Review of the Soap Association's Research Activities Pertaining to Water Supply and Sewage Treatment[1]

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FROM A GLANCE at our growing population, estimated to reach 220,000,000 in 1975, and the relatively constant volume of available surface waters, it is obvious that there are many challenging problems ahead for the sanitary engineer involved in developing adequate supplies of good water for the future. Improved waste disposal and treatment can help meet this challenge.


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The interests of the soap industry in cooperating in the solution of water supply and waste-water-disposal problems goes back a good many years and antedates the introduction of detergents. At the present time the Soap Association is happy to be supporting one of the research programs being set up by industry and by federal, state, and local agencies to deal with this broad question, but it does not overrate the relative importance of the detergent question among the many problems which the sanitary engineer faces.

Detergents are only one of the many substances which may find their way into surface waters from sewage or sewage effluent, from industrial wastes, from land drainage, or from the natural plant and animal processes which take place in surface waters. Reports of an installation on the Virgin Islands to distill fresh water from sea water and predictions that in the future waste heat from atomic reactors might be utilized for distillation give an idea of the scope of developments being directed toward the supplying of water for future needs. Even in this context the questions raised about detergents are deserving of attention.

The Association's research program deals with questions raised about the possible effects of the normal household use of detergents by the American public. There is considerable literature implying that the increased use of detergents is associated with the problems of frothing in some sewage treatment plants. In the literature it has been suspected that detergents might retard the biochemical processes in sewage treatment, might otherwise adversely affect efficiency of sewage treatment plants, and might pass unchanged from sewage treatment into surface waters. In water treatment, questions have been raised about sedimentation and coagulation, and frothing. These are queries to which our program has been directed so far.

Haney in 1954 (1) listed the various instances, reported in the literature, of trouble which has been associated with detergents and outlined the areas in which the Task Group on Synthetic Detergents of the A.W.W.A. thought research should be done.

[1] Dinner lecture.

Those who have expressed the belief that detergents will give the sanitary engineer serious trouble point to the amazing increase by the American public in detergent consumption. They suspect that the increased use is related to an apparent increase in problems encountered. On the other hand, sewage and water-works people had encountered the same type of problems before the advent of detergents, including frothing in sewage-treatment plants. Thus it is reasonable to expect that even if household detergents had not been invented, by now the number of such problems would be greater merely because the population has increased and particularly because the number of activated-sludge type of sewage-treatment plants has increased more rapidly even than the population.

With this background the Soap Association research program was set up to determine general problem areas, to define specific questions in a quantitative manner, to obtain data to contribute toward the understanding of the problems, and to take the lead in finding solutions if any real detergent problems were uncovered.

Several reports of the progress of the Association's research program have been made, which give more detailed descriptions of the various phases of the work (2, 3, 4, 5).

Our research program encompasses both of the major components of household detergents, the surface-active agent and the complex phosphate builders. Since alkyl benzene sulfonates (ABS) of the polypropylene type represent possibly 60% to 70% of all of the surface-active agents used in household detergents and since they stay popular with the consumer, our investigations of surfactants have concentrated chiefly on this important commercial material.

By way of definition, the term "detergent" is used in this discussion to refer to the complete product as it is found in the package on the grocery shelf or in barrels or drums for general bulk industrial uses. It is not used to refer to the surface-active component.

The Research Program

A. Research, Internal and External

Some of the Soap Association's research is being carried on internally by technical committees although a large part is being done externally, chiefly on the basis of grants to universities. For the period July 1, 1955 to July 1, 1957, $128,715 was spent on an outside research program; and for the period of July 1, 1957 to July 1, 1959, the Association's board of directors approved a budget of $118,000 for continuation of the outside research. While most of the internal manpower and the money for research came from member companies, since the organization in 1955 of the Subcommittee on Phosphates the Association has had the helpful support of several companies in the phosphate industry who are interested in participating in this program but who are not Soap Association members. The internal research, done by the Soap Association technical committees and not included in the research budgets mentioned above, represents a considerable additional contribution from the various member and nonmember companies who are supporting this program.

