UNITED STATES PATENT OFFICE.

BALTHASAR E. REUTER, OF CHICAGO, ILLINOIS, ASSIGNOR TO REUTER PROCESS COMPANY, OF CHICAGO, ILLINOIS, A CORPORATION OF ILLINOIS.

PROCESS OF BLEACHING FATS, OILS, AND THEIR DERIVATIVES.

1,219,486. Specification of Letters Patent. Patented Mar. 20, 1917.

No Drawing. Application filed November 8, 1912. Serial No. 730,214.

To all whom it may concern:

Be it known that I, BALTHASAR E. REUTER, a citizen of the United States, residing at Chicago, in the county of Cook and State of Illinois, have invented a certain new and useful Improvement in Processes of Bleaching Fats, Oils, and Their Derivatives, of which the following is a full, clear, concise, and exact description.

My invention relates to an improved process for bleaching fats and oils and derivative products resulting therefrom. In it, I include fats or oils either completely or partially decomposed, fatty acids resulting therefrom, glycerin and soap.

My present invention consists in subjecting these substances to successive steps, which effect their bleaching in a more perfect manner than has been done heretofore. My improved process differs from previous bleaching processes in that I find it advantageous to thoroughly wash the fatty acids resulting from the decomposition of the fats or oils before beginning the bleaching operation. After this step, the bleaching operation consists of successive steps, no one of which is sufficient to completely bleach all of the material which is undergoing treatment, whether this material be the fats and oils or decomposed and washed fatty acids.

In carrying out my invention, various bleaching agents may be employed, as decroline, blankit, or any one of a wide series of similar hydro-sulfite compounds. In using these bleaching reagents, I find it desirable in carrying out my improved process to employ a sufficient amount of sufuric acid, in connection with each one of the bleaching operations, to completely decompose the quantity of bleaching reagent which is employed. As before stated, this amount of bleaching reagent is not sufficient to wholly accomplish the bleaching of the desired amount of material being treated.

A marked advantage of treating the material to be bleached in successive steps rather than in a single operation is that each one of the incomplete bleaching operations serves to remove from the material a certain portion of the coloring matter and completely separate it from the material by precipitation, thus leaving the material freer to yield to the bleaching operation of the next amount of bleaching agent used, as a result of which the complete bleaching operation effected by successive steps, each in itself insufficient to completely bleach the material, effects far more efficient and thorough bleaching than is possible where the material is treated in a single operation, even though in the single operation an amount of bleaching agent is used which ought to be sufficient to completely bleach the material.

Furthermore, the conditions under which the bleaching operation takes place must be such, in order to permit the bleaching agent to act upon the material being treated, that the bleaching agent is quickly and rapidly decomposed in intimate contact with the material treated, whether as a result of heating the solution or the presence of acid added for the purpose of decomposing the bleaching agent and, when it is attempted to bleach the material in a single operation, much of the efficacy of the bleaching agent is lost, owing to the fact that there is not a sufficient time interval for it to properly act upon and permeate the material being treated before it is itself considerably decomposed and, therefore, made correspondingly ineffective. By my process, this objectionable feature of previous bleaching processes is eliminated, since I use in each step of the bleaching operation only a small amount of the bleaching agent, and this comparatively small amount has better opportunity to act upon the material being bleached before the bleaching agent thus used is decomposed.

My improved process of bleaching fats and oils and their derivatives may be carried out with any fats and oils or any fatty acids regardless of their origin, as a result of which it will not be necessary in the present application to particularly describe any special means of producing fatty acids or other derivatives of fats and oils, but, on the other hand, it will be assumed that the fatty acids or other derivatives of the fats and oils are at hand and ready for bleaching and that it is only necessary to subject them to the steps of my process to produce the desired resulting bleached material.

In carrying out my process, use is made of a boiling tank provided with a mechanical agitator, provision being made to exclude air from the tank and to maintain the contents

of the tank under substantially atmospheric pressure during the successive steps of bleaching, as indicated in my co-pending application, Serial No. 698,615, filed May 20, 1912.

