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Protective Films for Pilot-Plant Deodorizers[1]

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IT has been demonstrated many times that contamination by even exceedingly small amounts of certain dissolved metals lowers the quality of edible fats and oils (2). Lundberg made an extensive review of work on the stability of fats and oils (3). In some products flavor deterioration is observed in the early development of rancidity or of "oxidized" flavors. Vegetable oils, particularly soybean oil, develop the off flavors generally described as "reverted," and Evans has described the limits of tolerance of deodorized soybean oil for a number of metals before flavor stability is impaired (1). The oils also have a tendency to develop increased amounts of peroxidized products, especially in the presence of iron at elevated temperatures. Considerable metallic contamination of oils that have undergone refining, followed by water washing and adequate bleaching to remove soaps, probably occurs in the deodorizer since it has been shown that adequate bleaching removes metallic soaps effectively (5). Metal picked up in the hydrogenator should be largely removed by a second bleaching operation. Unless metallic contamination is avoided as much as possible at every stage of processing, the presence of prooxidant metals is conducive to the breakdown of natural antioxidants when the oil is exposed to air.

In their work with cottonseed oil shortening, Ziels and Schmidt recognized the importance of the deodorization step in its effect on the quality of the oil (7). Their results undoubtedly have led to the increased use of aluminum and nickel in the construction of deodorizers. From a practical viewpoint there appears to be no ideal material for deodorizer construction. Glass or porcelain-lined equipment would be most desirable from the standpoint of oil quality. But glass or porcelain surfaces are reported to erode when subjected to steam impingement, they have limited resistance to mechanical and thermal shock, and they are not resistant to alkaline cleaning solutions. Aluminum does not lower oil quality appreciably, but it lacks structural strength and is not resistant to alkaline cleaners. Nickel has been used successfully, but it is relatively expensive and many fabricators are unfamiliar with it. Mild steel has desirable structural properties and is not as expensive, but it affects oil stability adversely; certain types of stainless steel are better in this respect but are more expensive.

The present work was undertaken primarily to determine the best material for constructing a pilot-plant unit for deodorizing soybean oil. Although pilot-plant operations and equipment should, in general, follow the lines projected for manufacturing practice, special considerations exist where edible oils are concerned. Pilot equipment must be cleaned frequently to prevent contamination of successive experimental runs and after the equipment has been cleaned or has been idle for some time it is often necessary to process several "washout" batches until a protective oil film is formed over the metal surfaces. Intermittent operation is typical of pilot experiments and necessitates frequent cleaning. Although metal scavengers may be used in commercial practice to deactivate metal contaminants, their use would interfere with experimental results in the pilot plant.

Laboratory tests show that the problem of contamination of soybean oil by metals during deodorization is one of simple fatty acid corrosion. Polishing the surface of mild steel or stainless steel greatly reduces corrosion of the metal while depositing certain protective films thereupon makes the surface nearly equal to a glass surface so far as the effect on the oxidative and flavor stability of soybean oil is concerned. The treatment may be accomplished without processing several preliminary batches of oil in the deodorizer.

Experimental Methods

Refined, bleached soybean oil was used for all the investigations reported here. One batch of refined, bleached oil was obtained from a commercial processor; other batches were prepared in pilot-plant equipment at the Northern Regional Research Laboratory. The oils were deodorized at 200° ± 1°C. in 500-ml. batches in 1-liter glass deodorizers arranged so that two flasks could be operated simultaneously from the same vacuum pump and dry ice condenser. A diagram of the apparatus is shown in Fig. 1. The flasks were heated by electrical resistance heaters and the temperature was maintained by an on-off controller.

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[1] Report of a study made under the Research and Marketing Act of 1946. Presented before the American Oil Chemists' Society Meeting, May 1-3, 1950, Atlanta, Ga.

[2] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

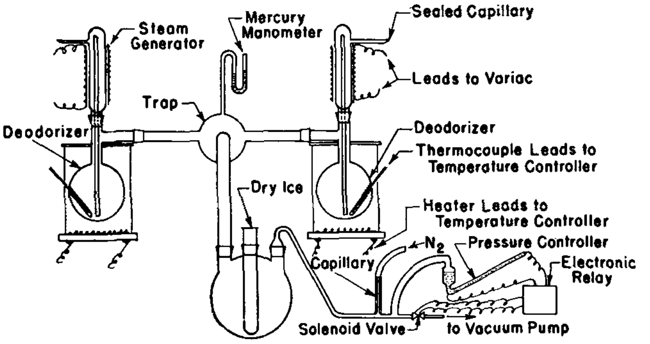

FIG. 1. Apparatus for deodorization of soybean oil.

