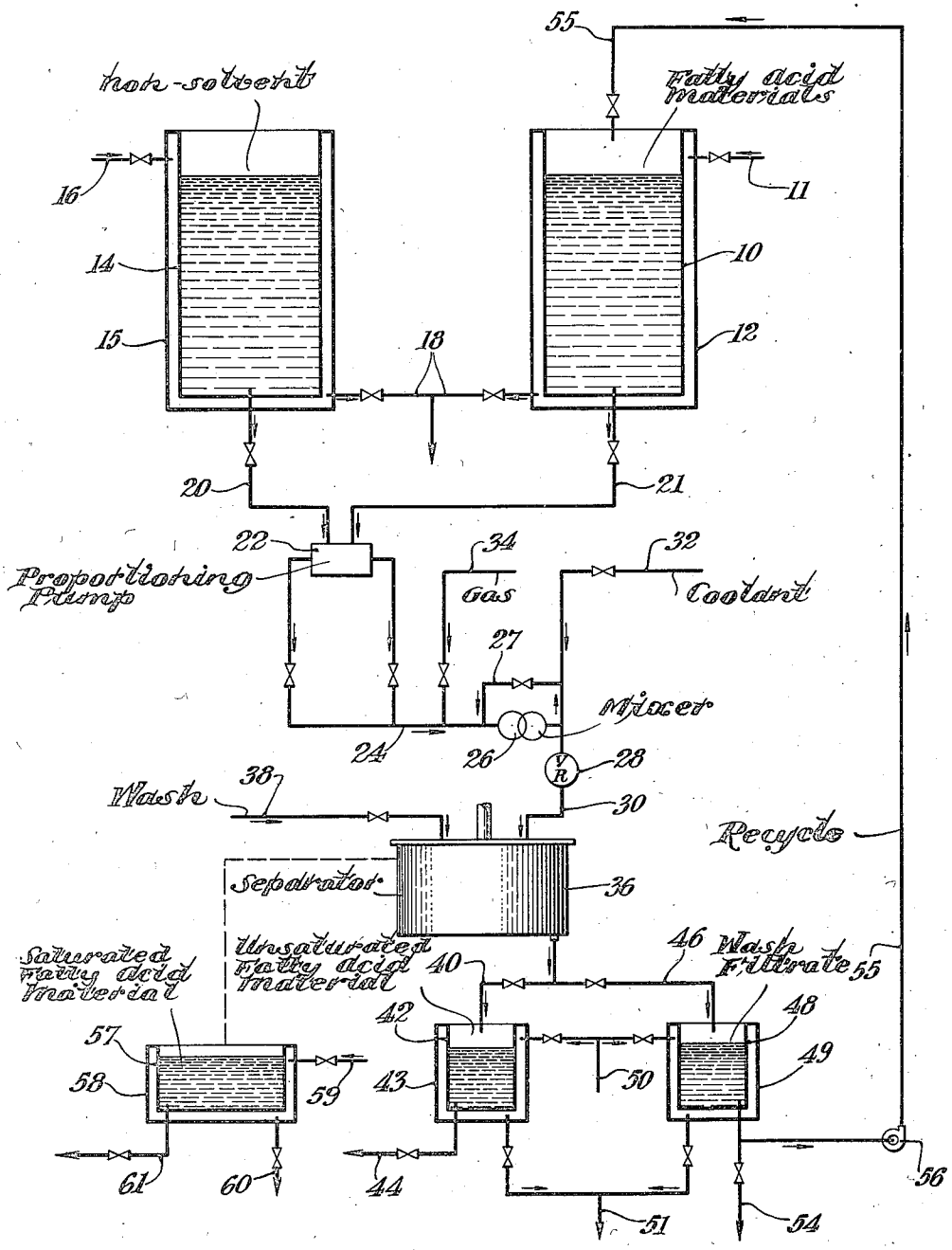
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SEPARATION OF DIFFERENT MELTING POINT MATERIALS

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SEPARATION OF DIFFERENT MELTING POINT MATERIALS

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9 Claims. (Cl. 252—303)

This invention relates to a method for the partial separation of various melting point materials from mixtures thereof and more particularly to the concentration of higher melting point materials from lower melting point materials in a mixture thereof. It is, in a sense, a fractional solidification and separation by the aid of an emulsion whereby separation is facilitated. It is a continuation-in-part and modification of the invention disclosed in my copending application, Ser. No. 274,412, filed May 18, 1939, and entitled "Separation of different melting point materials."

