

The following is a review article.

Biodegradability of Alkylbenzene Sulfonates

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This article puts in perspective various questions concerning the environmental impact of alkylbenzene sulfonates (ABS). Several points which are frequently overlooked or misunderstood are clarified:

1. Except for the nuisance aspects of its foaming tendency, ABS entering the environment as a component of household sewage is insignificant relative to other pollutants.

2. Any negative impact that ABS has—foaming, possible interaction with living organisms—comes from its surface active character. When it biodegrades sufficiently to lose its surface active character, it becomes innocuous. Hence, only “primary” biodegradation rates are important.

3. Linear ABS (LAS) biodegrades much faster than highly branched ABS (BAS). However, they both eventually completely degrade in an aerobic environment.

4. Neither BAS nor LAS degrade when the sewage does not receive adequate aerobic sewage treatment either in a treatment plant or in the receiving water. Therefore, in localities without adequate aerobic sewage treatment, there is no environmental incentive to use LAS in preference to BAS.

5. Realistic laboratory tests mirror the sewage treatment process. It is not reasonable, except for screening purposes, to run biodegradation tests in the absence of sewage.

6. Slight variations in the LAS structure—*isomer distribution, modest branching*—do not affect its biodegradation in realistic tests.

7. The important criteria for a country contemplating a switch from BAS to LAS are the concentration of ABS found in receiving waters and the adequacy of sewage treatment.

ABS, first used in the 1930's, has been the major surfactant for synthetic household detergents worldwide since the 1950's. Approximately 4.5 B lb/yr are presently produced. The basic chemistry and properties have long been established, but there have been changes from time to time in the source of the alkyl group. At first, chlorinated kerosene fractions were used. Now, the two common sources of the alkyl side chain are:

1. C₁₁-C₁₅ propylene oligomer. After alkylation and sulfonation reactions, this provides a highly branched ABS called BAS. BAS has sometimes been called TBS (tetrapropylene benzene sulfonate) and DBS (dodecyl benzene sulfonate). It was originally called ABS for alkylbenzene sulfonate. This latter term should only be used as a generic name covering all structures. (Note that ABS can be confused with the abbreviation for acrylonitrile/butadiene/styrene plastics.) It was developed in the 1940's, but has not been used in the industrial countries since the mid-1960's because of environmental concerns based on its relatively slow biodegradation rate. Worldwide usage is approximately 1.2 B lb/yr.

2. C₁₀-C₁₄ n-paraffin. After dehydrogenation or chlorination, alkylation, and sulfonation, this forms a linear side chain ABS called LAS. Because of its relatively fast biodegradation rate, it was developed in the 1960's to overcome the concerns with BAS. Worldwide usage is approximately 3.2 B lb/yr.

The switch from BAS to LAS in the industrial countries resulted from many observations that BAS was being found in sewage treatment plants and inland waterways at a concentration high enough to cause noticeable foam, and that the BAS did not quickly disappear, i.e., biodegrade.

A very large scientific effort was mounted by government and industry to analyze the problem and seek solutions. The large amount of scientific information collected has been well documented by R.D. Swisher in 1970 and again in 1987 (1). LAS was found to be a completely satisfactory cure for the problems (2-4).

LAS is much more quickly degraded by common microorganisms than BAS. In 1965, the United States detergent industry switched from BAS to LAS at an estimated cost of \$150 MM (5). The foaming problems disappeared.

During the last 20 years, there has been a tremendous increase in concerns, investigations, cleanup operations, laws, and regulations in the area of environmental affairs. Analytical techniques have grown much more sophisticated and risk assessment has matured.

It is very notable that the first item to gain widespread attention—the foaming surfactant—is no longer an issue. This is because of the success of LAS and realization that many other pollutants (pesticide residues, heavy metals, fluorocarbons, radon, acid rain, etc.) which are not as readily visible as the foaming surfactant can have a real, rather than just a cosmetic, environmental effect. Now many prestigious books on pollution and water quality hardly even mention ABS (6-8).

Nevertheless, the subject of ABS biodegradability is still of interest today for two reasons:

1. BAS is still used in many countries where LAS has not been needed. In those countries, the question of switching to LAS is reviewed from time to time. In this article, we present rational environmental criteria for making this decision.

