

## BIODEGRADABLE DETERGENTS FROM ANIMAL FATS

T. C. Cordon

Eastern Utilization Research and Development Division  
Agricultural Research Service, U.S.D.A.  
Philadelphia, Pennsylvania 19118

Disposal of waste material in a manner that will not pollute the environment is posing increasingly acute problems. Sewage disposal is a case in point. In 1963 (3) it was estimated that 44% of the sewage we produce received no treatment or settling only. No more than 36% received secondary treatment (activated sludge or trickling filter) and 20% was disposed of in septic tanks and cesspools. It is doubtful if the situation has improved since then; more likely it has worsened. The addition of incompletely treated or untreated sewage to our water systems is a major factor in the eutrophication that is despoiling our lakes and estuaries. The need for cleansing agents (detergents) that are rapidly biodegradable under all conditions is apparent.

Animal fats, which are by-products of the meat packing industry, are plentiful, low-cost starting materials for the manufacture of surface active agents including synthetic detergents. About 4.6 billion lbs. of inedible animal fats are produced each year; approximately half of this is exported as inedible fat. All or part of this could be used for detergent manufacture. The constituent fatty acids, stearic, oleic and palmitic acids are natural hydrophobic (water repelling) compounds that are easily biodegradable because of their linear character. Some surface active derivatives of the saturated fatty acids may not be adequately soluble because of the length of the hydrophobic chain. Hydrophilic (water-attracting) groups may be introduced at the double bond of oleic, at the terminal carboxyl group, or at the  $\alpha$ -C atom of saturated fatty acids. If left intact, the double bond in the middle of the chain promotes solubility. Straight chains of natural origin, if not adversely substituted, are attractive starting materials for the manufacture of biodegradable detergents.

In comparing the detergency of different members of a homologous series, it is usually found that maximum hot-water detergency occurs when the aliphatic chain is 16 or 18 C atoms long, but surface active agents with these chain lengths often have poor cold-water solubility. This laboratory has therefore directed research toward chemical modifications which will increase solubility without reducing detergency or biodegradability. Methods have included blending with more soluble detergents, the introduction



**Detergent and other Surface Active Properties.** It has been shown that tallow alcohol sulfates, sodium hexadecyl sulfate and sodium octadecyl sulfate have very good detergent properties. A method for increasing solubility of the slightly soluble tallow alcohol sulfate is to oxyalkylate prior to sulfation. We have found that small amounts of oxyalkylation can produce the desired solubility increase without loss in detergency and have shown that oxypropylation and oxybutylation are more effective than oxyethylation (18). These ether-alcohol sulfates are also good lime-soap dispersing agents, making them attractive agents for combination with soap.

The sodium salts of  $\alpha$ -sulfopalmitic and  $\alpha$ -sulfostearic acids are also good detergents in hot water but have poor room-temperature solubility. Solubility of sodium  $\alpha$ -sulfopalmitic or  $\alpha$ -sulfostearic acids may be improved by esterifying the carboxyl group with low-molecular weight alcohols.

Table I shows the detergency of some tallow-based detergents com-

TABLE I  
DETERGENCY<sup>1</sup>

| .25% Built Detergents <sup>2</sup> | Soft Water | Hard Water |
|------------------------------------|------------|------------|
| Tallow Alcohol Sulfate             | 101        | 98         |
| $\alpha$ -Sulfo Tallow Ester       | 92         | 88         |
| Ester-Soap mixture (1 - 1)         | 99         | 89         |
| ABS                                | 86         | 76         |
| LAS                                | 86         | 85         |

<sup>1</sup>.25% soap in soft water = 100

<sup>2</sup>.05% active ingredient + .20% inorganic builder.

pared to the alkylbenzene sulfonates now in common use. It is clear that the tallow-based detergents were equally as good or perhaps a little better than the petroleum-based products in these tests.

### BIODEGRADATION

Following use, detergents may be subjected to biodegradation in sewage treatment plants, water courses or soils where oxygen concentration, temperature and detergent concentration may vary markedly. We have simulated some of these natural conditions and subjected the detergents to the action of natural microflora. We have also followed the course of the biodegradation under conditions in which the inoculant and nutrient supply were controlled.