The pressure was held at 6 mm. of mercury absolute during all deodorizations by means of a solenoid valve between the dry ice trap and the vacuum pump. The solenoid was controlled by a mercury switch and a relay. Nitrogen was introduced continuously into the dry ice trap through a fine capillary tube to eliminate small fluctuations in pressure. Deodorizations were of 6 hours' duration and about 100 cc. of distilled water which previously had been boiled was vaporized and bubbled through the oil as the stripping agent. These conditions were chosen because of their similarity to commercial practice.

The effect of metals upon the oil being deodorized was obtained by introducing two metal strips 5″ x 15/16″ x 1/16″ into the deodorization flask after addition of the oil. The ratio of metal surface area to weight of the oil was thus about 20 square inches per pound of oil or about five times the ratio that is found in commercial batch deodorizers. The level of oil in the glass deodorizer flask was such that only about half of each metal strip was submerged although the entire strip was subject to the splashing action of the oil. During later experiments a single strip of metal comparable in surface area to the two strips previously used was rolled into a three-quarter cylinder of sufficient length to project into the neck of the flask where it was held fairly rigid—necessary because the motion of the two strips against the glass stem was sufficient to rub off the film coating the strips. This motion could be sufficiently vigorous to abrade even the metal. Unless noted otherwise, all metal strips except aluminum or those coated with aluminum were cleaned by immersing them for 2 to 4 hours in a hot aqueous solution containing 2% trisodium phosphate and 4% sodium hydroxide, followed by thorough rinsing in distilled water. Citric acid, when used as a metal scavenger in a series of tests with the same metal strips placed in the deodorizer, was added as a 20% aqueous solution just prior to heating the oil for deodorization.

The stability of the deodorized oil was determined by oxidizing a 25-cc. sample in a 1″ x 6″ open test tube immersed in a 100°C. oil bath with filtered air bubbling through the oil at the rate of 0.4 liters per minute. Aeration times were generally 6 hours for each oil, but some samples were aerated for both 4 and 6 hours. Peroxide values were determined on 10-g. samples of the oil by a slight modification of the Wheeler method (6). The iron analyses reported on four of the samples were made by the emission spectographic method of Melvin and Hawley (4).

Results and Discussion

The relative prooxidant effect produced by 13 commercial metals and alloys during the deodorization of soybean oil is shown in Figure 2. The composition of the alloys is given in Table I. Each point on a curve represents an average of two or more peroxide value determinations. As might be expected from results reported on cottonseed oil shortening, the alloys containing appreciable amounts of iron have a greater effect than the aluminum and nickel alloys, and copper has an extremely bad effect. Molybdenum, which is present to the extent of 2 to 3% in type 316 stainless steel, apparently decreases the prooxidant effect of stainless steel on the oil.

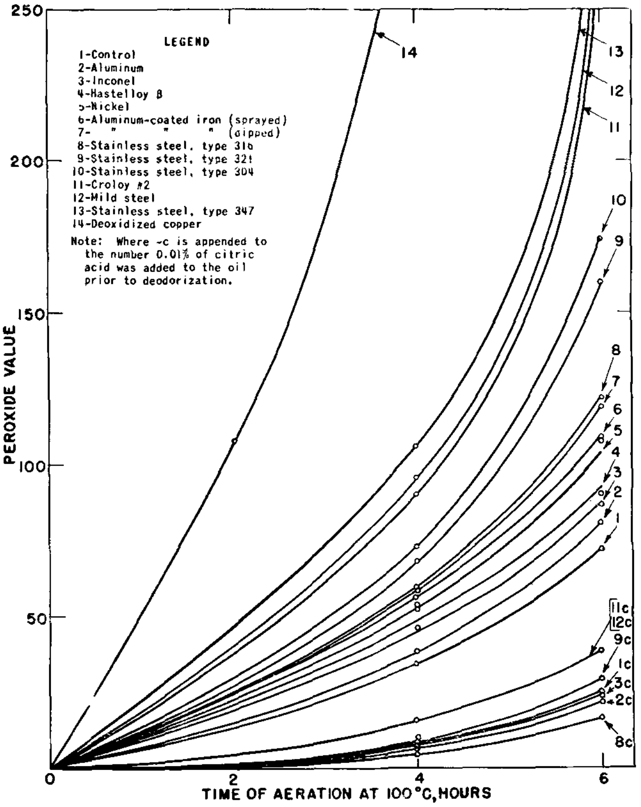

FIG. 2. Stability of refined, bleached soybean oil after deodorization with strips of metal present.

