United States Patent Office 2,812,343 Patented Nov. 5, 1957

2,812,343

METHOD OF MAKING REFINED FATTY ACIDS FROM SOAP STOCKS

Robert Palmer Cox and Robert M. Brice, Minneapolis, Minn., assignors to Archer-Daniels-Midland Company, Minneapolis, Minn., a corporation of Delaware

No Drawing. Application April 6, 1953, Serial No. 347,178

(Filed under Rule 47(a) and 35 U. S. C. 116)

8 Claims. (Cl. 260—413)

This invention relates to improved methods for the production of refined fatty acids from soap stock accumulations.

Fatty acids of commerce, as presently made, are principally obtained from refined glyceride oils. The fatty acids made from refined oil are usually produced by first hydrolyzing the oil so as to split off the glycerine from the triglyceride starting material and thereby release the fatty acids, washing to remove the glycerine, and then distilling off the fatty acids under vacuum. There results the finest grade of fatty acids. These fatty acids made from refined oil are water white when produced and retain their light color over long periods of time. They are useful in the production of light colored resins such as alkyd resins. The fatty acids obtained from refined oil are considered to be the finest available, but they have the disadvantage that they are produced from a relatively expensive starting material. A less expensive, secondary source of fatty acids is the soap stock formed during the alkali refining of oils.

Soap stock is the residue formed when an oil of animal or vegetable origin is refined with alkali, such as sodium hydroxide, sodium carbonate or the like. Among the oils commonly refined in this manner are whale and other marine oils, lard, tallow, and other animal oils and animal fats, and the vegetable oils such as linseed oil, soybean oil, corn oil, cottonseed oil, sunflower seed oil and many other oils obtained from oil-bearing seed or nut materials. All commercially available oils contain undesirable ingredients such as resins, gums, chlorophyll, phospholipins (phosphatides), mucilaginous substances, proteinaceous material, cellulosic fibres and hulls, color bodies, and various oxidation products, depending upon the age and character of the oil, and its methods of treatment and storage and other substances. The raw oil is usually filtered and then alkali refined. The alkali refining has the effect of converting the free fatty acid of the oil to alkali soaps, and, depending upon the type of alkali refining, there may also be produced some soap from the oily material itself. The oil is sometimes permitted to stand during the refining treatment to allow the soap stock to separate. Separation may be achieved by simply decanting, but there are many other modes of separation which are known in the art, all directed to attaining a better separation of the oil and soap stock. The most widely used separation method today, of course, is the continuous centrifugal separation process. The soap stock resulting from the refining of natural oils contains not only the alkali soaps of fatty acids, but also

about an equal amount of the starting oil entrained therein, together with color bodies, phosphatides, oxidation products, carbohydrates, gums, and the other minor constituents from the natural oil. Such soap stocks are usually accumulated and stored in tanks by the refiners, and in storage they undergo some oxidation and hydrolysis which produces more discoloration and undesired, complex compounds. The commercial value of the soap stock is much less than that of the refined oil from which it is produced, and such soap stock would represent a dead loss to the refiner unless suitable recovery methods were used to reclaim the valuable oils and acids of the soap stock.

One conventional method of reclaiming the oil and acids in the soap stock is to use it for the production of fatty acids. A typical prior art treatment of the soap stock might include treating the soap stock with dilute mineral acid to liberate free fatty acids, washing to free the resultant product from the mineral acid, and hydrolyzing in an autoclave with water. The resultant product, which contains the free fatty acids, some neutral oil, and residues, is then washed to separate the free glycerine from the resultant fatty acids. At this stage the fatty acids are crude and are contaminated with the color bodies, phosphatides and other nitrogenous materials, carbohydrates, sugars, waxes, etc. The crude fatty acids are then distilled off from the residues under vacuum. Because of the presence of foreign materials and colors, the distilled fatty acids, after a single distillation, are always much darker than the fatty acids made from refined oil, and this is particularly true where fractional distillation is not employed. Thus, in utilizing a still of the continuous commercial type, a certain amount of coloring material is carried over with the distilled acids and renders them considerably darker than the water white fatty acids produced from refined oil. In order to overcome this color disadvantage, in the past the soap stock has been more thoroughly washed and the crude fatty acids have been distilled several times. There is usually an improvement of color with each pass through the still. In addition, some improvement of color may be obtained by the use of bleaching clays.

