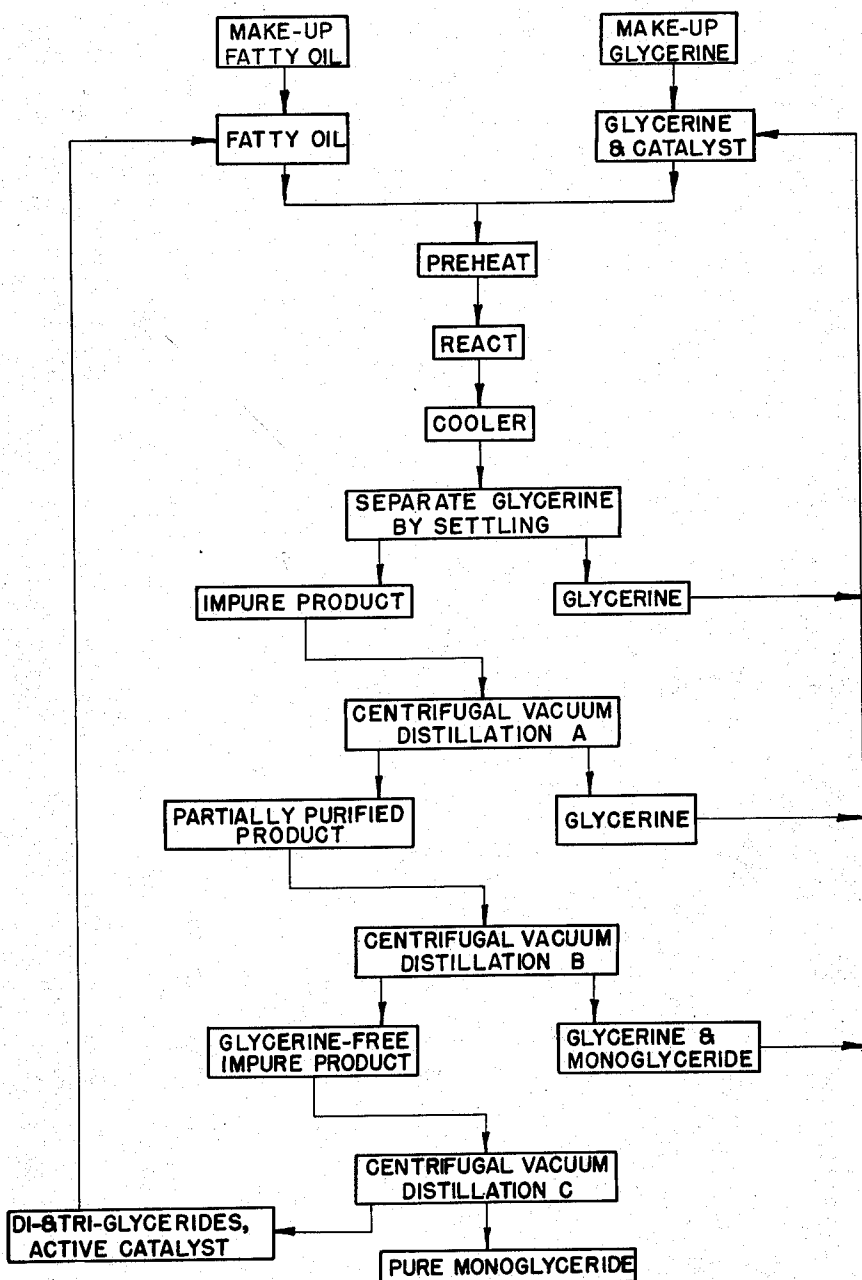


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PROCESS FOR SUBSTANTIALLY COMPLETE CONVERSION
OF FATTY MATERIAL TO PARTIAL ESTER
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CONVERSION OF FATTY MATERIAL TO
PARTIAL ESTER

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1

This invention relates to a process of treating fatty material and is particularly concerned with a process of effecting conversion of fatty material to a fatty acid partial ester of a polyhydric alcohol.

Fatty acid partial esters of polyhydric alcohols are prepared by reacting fatty material with an alcohol in the presence of an ester-interchange catalyst.

It is an object of this invention to provide a highly effective process for treating fatty material.

It is a further object of the invention to provide a commercially feasible process for preparing fatty acid partial esters of polyhydric alcohols.

Another object of the invention is to provide a process for effecting substantially complete conversion of fatty material to a partial ester.

Another object is to provide a novel process whereby substantially pure partial ester is prepared and recovered.

A further object of the invention is to provide a process whereby ester-interchange catalyst is retained in active form throughout the process.

Another object of the invention is to provide a cyclic process of preparing monoesters.

Another object of the invention is to obtain monoglycerides in high yield.

Another object of the invention is to provide a process whereby substantially all of the partial ester formed by alcoholysis of fatty material is recovered.

Other objects will be apparent from the drawing, description and claims.

These and other objects are attained by means of this invention which is best illustrated by reference to an embodiment thereof. The single figure of the drawings is a schematic representation of a process embodying the invention whereby a fatty oil such as soybean oil is converted to monoglyceride by reacting the soybean oil with glycerine in the presence of an ester-interchange catalyst.

