Patented Sept. 15, 1953

2,652,414

UNITED STATES PATENT OFFICE

2,652,414

PREPARATION OF SATURATED FATTY ACIDS OF IMPROVED COLOR AND COLOR STABILITY

David E. Terry and Donald T. Warner, Minneapolis, Minn., assignors to General Mills, Inc., a corporation of Delaware

No Drawing. Application November 13, 1950, Serial No. 195,461

2 Claims. (Cl. 260—419)

1

The present invention relates to the preparation of saturated higher fatty acids having improved color and color stability.

For many uses to which higher saturated fatty acids are put, it is essential that the fatty acids possess a light color and that the color be stable over long periods of time. Fatty acids for such uses are commonly produced by the fractional distillation of hydrolyzed animal and vegetable oils, or from waste products resulting from the processing of such oils. As first produced, these fatty acids have a fair degree of whiteness but are not sufficiently light in color to be used for many industrial applications. It is essential, therefore, to improve the initial color of these fatty acid products.

While the fatty acids initially produced by distillation may have a fair color, this color rapidly deteriorates on storage and the product after a reasonable storage period possesses such a poor color that it can be used only for uses which command a low price.

This degradation in color is apparently caused by certain color forming bodies which are present in the fatty acids initially, but which are colorless at that point. Upon exposure to air and light these color forming bodies are converted into compounds which are colored and which thus color the entire body of fatty acid.

It is essential, therefore, to treat these fatty acid products not only to improve their initial color, but to give the fatty acid products color stability for a long period of time.

Heretofore the improvement in color of fatty acids and fatty products has been accomplished by various bleaching methods, principally by means of the use of various bleaching agents such as the bleaching earths, activated carbon, and the like. These methods have been subject to various disadvantages. The bleaching agents employed are relatively expensive and in their use they entrain considerable quantities of the fatty acids being treated thereby reducing the yield of products obtained.

Some attempts have been made to use sulfuric acid in the purification of fats and fatty products. For example, British Patent 2,356 of 1868 refers to the use of sulfuric acid as an adjunct to the ordinary fuller's earth bleaching treatment. This patent describes the use of from 3% to 5% of strong sulfuric acid as a preliminary to fuller's earth bleaching. No conditions of treatment are given, and the treatment apparently is not particularly efficacious inasmuch as it serves only as a preliminary to the usual fuller's earth treatment. Moreover, the quantity of sulfuric acid employed is excessive from a cost standpoint. In addition, the operation is clearly a two-phase system which is very inefficient. The U. S. patent to Kraft, 153,350, likewise describes the use of sulfuric acid in the treatment of stearin. In this instance, however, the sulfuric acid has been employed in the form of dilute acid and the operation is likewise an inefficient two-phase system. Moreover, the details of the process are not described in the patent. U. S. patent to Huff, 2,434,699, describes a process of overcoming the disadvantages previously encountered in the use of sulfuric acid for the treatment of fatty materials containing saturated and unsaturated fatty acid bodies. The Huff patent is concerned with a method of producing a liquid sludge in such a treatment as compared with a solid sludge ordinarily produced. The Huff process involves the use of a mixture of sulfuric and phosphoric acid and is a two-phase process operated at low temperatures.

2

It has now been discovered that it is possible to treat higher saturated fatty acids by a controlled sulfuric acid treatment such that the initial color is improved and such that the color stability of the product is very materially enhanced.

It is therefore an object of the present invention to provide a novel process of treating higher saturated fatty acids with sulfuric acid under controlled conditions to improve the initial color and to improve the color stability of the fatty acids.

It is also an object of the present invention to provide a process of producing saturated higher fatty acids which retain their characteristic initial color and high temperature color stability to a marked degree even after long storage in the flaked form in air.

This improvement of the color and color stability of saturated higher fatty acids is readily accomplished by heating the fatty acid at a high temperature with sulfuric acid, washing the resulting acid mixture with water to remove the sulfuric acid, and then distilling the fatty acid. The sulfuric acid employed is concentrated sulfuric acid such as 66° Baumé acid, or higher concentration acid. It is employed in a quantity of from 1–3% by weight of the fatty acid. The temperature of treatment is within the range of approximately 150–175° C. and the time period may be varied from 2 to 6 hours. In this temperature range and with this quantity of sulfuric acid a homogeneous single phase is obtained which is extremely efficient for the treatment of the fatty acids.

The treatment involves the admixture of the sulfuric acid with the fatty acid in a liquid con-

dition. In the case of fatty acids which are liquid at normal temperature, the sulfuric acid may be added at room temperature. In the case of the higher melting fatty acids, these may be melted and the sulfuric acid added thereto. The resulting mixture is then heated to temperatures within the range mentioned above and agitated for the time period described. Following the treatment the mixture is cooled to about 100° C. and then washed with water until the sulfuric acid has been removed. The product is then dried and subjected to distillation. The product obtained upon distillation is much improved in color, in color stability on aging, and in color stability on heating to 205° C.