As set forth in the foregoing application, I have discovered that I can obtain a highly effective separation of mixtures of materials having different melting points by forming an emulsion of such a mixture with a non-solvent fluid and thereafter cooling the emulsion to such a temperature that one of the materials to be separated becomes filterable or separable by liquid-solid separation from the other. The non-solvent fluid surrounds the crystals or solid particles and the liquid particles and produces a free-filtering, free-draining, and free-settling system. I then separate the liquid from the solid material and thereafter separate out the non-solvent fluid.

It is well known that vegetable oils such as soybean oil are mixtures of the glycerides of higher fatty acids, some of which are saturated and some of which are unsaturated. The glycerides of the more unsaturated acids, particularly linoleic and linolenic acids, are valuable as drying oils; they have melting points that are comparatively low and are below the freezing point of water. The glycerides of other acids such as palmitic and stearic acids, which are saturated, and oleic acid have a much higher melting point and are commercially known as the saturated or non-drying oils and are useful primarily in the production of soap.

A typical approximate analysis of a mixture of soybean fatty acids as described in "Chemical and Metallurgical Engineering," volume XLII, No. 9, September, 1940, page 618 is as follows:

Fatty acids in soybean oil	Weight	M. P.
	Per cent	*°C.*
Palmitic acid (C_{16}, saturated)	8	61.8
Stearic acid (C_{18}, saturated)	5	68.8
Oleic acid (C_{18}, double bond)	27	15.4
Linoleic acid (C_{18}, 2 double bonds)	57	−11
Linolenic acid (C_{18}, 3 double bonds)	3	-------
	100	

The melting points of the several fatty acids above referred to are taken from Perry's "Chemical Engineers' Handbook" (1934 edition).

The separation of the relatively more saturated and relatively more unsaturated fatty acids or fatty acid glycerides from a mixture thereof is of considerable importance from the commercial standpoint in order that the objectionable qualities of one group do not influence the highly desirable qualities of the other group. In the past, such separation has been accomplished principally by distillation procedures; however, partial decomposition of the fatty acids or their glycerides occurs at the high temperatures required, and it has been found difficult to accomplish a sharp fractionation. Solvent fractionation of such materials has also been proposed, but attempts in this respect have not been entirely satisfactory.

The principal object of my invention is to effect a separation of a mixture of higher fatty acids or their glycerides into the relatively more saturated and the relatively more unsaturated constituents by forming an emulsion of the mixture and accomplishing a liquid-solid separation of the emulsion after cooling.

A more specific object of my invention is to fractionally separate a mixture of the saturated and unsaturated fatty acids and their derivatives obtained from soybean oil by forming a free-filtering emulsion of the mixture in which the higher melting point compounds, which are relatively saturated, are solidified.

In accordance with the preferred form of embodiment of my invention as shown on the attached drawing, which represents a general or typical flow sheet, it will be noted that I provide a tank 10 for receiving a mixture of fatty acids or their glycerides obtainable, for example, from soybean oil. Preferably, this tank is provided with a source of heating medium such as steam, which is conveniently introduced through line 11 into jacket 12 to maintain the mixture in a liquid condition. It is to be understood that, for the purposes of my invention, the material may be either the soybean oil itself or a mixture of the fatty acids characteristic thereof.

A supply of a suitable non-solvent liquid is maintained in tank 14, which is preferably jacketed as at 15 and is maintained at a predetermined temperature by means of the heating medium introduced through line 16. Preferably both materials are maintained at substantially the same temperature, and the condensate from the steam jackets is withdrawn through the lines 18.

Water is the preferable non-solvent liquid for forming the emulsion with the soybean fatty acids. Other liquids such as aqueous salt solutions or low boiling organic liquids may be selected; but it is preferable that the emulsifying material should form a suitable emulsion with the fatty acid materials and should have no substantial solvent or chemical effect thereon. I

have found water satisfactory with the soybean acids, and reference will hereafter be made to this as the emulsifying material.

Predetermined quantities of the non-solvent liquid and the mixture of fatty acid materials are drawn off through lines **20** and **21**, respectively, by the proportioning pump **22**, and the streams are consolidated in the manifold **24** and introduced into mixer **26**. This mixer may be any suitable type of emulsifier or homogenizer, but I find that in many cases a rotary pump is adequate if provided with a by-pass **27** and a relief valve **28**. It will be appreciated that, by adjusting the capacity of the mixer **26**, it will be possible to recycle the material through the mixer as many times as may be desirable so that the resulting material discharged through line **30** is suitably emulsified. I usually find it preferable to add an inert, non-solvent gas through line **34** to the mixture in manifold **24** to aerate or otherwise expand the emulsion, and in some cases I can use the gas to the exclusion of the liquid non-solvent. I prefer to directly cool the emulsion as by the direct introduction of a coolant at **32** into the by-pass line **27** where this is possible; otherwise, indirect cooling may be applied satisfactorily. Both direct and indirect cooling may be used if desired. Where direct cooling is used, it is frequently desirable to select as the cooling medium a non-reactive emulsifying material such as that contained in tank **14**.

