

UNITED STATES PATENT OFFICE

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PROCESS OF BLEACHING FATS AND OILS
AND MAKING SOAP THEREFROMLloyd F. Henderson, Reading, and Louis H. Libby,
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This invention relates to the bleaching of oils and fats and to the saponification of the same to yield soaps of light color.

Soaps are commonly made from glyceride oils and fats, and except when the oil or fat available is very high grade stock, it is often necessary to make the soap from stock which is dark in color. When soap is made from such dark colored stock the coloring matter is carried into the finished soap and renders it dark in color. This is undesirable because white soap or soap that is light in color has more appeal to the user and is freer from impurities. For this reason the art has developed numerous processes, the object of which is to produce lighter soaps from darker stocks.

One such process involves hydrolyzing or splitting glyceride oils and fats, and distilling the fatty acids to separate them from the coloring matter; the soap is then made from the fatty acids. This process requires extensive equipment, high temperatures and pressures for the splitting, high vacuum for distillation, and other requirements which are uneconomical, except in the case of very extensive installations. The odor from pitch and decomposition products resulting from the high temperatures used is often carried into the soap.

Another proposal has been to convert the glycerides into the corresponding methyl or ethyl esters of the fatty acids and to distill these; the soap is made from such esters with the liberation of the methyl or ethyl alcohol. Such a process is open to substantially all of the objections mentioned in connection with fatty acid distillation and to the additional problem created by the fire hazard incident to the presence of the alcohol.

A more common method has been to bleach the oil or fat with an earth bleach, such as fuller's earth, and especially an activated bleaching earth. The use of such an earth exerts a strong bleaching action, but the amount of bleaching that can be accomplished with a reasonable amount of earth is often not as great as that desired, particularly when it is necessary to utilize very dark stocks for the purpose of making soaps of especially light color. Generally it is not regarded as feasible to use more than 5% to 6% of earth in such a bleach because of the cost of the earth and the loss of stock that is absorbed in the earth along with the coloring matter. If this amount of earth does not obtain the desired bleaching action, it is generally regarded as essential for economic rea-

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sons, to use the stock for dark soap or some other purpose.

A wide variety of chemicals have been proposed as bleaching agents for oils and fats, including various oxidizing agents, reducing agents, halogen compounds, acids, etc. In general, none of these have obtained promising results nor have they been widely accepted commercially; the industry has relied primarily on earth bleach as the most satisfactory. If the desired amount of bleaching could not be accomplished with 6% of earth bleaching, the general practice has been to seek a better grade of stock as a raw material.

Another difficulty in connection with bleaching oils and fats is the tendency of the color to revert after the oil or fat is made into soap. Thus some bleaching processes produce an oil or fat of reasonably light color, but upon making soap from such bleached stock and acidifying the soap to form fatty acids, it will be found that the fatty acids are darker than the oil from which the soap is made. Probably latent coloring materials develop during the soap making. Many bleaching processes, therefore, which are suitable for making light oils and fats, per se, are not suitable when the oil or fat is to be made into soap.

With the desire in the art for lighter colored soaps, even in the case of the less expensive laundry soaps, and with the dwindling supplies of high-grade stocks for making light colored soaps, it has become increasingly important to utilize dark colored stocks for the purpose of making light soaps.

We have discovered that if the oil or fat from which the soap is to be made is given a preliminary treatment with both an acidic phosphoric compound and an oxygen liberating compound, preferably of the per type, following which the oil or fat is given a conventional earth bleach, is then made into soap, and the soap bleached with a reducing agent, it is possible to effect a remarkable bleaching action and to produce soaps which are of unexpectedly light color, considering the dark stock used as the raw material.

We appreciate that it has been proposed to use phosphoric acid in connection with the bleaching, and that oxidizing compounds have also been proposed as bleaching agents. It has not been proposed, insofar as we are aware, to use the combined action of an oxygen liberating compound and a phosphoric acidic compound in bleaching oil or fat, and particularly in combination with the subsequent treatment of the soap made therefrom by a reducing bleach. Our invention has revealed the unobvious fact that ap-

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parently the combined use of both an oxygen liberating compound and a phosphoric acidic compound, not only accomplishes an unexpected bleaching action on the oil or fat, as compared with the action of these materials separately, but leaves the oil or fat in a unique condition (although the phosphoric and oxygen liberating compounds are completely removed) such that soap made from the treated oil or fat is much more susceptible to the bleaching action of reducing agents added to soap than is the case with oils or fats treated by other processes. Thus the bleaching process of our invention is one in which there is a minimum, if any, color reversion when the stock is made into soap.

