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CENTRAL FILES 60-7-17

COPY No. _____

Contract No. W-7405-eng-26

HEALTH PHYSICS DIVISION

STUDIES OF THE USE OF COAGULANT AIDS IN THE LIME-SODA TREATMENT
OF LARGE-VOLUME, LOW-LEVEL RADIOACTIVE LIQUID WASTE

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DATE ISSUED

AUG 22 1960

Oak Ridge National Laboratory
Operated by
Union Carbide Corporation
For
U. S. Atomic Energy Commission
Oak Ridge, Tennessee

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found to affect coagulation. While the combination of aids used gave a rapidly settling floc, a drop in water temperature produced an increase in the amount of very fine particles having slower settling rates. Coagulant aids do not work well if the system contains complexing agents, such as versene and polyphosphates. Minor modifications in the plant will be necessary to get the best results.

Introduction

One of the major problems affecting nuclear energy installations is the safe and economical disposal of low-level radioactive wastes. Chemical treatment of the wastes by precipitation of the radioactive contaminants and subsequent removal by settling is one method for the decontamination of large-volume, low-level radioactive wastes. At the Oak Ridge National Laboratory, decontamination of 200 to 300 million gallons of process waste water per year, containing 200 to 500 curies of radioactivity, is accomplished by use of the lime-soda treatment process.^{1,2} The present paper deals with studies on the use of polyelectrolytes as coagulant aids to improve the efficiency of removal of radionuclides from the treated wastes.

The ORNL Process Waste Water System

Process wastes originate from equipment cooling water, floor drains, decontamination pad drains, storage canals, laboratory sinks, and miscellaneous low-activity operations. These wastes are conveyed by gravity through vitrified clay pipes to a central monitoring point from whence they are diverted either to the settling basin or to the equalization basin. A flow diagram of the system is shown in Fig. 1.

The ORNL Process Waste Treatment Plant is designed to handle up to a maximum of 0.5 million gallons per day (mgd) or 350 gpm. The plant closely resembles a conventional water softening plant, having the usual softening equipment and using standard softening chemicals. The plant is

Fig 1 Flow Diagram of Process Waste System

designed to reduce calcium carbonate in the effluent to 30 to 35 ppm with a maximum value of 50 ppm.²

Lime-soda treatment was chosen for the following reasons:

1) potential capability of removing more than 90% of radiostrontium and the rare earths; and 2) flexibility of operation under varying conditions of influent water quality, with the pH ranging from 2.0 to 10.0.

Increased removals of radiostrontium by the lime-soda process were found to be related to: 1) reduced concentration of calcium in the effluent, 2) excess dosages of lime and soda-ash (above stoichiometric quantities), and 3) successive precipitation of calcium in the presence of preformed calcite crystals.

Pilot plant tests with various types of coagulation and sedimentation units led to the design and construction of a horizontal-flow unit with provisions for alternative use of phosphate coagulation for radiostrontium removal and the addition of illite clay for removal of cesium. Provisions for recirculating the effluent for repeated treatment were also included. The following standards for chemical feed were established during full-scale operation of the plant and are still in use:

1. Lime - stoichiometric quantity
2. Soda-ash - 200 ppm in excess of stoichiometric amount
3. Grundite clay - 200 ppm (Grundite is a clay product containing 65 to 75% illite, 15 to 25% kaolinite, and 5 to 15% quartz, used mainly to remove cesium by sorption).

Under these conditions, the pH of the treated waste water is about 10.5, and removals are of the order of 80 to 90% for radiostrontium and about 90% for radiocesium.

