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MLM-331

Contract Number AT-33-1-GEN-53

MOUND LABORATORY

Operated By

MONSANTO CHEMICAL COMPANY

MIAMISBURG, OHIO

M. M. Haring
Laboratory Director

Classification changed to UNCLASSIFIED
authority of LASL Review 1/25/73 10/2/73
by Carletta L. Lewis
Reviewed by Paul Dowd 8/9/79

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PROCESS RESEARCH PROGRESS REPORT

Caution

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Date: April 1-30, 1949

This document is Prepared by: P. M. Hamilton

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PROCESS RESEARCH

Group 2

P. Hamilton, F. Middleton, H. Morris, T. Norton, and P. Schauer

Group 49

T. Brodbeck* and G. Schommer**

* Part time for chemical development of decontamination process using ultrasonic energy.

** On loan for development of ultrasonic equipment.

PERSONNEL DISTRIBUTION

	Problem	Group Number	No. of Man-Months
1.	Liquid Waste Disposal	2	4
2.	Combustible Waste Disposal	2	1
3.	Decontamination by Means of Ultrasonic Energy	49	1/4
		Total	5 1/4

ABSTRACT

Liquid Waste Disposal

The liquid waste disposal plant has been in hot operation since December, 1948. Until February, 1949 the influent was never above 100 c./min./ml. Since that month the quantity of activity has gradually increased to a maximum of 200,000 c./min./ml. and a minimum of 1,500 c./min./ml. The over-all efficiency in the removal of activity during February ranged from 95 to 99 per cent. Some difficulty has been experienced in treating the influent due to the variation in composition as compared with the solutions used in the original work. Most of the work of this group, for the past two months, has been devoted to determining the nature of those problems resulting in low efficiency.

During the month of March, 1949 a total of approximately [illegible],200,000 gallons of solution were received having an average of [illegible]2,000 c./min./ml. In the precipitation step an average of 95.4 per cent of the activity was removed. In the sand filtration step there was another 91.9 per cent removed for a total efficiency of 99.62 per cent. In April, 1949 approximately 750,000 gallons of solution were received having an average count of 40,000 c./min./ml. In the first step 99.2 per cent of the activity was removed while in the second step 92.3 per cent of the activity was removed. The total recovery was 99.95 per cent with a minimum of 99.92 and a maximum of 99.93 per cent.

Hot operation of the secondary concentration step was not started until the middle of this month. The sludge contained approximately 1 x 10[illegible] c./min./ml. In the first batch 99.7 per cent of the activity was removed from 70 gallons of the solution. The filtrate contained 2,500 c./min./ml. In the second batch 99.98 per cent of the activity was removed. The final count in the 70 gallons of filtrate was 800 c./min./ml.

Combustible Waste Disposal

Plans for modification of an area in the "HR" Building have been completed by the Engineering Department. The area is to be used for further development of the process for the disposal of combustible waste. It is expected that this area will be ready for installation of equipment sometime in August, 1949. An experimental steam-ejector nozzle and incinerator have been designed and are being constructed.

Decontamination by Means of Ultrasonic Energy

This investigation has been initiated to determine the possibility of using ultrasonic energy to decontaminate glassware, quartz, and plastics. A general discussion of the nature of this problem is given in this report.

DETAILED REPORT

Liquid Waste Disposal - F. Huddleston, R. Norton, and P. Schauer

A more detailed discussion of the work carried out during the past few months will appear in the next progress report. For the present, the general nature of the problem and results will be discussed.

Primary Concentration Step - For February, 1949 the average removal of activity from the influent was 97 per cent. In March, 1949 the average efficiency had increased to 99.6 per cent and in April, 1949 to 99.95 per cent.

The basic problem was the periodic increase of activity in the effluent from the primary process. The cause for the incomplete removal of activity can occur at either or both of two operations. The one is precipitation in a Dorr Clariflocculator and the other step is filtration through a Hardinge automatic backwash sand filter.

Investigation of the problem was first directed towards the latter step since little was known about the operation of the backwash mechanism. Analysis of the filtrate made during the backwash operation revealed that the activity was higher than before or after backwashing. A substantial reduction in activity was obtained by putting the two sand filters in series. The present method of operation consists of splitting the flow to both sand filters, then when it is necessary to backwash one of the filters the total flow is directed to the other filter. The filter requiring backwashing is thus taken out of operation during this process. More recent data has indicated that if there is proper flocculation in the first step there is an equal improvement in filtration.