Before the first step of the bleaching process and preparatory to subjecting the material to the bleaching process, the fats or oils or fatty acids to be treated are introduced into the boiling tank provided, and if this material is not already in the form of an emulsion; that is, finely subdivided with water between the particles, it is placed in that condition by any suitable means known in the art, one of which is described in my co-pending application referred to, as an incident of the decomposition of the fats and oils to produce fatty acids and glycerin. The process described in detail immediately below relates particularly to the bleaching of fatty acids, although it will be understood that fats and oils are similarly treated when it is desired to treat them without decomposition. With the fatty acids which are to be bleached in the form of an emulsion in the boiling tank, as referred to, water is added to the fatty acids to wash out the entrained glycerin, reagent and foreign matter, whereupon the mixture is brought to a boil and the agitator is started. The fatty acids and water will, as a result of the prior treatment, be in good emulsion and should be boiled and agitated for from 3 to 6 hours, thereupon adding sufficient sulfuric acid to break the emulsion, whereupon the water will settle out. The quantity of sulfuric acid necessary to be added will make the water test from .05% to .1% acid, the percentage being dependent upon the nature of the fats or oils or fatty acids undergoing treatment. After the additional sulfuric acid has been intermixed, the agitator is stopped and the steam turned off and the contents of the tank are allowed to settle for from one-half to one hour. The water which settles out as a result of this washing is then drawn from the tank at the bottom. The fatty acids remaining are then subjected to heat and agitation, whereupon a small quantity of distilled water is added, together with from .05% to .2% of the Badische Co.'s decroline. This mixture is boiled from 15 to 30 minutes. If thereupon the water does not settle out or if the bleaching agent is not thoroughly decomposed, a small quantity of sulfuric acid, sufficient to break the emulsion and decompose the bleaching agent, is added; this will cause the water to settle out in from 15 to 30 minutes, whereupon it is withdrawn from the bottom.

The remaining fatty acids are then again boiled and agitated, and a small quantity of water and from .05% to .2% of decroline added as before. The boiling and agitation are continued for from 15 to 30 minutes. If again the water does not settle out promptly upon turning off the heat and stopping the agitator, or if the bleaching agent is not thoroughly decomposed, sulfuric acid sufficient to break the emulsion and decompose the bleaching agent is added, whereupon the water will settle out in from 15 to 30 minutes. This second water is drawn off as before.

Again the fatty acids are boiled and agitated with the addition of .05% to .2% of decroline and a small quantity of water, and the boil is continued for 15 to 30 minutes, whereupon I add sufficient barium carbonate, sodium carbonate, or caustic soda to neutralize the sulfuric acid in the water; that is to say, the neutralizing agent is added until the water in the mixture tests neutral to methyl orange.

For the production of pure white soaps, it is desirable to add the small quantity of water with the decroline used in the last bleaching operation before neutralizing the mixture, and the same is true in connection with the use of other bleaching agents of the hydro-sulfite compounds as well as decroline. For this purpose, it is also desirable to precipitate the water and draw it off before adding the neutralizing material referred to. A further very desirable object secured by allowing the water to settle and drawing off the same before neutralizing the remaining mixture, is that in connection with glycerin, requiring considerable treatment to bleach and purify it, more of the effect of the bleaching agent contained in the water is secured upon the glycerin by treating it in this manner than as though the neutralizing agent were introduced before the water was permitted to settle. Furthermore, the settling operation tends to remove the free entrained glycerin that may be contained in the fatty acids and thus save it, where, if the neutralizing agent were introduced before the water was allowed to settle, this entrained glycerin would be lost, at least in considerable measure. In this manner, the bleaching agent is made to do double duty, for, after having acted upon the fatty acids to bleach them in the manner above described, sufficient of the bleaching agent remains in the water in effective condition to act upon and bleach the glycerin if permitted to come in contact with it. This would not occur if the water were not allowed to settle before the neutralizing operation. It is to be borne in mind in this connection that the wash water, containing the bleaching agent remaining from the bleaching operation, during any step contains a sufficient amount of the bleaching agent in effective condition to in part bleach glycerin with which it may be brought into contact, whether the glycerin results from the decomposition of the fats