Scope of the Present Study

Routine operation of the treatment plant is beset with some inherent difficulties. Low temperatures influence the removal of calcium; that is, the amount of calcium in the effluent increases significantly during cold weather. Increased time of contact produces an increased removal of calcium up to 50%, either in solution or in fine colloidal suspensions. It is not possible to increase contact time in the plant, since the designed retention time in the settling basin of the plant is only 129 minutes. Recycling the previously settled sludge from the settling basin back to the flash mixer is effective in reducing the amount of calcium carbonate in the effluent and can be practiced. Also, during cold seasons, the amount of suspended matter in the effluent increases. Complexing agents, such as versene and polyphosphates, are found to interfere with the treatment process, resulting in a significant increase in effluent hardness. Although the percentages of magnesium hydroxide and calcium carbonate in the suspended matter are difficult to evaluate, it is possible to estimate the distribution of activity in the solid as well as the liquid phase of the effluent. Gross beta counting of the filtered and unfiltered weekly composite samples of the effluent show that, on an average, 50% of the outgoing activity is associated with the solid phase.

The use of coagulant aids is one approach to the removal of suspended matter in the effluent. The slight increase in the cost of treatment might be justified on the basis of increased efficiency. Accordingly, the present study was made in order to select a suitable coagulant aid and to determine the conditions under which the aid would be more effective. As the current interest is in the use of polyelectrolytes as coagulant aids, the study was limited to polyelectrolytes only.

Coagulation

The use of coagulants and coagulant aids in water softening processes is widespread. The subject of coagulation itself, in its application as a means of decontaminating radioactive liquid wastes, has been treated extensively by several authors.^{3,4,5,6,7,8,9} Black describes the action of a coagulant when added to water as one involving three stages:¹⁰

1. The coagulant disperses itself in the solution and produces multivalent ions which serve to neutralize the negative charge on the color or turbidity colloids under proper conditions of pH and alkalinity.
2. The colloidal particles resulting therefrom lose their ability to repel similar colloids and may under proper conditions of pH acquire an opposite charge. This may permit the interaction by which one colloid neutralizes another in solution, the second stage of coagulation.
3. The third stage of coagulation consists in "conditioning" the colloidal forms to a point where they may cluster together and agglomerate into particles of a size suitable for settling.

The first stage of coagulation called "electrokinetic coagulation" has been presented by Kaufman⁷ and Nesbitt.⁴ This involves the neutralization or dispersal of charge of the zeta potential of the hydrophobic colloid by adding an electrolyte or oppositely charged ions to a point where the repulsive forces of stability of the colloid are decreased below a critical level at which the London - van der Waals forces serve to accomplish the agglomeration of the primary crystallinities.

The second and third stages of coagulation are termed "orthokinetic coagulation".⁷ Here the primary crystallinities aggregate to form larger lumps or submicron particles, and the predominant forces acting are the London - van der Waals forces. These second order forces between the molecular disperse systems are attractive forces believed due to the dipole moment of the molecule or the polarization of an atom by fluctuations in charge distribution in a second atom. Since the London - van der Waals forces of attraction vary inversely as the seventh power of the distance of separation of the particles, it is necessary that the particles approach one another closely for these forces to operate.

In recent years, a number of polyelectrolytes have been found to possess very good properties as aids for coagulation. These polymeric electrolytes, called in short polyelectrolytes, contain recurring units of small molecular weight chemically combined to form molecules of colloidal size, with each of the recurring units carrying one or more electrical charges or ionizable groups. Michaels¹¹ suggests that the most effective polyanionic polymers contain a mixture of ionic and unionized hydrophilic groups. The hydrophilic groups are adsorbed on

the particle surface while the ionized groups keep the polymer in an extended position, leading to particle bridging and agglomeration.

Experimental Method

The study of coagulant aids was divided into two main phases, namely:

1) laboratory tests to compare and select the most suitable aid and to evaluate the best possible conditions under which it will work and 2) plant trials to obtain data on plant performance of the coagulant aid selected.

Laboratory Tests

For investigation in the laboratory, samples of a number of coagulant aids were procured and tested by the conventional jar-test technique.¹² Table 1 lists these products by their trade names and manufacturers. Although the chemical compositions of these coagulant aids were not supplied, three were probably polyanionic electrolytes, possibly polyacrylamide. Hagan Nos. 7, 11, and 18 are mixtures of a polyampholyte and bentonite clay in varying proportions.¹⁶ The nature of the other aids is unknown.

