It is well known that it is difficult and sometimes impossible to separate fatty alcohols by distillation from the soaps obtained in the preparation of the same. Effectively, even if a good vacuum is employed, the higher alcohols will distill off at high temperature and in these conditions the soap is liable to react with said alcohols and form such decomposition products as unsaturated hydrocarbons, resin, tar, etc. Moreover, depending on the soap-content of the mixture, abundant and persistent froths are formed which make distillation quite difficult. Finally, there always remains a certain percentage of undistilled fatty alcohols in the residual soap.

It has been attempted to eliminate these inconveniences by using other methods of separation than distillation, for instance by washing the mixture of soap and fatty alcohols with water while taking advantage of the insolubility of heavy alcohols in water to separate same from the water-soluble soap.

However, it is found that in order to carry out such a separation such large amounts of water must be used that the separation proves to be almost impossible; effectively, within quite wide limits, soaps will act as emulsifiers, and emulsions of alcohols in soap water are obtained which it is almost impossible to resolve.

Applicant has now found, according to the present invention, that all the aforesaid inconveniences can be eliminated by dissolving the mixture of soap and heavy alcohols in a suitable solvent and then washing the solution with water. Under these conditions, with suitable amounts of water added to the solution in the solvent, there are obtained two layers: a layer more particularly loaded with solvent and which contains the alcohols and an aqueous layer enriched in soap.

It will be appreciated that by repeating the operation several times it is possible in this manner to separate all the heavy alcohols from the soaps.

It will also be appreciated that the performance of the said separation in continuous operation becomes quite easy since it is only necessary to run the soap and heavy alcohol solution and the wash water in opposite directions.

It is advantageous to use a low-boiling solvent in order that it can be separated easily by distillation from the heavy alcohols or the aqueous soap solution.

As solvents for the fatty alcohols there may be used such aliphatic or cyclic alcohols as propyl alcohol, butyl alcohol, amyl alcohol, lauryl alcohol, cyclohexanol, aliphatic or cyclic hydrocarbons, gasoline, benzene, toluene, cyclohexane, ketones, etc., or mixtures of such solvents.

On the whole, the present invention has for its object a method of separating heavy alcohols from their mixture with the soap obtained in preparing the same by causing the heavy alcohols to pass into a water-insoluble solvent in which soap is less soluble than in water.

The invention is applicable to all alcohols and alcohol mixtures whose molecular weights are higher than C6, and in particular to alcohols obtained by reducing coconut oil, palm oil, sperm oil, fish oils and to synthetic alcohols.

Various examples of the application of this invention are given hereinafter for the purpose of illustrating and by no means of limiting the invention.

(1) The product from the reduction in a butyl alcoholic medium of 250 kg. of fatty acids derived from palm oil or coconut oil, that is, about 1500 kg., is first of all washed with 300 litres of water in order to remove the sodium hydroxide.

The material resulting from this washing step is admixed with 600 kg. of benzene and 600 kg. of water. The mixture breaks up into two layers, viz.:

An upper layer weighing about 1000 kg.
A lower layer weighing about 1100 kg.
About 2% of soap are contained in the upper layer, and 7.4% in the aqueous layer.

In this case the coefficient of separation related to the soap comes out as 3.7 and it will be appreciated that by repeating the washing operation several times it is possible to completely separate the soap from the heavy alcohols.

Thus, by using another 600 litres of water in a further washing of the alcohol-containing layer, the amount of soap would become 10 times larger in the water than in the alcohol-containing layer.

Since the variation of the coefficient of separation then makes the washing the easier as the amount of soap is lower, practically after three times, no soap is present any longer in the alcohol-containing layer and the heavy alcohols are stripped therefrom.

(2) 3.7 parts by weight of spermacetum are saponified with 8 parts by weight of potassium hydroxide dissolved in 5 parts by weight of butanol.

To the solution there are added 100 parts by
volume of butanol and 5 parts by volume of xylene and to the mixture there are admixed 100 parts by volume of water.

The mixture breaks up into two layers and the coefficient of division of the soap amongst the two layers is 2.6 to the advantage of the aqueous layer.

