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METHOD FOR THE SEPARATION OF
STEARIN AND OLEIN FROM FATTY ACID
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4 Claims. (Cl. 87—4)

The present invention relates to a method for the separation of stearin and olein from fatty acid mixtures of all kinds containing them.

Hitherto stearin has generally been prepared by the so called double press method. This method takes considerable time and requires expensive apparatus. Apart from this, not all fatty acids can be expressed by this method. Moreover, it is necessary to endeavour to obtain a crystalline structure by mixing different fatty acids, so as to permit of the pressing operation. Further, olein, on account of its high melting point and its little crystalline form can only be worked up with difficulty to liquid olein.

Attempts have frequently been made to replace this pressing operation by employing solvents out of which to crystallize the stearin upon cooling. These however have been found to be impracticable owing to certain difficulties. The use of alcohol as a solvent has many times been proposed. Since, however, for reasons of economy the solvent, alcohol, must be recovered, the alcohol is distilled off and upon distillation an esterification of the alcohol with stearin or olein takes place so that the end products are valueless.

In British Specification No. 156,259, it has been proposed to separate lower from higher molecular weight fatty acids by making use of solvent. Alcohol was proposed as a solvent and it was proposed to reduce the power of the solvent to dissolve the high molecular acids, by addition of water or to precipitate the higher molecular weight fatty acids from a solution of the fatty acid mixture by addition of water. Further a method has been proposed in which liquid sulphur dioxide alone or in mixtures with other solvents was employed for washing out unsaturated fatty acids from fatty acid mixtures. This method necessitates a complicated apparatus and demands special precautionary measures on account of the troublesome and noxious odour. It is also not practicable to use petroleum benzine as a solvent for fatty acids, from which to crystallize out stearin upon cooling, since the fatty acids harden as a paste, from which no olein can be filtered off.

It has now been found according to this invention that stearin and olein can be practically and economically recovered from fatty acids of all kinds by crystallizing their solutions in oxygen free hydrocarbons such as petroleum benzine, benzene, toluene, chlorinated hydrocarbons and the like, or mixtures thereof, if before cooling, water is added to the fatty acid solution in

such a manner that it is dispersed colloiddally or as an emulsion in the solution.

Various methods of colloiddally dispersing or emulsifying the water in the fatty acid solution may be employed. For example, alcohols, or other bodies containing considerable quantities of water, and which dissolve wholly or partially in water as well as in the solvent or the fatty acids, may be added to the solution. Upon cooling the solid fatty acids separate out in an easily filterable form.

If high percentage alcohol is employed it dissolves without cloudiness. Since, however, very large quantities are necessary in order to attain the desired object, then upon distilling off the solvent, partial esterification would take place, whereby the end products would be useless. If, on the other hand, the operation is carried out with water only and without alcohols, a paste is obtained which filters very badly and only incompletely.

Apart from alcohols, ketones, aldehydes and so forth, emulsifiers can be employed such as sulphonation products of organic acids, alcohols, ketones and so forth, their esters or salts and also salts of fatty acids. Emulsifiers may also be used, together with alcohols, ketones, aldehydes or the like, for introducing the necessary water. The first group, namely the sulphonation products are employed in aqueous solution, whilst the salts of fatty acids are actually formed in the fatty acids by stirring in solutions or suspensions in water of oxides, hydroxides, or carbonates of metals or nitrogenous bases, the water being bound in colloidal form in the fatty acids.

If the fatty acids to be employed already contain water then account must be taken of this fact in order to effect colloidal dispersion of the water in the fatty acid solution when adding the alcohols, by employing higher concentrations.

In some cases the fatty acids to be employed may already contain an emulsifying agent, for example a small percentage of soap and in that case the addition of water alone permits of the formation of a colloidal solution or emulsion.

The wet crystalline mass obtained upon cooling the fatty acid solution is treated in a filter press. The olein remains in the solvent and can be easily filtered off. Hard cakes of the solid fatty acids remain in the filter press. Both products are freed from the solvent in known manner.

In this process the filter cloths can be used for a month without being clogged or stopped.

