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2,862,943

PURIFICATION OF FATTY ACIDS

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No Drawing. Application October 7, 1954
Serial No. 461,035

7 Claims. (Cl. 260—419)

The present invention relates to a process of purifying fatty acids to produce fatty acids of improved color and improved color stability.

The fatty acids of commerce are most often prepared from inexpensive and low grade starting materials such as foots, soap stocks, brown grease and various other residues. These starting materials generally contain either a very large percentage of free fatty acids or soaps together with entrained neutral oil and other foreign materials such as phosphatides and other materials. These starting materials are usually dark colored, foul smelling materials and it is desired to produce from them light colored to water white fatty acids. Usually the starting material is subjected to a hydrolysis or splitting procedure for the purpose of liberating the free fatty acids from soaps and neutral oils. The crude fatty acids thus obtained are usually decanted from any aqueous phase presented and then subjected to various distillation techniques to purify them. They may be subjected to a simple pot distillation without any appreciable fractionation in which event the product will be composed of substantially the whole mixture of fatty acids used in the starting material. Alternatively either the crude fatty acids or the distillate from the pot distillation may be subjected to a fractional distillation to produce products varying in chain length and in degrees of unsaturation. The distilled products obtained vary in color depending upon the nature of the starting material but in general they are very materially improved in color compared with the starting material. Nonetheless these distilled materials may still be quite highly colored as compared with a water white product and for most applications it is necessary to produce a fatty acid product of quite light color.

Improvement in color may also be obtained by bleaching following distillation. However, in some instances even the bleached product is unsatisfactory in color. This is particularly true with certain extremely low grade starting materials which are available at a very low price. Some of these materials are practically impossible to purify to a very light color by presently known techniques. It is possible to distill these materials repeatedly without getting a satisfactory color. Moreover, in the repeated distillations there are mechanical losses and continued polymerization resulting in considerable pitch losses.

By means of the present invention it is possible to treat low grade stocks and thus to produce fatty acids which have material improvements in color without the necessity for repeated distillations and without the large losses which result from repeated distillations.

It is, therefore, an object of the present invention to provide a novel process of purifying fatty acids, resulting in a fatty acid of improved color as compared with the product obtainable by ordinary distillation.

The invention involves the inclusion of a small amount

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of a boric acid compound in the fatty acids at the time of distillation. By this means the color bodies and the color forming bodies either remain behind in the distillation residue or are somehow converted into compounds which do not develop color. In any event the distillate obtained is very light in color and is very color stable.

The invention is applicable to fatty acids in general regardless of the source. Generally, however, commercial processes of preparing fatty acids start with the residues referred to above and, accordingly, the invention is particularly adapted to these residues inasmuch as they are very inexpensive and inasmuch as they present the greatest color problem. These starting materials may be derived either from animal or vegetable or marine fats or oils or from such materials as the fatty acid fraction of tall oil. For this purpose the fatty acid starting material may be subjected to any preliminary purification steps such as acidulation, splitting and the like. The crude fatty acids thus obtained may be mixed with a small quantity of boric acid and then distilled. In some instances, however, this direct treatment of the crude fatty acids may result in some gelation of the distillation residues and, accordingly, it is preferred to subject the fatty acid starting materials to a pot type distillation, and then include a small quantity of boric acid in the fatty acids when they are subjected to a redistillation and fractionation. In this way gelation of the residue in the first distillation is avoided and at the same time only a small quantity of boric acid need be used in the second distillation. However, the improvement in color is definitely noted regardless of whether the material being distilled is a crude fatty acid or a fatty acid which has already been once distilled. In the production of most commercial fatty acids, however, the crude material is subjected to at least two distillations and accordingly, it is preferred to include the boric acid treatment after the first distillation.

Most if not all of the boric acid remains in the still residue and at most a trace may be present in the distillate. Distillates have been analyzed for the presence of the boric acid and in general the results indicate the presence of no boric acid within the limits of the accuracy of the analytical method. It is possible that a minute trace of boric acid may be present in the distillate.

A variety of boric acid compounds may be used including orthoboric acid, tetraboric acid, boric oxide, and salts of the above acids such as borax. In view of the acidity of the fatty acids presumably these materials are present as boric acids during the distillation. The quantity of the boric acid depends upon the nature of the feed stock. The color of the feed stock is not necessarily indicative of the quantity of boric acid which may be necessary for its treatment. In some instances very highly colored feed stocks may be purified readily to a low color by small quantities of the boric acid. In other instances feed stocks which are not too dark may require substantial quantities of boric acid for the purification treatment. It is best to determine the optimum quantity experimentally with each quantity of feed stock. Generally quantities within the range of 0.001 to 1.0% based on the weight of the feed are suitable. Often from 0.1 to 0.5% is adequate. Larger quantities than 1% may be used but in general are unnecessary and, therefore, serve no useful purpose over the smaller quantities.

