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HYDROXYLAMINE STABILIZED FATTY MATERIALS

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The present invention relates to the stabilization of fatty materials including fats, fatty oils, fatty esters, fatty acids, fatty acid salts and the like, and more particularly to stabilization against oxidation and discoloration in the soaps that are prepared therefrom.

Fatty materials are notably subject to rancidity and exhibiting the accompanying objections of developing disagreeable odors and taste. This rancidity is due to an oxidation reaction and there have been numerous attempts in the past to control or retard this reaction by the addition of various substances to the fatty materials, and these attempts have met with varying degrees of success. So far as is known, however, none of these stabilizers have exhibited a protective action as measured by an improvement in the color of the soap prepared therefrom.

Fatty materials in their pure state are colorless. However, both animal and vegetable fats, occurring naturally, contain various bodies which impart a color to the fat or oil. This color increases in intensity upon storage and also when the oil is processed in connection with which high temperatures and other harmful conditions are employed. Darkening of oils and fats have been found to occur under these conditions even after they have been refined and bleached according to standard procedure.

The darkening of fatty materials above referred to is a particularly important problem in the field of soap preparation when the soap maker is interested in preparing a high grade white toilet soap. An off color is produced in the final product because of the color change in the fatty material from which it is prepared during storage and during processing, in which is involved such operations as filtering, bleaching, refining and in general prolonged exposure to high temperatures. This darkening is especially noted in the soap colors obtained from over-treated fats. The darkening that is apparent in the final soap may not be apparent in the fatty base so that conventional color readings on the parent fat or oil are of no value in estimating the color of the soap produced. The color of the soap in units of yellow and red is the important factor.

It is an object of the present invention to provide a process by means of which a fatty material may be stabilized against the development of color and rancidity.

It is another object of the present invention to provide a product, the fatty content of which is stabilized against development of objectionable odors, taste or color.

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It is still another object of the present invention to provide a process by means of which a soap derived from fatty material may be stabilized against the development of color and eventual rancidity.

Another object of the invention is to provide an improved process of making soap involving the incorporation of an inhibitor into the soap ingredients and then converting the treated ingredients into soap.

Another object of the present invention is to provide a soap product which is stabilized against developing objectionable odors or color.

Another object of the present invention is to provide a process by means of which fatty materials are stabilized against darkening, as indicated by the soap produced therefrom, when stored for long periods of time or when processed at elevated temperatures.

Another and more specific object of this invention is to provide a refined or unrefined tallow stabilized against development of an off-color in soap subsequently prepared therefrom.

A still further object of the invention is to provide a process for stabilizing refined or unrefined tallow to be used in soap making against darkening in color in the soap.

Other and further objects of this invention will be apparent from the following detailed description.

It has been found that the substance hydroxylamine, NH_2OH , exerts a pronounced stabilizing effect upon fatty materials particularly as measured by the color of the soaps prepared therefrom. This material when added to either a refined or unrefined fatty material in only small amounts shows a very effective preserving action both as an anti-oxidant to retard rancidity and as an agent to prevent or retard darkening during storage or during processing at elevated temperatures and that such preserving effect may be observed by studying the color of the resultant soap.

As seen in the examples set forth hereinafter, proportions of hydroxylamine, calculated in terms of the free base, in the neighborhood of .5% have proven very effective in exerting a stabilizing effect on the fatty materials. A range of .1 to 2% is, however, contemplated by this invention.

The following two examples will serve to illustrate the advantage derived from adding small amounts of hydroxylamine to refined tallows, which are subsequently converted into soap by a conventional process. In the example the data on washed saponification colors relate to the col-

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ors determined on the soap prepared from the fat under test. The soap was prepared by saponification in a kettle, settling, salting out the soap, washing, drying, milling and conversion of the soap into conventional forms, such as bars, chips, flakes, powders, etc.