TABLE I

Composition of Metal Alloys Used in Stability Tests

Sample	Fe	Ni	Cr	Mn	Mo	Si	Cu	Ti	C
Stainless steel type 304	73.2	8	18	0.75					.08
Stainless steel type 316 (Allegheny 18-8M)	63.7	14	18	1.75	2.5				.08
Stainless steel type 321	71.5	10	18					.4	.10
Stainless steel type 347	69.9	10	18	2.00					.10
Croloy type 2	96.9		2	0.5	0.5				.10
Inconel	6.5	79.7	13	0.25		0.25	0.2		.08
Hastelloy B	4.7	65.9		0.6	28.6	0.2			.05

Reference. Chem. Eng. *45*, No. 11 (1938).

The addition of 0.01% of citric acid to the oil prior to deodorization greatly reduces the effect of the metals on soybean oil. This is clearly shown in Figure 2. The stability of some of the samples deodorized with the various metals plus citric acid exceeded that of the control sample plus citric acid, probably because of a variation in the amounts of the acid used or other uncontrollable variables.

It has been suggested, but not demonstrated, that metal contaminants are present in vegetable oils in the form of soaps (7). Small amounts of fatty acid in the oil might attack the surface of any metals present and form soaps that would be washed from the surface of the metal by the oil during the violent agitation in the deodorizer. It was found (Table II) that strips of stainless steel type 347, previously cleaned with 5% caustic solution and rinsed, lowered only slightly the stability of a refined, bleached soybean oil having an acid value of 0.07%. However, when 0.3% of pure stearic acid was added to the oil and the oil was again deodorized with the same metal present, the stability of the oil was significantly lowered. The addition of the acid alone affected the stability but slightly, demonstrating that lowered stability resulted from the combination of metal, acid, high temperature, and violent agitation.

TABLE II

The Effect of Free Fatty Acid and Temperature on the Lowering of Oxidative Stability of Soybean Oil By Contact With Metallic Surfaces

Sample	Average peroxide value	
	Aeration at 100°C.	
	4 hr.	6 hr.
1. Soybean oil No. 1 deodorized in glass with no metal present, (% FFA = 0.07)	29	60
2. Oil No. 1 deodorized with stainless steel type 347, cleaned in hot 5% NaOH and rinsed, present	37	70
3. Oil No. 1 with 0.3% stearic acid added, deodorized in glass with no metal present	39	65
4. Oil No. 1 with 0.3% stearic acid added, deodorized with stainless steel type 347, cleaned as in 2, present	66	170
5. Soybean oil No. 2, control (% FFA = 0.09%)	....	47
6. Oil No. 2 heated at 100°C. under N_2 for 4 hr. and deodorized	....	46
7. Oil No. 2 heated at 200°C. under N_2 for 4 hr. and deodorized	....	34
8. Oil No. 2 with 0.3% distilled fatty acids added, and deodorized	....	103
9. Oil No. 2 with 0.3% distilled fatty acids added, heated at 100°C. under N_2 for 4 hr. and deodorized	....	78
10. Same as No. 9 except heated at 200°C	....	76
11. Oil No. 2 heated at 100°C. under N_2 for 4 hr. with strips of mild steel, cleaned in hot 5% NaOH and rinsed, present	....	35
12. Same as No. 11 except heated at 200°C	....	48
13. Same as No. 11 except 0.3% soybean fatty acids added to oil	....	82
14. Same as No. 13 except heated at 150°C	....	62
15. Same as No. 13 except heated at 200°C	....	800

NOTE: Samples 5 and 8 were deodorized in glass with no metals present. Samples 6, 7, and 9 through 15 were treated in a test tube as shown and then deodorized in glass with no metals present. Agitation in the test tube was provided by bubbling N_2 through the oil.