Apparatus and Procedure.— The conventional jar test, described by Cohen^{12,13} was used for evaluating the performance of the aids. The stirring device used, Fig. 2, is a ten-place laboratory stirrer designed at ORNL and described by Straub and Hensley.¹⁴ A suction sampler was assembled from Pyrex tees and Tygon plastic tubing such that samples could be taken from a set of five jars simultaneously at intervals of 1, 2, 3, 5, 10, and 30 minutes after settling. Since no change in the treatment

Table 1. Coagulant Aids Tested by the Conventional Jar-Test Technique

Trade Name	Form	Manufacturer
Hagan 7	Gray granules	Hagan Chemicals and Controls, Inc.
Hagan 11	-----do-----	-----do-----
Hagan 18	-----do-----	-----do-----
Hagan 50	White powder	-----do-----
Hagan X 323*	-----do-----	-----do-----
Separan 2610	White powder	Dow Chemical Company
Aerofloc 552	Dull yellow powder	American Cyanamid Company
Aerofloc 3000	White powder	-----do-----

* Material supplied as solution. Physical form unknown.

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Fig. 2. Laboratory Stirring Device.

procedure was contemplated, it was necessary to duplicate the plant conditions in these jar tests. Accordingly, the following time and speed sequences were used in the tests:

1. 85 seconds flash mixing at 288 rpm
2. 31 minutes coagulation at 81 rpm.

These time and speed sequences are typical of those in the flash mixer and the three coagulation basins, respectively. At 81 rpm, the stirrer used gave a paddle tip velocity of 64 feet per minute.

Turbidity measurements were made with a Klett-Summerson photo-electric colorimeter, using a 40 mm glass cell and a blue filter having a range of 400 to 465 millimicrons. Since the scale of this instrument is divided logarithmically, it was possible to relate the initial and final turbidities and to determine the percentage of turbidity remaining. Nevertheless, the instrument was calibrated with standard solutions of fuller's earth turbidity, and the resultant curve was used in the calculations.

A continuous sampler was set in the plant influent line to yield a 24-hour sample of influent waste water, and this was used in the tests. Analysis of the waste water for total, carbonate, noncarbonate, calcium, and magnesium hardnesses was made in accordance with standard procedure.¹⁵ With Model G Beckman pH meter, pH determinations were made. From these figures, dosages of lime and soda-ash solutions were calculated:

Lime	10 grams per liter
Soda-ash.....	50 grams per liter
Grundite clay (< 250 mesh).....	40 grams per liter

Filtered samples of the treated waste water, under the conditions mentioned on page 3, gave a final pH of 10.5.

The concentration of aids in stock solutions ranged from 0.25 grams per liter to 2.0 grams per liter. These concentrations were decided upon from consideration of the dosages suggested by the manufacturers.

A few exploratory tests were necessary to decide on the type of aid to be selected, its optimum concentration range, and the best combination. From these tests it was possible to eliminate some of the aids and some specific combinations. Hagan 50, Separan 2610, and Aerofloc 3000 were found to work better than the other aids tested. Combinations of these aids with Hagan 18 produced even better results. Thus, these combinations were used for detailed study.

Figure 3 gives the settling curves obtained for the combination of Hagan 50 and 18 and for Separan 2610. It is seen that an increase in the dosage of Separan 2610 from 2.0 ppm to 3.0 ppm produced very little change in the settling characteristics of the floc and in the final turbidity of the supernatant solution. The same effect is also seen for the combination of Hagan 50 and 18 in spite of the fact that the floc size for 0.75 ppm of Hagan 50 and 5.0 ppm of Hagan 18 was much larger than that for 0.5 ppm of Hagan 50 and 5.0 ppm of Hagan 18. The curves show a wide range of particle size distribution for coagulation without any aid, and a floc of more or less uniform size with good settling characteristics for coagulation in the presence of an aid. These tests were carried out at 25°C.