In converting soybean oil to monoglyceride in accordance with this invention, a predetermined amount of the oil is admixed with an excess of glycerine and with an ester-interchange catalyst. The amount of glycerine employed for alcoholysis of the oil may be varied; and, although stoichiometric or smaller amounts may be employed, it is preferable to use an excess of glycerine for optimum conversion of the oil to monoglyceride. The convertible fatty material in the soybean oil consists largely of fatty acid compounds present as free fatty acid or fatty acid esters, particularly

2

triglycerides, and the amount of glycerine used is calculated from the amount of convertible fatty material present in the oil calculated as fatty acid. Thus the glycerine may be used in stoichiometric amounts or less but is preferably employed in at least 40% excess over stoichiometric amounts and in many cases as much as 200% excess or more.

The alcoholysis is carried out in the presence of an ester-interchange catalyst which promotes the alcoholysis or esterification. Suitable ester-interchange catalysts include any of the well-known alkaline or acid catalysts which do not distill simultaneously with the desired partial ester. Catalysts desirably employed in the alcoholysis of soybean oil or similar vegetable oil or other fatty material are the metal compounds which react with fatty acid present in the reaction mixture to form a metal soap which functions as ester-interchange catalyst. Particularly suitable metal compounds are the alkali and alkaline earth metal oxides and hydroxides and desirably the bivalent metal compounds. Thus although such monovalent metal compounds as sodium hydroxide, sodium methylate, sodium carbonate and the like may be employed, particularly efficacious results are obtained by adding to the reaction mixture such bivalent metal compounds as strontium oxide, calcium oxide, calcium hydroxide, strontium hydroxide, barium oxide and the like. Strontium compounds are particularly desirable catalytic materials because of the enhanced yield of partial ester and its marked ability to prevent formation of color bodies during the process. The absence of color body formation is of particular importance in the cyclic process and particularly a continuously circulating process since color bodies formed in the process otherwise build up in the system and contaminate the product.

The amount of catalyst may be varied in accordance with well-known practice as, for example, from 0.01 mole to 0.5 mole of catalyst per equivalent of fatty material in the reaction.

The resulting mixture of soybean oil, glycerine, and catalyst is then reacted to cause alcoholysis of the fatty material and formation of substantial amounts of partial ester including monoglyceride. The temperature at which the reaction is carried out may vary from about 20° C. to about 300° C. depending upon the materials being treated. It is necessary, however, that the minimum reaction temperature be sufficient to cause substantial formation of the desired partial ester and it is desirable to carry out the reaction under

reaction conditions effective to cause substantially maximum formation of the desired partial ester in the composition resulting from the reaction. In the case of alcoholysis of soybean oil, substantial formation of monoglyceride is initiated above about 180° C. but desirably the reaction is effected at a temperature above about 230° C. The alcoholysis of soybean oil is preferably carried out above the minimum reaction temperature at which maximum amounts of monoglyceride are obtained and desirably in the range of about 250° to 260° C., although reaction is successfully effected at a temperature of about 180°–200° C. with longer reaction times.

After reaction has been effected, the monoglyceride formed in the reaction is separated from the composition resulting therefrom while the composition contains the catalyst in active form. During the reaction, the composition reaches an equilibrium concentration between the alcohol, the convertible fatty material and the partial ester formed by the reaction. The equilibrium system reached under reaction conditions is, however, a dynamic equilibrium. A change in the relative proportions of the components in the composition causes the equilibrium to shift in order to assume a new metastable condition. Thus, for example, removal of alcohol from the composition by phase separation or other means causes reversion of partial ester to be initiated whereby additional alcohol is liberated to take the place of alcohol separated from the composition. The speed of reversion of the partial ester is a function of the change in concentration of the alcohol, the temperature of the composition and the presence of catalyst. Elevated temperatures and presence of active catalyst in general accelerate the reversion. Lowering the temperature, however, causes alcohol to separate from the oil phase and radically changes the composition of the system.

The interrelationship of concentration, temperature, and catalyst determines the time interval necessary for substantial reversion of partial ester to be effected, and the time interval, for any given combination of conditions, which will elapse before substantial reversion of the partial ester to a more stable ester form occurs is denoted the reversion time interval.

To illustrate what is meant by reversion time interval, if soybean oil and glycerine are reacted at elevated temperatures in the presence of active catalyst, an equilibrium system is attained. If the temperature is maintained at the final reaction temperature and the concentration of the components of the system is maintained constant by preventing loss, decomposition or phase separation of the components, the system is maintained at static equilibrium for an infinite time interval.

When the system is cooled, however, glycerine separates from the reaction composition and reversion of partial ester is initiated. The lowered temperature slows the reversion and when the composition is rapidly cooled for example from a reaction temperature of 260° C. to a temperature of 100° C., the reversion time interval will be several hours. Thus, for example, 10% reversion does not occur in less than about 8 hours at 100° C.

When distillation is effected, however, to remove from the system either glycerine or partial ester or both, the reversion time interval is greatly shortened by the elevated temperatures employed. Substantial reversion of the order of

10% or more will occur in ten minutes when distillation of monoglyceride is effected from a glycerine-depleted composition at a temperature of 200° C. and at least 10% reversion will occur under distillation conditions at a temperature above about 230° C. in about 5 minutes.