**Biodegradation in River Water (19).** The "River Water Die-Away" test (8, 15) was used to determine the biodegradability of thirty-three anionic and six nonionic detergents. With the exception of a few samples used for

comparison, all river water used in these tests came from the Schuylkill River at Fairmount Park, Philadelphia. The solutions containing 5 ppm detergent were stored in the dark at 20°C, and aliquots were analyzed regularly for methylene blue active substance (MBAS) by the method of Degens (7). This method measures loss of surface activity only and not complete degradation.

**Degradation of Anionic Detergents.** Figure 1 illustrates the range of

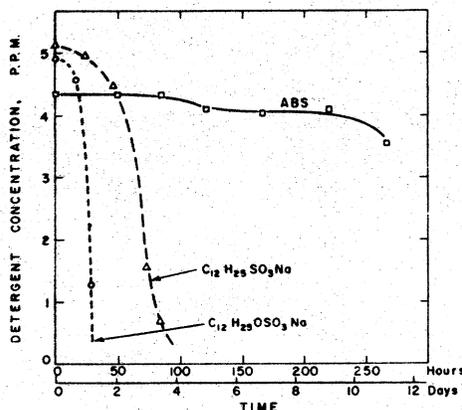


Figure 1. Biodegradation of Anionic Surface-Active Agents in Schuylkill River Water. materials being considered: the very "soft," or very easily biodegraded, sodium alkyl sulfate; the soft sodium alkanesulfonate; and the "hard," previously used, tetrapropyl type alkylbenzenesulfonate (ABS) that is quite resistant to biodegradation.

Table II shows the time required for the disappearance of 80% of the

TABLE II  
Biodegradation of Representative Anionic Detergents in Schuylkill River Water, 20°C.

| Compounds  | Time in hrs for 80% Degradation | Class |
|--|---------------------------------|-------|
| Sodium Sulfoethyl Palmitate<br>C <sub>15</sub> H <sub>31</sub> CO <sub>2</sub> C <sub>2</sub> H <sub>4</sub> SO <sub>3</sub> Na                                  | 19                              | I     |
| Sulfated Trioxyethylated Hexadecanol<br>C <sub>16</sub> H <sub>33</sub> (OC <sub>2</sub> H <sub>4</sub> ) <sub>3</sub> OSO <sub>3</sub> Na                       | 45                              | I     |
| Sodium Hexadecanesulfonate<br>C <sub>16</sub> H <sub>33</sub> SO <sub>3</sub> Na   | 98                              | II    |
| Sodium Methyl α-Sulfopalmitate<br>C <sub>17</sub> H <sub>35</sub> CH(SO <sub>3</sub> Na)CO <sub>2</sub> CH <sub>3</sub>  | 103                             | II    |
| Sodium Hexyl α-Sulfopelargonate<br>C <sub>7</sub> H <sub>15</sub> CH(SO <sub>3</sub> Na)CO <sub>2</sub> C <sub>6</sub> H <sub>13</sub>                           | 245                             | III   |
| Disodium α-Sulfophenylstearate<br>CH <sub>3</sub> (CH <sub>2</sub> ) <sub>7</sub> CH(Ph)(CH <sub>2</sub> ) <sub>7</sub> CH(SO <sub>3</sub> Na)CO <sub>2</sub> Na | 335                             | III   |
| ABS<br>C <sub>12</sub> H <sub>25</sub> C <sub>6</sub> H <sub>4</sub> SO <sub>3</sub> Na  | > 700                           | IV    |

surface activity of some representative compounds. From data of this kind the surfactants have been divided into four categories: (I) very soft, (II) soft, (III) moderately hard and (IV) hard.

The very soft (0-3 days) group includes the simple alkyl sulfates, and carboxylic esters and amides where the hydrophobic and hydrophilic parts may be easily separated by hydrolysis, with early destruction of surface activity. With the exception of a few unusual chemical structures, all the alkanesulfonates, the  $\alpha$ -sulfo fatty acids, their salts, esters and amides belong to the soft group (3-7 days).

