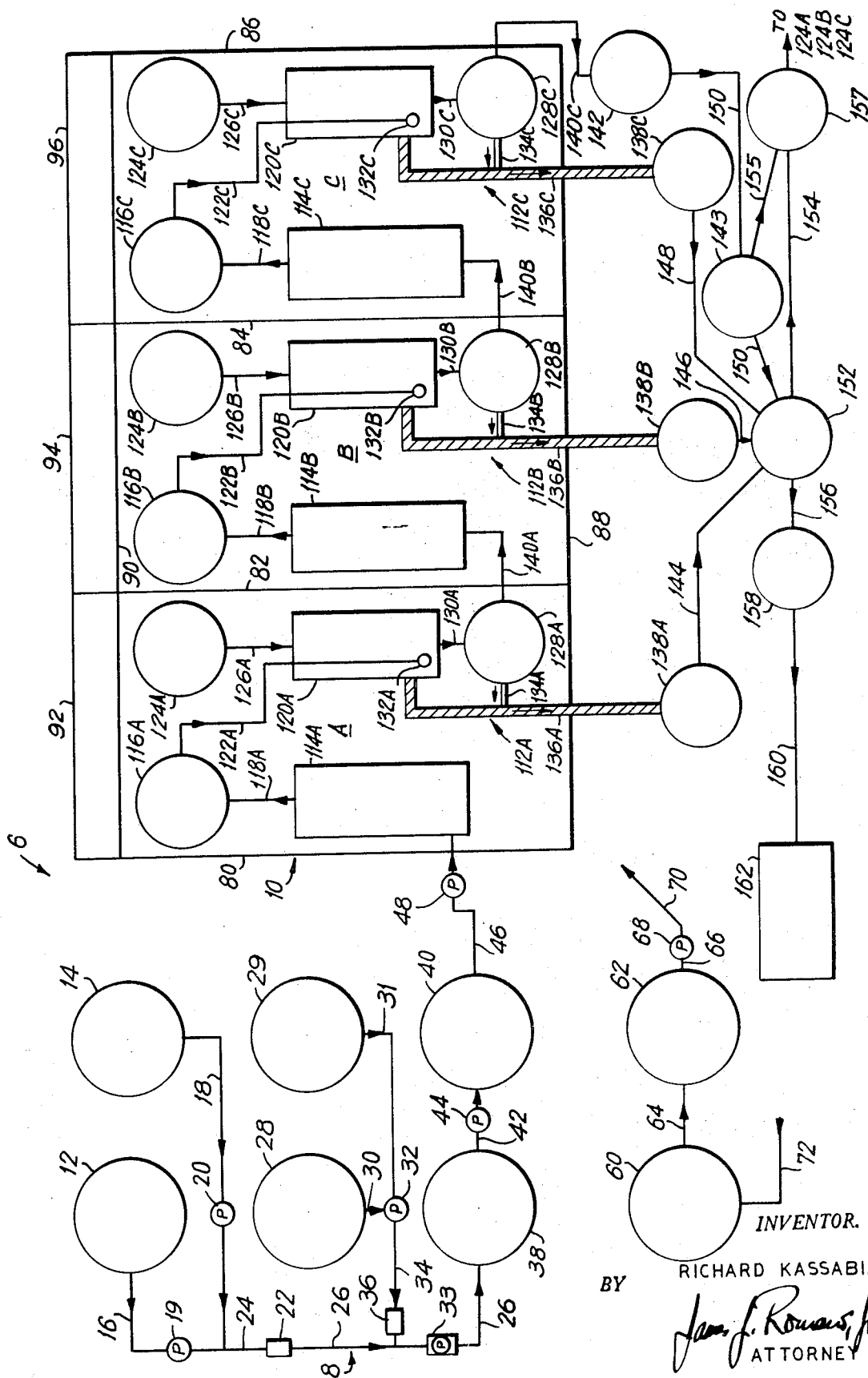


Jan. 4, 1972

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3,632,616

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OF FATTY MATERIALS INTO USEFUL FRACTIONS THEREOF
Filed April 24, 1968



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Filed Apr. 24, 1968, Ser. No. 729,489

Int. Cl. C11b 3/00

U.S. Cl. 260—428.5

12 Claims

ABSTRACT OF THE DISCLOSURE

Apparatus and process are provided for the selective fractionation of fatty materials into useful fractions thereof. Means are included to mix the fatty materials with a solvent blend, in which one of the solvents functions primarily as a diluent, to form a miscella which is then successively subjected to successively lower fractionation temperatures to selectively fractionate for removal the desired, useful fatty material fractions.

This invention relates to a new and improved apparatus and process for the fractionation of triglyceride or fatty acid mixtures into respective, commercially valuable fractions thereof and, more particularly but by no means exclusively, to apparatus and process for the fractionation of palm oil to obtain therefrom a fraction which is eminently suitable for use as a commercial substitute for cocoa butter.