Even with the added distillations and bleaching, the stock is much darker than is commercially desirable, and further, fatty acids from soap stock tend to darken at a very rapid rate upon aging. Their color instability is much more pronounced than is the case with fatty acids produced from refined oil. Other disadvantages are involved when such fatty acids from soap stock are utilized for the production of resins such as alkyd resins.

The object of the present invention is to devise a method of recovering water white fatty acids from soap stock accumulations.

A further object of the present invention is to provide a process for producing fatty acids from soap stock by which the fatty acids are characterized not only by light color, but also stable against color deterioration upon aging.

These and other objects of the invention have been achieved by devising a process for treatment of the soap stock which has been conventionally hydrolyzed, acidulated and water-washed, which includes drying the soap stock, treating it with particular mineral acids, re-

moving the sludges produced, drying, and then distilling off the high-grade fatty acids. When this process is followed, high grade fatty acids are produced with one pass through the still. The acids are either of water-white grade or very close thereto, and do not change during storage. The color stability is exceptional.

A salient feature of the invention is, therefore, the discovery that distinct color improvement may be obtained by treatment of the fatty acid material with certain concentrated strong mineral acids after hydrolysis and before distillation. Thus, the crude soap stock is given the usual preliminary treatment which consists of treating it with dilute mineral acid so as to convert the soaps of the soap stock to free fatty acids. The resultant mixture is then washed with water so as to remove the mineral acid. The mixture then contains the fatty acids from the soap of the soap stock, the neutral oil which had been occluded in the soap stock, and the color bodies, phosphatides and other unwanted materials. This mixture is passed into the autoclave, where, in the presence of moisture, and at high temperatures, the neutral oil is split into glycerine and additional free fatty acids. The material from the autoclave is then washed with water several times, and this has the effect of removing the glycerine, but the unwanted constituents, for the most part, remain behind distributed through the free fatty acids. Such unwanted materials must be removed. According to the present invention, at this stage the free fatty acids are dried by heating and mixing either in the presence of air or under vacuum and there is then added from 1 to 10% of strong concentrated mineral acid. The mineral acid is preferably added gradually, over a period of time, with constant agitation of the mass during the addition and for a considerable period of time after the addition is completed. The type of agitation does not appear to be critical. Either slow or extremely vigorous mechanical agitation appear to be equally efficacious. Simple air agitation may also be used. As the mineral acid, there may be used concentrated sulfuric acid or concentrated phosphoric acid. The 90% strength commercial sulfuric acid is desirable and where phosphoric acid is used, it should be of the usual concentrated commercial strength of about 85%. The strong mineral acid appears to have the effect of charring the color bodies and other unwanted materials in the crude fatty acids, to form a sludge, and these unwanted materials and color bodies may then be removed as a sludge with relative ease by settling and decanting, or by centrifuging. The centrifuged or decanted material is then distilled through a continuous still, one pass, and there are obtained purified fatty acids light in color and with excellent color stability. As herein set forth, the color standards are expressed in Gardner 1933 color standards.

In accordance with this invention it has been found that the fatty acids thus produced have a materially lighter color when produced and upon aging than fatty acids produced from soap stocks according to known procedures, and in addition they are suitable for the production of lightest colored alkyd resins and other uses in which light colors are requisite.

The invention is further illustrated with reference to the following exemplary data which will, however, be understood as not being a limitation on the invention:

EXAMPLE

2000 parts of commercial soap stock resulting from the alkali refining of soybean oil were preliminarily treated with about 800 parts of dilute sulfuric acid at a temperature of 35° C. for 8 hours so as to break down the soap fractions of the soap stock and split off the fatty acid. The charge was then thoroughly washed with water so as to remove the excess mineral acid and the resultant charge, without drying, was placed in an autoclave with water and a catalyst. Steam was added in the conventional manner to heat the mass and to keep it agitated.