Figure 4 shows the settling curves for 0.5 ppm of Hagan 50 and 5.0 ppm of Hagan 18, 2.0 ppm of Separan 2610 and 2.0 ppm of Aerofloc 3000. It is seen that the performance of Aerofloc and that of Separan 2610 were

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Fig. 3.

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Fig. 4.

close to each other with the difference that Aerofloc produced a wider range of particle sizes, yielding the sloping curve. Here again, the combination of Hagan 50 and 18 gave better results than the others.

Combination of Aids.- Another test was performed to see whether Hagan 18 would give the same supporting effect for Separan 2610 and Aerofloc as for Hagan 50. The resulting curves are shown in Fig. 5. The floc size for Aerofloc 3000 plus Hagan 18 was much larger than the other combinations, and the final turbidity was low. This series of jars also contained one jar in which both Separan and Aerofloc were used together. This combination produced an adverse effect on the settling characteristics of the floc as compared with the other combinations.

Effect of Agitation.- Figure 6 shows the settling characteristics of the floc obtained for the combination of Hagan 50 and 18 for what is termed "slow agitation." The time and speed sequences were as follows: 108 rpm for 3 minutes, 54 rpm for 5 minutes, 36 rpm for 10 minutes, and 30 minutes of settling. The results of this test gave settling curves for the same combinations with "fast agitation." Comparing these results with those shown in Fig. 3, it can be seen that "slow agitation" gave somewhat higher turbidities for the supernatant. However, the floc size was much larger at slow speeds than at high speeds.

Effect of Temperature.- Two tests were conducted to determine the effect of temperature on coagulation. In both cases, the samples were immersed in an ice bath and cooled to 40°F before the addition of lime, soda-ash, and clay. Figure 7 gives the results of the first of these tests with the "fast agitation" sequence, and Fig. 8 shows the corresponding results for the "slow agitation" sequence. The curves show

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Fig. 5.

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Fig. 6.

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Fig. 7.

Fig. 8.

that the aids did produce appreciable coagulation at low temperatures. It will also be noted that the slow speed sequence produced a higher turbidity than the high speed sequence.

Point of Addition.- Figures 9 and 10 show the results of tests where the aids were added before and after the other chemicals. The aid combinations used were Hagan 50 and 18 and Aerofloc 3000 and Hagan 18. At least in these jar tests, there seemed to be little or no effect on coagulation whether the aids are added before chemical treatment or after. However, more tests must be conducted before this effect can be determined conclusively.

Presence of Complexing Agents.- In the various services and laboratories at ORNL, it is common practice to use Turco and versene as decontaminating agents for cleaning surfaces, equipment, and clothing. Contaminated water resulting from such clean-up operations is released to the process waste water system. Very often, the amount of Turco in the influent waste stream is high enough to cause interference in the treatment process. It was of interest, therefore, to determine whether the aids would be as effective in the presence of Turco.

Tap water was spiked with varying amounts of Turco ranging from 100 ppm to 500 ppm, and coagulation was accomplished with the Hagan 50 and 18 combination. Two control jars, unspiked with Turco, were used, one with, and the other without the aids.

Tap water spiked with versene ranging from 10 ppm to 50 ppm also showed more or less the same effect as with Turco. Because versene is used in the complexometric titrations for hardness estimations, it was not possible to evaluate the final hardness of the treated water.

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Fig. 9.

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Fig. 10.

The jar tests showed that the use of coagulant aids does reduce the turbidity of the treated waste solution and that the combination of Hagan 50 and 18 produces uniformly good results under most of the conditions described above. Since this combination seemed well suited to the conditions existing in the treatment plant, a six-week plant trial was planned for further testing of these aids.