Our invention is to be distinguished from the known proposal to use such oxygen liberating compounds with sulfuric acid with or without a phosphate and our process is not the equivalent of such a proposal.

Our invention assumes particular importance in the treatment of very dark colored fats. Such dark colored greases have a high free fatty acid content as an inherent characteristic thereof. It is more difficult to bleach stock high in free fatty acids than stock which is more neutral. However, our process may be applied even to lighter colored stocks to obtain an improved result when extremely light or pure white soaps are wanted.

The manner in which the invention may be practiced will appear more fully from the description hereinafter of a preferred embodiment thereof.

The oil or fat to be bleached, in accordance with this illustrative example, is agitated with a small quantity of a commercial grade of phosphoric acid and a small quantity of hydrogen peroxide solution. While agitating the mixture of the fat and these agents, the temperature is maintained slightly elevated and the mixing continued at this temperature for the desired time. Following this the stock is settled and the sludge withdrawn. The stock is then washed with hot water (1 to 10%) to remove the phosphoric acid and the coloring matter and material that has coagulated, together with any unreacted hydrogen peroxide. The wash water and the water-soluble materials settle as a lower layer and can be withdrawn. Further washing with 5 to 50% hot water may be done so as to remove phosphoric acid and compounds thereof. Careful neutralization with alkali may be employed in connection with water washing at any stage.

The washed oil may be dried in accordance with the usual processes and is then bleached with fuller's earth or bleaching earth in the conventional manner. This involves mixing the stock with about 2% to 6% of earth, and agitating the mixture at an elevated temperature. After this the stock is filtered to remove the earth. This operation and variations thereof are well-known in the industry.

The bleached stock is next transferred to a soap kettle or a continuous soap making apparatus where it is processed in the customary manner for making a boiled soap, i. e., is saponified with caustic in the requisite amount and washed to remove glycerine, following which the soap is bleached with a small amount (0.01 to 5%) of a reducing agent, in accordance with the known technique, such as with sodium hydrosulfite or sodium formaldehyde sulfoxylate. The soap is then finished in the usual manner and converted into bars, cakes, flakes, powders or other forms. The soap may be admixed with builders and may

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have preservatives, coloring matter, perfume or any ingredient added thereto that are conventional in soap making processes. The finished products are of remarkable lightness in color and as far as is known, no other process, except distilling fatty acids and making soap therefrom, has resulted in the production of such light colored soap from low grade dark fats.

The acidic phosphoric compound to be used may be any phosphorus compound that is distinctly acid in nature, preferably any phosphoric acid, a salt thereof having an acid reaction, or a compound forming such an acid or salt under the reaction conditions. Phosphoric acid is commercially available and relatively inexpensive. Commercial orthophosphoric acid is available in concentrations of from 70 to 85% and these may be used conveniently. The water and impurities which make up the balance of such commercial acids are not detrimental to the process. Any other acidic phosphoric compound may be used, such as pyrophosphoric acid, and acidic salts of phosphoric acids, such as potassium or sodium dihydrogen phosphate or disodium dihydrogen pyrophosphate, and acidic pentavalent and organic phosphorus compounds.

The oxygen liberating compound may be, for example, a "per" type compound, such as hydrogen peroxide; an alkali metal peroxide, such as sodium peroxide; perborates such as sodium perborates; per carbonates, such as sodium per carbonate; and per sulfates, such as sodium persulfate. Hydrogen peroxide is commercially available in a 30% solution and this may be used with facility. The water and remaining impurities are not detrimental to the process. Other oxygen liberating compounds include chlorites, and hypochlorites, such as sodium chlorite and sodium hypochlorite.

An oxygen liberating compound is well known to be one of a class of compounds which contains more oxygen than a more stable member of the class under certain conditions; such compounds which liberate oxygen under the acidic and elevated temperature conditions of the reaction may advantageously be used in accordance with the invention. The oxygen is well known to be in an active state, sometimes known as "nascent" and is to be distinguished from ordinary oxygen (O_2) and air.