In order that the invention may be well understood

stood the following examples will be given by way of illustration only:—

Example 1

5 1000 kg. of melted twitchelled bone fatty acids with the following analysis numbers:

| | |
|-----------------------|--------|
| Acid number | 197 |
| Saponification number | 205 |
| Solidification point | 34° C. |
| 10 Iodine number | 49 |

are stirred together with 700 kg. commercial extraction spirit fractionated from crude petroleum with a boiling point of 105 to 175° C., in suitable vessels with cooling jackets and slowly operating stirring mechanism. 50–70 kg. of 75% methyl alcohol are added whilst stirring, to the clear solution at 35° C., whereupon a colloidal cloudiness is produced. The stirring is slowly continued with strong cooling until the temperature of the fatty acid solution is about 10–12° C. at which temperature it becomes a wet crystalline mass or pulp. The pulp is passed through filter presses at a moderate pressure. Very firm cakes are formed in the presses whilst the clear olein containing mother-liquor runs away.

The stearin cakes are taken out of the filter presses and melted. Thereupon the greater part of the added dilute alcohol is separated and is used with the next charge. All of the methyl alcohol still contained is distilled off from the cakes at about 90° C. and after this the solvent is driven off by blowing steam through. After distilling off the solvent the stearin has the following analysis numbers:

| | |
|---------------|-----------|
| Melting point | 52–54° C. |
| Iodine number | 5–7 |

If a higher melting point and a lower iodine number is to be obtained, the cakes coming from the filter presses are thoroughly stirred at 10–12° C. with about the same quantity of the aforesaid spirit as previously employed for about an hour, and passed again through the filter presses, the filtrate being used for a fresh charge. The stearin cakes treated as above described, after driving off the solvent now have the following constants:

| | |
|------------------|-----------|
| Melting point | 56–58° C. |
| 50 Iodine number | 2–3 |

The mother-liquor from the first pressing is cooled further to about 3° C. for separation of the small quantities of stearin still contained in the solution, whereby a further small crystallization takes place, and then filtered. This stearin is specially treated since it contains oxystearic acid which during the subsequent distillation is converted into iso-oleic acid. The filtrate is then freed from solvent whereby first of all, as before, methyl alcohol is obtained. There remains an olein with a solidification point of 10–11° C., which by cooling and filtering can be brought to any technically usual solidification point.

In the above example the following yields were obtained:—

| | |
|--|-----|
| | Kg |
| Stearin of melting point 52–54° C. | 440 |
| Intermediate product | 80 |
| 60 Olein of emulsification point 10–11° C. | 470 |
| 70 Lesses | 10 |

Example 2

Operating as in Example 1, 1000 kg. of twitchelled bone fatty acids are reacted with 30 litres of a 25% aqueous ammonia solution and the emulsification is carried out with vigorous stirring. The fatty acids so pretreated are stirred cold at 35° C. with 800 litres of heavy benzine of boiling point 100–125° C. The stirring is slowly continued with strong cooling until the temperature of the fatty acid solution is about 10–12° C. at which temperature it becomes a wet crystalline mass or pulp. The pulp is passed through filter presses at a moderate pressure. Very firm cakes are formed in the presses whilst the clear olein-containing mother-liquor runs away. Upon driving off the solvent from the stearin cakes the small percentage of fatty acid ammonia splits up again into fatty acid and ammonia.

Whilst in the foregoing specific examples of the application of this invention have been described it is to be understood that various modifications may be made without departing from the scope thereof.

I claim:—

1. The herein described method of recovering stearin and olein from fatty acid mixtures, which method comprises dissolving the fatty acid mixtures in an organic solvent selected from the class consisting of hydrocarbons free from oxygen, incorporating water in said solution and forming a colloidal dispersion or emulsion of the water in said solution, cooling the solution and thereby crystallizing its stearin content and recovering the crystallized stearin.

2. The herein described method of recovering stearin and olein from fatty acid mixtures, which method comprises dissolving the fatty acid mixtures in an organic solvent selected from the class consisting of hydrocarbons free from oxygen, introducing a soap containing water into said solution and forming a colloidal dispersion or emulsion of the water in said solution, cooling the solution and thereby crystallizing its stearin content and recovering the crystallized stearin.

3. The herein described method of recovering stearin and olein from fatty acid mixtures, which method comprises dissolving the fatty acid mixtures in an organic solvent selected from the class consisting of hydrocarbons free from oxygen, introducing alcohol containing water into said solution and forming a colloidal dispersion or emulsion of the water in said solution, cooling the solution and thereby crystallizing its stearin content and recovering the crystallized stearin.

4. The herein described method of recovering stearin and olein from fatty acid mixtures, which method comprises dissolving the fatty acid mixtures in an organic solvent selected from the class consisting of hydrocarbons free from oxygen, introducing into said solution a water emulsifying sulfonated organic product containing water and forming a colloidal dispersion or emulsion of the water in said solution, cooling the solution and thereby crystallizing its stearin content and recovering the crystallized stearin.

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