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LAMS-863

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PROGRESS OF LIQUID WASTE DISPOSAL STUDIES

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Progress of Liquid Waste Disposal Studies

The results of three experiments studying the use of activated sludges for removing plutonium from liquid wastes, presented in LAMS-823 and LAMS-833 have indicated that activated sludges will remove from 90 to 95 per cent of the radioactive material from waste solutions. Also, in LAMS-833, it was reported that preliminary experiments with weighting materials for activated sludges indicated ability of inert substances to remove plutonium from liquids.

This report contains results of continuation of the above work as follows:

1. A fourth experiment designed to obtain relative effectiveness of different types of activated sludges in removing plutonium from liquids and to simulate operation of a three-stage biological treatment plant.

2. Expansion of studies of the relative effectiveness of inert materials in removing plutonium from liquids.

Experiment 4 on activated sludges

On the basis of plutonium removal efficiencies that could be expected by the use of activated sludges (90 to 95%) it was evident that two, and perhaps three, stages of biological treatment would be necessary for producing a final effluent containing less than 70 counts per minute per liter of the radioactive material. Likewise, in operation of a multistage process, the amount of plutonium concentrated in the sludges and

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effluents would decrease from a maximum in the first stage to a minimum in the final stage of treatment. Approximating these conditions, a three-stage bottle experiment was conducted for a period of three weeks.

Description of Experiment:

The experiment was conducted in 5-gallon carboys with a total volume of 16 liters of material. Air was introduced through a carborundum ball with just enough volume to maintain the sludges in suspension insuring thorough mixing of the sludge particles in the liquid. Three different sludges were studied in parallel. The types of sludge selected were:

a. Activated sludge developed using 150 ml of screened primary solids as the daily nutrient. (The sewage solids contained 50% ash and 50% volatile matter).

b. Synthetic activated sludge maintained with a daily feed containing:

1 gram glucose (On the 11th day (12 January) the glucose was increased to 1.25 grams per feed.)

0.5 gram phenol (Phenol was introduced to reduce infection of *Sphaerotilus natans*.)

0.025 gram ammonium sulfate.

0.025 gram dipotassium phosphate.

2 ml canned milk

c. Synthetic activated sludge maintained on formula (b) as above and weighted with 1000 ppm of finely ground tufa

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(passing 200 mesh screen). The tufa, a volcanic ash available to the Los Alamos area, was used in an effort to increase the settleability of the synthetic sludge.

The same daily procedure was followed for each sludge throughout the experiment:

1. Stopped aeration at 8:30 AM and allowed sludge to settle for ½ hour.

2. Two liters of supernatant withdrawn for analyses. (22 hour sample).

3. Feed and plutonium spike added.

4. Tap water added to make up volume of mixture to 16 liters.

5. Mixture aerated until 2:00 PM at which time sludge was allowed to settle ½ hour.

6. Two liters of supernatant withdrawn for analyses. (5 hour sample)

7. Aeration until 8:30 AM the following day.

The five hour samples, step (6), were not taken on weekends.

Duration of the experiment was three weeks, each week representing a different cycle, or stage, of treatment. The only variation in the procedure throughout the three cycles was the amount of plutonium added in the spike. Concentrations of plutonium in the spikes were as follows:

First week: 1,000 counts per minute per liter added daily.

Second week: 9,100 counts per minute per liter added daily

Third week: 95,000 counts per minute per liter added daily.

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The spike consisted of plutonium dissolved in hydrochloric acid and diluted with distilled water. One microgram of plutonium is approximately 70,000 counts per minute.

Plutonium assay work was performed on filtered samples throughout the experiment. In most cases, the unfiltered sample was not subject to analysis because previous experience has shown that the values found on unfiltered samples are quite erratic and difficult of interpretation. However, during the final days of the experiment, plutonium assays were made on unfiltered samples to indicate the magnitude of the plutonium carry-out by suspended matter in the sludge.

The volumes selected for analyses were:

First week - 1000 ml.

Second week - 500 ml.

Third week - 100 ml.

Summary

1. Tables 1, 2, and 3 contain a summary of analytical data obtained from the three sludges studied in experiment 4.

2. Table 4 shows a summary of the average plutonium residuals in the filtered supernatants from the three sludges. These average results simulate what might be expected of a three stage biological treatment process, results of the third week indicating the first stage of treatment, the second week indicating the second stage and the ~~first~~ third week indicating the third stage of treatment.

