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Tall Oil Studies. III. Bactericidal Activity of Polyethenoxy Tallate Ozonides. Identification of the Active Principle

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IN the preceding article (1) it was shown that polyethenoxy tallates (tall oil-ethylene oxide condensates) could be successfully decolorized with ozone. We have shown herein that completely ozonized polyethenoxy tallates can be prepared and that these substances are bactericidal nonionic detergents. It was further noted that polyethenoxy tallate ozonides containing a larger proportion of fatty acid were more effective bactericides than those containing more rosin acid. Polyethenoxy oleate ozonide was then prepared and showed greater germicidal activity than the corresponding tall oil derivatives. Up to this point the fat-soluble, water-soluble ozonides had been assumed responsible for the germicidal activity. However it was soon found that decomposing the ozonides to their corresponding acids (*e.g.*, by hydrogen peroxide in aqueous acetic acid) not only increased bactericidal activity but that the active principle could be separated from the residual polyethenoxy esters by steam distillation. The activity was volatile with steam. The steam distillate was investigated and found to contain pelargonic acid (I)

$$CH_3—(CH_2)_7—COOH \qquad (1)$$

a known decomposition product from the ozonization of oleates. Pure pelargonic acid was then obtained, and its bactericidal activity was ascertained. It soon became evident that the active bactericidal principle was essentially pelargonic acid and that the activity varied with the pH of its solution. In connection with these results the effect of pH on the germicidal activity of the fatty acids in the 9- to 12-carbon range was studied and undecylic acid exhibited the highest activity (2). Figure 1 illustrates the bactericidal activity of pelargonic acid *vs.* pH.

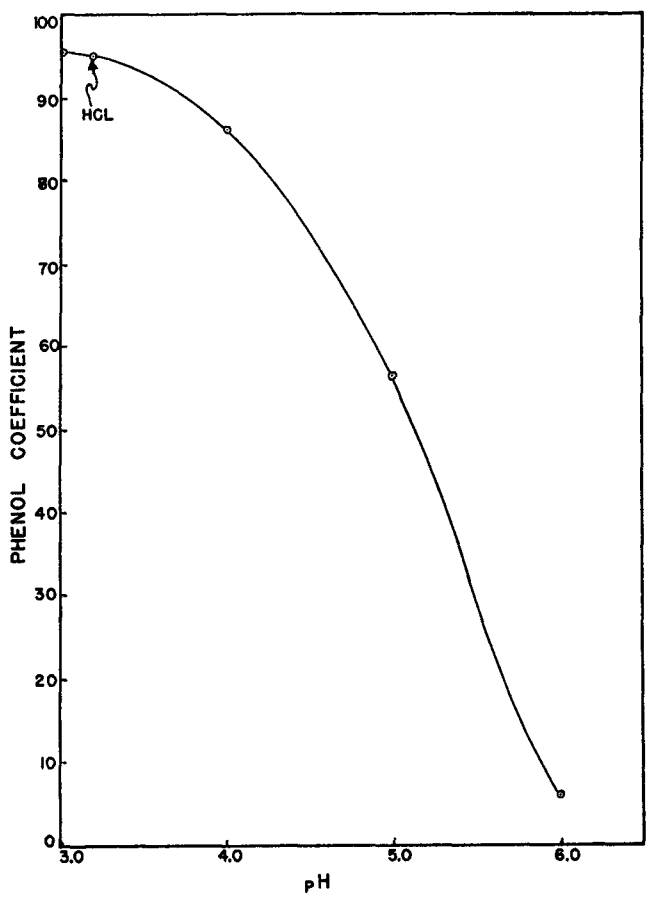

FIG. 1. Variation in "phenol coefficient" of pelargonic acid with pH.

Experimental Details

Preparation of Polyethenoxy Oleate Ozonide. Polyethenoxy oleate was prepared by condensation of pure oleic acid with 13 moles of ethylene oxide as described in a preceding article (3). Ozone, generated by the apparatus described in our previous report (1), was passed into 100 g. of polyethenoxy oleate until the gas was no longer absorbed. A total weight of 5.9 g. of ozone was absorbed by the ester giving polyethenoxy oleate ozonide as a water-soluble, light yellow oil with detergency values (*i.e.*, soil removal and whiteness retention in hard and soft water) comparable to the original polyethenoxy tallates (4). The ozonide possessed a phenol coefficient of 11 without acidification. Similar ozonides prepared from polyethenoxy tallates possessed considerably lower phenol coefficients. It was noted that the phenol coefficient decreased with increasing rosin acid content of the original tall oil.