Internal pressure was held to about 100 p. s. i. for six to ten hours. There occurred the usual splitting of the occluded whole oil fractions of the soap stock into glycerine and additional fatty acids. The resultant autoclaved split stock was washed several times with water so as to remove the resultant glycerine. The procedure to this point is standard commercial procedure. The mass was then charged into a vacuum drying still and was dried by holding for 1 hour at 93° C. at a pressure of 2–3 mm. Hg. The resultant dried stock was then cooled under vacuum to 32° C. and approximately half (sample A) of the resultant material was removed from the vessel and stored temporarily for treatment in accordance with the present invention, as hereinafter described. The remainder of the material in the vessel (sample B) was distilled without further treatment. The distillation of sample B was carried out in a batch still and the first 10% of the distillate was separately collected. The distillation was conducted at a pressure of 3 mm. of Hg and the temperature was slowly raised. At a still temperature of 250° C. distillation was discontinued and the system was cooled to 32° C. and the vacuum broken with an inert gas (CO_2).

Sample B (untreated)

Sample B, before distillation, had the following characteristics; acid value of 169.0; unsaponifiable 5.7%. The first 10% fraction, which was removed from the still at a pressure of 3 mm. of Hg and a still pot temperature of 213° C. and a top distillation temperature of 194° C., had a color when freshly distilled of 7.0 on the Gardner scale and a maximum aged color of 14.0 on the Gardner scale. It is noted parenthetically that throughout this specification the readings for maximum age color were made after one week's storage at ordinary temperatures. It will be understood that these conditions were continued for all indications of "maximum aged color." The main body of distillate, about 61.5% of the charge, had a color 194–199° C. Approximately 26.6% of the original charge remained behind in the still as pitch. The main body of distillate, about 61.5% of the storage, had a color when freshly distilled of 4.0 Gardner and a maximum aged color of 7.5 Gardner. A composite of the 10% first fraction and main body distillate portions had the following analysis: Color, 11.4 Gardner; iodine number, 129.2; saponification number, 198.9; acid number, 200. A maleic adduct was prepared according to an established test method for testing fatty acid color stability. The preparation of such maleic adduct was as follows:

There was weighed into a reaction flask 200 grams of the molten, well-mixed, sample of fatty acids to be tested. The fatty acids were then agitated at 100 R. P. M. under a flow of 400–500 ml. per minute CO_2, while heating to 100° F. At this temperature there was added 8.5 grams of maleic anhydride and the flask was connected to an air reflux condenser. Heat was applied to elevate the temperature to 450° F. in 15±2 minutes and the temperature was then held at approximately 450° F. for 30 minutes. Heat was then reduced to allow the temperature to go down to 100° F.

A sample of the reaction mixture was poured into a Gardner Holdt tube and the Gardner color read at 100° F. If the sample is cloudy at 100° F., it may be heated until the cloudiness disappears and the Gardner color then read. The resultant maleic adduct of the instant sample had a color of 15.5 Gardner when thus prepared.

An experimental alkyd resin was prepared using the following formulation:

	Grams
Fatty acids	297.6
Glycerol	150.4
Phthalic anhydride	207.0
Maleic anhydride	6.0

The resultant alkyd resin had a color of 7.0 on the Gardner scale at 50% non-volatile.

All tests hereinafter referred to involving the maleic adduct and experimental alkyd resins were made according to the aforesaid formulae and procedure.

Accordingly, sample B may be used as a basis of comparison for fatty acids produced in accordance with the improved processes of the present invention.