3. The removal efficiencies shown in Table 4 are computed on filtered effluents. Results of analyses on four

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unfiltered samples are tabulated in tables 1, 2, and 3. These results indicate that small amounts of suspended "hot" sludge in the final effluent will materially decrease the efficiency of treatment. Thus, it follows, that the final effluent from a biological process could be materially improved by filtration to remove the suspended matter.

4. A comparison of the average suspended solids (volume of sludge) in the mixed liquor shows the following for the three sludges.

Type of Sludge	Average suspended solids for three weeks in mixed liquor		Total volume of sludge that would be contained in 1000 gals. of mixed liquor.	
	Total ppm	% Ash	Pounds	Pounds of Ash
Fed on Sewage Solids	9400*	61.1	78.30	47.84
Synthetic	880	15.4	7.33	1.13
Synthetic(weighted)	2190	60.4	18.24	11.02

* Similar plutonium removal efficiencies were found in previous experiments with sewage sludges having much lower solids concentration in the mixed liquor.

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Table #1

Analytical Data

Experiment: No. 4A with a Sewage Solids Feed.

Date & Time 1/49		Counts added x 1000 Daily	Cumulative	per liter	Susp. Sol. in mixed liquor Total	Ash	EFFLUENT pH	Counts/min/lit unfilt.	filt.	Hours Aeration after last spike
3	1130	16	16	1	7500	48.3	-	-	-	0
4	0900	16	32	1			7.9	-	33	21.5
4	1400	--	32	-			7.5	-	142	5
5	0900	16	48	1			7.4	-	71	22
5	1400	--	48	-			7.6	-	125	5
6	0900	16	64	1			7.6	-	89	22
6	1400	--	64	-			7.5	-	101	5
7	0900	16	80	1			7.6	-	77	22
7	1400	--	80	-			7.7	-	120	5
8	1300	16	96	-			7.6	-	67	23
9	1200	16	112	1			-	-	77	23
10	0900								134	21
10	0900	146	258	9.1	10,700	58.9	7.3	-	---	0
10	1400	--	258	-			7.4	-	578	5
11	0900	146	404	9.1			7.4	-	278	22
11	1400	--	404	-			8.0	-	250	5
12	0900	146	550	9.1			7.9	-	174	22
12	1400	--	550	-			7.6	-	626	5
13	0900	146	696	9.1			7.8	-	321	22
13	1400	--	696	-	10,900	72	7.9	-	1032	5
14	0900	146	842	9.1			7.8	-	596	22
14	1400	--	842	-			7.9	-	1068	5
15	1400	146	988	9.1			7.8	-	562	24
16	1300	146	1134	9.1			7.7	-	445	23
17	0900							-	835	20
17	0900	1520	2654	95			7.8	-	---	0
17	1400	--	2654	-			7.7	-	16,800	5
18	0900	1520	4174	95			7.9	-	8500	22
18	1400	--	4174	-			7.6	-	8400	5
19	0900	1520	5694	95			7.5	-	6340	22
19	1400	--	5694	-			7.5	-	9500	5
20	0900	1520	7214	95			7.6	-	5600	22
20	1400	--	7214	-			7.6	-	8500	5
21	0900	1520	8734	95			7.5	-	5420	22
21	1400	--	8734	-			7.5	15,900	12,820	5
22	1300	1660	10,394	104				10,300	4610	23
23	1300	1660	12,054	104				20,900	7000	23
24	0900				8600	6[illegible].2	7.8	38,250	17,700	20

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Table #2
Analytical Data

Experiment: No. 4B with Synthetic Feed.