Further experiments to determine the influence of temperature on the corrosion of mild steel in soybean oil indicate that extensive contamination occurs at 200°C. but not at 150° if the acid content of the oil is about 0.4%. If the acid content is less than 0.1% however, the corrosion is not appreciable at either temperature under the conditions used. In these experiments strips of mild steel were placed in a large test tube containing soybean oil and the tube was heated in a brass cylinder, the temperature of the system being controlled electrically. High purity nitrogen was bubbled through the oil to provide agitation and to prevent oxidation of the oil. After 4 hours the oil was cooled and the metal strips were removed. The oil was then deodorized in glass apparatus. When the oil, containing 0.1% fatty acid either alone or with metal strips added, was heated in the test tube at 100° and 200°, its stability after deodorization was found to be substantially unaffected. The metal would be expected to lower the stability of the oil appreciably in the test made at 200° (see Sample No. 11). Its failure to do so probably is attributable to the relatively small degree of agitation which was used as compared with the amount which occurs in a deodorizer. The addition of 0.3% soybean oil fatty acids, which had been distilled in the laboratory to remove soaps, lowered the stability markedly for some reason, but the combination of fatty acids and metal strips affected the oil no more than did the acids alone, at either 100° or 150°. At 200° corrosion of the metal by fatty acids and the formation of metallic soaps that dissolved in the oil apparently occurred to an appreciable extent, giving the resultant oil extremely low stability.

The tests indicate that iron in the form of soaps acts as a prooxidant. It also is evident from the data in Table III that iron in the form of ferric citrate or ferrous sulfate is probably not a prooxidant but in the form of ferric chloride it is. It is believed that small amounts of citric or sulfuric acids added to the oil after deodorization stabilize the oil by reacting with any iron present and thereby deactivating it.

TABLE III

The Effect of Iron Salts on the Oxidative Stability of the Soybean Oil

	Average peroxide value	
	Aeration	
	4 hr.	6 hr.
1. Control	29	60
2. Ferric citrate (6 p.p.m. Fe) added to soybean oil before oil was deodorized in glass	31	66
3. Ferrous sulfate (5 p.p.m. Fe) added to oil before deodorization	32	65
4. Ferric chloride (3 p.p.m. Fe) added to oil before deodorization	64	127
5. 0.01% citric acid added before deodorization	6	26
6. 0.01% citric acid added after deodorization	2	13
7. 0.0003% H_2SO_4 added before deodorization	27	60
8. 0.0015% H_2SO_4 added before deodorization	24	51
9. 0.0004% H_2SO_4 added after deodorization	....	22
10. 0.002% H_2SO_4 added after deodorization	....	27
11. 0.0004% H_2SO_4 added to No. 4 (above)	....	75

From an understanding of the reaction by which extremely small amounts of metal may dissolve in soybean oil when it is deodorized, a basis is given for speculating on methods of preventing the corrosion. Commercial deodorizers acquire an oil film after several batches of oil are processed in them, and this film, sometimes called linoxyn, serves to protect the oil from metallic contamination. Since pilot-plant deodorizers usually operate intermittently and are cleaned frequently, another method of preventing corrosion is desirable.

Table IV gives the oxidative stability of soybean oil deodorized in the presence of metal strips cleaned with an 80-20 mixture of acetone and toluene, a mixture that was used for cleaning aluminum strips in all tests with that metal. The strips had been used previously in a deodorization and had stood several weeks without being cleaned. The oil on the strips became oxidized to a tacky film. The strips were placed in a Soxhlet extractor and extracted several times with the acetone-toluene mixture, after which they were removed, dried, and used in a deodorization. After the cleaning process several of the strips when examined visually and microscopically showed no evidence of any oil film remaining on them except that a slight iridescent sheen was noticeable in some

TABLE IV

Stability of Soybean Oil Deodorized in Presence of Acetone-Toluene Washed Metal Strips

	Average peroxide value	
	Aeration	
	4 hr.	6 hr.
1. Control (glass)	29 26 24 } 26	60 60 57 } 59
2. Aluminum	30	63
3. Aluminum metallized iron	30	61
4. Aluminum-dipped iron	32	63
5. Croloy 2	29	58
6. Nickel	26	52
7. Stainless steel, type 316	27	54
8. Boiler plate	32	60
9. Hastelloy B	29	54
10. Inconel	28	54
11. Stainless steel, type 304	38	76
12. Stainless steel, type 347	35	73
13. Boiler plate (8) cleaned with hot 5% caustic solution	49	95
14. Stainless steel, type 316 (7) cleaned with hot 5% caustic solution	37	76
15. Stainless steel, type 347 (12) cleaned with hot 5% caustic solution	37	70

places. Presumably a very thin film existed however because the stability of all samples was substantially the same as that of the control. Probably other solvents than acetone-toluene would have been equally effective but none was tested.