The moderately hard group (1-2 weeks) includes a number of compounds of unusual structure. These may possibly interfere with bacteriological activity because of exceptional wetting properties or because of phenyl or chlorine substitution in the alkyl chain.

**Degradation of Nonionic Detergents.** Figure 2 shows biodegradation

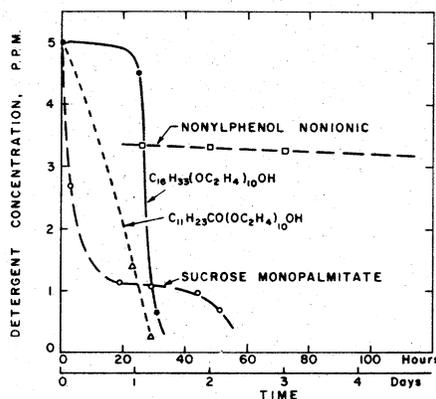


Figure 2. Biodegradation of Nonionic Surface-Active Agents.

curves for four nonionic detergents of different structure. Concentrations for these curves were determined by the surface-tension method. Sucrose monopalmitate showed very rapid initial degradation, followed by a slower degradation in the final stage which may indicate that some intermediates are surface active. Degradation of the ester type nonionic proceeded immediately and rapidly. The ether type appeared to go through a short induction or acclimation period before rapid degradation. The nonylphenol nonionic showed very little degradation in this time.

**Biodegradation in Activated Sludge (2).** Secondary treatment of sewage in an activated sludge plant involves (1) settling of the raw sewage, (2) aeration of the effluent, (3) settling to separate the solids (this sludge consists of cells of microorganisms), (4) return of a portion of the sludge to the aeration tank, and (5) anaerobic digestion of the solids from the aeration tank.

Tallow-based compounds were subjected to conditions similar to those found in the aeration section of sewage-disposal plants. A diagram of Ludzack's unit (13) used for this purpose is shown in Figure 3. These tests

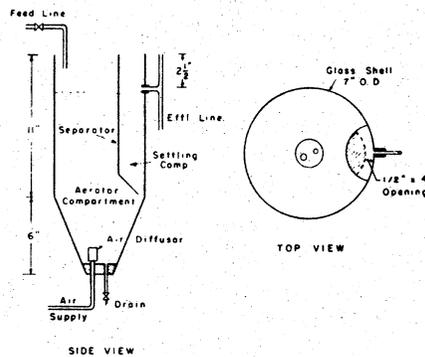


Figure 3. Model Activated Sludge Unit.

were started with sludge from a sewage disposal plant that treats mostly household sewage and the aerators were fed continuously with a weak suspension of trout chow containing the detergent under test. They were aerated continuously and the effluents analyzed daily for undegraded detergent by the methylene blue method.

The bar graph in Figure 4 compares the results obtained with two

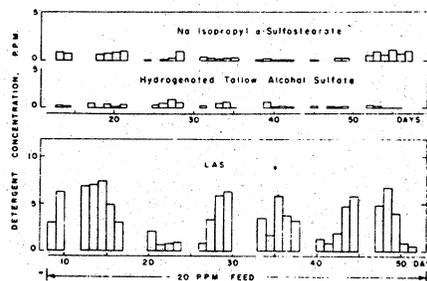


Figure 4. Degradation of Fat-Based Detergents and LAS in Activated Sludge.

fat-based detergents and LAS. Both tallow-based compounds were almost completely degraded, whereas LAS remained undegraded to the extent of 25 to 60 percent.

**Biodegradation in Anaerobic Digesters (14).** In the operation of a sewage treatment plant with secondary treatment, the solids that are not degraded in the activated sludge section are placed in digesters under anaerobic conditions for further breakdown. Thus any detergent that was adsorbed on the solid particles would be carried into the digester. The

experimental procedure used in these tests was patterned after that of Johnson and Bloodgood (11) and Hernandez and Bloodgood (10). In this procedure sludge from the anaerobic digester of a sewage plant was incubated at 35°C in closed bottles. The feed consisted of primary sludge equivalent to 2g of volatile solids per day. An equal amount of sludge was removed for analyses before adding the feed.