Having established a suitable emulsion with a large interfacial area between the crystallized material and the non-solvent, I am able to obtain a very high rate of separation in the liquid-solid separator **36**. Although this is indicated as a centrifugal type, it may be either a centrifugal filter having a foraminous filter basket, or it may be a clarifier with a solid bowl. It is, of course, in the contemplation of my invention that the separator, the primary function of which is to separate liquids from solids, may be any desired type including a pressure or a vacuum filter of the continuous or other type and having filter discs or a drum as may be desired. In some cases gravity settling may be practiced.

In the preferred form of embodiment of my invention and particularly for economical high speed operation, I find a centrifugal basket filter having a perforated rotating basket lined on the interior with a foraminous filtering medium to be most effective. This is rotated at such a speed that a high separating force, which may be in the range of 500 to 1,000 times gravity or more, is obtained.

The cake formed is normally a fine-grained, free-filtering, porous type; and, inasmuch as the filtering force is gravitational by nature, the entire cake is submitted to the filtering force without an external pressing action. It is, of course, possible and usually desirable to wash such cake with a wash liquid as from line **38**. Such wash liquid will readily permeate the cake. A uniform washing and drying of the cake are thus possible. The wash liquid may be either a diluent for the liquid material of the emulsion or a liquid which is inert with respect thereto and a non-solvent therefor. An inert and non-solvent wash liquid does not dilute the liquid part of the emulsion and yet serves as a suitable displacing medium which is especially effective where the solid part of the emulsion is porous as in the instant case.

With the proper temperature control, the liquid unsaturated fatty acid material that is separated from the saturated fatty acid material filter cake is withdrawn through the line **40** into tank **42** provided with a suitable heating jacket **43**. The unsaturated low melting point fatty acid material is removed through the line **44**.

The wash liquid filtrate may be withdrawn through the line **46** by suitable manipulation of the valves into tank **48** provided with jacket **49**. This filtrate may be either recycled through line **55** by means of the pump **56** or otherwise discharged as through the line **54**. It will be understood that tanks **42** and **48** are maintained at the desired temperature by the introduction of a heating medium at **50** and the discharge thereof at **51**.

The filter cake, which is the higher melting point material, may be removed from the separator **36** and collected in the tank **57**, which is also provided with a heating jacket **58**, to which a heating medium is supplied at **59** and from which the heating medium is removed at **60**. The saturated, high melting point fatty acid material is removed through line **61**.

As an example of the operation of my invention on soybean acids, an emulsion of soybean fatty acids having a melting point of 64° F. and an iodine number (Wijs) of 137.5 was made with water. Equal parts of water and acids were used, and the resulting emulsion was chilled as indicated on the following table:

	Run No.			
	4	5	6	7
Filtering temp.....°F..	70	60	50	45
Cake:				
Iodine number (Wijs).....	82.3	87.2	111.2	129.3
Melting point.....°F..	105	101	82	71
Cloud point.....°F..	.....	180	.....	144
Yield.....wt. %..	12.5	19.5	39	77
Filtrate:				
Iodine number (Wijs).....	144.9	149.3	155	155
Melting point.....°F..	52	46	28	23
Cloud point.....°F..	155	137	.....	65
Yield.....wt. %..	87.5	80.5	61	23

In a once-through operation without washing, it was possible to obtain a filtrate having an iodine number of 149.3 with an 80.5% yield or a filtrate having an iodine number as high as 155 with only a somewhat smaller yield. With a recycle ratio of filtrate to initial charge of two or three to one and with suitable washing with a non-solvent wash liquid, it is possible to obtain substantially theoretical yields.

The separation of soybean oil into fractions comprising respectively the relatively more saturated and the relatively more unsaturated constituents thereof can also be effected by my process. The soybean oil is emulsified with water for example, and the resulting emulsion is chilled to such a temperature that a portion of the soybean oil solidifies. Upon separation in a suitable manner, a filter cake comprising the relatively more saturated constituents and a filtrate comprising the relatively more unsaturated constituents are obtained.

The use of a diluent to thin out the soybean oil prior to emulsification thereof may be desirable to facilitate the formation of the emulsion. In such case a liquid which is a solvent for the soybean oil is used. Such liquid, however, should have a solvent power for the soybean oil such that when the emulsion is chilled only the relatively more unsaturated constituents remain in solution.

One of the principal advantages of the invention is that the admixed materials are separated by

their melting points rather than by their boiling points, and constant boiling mixtures or tarry decomposition products resulting from distillation are avoided. The process is far quicker and more economical than the usual methods of crystallization since it makes possible a better separation with truly continuous operation and accurate temperature control.

