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## PROCESS OF DECOLORIZING AND REFINING CRUDE COTTONSEED OIL.

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This invention relates to a new and improved process for treating certain vegetable oils; the present embodiment thereof being particularly adapted to the refining of crude cotton seed oil.

One of the objects of the invention is to provide a process which will permit of the reversing of present practice, viz: the first refining an oil with caustic and then bleaching or de-colorizing. Many attempts have been made to first bleach or de-colorize a crude cotton seed oil but without commercial success.

One of the objects of the invention is to provide a process which may be carried on more economically than have processes of this character hitherto employed.

Another object of the invention is to provide a process wherein certain water soluble compounds in combination with the oil are removed prior to the bleaching operation, which said impurities, if contained in the oil before bleaching, are detrimental to and prevent the successful de-colorizing of the oil, and which at the present time are removed by using an excess of caustic in the refining operation.

Another object of the invention is to provide a process wherein the oil is first decolorized before the removal of the fatty acids therefrom; whereby the quality of the soap stock later produced is vastly improved.

A further object of the invention is to provide a process whereby certain albuminous, mucilaginous, colloidal, coloring and other undesirable matters present in the crude oil are first removed and the oil decolorized, so that said oil is free from enzymes and ferments whereby the de-colorized oil may be stored for an indefinite length of time without deterioration before refining.

A still further object of the invention is to provide a process of refining oil of the above character wherein the amount of caustic usually employed in connection with the refining of such oil is greatly reduced while at the same time producing a soap stock which is vastly superior in color and quality than that produced in processes hitherto carried on.

Another object of the invention is to reduce or lessen the loss obtained by present refining methods, that is, to obtain a larger recovery of neutral or refined oil and limit the saponification to the neutralizing of the fatty acids contained in the oil. In other words doing away with the necessity of using an excessive quantity of caustic which is at present employed not only to neutralize the fatty acids contained in the oil but also to remove color and to remove other detrimental elements in the oil, but which at the same time saponifies a considerable quantity of neutral oil, which is thrown into the soap stock and greatly reduced in value.

Other objects and aims of the invention more or less specific than those referred to above, will be in part obvious and in part pointed out in the course of the following description of the steps and the relation of each step to one or more of the others thereof employed in carrying out my process, and the scope of protection contemplated will be indicated in the appended claims.

In carrying on my improved process a given quantity of oil, in the present instance crude cotton seed oil, is first put into a tank and then heated to a temperature not to exceed 110° F. This temperature, however, is not exact because in some instances the crude cotton seed oil may require a slightly higher or lower temperature. This heating operation is designed to redissolve the stearine which before heating may be present in the oil as a precipitate, due to chilling of the oil or low temperature thereof. When the oil has been heated as above described, water of approximately 10% of the weight of the oil and at a temperature ranging from 60° to 70° F. is sprayed upon the oil while the latter is being gently stirred or agitated. In treating some oils it its found that it is not necessary to heat the oil and this is true where the temperature is high enough so that no stearine is settled out of the oil. If the oil is high grade and if the room temperature or atmospheric temperature is high, no stearine will have settled from the oil and therefore the heating of the oil is not required.

The stirring or agitation of the oil is carried on until a complete "break" is obtained. When this break occurs during the agitation of the water and oil there are separated from the oil certain compounds which are carried in solution or suspension in the water which compounds are carried down to the

bottom of the tank with the water; such compounds contain albuminous, mucilaginous, colloidal and coloring matters or substances commonly classified as "Lipoids." These precipitated compounds also contain a certain amount of combined fatty acids. When the aforesaid matters have been carried down at this stage of the process the oil which up to this point had a cloudy or murky appearance will show clear.

The oil is then removed to another tank by siphoning or by any well known method wherein it is ready for the bleaching or de-colorizing operation.

In the bleaching or de-colorizing operation I preferably employ certain activated clays, preferably natural clays which when treated with acid by certain well known processes have the property of more efficiently de-colorizing oils, fats and greases than any material heretofore known or employed in this relation. I have found through practice that a material now known as Filtrol is one adapted for carrying on the bleaching operation better than any other activated clay which has come under my observation. Filtrol is acid treated clay believed to be of volcanic origin or deposited by thermal waters containing principally silica and some combined aluminum, acid soluble aluminum and acid soluble magnesium.

In carrying on the bleaching operation the decanted or siphoned oil is drawn off into a tank preferably equipped with mechanical means for agitation and is therein heated to a temperture preferably ranging from 210° to 220° F., and is maintained at such temperature during the de-colorizing operation. An activated clay material of the character above described may be added to the oil at any time after the latter has been raised to a temperature of 170° F. A vigorous agitation is maintained until the de-colorizing reaction has been completed. This de-colorizing operation which may vary according to the amount of oil treated may take from an hour to two hours to complete. During this operation any moisture which may exist in the activated clay or which may have remained in the oil after the first treatment is driven off so that the oil after filtration is reduced to what is known as a dry oil. In this connection I have found that the presence of a slight amount of water in the oil during this bleaching operation facilitates the de-colorizing of the oil or in other words assists the action of the activated agent in de-colorizing the oil.