I have discovered that in the process of this invention, separation of partial ester must be effected during the reversion time interval for optimum yield. Because of the shortened reversion time interval at elevated temperatures, I discovered that separation of the alcohol and partial ester must be effected simultaneously or that separation of the partial ester from a composition freed of a substantial portion of its unreacted alcohol must be effected rapidly if substantial reversion is to be obviated.

I have found that such separation of partial ester is successfully effected by spreading the composition, containing partial ester and active catalyst, in a thin film at a temperature below the decomposition temperature of the catalyst and subjecting the film to vacuum effective to distill partial ester therefrom. This is in contrast to pot type vacuum distillation where the reversion time interval is exceeded before separation is effected and objectionable reversion occurs.

Thus for optimum yield, monoglyceride present in a composition resulting from alcoholysis of soybean oil with glycerine in the presence of ester-interchange catalyst is separated from the composition, before the reversion time interval has elapsed, by vacuum distillation from a thin film leaving a distillation residue containing the catalyst in active form.

This separation is effected by maintaining the composition resulting from reaction substantially at reaction temperature and spreading successive portions on an evaporating surface maintained under vacuum so as to flash evaporate monoester and glycerine simultaneously and rapidly. Alternatively the composition is cooled to allow glycerine to separate from the composition and be removed by phase decantation whereafter the monoester and any remaining glycerine are simultaneously distilled rapidly from the glycerine-depleted composition by flash evaporation from a film as set forth hereinabove within the reversion time interval.

In the preferred separation, the separating is effected within the reversion time interval by cooling the reaction composition to about 100° C., causing a substantial proportion of the unreacted glycerine to separate from the composition, separating the layers thus obtained by phase decantation, thereafter vacuum distilling substantially all of the residual glycerine remaining in the composition therefrom by vacuum distillation of successive increments of the glycerine-depleted composition in film from under conditions ineffective to substantially distill monoglyceride therefrom, and thereafter progressively subjecting successive increments of the resulting substantially glycerine-free composition to vacuum distillation from a thin film under conditions effective to distill monoester therefrom.

Thus, separation of the desired ester is effected while the composition contains the catalyst in active form and without substantial reversion of the partial ester during the separation by spreading the composition obtained from the reaction in a thin film on an evaporating surface, maintaining the film at a temperature substantially below the decomposition temperature of the catalyst employed and subjecting the film to

vacuum effective to distill monoglyceride therefrom leaving a distillation residue containing substantially all of the original catalyst still in active form. The partial ester distilled from the film is condensed and cooled substantially immediately upon being distilled from the film so that the entire separation is effected during a time interval insufficient to effect substantial reversion of the partial ester. The separation is described in greater detail in my copending application Serial No. 105,271 filed July 16, 1949.

Distillation of monoglyceride or other partial ester is readily effected in this manner in a short time since, due to the thinness of the layer being distilled, substantially all the molecules of monoglyceride readily find their way to the distilling surface and escape from the heated film. Thus, no portion of the composition is subjected to objectionable local overheating nor held at an elevated temperature for an extended period of time.

Distillation is preferably effected by causing successive small increments of the composition being distilled to flow along an evaporation surface so that distillation of any given increment is completed in a time less than about five minutes. It is desirable to employ high vacuum substantially unobstructed path centrifugal distillation for effecting separation of the monoglyceride whereby relatively large quantities of monoglyceride may be distilled in a relatively short time. In the case of such high vacuum centrifugal distillation, the total distillation period for any given increment of the composition is less than a minute and usually only a few seconds. With such rapid distillation, the reversion time interval is not exceeded even with active catalyst present in the distilland. Thus the distillation of monoglyceride may be effected from a composition containing active catalyst at temperatures of the order of 130°-200° C. and distillation temperatures below about 225° C. are preferably employed.

The distillation is carried out under conditions effective to molecularly distill the monoglyceride. The pressure employed must be effective to cause distillation of the desired partial ester from the film of distilland at the temperature employed. In most cases, it is desirable to employ a vacuum below about 100 microns and preferably below about 50 microns mercury with pressures of the order of 1-20 microns or less being desirably employed.

If it is desired to obtain monoglyceride substantially free of alcohol, the unreacted glycerine in the composition resulting from the reaction is separated before vacuum distillation of the monoglyceride. Alcohols such as glycerine may be at least partially separated from the reaction mixture with fatty oils such as soybean oil by cooling and allowing the reaction composition to stand for a short time and separate into layers whereby a substantial part of the unreacted glycerine may be drawn off.

The remaining unreacted glycerine is then separated from the composition by subjecting the composition to high vacuum film distillation under conditions effective to distill off the glycerine without distilling a substantial amount of monoglyceride. The resulting glycerine-free composition is then vacuum distilled whereby monoglyceride substantially free of catalyst and containing less than 1% glycerine is obtained as distillate leaving a distillation residue comprising active catalyst and unreacted oil which con-

sists essentially of di- and triglycerides in the case of oils such as soybean oil.

