UNITED STATES PATENT OFFICE.

EUGENE E. AYRES, JR., OF CHESTER, PENNSYLVANIA, ASSIGNOR TO THE SHARPLES SPECIALTY COMPANY, OF WEST CHESTER, PENNSYLVANIA, A CORPORATION OF PENNSYLVANIA.

PROCESS WHEREBY NEUTRAL OILS CAN BE PROFITABLY RECOVERED FROM THEIR "FOOTS" OR SOAP-STOCK.

1,247,782. Specification of Letters Patent. Patented Nov. 27, 1917.

No Drawing. Application filed May 21, 1917. Serial No. 169,899.

To all whom it may concern:

Be it known that I, EUGENE E. AYRES, Jr., a citizen of the United States, residing at Chester, in the county of Delaware and State of Pennsylvania, have invented a certain new and useful Process Whereby Neutral Oils Can be Profitably Recovered from Their "Foots" or Soap-Stock.

My invention comprises, generally stated; first, the preliminary treatment of the "foots" to reduce the stability of the emulsion; second, the centrifugal separation of the treated emulsion into two portions, one (A) containing most of the neutral oil and some water and soap, the other (B) containing most of the water and soap with a little oil; and, third, the chemical treatment of the emulsion (A) discharged from the centrifuge in order to separate the oil in sufficiently neutral condition to be economically refined to neutrality.

The invention has for its primary object to provide a simple, efficient and economic method of recovering the neutral oil from the soap with which it is emulsified.

In the generally practised methods of refining oils, the crude oil is treated with a solution of caustic soda. The effect of this treatment is to neutralize the free fatty acids in the crude oil with the formation of soap, and to dissolve the impurities in the crude oil in the alkaline water. When the soap is precipitated in this way, a certain percentage of the neutral oil is usually emulsified and carried down with the soap and water. Heretofore this oil has not been recovered in its neutral state. The "foots" thus formed are sometimes sold as such to the soap makers but more often the soap emulsion is acidified to recover the fatty acids. In the latter case, the oil mixed with the fatty acids is sold simply as soap stock, as the oil cannot be profitably recovered from the high percentages of fatty acids. In other cases, the "foots" are treated in such a way as to recover glycerin from the oil. Where the percentage of oil in the "foots" is high, the refiner of vegetable oils has suffered considerable loss heretofore. A number of attempts have been made to recover this neutral oil by centrifugal methods, but the trials have not been commercially successful because of the high stability and the high viscosity of the emulsion.

I have found that by certain operations and reactions affecting the emulsifying agents it is possible by means of centrifugal force to separate the "foots" into two portions, one (A) of which will contain most of the oil, the other (B) containing most of the water and soap. Furthermore, I have found it possible to chemically break the emulsion (A) discharged from the machine because of the absence or changed character of emulsifying agents in the emulsified water.

There are, broadly speaking, two types of reactions that will affect the emulsifying agents in such a way as to reduce the stability of the emulsion. The first reaction will so change its chemical constitution that the emulsifying agents lose their property of stabilizing the emulsion. I have found that in most cases the interaction of substances already in solution will serve to bring about this first reaction when the "foots" are diluted with water. The rapidity of the reaction is hastened by the application of heat. The yield of oil recovered by such a procedure is sufficiently high in most cases for commercial profit.

But I have found that the yield of oil can be increased greatly in the majority of cases by assisting this reaction by the addition of chemical reagents. It is essential to select some reagent that will not salt the soap out of solution and will not decompose the soap to form fatty acids. I have found that a good type of reagent for the purpose is any alkali or alkaline salt. A convenient alkaline salt is normal sodium carbonate, which has the advantage of causing a minimum saponification loss of neutral oil. The effect of this first reaction to alter the chemical constitution of the emulsifying agent can be noted by (1) a reduction in viscosity, (2) a tendency to separate by gravity into two layers, and (3) ease of centrifugal separation.

A second reaction can be obtained which will partially or completely transpose the emulsifying agent from solution in the water to solution in the oil. I have found that the addition of any acid will reduce the

solubility of the emulsifying agent in water and increase the solubility in the oil. I have found that boric acid has a minimum effect on the soap while it is efficacious in causing this second reaction, thereby reducing the stability of the "foots" emulsion. However, if it is desired to recover the oil in its neutral state (not dissolved in fatty acids) it is essential that the soap be not decomposed.

Both of the reactions described are aided by the application of heat.

In carrying out the invention, I proceed substantially as follows: The "foots" from the refining kettle are diluted with from one-half to two parts of water (more or less water may affect the ease of centrifugal separation). The diluted mixture is then heated, preferably to boiling, after the addition of 2% of normal sodium carbonate or 2% of boric acid. When the sodium carbonate is used, I prefer to allow the hot liquid to settle for about one hour to permit a partial gravity separation, with formation of a lower layer containing very little oil, which can be run off before centrifuging the remainder, thus reducing the volume of liquid to be handled by the centrifuge. When boric acid is used, the period of settling is of no great advantage as there will be no very distinct gravity separation.