The prior art is, of course, replete with apparatus and processes for triglyceride oil and/or fatty acid mixture fractionations but, in most instances, each of the said prior art apparatus and processes are known to embody disadvantages when employed on commercial scale. More specifically, in some instances the said prior art apparatus and processes do not provide for sufficient fraction separation resolution, to thus render impractical the separation of closely related fractions or, alternatively, require the use of unduly large numbers of process stages to compensate for this lack of sufficient process resolution. In other instances, the said apparatus and processes of the prior art are far too complex and costly in fabrication, operation, and process material requirements for reasonable commercial application. In still other instances, the said apparatus and processes of the prior art are suitable for laboratory or pilot plant use but are inherently of such relatively small capacity as to be inapplicable to large scale commercial use.

It is, accordingly, an object of this invention to provide new and improved process and apparatus for the fractionation of triglyceride and fatty acid mixtures with extremely high fraction resolution.

Another object of this invention is the provision of apparatus and process as above which are of particularly simple and economical design, construction and manner of operation and which require therein the use of only readily available components of proven dependability, and of readily available process operating materials.

A further object of this invention is the provision of apparatus and process as above which are particularly adapted to economical, large-scale commercial use.

A still further object of this invention is the provision of apparatus and process as above which are particularly, though by no means exclusively, adapted to the fractionation from palm oil of a superior quality, eminently suitable commercial substitute for cocoa butter at a cost per unit weight of the latter far below currently prevailing costs of cocoa butter.

As disclosed in detail herein, the apparatus and process of my invention are directed toward the fractionation of

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a triglyceride into a plurality of the commercially valuable fractions thereof. More specifically, multi-stage fractionation apparatus are provided which comprise, for each stage thereof, fractionation means in the nature of a "cold" room wherein are contained fraction crystallization and separation means. A process feed stage is provided and functions to form a miscella of predetermined proportions from the triglyceride oil and a solvent blend, and to feed the said miscella to the first of the fractionation means stages. As utilized herein, one of the solvents of the solvent blend in essence functions as a diluent to dilute the solvent action of the other solvent to thus enable fractionation at relatively high temperatures.

In operation, the respective fractionation stage "cold" rooms, and crystallization and separation apparatus contained therein, are maintained at somewhat, although not greatly, differing temperatures and the miscella is fed to the first fractionation stage. Therein, the desired fraction is removed from the miscella through the combined effects of the "cold" room temperature and the action of the solvent blend on the triglyceride oil, both of which result in the crystallization of the particular fraction commensurate with the "cold" room temperature, and the subsequent separation if the thusly crystallized fraction from the miscella through the operation of the fraction separation means. This process is repeated in each fractionation stage with the miscella being fed, after processing in the first fractionation stage, to the second fractionation stage, etc., and only that fraction of the triglyceride oil which crystallizes, in the presence of the solvent blend, at each "cold" room temperature being separated in the said "cold" room. Means are provided to process the thusly separated fractions in conventional manner to enable the ready commercial utilization thereof.

The above and other objects and advantages of my invention are believed made clear by the following detailed description thereof taken in conjunction with the accompanying drawing wherein:

The figure is a schematic diagram, in flow sheet form, of apparatus constructed in accordance with a preferred embodiment of my invention.

Referring now to the figure, the preferred embodiment of my invention depicted therein constitutes fractionation apparatus, generally indicated at 6, particularly but by no means exclusively adapted to the selective fractionation of palm oil into a plurality of the useful fractions thereof with special emphasis being placed, in this instance, upon the fractionation therefrom of a superior quality, commercially acceptable substitute for cocoa butter at significantly lower cost per unit weight than that of the latter.

To this effect, the apparatus 6 will be seen to include a process blending and feed stage, generally indicated at 8, and a process fractionation stage, generally indicated at 10.

The process blending and feed stage 8 comprises solvent supply tanks 12 and 14, connected as shown by conduits 16, 18 and 24, respectively, to readily adjustable pump means 19 and 20, and readily adjustable liquid metering means 22 of suitable performance characteristics, all arranged in the depicted manner. In most currently contemplated utilizations of the apparatus and process of my invention, solvent supply tanks 12 and 14 will hold different solvents—for example acetone and isopropanol, respectively, as discussed in detail hereinbelow—whereby the readily adjustable pump means 19 and 20 may be adjusted to provide a wide range of solvent blends, by volume, from the solvent supply tanks to the liquid metering means 22 and therefrom to a solvent blend delivery conduit 26 as should be obvious.

For use in the fractionation of triglyceride oils, oil supply tanks 28 and 29 are provided for the supply of triglyceride oils which, in some instances, have been neutralized and washed in conventional manner, and are

connected as shown by conduits 30 and 31 to readily adjustable pump means 32 of suitable performance characteristics. Non-illustrated temperature control means in the nature, for example, of cooling or heating coils are incorporated in each of oil supply tanks 28 and 29 to enable the maintenance of the oils contained therein at desired temperature levels and in the liquid state. Pump means 32 are in turn connected as shown to conduit 26 by conduit 34 extending therebetween, and readily adjustable liquid metering means 36 are connected as shown in conduit 34. In-line mixer means 33 are arranged as shown in delivery conduit 26 downstream of the juncture thereof with conduit 34. Thus may be readily understood whereby proper adjustment of the respective pump means 20 and 32, and liquid metering means 22 and 36, will result in the delivery of a blend of miscella of the distinct solvents from solvent supply tanks 12 and 14, and the triglyceride oil from oil supply tanks 28 and/or 29 of desired volumetric proportions, to a point in conduit 26 just downstream of the juncture thereof with conduit 34.