The amounts of the phosphoric compounds and the per compound may be within the range of 0.01% to 2% of the oil or fat to be treated. There must be present in a substantial amount, i. e., more than a mere trace or impurity. The same amount of each need not be used. In the case of phosphoric acid the amount may be 0.1% or 0.25% to 2.0%. In the case of hydrogen peroxide the amount may be 0.25% or 0.5% to 1.0% to 2.0% (of a 30% solution). In the case of chlorites or hypochlorites, smaller amounts can be used, such as 0.05% to 1.0%. If the oxygen liberating compound is of an alkaline type, such as sodium percarbonate, or sodium perborate, the amount of the acidic phosphorus compound should be sufficient to place the mixture on the acid side during the reaction. The use of amounts more than 2% is not harmful but the increase in the improvement is not sufficient to justify amounts larger than this in a commercial operation.

The bleaching is carried out at a temperature sufficiently high so that the oil or fat is liquid. The temperature should not be above the charring temperature of the fat in the presence of the acid. A temperature of 80° to 90° C. probably repre-

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sents the upper limit. However, the optimum temperature is about 60° to 70° C. and in view of the desirable results that may be obtained at this temperature, there is no advantage of supplying additional heat for the higher temperatures. Because of the heat requirements, the minimum temperature to accomplish the desired result is recommended.

In the preferred embodiment, both the acidic phosphoric compound and the oxygen liberating compound are added to the stock and mixed with it simultaneously because this is the simplest method of handling. Substantially the same results can be obtained, nevertheless, by treating first with the acidic phosphoric compound and second with the oxygen liberating compound or vice versa. However, there is no point in going through two separate treating operations with the two chemicals, when the same result can be accomplished in a single operation using them both.

The treating time is preferably about $\frac{1}{2}$ to $1\frac{1}{2}$ hours. The agitation with the bleaching agents should be continued as long as any improvement in color is manifest. There is no point in continuing the treatment beyond this stage and the end point can be readily ascertained by the operator of the process.

While the illustrative example has been described as a batch process, it is obvious that it may be applied to a continuous process when somewhat shorter treating times probably can be used because of better agitating conditions.

As indicative of the results that may be obtained in accordance with the invention, a sample of crude very dark tallow was bleached using the process of the invention described herein, and compared with the same stock bleached with earth in the conventional manner. The color of the bleached tallows were determined, using a Lovibond colorimeter on a 1" column; the red readings are designated R, and the yellow readings are designated Y.

Table I

[Tallow]

Method of Bleaching	Color	
In accordance with invention.....	13.6 R	75 Y
Conventional Earth bleach.....	20.0 R	75 Y

Soap was made from the above two samples in accordance with the manner described previously and the soap was acidulated to liberate the free fatty acids therefrom. The color of these fatty acids was similarly measured on a 1" column and the results are as follows:

Table II

Fatty Acids from Soap

Method of Bleaching	Color	
In accordance with invention.....	5.9 R	75 Y
Conventional Earth bleach.....	22.0 R	75 Y

It will be seen that the fatty acids from the soap which was made from stock bleached in accordance with the invention is much lighter than the bleached tallow from which the soap was made. On the other hand, the fatty acids from soap made from conventional earth bleached tallow were darker than the tallow from which the soap was made. This demonstrates

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the susceptibility of the stock treated in accordance with the invention to the production of light colored soaps, and the absence of color reversion which is characteristic of stock bleached by other processes.

The combined action of phosphoric acid and hydrogen peroxide, as compared with these materials treated separately, is indicated by the following: a dark tallow, containing 27.7% free fatty acids and having a color too dark to read, was treated at 65° C. with 1 and 2% respectively of 85% phosphoric acid, 1 and 2% respectively of 30% hydrogen peroxide; and 1% each of 85% phosphoric acid and 30% hydrogen peroxide. Each of these was followed by a conventional bleach with 6% earth, and compared with a sample bleached only with the same amount of earth in the conventional manner. The color of the bleached stock, when using a 1" column on a Lovibond colorimeter, is set forth in the following table:

Table III

[Tallow too dark to read—27.7% F. F. A.]