This distillation residue is then recombined with the unreacted glycerine previously separated therefrom, the resulting admixture is replenished with additional oil and glycerine and the process repeated. These successive steps are repeated until the amount of monoglyceride being recovered as distillate is substantially equivalent to the amount of convertible fatty material being added whereby substantially complete conversion of the oil to monoglyceride is effected. The active catalyst is repeatedly recycled and it is necessary only to add make-up amounts of oil and glycerine at each cycle. The amount of catalyst remains substantially constant without substantial replenishment although small amounts may be added if necessary to make up for losses which may occur during the recycling.

Thus, by means of this invention, quantity production of pure monoglyceride is made possible employing only a small initial quantity of catalyst and converting substantially all the fatty material being treated to monoglyceride and the monoglyceride recovered is substantially free of catalyst and glycerine.

Any of the partial esters prepared by the alcoholysis of fatty material may be prepared in accordance with this invention. The fatty material converted may be any of the natural or synthetic fatty acid compounds including fatty acids or fatty acid esters or mixtures thereof and including either solid or liquid fats and fatty oils. The fatty acid compounds are preferably those fatty materials having from about twelve to about twenty carbon atoms in the fatty acid chain although fatty acid chains of from about eight to about twenty-six carbon atoms are suitable for use in practicing this invention. The fatty acid esters may include esters of either monohydric or polyhydric alcohols, as, for example, the fatty oils comprising a substantial proportion of triglycerides.

Suitable fatty materials which may be treated in accordance with this invention include marine oils such as fish oils and whale oil, animal fatty materials such as tallow, wool grease, hydrogenated tallow and the various other animal fats and fatty oils, and vegetable oils such as soybean oil, cottonseed oil, tung oil, coconut oil, palm oil, corn oil, olive oil, castor oil, peanut oil and the like, as well as such other fatty materials as the hydrogenated vegetable oils or shortenings. Partial esters of particularly good quality for certain uses are prepared by alcoholysis of free fatty acids such as stearic acid, oleic acid, lauric acid, palmitic acid, myristic acid and the like.

The alcohol employed in the reaction may be either monohydric or polyhydric; but where a monohydric alcohol such as methyl alcohol or ethyl alcohol is employed it is reacted with a fatty acid ester of a polyhydric alcohol to give a partial ester. Alcohols which are particularly suitable for alcoholysis are the aliphatic alcohols including the aryl-substituted alcohols and desirably the lower saturated polyhydric alcohols. Thus, suitable alcohols include the polyhydroxy alkanes such as glycerol, sorbitol, mannitol and the like; ether alcohols such as diglycerol, polyglycerols and the like; alkylene glycols such as trimethylene glycol, ethylene glycol, propylene glycol, diethylene glycol, polyethylene glycols, polypropylene glycols and the like; as well as such other alcohols as pentaerythritol and the like.

The catalyst employed in the reaction may be

any ester-interchange catalyst which will not distill with the desired partial ester and, although the metal compounds such as alkali and alkaline earth compounds and particularly the bivalent metal compounds are preferably employed, other suitable catalysts include well-known organic and inorganic acid catalysts such as phosphoric acid, sulfuric acid or similar inorganic acid, substituted inorganic acids such as alkyl sulfuric acid, organic acids such as aryl sulphonic acids, and the like, and other acid materials, as well as such other materials as tetramethyl ammonium hydroxide, aluminum chloride, and the like. The preferred strontium compounds are the fatty acid-reactive strontium compounds and particularly the water or alcohol soluble compounds. Suitable strontium compounds include strontium oxide, strontium hydroxide, strontium chloride, strontium bromide, strontium nitrate, strontium acetate, strontium tetraborate, strontium lactate and the like and such compounds are desirably employed where product color is a prime factor.

The alcoholysis reaction is carried out at a temperature substantially above the minimum temperature necessary for formation of the desired partial ester and desirably at a temperature sufficiently above the minimum reaction temperature to establish an equilibrium system with optimum yield of the desired partial ester in a period of from $\frac{1}{2}$ to 4 hours. The reaction is generally effected at temperatures of from about 20° C. to about 400° C. depending upon the materials being reacted and the reaction time desired. For example, the alcoholysis of soybean oil has been carried out at 180°–200° C. but is more preferably effected at 250°–260° or desirably at 285° C. The reaction mixture is preferably maintained at reaction temperature for about one-half hour although in some cases alcoholysis may be effected in fifteen minutes or less or the reaction may be carried on for as much as one or two hours or longer. When the reaction mixture is heated for extended periods, precautions should be taken to prevent substantial loss of the alcohol, which would shift the reaction equilibrium.

In the process according to this invention, it is desirable to preheat the reaction mixture before it is introduced into the reaction in order that the total reaction time may be minimized. The reaction mixture is preferably agitated during reaction and water evolved during the reaction should be continuously removed from the reaction zone.

The invention is particularly applicable for the preparation and recovery of monoesters such as monoglycerides but the invention may be employed in preparing any fatty acid partial ester such as diglycerides and the like. The desired partial ester may be separated and recovered with unreacted alcohol from the reaction in which case the amount of alcohol employed during the reaction may be adjusted to give a product of the desired composition.