**Analytical Procedures.** The course of the digestion was followed by collecting and measuring the gas evolved which was mostly methane and carbon dioxide. The experimental apparatus is shown in Figure 5. Gas

ANAEROBIC DIGESTER AND GAS COLLECTING CYLINDER

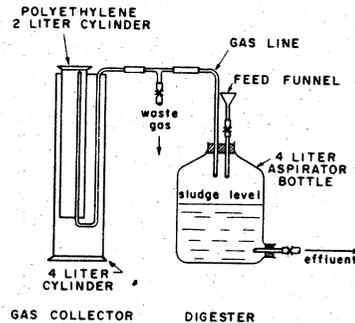


Figure 5. Experimental Apparatus Used for Anaerobic Sludge.

volume was measured daily. Detergent concentration was determined by extracting the detergent from the sludge sample with 95% ethyl alcohol and analyzing by the methylene-blue method.

The results of gas production from two experiments are shown in Table III. The uniformity of gas evolution from the controls confirmed

TABLE III

Gas Production of Anaerobic Digesters for 31 days

| Experiment Number | Digesters   | Volume of Gas Produced |                  |
|-------------------|---|------------------------|------------------|
|                   |   | Total ml               | Daily Average ml |
| 1                 | Control   | 32,580                 | 1053             |
|                   | LAS, $C_{12}H_{25}C_6H_4SO_3Na$   | 14,980                 | —                |
|                   | Sodium Isopropyl $\alpha$ -Sulfostearate<br>$CH_3(CH_2)_{15}CH(SO_3Na)CO_2CH(CH_3)_2$ | 33,930                 | 1093             |
| 2                 | Control   | 32,060                 | 1034             |
|                   | ABS, $C_{12}H_{25}C_6H_4SO_3Na$   | 14,830                 | —                |
|                   | Sodium Methyl $\alpha$ -Sulfostearate<br>$CH_3(CH_2)_{15}CH(SO_3Na)CO_2CH_3$          | 31,550                 | 1018             |
|                   | Hydrogenated Tallow Alcohol Sulfates<br>$R \cdot OSO_3Na$                             | 36,410                 | 1175             |

the reliability of the digester operation. Gas volumes produced from digesters containing ABS and LAS were significantly less than those of the control digesters indicating an adverse effect on the sludge-digestion process. The sludge in the digesters that contained the tallow-based detergents produced about the same quantities of gas as the controls, and in the case of hydrogenated tallow alcohol sulfates significantly more gas was produced.

The alcohol sulfates were completely degraded according to the analysis for MBAS. The detergent accumulated in all other instances. However, neither sodium isopropyl  $\alpha$ -sulfostearate nor sodium methyl  $\alpha$ -sulfostearate interfered with the normal digestion process.

Figure 6 illustrates the course of analysis for ABS and HTAS. ABS

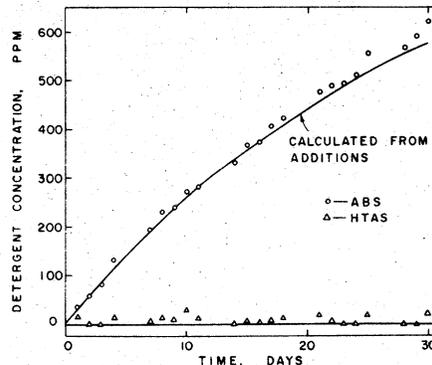


Figure 6. Detergent Analysis of Anaerobic Sludge.

values follow closely the curve for the calculated daily additions of detergent, whereas HTAS values follow closely the base or zero concentration line.