A miscella blend or make-up tank 38 is connected as shown to conduit 26 downstream of the said juncture, and is provided to insure proper blending of the solvent and triglyceride oil constituents of the said miscella prior to further utilization thereof. To this effect, the said make-up tank will preferably include non-illustrated agitator means to insure proper solvent-oil mixing and will, in addition, preferably include non-illustrated temperature control means to provide for the maintenance of the miscella at a desired temperature level.

A process feed tank 40 is connected to blend tank 38 by conduit 42, and readily adjustable pump means 44 are connected as shown in the latter. In the manner of tank 38, tank 40 also preferably includes non-illustrated agitator and temperature control means, respectively. The tank 40 is connected to the fractionation stage 10 by delivery conduit 46, and readily adjustable pump means 48 are connected in the latter to thus provide for the steady delivery of the miscella, at a desired rate, and at a desired temperature, to the said fractionation stage.

If desired, the process blending and feed stage 8 may further include rework tanks 60 and 62, respectively, series connected as shown by conduit 64, and connected to readily adjustable pump means 68 by conduit 66, and therefrom to an outlet conduit 70. An inlet conduit 72 may be utilized to connect tank 60 to any point in the fractionation stage 10 to thus provide for the storage of the miscella being processed in the event of fractionation stage failure. In like manner, outlet conduit 70 may be connected to any point in the fractionation stage 10 to provide for the re-introduction of the thusly stored miscella thereto after the said failure has been repaired.

Referring now to the fractionation stage 10, the same may be seen to include a plurality of adjacent "cold" rooms or sub-stages A, B, and C, respectively, formed as shown by heavily insulated walls 80, 82, 84, 86, 88, and 90, respectively, each of which is selectively maintainable at a desired temperature through the use of temperature control means as indicated generally at 92, 94, and 96, respectively. The temperature control means will preferably take the form of dual function, heat exchange means which are operable to lower the respective "cold" room temperatures if the desired levels thereof are below the prevailing ambient temperatures, and to conversely raise the said temperatures if the desired levels thereof are above the prevailing ambient temperatures. Thus, for example, means in the nature of large, non-illustrated hot and cold tanks containing heat transfer fluids in the nature of "THERMINOL FR" as marketed by the Monsanto Chemical Company, might be employed to operate the temperature control means 92, 94 and 96 and could, in addition, be called upon to provide both heating and cooling fluid where and if required at other points in the process. Such heat exchange means may, of course, be

operated in part through the use of readily available process steam.

Fractionation apparatus as generally indicated at 112A, 112B and 112C, respectively, are positioned as shown in the respective "cold" rooms, and are of substantially identical construction and manner of operation unless otherwise specifically noted. Thus, only one of the said fractionation apparatus, namely 112A, will be described in detail, it being understood that the correspondingly numbered structural components of the others, which accordingly bear different identifying letter designations, are of the same constructions and manners of operation.

Fractionation apparatus 112A comprises jacketed heat exchange unit means in the nature of scraped surface crystallizer means 114A which are connected as shown to conduit 46 and will thus function to receive the triglyceride oil-solvent miscella from process feed tank 40 and retain it for a period of time sufficient to reduce the temperature thereof to the ambient temperature within "cold" room A.

A slurry mixing, cooling and holding tank 116A is provided and connected as shown to scraped surface crystallizer means 114A by conduit 118A. The tank 116A functions to receive the crystal-miscella slurry from the crystallizer means 114A and retain the former for a period of time sufficient to provide crystal growth while maintaining the same in suspension in the miscella.

Pusher type centrifuge means 120A, including an inlet 132A, are connected as shown, by conduit 122A, to tank 116A to receive the cooled slurry and miscella therefrom.

A wash solvent blend holding tank is indicated at 124A and is connected to pusher type centrifuge means 120A by conduit 126A. The tank 124A functions to receive solvent blend from without the "cold" room A—as described hereinbelow—hold the said solvent blend for a period of time sufficient for it to assume the ambient temperature of "cold" room A, and introduce it, through conduit 126A, to pusher type centrifuge means 120A as a wash solvent blend to maintain a fraction in solution for purposes described in detail hereinbelow.

Self-thinker type centrifuge means are indicated at 128A, and the inlet thereof is connected as shown to an outlet of the pusher type centrifuge means 120A by conduit 130A.