2. Questions may arise in connection with biodegradability requirements for variations on the LAS structure or for LAS itself.

Therefore, the authors feel that the present discussion is warranted, even though some of the facts were established over 20 years ago. However, we will not provide detail on all the studies which have been covered so well by Swisher. Our aim is to discuss the points that are important in arriving at realistic public policy.

ABS IN THE ENVIRONMENT

Source. This discussion is concerned only with ABS which has been used as a surfactant in household detergents. After use, it always enters the environment—lakes, rivers, the ocean—as a component of household sewage. This

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situation is very important because it means that the ABS is always mixed with a noxious stream which must be treated or highly diluted to avoid polluting the receiving water (9). Of course, it should be noted that ABS could enter the environment from other sources; e.g., from industrial or agricultural uses. The conclusions reached here may not always apply in those cases.

Observance. The concentration of ABS in household sewage is often in the range of 1 to 20 ppm. The level found depends on many factors—per capita water usage, per capita detergent consumption, and types of detergent formulations used.

ABS in water at 1 to 20 ppm will foam when agitated. It was this tendency to foam that brought attention to BAS in receiving waters in the 1950's. Previously, the most commonly used surfactant, soap, was not observed foaming in sewage because soap precipitates with the hardness ions in the water and is quickly degraded.

Fate. The most effective way to treat sewage is to promote the oxidative assimilation of the organic matter by microorganisms. Both an adequate supply of microorganisms and oxygen must be provided. Man-made organic compounds, such as ABS or soap, which are present in domestic sewage are also assimilated by the microorganisms.

The total assimilation of organic compounds proceeds by a number of consecutive enzymatic steps which can proceed along various pathways. The pathway which follows the faster steps is favored. And the compound which contains structural features which are easily attacked is assimilated first. All this is true for ABS molecules as well as for other organic compounds. Degradation experiments indicate that the ABS molecule is normally attacked first on the end of the alkyl group, and then step-by-step the whole molecule is converted into CO₂, water, and sulfate ion (10-12). As discussed in greater detail below, the rate depends on the specific ABS molecular structure.

Problems. Clearly, ABS or any other surfactant component of household sewage is not a significant environmental problem as long as the sewage itself remains present. The sewage is the overriding problem. It contains pathogenic organisms and possesses a high biological oxygen demand (BOD), which is harmful to aquatic life. If, however, the ABS is still present after the sewage has been well treated, it may then be a problem if it is found to be harmful in some way.

This was the situation with the use of BAS in a number of localities in the industrial countries in the late 1950's and early 1960's (13, 14). In a number of instances, enough BAS remained after sewage treatment to cause foaming difficulties in sewage treatment plants and at their outfalls. Generally, the concentration at that point was in the 1-5 ppm range. After further dilution in the receiving waters below about 1 ppm, foaming was not observed.

Because of many studies showing the relatively innocuous toxicological nature of ABS, (15, 16) the U.S. Public Health Service allowed the presence of ABS (up to 0.5 ppm) in drinking water which was affected by the upstream use of detergents (17). There is no effect on taste from ABS when its concentration is below about 16-40 ppm (18, 19).

Surfactants, in general, do show some biological effects which are the result of their tendency to adsorb on biolog-

ical membranes. For example, surfactants can produce morbidity with sensitive fish when present at the 1-5 ppm level in clean water (20-22). This is not an important factor when the surfactant is present in untreated household sewage at the 1-5 ppm level because the sewage itself is inimical to the fish.

As stated earlier, when surfactants biodegrade, they are converted stepwise by oxidation into smaller molecules and are eventually fully assimilated. The oxidation intermediates from ABS generally contain an ionic carboxyl group as well as the sulfonate group. Because these molecules have gained the polar carboxyl group and have lost some of their hydrophobic hydrocarbon chain, they have lost their balanced hydrophobic/hydrophilic surfactant character. They no longer respond to surfactant analytical tests and they no longer exhibit surfactant behavior. They do not foam. They will not show the biological effects of the original surfactant (10).

