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PROCESS OF BLEACHING FATS AND OILS

Lloyd F. Henderson, Reading, and Louis H. Libby, Boston, Mass., assignors to Lever Brothers Company, Cambridge, Mass., a corporation of Maine

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17 Claims. (Cl. 260—423)

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This invention relates to the bleaching of very dark oils and fats.

Oils and fats are available in many grades, a large part of the available supply being in the form of very dark fats and greases. Except when the oil or fat available is very high grade stock, it is often necessary to use a stock which is dark in color for the purpose at hand. However, for many purposes a light colored oil or fat is desirable. When dark colored stock is used in making soap, 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. When the oil or fat is to be used in an edible product it is very important that it be of light color. Most other uses of oils and fats, such as for sizes and dressings in the textile and leather industry, require a light colored product. For this reason the art has developed numerous processes, the object of which is to produce lighter oils and fats from darker stocks.

When the oil or fat can be used as a fatty acid, such as in soap making, candle making, etc., it is possible to hydrolyze or split glyceride oils and fats, and distill the fatty acids to separate them from the coloring matter. 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 or other final product.

Another proposal has been to convert the glycerides into the corresponding methyl or ethyl esters of the fatty acids and to distill these; soap may be 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.

For many uses it is desirable to have the oil or fat in the original glyceride form, and many soap making operations even require or prefer the stock in this form. Under these circumstances, the oil or fat must be bleached while in the glyceride form if it is to be improved in color.

A very common method has been to bleach the oil or fat with a bleaching earth, such as fuller's earth, and especially with 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

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as that desired, particularly when it is necessary to bleach very dark stocks into relatively light colored oils and fats. 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 reasons, to use the stock for some purpose for which its dark color is not objectionable.

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 that are to be used in soap making 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 industry for lighter colored oils and fats, and with the dwindling supplies of high-grade stocks, it has become increasingly important to utilize dark colored stocks to augment the supply.

We have discovered that if an oil or fat, especially the low-grade dark fats and greases, is given a preliminary treatment with both an acidic phosphoric compound and an oxygen liberating compound, following which the oil or fat is given a conventional earth bleach, it is possible to effect a remarkable bleaching action and to produce stocks 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 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 stock with an earth bleach. Our invention has revealed the unobvious fact that apparently the combined use of both an oxygen liberating compound and phosphoric acidic compound places the oil or fat in a peculiar condition so that when treated with an earth bleach there is an unexpected bleaching action on the oil or fat, as compared with the action of these materials separately. Our invention is to be distinguished from the known proposal to use such oxygen liberating compounds with sulfuric acid, with or without a phosphate salt, and our process is not the equivalent of such a proposal.

When the oil or fat is to be used in making soap, the invention is significant because it 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 less susceptible to color reversion, especially if the soap is subjected to the bleaching action of reducing agents added to soap.

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 very difficult to bleach stock high in free fatty acids, and dark greases have generally heretofore been usable for making light products only after splitting and distillation.

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, preferably 10 to 25%, may be done so as to remove phosphoric acid and compounds thereof. Careful neutralization with alkali may be employed in connection with the water washing at any stage.

The washed oil may be dried in accordance with the usual processes, such as by heating to a temperature above 100° C., 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, such as 80° to 125° C. After this the stock is filtered to remove the earth. The earth bleaching operation and variations thereof are well-known in the industry.

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 compound.

The oxygen liberating compound may be, for example, a "per" type oxidizing compound, such as hydrogen peroxide; an alkali metal peroxide, such as sodium peroxide; per borates such as sodium perborate; per carbonates, such as sodium percarbonate; 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 contain 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 compound and the per compound may be within the range of 0.01% to 2% of the oil or fat to be treated. They 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. 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. In the case of phosphoric acid, the amount may be 0.1% or 0.2% to 2.0%. In the case of hydrogen peroxide, the amount may be 0.25% or 0.5% to 1.0% or 2.0% (of a 30% solution). The chlorites and hypochlorites can be used in a smaller amount, such as 0.05% to 1.0%. If the oxygen liberating compound is of a alkaline type, such as sodium perborate or percarbonate, the amount of the acidic phosphoric compound should be sufficient to place the mixture on the acid side during all of the reaction.

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 represents 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 ½ to 1½ 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, too dark to read the color thereof and containing 6.9% of free fatty acids (as oleic) was bleached using the process of the invention described herein, and compared with the same stock bleached with earth in the conventional manner, and with the same stock bleached only with phosphoric acid and only with hydrogen peroxide. 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 too dark to read—16.9% F. F. A.]

Sample	Pretreatment	Earth Bleach	Color	
		Per Cent		
1	1% of 85% H_3PO_4	6	30.0 R	75 Y
2	1% of 30% H_2O_2	6	17.5 R	75 Y
3	1% of 85% H_3PO_4+1% of 30% H_2O_2	6	8.5 R	75 Y
4	None	6	50.0 R	75 Y

The combined action of phosphoric acid and hydrogen peroxide, as compared with these materials utilized separately, is indicated also 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 II

[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
7	do	20	14.0 R	75 Y

From the foregoing it will be seen that a total of 2% of the combined bleach is markedly superior to 2% of either phosphoric acid or hydrogen peroxide. The color of the stock bleached in accordance with the invention is almost as good as the color of stock bleached with 20% earth. The loss of stock in the 14% additional earth would render the use of such large amounts of bleach entirely impractical as compared with the process of the invention.