**Biodegradation Under Microaerophilic Conditions.** While strictly anaerobic conditions are present in anaerobic digesters, detergents may also be disposed of under conditions of low oxygen concentration that are not strictly anaerobic. Such conditions are found in anaerobic ponds, cesspools, septic tanks and water-logged soils. Conditions simulating these as to oxygen concentration have been produced in the laboratory by bubbling nitrogen through river water. The concentration of oxygen in this system was less than 1 ppm and usually less than 0.5 ppm. Dissolved oxygen was determined by a polarographic method that employed a gold-silver electrode enclosed in a polyvinyl-chloride housing through which the only entrance for oxygen was a thin gas-permeable teflon membrane. This method gave good agreement with the standard Winkler procedure.

The biodegradation of representative detergents in this system was compared with biodegradation under aerobic conditions at 5 ppm and 10

ppm concentration and at 25° and 35° C. Loss of MBAS was used as the criterion of breakdown. The results for 10 ppm are given in Table IV. The alcohol sulfate and the sulfated hydroxy amides were degraded under

TABLE IV  
Biodegradation Time for Zero MBAS

| Compound   | 10 PPM   |                      | 10 PPM               |                      |
|--|----------|----------------------|----------------------|----------------------|
|  | Aerobic  | Micro-aerophilic     | Microaerophilic      |                      |
|  |          | 25°                  | 25°                  | 35°                  |
| Na Hexadecyl Sulfate<br>$C_{16}H_{33}OSO_3Na$                                  | 1-2 days | 9-10 days            | 9-10 days            | 3-4 days             |
| Sulfated Hydroxypropyl Stearamide<br>$CH_3(CH_2)_{16}CONHCH_2CH(CH_3)OSO_3Na$  | 2 days   | 6-7 days             | 11-15 days           | 9-11 days            |
| Na Hexadecyloxyethyl Sulfate<br>$C_{16}H_{33}OCH_2CH_2OSO_3Na$                 | 2 days   | 48-63%<br>in 11 days | 13-25%<br>in 17 days | 70%<br>in 17 days    |
| Na Hexadecyloxybutyl Sulfate<br>$C_{16}H_{33}OCH_2CH(C_2H_5)OSO_3Na$           | 3 days   | 0%<br>in 8 days      | 0%<br>in 11 days     | 15-20%<br>in 11 days |
| Na Isopropyl $\alpha$ -Sulfostearate<br>$C_{16}H_{33}CH(SO_3Na)CO_2CH(CH_3)_2$ | 6 days   | 0%<br>in 9 days      | 0-20%<br>in 14 days  | 7-9 days             |
| LAS <sup>1</sup><br>$C_{12}H_{25}C_6H_4SO_3Na$                                 | 9 days   | 0%<br>in 9 days      | 0%<br>in 18 days     | 0%<br>in 18 days     |

<sup>1</sup>Commercial product

all conditions but the process was much slower at lower oxygen levels. The ether alcohol sulfates were somewhat resistant to attack at low oxygen concentration but 25% to 70% breakdown did occur in about two weeks. Degradation was greater at the higher temperature. There was no degradation of the  $\alpha$ -sulfo esters under microaerophilic conditions at room temperature. However, at 35°C complete loss of MBAS occurred in 7 to 11 days. With LAS no loss of MBAS was found under microaerophilic conditions in these tests.

**Biodegradation in Soil (14).** Figure 7 shows the MBAS content of the effluents from solutions that contained 20 ppm of detergent after they were percolated through a local soil (Calfon silt loam). In 21 days the ABS degraded to the extent of 35%; in 39 days LAS was 83% degraded; and in 38 days sodium isopropyl  $\alpha$ -sulfostearate was 100% degraded continuously with variation of less than 0.5 ppm.

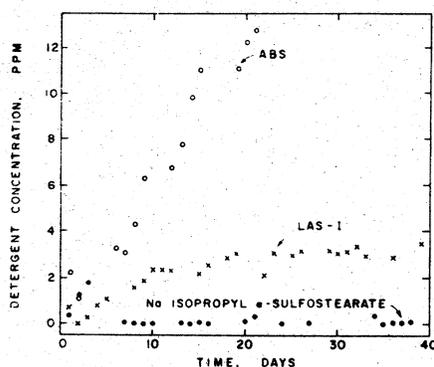


Figure 7. Detergent Analysis of Lysimeter Effluents.