The main bulk of the problem has been found to be either incomplete coprecipitation of aluminum hydroxide and polonium or poor flocculation or both. The factor governing coprecipitation is that of pH while the factors governing flocculation are both pH and the presence of deflocculating agents. The influent is known to contain sterox, tetrasodium pyrophosphate and citric acid.

The isoelectric point for precipitation of aluminum hydroxide is a pH of 8.8. This will, however, vary with the concentration of electrolytes in the solution. It has been found that good coagulation in the waste solution is obtained at a pH above 10 while the more complete coprecipitation is at a pH of 9. This conclusion is arrived at because of the decrease of activity in the supernatant and an increase of activity in the filtrate at a higher pH.

The sterox was found to have little effect upon the flocculation of the aluminum hydroxide. It would, however, in the presence of oil form an emulsion which could cause deflocculation. With the exception of the first few weeks of operation no appreciable quantities of oil have been observed. This, however, still remains as an important factor. The tetrasodium pyrophosphate is a deflocculating agent. The aluminum hydroxide which forms in the presence of this compound remains suspended in the solution as very fine particles. The pyrophosphate can be removed by addition of calcium chloride which forms the insoluble calcium pyrophosphate. In the case of citric acid no definite solution has been found. The citric acid forms a soluble complex with the aluminum hydroxide. It appears that this effect can be reduced by increasing the pH to 10 or above.

Some preliminary work has been done using ferrifloc in place of alum. The advantage of using ferrifloc would be precipitation at [illegible] pH. It would also permit a wider variation in the pH during pr[illegible] which is not now possible.

As an expedient method the clariflocculators were connected in series. A few preliminary experiments had indicated that a double precipitation would result in a more filterable material going to the sand filter. At about the same time calcium chloride was added to each influent solution and the pH controller set a 8.7. Data obtained with this method of operation has indicated that the filtrate from the filtration of the effluent from either clariflocculator is the same, however, the count in the effluent before filtration is higher in the first clariflocculator than in the second one. With this procedure there has been a greater build up of the sludge in the first unit. With the secondary process now in operation there will be a concentrated effort to lower the sludge bed in this unit. When the bed has been sufficiently reduced the clariflocculators will then be operated in parallel.

Secondary Concentration Step - The two problems in the secondary process were the low capacity of the Eimco filter due to a plugging of the nylon cloth, and the large amount of insolubles present after digestion of the aluminum hydroxide cake. The capacity of the Eimco filter was increased from 12 lbs./hr. to 30 lbs./hr. by revising the unit to serve as a precoat filter. A 60 x 350 mesh stainless steel cloth was used in place of the nylon cloth. A gear arrangement was used to move the knife blade a definite small distance with each revolution of the drum so that the cake and a small amount of filter aid were removed to give a clean surface with each pass of the drum through the slurry.

To take care of the large amount of insoluble material remaining after dissolving the cake, nylon filter bags are being used in place of "Filtros" cartridges. Not only is the cost reduced by one-twelfth but also the volume occupied by the bags in a drum is much less than with the cartridges. An analysis of the insolubles will be made to determine whether this volume can be further reduced.

Combustible Waste Disposal - H. Morris and P. Schauer

In the work carried out at Unit III it is believed that very little if any actual removal of activity in the steam-ejector nozzle was being accomplished by sudden condensation of the supersaturated steam on particles of activity in the flue gases. It is suspected that the reaction in the nozzle approached that of a spray nozzle but of higher efficiency than that normally obtained in commercial spray nozzles or atomizers.

An experimental steam nozzle has been designed and is under construction. The nozzle is to be used to substantiate the work of Yellot (Trans. of Am. Society of Mech. Eng., Supersaturated Steam, June, 1934, pp. 412-30) and to investigate the condition which results from passing the steam through two orifices which in effect may result in expandion and recondensation. It is hoped by this method that more effective use can be made of the sudden condensation of the steam in a supersaturated state.

The nozzle is to be provided with a transparent side through which the expanding steam can be observed with the aid of an intense beam of light passing axially through the nozzle. The size of the orifices can be varied by means of adjustable brass blocks.

The experimental incinerator design has been based on the down-draft principal. The feed is at the top of the unit and is provided with air-lock system for introductory of the contaminated material into the combustion chamber. A stainless steel liner wrapped with nichrome wire is to provide the means for ignition. The ash is to be collected in an ash hopper at the bottom of the combustion chamber from where it can then be dropped into drums for a possible further treatment such as leaching the activity with acids

Decontamination by Means of Ultrasonic Energy - R. Brodbeck and G. Sandmann

In the past glassware and quartz has been decontaminated by immersion in hot chromic acid or boiling in aqua regia for periods up to five or six hours. This method has been satisfactory in removing the activity from glassware such as beakers, but ground glass or quartz, such as stopcocks or ground glass joints cannot be satisfactory decontaminated by this means.