When the same tallow was bleached with 1% of 30% H_2O_2 and 1% of $H_4P_2O_7$ 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 advantage of the invention in bleaching oils and fats for soap making and 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 a somewhat lighter stock:

Table III

Treatment	Color of Tallow (1'' Col.)		Color of Fatty Acids from Hydrosulfite Bleached Soap (1'' Col.)	
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 reduction and represents a significant improvement.

Other examples, using other oxygen liberating compounds, and the results obtained are shown in the following tables:

Table IV

[Very dark tallow.]

Acidic Compound	Oxygen Liberating Compound	Conditions		Earth Bleach	Color 1'' Col.	
		Temp.	Time			
		° *C.*	*Hours*	*Percent*		
None	None			6	21.0 R	60 Y
1% H_3PO_4	1% H_2O_2	65	1	6	11.2 R	60 Y
Do	1% $NaBO_3 4H_2O$	65	1	6	9.6 R	60 Y

Table V

[Very dark tallow.]

Acidic Compound	Oxygen Liberating Compound	Conditions		Earth Bleach	Color 1" Col.	
		Temp.	Time			
		° C.	Hours	Percent		
None	None			6	30.0 R	75 Y
1% H_3PO_4	1% H_2O_2	65	1	6	14.0 R	75 Y
Do	0.1% $NaClO_2$*	65	1	6	12.5 R	75 Y

* Dissolved in 5% H_2O based on weight of tallow.

Table VI

[Very dark tallow.]

Acidic Compound	Oxygen Liberating Compound	Conditions		Earth Bleach	Color 1" Col.	
		Temp.	Time			
		° C.	Hours	Percent		
None	None			10	59.9 R	75 Y
1% H_3PO_4	1% H_2O_2	65	1	6	21.0 R	75 Y
Do	2% NaOCl*	65	1	6	20.0 R	75 Y

*2% of a 5% solution of NaOCl.

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 as fall within the scope of the following claims are intended to be within the invention.

We claim:

1. A process of bleaching dark oil and fat 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 the said acidic compound under the treating conditions, said phosphoric compound imparting those acidic conditions under which said oxygen liberating compound acts to achieve a pronounced lightening of the color of said stock, and then treating the stock with a bleaching earth.

2. A process of bleaching dark oil and fat stocks which comprises treating said stock with a phosphoric acid and a per type compound liberating nascent oxygen in the presence of said acid under the treating conditions, said phosphoric acid imparting those acidic conditions under which said per type oxygen liberating compound acts to achieve a pronounced lightening of the color of said stock, and then treating the stock with a bleaching earth.

3. A process of bleaching dark oil and fat 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, said phosphoric compound imparting those acidic conditions under which said per type oxygen liberating compound acts to achieve a pronounced lightening of the color of said stock, separating and washing the stock, and treating it with a bleaching earth.

4. A process of beaching dark oil and fat stocks, which comprises treating said stock with phosphoric acid, said phosphoric acid imparting those acidic conditions under which said hydrogen peroxide acts to achieve a pronounced lightening of the color of said stock, and hydrogen peroxide, and then treating the stock with a bleaching earth.

5. A process of bleaching dark oil and fat stocks, which comprises treating said stock with phosphoric acid, said phosphoric acid imparting those acidic conditions under which said sodium chlorite acts to achieve a pronounced lightening of the color of said stock, and sodium chlorite, and then treating the stock with a bleaching earth.

6. A process of bleaching dark oil and fat stocks, which comprises treating said stock with phosphoric acid, said phosphoric acid imparting those acid conditions under which said sodium perborate acts to achieve a pronounced lightening of the color of said stock, and sodium perborate, and then treating the stock with a bleaching earth.

7. A process of bleaching dark oil and fat 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, said phosphoric acid imparting those acidic conditions under which said oxygen liberating compound acts to achieve a pronounced lightening of the color of said stock, separating the stock from sludge and washing it with water, and treating the stock with a bleaching earth.

8. A process of bleaching dark oil and fat stocks, which comprises treating said stock with phosphoric acid and hydrogen peroxide at a temperature at which the stock is liquid, said phosphoric acid imparting those acidic conditions under which said hydrogen peroxide acts to achieve a pronounced lightening of the color of said stock, separating the stock from sludge and washing it with water, and then treating the stock with a bleaching earth.

9. A process of bleaching dark oil and fat 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 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 ¼ to 1½ hours, said phosphoric compound impart-

ting those acidic conditions under which said per type oxygen liberating compound acts to achieve a pronounced lightening of the color of said stocks, separating the sludge from the stock, washing the stock with water, drying the stock, bleaching the stock with a bleaching earth, and separating the bleached stock from the earth.