Preferably, however, the desired partial ester is separated in substantially pure form and the unreacted alcohol is recycled with the distillation residue containing the active catalyst whereby only make-up amounts of fatty material and alcohol are added to replenish the composition from which the partial ester has been separated.

In an example of the process in accordance with this invention as represented schematically in the drawings, a fatty oil such as soybean oil is admixed with glycerine in the proportions of

2.3 moles of glycerine per mole of fatty oil calculated as triglyceride and 0.1% by weight of strontium oxide based on the weight of the reaction mixture. The strontium oxide combines with fatty acid in the oil to form a strontium soap which functions as ester-interchange catalyst in the reaction. The resulting mixture is passed through a preheater wherein the temperature is raised to about 225°–230° and the heated mixture is then led into a pot reactor. In the reactor, the mixture is heated to 250°–260° C. and maintained at that temperature for 20 minutes to cause formation of monoglyceride. During the reaction, the mixture is constantly agitated and water vapor evolved during the reaction is vented from the reactor.

The composition resulting from the reaction, consisting primarily of active catalyst, unreacted glycerine, and mono-, di- and triglycerides, is then led through a cooling coil whereby the temperature of the composition is rapidly reduced to about 100° C.

The composition is allowed to stand without agitation for about five minutes whereupon a substantial proportion of the unreacted glycerine separates from the body of the composition and is drawn off.

The remainder of the composition which is poor in glycerine is continuously fed on to the rotor of a high vacuum substantially unobstructed path centrifugal still. The nature of centrifugal distillation is such that a single pass of a given increment of composition being distilled is usually effected in a second or two or at most in less than 2 or 3 minutes. Furthermore, with the composition spread in a thin film, the temperature of the composition can be rapidly changed. It is therefore possible to distill off glycerine in a relatively short time and at a relatively low temperature without initiating reversion of the monoglyceride.

Accordingly most of the residual glycerine remaining in the composition is separated therefrom by subjecting successive small increments of the composition to high vacuum centrifugal distillation at about 100 microns pressure and a rotor temperature of about 100° C., although pressures of 10–300 microns and temperatures of 90°–150° C. are suitable.

In order to ensure substantially pure monoglyceride being obtained as product, the distillation residue from the first distillation is immediately passed to another high vacuum unobstructed path centrifugal still whereon a second distillation is effected at about 20 microns pressure and a rotor temperature of about 120° C. whereby substantially all residual unreacted glycerine and a small amount of monoglyceride is distilled from the composition.

The resulting substantially glycerine-free distillation residue which still contains the catalyst in active form is immediately subjected to a third high vacuum centrifugal distillation at about 3 microns pressure and about 170° C. rotor temperature whereby about 30% by weight of the composition being distilled is distilled out as pure monoglyceride containing substantially no catalyst and less than 1% glycerine by weight. This distillate is immediately condensed and recovered.

The distillation residue from the third distillation is thereafter preferably molecularly distilled at a pressure of about 3 microns and a temperature of about 210° C. whereby any residual monoglyceride is stripped therefrom and returned to the system to be redistilled in the above-named

third distillation. In a continuous process of successive distillations, the entire series of distillations is readily effected in less than five minutes of actual distillation time for any given increment of composition.

The final distillation residue consisting mainly of diglycerides, triglycerides and active catalyst is then recombined with the unreacted glycerine cuts separated from the reaction composition prior to recovery of the pure monoglyceride and the recombined admixture is replenished with make-up amounts of fatty oil and glycerine correlated to the amount of monoglyceride recovered and the cycle is repeated successively. Since the original catalyst is retained intact throughout the process, no additional catalyst is added. It is desirable to separate from the distillation residue small amounts of polymeric material formed in the reaction before recycling.

By the time the third cycle is being effected, the amount of monoglyceride being recovered is substantially equivalent to the amount of convertible fatty oil being added to replenish the distillation residue. Thus, the process is readily continued for extended periods of time whereby substantially all of the convertible fatty material being treated is recovered as monoglyceride.

Thus, by means of this invention it is possible to obtain substantially complete yields of monoglyceride, diglycerides or other partial ester rather than the yields of 40-50% by weight based on the weight of fatty material obtained heretofore. Furthermore, the monoglyceride obtained contains less than 1% glycerine by weight as compared to commercially available monoglyceride preparations which contain 7% or more glycerine. The monoglyceride obtained is substantially free of fatty acids, diglycerides and triglycerides rather than being mixed with the almost equal amount of diglyceride normally formed during the reaction. The distillation residue is readily recycled since it contains no materials added to inactivate the catalyst. The cost of the catalyst becomes a negligible factor since it is reused repeatedly. There is substantially no by-product to be disposed of since substantially the entire amount of fatty material is converted to the desired partial ester. The process is capable of large-scale production of monoglycerides or other partial ester of high purity which was not feasible heretofore. Substantially all the monoglyceride formed in the reaction is recovered since substantial reversion is obviated.

While the invention has been described in considerable detail with reference to the preparation and recovery of monoglyceride, it will be understood that the invention is applicable to the preparation and recovery of any fatty acid partial ester of a polyhydric alcohol or mixtures thereof and that variations and modifications may be effected within the spirit and scope of the invention in accordance with the foregoing description and drawings and as defined in the appended claims.