Apparently the boric acid and the fatty acids undergo reaction at the elevated temperature employed for distillation. In some instances it may be desirable to prolong this heat treatment by subjecting the feed stock and the boric acid at an elevated temperature of 100–300° C.

for a period of time varying from five minutes to twenty-four hours. Tests appear to indicate that the subjection of the feed stock to these elevated temperatures for the periods indicated does not adversely affect the feed stock over the same treatment in the absence of the boric acid. Accordingly, while generally there is no need to employ periods of treating in excess of one or two hours the tests indicate that considerable leeway is permitted in the time and the temperature so that the operation may readily be fitted into most industrial processes.

Generally the heat treatment involved during the ordinary distillation is adequate to effect a pronounced improvement in color. In some instances a further improvement may be noted upon the use of the preliminary heat treatment. The exact treatment to be given to any feed stock can be readily determined in accordance with the above criterion. It appears that it is essential that the boric acid be present during the distillation step inasmuch as in certain tests in which the fatty acid and the boric acid have been heated and the boric acid removed subsequently by washing prior to distillation, the distillate obtained is not improved in color to the extent that it is when the boric acid is present during the distillation.

Example 1

The starting material employed in this instance was a green cottonseed distillate. This was a split acidulated cottonseed soap stock which had been subjected to a distillation for preliminary purification. The starting material is of particularly poor quality and the crude distillate employed as a starting material herein was even too dark to read on the Gardner scale. Various quantities of boric acid were added to this feed stock and various heat treatments were given prior to distillation, thereafter the products were distilled in a simple alembic flask at 5 mm. without any column and the results compared with similar fractions of a control which had not been subjected to any treatment. The first fraction was the first 5% of the material distilled. The second fraction was that obtained when the pot temperature was raised to 275° C. The third fraction was that obtained at a pot temperature of 300° C.

No.	Treatment	Fractions	Color (Gardner)
1	None	{First 5%----- To pot temp. 275° C-- To pot temp. 300° C--	14 8-9 11
2	.5% H ₃ BO ₃ , 2 hrs., 125° C.	{First 5%----- To pot temp. 275° C-- To pot temp. 300° C--	17 1 1-2
3	.1% H ₃ BO ₃ , 175° C., 5 min.	{First 5%----- To pot temp. 275° C-- To pot temp. 300° C--	4-5 2 4-5
4	.1% H ₃ BO ₃ , 22½ hrs., at 175° C.	{First 5%----- To pot temp. 275° C-- To pot temp. 300° C--	5+ 2 7
5	.05 H ₃ BO ₃ added to pot at 200° C. Distillation began in 12 min.	{First 5%----- To pot temp. 275° C-- To pot temp. 300° C--	5 2 5-6

¹ Product bumped over.

Example 2

In this instance crude split cottonseed oil foots were employed as the starting material, the distillation being conducted as in Example 1. The results are indicated in the following table:

No.	Treatment	Fractions	Color (Gardner)
1	None	{To pot temp. 275° C-- To pot temp. 300° C--	10 13
2	.5% H ₃ BO ₃ , 92-95° C., for 16 hrs.	{To pot temp. 275° C-- To pot temp. 285° C--	2-3 4

Example 3

In this instance crude soybean fatty acids (from soap-stock) were employed, and treated according to procedure

described in Example 1. The results are indicated in the following table:

No.	Treatment	Fractions	Color (Gardner)
1	None	{First 5%----- To pot temp. 275° C--	10 5
2	1% H ₃ BO ₃ , 125° C., 2 hrs.	{First 5%----- To pot temp. 275° C--	4 1-2
3	.1% H ₃ BO ₃ , 175° C., 5 min.	{First 5%----- To pot temp. 275° C--	6 2

Example 4

In this instance acidulated cottonseed soap stock was used as the starting material. The results are indicated in the following table:

No.	Treatment	Fractions	Color (Gardner)
1	None	{First fraction----- To pot temp. 275° C-- To pot temp. 304° C--	4 6 16
2	.5% H ₃ BO ₃ , 125° C., 2 hrs.	{First fraction----- To pot temp. 275° C-- To pot temp. 300° C--	6 <1 4

