and secondary stages.

12. Although the experimental data of the 39 runs can be fitted to adsorption isotherms, a more striking and significant relationship was identified on the basis of the data generated from the continuous-run pilot units. This relationship reveals that the concentration of each metal sorbed on the solids of each process liquid was directly related to total metal, and was

Table 2. *Overall Averages and Ranges for Different Parameters in Different Test Liquids**

Parameter			Raw Sewage	Primary Effluent	Mixed Liquor	Secondary Effluent
VSS		Avg.	62	36	1307	15
		Range	2-460	1-196	150-8106	1-220
SOC		Avg.	28	19	14	11
		Range	3-294	1-106	1-200	1-38
Aluminum	Total	Avg.	652	478	7179	472
		Range	63-5100	24-3032	526-21000	67-2732
	Soluble	Avg.	81	79	61	83
		Range	11-425	8-375	0-325	5-350
	% Soluble	Avg.	12.4	16.5	0.8	17.6
Cadmium	Total	Avg.	85	72	411	44
		Range	3-650	2-514	4-810	2-382
	Soluble	Avg.	16	14	15	13
		Range	1-305	1-295	1-98	1-67
	% Soluble	Avg.	18.8	19.4	3.6	29.5
Chromium	Total	Avg.	241	170	1292	162
		Range	18-1700	5-650	10-3150	31-1600
	Soluble	Avg.	4.2	4.0	4.0	3.9
		Range	2-17	2-9	2-9	2-5
	% Soluble	Avg.	1.7	2.4	0.3	2.4
Copper	Total	Avg.	330	281	3215	210
		Range	11-2900	3-913	4-8500	11-1866
	Soluble	Avg.	17	12	14	14
		Range	1-157	1-100	1-96	1-50
	% Soluble	Avg.	5.2	4.3	0.4	6.9
Iron	Total	Avg.	1778	1247	28184	1089
		Range	200-7000	200-3500	1048-8400	100-5800
	Soluble	Avg.	118	97	70	52
		Range	5-783	5-842	3-885	3-580
	% Soluble	Avg.	6.6	7.8	0.2	4.7
Lead	Total	Avg.	142	100	1971	64
		Range	0-1069	0-600	11-9000	0-1200
	Soluble	Avg.	24	27	24	18
		Range	2-197	2-248	2-474	2-211
	% Soluble	Avg.	16.9	27.1	1.2	28.3
Nickel	Total	Avg.	1349	794	6602	733
		Range	22-8500	5-15000	77-23000	10-5000
	Soluble	Avg.	319	297	290	250
		Range	8-1168	9-1479	5-975	3-849
	% Soluble	Avg.	23.6	37.4	4.4	34.1
Zinc	Total	Avg.	741	637	11589	514
		Range	100-5000	80-3400	1000-36000	100-4100
	Soluble	Avg.	90	74	79	65
		Range	2-1000	1-430	2-900	1-900
	% Soluble	Avg.	12.1	11.6	0.7	12.6

inversely related to TVSS present. In other words, at constant suspended solids, the metal per unit of solids increased with increasing total metal. However, at constant total metal, the metal per unit of solids increased with decreasing suspended solids concentration. Figure 1 presents one example, for nickel distribution in raw sewage.

13. A number of models were assessed for their accuracy in predicting the distribution of metals in each process liquid, between the soluble and solid phases. An investigation of the influence for the total metal concentration of the parameters VSS, SOC, and pH revealed that a model which related total metal to sludge-bound metal per unit weight of VSS and to VSS solids in the process liquid provided an accurate prediction tool for metals distribution. This model has been designated as Metals Distribution Model 3 in this report and model coefficients for each metal in each process liquid were derived. Model 3 yielded high correlation coefficients with the experimental data on each process liquid and each metal, ranging from a squared coefficient of 0.80 for nickel to 0.99 for chromium in raw sewage, and coefficients of 0.99 for all metals in mixed liquor (Table 3). At moderate to high suspended solids levels, a simplified model (termed Model 4) which directly relates total metal to sludge-bound metal is equally accurate, and Models 3 and 4 have been utilized as the basis for a model of the full-treatment system.

This project has resulted in the development of a full-system model to predict the removal of metals at each unit process across a combined treatment system. The full-system model relies upon submodels for (1) metals distribution, by process liquid, (2) primary clarifier performance in suspended solids removal, and (3) secondary treatment system performance in terms of sludge yield, and secondary clarifier performance.

A comparison of the full-system model to pilot-plant experimental data revealed that, where the full-system model was
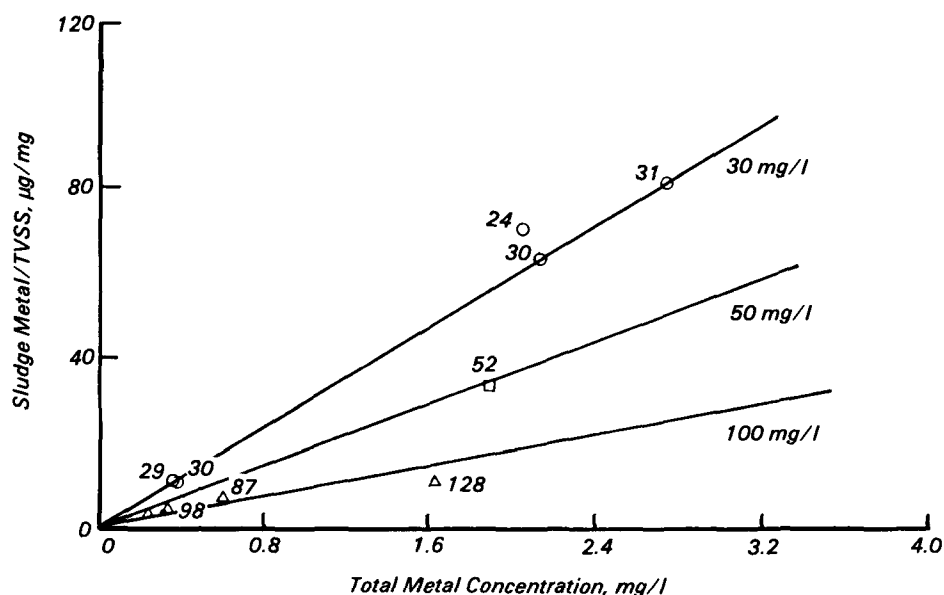
Figure 1. Adsorption isotherms of nickel in raw sewage at different VSS concentrations.

Table 3. Squared Correlation Coefficients for Metals Distribution Model 3

	Process Liquid			
Metal	Raw Sewage	Primary Effluent	Mixed Liquor	Secondary Effluent
Aluminum	0.959	0.749	0.999	0.852
Cadmium	0.970	0.837	0.997	0.720
Chromium	0.999	0.999	0.999	0.999
Copper	0.996	0.989	0.999	0.992
Iron	0.989	0.984	0.998	0.949
Lead	0.877	0.840	0.997	0.826
Nickel	0.803	0.560	0.986	0.909
Zinc	0.953	0.914	0.999	0.814

inaccurate, it failed through an inability to track the short-term solids balance around each unit process. These unit processes, while performing in a predictable fashion on a long-term average basis, perform in a more erratic fashion over short periods of days to weeks, sometimes exhibiting, for example, negative suspended solids removal in the primary clarifier or short-term interruptions in activated sludge yield. Metals removals are closely tied to the solids balances around the unit processes of the treatment system, and improved models to predict the short-term behavior of the systems in terms of solids are necessary before more accurate short-term modeling of metals dynamics will be possible.

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