Dec. 18, 1945. B. CLAYTON 2,390,990

PROCESS OF REFINING OIL AND FOR PRODUCING SOAP

Filed Aug. 15, 1941 3 Sheets-Sheet 1

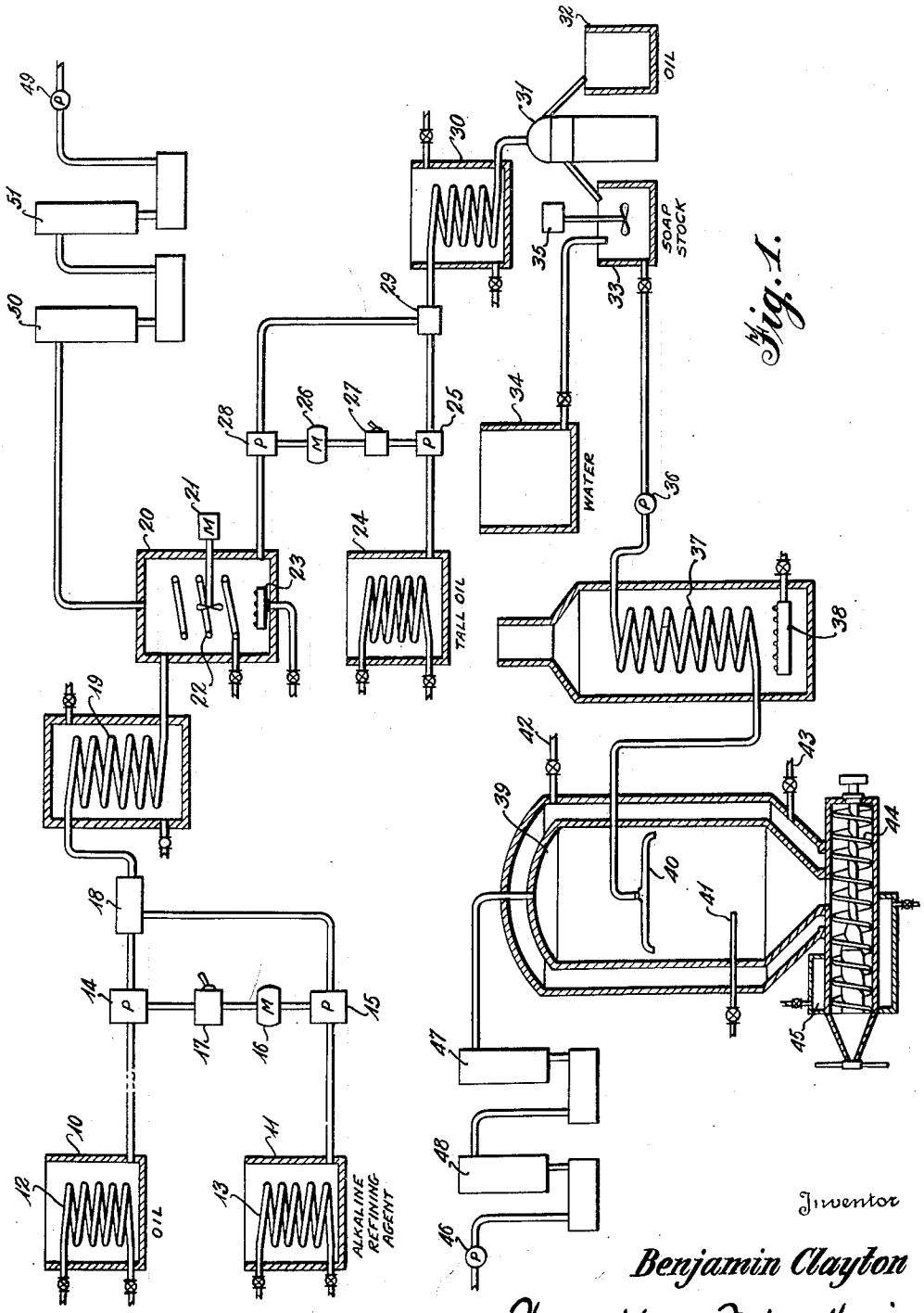



Fig. 1.