The term "ultrasonics" is used in acoustics to denote the frequencies which are beyond the limits of hearing of the human ear, i. e., about 20 kc. and up. Ultrasonic vibrations may be produced as high as 5×10^5 kc. Expressed in wavelength this means that ultrasonic waves in air (velocity = 330 m./s.) cover a range of 1.6 cm. to $.6 \times 10^{-4}$ cm., in liquids (velocity $\approx$ 1200 m./s.) the range of 6 cm. to 2.4×10^{-4} cm., and in solids (velocity $\approx$ 4000 m./s.) the range is from 20 cm. to 8×10^{-4} cm. The shortest ultrasonic waves are therefore already in the order of magnitude of the wave of visible light.

By reason of the small wave length it has been found possible to make use of ultrasonics in various fields. Many investigations of sound vibrations, and particularly measurements of velocity, may be had in much smaller space by the use of ultrasonics than with audible vibrations. The laws of sound, valid for the audible range, are also true for the ultrasonic range, though in the latter case further phenomena appear which have not previously been observed in the audible range.

The application of ultrasonic techniques to the decontamination problem is an outgrowth of the theory that the nature of the forces binding polonium to the glass is similar to that of the chemical bond, but is less intense. It is readily seen that in order to thoroughly decontaminate an article, this binding force must be overcome.

This has been accomplished by using frequencies of 100 cycles to 20 kc at a power output of from 2 to 20 watts passing through a citric acid solution (approximately pH-2) in which the contaminated article is immersed. The optimum results using a tank with the dimensions 7" x 7" x 7" and 2 liters of solution has been found at 200 cycles or harmonics of 200 cycles. Citric acid was used because of the fact that it forms a soluble complex with polonium. It has been observed that only solutions containing citric acid have shown any favorable results.

Tests of this system to date have shown an average of 65 per cent removal of activity. It has been found by Wood and Loomis that intense ultrasonic waves transform immiscible liquids such as water and oil or water and mercury, into very stable emulsions. The simplest explanation of this phenomenon is that particles of the heavier liquid are driven by the ultrasonic wave into the overlying liquid.

It has also been found that with the application of very high intensity ultrasonic energy at a frequency of 400 kc., true colloidal solutions were obtained with S, Pb, Sn, and Bi with particles as small as 10^{-6} cm. and showed a strong Tyndall effect. If the suspended particles have the form of rods or discs, ultrasonic waves of medium energy have a directive effect on the particles.

Important progress in ultrasonic dispersions of metals in liquids was made by Claus. He has found that if a cathode is exposed during electrolysis to ultrasonic energy, the metal deposited there, which is already finely divided, goes into the liquid in a highly dispersed form.

The biological effects of ultrasonic waves have been the subject of numerous investigations.

Harvey has observed the action of ultrasonics under the microscope, and found that cells, at first round, were distorted and stretched, losing their color in the process, and finally broken up and dispersed into a number of small spherical particles. He also found that the protoplasm or chloroplasts could be separated from the cell wall and coagulated elsewhere.

It is planned to make a cursory investigation of existing ultrasonic techniques and physical properties, such as methods of measuring energy level in the solution, power absorption, scattering of energy and velocities. It is not deemed necessary to investigate these properties from the standpoint of pure physics but rather to gain a general background of techniques.

A thorough investigation of projectors is proposed, which will include the use of piezo-quartz crystals, ammonium dihydrogen phosphate crystals, magentostriction oscillators, and rochelle salt crystals. This will involve methods of maximum transfer of energy through liquid media.

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Intensive tests of frequency, power output, and solar concentrations will be necessary when a prototype equipment is designed.

FUTURE PLANS

Liquid Waste Disposal

The immediate objective in the operation of the waste disposal process is to return to a parallel operation of the clarifloccolators. This depends upon reduction of the sludge level in the first unit. Also an elimination of the gassing which occurs in this unit may be necessary. Steps are being taken to determine and eliminate the source of this gas.

A more long-range program is an investigation to determine the advisibility of using ferriflos in place of alum as the precipitating agent. Two advantages that can be cited are: operation at a higher pH and possible wider variation in the pH during precipitation. The first point is important so far as a reduction in the complexing effect of the citric acid.

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