Example 1

A fresh fancy tallow, free fatty acids 4.0%, was used. 1,000 grams were refined with 49.2 ml. 20° Bé. caustic which would leave 5% excess NaOH in the aqueous phase. The caustic was added to the tallow at 51–54° C. with rapid stirring for 2–3 minutes; the temperature was then raised to 61–64° C. and slow stirring continued for 5–10 minutes. The foots were allowed to settle for two hours, keeping the mixture in a water bath at 65–70° C. The clear oil was then poured off through a 100 mesh stainless steel screen.

The refined tallow was bleached with 6% Bennett and Clark earth and 0.5% Norit F at 100–105° C. for 15 minutes and filtered by suction through a thin pad of Dicalite.

Washed saponification color

Crude tallow ----- 30Y-3.3R
Refined tallow ----- 20Y-3.4R
Refined and bleached tallow ----- 4.0Y-1.1R

One thousand grams of the same fancy tallow were also refined with the following mixture: 49.2 ml. 20° Bé. caustic, 10 grams hydroxylamine hydrochloride and 33.8 ml. of the 20° Bé. caustic to neutralize the hydrochloride. The hydroxylamine hydrochloride was dissolved in the caustic in an ice-bath with constant stirring. All other details of the refining were identical with the above refining. Similarly, the NH₂OH-refined tallow was bleached with 6% B. C. earth and 0.5% Norit F.

Washed saponification color

NH₂OH-refined tallow ----- 30Y-2.4R
NH₂OH-refined and bleached tallow -- 4.0Y-1.1R

The two samples of refined and bleached tallow were now stored in glass jars in a steam chest at approximately 75° C. for ten days.

After storage at 75° C.	Washed Saponification Color
Refined and bleached tallow -----	10Y-2.7R
NH ₂ OH-refined and bleached tallow -----	5.0Y-1.1R

The above samples of refined and bleached tallow were now stored at room temperature, 25–30° C. for 23 days.

After storage at room temperature	Washed Saponification Color
Refined and bleached tallow -----	35Y-5.3R
NH ₂ OH-refined and bleached tallow -----	8.0Y-1.3R

Example 2

A prime tallow, free fatty acids 2.9% was used. 2,000 grams of this tallow were refined with 71.4 ml. of 20° Bé. caustic, leaving 5% excess NaOH in the aqueous phase. The refining procedure was identical with that in Example 1. The refined tallow was aerated for 40 hours at 70–80° C. by bubbling clean air through the sample.

Another 2,000 grams of the prime tallow were refined with a mixture of 57.5 ml. 20° Bé. caustic (enough to neutralize the free fatty acids and leave an excess of 2.5% NaOH in the aqueous

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phase), 20 grams hydroxylamine acid sulfate and 77 ml. of 20° Bé. caustic to neutralize the acid sulfate. As in Example 1, the hydroxylamine salt was dissolved in the caustic in an ice-bath and the refining carried out as described above. The NH₂OH-refined tallow was aerated for 40 hours at 70–80° C.

	Peroxide Value	Washed Saponification Color
Refined tallow -----	6.0	50Y-7.4R
Aerated 40 hours -----	79.0	75Y-10.2R
NH ₂ OH-refined tallow -----	6.0	100Y-8.5R
Aerated 40 hours -----	6.5	75Y-7.9R

The following example will illustrate that the present invention is also applicable to an unrefined or crude fat.

Example 3

A choice tallow, free fatty acids 1.25%, was used. A portion of the tallow was bleached with 2.5% of an activated earth and tested for soap color. Another portion of the crude tallow was stored at 45° C. in the presence of iron. A third portion of the crude tallow was treated with a neutralized solution of hydroxylamine prepared as follows:

2.88 pts. anhydrous sodium carbonate (95%) were dissolved in 20 pts. water and added to 4.0 pts. hydroxylamine acid sulfate (84.3%) with constant stirring.

The treatment consisted of adding the freshly prepared hydroxylamine solution to 400 pts. of the crude tallow at 50° C. with rapid stirring. Stirring was continued for 15 minutes. The mixture was allowed to stand overnight at room temperature, then warmed up to approximately 65° C. and the fat layer decanted from the water layer. The treated fat was aged at 45° C. under the same conditions as the untreated sample. Both samples were aged for 7 days and then bleached with 2.5% of the activated earth.

