UNITED STATES PATENT OFFICE.

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PROCESS OF REFINING CRUDE COTTON-SEED OIL.

1,010,017. Specification of Letters Patent. Patented Nov. 28, 1911.

No Drawing. Application filed February 27, 1911. Serial No. 610,977.

To all whom it may concern:

Be it known that I, JESSE C. CHISHOLM, a citizen of the United States, residing at Dallas, in the county of Dallas and State of Texas, have invented certain new and useful Improvements in Processes of Refining Crude Cotton-Seed Oil, of which the following is a specification.

This invention relates to processes of refining oil, and particularly to a process of refining crude cotton-seed oil.

In the process of refining oil by the caustic soda method, the alkali employed not only neutralizes the fatty acids present but combines with a material quantity of the oil under treatment, saponifying it, thereby occasioning a considerable loss of oil.

I have discovered that if the acidity of the oil is overcome prior to the addition of the alkaline hydroxid thereto, the saponification of the oil by such hydroxid is in a very large measure avoided, the loss of oil due to saponification, being overcome.

In the practice of my process in its preferred form, I neutralize the oil by use of reagents which will combine with the fatty acids present without effecting the saponification of the oil, such non-oil-saponifying alkaline reagents reacting only with the constituents of the oil which it is desired to remove and not with the oil itself.

The non-oil-saponifying alkaline reagents which I prefer to employ are metal substituted bases, that is to say, compounds formed by replacing the hydrogen of an alkaline hydroxid with another metal, such for example, as sodium aluminate, sodium ferrite, and sodium zincate. These reagents not only neutralize the oil without materially effecting the saponification thereof but as a result of their reaction with the fatty acids present, produce metallic hydroxids which are particularly effective in lessening the subsequent action of alkaline hydroxid on the free oil and in removing the coloring matters and other impurities from the oil. The addition of metal substituted bases wherein a metal has replaced hydrogen, as the first step, is to neutralize the free fatty acidity of the crude oil (to lower saponification later by the caustic) together with the precipitate of aluminum hydroxid in the neutral oil. The presence of this precipitate aluminum hydroxid, on the addition of the caustic soda reunites with a portion of the same to form the original substituted base while any excess acts as free caustic soda, as on a neutral oil containing no precipitated material.

The non-oil-saponifying reagent, as for example, sodium aluminate, is added to the crude oil until the acidity of such oil is overcome, the mixture being neutral or slightly alkaline, as may be determined by tests with litmus or by the taste of the oil. The mixture is agitated preferably at an elevated temperature of from 75° to 90° F. until a perfectly homogeneous mixture of the oil and alkaline reagent is effected. There is then gradually added to the mixture from two to eight per cent. by weight, of a solution of alkaline hydroxid, preferably sodium hydroxid of from 12° to 22° Baumé. This alkaline hydroxid solution is added until a filtered sample of the mixture shows an oil of the required color.

Having obtained oil of the required color the addition of the alkaline hydroxid is continued and tests are made from time to time upon removed samples, by heating such samples to preferably from approximately 120° to 140° F., the alkaline hydroxid solution being added until such samples are found to have reached the "critical point", that is to say, the point at which the soap stock precipitate in the sample will settle rapidly and completely from the oil and be practically free from obtained oil. When this point is reached no further addition of the alkaline hydroxid is made. The oil is stirred or otherwise agitated while being treated with the alkaline hydroxid. The mass of treated oil is then heated to preferably from 100° to 120° F., the mixture being agitated during such heating. The heating and agitating are continued until the soap stock particles agglomerate and a test of a removed sample of the oil shows that the soap stock therein will settle rapidly and completely from the refined oil. The oil is then allowed to

stand preferably from two to twenty-four hours until the soap stock has completely settled. The supernatant oil is then drawn off and heated to preferably about 150° F. to free the same from water. If desired, air may be pumped through the refined oil to accelerate the drying operation. The refined oil is then filtered.