Plant Trials

The plant trials showed that it would be possible to obtain, under normal conditions, a good-sized floc of fairly quick settling characteristics by the use of coagulant aids Hagan 50 and 18. There are, however, several features which differentiated the plant trials from the laboratory jar tests. Variation in temperature had a more drastic effect on the size of the floc. The fact that complexing agents were present in the system during the plant trials had to be noted in judging the performance of the coagulant aids. Choice of the proper feed concentration for the aid solutions, point and mode of addition of the aids, and careful control of agitation were contributory factors to floc formation. Also, some mechanical modifications were necessary to get the best possible results.

Reference to some pertinent details of the plant equipment is necessary here. Culbreath² describes the details of the many features of the ORNL plant. The detention times and agitator speeds in the various units of the plant are as follows:

	Detention Time	Speed of Agitation (rpm)
Flash Mixer Basin	85 seconds	288
Coagulation Basins	31 minutes	81
Settling Basin	129 minutes	--

Both the flash-mixer equipment and the standard Vorti-Floc coagulators used in the plant were supplied by Infilco, Tucson, Arizona. The flash-mixer basin and the three coagulators are square in shape, separated by wooden baffles and provided with concrete fillets on all four sides of the bottom to prevent sludge accumulation. The flash mixer has a fixed-speed agitator, and each of the three Vorti-Floc units are driven by variable speed motors. The Vorti-Floc consists of two impellers, each of which is a horizontal plate with vertical blades supported by and fastened to the vertical shaft. The blades are designed to produce a vortex action in the liquid.

Two Transite baffles separate the third coagulator basin from the settling basin. Each of these baffles has ten holes, 6 inches in diameter, arranged in five rows of two each. At 350 gpm flow rate, the velocity of flow in each of the ten holes is 10.7 fpm.

A traveling Link-Belt sludge collector is used for sweeping the settled sludge into the sludge hopper.

Solutions of the aids, Hagan 50 and 18, were made and fed separately into the flash mixer. Two 55-gallon stainless steel drums equipped with Lightnin' mixers, were used for making the solutions. The solutions were drawn through Tygon tubing passing through Sigma pumps. By taking the tubes underneath the catwalk, where there are steam lines, freezing in cold weather was prevented. Feed rates of the solutions were controlled by regulating the speed of the Sigma pumps. Rotameters installed in the discharge of the pumps measured the flow rates.

The following concentrations were used at the start of the plant trials:

Aid No.	Suggested Dosage (ppm)	Dosage Used (ppm)	Feed Solution Concentration	Maximum Suggested Concentration
Hagan 50	0.25 to 1.0	0.5	0.5%	0.5 to 2.0%
Hagan 18	3.0 to 15	5.0	3.0%	3.0%

The containers for Hagan 50 were filled once each day and for Hagan 18 twice a day. After the start of the experiment it was found that a 3.0% solution of Hagan 18 was too viscous. Therefore, the concentration was reduced to 2.0% with three fillings per day. To prepare the solutions, the drums were partially filled with water, and the aids were added with the agitators "On". Shortly thereafter the remaining volume of water was added. Aid 50 dissolved completely in about one hour, and no further agitation was required. Hagan 18 required constant agitation.

In the beginning, the aids were fed independently into the flash mixer. After sometime it was evident that this point of introduction was not the most effective, and so Aid 50 was split into two portions, one going to the flash mixer and the other to the third coagulator basin. Aid 18 was fed into the flash mixer only.

The results were apparent 48 hours after the initial feeding. There was marked floc production in the coagulator basins. At the outset the floc size did not improve in the second and third basins but appeared to break up. To prevent this, the speed of the agitators was progressively reduced to 50 rpm in the second basin and to 30 rpm in the third basin.

This amounted to paddle tip velocities of 393 and 245 fpm, respectively. Also, the floc particles that came to the surface in the settling basin were much smaller than those in the third coagulation basin. To rectify this, the Transite baffles were interchanged with wooden baffles having 7-inch slots instead of 6-inch holes. This reduced the velocity of flow through the baffles from 10.7 fpm to 4.5 fpm.