Two factors that determine the extent to which a metal may dissolve in an oil are a) the area and b) the condition of the exposed metal surface. The importance of those factors is greater for pilot equipment than for commercial-sized equipment since the ratio of surface area to weight of oil is considerably less in a large deodorizer than in a small experimental unit. Table V summarizes the results obtained by cleaning or treating boiler plate (mild steel) in a number of different ways before placing the metal strips in the deodorizer and testing the stability of the oil. The same refined, bleached oil was used here as in the experiments with the solvent-cleaned metals. Treatment with dilute acids under conditions of surface activation increases the deleterious effect of the metal on the oil, while polishing or coating the metal with wax decreases the effect in the case of boiler plate. Since the particular wax used exhibited a slight antioxidant effect, no definite conclusion may be drawn about its use as a protective coating. With type 347 stainless steel a variety of treatments had little effect on the action of the metal towards the oil.

TABLE V

The Effect of Surface Condition of Metal on Oil Stability [a]

Treatment of metal before deodorization	Average peroxide value	
	Aeration	
	4 hr.	6 hr.
I. Mild Steel (hot-rolled)		
1. Cleaned with an acetone-toluene mixture	32	60
2. Cleaned in hot aqueous 5% NaOH solution	49	95
3. Cleaned in hot 5% NaOH solution, rinsed, and immersed in a 20% citric acid solution for one hour	67	148
4. Polished with fine grit and cleaned in hot 5% NaOH solution	34	68
5. Pickled in 3% H_2SO_4 solution for 4 hours	62	128
6. Cleaned in hot 5% NaOH solution, rinsed, and immersed in concentrated H_2SO_4 for 2 hours	49	86
7. Washed in a detergent solution and scrubbed with an abrasive soap	66	126
8. Polished with fine grit and cleaned in hot aqueous solution containing 10% NaOH and a detergent	45	90
9. Carefully wiped free of excess oil following a deodorization and used shortly thereafter	44	79
10. Cleaned with hot 5% NaOH solution, rinsed, and coated with a solid wax of the polymeric-ether-linked type	34	70
II. Mild Steel (cold-rolled)		
1. Electropolished to a smooth, shiny surface and cleaned with hot 5% NaOH solution	45	79
2. Polished with fine grit and cleaned in hot 5% NaOH solution	44	83
III. Stainless steel, type 347		
1. Control, no metal present but wax added to oil (see 10 above)	24	51
2. Cleaned in hot 5% NaOH solution	37	70
3. Cleaned in hot 5% NaOH solution, rinsed, and immersed in 20% citric acid solution for 1 hr.	37	69
4. Electropolished to a smooth bright surface and cleaned in hot 5% NaOH solution	39	71
5. Cleaned with hot 5% NaOH solution and coated with a wax (see 10 above)	34	68

[a] Metal was thoroughly rinsed with distilled water before being used in each case.

An oxidized oil film that had been washed with a suitable solvent to remove unoxidized and non-coherent oil produced a good coating on the metallic surfaces. Unless the film is cleaned with solvent, presumably some of the film is washed into the oil during a subsequent deodorization and carries some metallic soaps with it. A more convenient method of forming the oxidized film than allowing it to oxidize at room temperature is to heat it to about 150°C. for several hours, thereby accelerating oxidation. In Table VI is shown the correlation between oxidative stability and iron content for soybean oil deodorized in the presence of mild steel strips, either treated with a silicone varnish, an oil film, or cleaned with caustic. The strips coated with silicone and those coated with oil were heated in an oven for several hours to harden the silicone and to accelerate oxidation of the oil. A silicone varnish film baked on the steel according to the recommendations of the manufacturer was quite effective. It was hoped that such a film might be sufficiently resistant to caustic treatment that a deodorizer so treated might not need recoating through a long period of use even though frequently cleaned with caustic. The film did not however withstand hot 5% aqueous caustic, as was expected. It is not inferred that silicone varnish films do not meet the claims of the manufacturers with respect to alkali resistance. Failure may have been due to the type or condition of metal surface used. A fairly good correlation between stability and iron content was found, considering the possible degree of error inherent in the spectrographic method and in stability measurements.

TABLE VI

Correlation of Peroxide Values and Iron Analyses for Soybean Oil Deodorized With Film-Treated Metals Present

Type of metal strip present during deodorization	Peroxide value 6-hr. aeration	P.P.M. Fe
1. Control, no metal present	44	0.06
2. Mild steel coated with baked silicone varnish	57	0.10
3. Mild steel coated with oven-dried (150°C.) soybean oil film and washed in acetone	51	0.12
4. Mild steel cleaned in hot 5% NaOH	146	0.23

Table VII presents the results of taste panel evaluations of a soybean oil deodorized in the presence of type 316 stainless steel treated as shown. Samples were scored on a scale where a bland oil is given a top score of 10, and a very repulsive-tasting oil is given a score of 1. Unless a sample scores 6 or higher, it is not considered acceptable for consumer use. "Zero time," or fresh samples, were judged four at a time, but aged samples were judged in pairs.