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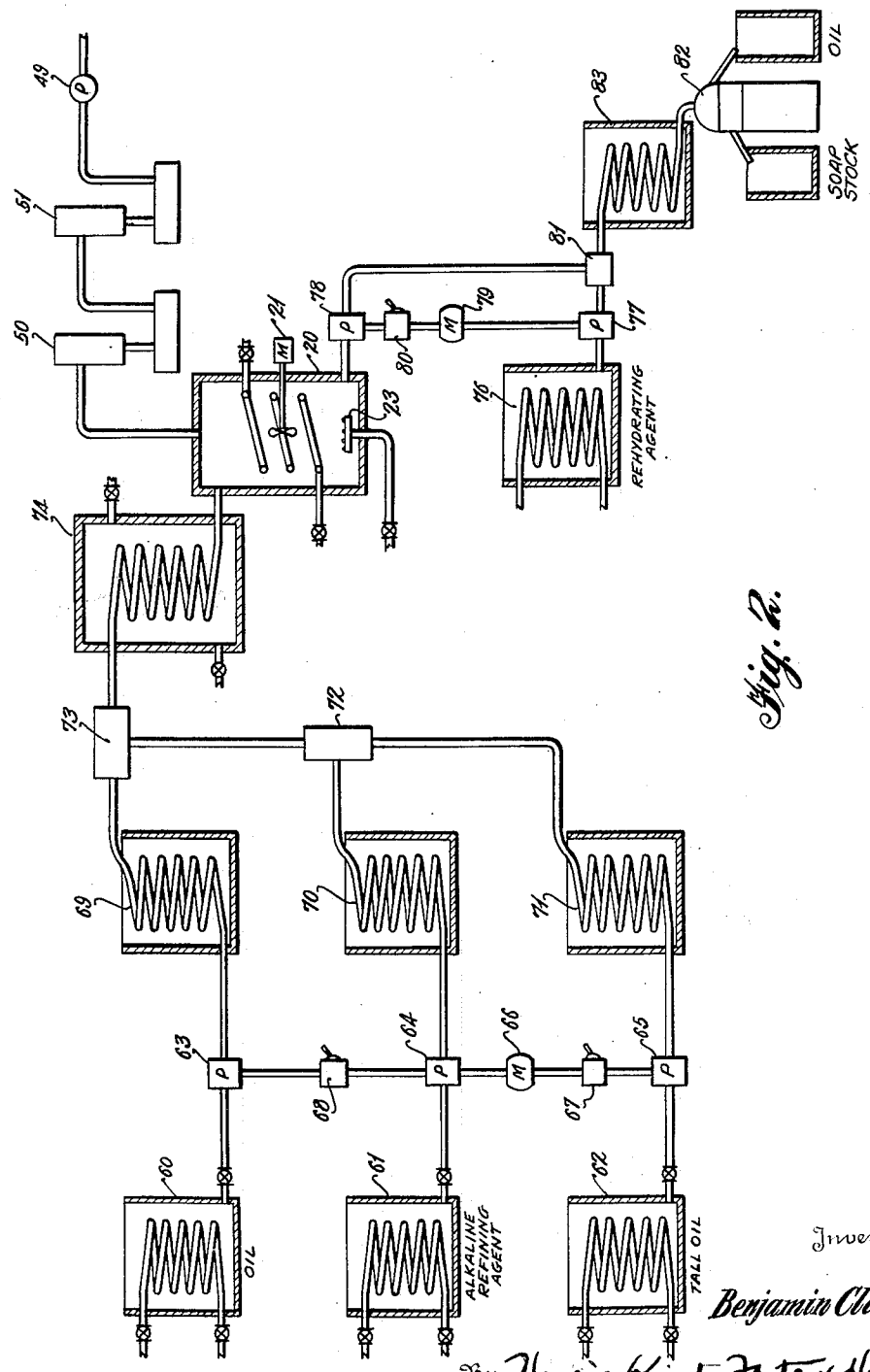



Fig. 2.

