

UNITED STATES PATENT OFFICE.

JESSE C. CHISHOLM, OF DALLAS, TEXAS.

PROCESS OF REFINING CRUDE COTTON-SEED OIL.

1,056,261.

Specification of Letters Patent.

Patented Mar. 18, 1913.

No Drawing.

Application filed October 11, 1911. Serial No. 654,048.

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, the process having its most important application to the treatment to crude cotton seed oil of high acidity, as for example oils containing from 1.5 to 12 per cent. acid or higher.

The principal object of the invention is to increase the yield of oil of the required color, odor and flavor.

In refining crude cotton seed oil by processes wherein soluble soap forming agents, as caustic alkalis, are employed, the alkali first added to the oil combines with the fatty acids present therein producing soluble soap in the oil. Upon the subsequent treatment of the oil with alkali there is a material loss of oil due to saponification. Now I have discovered that this loss of oil can be in a very large measure, overcome by first producing an insoluble soap with the fatty acids present in the oil and then treating the oil with caustic alkali. I have also discovered that the process is materially improved by the employment of an alkaline silicate in conjunction with the treatment specified. The formation of an insoluble soap in the crude cotton seed oil serves in a very large measure to prevent the loss due to saponification of oil, upon the addition of caustic alkali to the treated mass.

The insoluble soap-forming compounds which I employ may be any non-alkali metal oxid, hydroxid, phosphate, sulfate, sulfid, chlorid, or in fact any alkaline earth compound or other metallic compound, except compounds of the alkali metals, which will react with the organic acid or acids present in the oil, to produce an insoluble soap. The alkaline silicate may be added before, during or after the treatment of the oil with the insoluble soap forming agent.

In the practice of my process in its preferred form I determine the acidity of the crude oil to be refined. Having determined the amount of acid present in the oil I add thereto an insoluble soap forming agent in

approximately molecular proportion in respect of the acid present.

With the insoluble soap forming agent, which may advantageously be calcium or barium oxid or hydroxid I preferably add silicate of soda. The amount of silicate of soda to be added may vary widely. I have found that satisfactory results may ordinarily be obtained by adding the silicate of soda in an amount approximately equal to from 10 to 50 per cent. of the insoluble soap-forming agent employed. The silicate of soda and the insoluble soap forming agent may be added in aqueous solution, the solution being either saturated or diluted as preferred. After the insoluble soap-forming agent and silicate of soda have been added to the crude oil, preferably to the extent of approximately neutralizing it, the mixture is stirred or otherwise agitated at a temperature of approximately 75° to 90° F., the agitation being continued and the temperature maintained until the oil grains owing to the separation of soap stock. 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 110° to 120° F., the alkaline hydroxid solution 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 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.

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.

In a copending application for a process of refining crude cotton seed oil filed February 27, 1911, Serial Number 610,978 which application has now become Patent No. 1,007,642, issued October 31, 1911, I have

described and claimed a process consisting in treating the crude oil with an alkaline silicate until the acidity of the oil is approximately overcome, then subjecting it to the action of an alkaline hydroxid and separating the oil from the soap stock formed; and such process is not 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 an insoluble soap producing agent and an alkaline silicate until the acidity of the oil is approximately overcome, subjecting it to the action of an alkaline hydroxid, and separating the oil from the soap stock formed.

2. The process of refining crude cotton seed oil, which consists in reacting upon the fatty acids present to produce an insoluble soap therefrom while heating and agitating the oil at a temperature of approximately from 75° to 90° F., subjecting it to the action of an alkaline hydroxid 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 insoluble soap producing agent and an alkaline silicate while agitating the same at a temperature of approximately 75° to 90° F., until the oil grains, then subjecting it to the action of an alkaline hydroxid while heating and agitating, and separating the oil from the soap stock formed.

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

JESSE C. CHISHOLM.

Witnesses:

M. M. CRANE,
N. M. COOK.