**Metabolism by Sewage Microorganisms (4, 6).** The presence of anionic detergents in wastewater has usually been determined by measuring the concentration of MBAS. The method depends on the formation of a colored, chloroform-soluble salt of the detergent with methylene blue. The salt is no longer exclusively chloroform soluble when the carbon chains are shortened to about 6 to 8 carbon atoms. This method can show greatly decreased amounts of detergent, or even none, when, in fact, a good part of the original material may be present as smaller molecules.

To obtain more complete information on the mechanism of detergent biodegradation, the Esso Research Biodegradation Test was modified to determine the ability of microorganisms to utilize fat-based detergents as the sole source of carbon and energy. The course of the biodegradation was followed by measuring loss of carbon and MBAS and formation of sulfate ions. Carbon and MBAS were determined using automated analyzers. Sulfate ion concentration was determined by a turbidimetric method developed in our laboratory (5). This procedure includes a measure of inherent turbidity not produced from  $\text{SO}_4$ , that may be caused by growth of microorganisms or by the formation of insoluble compounds from detergent degradation. Turbidity from a non-detergent, glucose, is compared with that from two detergents in equivalent amounts in Figure 8. The alcohol sulfate and ether alcohol sulfate showed much larger maximum turbidity values, possibly due to the separation of insoluble long chain alcohols and ether alcohols.

**Alcohol Sulfates and Ether Alcohol Sulfates.** Sodium hexadecyl sulfate was rapidly attacked as shown in Figure 9. Most of the degradation took place in the first 3 days and by the end of the test 94% of the carbon had been lost and 96% of the theoretical  $\text{SO}_4^{2-}$  was present. The curves for the ether alcohol sulfates were similar to that for the alcohol sulfate, but degradation was not as rapid. Figure 10 shows an example. There was

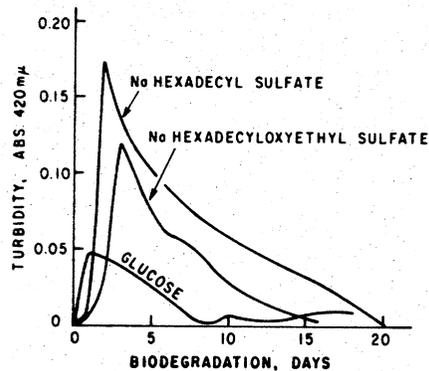


Figure 8. Comparison of Turbidity Formed in Degradation of Detergents and Glucose.

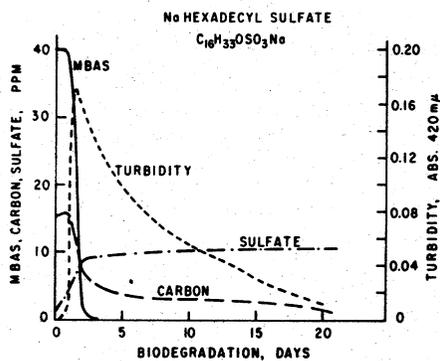


Figure 9. Biodegradation of an Alcohol Sulfate.

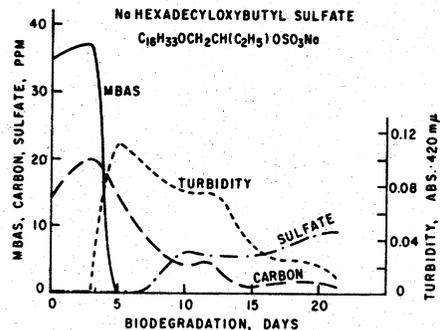


Figure 10. Biodegradation of an Ether Alcohol Sulfate.

some difference in the rate of degradation depending on the number of ether groups. The isopropyl ether was more resistant than the ethyl or butyl compounds.