Therefore, in terms of controlling environmental problems from ABS such as foaming, it is essential to measure the surfactant concentration itself. It is not meaningful to measure the nonfoaming oxidation intermediates which are not surface active. There is no reason to suspect that these oxidation intermediates are any more troublesome than the other partially oxidized organic compounds from the sewage. Recently, the prestigious British "Standing Technical Committee on Synthetic Detergents" concluded that "there was no cause for concern" from "the presence of traces of chemical residues of detergent origin" after sewage treatment (23).

Biodegradation Tests. Many tests have been devised to measure the rate of decay of surfactants. Some are batch, some continuous, some "fill-and-draw" semicontinuous. There can be a wide range of biological activity in the tests, depending on the concentration and types of organisms, the organic substrates previously assimilated by the organisms, and the rate of aeration. Therefore, test results (e.g., percent remaining in continuous tests or time required for 90% removal in batch tests) are not meaningful unless test conditions are specified. When comparing two surfactants, it is essential to run the same test on both.

The best tests are those which simulate what would actually happen to the surfactant as a component of sewage, i.e., which simulate sewage treatment. The surfactant should be dissolved in real or simulated sewage at the concentration at which it would be found in use. The mixture should be treated in a realistic way. Usually, this means simulating a well-run secondary treatment system using retained activated sludge.

Such activated sludge tests, however, are relatively complicated to run, and very often simple batch "die-away" tests are run instead. These die-away tests are very useful screening tests, but the results should be treated with caution. If the biological system is not fully acclimatized or is too dilute, the test may show differences between surfactants that are not seen in a real situation. Because of bacterial inhibition or lack of acclimatization, one may see no decay for long periods, whereas with an active bacterial system, the same surfactant will rapidly decay (24). One can even see a reversal in relative rates of decay of surfactants in these weak systems. In fact, Swisher shows a test in which BAS decayed faster than LAS (25).

Therefore, even in the batch die-away tests, one should

ensure that an active, robust biological system is present. This can easily be done by adding a few percent of secondary sewage treatment effluent to the medium. Even this, however, does not avoid all acclimatization worries. Concerns about acclimatization are answered by running continuous tests or by preparing the bacteria for a batch test by allowing them to degrade the surfactant in a preliminary exposure period.

These points were taken into consideration when the various standard tests were drawn up. The ASTM, for example, describes two tests: a relatively simple "shake-flask" die-away test and a more reliable activated sludge sewage treatment test (26).

Once a reliable test has been run, there is no obvious way to decide what is a satisfactory degree of removal. The decision is completely arbitrary. One could logically compare the removal with the removal of other organic components of the sewage itself. One could correlate with the decrease in Biological Oxygen Demand (BOD) or Ultimate Oxygen Demand (UOD) of the whole sewage.

A more sensible approach is to correlate laboratory tests with behavior in the field and then to establish what laboratory test result correlates with avoidance of environmental problems. This was done in the 1960's with ABS. It was determined that LAS was almost completely removed by normal secondary sewage treatment in the field (2, 3, 4). BAS was only about 65% removed (12).

At these removal levels, enough BAS remained to give foaming difficulties, whereas the lower amount of LAS did not. Therefore, the laboratory tests were designed so that LAS removal was at least 80-90%, while BAS removal was low.

The position of requiring about 90% removal of an ABS fits in well with the normal removal of about 90% of the sewage BOD during a good sewage treatment process (27, 28). For environmental protection, it is not reasonable to require a higher level of removal of an organic pollutant unless it is considered more harmful than the sewage itself. This is not true with ABS.