The invention should be understood as not being limited to the above proportions of the chemicals used, and, as indicated, the use of sodium carbonate or boric acid is not essential to the preliminary treatment of the emulsion, although I have found that the use of these reagents causes a higher yield of oil in every case and a greater uniformity of results. Heat is not essential although it is an advantage.

The next step in the process is to pass the treated "foots" emulsion through a centrifugal separator. The lighter portion will be an oil emulsion containing, as a rule, some water and soap (emulsion A). The heavier portion will contain most of the water and soap with a little oil (emulsion B). The efficiency of the centrifugal separation is dependent upon the thoroughness of the preliminary treatment, and, as indicated, the yields of oil will be higher when the reagents are used to affect the emulsifying agents.

The emulsion of oil and water (A) discharged from the centrifugal separator can be rendered unstable readily by any one of the procedures mentioned below:

1. Salting the soap out of solution.
2. The addition of an electrolyte.
3. Freezing and remelting.
4. Acidifying with an acid such as sulfuric acid.

The emulsion treated in this way can then be separated either by gravity or by a centrifugal separator.

In salting the soap out of solution, the emulsion discharged from the centrifugal machine is heated with gentle stirring and say 1% common salt is added, though different amounts may be used. When the salt has dissolved, the emulsion is permitted to stand hot until the separation is complete. While the soap can be salted out by means of substances other than common salt, I prefer the latter because of its low cost.

As a specific example of the operations and results of my invention, the following is submitted: To 100 pounds of "foots" was added 150 pounds of water and 4 pounds of sodium carbonate in solution. The mixture was boiled for five minutes and settled one hour. Then 73 pounds of soap solution were drawn off from the bottom. The liquid remaining in the tank was thoroughly mixed and passed through the centrifugal machine at the rate of 1280 pounds of water and 152 pounds of emulsion per hour. The water run off from the tank after settling contained 3% neutral oil. The water discharged from the machine contained 2.57% neutral oil. The emulsion discharged from the machine contained 71% neutral oil. The original soap stock contained 25% neutral oil. The emulsion from the machine was broken by the addition of common salt and then separated by gravity. The separated oil contained about $\frac{1}{10}$ of 1% acidity. After refining by the usual method, the oil was pronounced of good marketable quality. The weight of recovered oil was 15.5 pounds. In some of my experiments, I have recovered as high as 95% of the oil originally present in the "foots."

The above operations and results in the specific example given were with relation to the "foots" obtained from cotton seed oil obtained from the ordinary caustic soda treatment.

Having described my invention, I claim:

1. In the recovery of neutral oil from its "foots", the process which comprises diluting the "foots" with water so as to reduce the stability of the "foots" emulsion, separating by centrifugal action a part containing the bulk of the oil and a part containing the bulk of the water and soap, treating the part containing the bulk of the oil so as to reduce the stability of the emulsion, and separating out the oil from the last named product.

2. In the recovery of neutral oil from "foots", the process which comprises adding water to the "foots" and heating, subjecting the product to centrifugal action with separation of a part containing the bulk of the oil, treating said part with a chemical reagent capable of reducing the stability of the emulsifying agents found with the oil, and separating the oil from the liquid in which it is contained by means of its lower gravity.

3. In the recovery of neutral oil from "foots", the process which comprises the addition to the "foots" of a chemical reagent adapted for reducing the stability of the emulsifying agents, separating from the product a part containing the bulk of the oil and a part containing the bulk of the water and soap, treating the part containing the bulk of the oil with a chemical reagent adapted for breaking the emulsion, and separating oil from the latter product by means of differences in specific gravity.

4. In the recovery of neutral oil from "foots", the process which comprises the addition to the "foots" of water and a chemical reagent for reducing the stability of the emulsifying agents, separating from the product by means of differences in specific gravity a part containing the bulk of the oil, and salting the part containing the bulk of the oil.

5. In the recovery of neutral oil from "foots", the process which comprises the addition to the "foots" of water and a chemical reagent adapted to reduce the stability of the "foots" emulsion and heating, separating the bulk of the soap solution and the bulk of the oil of the product by means of their difference in specific gravity, subjecting the bulk containing the oil to centrifugal action, and adding salt to the oil emulsion separated by the centrifugal action.

6. In the recovery of neutral oil from "foots", the process which comprises the addition to the "foots" of more than half its weight in water and a chemical reagent for reducing the stability of the "foots" emulsion and heating, settling the heated product and separating the bulk of the soap solution from the bulk of the oil, subjecting the solution containing the bulk of the oil to centrifugal action for separation of the bulk of the water and soap from the bulk of the emulsified oil, treating the emulsified oil with salt, and separating the oil from the salted product by gravity.

In testimony whereof I have hereunto set my name this 19th day of May, 1917.

EUGENE E. AYRES, JR.