TABLE VII

Organoleptic Evaluation of Soybean Oil Deodorized With Type 316 Stainless Steel

	Flavor Scores and Significant Difference[a]					
Sample pairs	204-205	204-206	204-207	205-206	205-207	206-207
Zero time[b]	7.9 + 7.1	7.9 + 8.6	7.9 + 7.5	7.1 ** 8.6	7.1 + 7.5	8.6 * 7.5
Three days' storage at 60°C	5.7 + 4.8	4.9 + 4.9	4.8 + 3.9	3.8 ** 5.8	4.5 + 5.0	5.9 + 5.0

	204	205	206	207
0 time peroxide value	0.21	0.28	0.18	0.24
Average peroxide value after 3 days' storage at 60°C	5.7	4.5	4.6	5.2
Average peroxide value after 8 hours' aeration at 98.5°C	70.3	97.0	65.3	75.5

Identification of Samples—Treatment of metal strips present during deodorization:

204—Control sample—no metal present.
205—Stainless steel cleaned with 5% aqueous caustic and rinsed with water.
206—Stainless steel from deodorization 205 stored 3 days and cleaned with refluxing acetone-toluene mixture.
207—Stainless steel polished with No. 500 grit, cleaned with 5% aqueous caustic and rinsed with water.

[a] Method of Moser, Jaeger, Cowan, and Dutton, J.A.O.C.S. *24*, 291, 1947.

[b] All four samples were tasted at one time to obtain the zero time flavor scores. + = no significant difference; * = a significant difference; and ** = a highly significant difference.

Sample 206 was deodorized with strips of type 316 stainless steel present which previously had been used in a deodorization and then stored 3 days at room temperature, followed by cleaning in a Soxhlet extractor with an 80-20 acetone-toluene mixture. The sample of oil was given the highest initial flavor score, and after 3 days' storage at 60° C. it was scored as being better by a highly significant difference than Sample 205, an oil deodorized in the presence of strips of caustic-cleaned stainless steel. It is of interest to note that polishing alone was fairly effective in preventing contamination of the oil by metal.

Summary

With mild agitation, soybean oil is not appreciably contaminated by contact with clean steel surfaces unless the fatty acid content is above 0.1% and the temperature above 150°C.

It has been shown that an oxidized oil film is an effective coating for metals used in the construction of pilot-plant deodorizers. Such a film, to be most effective, should be washed free of unoxidized, noncoherent oil with a suitable solvent. If the metal is properly coated an oxidation-stable and flavor-stable oil can be obtained from the deodorizer, providing the oil has been properly refined and bleached. The oil film is nearly the equivalent of a glass surface in its effect on oil stability, is easily formed on a metallic surface, and can be readily removed when the metal is cleaned with caustic.

A silicone varnish film baked on the metal surface is somewhat less effective than an oil film. Other methods of treating steel or stainless steel, which include coating the metal with a polymeric ether-linked wax, grit-polishing, and electropolishing, are fairly effective in reducing corrosion of the metal during deodorization.

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Alcohols From Menhaden Oil by Sodium Reduction

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IN the classical Bouveault-Blanc procedure for the reduction of esters, large excesses of both sodium and reducing alcohol are used (10). Recent modifications in the process however have lowered the quantities of sodium and alcohol required to nearly the theoretical amounts (4). These improvements have increased interest in the application of the sodium reduction method to the commercial production of fatty alcohols from natural oils (6).

The sodium reduction of saturated glycerides, coconut oil for example, is being carried out on a commercial scale. This is made possible not only by the improved efficiency in the use of sodium but also by the ease of recovery of caustic soda and glycerol. Moreover, when it is desired to preserve the ethylenic unsaturation in alcohols derived from unsaturated glycerides, as cotton seed and sperm oils, the sodium reduction process offers a possible route. Catalytic hydrogenation of an ester for the most part simultaneously saturates these olefinic linkages.

The reduction of the polyunsaturated esters occurring in highly unsaturated vegetable and fish oils to produce alcohols with one or more double bonds per molecule has never been fully investigated. A

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[1] Presented at the 23rd Fall Meeting of the American Oil Chemists' Society at Chicago, Ill., on November 1, 1949.