As discussed above, it is only appropriate to use biodegradability test methods which measure the amount of

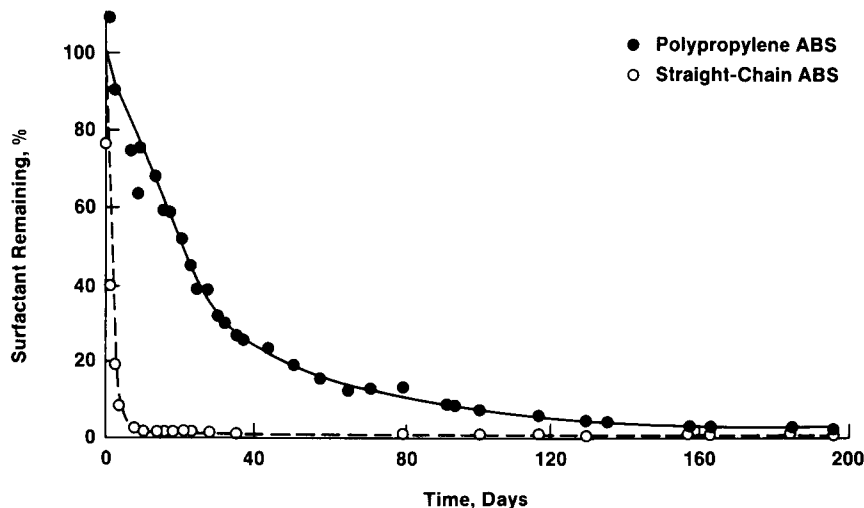
intact surfactant. So-called "ultimate" biodegradability tests which measure the overall conversion to CO_2 and water are not appropriate because the oxidation intermediates present no problem as they proceed down the path to total oxidation (10). So, while the rate of loss of surfactant is important, the rate of conversion of the oxidation intermediates is not.

BIODEGRADATION RATES OF ABS

Biodegradability of BAS. As stated above, BAS can give environmental foaming problems and does not pass laboratory biodegradation tests, whereas LAS cures these problems and passes the tests. Many people have misconstrued this difference by saying LAS is "biodegradable" and BAS is "not biodegradable." Even the most scholarly and authoritative sources occasionally slip into this mistaken terminology (29). On the contrary, there is widespread and overwhelming evidence that all the various ABS structures will fully biodegrade, given enough time. Early on, Sharman (30) showed this for BAS in an elegant fish tank experiment. His decay curves for BAS and LAS are reproduced in Figure 1. Robeck *et al.* (11) who also used radioactively tagged BAS similarly reported up to 98% removal in soil lysimeter experiments. Even Swisher's early, simple die-away tests showed about 85% removal in 30 days (25).

BAS is made from propylene oligomer which consists of hundreds of branched isomers and homologs centering around C_{12} . The highly branched olefins form BAS molecules which are very slow to degrade. The less branched ones form BAS molecules which degrade faster. This causes the die-away curves for BAS to be concave upwards, as seen in Figure 1. The faster components degrade first.

In a second series of experiments, Sharman *et al.* (31) showed that BAS remaining in secondary sewage treatment plant effluent can be recovered by a foam separation process and recycled to the aeration zone where more of it is decomposed. In this way, the overall BAS removal was increased from about 65% to 85% during



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FIG. 1. Disappearance of surfactants in active, stable fish tank experiments • Polypropylene ABS, ○ Straight-Chain ABS. Reprinted by permission from *Nature*, Vol. 201, p 705, Copyright © 1964, Macmillan Journals Limited.

normal sewage treatment. With higher foaming efficiency equipment and modifications in the aeration equipment, even higher removals could be obtained. This approach was tried on a commercial scale in the United States but was not needed after the industry switch to LAS. Again, these experiments showed that all the BAS molecules will progressively degrade.

There were some literature suggestions that BAS disappearance came from removal by adsorption rather than biodegradation (32); but a number of studies (33), including Sharman's (31), have shown adsorption to be minor effect.

Therefore, the slow but eventually complete degradation of BAS has been well documented. The rate is actually comparable to that of some of the natural components in sewage. These slower components can be measured in a UOD test instead of the usual, five day BOD test. After normal sewage treatment, about 25-30% of the UOD is still present (34).

Biodegradability of LAS. LAS degrades rapidly during sewage treatment and in reasonable laboratory tests. The straight-chain character allows the fast degradation to occur.

LAS consists of a mixture of relatively few isomers and homologs. In certain mild tests, one can see differences in degradation rates between these isomers and homologs (35); but in more active tests, these differences disappear (24). In continuous tests, removals are very high, and in die-away tests, the decay curves are almost linear.