I preferably carry on this de-colorizing operation under vacuum which method aids in removing the moisture from the oil and de-colorizing material without the necessity of using excessive temperatures.

The mass is then pumped from the agitating tank and discharged into a filter press of any approved type, the clear bleached or filtered oil being discharged to storage tanks, or after cooling it may be immediately refined. The solid matter which has been compressed into cake form in the press may be then blown with air and steamed to remove any excess of oil remaining therein; or preferably the cake may be removed to an extractor and the retained oil recovered by suitable solvents.

Referring now to the first step of my process, to wit, the treatment of the oil with the cold water, the settlings are first drawn off into a settling tank and after standing for awhile will separate into three layers, oil, solids and coloring matter, and water. The oil is skimmed off and run back into the crude storage. The water is then drawn off from the bottom and if desired the mass remaining therein may be heated or salted out to obtain any entrained oil. The mass remaining in the settling tank is then discarded or may be employed as a fertilizer inasmuch as I have found it to be very rich in nitrogenous substance.

From this point in my process present known methods and equipment for refining with caustic alkali may be employed. Referring now to the de-colorized oil produced by the treatment of the activated clay aforesaid, the latter is discharged either direct from the filtration tank or from storage into the refining tank which may be of any well known type of construction now employed in the refining of vegetable oils. At this point it may be noted that the process as thus far carried on has removed from the oil all substance which it is desired to remove except the free fatty acids. The oil has also by my process as above described been reduced to its final color, that is to say, such color as is desired in the finished oil for commercial purposes. The refining operation now carried on therefore merely consists in mixing with the oil substantially the theoretical amount of caustic alkali necessary only to remove the free fatty acids. This refining process may be conveniently started at about 108° F. or 110° F. and slightly increased to, say, 112° F. or 115° F., which facilitates the "break" or formation of soap stock in the oil. The granular soap stock is separated from the oil by settling. The only water present at this time is that added in the caustic alkali solution which unites and settles with the soap stock. In this connection I preferably add a very slight excess of caustic alkali, say for instance five hundredths of one per cent over the theoretical amount required.

I also find that the soap stock produced by the action of the caustic alkali with the free

fatty acids is a granular or grainy substance, and inasmuch as it contains no impurities of any character, the neutral oil thrown down with the soap stock may be removed therefrom by filtration instead of by present methods, and thereby recover a greater percentage of neutral oil. The soap stock thus produced is white and free from all impurities and is ready for the soap kettle and does not require to be split with acid and distilled, as does the soap stock produced by present practice.

This application is a continuation in part of my application entitled Process for decolorizing raw cottonseed oil, Serial No. 673,603, filed November 8, 1923.

Having thus described my invention what I claim as new and desire to secure by Letters Patent is:

1. The herein described process of purifying a vegetable oil, which comprises: heating said oil to a temperature not to exceed 110° F.; intimately mixing water with said heated oil; removing from the oil the water and substances carried thereby; adding an activated clay to the oil in such a manner as to produce an intimate mixture of said oil and clay; and separating from the oil the clay and any substances carried thereby.

2. The herein described process of purifying a vegetable oil, which comprises: heating said oil to a temperature of about 110° F.; intimately mixing water with said heated oil; removing from the oil the water and substances carried thereby; adding an activated clay to the oil in such a manner as to produce an intimate mixture of said oil and clay; and separating from the oil the clay and any substances carried thereby.

3. The herein described process of purifying a vegetable oil, which comprises: agitating said oil in the presence of water; removing from the oil the water and substances carried thereby; adding an activated clay to the oil in such a manner as to produce an intimate mixture of said oil and clay; separating from the oil the clay and any substances carried therein; adding to the oil sufficient caustic alkali to neutralize the free fatty acids contained in said oil; and thereafter removing from said oil the reaction products of said caustic alkali.

4. The herein described process of purifyiny a crude cotton seed oil, which comprises: heating said oil to a temperature not to exceed 110° F.; intimately mixing water with said heated oil; removing from the oil the water and substances carried thereby; adding an activated clay to the oil in such a manner as to produce an intimate mixture of said oil and clay; separating from the oil the clay and any substances carried therein; adding to the oil sufficient caustic alkali to neutralize the free fatty acids contained in said oil; and thereafter removing from said oil the reaction products of said caustic alkali.

5. The herein described process of purifying a crude cotton seed oil, which comprises: heating said oil to a temperature of about 110° F.; intimately mixing water with said heated oil; removing from the oil the water and substances carried thereby; adding an activated clay to the oil in such a manner as to produce an intimate mixture of said oil and clay; separating from the oil the clay and any substances carried therein; adding to the oil sufficient caustic alkali to neutralize the free fatty acids contained in said oil; and thereafter removing from said oil the reaction products of said caustic alkali.