Although described in detail hereinbelow, it is considered well to note at this juncture that the separation of a triglyceride oil fraction from the miscella in "cold" room A is effected by the combined action of the reduced temperature prevailing in the scraped surface crystallizer means 114A, the slurry mixing, cooling and holding tank 116A, and the respective centrifuge means 120A and 128A, due to the respective dispositions thereof in "cold" room A, and the action of the solvent blend on the triglyceride oil, both of which result in the crystallization of the particular fraction, commensurate with the said "cold" room temperature, and resultant centrifugal separation of the thusly crystallized fraction from the still liquid, remaining portion of the miscella through the action of the said centrifuge means.

"Moyno" type pump means 136A are connected as shown to the crystallized fraction output of the pusher type centrifuge means 120A, and a crystallized fraction discharge means 134A extend as shown from the crystallized fraction outlet of the self-thinker centrifuge means 128A to the said "Moyno" type pump means. Thus the triglyceride oil fraction separated from the miscella through the operation of the respective centrifuge means 120A and 128A may be conveyed to the said "Moyno" type pump means.

The "Moyno" pump means extend as shown through cold room wall 88 to discharge into a separated fraction holding tank 138A. Thus, the fraction separated from the triglyceride oil through the operation of the apparatus of "cold" room A, and any small quantity of solvent

blend which may remain therewith, may be transferred to holding tank 138A for further processing.

A miscella discharge conduit 140A extends as shown from self-thinker type centrifuge means 128A, through heavily insulated wall 82, to the inlet of the scraped surface crystallizer means 114B of "cold" room B. Thus the miscella, minus, of course, the triglyceride oil fraction separated therefrom through the operation of the elements of "cold" room A, may be conveyed to "cold" room B for the separation therefrom of another triglyceride oil fraction commensurate with the temperature prevailing in "cold" room B.

Fractionation apparatus 112B and 112C are of substantially the same construction as fractionation apparatus 112A and are, of course, connected in series therewith; with the miscella discharge conduit 140A of self-thinker type centrifuge means 128A extending, as described hereinabove, through cold room wall 82 into connection with the inlet of scraped surface crystallizer 114B, and miscella discharge conduit 140B of self-thinker type centrifuge means 128B similarly extending into connection with the inlet of scraped surface crystallizer 114C.

The "Moyno" type pump means 136B from "cold" room B extend as shown to fraction holding tank 138B, while the "Moyno" type pump means 136C from "cold" room C extend as shown to fraction holding tank 138C.

The miscella discharge conduit 140C from the self-thinker type centrifuge means 128C extends as shown to a processed miscella holding tank 142.

Each of the separated fraction holding tanks 138A, 138B and 138C, and the processed miscella hold tank 142 is connected as shown, by conduits 144, 146, 148 and 150 to solvent stripper means 152 whereby the solvent blend remaining with the said separated fractions and processed miscella may be readily stripped therefrom. An evaporation means 143 is interposed in conduit 150 since a relatively large amount of solvent blend is present in the processed miscella from tank 142. Thus the evaporation means 143 will function to remove most of this solvent blend before the processed miscella is fed to the solvent stripper means 152. Solvent blend return lines 154 and 155 extend respectively from the said solvent stripper and evaporation means for the supply of the solvent blend to a solvent blend reconstitution tank 157. After solvent blend reconstitution, in accordance with the desired solvent blend proportions, the reconstituted solvent blend may, if desired, be supplied for solvent blend wash purposes, to the respective "cold" room, wash solvent blend holding tanks 124A, 124B and 124C.

Deodorizer means 158 are connected to solvent stripper means by conduit 156 and will, of course, function to deodorize the respective separated fractions prior to the transport thereof, as indicated by 160, to fraction collection means 162.

OPERATION

In the utilization of the depicted fractionation apparatus of my invention for the fractionation of, for example, palm oil into a plurality of the fractions thereof, with particular emphasis being placed upon the economical production of a commercially acceptable substitute for cocoa butter, oil supply tanks 28 and 29 are filled with palm oil which has been neutralized and washed in conventional manner to insure that no soap is present therein. Similarly, solvent supply tank 12 is filled with acetone of commercially available grade, and solvent supply tank 14 is filled with isopropanol of 99% purity, again a commercially available grade, it being here emphasized that other and different solvents may find satisfactory utilization in this process as discussed in detail hereinbelow.

This filling of the respective oil and solvent supply tanks may be effected in any convenient manner, as for example through the utilization of non-illustrated feed pump and tank inlet means, it being noted that non-illustrated continuously operable tank filling means may

be employed to maintain a constant supply of palm oil and solvent to the said tanks and thus enable substantially continuous operation of the process. Alternatively, and since in practice the respective oil and solvent supply tanks would be of very large capacity, batch type filling thereof could, of course, be employed.