Sample	Pretreatment	Earth Bleach	Color	
		Per cent		
1.....	1% of 85% H_3PO_4	6	41.0 R	75 Y
2.....	2% of 85% H_3PO_4	6	39.6 R	75 Y
3.....	1% of 30% H_2O_2	6	41.0 R	75 Y
4.....	2% of 30% H_2O_2	6	32.0 R	75 Y
5.....	1% of 85% H_3PO_4 +1% of 30% H_2O_2	6	18.0 R	75 Y
6.....	None.....	6	87.5 R	75 Y

When the same tallow was bleached with 1% of 30% H_2O_2 and 1% of $H_3P_2O_7$ at 40° C., it had a color of 19 R and 50 Y, and when bleached similarly at 50° C., it had a color of 23.5 R and 50 Y. When the same tallow was bleached with 1% of H_2O_2 and 1% of KH_2PO_4 at 65° C., it had a color of 28.6 R and 75 Y.

As indicative of the results that may be obtained in accordance with the invention in the treatment of an initially lighter colored stock, the process was applied to the treatment of a tallow having an original color of 13.5 red and 50 yellow when measured on a 1" column, using a Lovibond colorimeter. The results are set forth in the following table:

Table IV

Tallow having 13.5 R and 50 Y (Original Color) (1" Col.)

Pretreatment	Earth Bleach	Color (1" Col.)	
	Per cent		
1% of 85% H_3PO_4	4	7.2 R	50 Y
1% of 30% H_2O_2	4	4.1 R	50 Y
1% of 85% H_3PO_4 +1% of 30% H_2O_2	4	3.5 R	35 Y
None.....	4	8.0 R	50 Y

The improvement of the stock treated in accordance with the invention, as compared with the conventional earth bleach, is readily apparent, the difference between 3.5 and 8 readings on the red scale being a tremendous difference in color. This is not possible with the use of either of these materials alone and this would be true even though larger amounts than 1% were used separately.

As further indicative of the tendency of soap made from conventionally bleached stock to revert in color, and the improvement of the soap when the stock is bleached in accordance with

the invention, the following results were obtained in connection with another illustrative stock:

Table V

Treatment	Color of Tallow (1" Col.)		Color of Fatty Acids from Hydrosulfite Bleached Soap (1" Col.)	
	R	Y	R	Y
In accordance with invention.	6.5 R	75 Y	5.0 R	75 Y
Conventional Earth Bleach	6.9 R	75 Y	7.6 R	75 Y

It will be understood that further improvements in color in stocks which have already been reduced in color are difficult to achieve, and therefore an improvement of a reading from 7.6 R to 5.0 R on the Lovibond scale is a great color re-

From the above results it will be seen that the fatty acids from the soap are lighter in color than the stock from which the soap was made, indicating the susceptibility of the stock to absence of color reversion when the soap is bleached. The improvement in the yellow color also is notable. The color of the fatty acids from the soap made in accordance with the invention, as compared with the other processes included in the table, is particularly significant.

Other examples, using other oxygen liberating compounds, and the results obtained, both in the color of the bleached fat and the color of the fatty acids of soap made therefrom in the same way as indicated heretofore, are shown in the following tables:

Table VII

[Very dark tallow.]

Acidic Compound	Oxygen Liberating Compound	Conditions		Earth Bleach	Color (1" Col.)			
		Temp.	Time		Fat		Soap Fatty Acids	
		°C.	Hours		R	Y	R	Y
None	None			Per cent	30.0 R	75 Y		
1% H ₃ PO ₄	1% H ₂ O ₂	65	1	6	14.0 R	75 Y	6.5 R	50 Y
Do	0.1% NaClO ₂ *	65	1	6	12.5 R	75 Y	6.4 R	50 Y

* Dissolved in 5% H₂O based on weight of tallow.

Table VIII

[Very dark tallow.]

Acidic Compound	Oxygen Liberating Compound	Conditions		Earth Bleach	Color (1" Col.)			
		Temp.	Time		Fat		Soap Fatty Acids	
		°C.	Hours		R	Y	R	Y
None	None			10	59.9 R	75 Y		
1% H ₃ PO ₄	1% H ₂ O ₂	65	1	6	21.0 R	75 Y	12.0 R	75 Y
Do	2% NaOCl*	65	1	6	20.0 R	75 Y	10.2 R	75 Y

* 2% of a 5% solution of NaOCl.

duction and represents a significant improvement.