In tests simulating good sewage treatment, pure, straight-chain ABS molecules show very high removal values of about 99% (12). Practical LAS products, however, do not show removal levels that are quite as high, because these LAS products contain minor amounts of ABS impurities which are slower to degrade (36). Commercial LAS products are usually only 90-97% degraded in continuous tests. In batch die-away tests, the decay

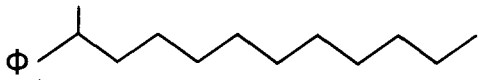
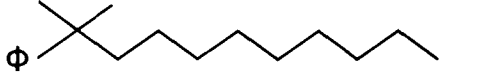
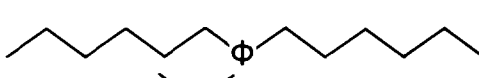
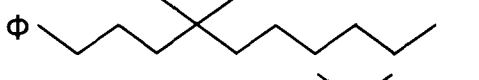
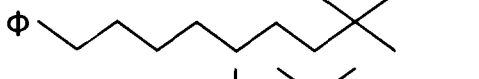
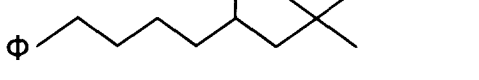
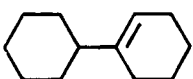
	<u>Percent Remaining</u>
1. BAS	35
2. LAS	~2
<u>Alkylate Precursor:</u>	
3. 	0.4
4. 	0.3
5. 	1.2
6. 	1.8
7. 	16
8. 	44
<u>HF/Benzene Alkylates from:</u>	
9. Pure n-Dodecene	0.3
10. Ziegler Dimer of 1-Hexene (2-Butyl-1-Octene)	3.3
11. 	44
12. Dialkylate from 1-Hexene	80

FIG. 2. ABS removal in sewage treatment tests. ϕ = Phenyl

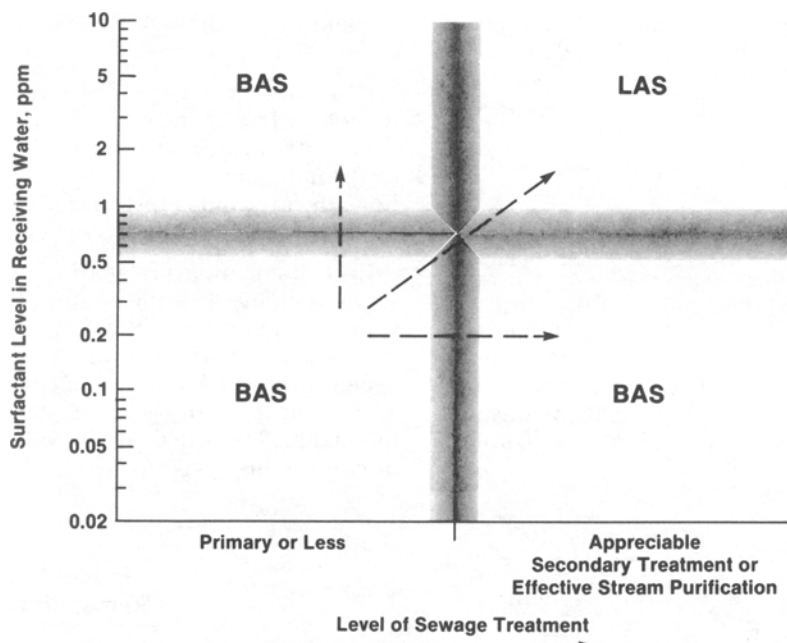


FIG. 3. Choice of BAS or LAS is dictated by local conditions.

curves show an appreciable slowdown in rate at about 5–15% remaining.

However, the presence of these minor amounts of slower degrading impurities in LAS is not a problem. They, like BAS, will fully biodegrade, given enough time. The amount is small enough so that after sewage treatment, the residual ABS concentration is below the level which can cause foaming difficulties.

Other ABS Structures. Many studies have shown that the rate of biological oxidation of an ABS molecule is highly dependent on its structure. It is generally true that straight-chain molecules degrade faster than branched ones, but there are subtleties in behavior that are not well understood. Therefore, any new ABS structure needs to be tested to determine whether, as discussed above, it can pass the tests established in the industrial countries where sewage is well treated.

