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PRODUCTION AND PURIFICATION OF
FATTY OILS

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This invention relates to the production and/or purification of fatty oils by a process involving solvent extraction and subsequent precipitation of the oil from the solution by cooling.

One object of the invention is to provide an improved method of freeing the extracted fatty oil from the solvent. Another object of the invention is to provide a method of removing coloring matter, oxidized material, phosphatides, etc., from the fatty oil. Still another object is to provide a method for separating a fatty oil into various fractions of different degrees of unsaturation. Other objects of the invention will become apparent as the description thereof proceeds.

By the term "fatty oils" as used herein and in the appended claims is meant oils which are derived from fatty oil containing raw material of vegetable or animal origin and which are regarded chemically to be the glycerol esters of high molecular weight aliphatic acids, either saturated or unsaturated and embrace the drying, semi-drying and non-drying oils.

Fatty oils are, at present, produced from fatty oil containing raw materials of either vegetable or animal origin by cooking and allowing the oil to settle out; by pressing, or by solvent extraction. The present invention is concerned with improvements in the methods involving solvent extraction. For the purpose of this description of the present invention a solvent process is considered to be any process in which the fatty oil in the course of its preparation and/or purification is dissolved in solvents and subsequently separated therefrom. Thus, if the oil is pressed from vegetable or animal material and subsequently dissolved in solvents it is considered a solvent process and the improved methods of the present invention are applicable thereto.

In the prior art, fatty oil is recovered from a solvent solution by distillation of the solvent by the application of heat, vacuum, or both heat and vacuum. Thus, the recovered fatty oil contains any non-volatile impurities which may be contained in the solution whereas the solvent is recovered substantially pure.

I have now found that when a solvent solution is sufficiently cooled, as by the use of artificial refrigeration, the fatty oil will precipitate substantially completely out of the solution in a practically pure state as an easily filterable solid mass which may be readily separated from any contaminating impurities which remain dissolved in the solvent. I have further found, when cooling fatty oils of the drying and semi-drying types according to the present invention, that at different temperatures various fractions of the oil differing from each other particularly with respect to degree of unsaturation separate out of the solution. Hence, the present invention provides a means for obtaining a great variety of different

oil fraction from the same fatty-oil containing raw material.

I am aware that the prior art teaches that a portion of the saturated fatty oils may be removed from the bulk of the fatty oils by chilling either the oil itself or a solution thereof in a suitable solvent and subsequently removing the precipitated solids by filtration. Where solvents are used the bulk of the oil is then recovered, according to these prior art methods, by distilling the solvent, leaving the non-volatile impurities in the fatty oil. In contradistinction to such methods, the present invention contemplates a precipitation of the bulk of the fatty oil in solid condition and the subsequent separation thereof from the solvent which contains the impurities by filtration.

Any solvent commonly used in extracting fatty oil from vegetable or animal oil containing raw materials and which does not solidify at the temperatures employed may be used in practicing the present inventions. Such solvents will include the coal tar derivatives, e. g. mineral spirits, petroleum derivatives, e. g. petroleum spirits, petroleum ether, etc., natural derivatives, e. g. turpentine, the alcohols, e. g. methyl, ethyl, isopropyl, propyl, etc., the ketones, e. g. acetone, methyl-ethyl ketone, etc., the ethers, e. g. ethyl ether, etc., the acetates, e. g., ethyl acetate, etc., the halogenated paraffins, e. g. carbon tetrachloride, chloroform, etc. Since, the only requirements of a solvent for use in practicing the invention are: (1) that it be capable of dissolving the fatty oil, and (2) that it be substantially non-freezing, i. e. it will not solidify at the low temperatures employed, a wide choice of solvents is permitted without departing from the scope of the invention.

I have found that substantially all of the fatty oil in solvent solution will be precipitated when the solution is cooled to temperatures between about -40°C . and -70°C . When drying oils, such as linseed oil, are treated according to the present invention, the degree of unsaturation of such oils may be enhanced. For instance, I have found, that the more saturated constituents of the drying oil tend to precipitate at higher temperatures, for instance, between about -20°C . and about -40°C . than the more unsaturated constituents. When linseed oil is treated at a temperature of about -65°C . over 90% of the oil will be recovered in a highly unsaturated condition. The present invention, therefore, by enhancing the degree of unsaturation is especially adapted for improving the drying qualities of drying oils.

The general preferred method of practicing my invention is first to extract the fatty oil from the vegetable or animal fatty oil containing raw material by any suitable means, either with or without the use of solvents. Such methods may

include Soxhlet extraction, continuous extraction, dissolving decanted oil, soaking, etc. If no solvent has been employed the raw oil is dissolved in a solvent. The fatty oil solution is filtered to remove suspended, insoluble material. The solution may then be cooled either to precipitate at once the bulk of the dissolved oil or to bring about fractional crystallization of the oil into portions of varying degrees of saturation. The oil is precipitated in a solid, easily filterable form, separated from the liquid solvent containing the dissolved impurities in any convenient manner, as by decantation or filtration. The solid oil is then melted, and freed from any residual small amounts of solvent by known procedures. The separated remaining solvent may be purified by distillation and the residual material recovered.

