UNITED STATES PATENT OFFICE.

JESSE C. CHISHOLM, OF DALLAS, TEXAS.

PROCESS OF REFINING CRUDE COTTON-SEED OIL.

1,056,264. Specification of Letters Patent. Patented Mar. 18, 1913.

No Drawing. Application filed October 26, 1911. Serial No. 656,813.

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 has particular reference to an improved process of refining crude cotton seed oil.

It is the purpose of the invention to increase the yield of refined oil of the required color, odor and flavor.

In Letters Patent No. 1,010,017, for a process of refining crude cotton seed oil, granted to me under date of November 28, 1911, I have described and claimed a process of refining cotton seed oil by subjecting it to the action of a metal substituted base wherein a metal has replaced hydrogen, then to the action of an alkaline hydroxid and separating the oil from the soap stock formed. In Letters Patent No. 1,007,642, for a process of refining crude cotton seed oil, granted to me under date of October 31, 1911, I have described and claimed a process of refining cotton seed oil which consists in subjecting the oil to the action of an alkaline silicate until the acidity of the oil is overcome, then subjecting it to the action of an alkaline hydroxid and finally separating the oil from the soap stock formed. In still another application for a process of refining crude cotton seed oil Serial No. 656,811, filed October 26, 1911, I have described and claimed a process which consists in subjecting the oil to the action of an alkaline silicate and an alkaline carbonate until the acidity of the oil is approximately overcome, then subjecting it to the action of an alkaline hydroxid and finally separating the oil from the soap stock formed. Now I have discovered that in some cases advantageous results may be obtained by first treating the oil with an alkaline silicate mixed with an alkaline carbonate or hydroxid until the oil is approximately neutralized then subjecting it to the action of a mixture of alkaline hydroxid and a metal substituted base wherein a metal has replaced hydrogen and separating the oil from the soap stock formed.

In the practice of my process crude cotton seed oil is treated with an aqueous solution of a water soluble alkaline silicate preferably sodium silicate ($Na_2Si_4O_9$) together with an aqueous solution of an alkaline carbonate or hydroxid preferably sodium carbonate (Na_2CO_3) until the acidity of the oil is approximately overcome. The proportions of the alkaline silicate to the alkaline carbonate or hydroxid in the mixture may vary widely. I have found however that satisfactory results may be obtained by employing a mixture of equal parts by weight of an aqueous solution of alkaline silicate of approximately 40° Baumé and an aqueous solution of an alkaline carbonate of approximately 30° Baumé. The mixture is added to the oil until the latter is approximately neutralized. It may, without interfering with the results desired, be added until the oil is slightly alkaline. This ordinarily requires from one to three parts by weights of the alkaline mixture to one hundred parts by weight of crude oil. After the solution is added to the oil the mixture is stirred or otherwise agitated at a temperature of from approximately 75° to 90° F., the agitation being continued and the temperature maintained until the oil "grains" from the separation of soap stock and the yellow color of the foam on the oil changes to a darker shade. This effect takes place when the fatty acids contained in the oil are neutralized by the action of the alkaline mixture which has been added thereto. There is then gradually added to the treated oil a mixture of an alkaline hydroxid, or carbonate, preferably sodium hydroxid, and a metal substituted base, such for example, as sodium aluminate, sodium ferrite, and sodium zincate, wherein the hydrogen of an alkaline hydroxid is replaced by another metal. The mixture referred to may advantageously comprise equal parts by weight

of an aqueous solution of sodium hydroxid of from 12° to 22° Baumé and an aqueous solution of sodium zincate of from 15° to 30° Baumé. It ordinarily requires from two to eight per cent. by weight of this mixture to satisfactorily treat the oil. The alkaline mixture is added until a filtered sample of the treated oil shows an oil of the required color. Having obtained oil of the required color the addition of the mixture 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 mixture being added until such samples are found to reach 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 contained oil. When this point is reached no further addition of the alkaline mixture is made. The oil is stirred or otherwise agitated while being treated with the mixture. The mass of treated oil is then heated to preferably from 100° to 120° F., and is 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.

While in the practical operation of my process I prefer to heat the oil as indicated, some measure of success in the practice of the process 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 set forth, without any application of heat whatever, but the practice of the process can be carried out most effectively under the conditions of temperature hereinbefore set forth.

While I have set forth in detail the strength of the solutions and the proportions of the various reagents which I prefer to employ, it is to be understood that my process is not restricted thereto, but that the strength of such solutions and the proportions may be widely varied, without departing from the spirit of my invention or the scope of the appended claims. This wide variation in the strength of solution and the proportions of the reagents is necessitated by reason of the widely varying characters of the crude cotton seed oils received for refining.

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 an alkaline silicate until the acidity of the oil is approximately overcome then subjecting it to the action of an alkaline compound and a metal substituted base wherein a metal has replaced hydrogen and separating the oil from the soap stock formed.

2. The process of refining crude cotton seed oil, which consists in subjecting it to the action of an alkaline silicate and another alkaline compound until the acidity of the oil is approximately overcome, then subjecting it to the action of an alkaline compound and a metal substituted base wherein a metal has replaced hydrogen and separating the oil from the soap stock formed.

3. The process of refining crude cotton seed oil, which consists in subjecting it to the action of an alkaline silicate and an alkaline carbonate until the acidity of the oil is approximately overcome, then subjecting it to the action of an alkaline hydroxid and a metal substituted base wherein a metal has replaced hydrogen and separating the oil from the soap stock formed.

4. The process of refining crude cotton seed oil, which consists in subjecting it to the action of heat and a mixture of an alkaline silicate and an alkaline carbonate until the acidity of the oil is approximately overcome, then subjecting it to the action of heat and a mixture of an alkaline hydroxid and a metal substituted base wherein a metal has replaced hydrogen and separating the oil from the soap stock formed.

5. The process of refining crude cotton seed oil, which consists in agitating the oil at a temperature of approximately from 75° to 90° F., a mixture of an alkaline silicate and an alkaline carbonate being added to the oil until the acidity of such oil is approximately overcome, maintaining the temperature of the mixture at approximately from 75° to 90° F., until the separation of the soap stock is apparent in the oil, adding an alkaline hydroxid and a metal substituted base wherein a metal has replaced hydrogen, maintaining the temperature of the oil until a test sample of the treated oil shows the required color, adding additional quantities of the last mentioned alkaline mixture until in a test sample of the mixture removed and heated to from approximately 100° to 140° F., the soap stock precipitates and separates readily and completely

from the oil, then heating the mass of treated oil to preferably from 100° to 120° F., such heating being continued until the soap stock settles rapidly and completely from the oil, allowing the soap stock to settle and separating the oil from the soap stock formed.

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

JESSE C. CHISHOLM.

Witnesses:
M. M. CRANE,
N. M. COOK.