B. Analysis of Water and Sewage for ABS

Water. The need for reliable methods of analysis to measure the extremely low concentrations of ABS or other surfactants in water and sewage was recognized early by the American Water Works Task Group, by the Ohio River Valley Water Sanitation Commission, the U. S. Public Health Service, and, of course, the Soap Association. Without good analytical tools any research project will bog down. The colorimetric procedures in general use are not specific in that many substances, including urine, bile salts, tea, coffee, and distilled water extracts of such things as straw, leaves, and rotted wood, interfere and give erroneously high results. One of the highly surface-active bile salts, "sodium taurocholate," is, as a matter of fact, a sulfonate. Because of the weaknesses of the colorimetric procedures the Soap Association group early in the game gave top priority to the analytical problem. It was agreed that results would be obtained more quickly if this work were done internally by a Soap Association technical committee.

After several years of cooperative work the Subcommittee for Analysis of ABS published in December 1956 (6) the details of a method which more accurately measures low concentration of ABS that might be present in some surface waters. The new method, which followed leads reported by the U. S. Public Health Service, involves the removal of the ABS by adsorption on an activated carbon column. Adsorption on carbon begins to separate the ABS from the larger amounts of other organic matter present. The removal of the ABS from the carbon is followed by further purification by chemical methods. By obtaining quantitatively a sufficient amount of pure ABS from a given sample, it is measured qualitatively and quantitatively by the infrared spectrophotometer.

Sewage. The method developed for successfully analyzing water has been discarded for use with sewage in favor of a simpler extractive method, eliminating the adsorption and desorption on carbon. It was possible to do this because the higher concentration of ABS in sewage (1–10 p.p.m.) compared to about 0.05–0.2 p.p.m. normally found in surface waters made it feasible to concentrate ABS by evaporation and extraction. The program for the future is a) to perfect the procedure now under study, and b) to proceed to cooperative testing. It is hoped that the work will be completed in the coming months.

C. Shorter Methods

The infrared method is admittedly too complex for routine use in water and sewage treatment plants, and thus a shorter method is still needed. On the recommendation of the Analytical Committee, the Soap Association placed a fellowship at the University of Southern California under the direction of Prof. O. H. Miller.

The possibility of finding ABS-dye complexes, the solubilities of which are sufficiently different from interfering substances to permit selective extraction, is being explored. While this project has only been operating since August 1, 1957, the first phase of the investigation is near completion. Already the behavior of a large number of the ABS-dye complexes has been systematically explored, both in the presence and absence of certain interfering substances. Several combinations of dyes and solvent have been found which show excellent promise. Also other organic reagents which complex with ABS will be studied. The progress of the work on this project has been remarkably good.

From work on the determination of ABS it became apparent that ABS is only a part of the total organic matter of surface waters and sewage, and thus there is a great need for analytical methods to measure qualitatively and quantitatively the other organic compounds present in order to understand the part they may play in these questions.

Analysis of Surface Waters for Phosphates. Another internal project is the development of reliable and practical methods for measuring and differentiating between the various types of phosphates in surface waters. The speculation that household detergents might affect coagulation and sedimentation in water treatment first focused on the surface-active agent. When it became apparent that this hypothesis was not sound, the speculation shifted to the complex phosphates. Thus methods of analysis were needed which would permit answers to such questions as "is the concentration of complex phosphates present in the surface water supply of a water treatment plant sufficiently high to produce adverse effects?" To answer this question it is necessary to know how much of the various types of phosphates is present.

The Subcommittee on Phosphates has developed new analytical procedures that are scheduled to be published this year. In this work the committee has made a significant contribution to the knowledge of phosphates in surface waters by the discovery and recognition that organic phosphates, presumably from natural sources, are usually a significant part of the total phosphate present. By the methods ordinarily used for water analyses any natural or other organic phosphates would show up erroneously as inorganic polyphosphates. The new method avoids this error by including a step which determines the total phosphate present by a "wet-ashing" procedure. This total phosphate is the sum of all orthophosphate, all inorganic polyphosphate, all hydrolyzable organic polyphosphate, and all non-hydrolyzable organic phosphate. Another step involves boiling the sample with aqueous acid and obtains the first three above. In still another step orthophosphates are determined by a refinement of standard procedures. These three determinations permit the calculation of a) total phosphate, b) orthophosphate (as in earlier methods), c) hydrolyzable phosphate which is the sum of the hydrolyzable organic and inorganic polyphosphate, and d) nonhydrolyzable organic phosphate.