Date & Time 1/49		Counts added x 1000 Daily	Cumulative	per liter	Susp. Sol. in mixed liquor Total	%Ash	EFFLUENT pH	Counts/min/lit. Unfilt.	Filt.	Hours Aeration after last spike
3	1130	16	16	1	930	17.3		-		
4	0900	16	32	1			7.2	-	95	21.5
4	1400	--	32				7.8	-	177	5
5	0900	16	48	1			7.3	-	104	22
5	1400	--	48				7.0	-	148	5
6	0900	16	64	1			7.6	-	126	22
6	1400	--	64	-			7.5	-	158	5
7	0900	16	80	1			7.6	-	206	22
7	1400	--	80	-			7.6	-	176	5
8	1300	16	96	1			8.0	-	238	22
9	1200	16	112	1			-	-	248	23
10	0900	--					-	-	238	20
10	0900	146	258	9.1	760	16.5	7.9	-	---	0
10	1400	--	258	-			7.7	-	696	5
11	0900	146	404	9.1			7.9	-	498	22
11	1400	--	404	-			7.9	-	790	5
12	0900	146	550	9.1			8.0	-	743	22
12	1400	--	550	-			7.9	-	920	5
13	0900	146	696	9.1			7.9	-	764	22
13	1400	--	696	-	730	15.3	7.5	-	1180	5
14	0900	146	842	9.1			7.9	-	1068	22
14	1400	--	842	-			8.0	-	1380	5
15	1400	146	988	9.1			8.1	-	1055	23
16	1300	146	1134	9.1			8.2	-	1370	22
17	0900								1260	20
17	0900	1520	2654	95			7.9	-	---	0
17	1400	--	2654	-			7.7	-	5950	5
18	0900	1520	4174	95			8.1	-	4400	22
18	1400	--	4174	-			7.7	-	6550	5
19	0900	1520	5694	95			7.9	-	6000	22
19	1400	--	5694	-			7.6	-	7730	5
20	0900	1520	7214	95			7.9	-	6620	22
20	1400	--	7214	-			7.7	-	10,680	5
21	0900	1520	8734	95			7.8	-	7660	22
21	1400	--	8734	-			7.7	9960	7860	5
22	1300	1660	10,394	104				12,490	5250	22
23	1300	1660	12,054	104				35,400	16,100	23
24	0900				1100	12.5	8.1	80,300	40,400	20

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Table #3

Analytical Data

Experiment: No. 4C Synthetic (weighted)

Date & Time 1/49		Counts added x 1000 Daily	Cumulative	per liter	Susp. Sol. in mixed liquor Total	%Ash	EFFLUENT pH	Counts/min/lit. Unfilt.	Filt.	Hours Aeration after last spike
3	1130	16	16	1	1920	62.9	-	-	-	0
4	0900	16	32	1			7.4	-	106	21.5
4	1400	--	32	-			7.8	-	145	5
5	0900	16	48	1			7.6	-	131	22
5	1400	--	48	-			7.6	-	174	5
6	0900	16	64	1			7.8	-	148	22
6	1400	--	64	-			7.7	-	168	5
7	0900	16	80	1			7.7	-	156	22
7	1400	--	80	-			7.6	-	224	5
8	1300	16	96	1			7.8	-	148	22
9	1200	16	112	1			-	-	160	23
10	0900	--					-	-	188	20
10	0900	146	258	9.1	2110	60.5	8.2	-	---	0
10	1400	--	258	-			7.9	-	610	5
11	0900	146	404	9.1			8.1	-	470	22
11	1400	--	404	-			7.7	-	765	5
12	0900	146	550	9.1			7.9	-	536	22
12	1400	--	550	-			7.7	-	664	5
13	0900	146	696	9.1			8.2	-	640	22
13	1400	--	696	-	2120	64.7	7.9	-	550	5
14	0900	146	842	9.1			8.1	-	612	22
14	1400	--	842	-			7.8	-	731	5
15	1400	146	988	9.1			8.1	-	585	23
16	1300	146	1134	9.1			8.2		626	22
17	0900	--		-			-		720	20
17	0900	1520	2654	95			8.1	-	---	0
17	1400	--	2654	-			7.9	-	7320	5
18	0900	1520	4174	95			7.9	-	5325	22
18	1400	--	4174	-			7.5	-	7360	5
19	0900	1520	5694	95			8.0	-	4360	22
19	1400	--	5694	-			7.8	-	5730	5
20	0900	1520	7214	95			8.0	-	3900	22
20	1400	--	7214	-			7.9	-	8000	5
21	0900	1520	8734	95			7.9	-	5390	22
21	1400	--	8734	-			7.9	34,200	7860	5
22	1300	1660	10,394	104			-	12,800	11,220	23
23	1300	1660	12,054	104			-	20,600	5840	23
24	0900	--		-	2500	53.7	8.1	50,800	19,400	20

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Table #4

Tabulation of Average Analytical Results shown in Tables 1,2, and 3.