Aliphatic and Aromatic Sulfonates. Figure 11a shows that the biodeg-

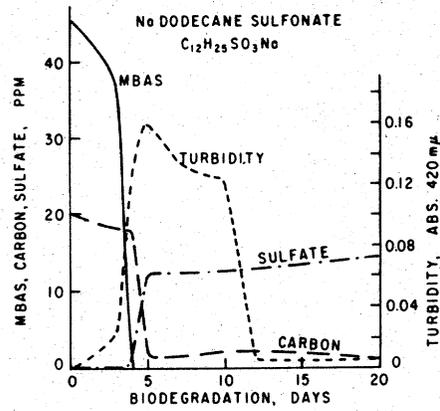


Figure 11a. Biodegradation of an Alkane Sulfonate.

radation of sodium dodecanesulfonate was more rapid and complete than for a linear alkylbenzenesulfonate, Fig. 11b. The value for MBAS

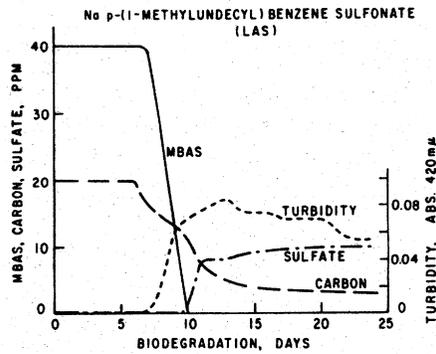


Figure 11b. Biodegradation of LAS.

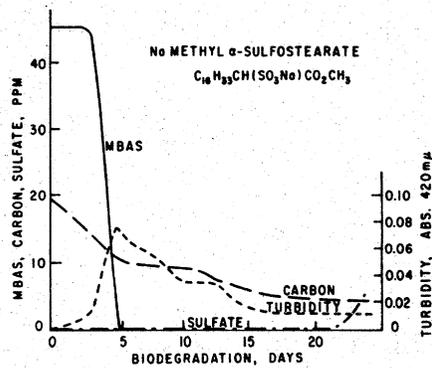


Figure 12. Biodegradation of an  $\alpha$ -Sulfo Fatty Acid Ester.

dropped to zero in 3-4 days compared to 9 days for LAS; and loss of C and  $\text{SO}_4$  formation was nearly quantitative compared to 89% and 84% respectively for LAS. Other surface active aliphatic sulfonates were also completely degraded (6).

**Esters of  $\alpha$ -Sulfo Fatty Acids.** With the exception of one highly fluorinated compound all the  $\alpha$ -sulfo esters we have tested, including sulfosuccinates, were aerobically biodegradable in the sense that the MBAS was reduced to zero. However, a small residue of the carbon remained in the case of the  $\alpha$ -sulfo esters and sulfate ion was not formed until the end of the test. An example is shown in Figure 12. A series of compounds that might result from the incomplete breakdown of  $\alpha$ -sulfo esters was prepared and tested. Disodium  $\alpha$ -sulfosuccinic acid was found to be resistant to biodegradation, Figure 13. It appears that small amounts of

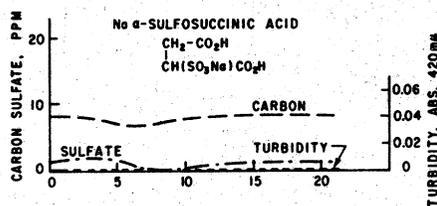


Figure 13. Biodegradation of a Sulfosuccinate.

$\alpha$ -sulfosuccinates may be formed as intermediates in the biodegradation of  $\alpha$ -sulfo esters. Succinates are non-toxic and are used in the food industry.

## DISCUSSION

It has been shown that inedible animal fats are a plentiful, low-cost source of starting material for the manufacture of detergents. Within the group of products excellent detergents, wetting agents, and lime-soap dispersing agents are found. The latter compounds would be especially useful in mixtures with soap to disperse the lime-soap curds that form in hard water.

Many of these compounds are biodegradable under a variety of natural conditions and some, like the alcohol sulfates, degrade in the absence of oxygen. This is in contrast to the alkylbenzenesulfonate-type compounds which were found to be completely resistant to biodegradation under anaerobic conditions.