D. Water Treatment—Coagulation and Sedimentation

Closely related to the Phosphate Subcommittee's study of analytical methods is the project at the University of Illinois, started in June 1955 under Prof. J. C. Dietz and since then completed under Prof. R. S. Engelbrecht. A partial report on this project was given by Professor Dietz at the Purdue Conference on Industrial Wastes in May 1957, and his paper will appear in the proceedings of this conference.

The first phase of this study tackled the question of how much ortho and complex phosphates might be found in surface waters in Illinois streams. Three surveys were made. The first survey concentrated on the sampling of lakes and reservoirs. A second survey sampled a number of streams throughout the state. The third survey, which was continued from

April to December 1956, concentrated upon seven locations along the Kaskaskia River. The average phosphate concentrations determined by these surveys, together with the number of samples, are shown in the following table. All concentrations are as P_2O_5.

Survey	No. of samples	p.p.m. Ortho and hydrolyzable	p.p.m. Ortho	p.p.m. Hydrolyzable [a]
Lakes and reservoirs...	9	0.081	0.036	0.045
Illinois streams...........	27	0.657	0.411	0.246
Kaskaskia river...........	125	0.400	0.200	0.200

[a] Apparent complex phosphate.

In general, more than 50% of the phosphate found in the streams is ortho. The "hydrolyzable" portion consists of both inorganic polyphosphate and organic phosphate. The investigation also shows that much of the phosphates in streams comes from drainage of agricultural land—that the complex phosphates are converted to the simpler ortho form, and that this conversion is accelerated by the biological populations present in natural waters. The degradation rate of the same phosphate compound can vary considerably, with variation in mineral and other water characteristics. The rate of hydrolysis in natural waters increases with increase of temperature.

From studies with "jar" tests and on a pilot coagulation unit constructed in connection with the project, it was found that whereas complex phosphates at levels of 0.5 p.p.m. P_2O_5 and greater do exert interferences with coagulation and sedimentation, the interference can be reduced to an insignificant effect by practical increases in coagulant dosage or by an increase in settling time, also that the effects of levels of phosphates found to exist in Illinois waters do not appear to be significant from a practical point of view.

In summary, this project shows that phosphate levels in surface waters in Illinois are of a small magnitude, and only a portion of these is attributable to phosphates from household detergents. The project illustrates the use of the quantitative approach to the problem. It is intended that the results of these investigations will appear in the near future in three technical papers, completing the work on this project.

E. Foaming Problems—General

Foaming on rivers, in sewage-treatment plants, and in water-treatment plants is particularly noticeable to treatment plant operators and others in the field of sanitary engineering. One of the most difficult questions to answer is how much, if any, do household detergents contribute in any given instance of foaming or frothing. More knowledge of complex foam systems is needed so that it will be possible to identify all of the significant factors when a foam develops under unknown conditions, as is true with the foams being discussed.

It is known, on the one extreme, that when enough of one or more surface-active agents is present, voluminous, stable foams will form during any type of agitation, especially during aeration. On the other extreme, and not so widely realized, is the fact that low concentrations of one or two parts per million of surfactants in water, do not produce the stable, voluminous foams which are typical of high concentrations. The manifestation of foaming at these low concentrations is more accurately described as a "tendency to bubble."

It is recognized that the foaming tendencies of surface-active agents may be augmented by the presence of organic or inorganic substances. Again, better quantitative information is needed about specific foam builders likely to be present in water and sewage.

Physical chemists dealing with foams have recognized that the surface-active agents concentrate in the foam and are thus removed from the solution with which the foam is in contact. In the present situation the significance of this fact is that the amount of surfactant found in a foam cannot be taken as a measure of the amount of surface-active material present originally in the water.