What I claim is:

1. The process which comprises admixing a fatty material, an alcohol and an ester-interchange catalyst, reacting the resulting mixture at a reaction temperature effective to cause substantial formation of a desired fatty acid partial ester in the composition resulting from the reaction, separating said partial ester from said composition while said composition contains said catalyst in active form, said separating being effected by spreading said composition in a thin

film while maintaining said film at a temperature substantially below the decomposition temperature of said active catalyst and subjecting said composition while in said thin film to vacuum effective to distill said partial ester therefrom leaving a distillation residue containing said active catalyst, replenishing said distillation residue with make-up amounts of said fatty material and said alcohol and again effecting reaction of said replenished residue and separating partial ester formed thereby, and repeating said process at least until the amount of said partial ester being recovered is substantially equivalent to the amount of convertible fatty material being added to the distillation residue whereby substantially complete conversion of said fatty material to said partial ester is effected.

2. The process which comprises admixing a fatty material, an alcohol and an ester-interchange catalyst, reacting the resulting mixture at a temperature effective to cause substantial formation of a desired fatty acid partial ester in the composition resulting from the reaction, separating from said composition a substantial proportion of said alcohol remaining in said composition after said reaction, separating said partial ester from the remainder of said composition while said composition contains said catalyst in active form, said last-named separating being effected by spreading said composition in a thin film while maintaining said film at a temperature substantially below the decomposition temperature of said active catalyst and subjecting said film to vacuum effective to distill said partial ester from said composition leaving a distillation residue containing said catalyst in active form, recombining said distillation residue and said alcohol separated from said composition, adding thereto make-up amounts of said fatty material and said alcohol, repeating the reaction and separation of said partial ester formed thereby, and continuing the process until the amount of said partial ester being distilled from said composition is substantially equivalent to the amount of convertible fatty material being added to said distillation residue whereby substantially complete conversion of said fatty material to said partial ester is effected.

3. The process which comprises admixing fatty material with an excess of an alcohol and an ester-interchange catalyst, reacting the resulting mixture to cause maximum formation of a desired fatty acid partial ester of a polyhydric alcohol in the composition resulting from the reaction, and during the reversion time interval before substantial reversion of said partial ester is effected and while said composition contains said catalyst in active form, separating said partial ester from said composition by spreading said composition in a thin film while maintaining said composition at a temperature substantially below the decomposition temperature of said active catalyst and subjecting said film to vacuum effective to distill said partial ester therefrom leaving a distillation residue of higher ester in admixture with said active catalyst, adding to said residue make-up amounts of said fatty material and said alcohol and again reacting the resulting replenished mixture and separating said partial ester as in said first-named reaction and separation and thereafter again replenishing the distillation residue, and repeating said process until the amount of said partial ester being recovered is substantially equivalent to the amount of convertible fatty material being added where-

11

by substantially all of said fatty material is converted and recovered as said partial ester.

4. The process which comprises admixing fatty material with an excess of a polyhydric alcohol and a bivalent metallic ester-interchange catalyst, reacting the resulting mixture under conditions effective to establish an equilibrium composition containing substantially a maximum amount of a fatty acid monoester, recovering substantially all of said monoester present in said composition during the reversion time interval, which time interval is insufficient to permit substantial reversion of said monoester, said recovering being effected while said composition contains said catalyst in active form by first separating unreacted polyhydric alcohol from said composition and thereafter separating said monoester from said composition, said last-named separating being effected by subjecting successive portions of said composition to high-vacuum substantially unobstructed path centrifugal distillation at a temperature substantially below the decomposition temperature of said catalyst under conditions effective to distill said monoester from said composition leaving a distillation residue containing said catalyst in active form, recombining said separated unreacted polyhydric alcohol and said distillation residue, replenishing the resulting admixture with make-up amounts of said fatty material and said polyhydric alcohol, and repeating the reaction and separation of said monoester and replenishing of the distillation residue until the amount of said monoester being recovered is substantially equivalent to the amount of fatty material being added to replenish the distillation residue.

5. The process which comprises admixing a fatty oil with an excess of glycerine and an ester-interchange catalyst comprising a bivalent metal compound, reacting the resulting mixture under conditions effective to cause substantially maximum formation of monoglyceride in the composition resulting from said reaction, thereafter within a reversion time interval insufficient to permit substantial reversion of said monoglyceride and while said composition contains said catalyst in active form, separating said monoglyceride from said composition by subjecting successive portions of said composition to molecular distillation leaving a distillation residue containing said catalyst in active form, replenishing said distillation residue with additional fatty oil and glycerine, and repeating the reacting, separating and replenishing a plurality of times whereby the amount of additional fatty oil being added is substantially equivalent to the amount of monoglyceride being separated from the composition resulting from the reaction.

6. The process which comprises converting substantially all of the fatty constituents of a vegetable oil to monoglyceride, said converting being effected by reacting a predetermined amount of said vegetable oil with an excess of glycerine in the presence of a bivalent metal soap catalyst at a temperature effective to cause substantial formation of monoglyceride in the composition resulting from the reaction, separating said monoglyceride from said composition while said composition contains said catalyst in active form, said separating being effected by subjecting successive portions of said composition to high vacuum unobstructed path centrifugal distillation at a temperature substantially below the decomposition temperature of said catalyst under conditions effective to distill monoglyceride from said

12

composition leaving a distillation residue containing said catalyst in active form, replenishing said distillation residue with additional vegetable oil and glycerine, and repeating said process successively until the amount of monoglyceride being separated is substantially equivalent to the amount of vegetable oil being added to the distillation residue.