Prior to the commencement of operation, each of pump means 19, 20 and 32, and liquid metering means 22 and 36 are adjusted to provide a solvent blend of approximately 25% by weight of acetone and approximately 75% by weight of isopropanol, commonly referred to as aciso 2575 by those skilled in this art, to conduit 26, and a blend or miscella of approximately 50% by weight of neutralized palm oil and 50% by weight of the said solvent blend, to miscella make-up tank 38 and, as follows, process feed tank 40. It is here again emphasized, however, that other and different solvent blends may be satisfactorily utilized and that as a general range for the fractionation of palm oil using acetone and isopropanol, solvent blends ranging from 5% to 50% by weight of acetone and 95% to 50% by weight of isopropanol may be used. Too, the proportion of the palm oil fatty material to solvent blend may range from a ratio of one part by weight of palm oil to two parts by weight of solvent blend, to two parts by weight of palm oil to one part by weight of blend.

In addition, and with aciso 2575 as the solvent blend, heat exchanger means 92, 94, and 96 are regulated by the non-illustrated control means associated therewith to provide and maintain temperatures ranging from 70° F. to 80° F. in cold room A, 55° F. to 65° F. in cold room B, and 45° F. to 55° F. in cold room C, respectively. Preferably, said temperatures will be approximately 75° F. in cold room A, 60° F. in cold room B, and 50° F. in cold room C. In this regard it is considered well to note that the approximately 25-75 solvent blend by weight of acetone and isopropanol has been determined to result in very desirable crystallization characteristics, at the respective, relatively high sub-stage temperatures referred to hereinabove, within the respective fractionation apparatus 112A, 112B and 112C; it being noted that the isopropanol appears to function primarily as an acetone diluent in that the use of a significantly larger proportion of acetone would require a lower temperature for the respective sub-stage crystallizations, and especially insofar as the crystallizations of the lower melting point disaturated and/or tri-unsaturated triglycerides are concerned.

This is to say that the diluted solvent power of the acetone (as reduced by the isopropanol addition) causes the respective fatty fractions to be on the verge of precipitation whereby the latter can be selectively separated by the relatively slight, e.g. approximately 15° F., changes in temperature occurring between the respective "cold" rooms A, B and C. Thus, although the isopropanol is normally considered a solvent, it may, in this instance, be referred to as a diluent. Of additional significance is the fact that, since the respective fatty fractions are on the verge of precipitation, the relatively moderate "cold" room temperatures of crystallization can be employed as compared, for example, with the much lower temperatures of crystallization required by the somewhat similar processes of the prior art, whereby the significant economics of operation made possible by the process of the invention are believed made obvious. Further, the use of an approximately 1 to 1 ratio of oil and solvent blend made possible by the process of the invention greatly enhances the commercial feasibility thereof as compared, for example, with the somewhat similar processes of the prior art which requires 3 to 1 or even greater solvent to oil ratios and are thus forced to operate to overcome the significant economic disadvantages inherent in the fractionation of an extremely diluted oil. Too, by the use of the disclosed relatively high "cold" room temperatures, and the oil-solvent blend ratio and attendant avoidance of extreme

oil dilution, the viscosity of the miscella is maintained in a range very conducive to proper miscella flow rates.

A typical plant constructed in accordance with the teachings of this invention for the selective fractionation of palm oil to produce a commercially acceptable substitute for cocoa butter might operate at an overall flow rate of two tons per hour and provide for an approximately three hour retention time for the miscella in each of the "cold" rooms. This would thus require an initial flow rate in the process feed stage 8 of approximately one ton per hour of palm oil and approximately one ton per hour of the solvent blend whereby pump means 20, 32, 44 and 48, and liquid metering means 22 and 36 may be adjusted accordingly. Of course, the removal of a crystallized fraction, for example, through discharge conduit 136A from "cold" room A, would require the addition of sufficient solvent blend to the miscella to retain this flow rate through the remaining fractionation stages, and this would be accomplished through the control of the flow of wash solvent blend from the respective wash solvent blend holding tanks 124A, 124B and 124 C to the respective pusher-type centrifuge means 126A, 126B and 126C, so that the amount of oil removed, in crystallized fraction form from each "cold" room, would be replenished by an approximately equal amount of wash solvent blend. Further, the addition of wash solvent blend as described above would insure that fractions which are not to crystallize in any particular "cold" room will remain substantially in solution and pass therethrough for crystallization in a succeeding cold room, or removal with the processed miscella through conduit 140C from "cold" room C.

In this latter regard it is noted that palm oil contains a relatively small, but very valuable beta carotene fraction, and that the addition of the wash solvent blend will function to retain this carotene fraction in solution for discharge with the processed miscella whereby the latter may, if desired, be further processed through the use of a similar solvent fractionation process to further concentrate the said carotene fraction to significant economic advantage.

Once steady state operational conditions have been reached, operation of the depicted apparatus would thus entail the flow to and through the respective miscella make-up tank 38 and process feed tank 40 of approximately two tons per hour of the miscella with the said tanks functioning to both insure satisfactory mixing of the solvent and oil to form the miscella, and the maintenance of the latter at a temperature above that at which crystallization of the first triglyceride fraction will commence, in this instance a temperature above 80° F.