Sample A (treated)

Sample A was prepared and temporarily removed from the vessel as aforesaid, and was charged into a treating vat equipped with a temperature measuring thermometer, a mechanical stirrer, and a device for addition of the treating acid. The base stock was heated to 32° C., and 3% by weight of sample A of 90% commercial sulfuric acid was added gradually over a period of 30 minutes while maintaining a temperature of 32° C. The thus acidulated stock was agitated for 90 minutes at 32° C., and then was transferred to a holding vessel, where it was held for 48 hours at approximately 38° C., during which time the mixture separated into a sludge layer and a relatively clear supernatant layer. The settled layer constituted less than 1% of the total volume of the batch. The supernatant layer was removed from the sludge by careful decanting and was washed by twice boiling for 30 minutes with an equal volume of water. The water was removed by settling after each wash. The thus washed fatty acids were then dried by holding in a vacuum vessel at a temperature of 90°–95° C. at a reduced pressure in the neighborhood of 2–5 mm. of Hg. The removal of traces of water may also be accomplished by holding at 90°–95° C. and agitating by blowing air through the batch. The thus dried acid-treated fatty acids had the following analysis: Acid number, 161; unsaponifiable, 5.5%.

Sample A was then charged into a batch still. A 10% topping fraction was removed. Distillation was carried out under a vacuum of approximately 3.5 mm. of Hg. The first 10% fraction was removed at a still pot temperature of 217° C. with a top distilling temperature of 194° C. The main distillate, which amounted to about 63% of the batch, was collected in the distilling range of 194° C. to 199° C. Approximately 25.6% of the original batch remained as pitch in the bottom of the still.

The initial 10% of distillate had a color when freshly distilled of 2.9 Gardner and a maximum aged color of 3.2 Gardner, while the main fraction of distillate had a color when freshly distilled of 2.3 Gardner and a maximum age color of 2.5 Gardner. A composite of the distillate fractions had the following analysis: Color when freshly distilled, 2.3 on the Gardner scale; iodine number, 130.2; saponification number, 195.1; acid number 200.8. A maleic adduct was prepared as previously described, such adduct having a color of 6.2 Gardner. An experimental alkyd resin was also prepared from the fatty acids and it had a color of 1.0 Gardner at 50% non-volatile.

The invention is further illustrated by the following data, which for convenience of presentation is tabulated. In all of the runs shown in Table I, the soap stock was preliminarily treated with dilute mineral acid so as to liberate the fatty acids from the soap stock and then washed with water for the removal of the mineral acid. The soap stock was then treated in the autoclave with water and a catalyst at elevated temperatures to split the neutral oil content of the soap stock into fatty acids and glycerine, and the thus autoclaved material was additionally washed for the removal of the thus liberated glycerine. After removal of the glycerine the resultant fatty acids, which still contained the color bodies and unwanted materials, were then treated with strong sulfuric acid as indicated in the second column. This acidulation treatment consisted of agitating the stock for two hours at 90° F. in the presence of the indicated quantity of strong (commercial) 90% sulfuric acid. After the acid treatment the batch was in each instance agitated for two hours at 90° F. and then permitted to settle for 48 hours at 100° F. The settled layer was then drawn off and the supernatant layer washed and dried under a vacuum for removal of traces of moisture. The soap stock starting material in each instance was from the alkali refining of soybean oil.

TABLE I

Run Code No.	Percent H_2SO_4 Used by Weight of the oil charge	Distillate Fraction	Relative Gardner Color	
			Fresh	1 week
SSS-0	None	Initial 10%	7.0	14.0
		Main	4.0	7.5
SSS-0	1.0	Initial 10%	3.9	5.9
		Main	2.9	4.1
SSS-0	3.0	Initial 10%	2.9	3.2
		Main	2.3	2.5
SSS-0	5.0	Initial 10%	2.3	3.1
		Main	1.8	2.3
SSS-22	None	Initial 10%	7.6	10.8
		Main	4.4	5.1
	3.0	Initial 10%	4.0	4.2
		Main	3.3	3.2
SSS-39	None	Initial 10%	4.4	6.4
		Main	3.4	4.5
		Total Comb	3.5	4.7
	3.0	Initial 10%	2.7	2.5
		Main	2.0	1.9
SSS-49	None	Initial 10%	3.4	3.8
		Main	2.1	2.3
		Total Comb	2.5	2.7
SSS-49	3.0	Initial 10%	1.5	1.7
		Total Comb	1.5	1.7

The data in this table clearly shows the effectiveness of the concentrated acid treatment in producing light colored fatty acids. It appears that the use of 3% of 90% sulfuric acid gives optimum results, but the exact figure can be expected to vary somewhat with different batches.