6. A process for refining vegetable oils which comprises heating the oil, washing it with water and subjecting it to the action of a clarifying and bleaching agent, removing said agent together with the impurities carried off thereby, then mixing the decolorized oil still containing its fatty acids but otherwise purified, with a saponifying agent in proportions adapted to effect a maximum saponification of fatty acids, and a minimum saponification of the oil, and separating the oil and soaps.

7. A process for refining vegetable oils which comprises heating the oil, adding water to said oil with gentle but thorough agitation so that the water is thoroughly mixed with the oil without forming a permanent emulsion, removing from the oil the water and any substances carried therein, adding a decolorizing agent to the oil in such a manner that it is intimately mixed therewith, removing said agent together with the impurities carried off thereby, then mixing the decolorized oil still containing its fatty acids but otherwise purified, with a saponifying agent in proportions adapted to effect a maximum saponification of fatty acids, and a minimum saponification of the oil, and separating the oil and soaps.

8. A process for refining vegetable oils which comprises heating the oil to a temperature below the boiling point of water, adding water to said oil in such a manner that the water is thoroughly mixed with the oil without forming a permanent emulsion, removing from the oil the water and any substances carried therein, adding a decolorizing agent to the oil in such a manner that it is intimately mixed therewith, removing said agent together with the impurities carried off thereby, then mixing the decolorized oil still containing its fatty acids but otherwise purified, with a saponifying agent in proportions adapted to effect a maximum saponification of fatty acids, and a minimum saponification of the oil, and separating the oil and soaps.

9. A process for refining vegetable oils which comprises heating the oil to a temperture of about 110° F., adding water to said oil in such a manner that the water is thoroughly mixed with the oil without forming a permanent emulsion, removing from the oil the water and any substances carried therein, adding a decolorizing agent to the oil in such a manner that it is intimately mixed therewith, removing said agent together with the impurities carried off thereby, then mxing the decolorized oil still countaing its fatty acids but otherwise purified, with a saponifying agent in proportions adapted to effect a maximum saponification of fatty acids, and a minimum saponification of the oil, and separating the oil and soaps.

10. The herein described process of purifying a vegetable oil, which comprises: heating the oil; intimately mixing aproximately 10% of warm water with said oil until a "break" occurs separating the oil from the water and substances carried thereby; heating said oil to a temperature of approximately 210° F. to 220° F.; adding an activated clay to said oil in such a manner as to produce an intimate mixture of said oil and said slay; and separating from said oil the clay and any substances carried thereby.

11. The herein described process of purifying a vegetable oil, which comprises: heating the oil; intimately mixing approximately 10% of water at a temperature of from 60 to 70° F. with said oil to produce a "break"; separating the oil from the water and substances carried thereby; heating said oil to a temperature of approximately 210° F. to 220° F.; adding an activated clay to said oil in such a manner as to produce an intimate mixture of said oil and said clay; and separating from said oil the clay and any substances carried thereby.

12. The herein described process of purifying a vegetable oil, which comprises: heating the oil; intimately mixing approximately 10% of warm water with said oil until a "break" occurs; separating the oil from the water and substances carried thereby; heating said oil to a temperature of approximately 210° F. to 220° F.; adding an activated clay to said oil at any time after said oil has been heated to 170° F. in such a manner as to produce an intimate mixture of said oil and said clay; and separating from said oil the clay and any substances carried thereby.

13. The herein described process of purifying a vegetable oil, which comprises: heating the oil; intimately but gently mixing approximately 10% of water with said oil until a "break" occurs separating the oil from the water and substances carried thereby; heating said oil to a temperature of approximately 210° F. to 220° F.; adding an activated clay to said oil in such a manner as to produce an intimate mixture of said oil and said clay; and filtering under vacuum from said oil the clay and any substances carried thereby.

14. The herein described process of purifying a vegetable oil, which comprises: heating the oil to an initial temperature not to exceed approximately 110° F.; intimately mixing approximately 10% of warm water with said oil until a "break" is produced; separating the oil from the water and substances carried thereby; heating said oil to a temperature of approximately 210° F. to 220° F.; adding an activated clay to said oil in such a manner as to produce an intimate mixture of said oil and said clay; and separating from said oil the clay and any substances carried thereby.

15. The herein described process of purifying a vegetable oil, which comprises: heating the oil to an initial temperature not to exceed approximately 110° F.; intimately mixing approximately 10% of water with said oil until a "break" occurs; separating the oil from the water and substances carried thereby; heating said oil to a temperature of approximately 210° F. to 220° F.; adding an activated clay to said oil at any time after the temperature of said oil has reached 170° F. in such a manner as to produce an intimate mixture of said oil and said clay; and separating from said oil the clay and any substances carried thereby.

In testimony whereof, I affix my signature.

WALTER S. BAYLIS.