Another interesting point made by the A.W.W.A. Task Group Report (1) is that the existence of natural surfactants can be demonstrated by soaking grass, straw, or leaves in water. These extracts produce a substantial lowering of the surface tension and do foam. Raw waters may be expected to contain various amounts of natural surfactants.

F. Foaming at Water-Treatment Plants

The classic example of foaming in water treatment, which has had wide and repeated pick-up in the literature, was the situation at Wheeling, W. Va., where around Thanksgiving of 1953 large quantities of froth formed both on the Ohio River and in the water-treatment plant there. The froth was accompanied by bad taste, discoloration, and extreme difficulty with filtering and settling. The river was low and almost in pool, which probably resulted in the presence of high concentrations of various organic and inorganic substances. When frothing started, the river water appeared to contain about 1 p.p.m. of "apparent ABS," using the methylene blue procedure which would include amounts of the interfering substances present. With such a low level of ABS in the water it was extremely difficult to postulate how detergents might have produced large quantities of stable foam. On the other hand, the foam did contain a high concentration of ABS chiefly adsorbed on finely divided solids held and suspended in the foam. Because of the presence of ABS many people concluded that the foam was due to ABS, but since that time river water with no foam and the same concentration of ABS has been treated by the water works at Wheeling, W. Va., without trouble. The fact that the combination of the foaming in the river, the water quality difficulties, and the water-treatment problems has not occurred again at Wheeling—or for that matter elsewhere—in retrospect adds mystery to the situation. If detergents from domestic sewage caused the Wheeling trouble, it is not easy to explain why the same kind of trouble has not happened there again.

There are also examples of foaming of raw water in water-treatment plants which appear to be caused by foaming agents other than ABS. In helping to trace the cause of one instance of this type of foaming, it was found that fractions of a part per million of "apparent ABS" by methylene blue, much too little to account for the foaming, were present when the foaming occurred. Later, during a routine check on the "apparent ABS" content of the river, greater amounts of ABS were present at a time when no foaming occurred. Other minor-nuisance foaming problems in water-treatment plants on the Ohio River have been investigated with confirmatory results. These cases point to the need for quantitative information in order to know specifically what causes any given foam, and conversely they point to the error of generally assuming that if there is a foam, it is caused by household detergents.

In Kansas, where in periods of drought, water may become critically scarce in some localities, there have been two opportunities where unusually high concentrations of ABS in the water supply and in the raw water have been studied, using the infrared analytical procedure in the investigation.

In January 1956 the Kansas State Board of Health advised the Soap Association technical group that in a city of about 6,500 population there was a slight tendency for bubbles to form on the surface of their drinking water, as for example when it was drawn from a tap. It turned out that the tap water when examined had an ABS content of approximately 1.25 p.p.m. by the infrared method. The tap water was excellent in clarity, color, taste, and odor. With one minor exception there were no operating problems in the water-treatment plant for which detergents could be suspected. The minor exception involved the formation of an extremely stable foam, characterized by the operating personnel as a nuisance rather than as a serious problem, which developed on the re-carbonation basin. Laboratory attempts to duplicate this foaming by bubbling carbon dioxide through samples of the water in question were unsuccessful. As a matter of fact, it is not known how such a voluminous, stable foam could be formed with as little as 1.5 to 1.25 p.p.m. ABS unless some other foaming agent were present.

At the time of the investigation a moderate amount of froth formed on the creek below a dam at the city's raw water source, and the froth was fairly stable. When the raw water was brought into the laboratory, the frothing effect could be reproduced qualitatively. The raw water which foamed contained approximately 1.5 p.p.m. ABS while the tap water on which stable foams would not form contained approximately 1.25 p.p.m. ABS. This difference in ABS content could hardly explain the difference in the foaming tendencies of the two waters. As the raw water was somewhat colored, it is likely that substances removed by the water-treatment process could have contributed to the formation of the stable foams at the dam.

