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SEPARATION AND PURIFICATION OF FATTY ALCOHOLS

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1 Claim. (Cl. 260—643)

This is a continuation-in-part of my co-pending application for "separation and purification of fatty acids and glycerides," filed March 19, 1959, Ser. No. 800,404.

This invention relates to the separation and purification of fatty alcohols. More particularly, it relates to the recovery, separation, and refining of saturated fatty alcohols and unsaturated fatty alcohols from mixtures containing both saturated and unsaturated fatty alcohols.

A very important feature of this invention resides in the use of a particular solvent selected from a particular group of solvents to separate and/or purify the desired products, which solvent may be used within moderate temperature ranges. These temperature ranges comprise highs which may be characterized as warm, and those which may be characterized as "room temperature." The exact temperatures involved are of course more particularly pointed out below, but this general description indicates one value of the invention, in eliminating the necessity of extreme temperatures.

Many fatty alcohols, both saturated and unsaturated, are well known in the art, and it is not necessary to exhaustively list here the many known fatty alcohols, nor to set forth their various sources. In addition, the uses and importance of fatty alcohols are well known in the art, and it is not deemed necessary to set those forth herein.

It is an object of this invention to separate, refine, and purify saturated fatty alcohols from mixtures containing saturated and unsaturated fatty alcohols.

It is an object of this invention to separate, purify, and refine unsaturated fatty alcohols from mixtures containing saturated and unsaturated fatty alcohols.

It is another object of this invention to recover, separate, and purify fatty alcohols by means of differential solution in a particular solvent selected from a group of solvents.

It is another object of this invention to separate, refine, and purify fatty alcohols, by a solvent process, minimizing the temperature ranges required, the amount of solvent required, the cost of the process, and resulting in quantitative separations.

A still further object of this invention is to separate a mixture of saturated and unsaturated fatty alcohols into fractions which are respectively relatively rich in solid or high melting-point components and respectively relatively rich in liquid or low melting-point components.

Other aims and objects of this invention are made apparent in the following disclosure and claim.

The most important aspect and feature of this invention depends on the process of utilizing the differing solubility of the materials to be separated, as set forth above, in a solvent selected from the group consisting of nitrated members of the methane (paraffin) series, whose general formula is $C_nH_{2n+1}NO_2$, and isomers thereof. Of this group the preferred members are those in which n equals 4 or less, that is, nitro-methane, nitro-ethane, nitro-propane, and nitro-butane. For example, suitable solvents include 1-nitropropane,

$$CH_3CH_2CH_2NO_2$$

and the isomer 2-nitropropane, $(CH_3)_2CHNO_2$.

In any one of these solvents, the unsaturated fatty alcohols are completely, substantially completely or very

highly soluble at room temperatures, while the saturated fatty acohols are completely, substantially completely, or very highy insoluble at room temperatures.

The solvents recited above are individually referred to herein as "solvent" or "nitrated paraffin series solvent."

In general, the method of this invention comprises dissolving saturated fatty alcohols mixed with unsaturated fatty alcohols, in warm solvent and then cooling to room temperature while stirring constantly. The saturates then crystallize out in easily filterable form. The mixture is filtered. The filter cake containing the saturates are then washed out with the same solvent, and the residual solvent is recovered by the usual methods, as set forth below.

The filtrate, containing the unsaturated fractions, is then distilled under vacuum to recover the solvent by usual methods. The filter cake may be rid of the solvent by distillation under vacuum, or by steam distillation.

The exact temperatures involved in the practice of this method are not deemed quantitatively essential to the invention, but it is observed that the "warm" or higher temperature is approximately 50 degrees C. or higher. The "room temperature" or lower temperature is generally between approximately 12 and 16 degrees C.