As illustrative of the results that can be obtained in applying the invention to a medium grade stock, this stock was treated in accordance with the invention, utilizing 1% each of 30% hydrogen peroxide and 85% phosphoric acid, the treating being carried on at 65° C. for 1 hour with mechanical agitation. The stock was then washed, dried at 105° C. at atmospheric pressure and bleached for 15 minutes at 105° C. by treatment with 4% special "Filtrol." The color of the stock following this treatment, and the color of fatty acids from soap made from this stock and bleached with 0.3% "Lykapon," is set forth in the following table, and compared with the results obtained by using hydrogen peroxide alone, phosphoric acid alone, and the special "Filtrol" bleach alone:

Table VI

[Tallow having original color of 13.5 R and 50 Y (1" Column).]

Pretreatment	Earth Bleach	Bleached Tallow (5¼" Col.)		Fatty Acids from Soap Bleached with 0.3% Lykapon				
				(5¼" Col.)		(3" Col.)		
1% of 85% H ₃ PO ₄	Per cent	4	57 R	75 Y	26.5 R	75 Y	13.5 R	50 Y
1% of 30% H ₂ O ₂		4	27 R	75 Y	26.5 R	75 Y	14 R	50 Y
1% of 85% H ₃ PO ₄ +1% of 30% H ₂ O ₂		4	18.5 R	75 Y	14.0 R	75 Y	7.8 R	50 Y
None.....		4	60 R	75 Y	37.0 R	75 Y	16.5 R	75 Y

It will be obvious to those skilled in the art that the invention is capable of operation with a larger variety of treating agents than those specifically listed as illustrative, and with a wide variety of procedures other than that described in the preferred embodiment. All such variations are intended to be within the invention as fall within the scope of the following claims.

We claim:

1. A process of making light colored soaps from relatively darker stocks, which comprises treating said stock in the presence of a phosphoric compound selected from the group consisting of phosphoric acids and acid phosphates, and a compound liberating nascent oxygen in the presence of said acidic compound under the treating conditions, treating the stock with a bleaching earth, saponifying the stock to form soap, and bleaching the soap.

2. A process of making light colored soaps from

relatively darker stocks, which comprises treating said stock with a phosphoric acid and a compound liberating nascent oxygen in the presence of said acid under the treating conditions, treating the stock with a bleaching earth, saponifying the stock to form soap, and treating the soap with a reducing bleach.

3. A process of making light colored soaps from relatively darker stocks, which comprises treating said stock in the presence of a phosphoric compound selected from the group consisting of phosphoric acids and acid phosphates, and a per type compound liberating nascent oxygen in the presence of the said acidic compound under the treating conditions at a temperature at which the stock is liquid, separating and washing the stock, treating it with a bleaching earth, saponifying the stock to form soap, and treating the soap with a reducing bleach.

4. A process of making light colored soaps from relatively darker stocks, which comprises treating said stock with phosphoric acid and hydrogen peroxide, treating the stock with a bleaching earth, saponifying the stock to form soap, and bleaching the soap.

5. A process of making light colored soaps from relatively darker stocks, which comprises treating said stock with phosphoric acid and hydrogen peroxide, treating the stock with a bleaching earth, saponifying the stock to form soap, and treating the soap with a hydrosulfite bleach.

6. A process of making light colored soaps from relatively darker stocks, which comprises treating said stock with phosphoric acid and sodium chlorite, treating the stock with a bleaching earth, saponifying the stock to form soap, and treating the soap with a hydrosulfite bleach.

7. A process of making light colored soaps from relatively darker stocks, which comprises treating said stock with phosphoric acid and sodium perborate, treating the stock with a bleaching earth, saponifying the stock to form soap, and treating the soap with a hydrosulfite bleach.

8. A process of making light colored soaps from relatively darker stocks, which comprises treating said stock with a phosphoric acid and a compound liberating nascent oxygen in the presence of said acid under the treating conditions at a temperature at which the stock is liquid, separating the stock from sludge and washing it with water, treating the stock with a bleaching earth, saponifying the stock to form soap, and treating the soap with a reducing bleach.

9. A process of making light colored soaps from relatively darker stocks, which comprises treating said stock with phosphoric acid and hydrogen peroxide at a temperature at which the stock is liquid, separating the stock from sludge and washing it with water, treating the stock with a bleaching earth, saponifying the stock to form soap, and treating the soap with a hydrosulfite bleach.