The washing operation is repeated several times till the soap is completely separated from the heavy alcohols. After three washes there remains no soap in the alcoholic layer; the alcohols are stripped therefrom.

(3) 9 parts by weight of lanolin are saponified with two parts by weight of potassium hydroxide dissolved in 10 parts by weight of butanol.

The product obtained is admixed with 80 parts by volume of butanol, 20 parts by volume of toluene and to the whole there are added 280 parts by volume of water.

The coefficient of division of the soap amongst either layer is then 1. The operation is continued in the same manner as in the preceding examples.

The same saponification product as above, if admixed with a mixture of 100 parts by volume of acetone and 10 parts by volume of xylene will give a coefficient of division of 1.9.

(4) 5 parts by weight of beeswax are treated with 5 parts by weight of potassium hydroxide in 10 parts by volume of amyl alcohol and then admixed with 100 parts by volume of amyl alcohol and 20 parts by volume of xylene and then with 100 parts by volume of water.

The coefficient of division amongst either layer is 1.1. The operation is continued thereafter in the same manner as in the preceding examples.

(5) 25 parts by weight of palm oil dissolved in 100 parts by weight of lauryl alcohol are reduced with sodium.

The raw product is washed with water in order to eliminate the sodium hydroxide as a 36° Bé. solution; the remaining oil, that is, about 150 parts by volume, is admixed with 100 parts by volume of water.

The soap content of the oily portion is four times less than that of the aqueous portion. The operation is continued like in the preceding examples.

(6) 250 parts by weight of palm oil dissolved in 100 parts containing 10% of a 100–140° C. gasoline fraction are reduced with sodium. After washing with water to eliminate the sodium hydroxide as a 36° Bé. solution the remaining oil is admixed with an equal volume of water.

The amount of soap present in the oil is then 14 times less than the amount contained in the water. The operation is continued in the same manner as in the preceding examples.

The soap present in the water is three times larger than the amount of soap in the oil.

The operation is then continued in the same manner as in the preceding examples.

It is particularly advantageous to perform the method according to this invention in an apparatus designed for continuous operation equipped with one or several columns and to combine the washing with the necessary distillation apparatus so that the solvent or solvent mixture can be used in a closed circuit.

Two examples of apparatus enabling to carry out the present invention are shown diagrammatically for the purpose of illustration and by no means of limitation in Figs. 1 and 2 of the appended drawing.

In the embodiment illustrated in Fig. 1, a tank B is fed through a pipe 1 with the mixture of heavy alcohols and soap which are to be separated. The solvent recovered in the continuous operation flows into the tank through a pipe 14. The mixture of solvent, soap and alcohol is flowed through a pipe 2 into a washing column L. The water necessary for separating the soap is fed into the column through a pipe 3. Said water flows out through the pipe 7 as an aqueous soap solution that contains solvent and it is led into a distillation column D in which the soap solution is to be separated from the solvent and from which the solvent recovered is led into the tank B through the pipe line 9 and the condenser C.

The solution flows out at 8. The solution of heavy alcohols in the solvent is discharged from the washing column L at 5 and is led into a column D in which said heavy alcohols are separated from the solvent.

The heavy alcohols are discharged at 8 while the solvent recovered flows out at 10, is condensed in the condenser C and is led back into the tank B through the pipe 14. However, a portion of said solvent is used: on one hand by means of a pipe 4 to remove the last traces of heavy alcohols from the soap in the solution discharged through pipe 7, on the other hand to provide for the retrogradations necessary for the operation of the columns D1 and D2 and this, through the pipes 12 and 13. Besides, this system may be equipped with automatic regulators.

Another way of carrying the invention into effect consists in separating the alcohols from the soap and to continuously regenerate the fatty acids present therein so that the apparatus will deliver the alcohols to be separated and the regenerated fatty acids. This can be accomplished in the apparatus shown in Fig. 2.

The tank B is supplied through a pipe 1 with the mixture of heavy alcohols and soap to be separated. Said tank is fed through a pipe 14 with the solvent reclaimed in the continuous operation. Said mixture of solvent, soap and alcohol is led through a pipe 2 into a washing column L.