While I have shown and described a preferred form of embodiment of my invention, I am aware that modifications may be made thereto; and I, therefore, desire a broad interpretation of my invention within the scope and spirit of the disclosure herein and of the claims appended hereinafter.

I claim:

1. The method of separating a mixture of materials selected from the group consisting of higher saturated and unsaturated aliphatic fatty acids and their glycerides into fractions which are respectively relatively more saturated and relatively more unsaturated, which comprises maintaining the mixture in liquid condition, forming an emulsion thereof with a non-solvent, non-reactive fluid, cooling said emulsion to a temperature such that the relatively more saturated compounds are substantially solidified in a highly dispersed phase, and thereafter making a liquid-solid separation of the chilled emulsion to separate the solidified compounds from the remaining liquid material.

2. The method of separating a mixture of materials selected from the group consisting of higher saturated and unsaturated aliphatic fatty acids and their glycerides into fractions which are respectively relatively more saturated and relatively more unsaturated, which comprises maintaining the mixture in liquid condition, forming an emulsion thereof with a non-solvent, non-reactive liquid, cooling said emulsion to a temperature such that the relatively more saturated compounds are substantially solidified, and thereafter centrifugally separating the remaining liquid material from the solidified compounds.

3. The method of separating a mixture of materials selected from the group consisting of higher saturated and unsaturated aliphatic fatty acids and their glycerides into fractions which are respectively relatively more saturated and relatively more unsaturated, which comprises maintaining the mixture in liquid condition, forming an emulsion thereof with a non-solvent, non-reactive liquid and an inert, non-solvent gas, cooling said emulsion to a temperature such that the relatively more saturated compounds are substantially solidified, and thereafter making a liquid-solid separation of the chilled emulsion to separate the solidified compounds from the remaining liquid material.

4. The method of separating a soybean fatty acid mixture into fractions which are respectively relatively more saturated and relatively more unsaturated, which comprises maintaining the fatty acid mixture in liquid condition, forming an emulsion thereof with water, cooling said emulsion to a temperature such that the relatively more saturated fatty acids are substantially solidified, and thereafter making a liquid-solid separation of the chilled emulsion to separate the solidified fatty acids from the remaining liquid material.

5. The method of separating a soybean fatty acid mixture into fractions which are respectively relatively more saturated and relatively more unsaturated, which comprises maintaining the fatty acid mixture in liquid condition, forming an emulsion thereof with water and an inert, non-solvent gas, cooling said emulsion to a temperature of approximately 50° F. to solidify the relatively more saturated fatty acids, and thereafter making a liquid-solid separation of the chilled emulsion to separate the solidified fatty acids from the remaining liquid material.

6. The method of resolving mixtures of soybean fatty acids into the relatively more saturated acids and the relatively more unsaturated acids, which comprises maintaining in liquid condition a mixture of soybean fatty acids having an iodine number of approximately 137 (Wijs), forming an emulsion of said acids with water, the acids and the water being present in substantially equal quantities, cooling the emulsion to a temperature of approximately 50° F. to solidify the relatively more saturated fatty acids, and separating the solidified relatively more saturated fatty acids comprising approximately 40% of the charge and having an iodine number of approximately 110 from the remaining liquid containing the relatively more unsaturated fatty acids comprising approximately 60% of the charge and having an iodine number of approximately 155.

7. The method of separating soybean oil into fractions which are respectively relatively more saturated and relatively more unsaturated, which comprises maintaining the soybean oil in liquid condition, forming an emulsion thereof with water, cooling said emulsion to a temperature such that the relatively more saturated glycerides in said oil are substantially solidified, and thereafter making a liquid-solid separation of the chilled emulsion to separate the solidified glycerides from the remaining liquid material.

8. The method of separating soybean oil into fractions which are respectively relatively more saturated and relatively more unsaturated, which comprises maintaining the soybean oil in liquid condition, forming an emulsion thereof with water and an inert gas, cooling said emulsion to a temperature such that the relatively more saturated glycerides in said oil are substantially solidified, and thereafter making a liquid-solid separation of the chilled emulsion to separate the solidified glycerides from the remaining liquid material.

9. The method of separating soybean oil into fractions which are respectively relatively more saturated and relatively more unsaturated, which comprises dissolving the soybean oil in a liquid diluent, forming an emulsion thereof with water, cooling said emulsion to a temperature such that the relatively more saturated glycerides in said oil are substantially solidified, and thereafter making a liquid-solid separation of the chilled emulsion to separate the solidified glycerides from the remaining liquid material.

AUGUST HENRY SCHUTTE.