7. The process which comprises successively and repeatedly reacting fatty material and polyhydric alcohol in the presence of an ester-interchange catalyst under conditions causing substantial formation of a monoester in the composition resulting from the reaction, separating said monoester from said composition, while the catalyst is in active form, by molecularly distilling said monoester from said composition leaving a distillation residue containing said active catalyst, and replenishing said distillation residue with additional fatty material and polyhydric alcohol, said process being continued at least until the amount of said monoester being distilled from said composition is substantially equivalent to the amount of said fatty material being added to said residue.

8. The process which comprises the steps of reacting a fatty acid compound with a polyhydric alcohol in the presence of a bivalent metal soap catalyst under conditions effective to cause substantial formation of monoester in the composition resulting from the reaction, separating said monoester and unreacted polyhydric alcohol from said composition and from each other, while said composition contains said catalyst in active form by repeated high vacuum substantially unobstructed path centrifugal distillation under conditions leaving a distillation residue substantially free of polyhydric alcohol and monoester but containing said catalyst in active form, recombining said separated unreacted polyhydric alcohol with said distillation residue, replenishing the resulting mixture with additional fatty acid compound and polyhydric alcohol, and successively repeating said steps at least until the amount of said monoester being separated from said composition is substantially equivalent to the amount of fatty acid compound being added to replenish said distillation residue.

9. The process which comprises repeating the successive steps of reacting a fatty acid compound with glycerine in the presence of a bivalent metal soap catalyst under conditions effective to cause substantially maximum formation of monoglyceride in the composition resulting from the reaction, recovering substantially pure monoglyceride formed in said reaction by separating said monoglyceride from said composition during a time not substantially greater than the reversion time interval, which reversion time interval is insufficient to permit substantial reversion of said monoglyceride, and while said composition contains said catalyst in active form leaving a residue containing said active catalyst, said separating being effected by first treating said composition to remove therefrom substantially all unreacted glycerine, said treating including subjecting successive portions of said composition to high vacuum unobstructed centrifugal distillation under conditions causing substantially all of said glycerine to distill from said composition without distilling a substantial amount of monoglyceride therefrom, and thereafter subjecting successive portions of said composition substantially freed of glycerine to high vacuum substantially unobstructed path centrifugal distillation

under conditions effective to distill substantially pure monoglyceride therefrom at a temperature substantially below the decomposition temperature of said active catalyst, recombining said residue containing said active catalyst with said unreacted glycerine removed therefrom, and replenishing the resulting recombined mixture with additional fatty acid compound and glycerine.

10. The process of converting fatty material to fatty acid partial ester of polyhydric alcohol which comprises establishing a cyclic system by repeatedly in successive stages of said system effecting ester-interchange in a reaction mixture between fatty material and an alcohol in the presence of an ester-interchange catalyst under conditions causing substantial formation of a fatty acid partial ester of polyhydric alcohol in the composition resulting from the reaction, separating said partial ester from the remainder of said composition while said catalyst is in active form by progressively spreading said composition in a thin film and vacuum distilling said partial ester from said composition in said thin film leaving a distillation residue containing said catalyst in active form, and recycling said distillation residue to again effect ester-interchange in the reaction mixture, progressively withdrawing from said cyclic system partial ester formed in said reaction mixture and progressively adding to said cyclic system fatty material and alcohol in amounts substantially equivalent to said partial ester being withdrawn from said system.

11. In the process of converting fatty material to a fatty acid partial ester of a polyhydric alcohol by reacting fatty material with an alcohol in the presence of an ester-interchange catalyst, the method which comprises establishing a continuously circulating system containing said ester-interchange catalyst in active condition throughout said system, progressively adding to said system reacting proportions of said fatty material and said alcohol, effecting an ester-interchange reaction between said fatty material and said alcohol in the course of said circulating in said system, and progressively removing from said system partial ester produced in said reaction, said removing being effected by progressively spreading the composition resulting from said reaction in a thin film and vacuum distilling said partial ester from said composition leaving a distillation residue containing said catalyst in active form, coordinating the quantities of fatty material and alcohol progressively added to said system with the amount of partial ester progressively removed from said system and thereby maintaining predetermined equilibrium conditions in said system, the quantity of catalyst in said system remaining substantially constant without substantial replenishment.

12. The process which comprises repeatedly and successively subjecting a reaction mixture comprising fatty material in admixture with a polyhydric alcohol and a strontium soap ester-interchange catalyst to an alcoholysis reaction, separating from said reaction mixture polyhydric alcohol fatty acid partial ester formed in said reaction, and adding to said reaction mixture reactive amounts of fatty material and polyhydric alcohol, said strontium soap catalyst being in active form throughout said process, said separating being effected following said reaction by progressively spreading said reaction mixture in a thin film on a vaporizing surface, maintaining said film at a temperature substantially below

the decomposition temperature of said strontium soap catalyst, and subjecting said reaction mixture while in said thin film to vacuum effective at said temperature to distill said partial ester from said reaction mixture.