It is found that to obtain satisfactory commercial saturated fatty alcohol fractions from the mixture, it is necessary to subject the filter cake of saturates to an additional throughput. That is, the filter cake obtained as described above is redissolved and recrystallized and filtered again. The filter cake obtained from this second throughput is the commercial product. The filtrates obtained from each of the two throughputs constitute commercially satisfactory products. It is of course desirable that the minimum amount of solvent be used in the process. It has been found that a ratio of 4 parts of solvent to 1 part of mixed saturated and unsaturated fatty alcohols is a satisfactory and preferred approximate ratio. Obviously, greater ratios of solvent may be used in the process. The exact preferred ratio set forth herein is not to be considered a necessary element of the invention in itself. Depending on the material to be processed, it is apparent that the ratio of solvent may be reduced somewhat from the preferred ratio, and as a matter of fact the exact ratio used is not critical. As less solvent is used, below the ratio set forth as preferred, the feasible practice of the process, becomes much more difficult.

The practice of this method is made clearer by consideration of the following example.

Example I

A sample was provided of a mixture of fractions of saturated fatty alcohols and unsaturated fatty alcohols, said mixture having been obtained from sperm oil. The sample had an acid value of 1.5. It had a saponification value of three. It had an iodine value of 51–55, and was mainly cetyl alcohol and oleyl alcohol.

One part of the mixture was mixed with four parts of nitropropane, and was warmed. The fractions dissolved. The mixed solvent and fatty alcohols were then cooled while being stirred, to approximately 12 degrees C., at which point crystallization of the saturated fractions occurred. The mixed solvent and fractions was then filtered.

The filter cake, comprising mainly saturates, was redissolved in the same solvent, in the same ratio; that is, one part material to be treated, 4 parts solvent, and warmed. The mixture of solvent and material to be treated was then cooled again to approximately 12 degrees C., during constant stirring, at which point recrystallization had occurred. The filter step was again applied.

The filtrates resulting from each of the two recrystallization steps were combined. The solvent which was admixed with the filtrate was removed by vacuum distillation, and the solvent admixed with the filter cake was also removed by vacuum distillation.

The liquid fraction, or filtrate (after removal of solvent) had a saponification value of 2, and an iodine value of 82. This fraction comprised mainly oleyl alcohol.

The solid fraction (after removal of solvent) showed a saponification value of 0, and an iodine value of 2.8. This fraction comprised mainly cetyl alcohol.

It is understood that additional repeated throughputs may be performed, resulting in still higher degrees of separation or purification.

In this specification, the terms, "separation," "purification," and "refining," are loosely used in an interchangeable and equivalent manner. In common usage, the terms "purification" and "refining" would refer to the removal of relatively minor portions of undesirable material from the material treated, but it is apparent that such processes are merely one form of the broader process of "separation."

The example cited above is merely illustrative of many performed experiments. The same process as set forth in the example was carried out using others of the named solvents and their isomers with equivalent results. It has been found that homologues of the nitrated paraffin (methane) series above nitrobutane are not as satisfactory for use in the process, although the same differential solvent effect is present. Also, the homologues above nitrobutane are much more difficult to obtain commercially.

A characteristic of this group of solvents which makes them remarkably apt for this process is that the solubility of saturates in them at room temperature is almost nil. This makes for very high degrees of separation or purification, which may be characterized as quantitative.

Other known solvents are miscible with water, tending to azeotropism. The recovery of such solvents from the water is difficult. The solvents of this invention are

partially azeotropic, but are not miscible with water. The solvents of this invention entirely dissolve the material to be treated at slightly elevated temperatures, yet provide quantitative separation at room temperatures. The lack of the necessity of refrigeration below room temperature is important.

Another advantage of the process of this invention is that the crystals obtained in the filter cake are much better than those obtained by other processes for ease in filtering. The need for filter aids may often be eliminated in the practice of this invention.

The scope of this invention is to be determined by the appended claim, and is not to be limited by the specific examples and description above, which are intended to be exemplary and not limiting.

I claim:

The separation of a mixture comprising mainly cetyl alcohol and oleyl alcohol comprising dissolving one part of said mixture warm in approximately four parts of a solvent consisting of one of the nitrated members of the methane series of hydrocarbons having the general formula $C_nH_{2n+1}NO_2$ wherein n is an integer from 1 to 4, cooling to approximately 12 degrees C. while constantly stirring, and filtering out crystallized solid matter, mainly cetyl alcohol, whereby the original said mixture is separated into saturated and unsaturated fractions.

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