10. A process of making light colored soaps from relatively darker stocks, which comprises adding to said stock 0.1% to 2.0% of a phosphoric compound selected from the group consisting of phosphoric acids and acid phosphates, and 0.1% to 2.0% of a compound liberating nascent oxygen in the presence of said acidic compound under the treating conditions, agitating the mixture at a temperature above the melting point of the stock but below 90° C. for $\frac{1}{4}$ to $1\frac{1}{2}$ hours, separating the sludge from the stock, washing the stock with water, drying the stock, bleaching the stock with a bleaching earth, separating the stock from the bleaching earth, saponifying the stock to form

soap, and treating the soap with a reducing bleach.

11. A process of making light colored soaps from relatively darker stocks, which comprises adding to said stock 0.1% to 2.0% of a phosphoric acid and 0.1% to 2.0% of a peroxide type compound liberating nascent oxygen in the presence of said acid under the treating conditions agitating the mixture at a temperature above the melting point of the stock but below 90° C. for $\frac{1}{4}$ to $1\frac{1}{2}$ hours, separating the sludge from the stock, washing the stock with hot water, drying the stock by heating it to a temperature above 100° C., bleaching the stock with 2% to 6% of a bleaching earth, separating the stock from the bleaching earth, saponifying the stock to form soap, and treating the soap with a reducing bleach.

12. A process of making light colored soaps from relatively darker stocks, which comprises adding to said stock 0.1% to 2.0% of about 85% phosphoric acid and 0.1% to 2.0% of about 30% hydrogen peroxide, agitating the mixture at a temperature of about 40° to 70° C. but above the melting point of the stock, for $\frac{1}{4}$ to $1\frac{1}{2}$ hours until the bleaching action substantially ceases, separating the sludge from the stock, washing the stock with 10 to 25% hot water, drying the stock by heating it to a temperature above 100° C., bleaching the stock with 2% to 6% of a bleaching earth, separating the stock from the bleaching earth, saponifying the stock to form soap, and treating the soap with a hydrosulfite bleach.

13. A process of making light colored soaps from relatively darker stocks, which comprises adding to said stock 0.1% to 2.0% of about 85% phosphoric acid and 0.05% to 1.0% of sodium chlorite, agitating the mixture at a temperature of about 40° to 70° C. but above the melting point of the stock, for $\frac{1}{4}$ to $1\frac{1}{2}$ hours until the bleaching action substantially ceases, separating the sludge from the stock, washing the stock with 10 to 25% hot water, drying the stock by heating it to a temperature above 100° C., bleaching the stock with 2% to 6% of a bleaching earth, separating the stock from the bleaching earth, saponifying the stock to form soap, and treating the soap with a hydrosulfite bleach.

14. A process of making light colored soaps from relatively darker stocks, which comprises adding to said stock 0.1% to 2.0% of about 85% phosphoric acid and 0.1% to 2.0% of sodium perborate, agitating the mixture at a temperature of about 40° to 70° C. but above the melting point of the stock, for $\frac{1}{4}$ to $1\frac{1}{2}$ hours until the bleaching action substantially ceases, separating the sludge from the stock, washing the stock with 10 to 25% hot water, drying the stock by heating it to a temperature above 100° C., bleaching the stock with 2% to 6% of a bleaching earth, separating the stock from the bleaching earth, saponifying the stock to form soap, and treating the soap with a hydrosulfite bleach.

15. A process of making light colored soaps from relatively darker stocks, which comprises adding to said stock 1% of about 85% phosphoric acid and 1% of about 30% hydrogen peroxide, agitating the mixture at a temperature of about 60° to 70° C. for about one hour, settling and separating the sludge formed from the stock, washing the stock with hot water, drying the stock by heating it to a temperature of about 105° C., bleaching the stock with 2% to 6% of a bleaching earth at a temperature of about 105° C., separating the stock from the bleaching earth,

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saponifying the stock to form soap, and bleaching the soap with sodium hydrosulfite.

16. A process of making light colored soaps from relatively darker stocks, which comprises treating said stock with nascent oxygen in the presence of a phosphoric compound selected from the group consisting of phosphoric acids and acid phosphates, treating the stock with a bleaching earth, saponifying the stock to form soap, and bleaching the soap.

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