In the following series of runs variations were made in the type of soap stock, that is to say, whether derived from the alkali refining of corn oil, cottonseed oil or soybean oil; and in another series of comparative runs the variation was with and without drying prior to acidulating with strong mineral acid according to the present invention. Additional variations in the amounts and type and temperature of reacting conditions when acidulated with strong mineral acid are also included. In all runs the basic conditions were the same, namely the soap stock was originally treated with dilute mineral acids to split off the fatty acids from the soap component of the soap stock and then washed so as to free the stock from mineral acids. Then the so treated stock was autoclaved in the presence of moisture to split the oil component of the soap stock into fatty acid and glycerine and the glycerine was then washed out, as previously described. In all runs the stock was then dried as previously described, except in one run where it was omitted in order to show what variations in effect is thereby produced. The type and amount of strong mineral acid used in the treatment is indicated in the table. In all runs, after treatment with the strong mineral acid for the times and at the temperatures indicated, the batch was then permitted to stand for 48 hours at approximately 100° F., whereupon it separated into two layers, and the supernatant layer containing the fatty acids was then decanted and distilled in a batch still, the first 10% topping fraction being separated from the main fraction of distilled acid. Gardner color readings were taken after one week standing.

From the following runs of Table II the following conclusions may be made: The results obtained when utilizing the refining procedure of the present invention were of substantially the same order of magnitude when utilizing soap stock from corn oil, cottonseed oil or soybean oil, and the process was equally effective on all such original starting materials. Slightly better results are obtained by drying the autoclaved stock just before addition of the strong mineral acid for treatment in accordance with the present invention. Increasing the temperature of reaction did not substantially increase the effectiveness when using either 3% of 90% strength sul-

furic acid or 1% of 85% strength phosphoric acid, nor did increasing the temperature significantly improve the results in using 3% of 85% strength phosphoric acid. Likewise, the rate of agitation does not appear to be critical. In all of the runs the color numbers on the Gardner scale were within the range of good grade commercial fatty acids.

Further room for varying the process may be found in the desludging conditions. Experimentation has revealed that for optimum results, the acid refined oil should be desludged prior to washing with water preparatory to distillation, and also that the desludged oil should be treated in some manner to remove any oil-soluble reaction products formed by the acid. In the preceding discussion, and in the example, a water wash or successive water washes were used to remove the undesired by-products of the acid refining. Other possible ways to eliminate these by-products will suggest themselves to those skilled in the art. For instance, the batch may be agitated with $CaCO_3$ or $Ca(OH)_2$ and recentrifuged prior to distillation. Alternatively, the batch may be distilled in the presence of a strong base such as KOH. The preferred treatment, however, consists of successive washes with hot water, as has been described in detail. These conclusions are based upon the following experimental data:

TABLE II

Run Code No.	Type of Stock	Variable Being Considered	Conditions With Standard Control	Experimental Conditions	Distillate Fraction	Gardner Color (1 week)
XVII-37	Corn	Type of stock	See Note 1	5%-90% H_2SO_4	Initial 10%	5.1
					Main	4.2
XVII-38	Cottonseed	do	do	do	Initial 10%	7.1
					Main	5.8
XVIII-126	Soya	Wet vs. dry stock	Vacuum dried prior to acidulating.		Initial 10%	4.7
					Main	3.6
XVIII-121	do	do		Not vacuum dried	Initial 10%	5.1
					Main	4.6
XVII-29	do	Type of mineral acid	3%-90% H_2SO_4		Initial 10%	3.2
					Main	2.5
XVII-48	do	do		3%-85% H_3PO_4	Initial 10%	5.4
					Main	3.4
XVII-50	do	do		3% H_3PO_4 and H_2SO_4 (50:50).	Initial 10%	3.8
					Main	3.2
XVII-29	do	Reaction time and temp	2 hrs. at 90° F.		Initial 10%	3.2
					Main	2.5
T-III-17	do			2 hrs.—120° F	Initial 10%	3.8
					Main	2.4
XVII-48	do	H_3PO_4 at diff. reaction time and temperature.	3% H_3PO_4 for 2 hrs. at 90° F.		Initial 10%	5.4
					Main	3.4
T-III-3	do			1% H_3PO_4 for 2 hrs. at 120° F.	Initial 10%	7.8
					Main	3.9
XVII-47	do			1% H_3PO_4 for 1 hr. at 300° F.	Initial 10%	6.9
					Main	3.6
XVII-51	do			1% H_3PO_4 for 1 hr. at 450° F.	Initial 10%	4.4
					Main	3.1
XVIII-65	do	Speed of agitation	Slow-paddle type		Initial 10%	4.0
					Main	2.8
XVIII-88				Vigorous using Eppenbach mixer.	Initial 10%	6.2
					Main	3.4

NOTE 1.—After autoclaving, removal of glycerine, and drying, the fatty acids are agitated for two hours at 90° F. with 3% of commercial 90% strength sulfuric acid and allowed to settle for 48 hours at 100° F. and decanted, washed and distilled.

TABLE III.—THE DESLUDGING AND RECOVERY OF THE ACID REFINED SOYA AUTOCLAVE SPLIT STOCKS[1]

Sample Code No.	Method of Desludging	Treatment of Desludged Oil		Distillation of Acid Refined Stocks	
		Water[2] Washed	Other Treatments	Fraction	Gardner Color (1 wk.)
XVII-29	Settled	Yes	Standard Wash	Initial 10%	3.2
				Main	2.5
XVII-36	Sludge not removed.	Yes	do	Initial 10%	6.5
				Main	4.5
T-III-17	Centrifuged[3]	No	None	Initial 10%	4.9
				Main	3.9
T-III-19	do	No	¼% Filteral at 120° F. for 1.0 hr	Initial 10%	3.8
				Main	2.6
T-III-21	do	No	Distilled with equivalent amt. KOH to neutralize H_2SO_4.	Initial 10%	5.4
				Main	3.1
T-III-28	do	No	Stirred with $CaCO_3$, then recentrifuged.	Initial 10%	4.5
				Main	2.7
T-III-32	do	No	Distilled centrifuged stock in presence of $CaCO_3$.	Initial 10%	5.0
				Main	4.0
T-III-30	do	No	Stirred with $Ca(OH)_2$, then recentrifuged.	Initial 10%	4.5
				Main	3.2
T-III-33	do	No	Distilled centrifuged stock in presence $Ca(OH)_2$.	Initial 10%	4.6
				Main	2.6
XVII-31	Base stock distilled directly			Initial 10%	9.2
				Main	5.8

[1] All experimental work based on soya stock SSS-0. After autoclaving, removal of glycerine, and drying the fatty acids are agitated for two hours at 90°F. with 3% of commercial 90% strength sulfuric acid and allowed to settle for 48 hours at 100°F. and decanted, washed and distilled.

[2] The standard water wash consisted of two equal volume H_2O boils for a total period of 1.0 hr. after which acids separated and vacuum dried prior to distillation.

[3] Samples centrifuged in laboratory centrifuge at 2800 R. P. M. for 30 min. in presence of .50% Hyflo.

One of the advantages of the present invention is that the final distillation of the fatty acids that are treated in accordance herewith may be made in a continuous production still rather than a batch still. The fatty acids, after treatment with the strong mineral acid and after settling or centrifuging may be introduced directly into the continuous still, and the fatty acids may be distilled off continuously. The color of the so-produced fatty acids is comparable with the best grades of fatty acids produced from neutral oil and re-distillation is not required. Thus, considerable increases in the distillation capacity of a plant may be effected without installation of additional equipment, by utilizing the present invention.