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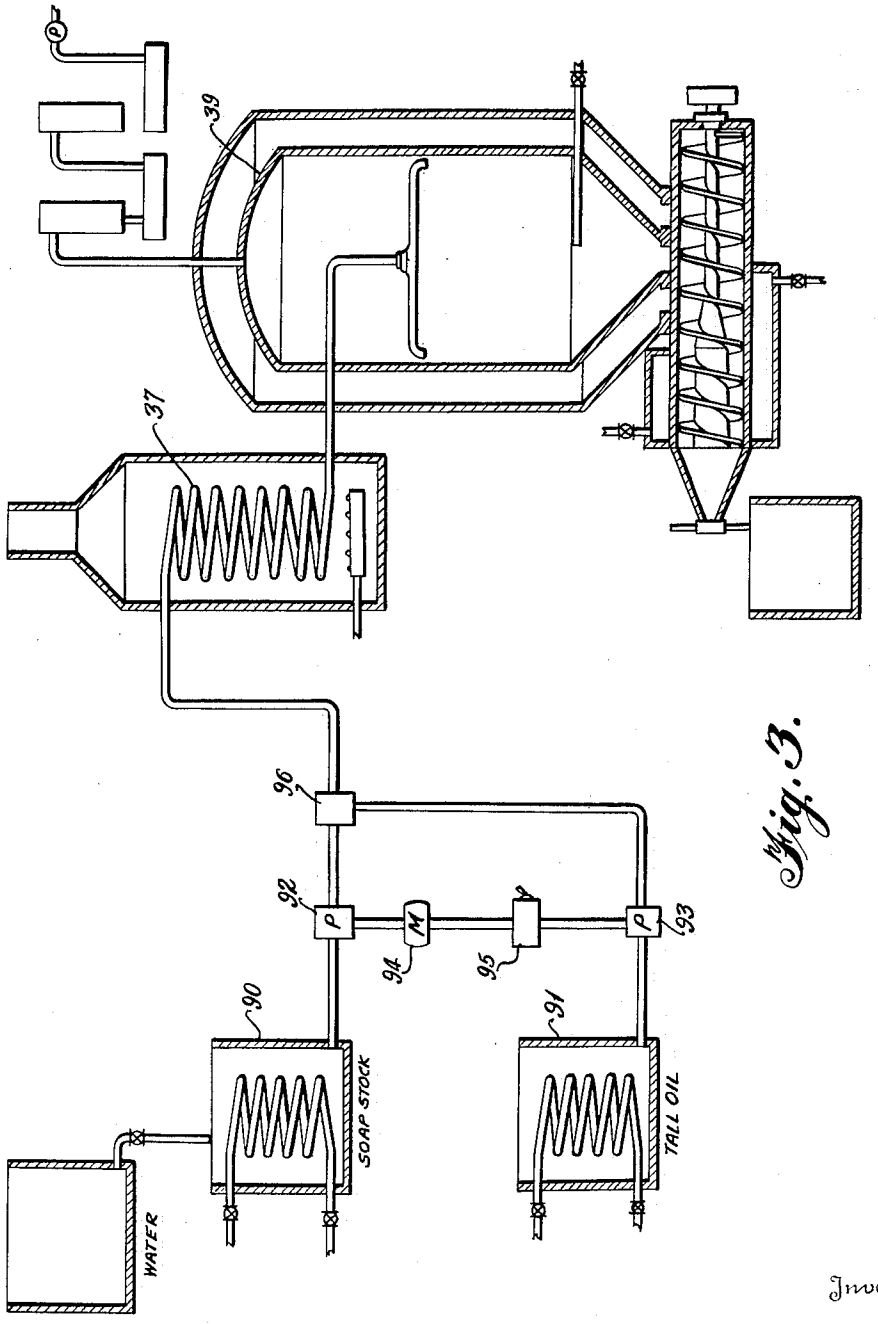



Fig. 3.

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Patented Dec. 18, 1945

2,390,990

# UNITED STATES PATENT OFFICE

2,390,990

## PROCESS OF REFINING OIL AND FOR PRODUCING SOAP

Benjamin Clayton, Houston, Tex., assignor, by mesne assignments, to Refining, Unincorporated, a Texas partnership

Application August 15, 1941, Serial No. 407,081

12 Claims. (Cl. 260—425)

The present invention relates to a process of refining oil such as animal or vegetable oil and for simultaneously producing a novel soap product containing tall oil soap.

In the liberation of cellulose fibers from wood by digestion of the wood with acid or alkali, a waste liquor is produced which contains an oily, resinous material. This material is composed chiefly of a mixture of fatty and rosin acids, together with some unsaponifiable material. It is dark colored and has a strong objectionable odor. The term tall oil as used herein is intended to refer to this material when in an acid condition, that is, when it contains free fatty and rosin acids. The term tall oil soap is used herein to designate soaps produced by the saponification of these fatty and rosin acids by means of an alkali. These two terms are intended to designate the material when in the particular condition referred to without regard to whether the material was originally derived from an acid digestion process such as a sulphite process, or from an alkaline digestion process such as a sulphate process.

Large quantities of this material have been available in commerce, but only relatively small quantities thereof have actually been used for detergent purposes because of the numerous difficulties encountered in making a satisfactory detergent from this raw material. Crude tall oil soaps have been of little detergent value. Even tall oil soap produced from oil from which the unsaponifiable material has been removed have been found unsatisfactory because of their extreme stickiness. This, together with the dark color and objectionable odor, has resulted in an extremely limited use of the soap. Furthermore, tall oil soaps produced at present are unstable and deteriorate unless used promptly after being made.

The present invention provides a method whereby tall oil may be converted into a valuable detergent product and may at the same time assist in a process of refining animal and vegetable oils. The tall oil soap resulting from the present invention is relatively light colored and odorless, and is not sticky. Furthermore, it is relatively stable. The present invention also provides for the production of a detergent containing a mixture of tall oil soap and fatty acid soaps which materially improves the ordinarily inferior detergent properties of the tall oil soap.