Example I

Crushed flaxseed is treated with warm acetone until practically all the linseed oil has been extracted. Sufficient acetone is used so that the resulting solution will contain about 10% linseed oil. The solution is cooled to -25°C . and filtered. About 3% of the linseed oil, i. e., the more saturated portion thereof will precipitate. The precipitate will also contain waxes, phosphatides, saturated glycerides, mucilaginous materials, etc. This fraction is discarded or used as a by-product. The remaining solution is then cooled to -68°C . and filtered. This will remove from solution all but about 4% of the oil. The portion of the oil remaining in solution is composed mainly of coloring matter, oxidized material, etc. The acetone is recovered by distillation and the residue may be considered a by-product.

The oil which precipitates out at -68°C . is the main fraction and consists of about 92% of the total oily material in the seed. It is freed if necessary from retained acetone by vacuum and heat treatment. This oil differs from that prepared by ordinary methods in that it contains no ash, phosphatides or waxes. Also, the unsaponifiable matter, coloring material and oxidized oil are materially reduced. It has many advantages over ordinary raw oil; it will not "break" on heating—i. e. will not precipitate out the dissolved non-oleaginous matter, has a higher iodine number, is lighter in color, and need not be aged to remove mucilaginous material, it dries slightly faster, and can be bleached with clay to a very light color.

Example II

A solution of 10% linseed oil in acetone is cooled to -38°C . and filtered, then the solution chilled to -75°C . and filtered. The following fractions were obtained.

Precipitating temp.	Percent weight	Iodine number
$^{\circ}\text{C}$.		
20 to -38	11.8	121
-38 to -75	85.4	196
(1)	2.8	191

¹ Not precipitated.

The iodine number of total oil in the seed was 186. Thus, the middle fraction contains a high percentage of the total oil with an increased iodine number. Therefore, the oil has been fractionated, so that a large proportion of the oil has an iodine number of 10 points higher than the total oil. This is considered an advantage when the oil is to be used as a paint vehicle. Fur-

ther, the oil has been purified by this treatment. By choosing other precipitating temperatures, fractions of oil having higher or lower iodine numbers may be obtained.

Example III

Tung oil is extracted from the tung apple with acetone. A 10% solution of the oil is cooled to -65°C . At that temperature 96% of the tung oil has precipitated from the solution. After filtering and freeing from the retained solvent, a very much lighter oil containing substantially no impurities is obtained than may be manufactured by pressing or extraction and distillation of the solvent.

Example IV

Castor oil is extracted from the crushed castor bean using a quantity of acetone so that the resulting solution will contain 10% castor oil. The acetone oil solution is then cooled to -65°C . and the precipitated oil recovered as in the previous examples. 94% of the total castor oil is obtained in this manner which is lighter in color and purer than is obtained by pressing.

It will be seen from the foregoing description and the illustrative examples given that my invention comprises broadly a method of purifying fatty oils by refrigeration, and embraces a means for the fractional crystallization of fatty oils which contain saturated and unsaturated constituents whereby the relative proportion of unsaturated constituents is raised while that of the saturated constituents is reduced.

I claim:

1. Method for the treatment of fatty oils containing saturated and unsaturated constituents which comprises cooling a solution of a fatty oil in a non-freezing solvent to a temperature at which the more saturated constituents will precipitate out of solution, separating from the solution the precipitated more saturated constituents together with precipitated impurities, and further cooling the solution to a temperature at which the major portion of the remaining more unsaturated constituents precipitate out of solution.

2. Method for the treatment of fatty oils containing saturated and unsaturated portions which comprises cooling a solution of a fatty oil in a non-freezing solvent to temperatures between about -20°C . and down to -40°C ., separating from the solution the precipitated more saturated constituents together with precipitated impurities, further cooling the solution to temperatures between -40°C ., and down to about -70°C . until the major portion of the remaining more unsaturated constituents are precipitated out of solution and thereafter separating the precipitated material from the solvent.

3. Method for the production of highly unsaturated linseed oil which comprises extracting flaxseed with acetone, filtering the resulting solution, cooling it to about -25°C ., separating therefrom precipitated solids, further cooling the solution to about -68°C . and separating the precipitated, highly unsaturated linseed oil from the solvent.

4. Method according to claim 1 in which the fatty oil treated is linseed oil.

5. Method according to claim 1 in which the fatty oil treated is tung oil.

6. Method according to claim 1 in which the fatty oil treated is castor oil.

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