Fatty acids prepared according to the instant invention have a residual nitrogen content of only 0.45 to 0.90 mg. per 100 gms. It is known that by the treatment of this invention more than 70% of the nitrogen present in the autoclave split stock is removed. It is believed that the foregoing may be responsible for the very light color of the fatty acids of this invention and of the maleic adduct and alkyd resins made therefrom and for the exceptional color stability upon aging of the fatty acids hereof.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that we do not limit ourselves to the specific embodiments herein.

What is claimed is:

1. The method of producing light fatty acids having a color when freshly distilled of from about 1.5 Gardner 1933 standard to about 6 Gardner 1933 standard and a maximum aged color of about 1.7 Gardner 1933 standard to about 7 Gardner 1933 standard from soapstock which comprises treating an acidulated, washed, autoclaved and dried soapstock with 1% to 5% of a concentrated acid selected from the group consisting of phosphoric and sulfuric acids and mixtures of the two by gradually adding the acid to an agitated mass of the soapstock at about 32° C., maintaining the acidulated soapstock in an agitated condition at about 32° C. for a period of time after acid addition has been completed, allowing the mass to stand for a substantial period of time at about 38° C., removing the clear oil from the sludge which thereupon settles out, washing to remove water-soluble products of the acidulation, and then distilling off the fatty acids.

2. An improved method of recovering from relatively dry soap stock accumulation, a main body of light colored mixtures of saturated and unsaturated fatty acids having a color when freshly distilled of from about 1.5 Gardner 1933 to about 6 Gardner 1933 comprising in combination the steps of acid treating the split dried soap stock accumulation with a mineral acid selected from the group consisting of concentrated sulfuric acid, concentrated phosphoric acid and mixtures of the two acids in an amount of from about 1% to about 10% by weight of the soap stock, agitating and heating the soap stock and acid mixture to a temperature of about 90° F. to about 450° F. for a period of about 1 hours to about 2 hours for selectively reacting relative components of the mixture at specific reaction temperatures, as related to the mixture, without discoloring and destroying the end product yield of fatty acids beyond the limits defined, settling the sludge in a quiescent stage for a period of about 2 to about 48 hours at a temperature near approximately the relative temperature of the acid treatment reaction without affecting the final main body color, separating the acid formed sludge from the liquid soap stock, washing the liquid soap stock to remove water soluble products formed by the said acid treatment, and recovering a main body of high grade fatty acids having a color value within the color limits defined and with at least 70% of any nitrogen present in the original stock removed.

3. An improved process of producing from relatively dry soap stock accumulation, a main body of light colored mixtures of saturated and unsaturated fatty acids having a color when freshly distilled of from about 1.5 Gardner 1933 to about 6 Gardner 1933, comprising in combination the steps of acid treating the said relatively dry soap stock and from about 1% to about 10% mineral acid selected from the group consisting of at least about 90% concentrated sulfuric acid, at least about 85% concentrated phosphoric acid and mixtures of said mineral acids, subjecting the soap stock and mineral acid to agitation for a period of about 1 hour to about 2 hours at a temperature of from about 90° F. to about 450° F. for selectively reacting relative components of the mixture at specific reaction temperatures, as related to the mixture, without discoloring and destroying the end product yield of fatty acids, permitting the agitated batch to stand in a quiescent state for a period of from about 2 hours to about 48 hours at a temperature of approximately 100° F. to settle and separate the reaction products as a supernatant layer and sludge, separating the supernatant layer containing the fatty acids from the sludge, treating the separated supernatant layer to remove undesirable reaction products formed by the acid treatment, and distilling the treated supernatant layer to recover a main body of fatty acids within the color limits defined and with at least 70% of any nitrogen present in the starting stock removed.