Supplies of Hagan 18 ran out after 3 weeks. Until fresh supplies could be received, Hagan 11 was substituted for 2 weeks, since it was known that Aid 18 and 11 were the same material but of different compositions. An initial dosage of 1.5 ppm of Hagan 11 was later reduced to 1.25 ppm with better results. Hagan 11 produced a floc comparable to that of Hagan 18 (both in conjunction with Hagan 50), but the over-all results favored the use of Hagan 18.

Several difficulties were encountered in feeding the aids at a constant rate. The high viscosity of the solution of Hagan 18 placed a limit on the concentration used. Also, there was an accumulation of fine grains of sand in the Tygon tubing and in the valves. These particles of sand may have come from the bentonitic clay used in the preparation of the acid. Periodic flushing with water helped to keep the tubing free of this accumulation. The Sigma pumps used were very unsatisfactory, because at night when the temperature dropped considerably, the Tygon tube became rigid and the flow extremely low. Hagan 50 was found to contain some impurities which built up gradually in the tube and in the rotameter. Flushing with water did not help. Since the solution of Hagan 50 was highly viscous, even at a concentration of 0.5%, the aid did not distribute

itself quickly enough. The only way this could be corrected was to use a more dilute solution.

Conditions in the plant were not uniform and normal during the trials. During very cold days, there was a marked decrease in the floc size and an increase in the amount of carry-over of solids in the effluent. Increasing the dosage of Hagan 50 from 0.5 ppm to 0.75 ppm and splitting the aid solution by feeding part of it into the flash mixer and the rest into the third basin helped to overcome the effect of temperature somewhat.

During the plant trials it was noted that waste solutions containing Turco, a proprietary product used in decontamination operations elsewhere in the Laboratory, were let into the equalization basin and finally into the treatment plant. Analysis of the influent for phosphates showed amounts ranging from 10 to 25 ppm. During continuous operation of the plant it was rather difficult to estimate exactly when these phosphates were coming into the plant. The poor performance of the aids is believed to be due to the influx of Turco into the plant, and would be so expected, based on the results observed in the jar tests.

Mechanical modifications of the plant produced some undesirable results. Slowing the agitators in the coagulators progressively, so that the velocity through the wooden baffles separating the third basin and the settling basin was reduced, resulted in a considerable sludge build-up in the third basin. Since the settled sludge was closely packed, washing the basin with a water jet did not improve matters. The agitation speed was increased for about 30 minutes to stir up the sludge once again, and this destroyed the floc already formed. Also, the horizontal slots in the

wooden baffles short-circuited the sludge back into the third basin. Circular holes, larger than those in use previously, might help to overcome this effect.

There was a significant reduction in the turbidity of the effluent as a result of coagulation with an aid. That the aids do produce a good floc of fairly uniform size and quick settling characteristics, even at low temperatures, was very evident in these trials. Analytical data on several weekly samples confirmed the beneficial effects of the aid. However, results were inconsistent, probably due to the uncontrollable factors described previously. A much longer observation period, including work with equipment to study the particle-size distribution, is necessary to evaluate properly the effect of coagulant aids on the removal of radioactivity from low-level liquid wastes.

Summary

The results of the jar tests and the plant trials were as follows:

1. The combination of aids Hagon 50 and 18 produced uniformly good results under normal operating conditions. A dosage of 0.5 ppm of Aid 50 and 5.0 ppm of aid 18 seems to be the optimum dosage. During cold weather it was necessary to increase the dosage of aid 50 to 0.75 ppm. The upper limit for aid 18 was 5.0 ppm.
2. Combinations of Hagon 18 with Separan 2610 and Aerofloc 3000 are promising.
3. For better mixing it is advisable to use a feed concentration of less than 0.5% for aid 50 and about 1.0% for aid 18. Dry feeding of aid 18 is desirable.

4. Some structural modifications are necessary in the plant for better results.

5. The aids are not effective in the presence of Turco and versene.

Acknowledgment

The authors wish to acknowledge with thanks the assistance rendered by Harvey J. Wyrick and R. L. Newton in the plant trials.

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