and oils producing the fatty acids bleached by the process, or whether the glycerin be the result of the decomposition of fats or oils at some other time or by some other process, and by my process, I neutralize the wash water containing the residue of bleaching agent referred to, whereas, in other processes, this wash water is thrown away and no valuable result is secured from it. When the water is settled from the mixture and drawn off before the neutralizing operation from the last bleaching boil, if the water does not promptly settle out, or if the bleaching agent is not thoroughly decomposed, a small quantity of sulfuric acid, sufficient to break the emulsion and decompose the remaining bleaching agent, is added, and this will cause the water to settle out in from 15 to 30 minutes, whereupon it is drawn from the bottom of the tank. After neutralizing, the agitator is stopped and the steam turned off and the water allowed to settle to the bottom. The water which settles out is withdrawn from the bottom of the tank, whereupon the agitator is run for from 15 to 30 minutes. This finishes the fatty acids, which will then be ready for the manufacture of soaps, stearic acids, candles, or for any other purposes to which high grade fatty acids of good color are adaptable.

It will be understood that, while I have described my bleaching process above as consisting of three successive steps, I do not limit myself to this particular number of bleaching steps, but may use any other number, depending upon the particular condition and mixture of the fatty acids, fats or oils to be treated. An essential feature of my process consists in bringing into contact with the fatty acids, fats or oils to be treated a comparatively small amount of the bleaching agent at a time, as a result of which the bleaching agent has ample opportunity to effectively attack the coloring matter of the fatty acids, fats or oils. In view of this, it will at once appear that the quantity of bleaching agent required in each boiling operation may, instead of being introduced all at once, be introduced gradually during the boiling operation, which obviates the disadvantage of introducing at the same time all of the bleaching agent used. It is important in introducing the bleaching agent gradually instead of all at once to be certain that there is a sufficient amount of sulfuric acid present in the solution at all times to completely decompose the bleaching agent added and to break the emulsion. Otherwise, the full effect of the bleaching agent is not secured. An essential feature of my present invention consists in providing in the solution at all times a sufficient quantity of sulfuric acid to decompose the bleaching agent added and to break the emulsion, for, by this means, good results are secured with a bleaching process consisting of a single boiling operation, provided the bleaching agent is added slowly throughout the boil, although, as poined out above, the results are superior if the bleaching operation consists of a number of successive boils.

It is to be understood that the quantity of bleaching agent required in each boil is determined largely by the nature of the fatty acids, fats or oils to be treated, as well as by the number of boils to which the material is subjected to complete the bleaching operation. Ordinarily, the amount of bleaching agent required is within the range given in connection with each step of the bleaching process described above, whether decroline is used, as referred to, or whether some one of the other bleaching agents of the hydro-sulfite family known in the art is employed.

In connection with the bleaching of soap, my improved process may be used to advantage as follows: After the saponification of the soap and the preliminary settling changes and during the final settling or finishing change, from about half an hour to two hours before the end of the final finishing change I add to the mixture from .05% to .6% of one of the hydrosulfite bleaching compounds, of which "blankit" is representative and commonly known to those in the art, with the effect that the bleaching compound is retained in the solution in contact with the material which it is desired to bleach. The subsequent boiling of from half an hour to two hours serves to thoroughly decompose the reagent and bring it into contact with the soap mass being treated. This is superior to processes heretofore used, in that in the art heretofore the bleaching compound has been added either during the saponification change or immediately thereafter, with the result that a large part of the bleaching agent and, therefore, a large part of the effectiveness of such agent is removed by the saponification change and first settling change, which difficulty is avoided by my process, as pointed out. To prepare the blankit for introduction into the soap mixture, it is advisable to add to it enough alkali solution to make a thin paste, so that upon introducing this paste into the soap mass it will thoroughly mix therewith.