4. An improved method of recovering from hydrolyzed and relatively dry split soap stock material, a main body of light colored fatty acid material having a color when freshly distilled of from about 1.5 Gardner 1933 to about 6 Gardner 1933, the combination of steps comprising acid treating the said split soap stock material with from 1% to about 10% by weight concentrated sulfuric acid for a period of at least about 1 hour and under a temperature condition of about 90° F. to about 100° F., agitating the soap stock during said acid treating thereof, permitting the acid treated soap stock to stand in a quiescent state for a period of from about 2 to about 48 hours at an approximate temperature of 100° F. to settle and separate the reaction products as a supernatant liquid layer and sludge, separating the supernatant layer containing liquid fatty acids from the sludge, treating the separated liquid layer to remove undesired by-products of the acid treatment, and distilling the treated separated liquid layer to recover a main body of fatty acids falling within the color limits defined.

5. The process of recovering high grade fatty acids having a color when freshly distilled of from about 1.5 Gardner 1933 to about 6 Gardner 1933 and a maximum aged color of about 1.7 Gardner 1933 to about 7 Gardner 1933 from acidulated, washed, autoclaved and dried split soap stock which comprises acidulating the split soap stock with an acid selected from the group consisting of concentrated sulfuric acid, concentrated phosphoric acid, and mixtures of these acids, agitating, heating and settling the split stock at a temperature of about 90° F. to about 100° F., separating the sludge formed by the acidulation from the resulting clear liquid, washing this clear liquid to remove the water-soluble reaction products from the thus treated soap stock, and then recovering the fatty acids.

6. The improvement in recovering fatty acids having a color when freshly distilled of from about 1.5 Gardner 1933 to about 6 Gardner 1933 and a maximum aged color of about 1.7 Gardner 1933 to about 7 Gardner 1933 from an acidulated, washed, autoclaved split soap stock which comprises acidulating the split soap stock with about 3% by weight of at least about 90% concentrated sulfuric acid, treating the split soap stock not substantially in excess of 100° F. for a period of from about 2 to 48 hours, separating the acid sludge from the soap stock, washing the soap stock with hot water to remove water-soluble acid reaction products, and then distilling the treated soap stock to recover the fatty acids contained therein.

7. The improved method of recovering fatty acids hav-

ing a color when freshly distilled of from about 1.5 Gardner 1933 to about 6 Gardner 1933 and a maximum aged color of about 1.7 Gardner 1933 to about 7 Gardner 1933 from an acidulated, washed, autoclaved and dried split soap stock which comprises acidulating the dried split soap stock with about 3% by weight of the soap stock of about 90% or more concentrated sulfuric acid, agitating the acidulated soap stock to obtain thorough mixing and reaction at a temperature of about 90° to about 100° F., settling out the sludge formed by the acidulation, decanting the clear liquid from the sludge, repeatedly washing the liquid with water to remove water soluble products formed by the acidulation, and then distilling off the fatty acids.

8. The method of recovering fatty acids having a color when freshly distilled of from about 1.5 Gardner 1933 to about 6 Gardner 1933 and a maximum aged color of about 1.7 Gardner 1933 to about 7 Gardner 1933 from acidulated, washed, hydrolyzed and dried split soap stock which comprises acidulating the dried split soap stock with about 3% by weight of a concentrated acid selected from the group consisting of sulfuric acid, phosphoric acid, and

mixtures of the two, in concentrations on the order of 90% and 85% respectively, agitating the acidulated soap stock while the acid is being added and for a period thereafter while maintaining the soap stock at about 32° C., separating the sludge formed thereby from the treated soap stock, washing to remove water-soluble products formed by the acid treatment from the soap stock, and distilling off the fatty acids.

References Cited in the file of this patent

UNITED STATES PATENTS

1,964,875	Freiburg	July 3, 1934
2,434,699	Huff	Jan. 20, 1948
2,652,414	Terry et al.	Sept. 15, 1953
2,694,082	Palmquist	Nov. 9, 1954

OTHER REFERENCES

Bailey: Cottonseed, 1948, pages 815–821.

Andersen: Refining of Oil and Fats, 1953, pages 70 and 71.