Decomposition of Polyethenoxy Oleate Ozonide with Hydrogen Peroxide. The polyethenoxy oleate ozonide (20 g.) prepared above was dissolved in a mixture consisting of 100 ml. of water and 25 ml. of glacial acetic acid. Ten ml. of 30% hydrogen peroxide was added to the mixture, which was allowed to reflux for 1½ hours. A further quantity of hydrogen peroxide (2 ml.) was added with an additional reflux of 1½ hours. The product was then concentrated to a light yellow residual oil weighing 22 g., which was

TABLE I

Variation in Bactericidal Activity of Pelargonic Acid

pH	Dilution	Time in Minutes		
		5	10	15
6	1: 500	−	−	−
	1: 1,000	+	+	+
5	1: 7,000	−	−	−
	1: 8,000	+	+	+
4	1: 11,000	−	−	−
	1: 12,000	+	+	+
3	1: 12,500	−	−	−
	1: 13,000	+	+	−
	1: 14,000	+	+	+
Phenol	1: 120	−	−	−
	1: 130	+	−	−
	1: 140	+	+	−
	1: 150	+	+	+

water-soluble and still possessed fair detergent properties.

The phenol coefficient of the acidic product had increased considerably. The actual value depended upon the pH of the test solution. Ten g. of the above oil was steam-distilled in the customary manner, and the oil obtained in the steam distillate was separated from the aqueous solution and dried. It had a b.p. of 250° C., and it gave a *p*-bromophenacyl ester melting at 66°C. At a pH of 4 it gave a phenol coefficient of 85 (see Fig. 1). Since pelargonic acid possessed identical physical constants (5), the active bactericidal principle in the polyethenoxy oleate ozonide was assumed to be pelargonic acid. In cases where the ozonide had been decomposed to carbonyl products, such as zinc dust, the bactericidal activity was considerably less.

Bactericidal Activity of Pelargonic Acid vs. pH. To 1 g. of pelargonic acid and 490 ml. of distilled water was added sufficient *N* sodium hydroxide solution to bring the pH to 9.5. The solution was diluted to 500 ml. with water, and from this stock solution further dilutions of sodium pelargonate were prepared (see Table I). Fifty-ml. fractions of each dilution were titrated separately with 20% aqueous acetic acid to pH values of from 3 to 6. In the actual phenol-coefficient test (6) 5 ml. of sodium pelargonate solution was placed in each test tube and the pH adjusted with 20% acetic acid from the data previously determined. The volume of acetic acid necessary to adjust the pH from 9.5 to from 3 to 6 was so small as to have a negligible effect on the concentration of pelargonic acid. The phenol coefficient tests were performed, using *Staphylococcus Aureus* as the test organism. The results of the bactericidal tests are recorded in Table I, and Figure 1 represents the phenol coefficient of pelargonic acid as a function of the acidity. Further results with other organisms are reported elsewhere (2).

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Yield and Chemical Composition of Sesame, Sesamum indicum L., as Affected by Variety and Location Grown[1]

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SESAME has been grown in the Eastern Hemisphere since time immemorial as a source of edible oil and nutritious protein food for human consumption. Sesame oil is noted for its excellent flavor and stability. The protein meal is especially rich in the amino acid methionine. Most of the sesame seed imported into the United States enters the confection and bakery trade as whole seed and is probably most familiar as a topping on hard rolls and French bread.

Budowski and Markley (2), in a review paper listing 258 references, discussed world production, imports into the United States, processing, and other topics as well as the chemical and physiologic properties of sesame oil. Swingle (9) prepared a library list of 216 references covering the literature on genetics, cultural practices, history of production, marketing statistics, chemistry, nutrition, and utilization of sesame and its derived products. More recent work with this species in the Western Hemisphere and India was reported at the First International Sesame Conference (7).

Sesame seed is well received by the oilseed processing industry of the United States. Sesame has not been grown on a commercial scale in this country because of the large amount of hand labor required in harvesting. In normal dehiscent (shattering) varieties the capsules open at maturity, and considerable care is required to prevent excessive loss of seed. It was only after the discovery of a single indehiscent plant by Langham (5) in 1943 that the complete mechanization of the crop became a possibility. The indehiscent character is under the control of a single recessive gene, but there must be several modifying genes. The future of sesame production in the United States is largely dependent upon the successful development of satisfactory indehiscent varieties. Breeding programs have been initiated, and progress is being made toward development of such varieties.

[1]Contribution of the Division of Tobacco, Medicinal, and Special Crops, Bureau of Plant Industry, Soils, and Agricultural Engineering, and the Southern Regional Research Laboratory, Bureau of Agricultural and Industrial Chemistry, U. S. Department of Agriculture.

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