It is therefore an object of the invention to provide a method for converting tall oil into a purified detergent product.

Another object of the invention is to provide a method of producing a detergent product containing tall oil and fatty acid soaps.

A further object of the invention is to provide a method of producing an improved detergent product containing tall oil and soap stock.

Another object of the invention is to provide a tall oil detergent containing phosphatides in sufficient quantity to improve the detergent characteristics of the tall oil soap.

A still further object of the invention is to provide a method of refining animal and vegetable oils in which tall oil facilitates the separation of soap stock from the neutralized oil.

These and other objects of the invention will be apparent to those skilled in the art from a description of the drawings in which:

Fig. 1 represents a preferred embodiment of the invention wherein tall oil is employed to assist in the separation of soap stock from neutralized oil in a glyceride oil refining process and wherein the tall oil and the soap stock are converted into a purified product; and

Figs. 2 and 3 represent modifications of the process of Fig. 1.

With reference to Fig. 1, 10 represents a source of supply for the oil to be refined and 11 represents a source of supply for an alkaline refining agent. The temperature of the oil and of the refining agent may be preliminarily adjusted to any desired temperature by means of coils 12 and 13, respectively. Pump 14 driven by motor 16 through the variable speed device 17, and pump 15 also driven by motor 16, withdraw proportioned streams of oil and refining agent from the respective supply tanks and force them into the mixing device 18 wherein the two reactants are intimately mixed in order to assure complete reaction. The refining agent reacts with the free fatty acids present to form soap stock. In addition, other impurities such as gums, phosphatides, and the like, if present in the oil being treated, are also precipitated. The mixture of oil and soap stock may then be passed through a heating coil 19 in which sufficient heat may be added to vaporize various impurities, including water or other solvent employed for the refining agent, upon discharge of the mixture into a lower pressure zone. The temperature of the mixture leaving coil 19 will usually range between 140 and 230° F. The heated mixture can then be discharged into dehydration chamber 20 which is maintained under a lower pressure than the coil 19 and which preferably is maintained at a high vacuum. The mixture of oil and

soap stock may be completely dehydrated in chamber 20 or any desired portion of the water or impurities may be removed. The mixture of oil and soap stock may be agitated by means of agitator 21 so as to cause all portions of the mixture to become uniformly dehydrated. Additional heat may be supplied to the mixture by means of the coil 22. The vaporization of impurities may be assisted by introducing steam through distributor 23. Introduction of the steam also assists in the agitation so that agitator 21 may be completely or partially dispensed with. The temperature in the dehydration chamber may vary with the material treated as well as with the results desired. Ordinarily the temperature will range between 160 and 250° F.

In order to prepare the dehydrated mixture for centrifugal separation, it is preferred to add a rehydrating medium. It has been found that rosin soap, such as tall oil soap, and also naphthenic acid soap may be used to advantage in rehydrating the dehydrated mixture. A stream of such rehydrating agent may be withdrawn from tank 24 by means of pump 25, driven from motor 26 through the variable speed device 27. A proportioned stream of the dehydrated mixture may be withdrawn from the chamber 20 by means of the pump 28 also driven by motor 26. These proportioned streams may be mixed in mixer 29 and may then be forced through the heat exchange device 30 in order to regulate the temperature to that suitable for centrifugal separation. The temperature for separation may vary depending on the particular mixture being separated but will generally be between 100 and 250° F. The thus prepared mixture is then subjected to separation in centrifuge 31 to yield a refined oil which is discharged into tank 32 and a soap stock which is discharged into tank 33. The oil may be withdrawn for further purification such as washing, drying, etc., according to conventional practice.