13. The method of preparing a monoester composition substantially free of objectionable color contaminants, which method comprises establishing a cyclic system having an ester-interchange catalyst comprising a strontium compound maintained in active form throughout said system, said cyclic system comprising repeating the successive steps of (1) effecting reaction in a reaction zone between a fatty material and an alcohol in the presence of said catalyst under conditions effective to convert a substantial proportion of said fatty material to a fatty acid monoester of a polyhydric alcohol, (2) separating monoester formed in said reaction from the mixture resulting from said reaction, said separating being effected while said catalyst is maintained in active form by progressively spreading the mixture resulting from said reaction in a thin film and vacuum distilling said monoester therefrom, and (3) returning said mixture from which said monoester has been separated to said reaction zone, withdrawing separated monoester from said system and adding to said system fatty material and alcohol in amounts correlated to the amount of monoester withdrawn.

14. The cyclic process for preparing monoglyceride of uniform color which comprises establishing a cyclic system comprising the successive steps of (1) reacting in a reaction zone fatty material including a substantial proportion of triglyceride with glycerine in the presence of a strontium soap ester-interchange catalyst, (2) separating in a separating zone monoglyceride formed in the reaction from the composition resulting from said reacting while said composition contains said catalyst in active form, said separating being effected by progressively spreading said composition in a thin film and vacuum distilling monoglyceride from said composition, and (3) returning the resulting monoglyceride-depleted composition containing said active catalyst to said reaction zone, withdrawing from said system monoglyceride separated in said separating zone and adding to said system fatty material and glycerine in amounts correlated to the monoglyceride being withdrawn from said system and effective to maintain predetermined equilibrium conditions in said system.

15. The method of producing monoglycerides which comprises reacting triglyceride fatty material with glycerine in the presence of a metal soap catalyst and thereby forming a composition containing a substantial amount of monoglyceride, separating monoglyceride from the remainder of said composition while said composition contains said catalyst in active form by subjecting said composition to thin film high vacuum distillation, recycling said remainder of said composition including said catalyst in active form, adding to the recycling remainder of said composition amounts of triglyceride fatty material and glycerine substantially equivalent to said monoglyceride separated from said composition, and repeating said reacting, separating, recycling and adding.

16. The process of preparing a monoglyceride product which comprises establishing a continuously circulating system containing ester-interchange catalyst maintained in active form

15

throughout said system, effecting alcoholysis of fatty material with glycerine in the course of said circulating system, and adding fatty material and glycerine to said circulating system and vacuum distilling monoglyceride from said system in correlated amounts effective to maintain predetermined equilibrium conditions in said system, said vacuum distilling being effected from a thin film at a pressure below 100 microns Hg.

17. The process which comprises the sequential steps of reacting a mixture of fatty acid and glycerine in the presence of an ester-interchange catalyst and thereby forming a composition containing a substantial amount of monoglyceride, separating unreacted glycerine from said composition, progressively spreading the resulting glycerine-depleted composition in a thin film and vacuum distilling monoglyceride from said composition while said catalyst is in said composition in active form and leaving a distillation residue containing said active catalyst, and replenishing said distillation residue with additional fatty acid and glycerine in amounts correlated to the amount of monoglyceride distilled from said composition and thereby forming a reconstituted mixture for said reacting, and repeating said reacting and said vacuum distilling with said reconstituted mixture.

18. The process which comprises the sequential steps of reacting a mixture of fatty material and glycerine in the presence of an ester-interchange catalyst and thereby forming a composition containing a substantial amount of monoglyceride, separating a substantial portion of the unreacted glycerine from said composition by phase separation, vacuum distilling substantially all of the remaining unreacted glycerine from said composition without distilling off substantial amounts of monoglyceride, and thereafter progressively spreading said composition in a thin film and vacuum distilling monoglyceride from said composition while said catalyst is in said

16

composition in active form and leaving a distillation residue containing said active catalyst, and replenishing said distillation residue with additional fatty material and glycerine in amounts correlated to the amount of monoglyceride distilled from said composition and thereby forming a reconstituted mixture for said reacting, and repeating said sequential steps with the reconstituted mixture at least until the amount of fatty material and glycerine added to replenish the distillation residue is substantially equivalent to the amount of monoglyceride distilled from the composition resulting from the reacting.

19. The process which comprises the sequential steps of reacting a mixture of vegetable oil and glycerine in the presence of a strontium soap catalyst and thereby forming a composition containing a substantial amount of monoglyceride, separating unreacted glycerine from said composition, progressively spreading said composition in a thin film and vacuum distilling monoglyceride from said composition while said composition contains said catalyst in active form and leaving a distillation residue containing said active catalyst, and replenishing said distillation residue with additional vegetable oil and glycerine in an amount substantially equivalent to the amount of monoglyceride distilled from said composition and thereby forming a reconstituted mixture for said reacting, and repeating said sequential steps with said reconstituted mixture.

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REFERENCES CITED

The following references are of record in the file of this patent:

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Number	Name	Date
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