At the A.W.W.A. meeting in Dallas in April, 1958, public health officials reported on a situation in a city of 12,000 population in Kansas where, because of drought conditions, the only water supply available was the treated, highly chlorinated effluent of its sewage-treatment plant. As stressed in the report, the re-circulation of chlorinated sewage effluent to supplement deficient water supplies should be considered only for severe emergencies. During water treatment, even though the level of ABS reached approximately 4 p.p.m., the only operating difficulty which occurred was serious frothing during agitation in the re-carbonation basin at the water-treatment plant. Other problems which might have been expected from earlier predictions in the literature did not occur. While many persons obtained their drinking water from other sources, it appears that a large segment of the people accepted the treated water for drinking purposes. There were no appreciable effects in the operation of the sewage-treatment plant.

In both these situations in Kansas public health officials who were involved reported that there were no public health problems encountered. In passing, it should be observed that the unusually high concentrations of ABS encountered did not produce the adverse effects which had been suspected of detergents in other situations, of interfering with coagulation and sedimentation in water treatment and of retarding the bacterial processes in sewage treatment.

The 1 p.p.m. and 4 p.p.m. ABS in drinking water obviously are extremely unusual. For example, the Ohio River, which has been monitored by P&G at Cincinnati during the past several years, has averaged 0.2 p.p.m. "apparent ABS" by methylene blue. Similar results are being obtained at a second monitoring point recently established by American Cyanamid at Willow Island, near Parkersburg, W. Va. The Ohio River monitoring for ABS is being done in cooperation with the Detergent Subcommittee of Orsanco.

From these two unusual experiences in Kansas it might be reasonable to conclude tentatively that while it is likely that, when tap water reaches about 1 p.p.m. of ABS, a slight tendency to bubble may become noticeable, no other undesirable water quality characteristics appear.

G. Removal of ABS in Water Treatment

In the event that the desire or need should arise for the removal of ABS during water treatment, the Soap Association has underwritten a research project at Johns Hopkins to investigate this possibility. This project, which was started last year, is under the direction of Prof. Charles E. Renn and is already tracking down several interesting leads. The use of activated carbon to adsorb and remove ABS is one possibility. Another possibility involves deliberately frothing the water, thereby concentrating the ABS in the foam. The foam is then separately removed and destroyed. Most intriguing seems to be the possibility of adsorption of ABS on activated carbon or other suitable solids with deliberate froth removal, in which the solids would be entrapped with the foam and the combined solids-foam system separately removed.

H. Frothing in Sewage-Treatment Plants

Foam first reared its frothy head in sewage-treatment plants long before the advent of detergents. When froth develops to photogenic heights, it is likely to receive considerable newspaper publicity which, more frequently than not, associates it with detergents. Frothing is now classed as no more than a nuisance and sewage plant operators are handling it with increasing success by the means at their command, such as the control of suspended solids, the use of surface sprays, and foam-breaking additives.

Because the frothing problem is still troublesome to sewage works operators and is of interest to the soap industry, a research project was placed at the University of Wisconsin under the direction of Prof. G. A. Rohlich and Assistant Professor L. B. Polkowski. It is directed toward determining the various causes of frothing in sewage-treatment plants and, more important, toward finding means of eliminating or at least minimizing the froth.

In response to a questionnaire directed by these Wisconsin men to a large number of activated-sludge plants, 97 plants stated that frothing was present to some degree while four plants reported no frothing; 50 plants reported that frothing was a daily occurrence while 49 plants said that it was not. The large majority of plants who encountered frothing expressed the belief that it has no effect on plant efficiency.

Among the factors or conditions which appear to influence frothing are protein degradation products, ABS content, suspended solids concentration, the degree of purification, temperature, and pH. These factors appear to be involved simultaneously, and the effects can be synergistic. For example, in water solutions containing 3 p.p.m. ABS foam persistencies are quite low and do not exceed three minutes. The addition of about 15 p.p.m. of a protein degradation product, which by itself also gives quite low foam persistencies, resulted in about a six-fold increase. With the addition of 30 p.p.m. of the protein degradation product the persistency increased almost 25 times. With ABS concentration of 1 p.p.m. or less and concentrations of protein degradation product of 300 p.p.m., persistencies are quite low. The Wisconsin studies confirm the reports that increasing suspended solids tends to reduce frothing. Decreased froth persistency is observed as the sewage temperature drops from 72° to 54°F. Froth persistency decreased as pH was decreased from 9.0 to 6.5. In these experiments pH was reduced from normal by adding acetic acid, was increased by adding lime.