It will be found that the soap stock thus separated from the oil may contain considerable quantities of impurities. The treatment in chamber 20 will remove some of the impurities, but in the event that a highly purified product is desired, further purification may be found necessary, particularly where crude or unrefined tall oil is employed. If the soap stock in tank 33 does not contain sufficient water to make a flowable mixture, additional water or other viscosity reducing substance may be withdrawn from tank 34 and mixed with the soap stock. An agitator 35 serves to maintain the mixture within tank 33 relatively uniform. A stream of the soap stock may be withdrawn by means of pump 36 and forced through coil 37 heated by burner 38. Preferably, sufficient heat is added in coil 37 to vaporize substantially all the vaporizable impurities upon the reduction of pressure. The thus heated stream may then be discharged into vapor separating chamber 39, preferably maintained under vacuum, through rotary nozzle 40. This nozzle may spray the heated soap stock in a thin film along the walls of chamber 39. Thus a large surface area will be presented to facilitate the removal of undesirable vaporizable materials. It is preferred to remove substantially all vaporizable impurities within chamber 39. Moreover, the temperature within this chamber is preferably sufficient to maintain the resulting substantially anhydrous soap molten. A steam pipe 41 may be provided to assist in the distillation of the impurities. Chamber 39 may likewise be provided with a jacket through which a heat exchange medium may be circulated by means of pipes 42 and 43. The molten anhydrous soap falls into the bottom of the chamber and may be withdrawn by means of the screw conveyor 44 which may be of the type disclosed in U. S. Patent No. 2,142,984 to Thurman. A cooling jacket 45 may be provided to remove sufficient heat to cause the soap to be solidified. Thus a seal may be formed which prevents the entrance into chamber 39 of air, which would deleteriously affect the soap at the elevated temperatures employed. Furthermore, the solidified soap is subjected to a milling or plodding action which results in the production of a relatively uniform product. The solidified soap may be discharged from conveyor 44 or it may be extruded or otherwise converted into some other desirable form.

Vapors liberated in chamber 39 are withdrawn by means of vacuum pump 46 and passed through condensers 47 and 48, wherein they may be fractionally condensed to partially separate the vapors into the respective constituents. The vapors liberated in chamber 20 may likewise be withdrawn by means of vacuum pump 49 through condensers 50 and 51.

The oil employed in carrying out the process may be crude animal or vegetable oils or may be partially refined, such as wholly or partially degummed oil. The process is particularly adapted to the treatment of degummed oils for the reason that the oil and soap stock produced in the refining of a preliminarily degummed oil have a tendency to stratify into three layers, an oil layer, a soap layer and an intermediate layer containing both oil and soap. In separating such a mixture it will be found that the soap stock will contain substantial amounts of oil or the oil will contain substantial amounts of soap stock. The tall oil soaps as well as naphthenic acid soaps and rosin soaps in general prevent this stratification. The dehydration treatment also is beneficial in this respect.

The alkaline refining agent employed may be any agent which will react with free fatty acids to form soaps. It may consist of a caustic alkali such as sodium or potassium hydroxide or it may consist of an alkaline salt such as soda ash, sodium phosphate, sodium silicate, or the corresponding potassium or alkaline earth salts and the like. In the use of soda ash large excesses such as from three to twelve times the amount theoretically required may be employed. The excess soda ash acts as a softening agent and aids in separation. When such a large excess is employed, the material withdrawn from tank 24 may consist of tall oil as distinguished from the use of preformed tall oil soaps. That is, the excess of soda ash employed may be relied upon to convert the acidic tall oil from tank 24 into soap before the mixture is separated in centrifuge 31.

In view of the fact that some of the impurities are removed in chamber 20 it will be apparent that the subsequent purification in chamber 39 may not be necessary where only a partially purified product is desired. Where, however, a high quality product is desired, the purification treatment in chamber 39 may be employed.

The soap product produced by the process contains a mixture of tall oil soaps together with fatty acid soaps. The product is extremely soluble as compared with a straight tall oil soap and in addition has had more of the odor producing compounds destroyed or volatilized and has

a materially improved color. The detergent properties of the fatty acid soap complement the high solubility and low sensitivity to graining out and resistance to jelling which are the characteristics of a straight tall oil soap. It has been found that this product may be further improved by the incorporation of a small amount of phosphatides. The emulsifying properties of phosphatides, particularly those derived from corn and cottonseed oil, contribute considerably to the detergent properties of the tall oil soap. Even small quantities of phosphatides added to a straight tall oil soap improve its properties over the untreated tall oil soap. It is preferred, however, to employ the phosphatides in a soap product containing both fatty acid soaps and tall oil soaps.

Crude vegetable oils such as corn and cottonseed oil contain phosphatides. In employing the present process on such crude oils some of the phosphatides will be found in the soap stock resulting from the refining operation. However, high temperatures and caustic alkalies deleteriously affect phosphatides and the quantity of this material finally found in the soap stock will depend upon the extent to which the phosphatides have been destroyed. In a process in which a mild alkali is employed and in which the temperatures used are not unduly elevated, it will be found that the soap stock resulting will contain appreciable quantities of phosphatides. It is preferred, however, to add phosphatides to the soap product as the same passes through the cooling section of the conveyer **44**. In this way the milling action of the conveyor may assist in distributing the phosphatides uniformly throughout the mixture. The phosphatides to be thus incorporated in the soap product may be derived from any source. A suitable method of securing phosphatides is to preliminarily degum the oil in any conventional manner, which precipitates the gums and the phosphatides which are then separated from the oil. The phosphatides may be isolated from the precipitated mixture and incorporated in the soap or in the event that a highly purified soap is not desired the phosphatides together with the precipitated impurities may be directly incorporated within the soap without being isolated. It will be apparent, however, that a higher quality product may be obtained when the phosphatides are isolated from the impurities before being incorporated in the soap.