As the miscella is flowed from tank 40 to the heat exchange unit which takes the form of the jacketed, scraped surface crystallizer 114A of "cold" room A, it will rapidly assume the 75° temperature of the said jacketed crystallizer whereby crystallization of the first fraction hereinafter referred to as S₁ will begin. Accordingly, the scraping action of crystallizer 114A will insure that these crystals which do form will be continuously scraped from the walls thereof for re-introduction to the miscella slurry so as not to interfere with the occurrence of the desired miscella-jacket heat exchange relationship.

The resultant crystal-miscella slurry is then flowed to slurry mixing and cooling tank 116A wherein it is retained for a period of time sufficient to promote further crystal growth. In instances wherein a three hour "cold" room retention time is utilized, the period of retention in the tank 116A might approximate 2.0 hours or less.

Flow of the slurry from cooling tank 116A to pusher-type centrifuge means 120A is then effected whereby the latter will function, through conventional centrifuge operation, to remove the crystallized fraction S₁ from the miscella or mother liquor and deliver the former to the "Moyno" type pump means 136A. The addition of the wash solvent blend to the centrifuge means 120A will enable the washing of the S₁ fraction crystals in addi-

tional solvent blend to insure the removal of substantially all of the remaining miscella therefrom.

The remaining miscella or mother liquor is then introduced to self-thinker type centrifuge means 128A which will operate to remove any remaining, minor quantities of S₁ fraction crystals from the miscella and effect the delivery thereof to "Moyno" type pump means 136A through delivery conduit 134A. This makes clear whereby the combined action of the respective centrifuge means 120 and 128A, and the introduction of wash solvent blend, will insure a substantially complete removal of the crystallized S₁ fraction from the miscella and delivery of the former to the pump means 136A. As a result, the crystallized S₁ fraction will be supplied from "cold" room A, through "Moyno" type pump 136A, to the S₁ holding tank 138A, while the miscella or mother liquor, including the additional solvent blend introduced thereto from wash solvent blend holding tank 124A, will be supplied, through conduit 140A, to the inlet of the scraped surface crystallizer 114B of "cold" room B.

Operation of the fractionation stage 112B in "cold" room B and of fractionation stage 112C in "cold" room C will be substantially as described hereinabove for fractionation stage 112A in "cold" room A. Thus fractionation stage 112B will function to crystallize, at approximately 60° F., and separate from the miscella a second fraction, hereinafter referred to as S₂ and discharge the same through "Moyno" type pump means 136B to S₂ holding tank 138B, while fractionation stage 112C will function to crystallize, at approximately 50° F., and separate from the miscella a third fraction, hereinafter referred to as S₃, and discharge the same through "Moyno" type pump means 136C to holding tank 138C. In addition, fractionation stage 112C will function to discharge the solvent-blend-diluted residual miscella, hereinafter referred to as S₄, to S₄ holding tank 142 through conduit 140C.

The respective separated fractions, S₁, S₂ and S₃, and the residual miscella S₄, are then delivered, through conduits 144, 146, 148 and 150, to solvent stripper means 152 wherein the solvent blend is stripped therefrom and returned, through return conduit 154 and reconstituted solvent tank 157, to the respective wash solvent blend holding tanks 124A 124B and 124C for re-use as described hereinabove.

With regard to the solvent stripping from the fraction or processed miscella S₄, it has been noted that, at the point where approximately half of the solvent blend, containing most of the acetone due to the lower boiling point thereof, has been stripped therefrom, two immiscible layers appear with one of the said layers being highly pigmented to indicate that it contains a relatively high concentration of the beta carotene from the original palm oil. Accordingly, it is envisioned that, through the use of a relatively simple physical separation of the said layers at this point, the subsequent recovery of a beta carotene-rich fraction from the highly pigmented layer or carotene liquid as it may be termed, could be greatly simplified to decided economic advantage.

The respective separated fractions S₁, S₂, S₃ and S₄—the latter, only, being first bleached by non-illustrated bleaching-means—are then delivered through conduit 156 to deodorizer means 158 for deodorization in preparation for the use thereof in foodstuffs and/or pharmaceuticals. After deodorization, the S₁, S₂, S₃ and S₄ fractions are delivered by conduit 160 to fraction collection means, as indicated at 162, which may take any form convenient to the collection and packaging of the said fractions. Preferably the fraction collection means 162 will include flaking drum means to put the respective fractions S₁, S₂ and S₃ in flake form for final collection and packaging, it being understood that the S₄ fraction will be in liquid form at the ambient fraction collection point temperatures.

In practice, this solvent stripping, deodorization and fraction flaking and collection will be accomplished se-

quentially. Thus, and through the use of non-illustrated value or similar flow control means in each of conduits 144, 146, 148 and 150, the system may be arranged, for example, to first deodorize and deliver for collection fraction S₁, then fraction S₂, then fraction S₃ and finally fraction S₄.

For convenience of description, the composition of the miscella at the entrance and exit thereof from each of the fractionation stages 112A, 112B and 112C, and various significant chemical and physical properties of the respective, separated palm oil fractions and residual miscella, are listed directly hereinbelow in Table A.