Washed saponification color

Freshly bleached tallow ----- 7Y-1.1R
Untreated tallow, aged 7 days and bleached ----- 20Y-3.2R
NH₂OH treated tallow, aged 7 days and bleached ----- 7Y-1.5R

The above examples indicate that hydroxylamine so stabilizes the soap color of a tallow that a light colored tallow if treated immediately could be processed without danger of darkening or could be stored for long periods of time.

Also, Example 2, by means of reference to the peroxide values, clearly shows that oxidation of fat which results in rancidity is greatly inhibited. Upon aeration, the sample of refined tallow containing the hydroxylamine showed an increase of only .5 in the peroxide value while an identical sample without the addition of this substance exhibited an increase in peroxide number of 73.

Other methods of incorporating the hydroxylamine may be used, such as stirring a mixture of the dry hydroxylamine salt and sodium carbonate with the refined tallow and then filtering or adding the mixture of the dry hydroxylamine salt and sodium carbonate to a refining operation immediately after the caustic has been added.

Still another method is to dissolve pure hydroxylamine in the caustic before refining.

The present invention has been illustrated

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specifically by experiments dealing with the treating of tallow to be used in the manufacture of soap. It is to be understood, however, that the invention has equal application in the stabilization of all types of fatty materials.

While I have shown the formation of the hydroxylamine in situ, it is to be understood that hydroxylamine may be first formed and then added to the fat before or during saponification.

It is understood that hydroxamic acids derived from hydroxylamine also have anti-oxidant properties, but it has been shown that these acids are not produced in the practice of this invention. It is also known that whereas hydroxamic acids stabilize fats they have no tendency to stabilize colors of soaps produced therefrom.

The term "fatty material" as used in the claims is intended to mean fats, fatty oils, higher fatty acids, higher fatty acid esters, and higher fatty acid salts.

Obviously, many modifications and variations of the invention hereinbefore set forth may be made without departing from the spirit and scope thereof, and, therefore, only such limitations should be imposed as are indicated in the appended claims.

Having thus described my invention, I claim:

1. The process of stabilizing a fatty material comprising incorporating therein a small amount of hydroxylamine.

2. A stabilized composition of matter comprising a fatty material and a small amount of hydroxylamine.

3. A stabilized tallow comprising in addition to said tallow a small amount of hydroxylamine.

4. The process of stabilizing tallow comprising adding to said tallow a small amount of hydroxylamine.

5. The process of stabilizing tallow against darkening comprising adding a small amount of hydroxylamine to said tallow in the proportion of .1 to 2% based on the weight of said tallow.

6. A tallow stabilized against darkening comprising in addition to said tallow .1 to 2% of hydroxylamine.

7. The process of stabilizing tallow against darkening comprising refining and bleaching said

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tallow and adding to said refined and bleached tallow a small amount of hydroxylamine.

8. The process of stabilizing tallow against darkening comprising refining and bleaching said tallow in the presence of a small amount of hydroxylamine.

9. A stabilized composition of matter comprising a refined and bleached tallow and a small amount of hydroxylamine.

10. The process of stabilizing fats, normally tending to deteriorate and form color when converted into soap, which comprises incorporating into said fat prior to conversion into soap a hydroxylamine in sufficient amount to substantially retard such deterioration and color formation.

11. In the process for manufacture of soap wherein a fat is converted into soap, the step of incorporating into the fat a small amount of a hydroxylamine.

12. A soap product stabilized against color and odor formation, which comprises a fatty acid soap and a small amount of hydroxylamine.

13. A soap product, comprising a tallow soap, said soap normally tending to deteriorate and form color on storage, and hydroxylamine in sufficient amount to substantially retard such deterioration and color formation.

14. In the manufacture of soap, the improvement which comprises saponifying a fat in the presence of a hydroxylamine whereby hydroxylamine is formed in situ during saponification, said fat normally tending to form a soap which is unstable in color and said hydroxylamine being sufficient to substantially stabilize said color.

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