A number of modifications of the process of Fig. 1 will be apparent. Thus, instead of using preformed tall oil soap for rehydration, it is possible to use tall oil which may be converted to soap in mixer **29** by means of excess alkaline refining agent previously withdrawn from tank **11**. Alternatively proportioned streams of tall oil and alkali may be mixed to form tall oil soaps in stream flow, which mixture may be used to rehydrate the dehydrated oil-soap stock mixture before separation.

In Fig. 2 I have illustrated an embodiment in which the tall oil or tall oil soap may be mixed with the oil and the refining agent before the dehydration treatment. Proportioned streams of oil, alkaline refining agent and tall oil may be withdrawn from supply tanks **60**, **61** and **62**, respectively, by means of pumps **63**, **64** and **65** driven by a motor **66** and the variable speed devices **67** and **68**. These materials may be preheated in the supply tanks if desired. Further heat may be added to the proportioned streams in coils **69**, **70** and **71**. The tall oil may be mixed with an alkaline refining agent in a mixer **72**. In the event that tall oil is used, it will be apparent that the acids therein will be neutralized by the alkali to form tall oil soaps. If tall oil soaps are withdrawn from tank **62** they will be unaffected by the alkali. In any event the mixture leaving mixer **72** will contain tall oil soaps and excess alkali. This mixture may then be mixed with the stream of oil in mixer **73** wherein the free fatty acids present in the oil will be converted into soaps. The resulting mixture may then be passed through heating coil **74** to add sufficient heat to the mixture to vaporize at least a portion of the water and other impurities upon discharge into the vaporizing chamber **20** which may be identical with that shown in Figure 1. Vapors released in the chamber **20** may be removed and condensed in condensers **50** and **51** and the chamber **20** may be maintained under a vacuum by means of pump **49**. Any desirable quantity of moisture and vaporizable impurities may be removed in chamber **20**. It has been found, however, that the greatest advantages are obtained when dehydration is substantially complete, and accordingly it is preferred to substantially completely dehydrate the mixture.

In order to separate the soap stock from the neutralized oil it has been found desirable to rehydrate the dehydrated mixture. Suitable rehydrating media may be water, aqueous solutions of various electrolytes such as sodium sulfate, sodium carbonate, etc. Other possible rehydrating agents are aqueous solutions of sodium bicarbonate, sodium lactate, sodium tartrate, sodium naphthenate, sodium citrate, sodium pyrophosphate, ammonium sulfate, ammonium chloride, calcium sulfate, magnesium sulfate, borax, disodium phosphate, trisodium phosphate, sodium thiosulfate or sodium thiocyanate, as well as equivalent potassium salts. In the present process in which tall oil is used, it has been found that the tall oil soaps assist in softening the soap stock and thus facilitate separation. Accordingly water alone may be used for rehydration to produce a readily separable mixture. A proportioned stream of rehydrating agent from tank **76** and a proportioned stream of dehydrated mixture from chamber **20** may be withdrawn by means of pumps **77** and **78**, motor **79** and variable speed device **80**. These proportioned streams may be mixed in mixer **81** after which the resulting mixture may be centrifugally separated in centrifuge **82**. In the event that the temperature of the mixture in **81** is not suitable, heat exchange coil **83** may be employed to adjust the temperature to one suitable for centrifugal separation. It will be apparent that this embodiment of the invention differs essentially from the embodiment in Figure 1 in that the tall oil soap is subjected to the vacuum conditions in chamber **20**. In this way it is possible to remove some of the vaporizable impurities from the tall oil soap as well as from the soap produced by neutralization of the free fatty acids in the oil. It will be apparent therefore that the soap stock discharged from centrifuge **82** will be of a somewhat higher purity than that discharged from centrifuge **31** in Figure 1. Where a soap product of lesser purity may be acceptable, the material discharged from centrifuge **82** may be used directly without further purification. Where a higher quality product is desired, however, it is preferred to further purify the product by the reheating and vacuum treatment disclosed in Figure 1.