TABLE A

Exit process Feed Stage 8	Exit fraction			
	Stage 112A	Stage 112B	Stage 112C	Stage 112C
Fraction from each stage	S ₁	S ₂	S ₃	S ₄
Form of fraction	Crystalline	Crystalline	Crystalline	Liquid
Amount recovered per ton, lbs.	100	400	400	1,100
Approximate recovery temp., ° F	75	60	60	-----
Approximate percent palm oil in miscella at this point	60.0	47.5	37.5	27.5
Approximate percent isopropanol in miscella at this point	37.5	39.4	46.8	54.4
Approximate percent acetone in miscella at this point	12.5	13.1	15.7	18.1
Melting point of fraction	60.2	34.0	31.0	10.1
Iodine value of fraction	10.8	30.0	33.7	61.1
Refractive index	-----	1.4565	1.4568	-----
Approximate triglyceride comp.	GS ₃	GS ₂ U	GS ₂ U	GSU ₂ -GU ₃

The S₁ fraction is essentially a tri-saturated fraction and is suitable for all applications wherein hydrogenated flakes and saturated fats of similar nature are utilized. A distinct advantage of the S₁ fraction resides in the fact that it has not been hydrogenated and would thus not contain any isomerized chains as are normally found in hydrogenated fats. As a result, the S₁ fraction will have a lower melting point than tri-saturated triglycerides containing isomerized fatty acids and will be particularly useful, for example, as a binder in peanut butter and/or halvah to prevent oil separation, and in candy coatings. This is to say that the S₁ fraction can satisfactorily perform the functions of hydrogenated oils without giving rise to the elevated melting points encountered with isomerized oils. Fuller discussion of this advantage of the S₁ fraction may be found on page 540A, September Meeting Report of the J. Am. Oil Chemists Soc., vol. 44 of November 1967.

The S₄ function is well suited for use as a salad or margarine oil and again, with an iodine value in the range of 60 to 70, offers the significant advantage of having almost no "iso" acids present since it has also not been hydrogenated. The highly pigmented or carotene liquid layer of the S₄ fraction is, as discussed above, rich in beta carotene and can be used as a highly economical source thereof in the production of beta carotene for use as a food coloring agent. Alternatively, the said carotene liquid, or CL as it is hereinafter referred to, can be bleached and incorporated for use as above with the S₄ fraction.

The S₂ and S₃ fractions are each, as discussed hereinabove, eminently suited for large scale commercial use as substitutes for cocoa butter at prices well below the current prices for the latter. This eminent suitability of the S₂ and S₃ fractions as cocoa butter substitutes, and further information on the chemical properties of the S₁, S₄ and CL fractions for the respective uses thereof discussed above, are believed clearly illustrated by the following Report of Analysis I which presents the results of chemical analyses conducted by a highly qualified, independent chemistry laboratory at my request on my samples of the fractions S₁, S₂, S₃, S₄, CL as produced in accordance with the process of my invention as disclosed herein, and two samples of cocoa butter of commercially available grade. Also presented are the AOCS values for cocoa butter.

REPORT OF ANALYSIS I

Sample identification	Cocoa butter							
	S ₁	S ₂	S ₃	S ₄	CL	A	B	AOCS
Melting point, ° C. (closed cap.)	60.2	34.0	31.8	8.5	11.8	32.1	32.9	30-35
Iodine value (Wijs)	10.82	29.41	33.70	60.65	61.65	46.14	47.17	32-40
Unsaponifiables, percent	0.15	0.30	0.29	0.60	0.52	1.19	0.62	<1
Refractive Index at 40° C.	1.4565	1.4568	-----	-----	-----	1.4575	1.4583	1.456-1.458

Consideration of Report of Analysis I is believed to make clear the eminent suitability of the S₂ and S₃ fractions for use as substitutes for cocoa butter, by making clear the marked similarity therebetween in the critical areas of refractive index and melting point. Although some divergence may be noted between the S₂ and S₃ fraction iodine values and the cocoa butter sample iodine values, this is shown to be of no particular significance by the fact that African cocoa butters, which are in wide commercial use today, exhibit iodine values ranging as do the iodine values of the S₂ and S₃ fractions, from 29 to 34. Also of particular significance, and again as made

clear by consideration of this Report of Analysis I, is the extremely high resolution made possible by the process of the invention despite the relatively small, and thus highly advantageous from an economic viewpoint, fractionation stage temperature differentials employed. This high resolution is illustrated by the relatively small iodine value differentials between the respective S₁, S₂, S₃ and S₄ fractions which clearly establish the ability of the process of the invention to separate out fractions within very narrow iodine value ranges.

The mark of similarity in purely physical properties—which are, of course, of great significance insofar as the feasibility of the commercial utilization of the S₂ and S₃ fractions as substitutes for cocoa butter in applications such as the making of chocolate is concerned—between S₂ and S₃, and cocoa butter are believed clearly illustrated by the following Report of Analysis II which presents the results of dilatometric analyses conducted by the same laboratory and under the same circumstances as described hereinabove with regard to Report of Analysis I.

