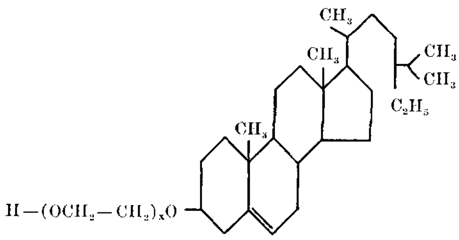
Tall Oil Studies. IV. Synthesis and Comparative Detergency of Built Polyethenoxy Sitosterols

J. V. KARABINOS and A. T. BALLUN, Research Laboratories,
Blockson Chemical Company, Joliet, Illinois

In conjunction with the earlier studies (1) on the polyethenoxy nonionic detergents from the rosin and fatty acids of tall oil, it was noted that one of the unsaponifiable constituents of tall oil, namely, β-sitosterol (2) contains a hydroxyl group which could be converted into a polyethenoxy derivative. In a subsequent study with 12-hydroxystearic acid (3) it was shown that the carboxy and primary alcoholic groups were condensed with ethylene oxide in preference to the secondary hydroxyl group even in the presence of a more alkaline catalyst such as potassium hydroxide. Though these results indicated that ethenoxification of a tall oil containing a sitosterol would probably leave the latter substance unchanged in the polyethenoxy tallate condensate, it seemed desirable to prepare the polyethenoxy ether of tall oil β-sitosterol independently and examine its detergent properties in a built mixture in comparison with the other nonionic detergents previously cited (1, 3).

Preparation of Polyethenoxy β-Sitosterol

The crystalline tall oil sterol (m.p. 131-133°C.) was isolated and generously supplied by F. J. Ball of the West Virginia Pulp and Paper Company and used in these experiments without further purification. The sterol (200 g.) was condensed progressively with weighed quantities of ethylene oxide in the presence of potassium hydroxide (0.5 g./100 g. of sterol) at 170-190°C. as described previously (3). No reaction was observed using potassium carbonate catalyst. The polyethenoxy β-sitosterol thus prepared is represented by the following structural formula where the number of ethenoxy units, given by x, was determined by accurately weighing the reactants and final product.



x = 8, 12, 16, 18, 20, 22, 24, 26

Discussion

Detergency tests, *i.e.*, soil removal and whiteness retention, were carried out in hard and soft water on each of these substances using 20% by weight of the polyethenoxy sterol in the test mixture and assuming 100% detergency values for the same standard previously employed (1). The detergency tests depicted in Figure 1 show the same typical curves previously illustrated (1, 3) in which the ethenoxy compound gives increasing detergency with ethenoxy chain length to an optimum point whereupon these values level off. As with the polyethenoxy alkanoates, this optimum detergency value is reached when the number of ethenoxy units reaches approximately two-thirds of the number of carbon atoms in the hydrophobic or sterol portion of the molecule. This empirical rule cited previously (3) seems to hold equally well for a highly condensed polycyclic molecule like the sterol as well as for the polyethenoxy alkanoates.

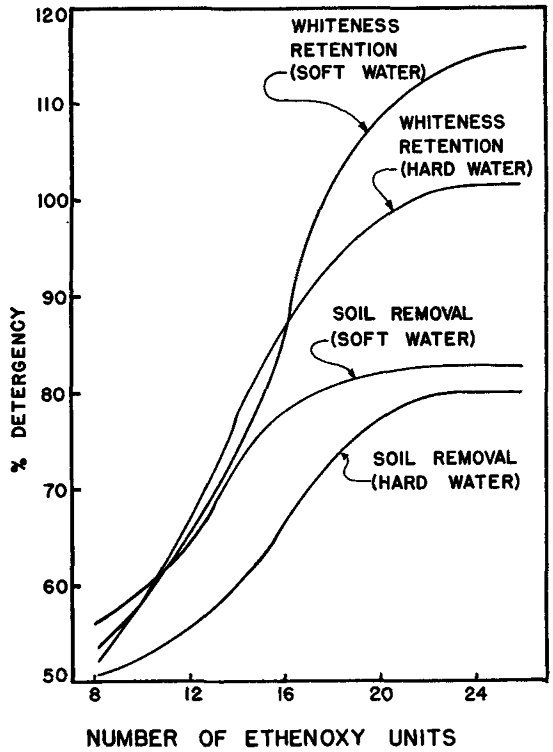


FIG. 1. Variation in detergency of polyethenoxy tall oil sitosterol with number of ethenoxy units.

It should be pointed out however that the polyethenoxy sitosterols do not attain as high detergency values, particularly with respect to soil removal, as do certain tall oils or fatty acids. However it is quite possible that the polyethenoxy sitosterols being water-soluble or water-dispersable sterol derivatives may possess other interesting uses.

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