Tests run on pure compounds illustrate this complexity. Figure 2 lists percent ABS remaining for various ABS structures in tests that provided good sewage treatment (12). Some of the results are surprising. Quaternary branching, which is thought to impede biological assimilation, has no effect on surfactant disappearance when it is next to the benzene ring (No. 4 vs No. 3), has only a slight effect when it is part way down the alkyl chain (No. 6), and has a clear negative effect when it is at the end of the chain (No. 7). Note, however, that Compound No. 7 degrades faster than BAS. Apparently, BAS is slower because it contains molecules with a higher degree of branching, like Compound No. 8. However, even some structures, such as No. 11 and No. 12, which don't appear highly branched are very slow to degrade. Apparently, there are other very important structural or shape factors which influence the enzymatic processes involved. It is very surprising to see the large difference between Compounds No. 5 and No. 12.

Overall, while one can say that highly branched struc-

tures are poor, it is not true that all unbranched structures are good (Compound No. 12). It is true that molecules with some branching, preferably away from the end of the chain, are equal to commercial LAS and will pass the biodegradability tests. New ABS molecules must be tested to be sure.

It is also interesting to note that Compound No. 4, with branching next to the benzene ring, showed a very high conversion, 70%, to inorganic sulfate during the test. Inorganic sulfate appears when the molecule is completely degraded. The branches did not block the stepwise complete decomposition.

CRITERIA FOR CHOICE OF ABS

This discussion has emphasized the significant difference in aerobic biodegradability between BAS and LAS. Because of this difference, LAS is used in the industrialized countries. However, a large amount of BAS is still used worldwide. Does this mean that BAS is causing a pollution problem in the areas where it is used?

The answer in many cases is "No, it is not, for two simple reasons." Either the amount being used is too low to produce the foaming problem or the sewage that it is found in is not being treated adequately before the receiving water flows out to the sea. These two aspects are incorporated into Figure 3, a diagram which suggests the circumstances under which a given country might decide that LAS was preferable to BAS.

In Figure 3, we see four quadrants. In both lower quadrants, BAS is not a pollution problem because the usage level is such that the concentration in the receiving waters is below about 1 ppm, the foaming threshold.

In the upper left-hand quadrant, the concentration is above 1 ppm, but adequate sewage treatment is not occurring, either in treatment plants or in the rivers. The inadequate nature of many river or canal systems can

often be observed by measuring the dissolved oxygen content. If it is low because of the effect of a heavy sewage load, neither BAS nor LAS will degrade. Therefore, in this quadrant, BAS is still acceptable for use because the sewage itself is the overriding pollution problem.

Only in the upper right quadrant, where the sewage is receiving adequate treatment, is LAS needed. This quadrant corresponds to the situation in parts of the United States, Europe, and Japan.

Despite the logic of Figure 3, some developing countries have required the use of LAS simply because the industrialized countries require it. The more intelligent approach has been to study the local situation to see if any serious problem exists with the use of BAS and to decide whether switching from BAS to LAS would produce any observable benefit. For example, studies have been made in Indonesia, India, Malaysia, Mexico, the Philippines, and Thailand (37-39). In many countries, the conclusion was to continue BAS use. Apparently, BAS has cost and/or property advantages over LAS.

There is a corollary to the finding that it is appropriate to use BAS in many countries. If, in the future, the situation in a given country moves toward the upper right quadrant of Figure 3, that country could decide that the level of biodegradability required for the ABS does not need to equal that for countries which are well into the quadrant. That is to say, they could opt for a BAS/LAS mixture or for some other ABS product not quite equal to LAS in biodegradability.

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ERRATUM

In the article, "Synergistic Stabilization of Perfluorocarbon-Pluronic F-68 Emulsion by Perfluoroalkylated Polyhydroxylated Surfactants," which appeared in the October 1989 issue of *JAOCs* (pp. 1515-1523) the trademark appeared without the accompanying company identification of "a product of BSAF, Wyandotte, Michigan."