In the following examples the Coleman Junior Spectrophotometer was used for the determination of the color of the samples. Color of the samples was determined at five wave lengths—400, 425, 450, 475, and 500 millimicrons. The color values are stated in "percentage transmittance" compared with distilled water values=100 at each of the wave lengths. Thus, for example, a tube of distilled water was placed in the instrument and the instrument was adjusted such that the reading was equal to 100% at that wave length. Then the distilled water tube was removed and the fatty acid sample was placed in the instrument. The instrument reading was then recorded as a measure of the fatty acid color at that wave length. The instrument readings at the five wave lengths were then averaged numerically and this average figure was designated as the color value in these examples.

In the heat stability test the color of the sample was first determined and the color value was recorded. The melted sample was then poured into a 50 ml. Nessler tube and heated for 2 hours at 205° C. The color of the heat treated sample was again determined in the Spectrophotometer and the value was expressed as the color value after heating.

The storage of the fatty acid samples for the tests after 1, 2 and 4 weeks was accomplished by flaking the fatty acid samples and then storing them in glass bottles with free exposure to the air. These samples were tested at intervals for color stability both before and after heating at 205° C.

Example 1

Four hundred parts by weight of commercial palmitic acid (90% palmitic acid) having a color value of 60 (before heating) and 13 (after heating 2 hours at 205° C.) were melted and 4 parts by weight of 66° Bé. sulfuric acid were added. The mixture was heated for 2 hours at 150° C. and then gradually cooled to about 100° C. The mixture was then washed with 4 portions of warm distilled water, the melt was dried in vacuo, and distilled without fractionation. Color stability was then tested and the results in color values are given in the following table:

	Before Treating	Treated and Freshly Distilled	Flaked Product		
			1 Week	2 Weeks	4 Weeks
As melted	60	84	81	84	83
After heating 2 hours at 205° C.	13	62	70	52	54

Control:
Freshly distilled and untreated, 77.
Freshly distilled and untreated, heated 2 hours at 205° C., 39.

Example 2

Example 1 was repeated on the same starting material and the color values are those indicated in the following table:

	Before Treating	Treated and Freshly Distilled	Flaked Product	
			1 Week	10 Weeks
As melted	60	83	84	78
After heating 2 hours at 205° C.	13	69	61	53

Example 3

Five hundred two parts of melted commercial palmitic acid (color values=60 before heating, 13 after heating) and 5 parts of concentrated sulfuric acid were heated for 4 hours at 150° C. and then cooled to about 100° C. The mixture was then washed with warm water to remove the acid, dried in vacuo, and distilled without fractionation. Color and color stability of the resultant product in terms of color values is given in the following table:

	Before Treating	Treated and Freshly Distilled	Flaked Product	
			1 Week	2 Weeks
As melted	60	82	84	82
After heating 2 hours at 205° C.	13	67	60	60

Example 4

100 parts of a commercial distilled stearic acid (90% stearic acid) were heated with 1 part by weight of concentrated sulfuric acid for 2 hours at 150° C. The melt was then washed with warm water, dried in vacuo, and distilled without fractionation. Color stability of this product was determined and the values are reported as follows in terms of color values:

	Before Treating	Treated and Freshly Distilled	Flaked Product	
			1 Week	2 Weeks
As melted	48	72	73	74
After heating 2 hours at 205° C.	1	46	48	50

Example 5

One hundred parts of a commercial grade of saturated fatty acids (55% palmitic–45% stearic acid) were heated with 3 parts by weight of concentrated sulfuric acid for 2 hours at 150° C. The melt was then washed with warm water, dried in vacuo and distilled without fractionation. The color stability of this product was determined and the values are reported below in terms of color values.

	Before Treatment	Treated and Freshly Distilled	Flaked Product (1 week)
As melted	5	55	56
After heating 2 hours at 205° C.	0	42	40

The process is applicable to higher saturated fatty acids in general. Those derived from ordinary animal and vegetable fats and oils will con-

tain from about 8 to about 22 carbon atoms. The process may be applied to isolated saturated fatty acids, to selected fractions of the saturated fatty acids derived from the fats and oils, or to the complete mixture of saturated fatty acids occurring in a fat or oil. It will be apparent that other variations may also be made in the process without departing from the spirit of the invention.

We claim as our invention:

1. Process of treating higher saturated fatty acids which comprises mixing from 1% to 3% by weight of concentrated sulfuric acid with the fatty acid, heating the mixture to a temperature within the approximate range of 150–175° C. for a period of from approximately 2 to 6 hours, thereafter washing the mixture with water to remove sulfuric acid, and distilling the residue to recover the fatty acid.

2. Process of treating higher saturated fatty acids which comprises mixing the fatty acid with approximately 1% by weight of concentrated sulfuric acid, heating the mixture of fatty acid and sulfuric acid to a temperature of about 150° C. for about 2 hours, washing the mixture with water to remove the sulfuric acid, and distilling the residual fatty acid.

DAVID E. TERRY.
DONALD T. WARNER.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
1,106,509	Hofmann	Aug. 11, 1914
2,434,699	Huff	Jan. 20, 1948

FOREIGN PATENTS

Number	Country	Date
419	Great Britain	Feb. 10, 1869
10,000	Great Britain	1843