The need to utilize materials as cleaning agents that will be easily degraded and will not persist to contribute to pollution should receive increased emphasis. Eutrophication, the process of nutrient enrichment in water, is very much in the public eye because of the growth of nuisance plants, especially algae, that this permits. Much work is being done to find ways of preventing the addition of phosphorus to our water courses.

Phosphorus is one essential nutrient for the growth of algae and it is believed that reducing the amount of phosphorus entering the water courses would alleviate the situation. A major source of the phosphorus ion in wastewater is from detergent formulations.

Evidence questioning whether phosphorus is actually the limiting nutrient responsible for algal blooms has been assembled by Kuentzel (12). He points out the fundamental fact that aerobic bacteria require oxygen to degrade organic matter and produce carbon dioxide ( $\text{CO}_2$ ) and that algae require  $\text{CO}_2$  to photosynthesize organic matter and produce oxygen. A nutrient cycle is diagrammed in Figure 14. Organic matter

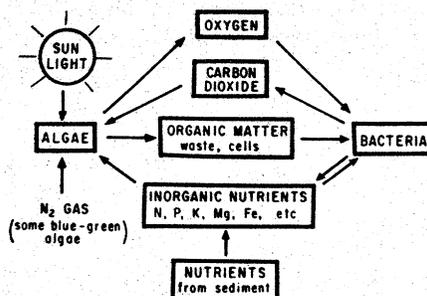


Figure 14. Nutrient Cycle Between Algae and Bacteria.

added to the system as wastes is converted to  $\text{CO}_2$  which is used by the algae to produce more organic matter. Dead algal cells are decomposed by the bacteria producing more  $\text{CO}_2$  and releasing mineral nutrients including phosphorus. Once established in a given body of water such a cycle would be very difficult to break. An idea of the magnitude that this process can attain is given by Harlow (9) who estimated that algae produce nearly 10 billion pounds of organic matter per year in the western basin of Lake Erie. This is about 18 times as much as contributed by all wastewater effluents combined. Kuentzel cites data showing that algal blooms occur when as little as 0.01 ppm of soluble phosphorus is present and that many of our lakes and estuaries already contain this amount or more. In view of this information it seems highly unlikely that stopping phosphorus addition to lakes that already contain .01 ppm would have any substantial effect on algal blooms. According to this concept the major effort should be to stop pollution with organic wastes.

#### SUMMARY

Animal fats, which are byproducts of the meat packing industry, are plentiful, low-cost starting materials for the manufacture of surface active agents including detergents. Some surface active derivatives of the saturated fatty acids may not be adequately soluble because of the length of the hydrophobic chain.

Solubility may be increased by the introduction of hydrophilic groups at the double bond of oleic acid, at the terminal carboxyl group, or at the  $\alpha$ -C atom of saturated fatty acids. Research has been directed toward the chemical modifications which will increase solubility without reducing detergency or biodegradability. The syntheses of  $\alpha$ -sulfo acids and esters, alcohol sulfates and ether alcohol sulfates are briefly described. Biodegradation of anionic and nonionic tallow-based detergents in river water was determined using the "River Water Die-Away" test. A laboratory model of the aeration section of an activated sludge sewage disposal plant was used to determine biodegradation in this system. Biodegradation was followed in the complete absence of oxygen in anaerobic sludge digesters and under conditions of low oxygen concentration, less than 1 ppm (microaerophilic), in river water. Percolation of detergent containing solutions through soil (lysimeter tests) was used to follow biodegradation in soil. The ability of sewage microorganisms to use detergents as the sole source of carbon and energy was determined in a system in which the nutrients and inoculum were controlled.

Representative compounds of each of groups of fat-based detergents under test could serve as the sole source of carbon and energy for sewage microorganisms. These detergents were rapidly degraded under aerobic conditions and some, like the alcohol sulfates, were biodegradable under anaerobic conditions. Linear alkylbenzene sulfonate did not degrade under anaerobic or microaerophilic conditions.

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