Example 5

In this instance the fatty acid fraction of tall oil was employed as the starting material. This was composed essentially of fatty acids and contained approximately 1% rosin acid. The results are indicated in the following table:

No.	Treatment	Fractions	Color (Gardner)
1	None	To pot temp. 275° C--	5-6
2	0.05% H ₃ BO ₃ , 1½ hrs., 175° C.	To pot temp. 275° C--	1+
3	.5% H ₃ BO ₃ , 175° C., 5 min.	To pot temp. 275° C--	2-3

Example 6

In this instance the green cottonseed distillate referred to in Example 1 was used as a starting material. The results are indicated in the following table:

No.	Treatment	Fractions	Color (Gardner)
1	None	{First 5%----- To pot temp. 275° C-- To pot temp. 275-300° C.	14 8-9 11
2	.1% B ₂ O ₃ , 175° C., 5 min.	{First 5%----- To pot temp. 275° C-- To pot temp. 275-300° C.	5 2- 4+
3	.155% Na ₂ B ₄ O ₇ , 175° C., 5 min.	{First 5%----- To pot temp. 275° C-- To pot temp. 275-300° C.	6-7 2+ 6-7

Example 7

In this instance the starting material was a fractionated distilled cottonseed fatty acid. The results are indicated in the following table:

No.	Treatment	Fractions	Color (Gardner)
1	None	{First fraction----- To pot temp. 260° C--	6 4+
2	.01% H ₃ BO ₃ , 125° C., 2 hrs.	{First fraction----- To pot temp. 275° C--	6 2
3	.005% H ₃ BO ₃ , 125° C., 2 hrs.	{First fraction----- To pot temp. 275° C--	6 2

Example 8

In this instance cottonseed fatty acids were being subjected to redistillation continuously in a fractionating column. The cottonseed fatty acids were fed in a stream

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to a fractionating column and an unsaturated fraction was taken off having a 5+ color. Thereafter the feed material was subjected to the injection of 0.1% boric acid into the feed line (in the form of a slurry of the boric acid in the fatty acid feed) and it was observed that the color of the unsaturated fraction coming over was reduced to 3.

Now, therefore, I claim:

1. Process of improving the color of fatty acids which comprises distilling fatty acids in the presence of boric acid said fatty acids being substantially free from neutral oil.

2. Process of improving the color of low grade fatty acids derived from an industrial residue which comprises distilling said fatty acids in the presence of an inorganic oxygen containing boric acid compound in the quantity of from 0.001 to 1.0% based on the weight of the fatty acid said fatty acids being substantially free from neutral oil.

3. Process of improving the color of fatty acids derived from soap stock which comprises distilling said soap stock fatty acids in the presence of an inorganic oxygen containing boric acid compound in the quantity of from .001 to 1.0% based on the weight of the fatty acids said fatty acids being substantially free from neutral oil.

4. Process of improving the color of crude fatty acids derived from foots which comprises distilling said acids in the presence of an inorganic oxygen containing boric acid compound in the quantity of from .001 to 1.0% based on the weight of the fatty acids said fatty acids being substantially free from neutral oil.

5. Process of improving the color of the fatty acid fraction of tall oil which comprises distilling said fatty acids in the presence of an inorganic oxygen containing

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boric acid compound in the quantity of .001 to 1.0% based on the weight of the fatty acids said fatty acids being substantially free from neutral oil.

6. Process of improving the color of fatty acids which comprises continuously feeding a stream of fatty acids stock containing an inorganic oxygen containing boric acid compound in the proportion of .001 to 1.0% based on the weight of the fatty acid into a distillation column, continuously vaporizing fatty acids in said column and continuously withdrawing fatty acid vapors and condensing them to obtain a fatty acid distillate of improved color said fatty acids being substantially free from neutral oil.

7. Process of improving the color of fatty acids which comprises continuously feeding a stream of fatty acids feed material into a fractionation column, continuously injecting a stream of a fatty acid slurry of an inorganic oxygen containing boric acid compound in such a proportion that the stream thus obtained contained a boric acid compound in the proportions of .001 to 1.0% based on the weight of the fatty acid, continuously vaporizing fatty acids in said fractionation column, continuously withdrawing fatty acid vapors therefrom and condensing said vapors to produce distilled fatty acids of improved color said fatty acids being substantially free from neutral oil.

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