Extending the investigation of methods to eliminate frothing to include practical studies of various engineering variables is part of the future program at the University of Wisconsin.

It becomes apparent that the factors involved in frothing are numerous and complex, and to single out any one factor as having the sole responsibility for the frothing which is experienced in plants would seem to be unwarranted by the facts now available. It is hoped that the information being obtained in the Soap Association's research program is helping to bring about a better understanding of the problems involved.

I. Biological Degradation of Tetrapropylene Benzene Sulfonate

Questions raised about detergents in sewage treatment which stimulated the Soap Association research program are these. Do detergents retard the biochemical process involved in sewage treatment? Do they otherwise adversely affect efficiency? Do the detergents pass unchanged through sewage treatment into surface waters?

At first these questions were asked about surface-active agents in general, but it has become apparent that surface-active agents which are easily decomposed in the biological processes of sewage treatment are not involved, and thus recently the questions have been directed more specifically to the polypropylene type of ABS, which is inherently more stable.

In looking for answers to these questions, the Soap Association set up two projects, the first, a study of the bacteriology of the degradation of the ABS molecule at M.I.T. under Prof. Ross E. McKinney and Prof. Rolf Eliassen, and second, a study of the fate of ABS in sewage treatment at the University of California under Prof. P. H. McGauhey and Prof. E. S. Crosby of the Sanitary Engineering Laboratory. At California the radioactive tracer techniques suggested by House and Fries (7) were refined and improved.

From these studies there is no evidence that the amount of ABS likely to be present in normal domestic sewage (5 to 10 p.p.m.) has any toxic effects on activated sludge. The organisms remained active and multiplied in a normal fashion. The BOD reduction was typical of activated sludge without ABS present. Both the M.I.T. and California projects came to the same conclusions on this point even though the experimental approaches were quite different. Further there was no evidence that the presence of relatively high levels of ABS would adversely affect the efficiency of the activated sludge process.

The work at both projects has established that ordinary sewage bacteria decomposed a major portion of the ABS entering a sewage plant. The radioactive tracer technique shows that approximately 60% is removed by the activated-sludge process. This is in marked contrast to earlier assumptions that ABS would pass through sewage treatment in an unaltered state.

Investigation at M.I.T. of specialized strains of bacteria capable of completely destroying ABS was discontinued, and more interesting leads are being studied which indicate that improved hydraulic and biological design of aeration tanks could bring about 80% removal of ABS. The design changes could very well be practical in conventional plants.

At the University of California at the start of the project it was necessary to attempt to use colorimetric methods to determine the approximate level of ABS in the natural sewage used in the experiment. These attempts gave entirely unsatisfactory results, which in fact were at times misleading in material balance studies. Only through the use of radiological tests was it possible to obtain reliable results.

J. Foam Removal by Induced Frothing

Work has been done both at the University of California and the University of Wisconsin to put to practical use the knowledge that a surface-active agent will concentrate in the froth it forms. In preliminary experiments forced frothing of settled raw sewage or of sewage effluents by aeration followed by removal of the froth reduces the ABS to 1 p.p.m. or less. It is likely that greater reductions could be obtained by improved foaming methods. The possibility of applying this technique to large-scale operations appears to be promising, and additional experiments indicate that the froth removed might be disposed of by incineration with waste-digester gas. The work on removal by forced frothing is relatively recent, and the investigators have high hopes that this development will have practical application.

Conclusion

In conclusion, the Soap Association's research program to date, in addition to contributing to the technical knowledge, has had very constructive effects in at least two somewhat separate but important areas. First, the research program has corrected a number of misconceptions about the possible effects of the normal use of household detergents by the American public on water supplies and sewage treatment, misconceptions which otherwise might have been accepted by default; and second, it seems reasonable to assume from the findings so far that it will be possible, if necessary, to develop technological processes capable of handling any problems which may arise.

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