Similarly, glycerin may also be bleached or purified by my process by adding thereto from .05% to 1% of bleaching compound, such as decroline or blankit, this addition being made either to the glycerin water before it is concentrated or to the crude glycerin before it goes to the still, and, after the addition of the bleaching compound, the mixture is boiled for from half an hour to

two hours to thoroughly intermingle the bleaching compound and the glycerin. To facilitate the decomposition of the bleaching compound, if decroline is used, it is advisable to introduce into the mixture a small amount of sulfuric acid sufficient to decompose the bleaching compound used, whereas if blankit is employed, it is advisable to mix the blankit with a sufficient quantity of alkaline solution to dissolve the bleaching compound used.

In connection with my bleaching process, whether used to bleach fatty acids, fats, oils, soap, or glycerin, it is advisable to make a thin paste of the bleaching compound before adding it to the mixture to be bleached, and this is done either with water or an alkaline solution, depending upon the nature of the bleaching compound used, for certain of these compounds, as, for example, decroline, require the addition of acid to decompose them, whereas with others of these compounds, as, for example, blankit, the decomposition is facilitated by dissolving them in alkali.

In bleaching most fats and oils and some fatty acids by means of blankit, it is advisable, in order to secure the best results, that the mixture shall be made neutral to methyl-orange before introducing the bleaching compound.

The compound known to the trade as "decroline" and hereinbefore referred to is fully described in patent to Karl Reinking *et al* No. 793,610, dated June 27, 1905.

Others of the hydro-sulfite compounds which are well known in the art and may be used in carrying out my bleaching process are rongalite and palatinit, which, as well as decroline and blankit, are manufactured by the Badische Company, although my process is not limited to the use of any of the bleaching compounds which have been specifically mentioned above, since it may be carried out by means of others of the numerous series of hydro-sulfite compounds known in the art.

While I have shown and described my improved process of bleaching as pointed out above, I do not wish to limit myself to the exact steps described, as I am aware that many minor changes may be made in them without departing from the spirit of my invention, and I claim such equivalents as may suggest themselves to those skilled in the art.

What I claim is:

1. The process of bleaching fats, oils, fatty acids, glycerin, or soap, which consists in forming an emulsion of the material treated by the addition and mixture therewith of a sufficient quantity of water, boiling the mixture, excluding air from contact with the mixture and at the same time maintaining the mixture under substantially atmospheric pressure, adding a hydro-sulfite bleaching agent, slowly and in small successive amounts to the mixture throughout the complete bleaching operation to prevent wasteful decomposition of the bleaching agent if added rapidly or in large amounts, and maintaining the mixture in a sufficiently acid condition to decompose the bleaching agent as it is added to the mixture.

2. The process of bleaching fatty acids which consists in successively boiling an emulsion of the fatty acids with a hydro-sulfite compound, maintaining the mixture under substantially atmospheric pressure excluding air from contact with the fatty acids during the boiling operations and maintaining the mixture in a sufficiently acid condition to decompose the bleaching agent as it is added to the mixture.

3. The process of bleaching fatty acids which consists in intimately mixing with the fatty acids a sufficient quantity of water to form an emulsion, excluding air from contact with the mixture by maintaining a steam seal above the mixture and repeatedly subjecting the mixture to the following treatment: heating and agitating the mixture with water, together with from .05% to 2% of hydro-sulfite compound, thereupon boiling from 15 to 30 minutes and maintaining the mixture at substantially atmospheric pressure, adding sufficient sulfuric acid to break the emulsion and decompose the bleaching agent, and settling out and withdrawing the water.

In witness whereof, I hereunto subscribe my name this 31st day of October, A. D. 1912.

BALTHASAR E. REUTER.

Witnesses:
LESLIE W. FRICKE,
ALBERT C. BELL.