Type of Activated Sludge	Plutonium Added in counts/min/liter			Counts/min/liter of Plutonium Remaining after Aeration Period			
	1st.week	2nd.week	3rd.week	5 hrs.	%Removal	22 hrs.	%Removal
Fed on Sewage Solids	1,000			122	87.8	78	92.2
Synthetic	1,000			165	83.5	180	82.0
Synthetic (weighted)	1,000			178	82.2	148	85.2
Fed on Sewage Solids		9,100		711	92.1	459	94.9
Synthetic		9,100		993	89.1	965	89.5
Synthetic (weighted)		9,100		664	92.5	598	93.4
Fed on Sewage Solids			95,000	11,204	88.2	6,245	93.4
Synthetic			95,000	7,754	91.8	12,347	87.0
Synthetic (weighted)			95,000	7,754	91.8	7,919	91.6

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Effectiveness of Inert Materials in Removing Plutonium from Liquids

The use of finely ground tufa (passing 200 mesh screen) as a weighting material in experiment four on activated sludges has been described. In order to round out the study of the sludges it was desirable to learn the plutonium removal properties of the tufa alone. Accordingly, 5000 ppm of finely ground tufa was introduced into 3 liters of tap water spiked with 98,000 counts per minute per liter of plutonium. The solution was aerated, settled and filtered. Analyses performed on the filtered supernatant showed 9,000 counts per minute per liter remaining after 3 hours aeration and 125 counts per minute per liter remaining after 20 hours aeration.

Results of the experiment on tufa indicated that the studies should be expanded to include other materials.

Comparison of Various Inert Materials

A series of experiments was performed using 5000 ppm each of activated carbon, celite, kaolin, and finely ground pumice. These materials were introduced into solutions of tap water, each containing 95,000 counts per minute per liter of plutonium, and aerated for 20 hours. Analyses in each case were performed on filtered supernatants. The concentrations of plutonium remaining in solution after aeration were as follows:

		% reduction
Activated carbon	850 counts per minute per liter	99.6
Celite	6840 counts per minute per liter	92.6
Kaolin	3160 counts per minute per liter	96.6
Tufa		99+
Pumice		

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Pumice 8020 counts per minute per liter 91.4

Comparison of Various Carbons

Inspection of the removal efficiencies showed activated carbon highly superior to the other materials. Accordingly, a series of experiments was performed to compare the relative efficiencies of different carbons. 5000 ppm of each of 5 different carbons were introduced into solutions of distilled water containing 98,000 counts per minute per liter of plutonium. The concentrations of plutonium remaining in solution after 15 hours aeration followed by filtration were:

Type of Carbon		
Darco G-60	445 counts per minute per liter	
Fisher	272 counts per minute per liter	
Norite	865 counts per minute per liter	(High count may have been due to poor filtering)
Nuchar	288 counts per minute per liter	
Pellet Size	19,200 counts per minute per liter	

Comparison of Carbon Dosages

Of the carbons studied Darco Grade G-60 was of coarser grain and much easier to handle. On that basis it was chosen for further experimentation. Dosages of 100, 300, 1000, and 2500 ppm were aerated in distilled water containing 113,000 counts per minute per liter of plutonium. Samples were collected for analysis after 1½ hours and 17 hours aeration. Results of analyses on the filtered supernatants are tabulated below:

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Table II

Carbon Dosage	Concentration remaining in filtered supernatant after 1½ hours counts/min/liter	1½ hours % reduction	17 hours counts/min/liter	17 hours %reduction
100 ppm	64,750	42.7	15,420	86.4
300 ppm	3,225	97.1	1,625	98.6
1000 ppm	650	99.4	464	99.6
2500 ppm	325	99.7	210	99.8

Comparison of Carbon and Tufa.

The use of 5000 ppm of finely ground tufa (passing 200 mesh screen) was compared with 300 ppm of Darco G-60 activated carbon. The materials were introduced into distilled water containing known plutonium spikes and aerated for 17 hours.

Plutonium Spike Counts/min/liter	pH	Concentration remaining in filtered supernatant 5000 ppm tufa counts/min/liter	5000 ppm tufa %reduction	300 ppm Activated Carbon counts/min/liter	300 ppm Activated Carbon %reduction
1,120	8.2	443	60.5	86	92.3
11,200	3.2	369	96.7	1390	87.6
11,200	7.9	3040	72.9	1720	84.7

Effect of Mechanical Stirring.

One experiment was performed introducing 300 ppm of Darco G-60 activated carbon into distilled water spiked with 113,000 counts per minute per liter of plutonium. The mixture was stirred for 18 hours, filtered and analyzed. The plutonium concentration remaining in the filtered supernatant was 700 counts per minute per liter, a reduction of 99.4%.