I have found that improved results may be obtained in the practice of my process by subjecting the crude oil to a preliminary treatment, consisting in heating it to about 80° F. and subjecting it to agitation prior to the addition of the metallic substituted base thereto. When the oil is subjected to this preliminary treatment such metal substituted base is preferably added immediately after such treatment without allowing the oil to cool, the remainder of the process being carried out exactly as hereinbefore set forth. When the preliminary treatment is omitted the crude oil may be heated either before or after the addition of the metal substituted base. I have also found that the treatment of crude cotton-seed oil with a metal substituted base alone, particularly when the oil is heated and agitated, improves the character of such oil materially.

While in the practical operation of my process I prefer to heat the oil as indicated, some measure of success may be obtained without subjecting the oil to artificial heat. In summer a considerable measure of success may be obtained throughout the southern States without any application of artificial heat. At other seasons of the year and in other sections of this country some measure of success may be obtained by prolonged treatment with the reagents herein set forth, without any application of heat, whatever, but the practice of my process can be carried out most effectively under the conditions of temperature hereinbefore set forth.

In two separate applications for processes of refining crude cotton-seed oil, designated as Cases A and C, and filed concurrently herewith I have described and claimed, first, a process of overcoming the acidity of the crude oil and subsequently treating the neutral or slightly alkaline oil with an alkaline hydroxid, (Case A Serial No. 610,976,) and second, a process of overcoming the acidity of the oil by the use of an alkaline silicate, and subsequently treating the neutral or alkaline oil with an alkaline hydroxid (Case C, Serial No. 610,978). The first mentioned process is not broadly claimed herein, and the last named process is not specifically claimed herein.

Having thus described my invention, I claim:—

1. The process of refining crude cotton-seed oil, which consists in subjecting it to the action of a metal substituted base wherein a metal has replaced hydrogen until the fatty acids present are neutralized, then to the action of an alkaline hydroxid and separating the oil from the precipitate formed.

2. The process of refining crude cotton-seed oil, which consists in subjecting it to the action of heat and a metal substituted base, wherein a metal has replaced hydrogen, then to the action of heat and an alkaline hydroxid, and separating the oil from the precipitate formed.

3. The process of refining crude cotton seed oil, which consists in adding a metal substituted base wherein a metal has replaced hydrogen, agitating the mixture at an elevated temperature, adding an alkaline hydroxid thereto, agitating the mixture at an elevated temperature, and separating the oil from the precipitate formed.

4. The herein described process of refining crude cotton-seed oil, which consists in adding an aqueous solution of a metal substituted base wherein a metal has replaced hydrogen until the fatty acids present in the oil are neutralized, agitating the mixture at an elevated temperature, adding an aqueous solution of an alkaline hydroxid, agitating the mixture at a temperature of from approximately 75° to 90° F., maintaining the temperature of the oil until a test sample of the mixture shows the required color, adding additional alkaline hydroxid until a test sample of the mixture removed and heated to approximately 120° to 140° F. the soap stock precipitate separates rapidly and completely from the oil, then heating the mass of treated oil to approximately from 100° to 120° F., such heating being continued until the soap stock will settle rapidly and completely from the oil, allowing soap stock to settle, and separating the oil from such soap stock.

5. The process of refining crude cotton-seed oil, which consists in heating it to a temperature of approximately 80° F. while agitating the same, adding a solution of a metal substituted base wherein a metal has replaced hydrogen, agitating the mixture at an elevated temperature, adding an alkaline hydroxid, agitating the mixture at an elevated temperature, and separating the oil from the precipitate formed.

6. The process of refining crude cotton-seed oil, which consists in subjecting it to the action of a non-oil-saponifying basic compound then to the action of an oil saponifying base, and separating the oil from the precipitate formed.

7. The process of refining crude cotton-seed oil, which consists in subjecting it to the action of heat and a non-oil-saponifying basic compound then to the action of an oil-saponifying base, and separating the oil from the precipitate formed.

8. The process of refining crude cotton-seed oil, which consists in subjecting it to the action of a non-oil-saponifying basic compound until the fatty acids present are neutralized, then to the action of an oil-saponifying base, and separating the oil from the precipitated formed.

In testimony whereof I affix my signature in presence of two witnesses.

JESSE C. CHISHOLM.

Witnesses:
C. W. PARKER,
B. P. FISHBURNE.