10. A process of bleaching dark oil and fat stocks, which comprises adding to said stock 0.25% to 2.0% of a phosphoric acid and 0.25% 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 ¼ to 1½ hours, said phosphoric acid imparting those acidic conditions under which said per type oxygen liberating compound acts to achieve a pronounced lightening of the color of said stock, 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, and separating the bleached stock from the earth.

11. A process of bleaching dark oil and fat stocks, which comprises adding to said stock 0.25% to 2.0% of a phosphoric acid and 0.25% 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, continuing the agitating for ¼ to 1½ hours until the bleaching action substantially ceases, said phosphoric acid imparting those acidic conditions under which said hydrogen peroxide acts to achieve a pronounced lightening of the color of said stock, 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, and separating the bleached stock from the earth.

12. A process of bleaching very dark grease stocks, which comprises adding to said stock 0.25% to 2.0% of about 85% phosphoric acid and 0.25% 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 about ¼ to 1½ hours until the bleaching action substantially ceases, said phosphoric acid imparting those acidic conditions under which said hydrogen peroxide acts to achieve a pronounced lightening of the color of said stock, 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, and separating the bleached stock from the earth.

13. A process of bleaching very dark grease stocks, which comprises adding to said stock 0.5% to 1.0% of about 85% phosphoric acid and 0.5% to 1.0% 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 ¼ to 1½ hours until a substantial bleaching action is obtained, said phosphoric acid imparting those acidic conditions under which said hydrogen peroxide acts to achieve a pronounced lightening of the color of said stock, settling and separating the sludge from the stock, washing the stock with 5 to 50% 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 at a temperature of about 80° to 125° C. and separating the bleached stock from the earth.

14. A process of bleaching very dark grease stocks, which comprises adding to said stock 0.5% to 1.0% of about 85% phosphoric acid and 0.5% 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 ¼ to 1½ hours until a substantial bleaching action is obtained, said phosphoric acid imparting those acidic conditions under which said sodium perborate acts to achieve a pronounced lightening of the color of said stock, settling and separating the sludge from the stock, washing the stock with 5 to 50% 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 at a temperature of about 80° to 125° C. and separating the bleached stock from the earth.

15. A process of bleaching very dark grease stocks, which comprises adding to said stock 0.5% to 1.0% of about 85% phosphoric acid and 0.05 to 1.0% of sodium chloride, agitating the mixture at a temperature of about 40° to 70° C. but above the melting point of the stock for ¼ to 1½ hours until a substantial bleaching action is obtained, said phosphoric acid imparting those acidic conditions under which said sodium chloride acts to achieve a pronounced lightening of the color of said stock, settling and separating the sludge from the stock, washing the stock with 5 to 50% 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 at a temperature of about 80° to 125° C. and separating the bleached stock from the earth.

16. A process of bleaching very dark grease stocks, which comprises adding to said stock 0.5% to 1.0% of about 85% phosphoric acid and 0.05 to 1.0% of sodium hypochlorite, agitating the mixture at a temperature of about 40° to 70° C. but above the melting point of the stock for ¼ to 1½ hours until a substantial bleaching action is obtained, said phosphoric acid imparting those acidic conditions under which said sodium hypochlorite acts to achieve a pronounced lightening of the color of said stock, settling and separating the sludge from the stock, washing the stock with 5 to 50% 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 at a temperature of about 80° to 125° C., and separating the bleached stock from the earth.

17. A process of bleaching dark oil and fat 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, said phosphoric compound imparting those acidic conditions under which said nascent oxygen acts to achieve a pronounced lightening of the color of said stock, and then treating the stock with a bleaching earth.

LLOYD F. HENDERSON.
LOUIS H. LIBBY.

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Certificate of Correction

Patent No. 2,483,413 October 4, 1949

LLOYD F. HENDERSON ET AL.

It is hereby certified that errors appear in the printed specification of the above numbered patent requiring correction as follows:

Column 4, line 46, for "0.2%" read *0.25%*; column 5, line 23, for "6.9%" read *16.9%*; column 7, line 74, beginning with the words "acid, said" strike out all to and including "then" in column 8, line 29 and insert instead *acid and hydrogen peroxide, said phosphoric acid imparting those acidic conditions under which said hydrogen peroxide acts to achieve a pronounced lightening of the color of said stock, and then*; same column 8, line 33, beginning with "acid, said" strike out all to and including "then" in line 37 and insert instead *acid and sodium chlorite, said phosphoric acid imparting those acidic conditions under which said sodium chlorite acts to achieve a pronounced lightening of the color of said stock, and then*; line 40, beginning with "acid, said" strike out all to and including "then" in line 44 and insert instead *acid and sodium perborate, said phosphoric acid imparting those acidic conditions under which said sodium perborate acts to achieve a pronounced lightening of the color of said stock, and then*; column 9, line 3, for "stocks" read *stock*; column 11, line 11, list of references cited, for "Australia" read *Austria*; column 12, line 4, for "pp. 2746" read *pp. 274–6*;

and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 31st day of January, A. D. 1950.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.