The Use of Carbon on Plant Wastes.

Samples of plant wastes were composited on January 12 and January 17. One liter samples were taken from each composite.

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The pH was adjusted to 8.3 with lime, 300 ppm of Darco G-60 carbon was introduced and the resulting mixtures aerated for 18 hours. Plutonium determinations were performed on the samples before treatment and on the filtered effluent after treatment.

Table 3

Composite Plant Waste	Plutonium Concentration in counts per minute per liter Before Treatment	After Treatment	% reduction
January 12	7200	450	93.7
January 17	6230	116	98.1

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Preliminary Investigations on the Effectiveness of Chemical Coagulation for Removing Plutonium from Solution

In the experiment studying the effectiveness of carbon for removing plutonium from plant waste a floc, or precipitate, was formed in the solution. This suggested the use of coagulants commonly known in the water and sewage treatment fields. The following experiments were performed using lime and ferric chloride:

Treatment of Distilled Water Containing Known Plutonium Spikes.

Three samples of distilled water were spiked with plutonium. 20 ppm of $FeCl_3$ was introduced into each sample and the pH was adjusted to about 10.5 with lime. The solutions were stirred until a slight precipitate formed, settled, filtered and analyzed for plutonium.

Plutonium Concentration in Counts per Minute per Liter	
Before Treatment	After Treatment
100,000	164
10,000	16
1,000	30

Treatment of Plant Waste with Lime.

Three composite daily samples of plant waste were collected. The pH was adjusted to 8.6 with lime. A heavy precipitate was formed after stirring. Analyses were performed on the filtered supernatants.

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Composite Daily Waste	Plutonium concentration in counts per minute per liter Before Treatment	After Treatment
January 17	4290	244
January 20	4660	116
January 21	3830	522

Comparison of filtration, treatment with lime and treatment with lime and 20 ppm of ferric chloride.

Two composite daily samples of plant wastes were treated by each of the three methods. Heavy precipitates were formed upon addition of the coagulants.

Method of Treatment	January 11 Waste PH	January 11 Waste Pu Concentration in C/M/L	January 12 Waste PH	January 12 Waste Pu Concentration in C/M/L
Untreated	2.6	3260	3.0	8900
Filtration	2.7	1680	3.3	1270
Lime	8.8	42	9.8	lost
Lime and 20ppm $FeCl_3$	8.5	84	8.7	12

Summary

1. The above series of experiments on the use of inert materials and coagulants indicate that plutonium can be removed from solution and concentrated into the inert material or into the precipitates formed by the coagulants.

2. The possibilities of treatment by chemical coagulation are quite attractive. Coagulation, sedimentation, and filtration apparently accomplish the purpose of removing plutonium from liquids. However, there is no assurance that chemical treatment will reduce the concentration of other toxic chemicals or remove the plutonium in the presence of fluorides, citrates or

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other plutonium complexing materials. Further studies will be necessary to evaluate these factors.

Future Course of Experimental Studies

Plutonium removal results obtained by using lime and ferric chloride indicated that the use of coagulants may prove superior to the activated sludge process for the treatment of laboratory wastes containing plutonium. Accordingly, after a series of conferences with Dr. E. R. Jette, Dr. C. C. Ruchhoft, Mr. A. E. Gorman and Mr. Guy R. Scott, the decision was made to withhold further exploration of the activated sludge process until more complete laboratory studies are made using coagulants. Dr. Ruchhoft outlined the following program for this work:

I. Tech Area Waste Study

A. Continued analysis of composite waste from Tech Area. Determinations of halogens, fluorides, chlorides, sulfates, suspended solids, total solids.

B. Study of the effectiveness of the various chemical coagulants such as Alum, $FeCl_3$, Ferrisul, copperas, NH_4OH, CaO, MgO, sodium silicate, etc. Make observations of the effect of pH and temperature.

C. Determine effect of various speeds and times of mixing on the effectiveness of coagulation with the coagulant selected.

D. Determine the quantitative yield of the sludge produced under the optimum conditions of mixing, coagulation and

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settling.

E. Determine the filterability of the sludge and the filter aids needed.

F. Glass tube sand filter studies for supernatants.

G. Expanded study of removal possibilities with activated carbon treatment.

II. Similar studies on DP Area Wastes.

III. Studies on laundry Waste treatment.

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