A further embodiment of the invention is shown

in Figure 3. In this embodiment soap stock previously separated from the refined oil may be admixed with tall oil or tall oil soap and the combined mixture may then be subjected to a purification treatment. In the event that tall oil, in which free acids exist, is used, it is preferred to employ a soap stock containing excess alkali so that the admixture of the two will convert the free acids of the tall oil into soap. In the event that preformed tall oil soap is used, excess alkali in the soap stock is not necessary although such excess alkali may be employed where the finished soap product is desired to be alkaline. Referring to Figure 3, soap stock either with or without dilution may be preheated to form a flowable mixture in tank **90**. The tail oil may likewise be preheated in tank **91**. Proportioned streams of these two substances may be withdrawn by means of pumps **92** and **93**, motor **94** and variable speed device **95**. The proportioned streams may be mixed in mixer **96** after which the resulting mixture may be heated in coil **37** to a temperature sufficient to remove vaporizable impurities upon discharge into the vacuum chamber **39** which may be the same as the chamber **39** illustrated in Figure 1. The remainder of the treatment of the product may be substantially the same as that previously described with reference to Figure 1.

While any soap stock may be employed it is preferred to employ an alkaline soap stock such as that produced in accordance with the process described in my prior Patent 2,190,594. The soap stock produced according to that process usually contains a large excess of soda ash. This excess may be utilized to convert tall oil into soaps. The relative proportions of tall oil and soap stock depend upon the amount of excess soda ash present in the soap stock and the type of product desired. If substantially equivalent amounts of soda ash and tall oil acids are used the reaction will go to substantial completion in chamber **39** wherein the carbon dioxide evolved may be readily separated from the reaction mixture. In view of the rapid nature of the reaction between the two constituents it is not necessary to provide any prolonged reaction chamber, but on the contrary, the mixture may be rapidly heated and discharged into the vacuum chamber immediately following the mixing step. The character of the resultant product may be controlled to some extent by choosing the amount and character of the soap stock added. Thus the soap stock may consist of sodium soaps, potassium soaps, or any other type of soap depending upon the characteristics desired. Furthermore, it has been found that the melting point of tall oil soaps is lowered in the presence of fatty acid soaps.

While other temperature conditions may be employed, it has been found that best results are obtained when the soap mixture passing through heating coils **37** before discharge into vacuum chamber **39** is heated to a temperature of from about 250° to about 600° F. It will be apparent, however, that the point at which heat is added to the reactants is of relative unimportance. It is merely necessary that the mixture before discharge into chamber **39** be at a sufficient temperature to vaporize the desired proportion of impurity. This may be attained by heating the mixture of reactants or by raising the temperature of the individual reactants to the final temperature or by partially heating the separate reactants and further heating the resultant mixture.

The present process employing stream flow permits the employment of high temperatures for organic materials which ordinarily rapidly decompose at such temperatures. The rapidity of flow and the resultant agitation prevent local overheating and thus there is little danger of decomposition.

While various embodiments of the invention have been described with reference to mixing of the reactants in stream flow, it will be apparent that the invention is in no way limited thereto. Thus the refining reaction may be conducted in a conventional kettle and after the reaction has been conducted to the extent desired, a stream of the mixture may be withdrawn and subjected to the remaining portion of the described process in stream flow. In view of the previously described advantages accompanying stream flow heating as distinguished from heating in batch, particularly where high temperatures are employed, it is preferred that all high temperature heating be conducted in stream flow as described with reference to the continuous operation.

In Figs. 1 and 2 the invention has been described with reference to dehydration of the mixture of refined oil and soap stock. While the best results are obtained when this step is employed, the invention may likewise be practiced by omitting this step.

The present invention may also be employed in the production of grease. A proportioned stream of tall oil, preferably preheated to from 450° to 600° F., may be mixed with a proportioned stream of alkali such as calcium hydroxide or a mixture of calcium hydroxide and other alkalies in sufficient quantity to saponify the tall oil. Fatty acid soaps such as soap stock may likewise be employed in the mixture, depending upon the type of product desired. The resulting tall oil soap, with or without the added fatty acid soap, may be heated in coil **37** and flashed to the vapor separating chamber for the removal of various impurities. A mineral oil lubricant may be proportioned into conveyor **44** before extrusion of the grease so as to be thoroughly incorporated therein. Furthermore, various addition agents may likewise be introduced in the conveyor or at any other point. In this manner a continuous process for the manufacture of grease may be provided which will produce a relatively high quality grease from low quality starting material at a relatively low cost.

The term "animal oils" as used herein may include fish oils such as sardine, menhaden, herring, etc. These oils may be in the crude or partially refined state. The present application is a continuation-in-part of Serial Number 398,489 filed June 17, 1941, which in turn is a continuation-in-part of Serial No. 296,685, filed September 26, 1939, now Patent No. 2,249,701, granted July 15, 1941. The present application is also a continuation-in-part of Serial Number 371,811, filed December 26, 1940, now Patent No. 2,327,502, granted August 24, 1943.