REPORT OF ANALYSIS II

Sample identification	S ₂	S ₃	Cocoa butter
Solid fat index AOCS (Cd 10-57) at—			
10.0° C.	53.5	58.8	66.9
21.1° C.	43.8	47.0	53.9
26.7° C.	29.4	26.9	16.1
33.3° C.	0	0	0
37.8° C.	0	0	0
40.0° C.	0	0	0

Of particular significance here is the fact that each of the S₂ and S₃ fractions change completely to a liquid, as indicated by the 0 value for the solid fat index—the percent of solids in the fat—in the same narrow temperature range as does commercially available cocoa butter. Thus it is assured that the S₂ and S₃ fractions would be every bit as palatable as cocoa butter. In summary with regard to the remarkable similarity between the S₂ and S₃ fractions and cocoa butter, it may be fairly alleged that sophisticated chemical methods in the nature of gas chromatography or the like would probably be required to distinguish therebetween.

As discussed hereinabove, other and different solvents could find satisfactory utilization in the formation of the solvent blend. Thus, for example, it is believed that any of ethyl acetate, hexane or methyl ethyl ketone could be considered for substitution for the acetone, while it is similarly believed that any of butanol or ethanol could be substituted for the isopropanol. An advantage of the use of acetone and isopropanol does reside in the fact that the same are both relatively economical and readily available in large quantities.

Referring again to the relatively small temperature differentials required between the respective "cold" rooms A, B and C, it may be understood that this makes possible the selective fractionation of each of tri-saturated, di-saturated mono-unsaturated, mono-saturated di-unsaturated, and tri-unsaturated glycerides. Examples of triglyceride oils other than palm oil to which the fractionation apparatus and process of my invention are particularly applicable are tallow and fish oils, and other vegetable oils.

Various changes may be made in the disclosed apparatus and may, for example, take the form of substituting rotary vacuum filter means for the respective centrifuge means.

Although a specific embodiment of the invention has been described in considerable detail for illustrative purposes, many modifications can be made within the spirit of the invention. It is therefore desired that the protection afforded by Letters Patent be limited only by the true scope of the appended claims.

What is claimed is:

1. In a process for the separation of one or more fractions from a fatty material in the nature of a fat or fatty acid, the steps of, mixing said fatty material with a blend of acetone and isopropanol wherein the latter acts as a diluent to dilute the acetone to form a fatty material-solvent blend miscella, subjecting said miscella to the crystallization temperature of said fraction in said miscella to crystallize the latter, and removing said crystallized fraction from said miscella.

2. In a process as in claim 1 wherein, said fatty material and blend are mixed in proportions ranging from a ratio of one part by weight of fatty material to two parts by weight of blend, to a ratio of two parts by weight of fatty material to one part by weight of blend.

3. In a process as in claim 1 wherein, said fatty material and said blend are mixed in a ratio of approximately one part by weight of said fatty material to approximately one part by weight of said blend.

4. In a process as in claim 1 wherein, said isopropanol and said acetone are mixed in proportions ranging from a ratio of one part by weight of acetone to nineteen parts by weight of isopropanol, to a ratio of one part by weight of acetone to one part by weight of isopropanol.

5. In a process as in claim 1 wherein, said acetone and

said isopropanol are mixed in a ratio of approximately one part by weight of acetone to three parts by weight of isopropanol.

6. In a process as in claim 1 wherein, said separation is carried out in three stages with said miscella being initially subjected to a first temperature ranging from 70° F. to 80° F. to crystallize a first fraction for removal therefrom, said miscella minus said first fraction then being subjected to a second temperature ranging from 55° F. to 65° F., to crystallize a second fraction for removal therefrom, and said miscella minus said first and second fractions then being subjected to a third temperature ranging from 45° F. to 55° F. to crystallize a third fraction for removal therefrom.

7. In a process as in claim 6 wherein, said first temperature is approximately 75° F., said second temperature is approximately 60° F. and said third temperature is approximately 50° F.

8. In a process as in claim 3 wherein, said acetone and said isopropanol are mixed in a ratio of approximately one part by weight of acetone to three parts by weight of isopropanol.

9. In a process as in claim 8 wherein, said separation is carried out in three stages with said miscella being initially subjected to a first temperature ranging from 70° F. to 80° F. to crystallize a first fraction for removal therefrom, said miscella minus said first fraction then being subjected to a second temperature ranging from 55° F. to 65° F. to crystallize a second fraction for removal therefrom, and said miscella minus said first and second fractions then being subjected to a third temperature ranging from 45° F. to 55° F. to crystallize a third fraction for removal therefrom.

10. In a process as in claim 9 wherein, said first temperature is approximately 75° F., said second temperature is approximately 60° F. and said third temperature is approximately 50° F.

11. In a process as in claim 1 wherein, said fatty material is palm oil.

12. In a process as in claim 6 wherein, said fatty material is palm oil, and said second and third fractions thereof will constitute a cocoa butter substitute

References Cited

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U.S. Cl. X.R.

23—273 F; 99—122 R