The tall oil or tall oil soaps used in the process may be crude or refined products derived from any desirable source. Preferably, the tall oil or tall oil soap used is that prepared according to Thurman application Serial Number 402,872, filed July 17, 1941.

While various modifications of the process have been described in detail, it is understood that the invention is not limited thereto but may be varied within the scope of the appended claims.

I claim:

1. The process of refining animal and vegetable oils, which comprises, subjecting a relatively dry mixture containing neutralized oil and soap stock to centrifugal separation in the presence of a

hydrating agent containing a soap selected from the group consisting of rosin and naphthenic acid soaps.

2. The process of refining animal and vegetable oils containing free fatty acids, which comprises, adding to said oils a neutralizing reagent for said free fatty acids which will not attack neutral oil, carrying the neutralization reaction to substantial completion in a vacuum to produce a relatively dry mixture of oil and soap stock, thereafter continuously centrifugally separating soap stock from said oil in the presence of a rehydrating solution and rosin soap for causing the soap stock to flow continuously from the centrifugal separator employed.

3. The process of refining animal and vegetable oils containing free fatty acids, which comprises, adding to said oils a neutralizing reagent for said free fatty acids which will not attack neutral oil, carrying the neutralization reaction to substantial completion in a vacuum to produce a relatively dry mixture of oil and soap stock, thereafter continuously centrifugally separating soap stock from said oil in the presence of a rehydrating solution and naphthenic acid soap for causing the soap stock to flow continuously from the centrifugal separator employed.

4. The process of refining animal and vegetable oils, which comprises, mixing an aqueous alkaline reagent with said oils to form a mixture of soap stock and oil containing water, delivering a stream of said mixture into a vapor separating chamber and separating vapors therefrom at a temperature sufficiently high to at least partially dehydrate said soap stock, continuously delivering a stream of said mixture of oil and dehydrated soap stock to a continuous centrifugal separator, admixing a stream of rehydrating solution containing rosin soap with said stream of oil and dehydrated soap stock to rehydrate said soap stock, and facilitate separation thereof, and continuously centrifugally separating the rehydrated soap stock from said oil.

5. The process of refining animal and vegetable oils, which comprises, mixing an aqueous alkaline reagent with said oils to form a mixture of soap stock and oil containing water, delivering a stream of said mixture into a vapor separating chamber and separating vapors therefrom at a temperature sufficiently high to at least partially dehydrate said soap stock, continuously delivering a stream of said mixture of oil and dehydrated soap stock to a continuous centrifugal separator, admixing a stream of rehydrating solution containing naphthenic acid soap with said stream of oil and dehydrated soap stock to rehydrate said soap stock, and facilitate separation thereof, and continuously centrifugally separating the rehydrated soap stock from said oil.

6. The process of refining animal and vegetable oils, which comprises, subjecting a relatively dry mixture containing neutralized oil and soap stock to centrifugal separation in the presence of a hydrating agent containing tall oil soap.

7. The process of refining animal and vegetable oils, which comprises, mixing oil with an alkaline refining agent to produce a mixture containing neutralized oil and soap stock and continuously centrifugally separating said mixture from said oil in the presence of a soap selected from the group consisting of rosin and naphthenic acid soaps.

8. The process of refining animal and vegetable oils, which comprises, producing a relatively dry mixture containing neutralized oil and soap stock, mixing a proportioned stream of said mixture with a proportioned stream of a hydrating agent containing tall oil soap, and continuously centrifugally separating said soap stock and said tall oil soap from said neutralized oil.

9. The process of refining animal and vegetable oils, which comprises, producing a relatively dry mixture containing neutralized oil, soap stock, and excess alkaline refining agent, mixing a proportioned stream of said mixture with a proportioned stream of tall oil, maintaining the resulting mixture for a sufficient period of time to convert said tall oil into tall oil soaps, and continuously centrifugally separating said tall oil soap and said soap stock from said oil.

10. The process of refining animal and vegetable oils, which comprises, mixing a proportioned stream of said oil, a proportioned stream of an alkaline refining agent, and a proportioned stream of a member of the group consisting of tall oil and tall oil soap, to produce a mixture containing neutralized oil, soap stock and tall oil soap, subjecting said mixture to a dehydration treatment, and thereafter rehydrating said mixture and separating said soap stock and tall oil soap from said neutralized oil.

11. The process as defined in claim 7 in which the mixture is centrifugally separated in the presence of tall oil soap.

12. The process as defined in claim 7 in which tall oil is added to the mixture of neutralized oil and soap stock so that said mixture is separated in the presence of tall oil soap to facilitate such separation.

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