The water necessary for separating the soap is fed into the column through a pipe 3. Said water flows out through a pipe 7 as an aqueous solvent-containing soap solution which is fed into a washing column L.

A mineral or organic acid is fed into said column L through a pipe 15 to wash the soap solution and set free the fatty acid; it flows out as an aqueous solution of sodium salts. The solvent-containing fatty acid is discharged at 17 and led to a distillation column D in which the solvent is separated from the fatty acids and then recycled as in the preceding example while the fatty acid recovered is discharged at 18.

A return of the said acids to the bottom of column L (pipe 16) is provided in order to strip the salt solution discharged at 16 from any solvent that might remain dissolved therein.

As to the solvent that contains the heavy alco-
hols and that is discharged from the column L, its circuit is the same as in the former case.

The solvent containing the heavy alcohol is discharged at 5 and led to a distillation column D. The heavy alcohol flows out at 6 and the solvent at 10. It is condensed from the top of condenser C, from which it is discharged through the pipes 14 and 11 to perform the same function as in diagram No. 1, that is, washing in the bottom of column L, retrogradation in the distillation apparatus, the solvent being returned to tank C. It is quite evident that any other suitable layout might be used; in particular, the columns 1 and 2 might be divided into two sections each of which would do the work that is effected in the upper and the lower portions of each said columns.

What I claim is:

1. A continuous method for the separation of high molecular fatty alcohols from their mixtures with soap obtained in the preparation of the same comprising admixing a high molecular alcohol and soap mixtures with a solvent for the high molecular alcohol in which the soap is less soluble than in water and introducing the resulting mixture into the middle portion of a washing-tower, introducing wash-water into the top portion of said wash-tower, introducing aqueous soap solution containing a small quantity of the solvent from the bottom of the tower, feeding the same into the top part of a first distillation column, condensing solvent vapors that escape from the top of the said first distillation column, tapping solution of high molecular alcohol from the top of the aforesaid washing tower, leading said last named solution into a second distillation column in which the solvent is separated from the high molecular alcohol, condensing solvent vapors that escape from the top of the said second distillation column, and re-using the solvent condensate from the first and the second distillation columns to treat further amounts of the high molecular alcohol and soap mixture.

2. A continuous method for the separation of high molecular fatty alcohols from their mixtures with soap obtained in the preparation of the same comprising admixing a high molecular alcohol and soap mixtures with a solvent for the high molecular alcohol in which the soap is less soluble than in water and introducing the resulting mixture into the middle portion of a washing-tower, introducing wash-water into the top portion of said wash-tower, introducing aqueous soap solution containing a small quantity of the solvent from the bottom of the tower, feeding the same into the top part of a first distillation column, condensing solvent vapors that escape from the top of the said first distillation column, tapping solution of high molecular alcohol from the top of the aforesaid washing tower, leading said last named solution into a second distillation column in which the solvent is separated from the high molecular alcohol, condensing solvent vapors that escape from the top of said column, introducing a smaller flow of the same solvent into the lower portion of said wash-tower, discharging aqueous soap solution containing a small quantity of the solvent from the bottom of the main washing-tower, introducing said last named solution into an additional washing-tower, introducing an acid into the top of said washing tower, tapping the thus formed aqueous alkali salt solution from the bottom of said additional washing-tower, tapping the solvent solution of fatty acid from the top of said tower, and separating the fatty acid from the solvent in a second distillation column, condensing solvent vapors that escape from the top of the same, and re-using the solvent condensate from the columns to treat further amounts of the high molecular fatty alcohol and soap mixtures.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

<table>
<thead>
<tr>
<th>Number</th>
<th>Name</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,814,684</td>
<td>Youz</td>
<td>July 14, 1931</td>
</tr>
<tr>
<td>2,070,518</td>
<td>Rosser et al.</td>
<td>Feb. 23, 1937</td>
</tr>
<tr>
<td>2,080,111</td>
<td>Bump</td>
<td>May 11, 1937</td>
</tr>
<tr>
<td>2,148,846</td>
<td>Von Retze et al.</td>
<td>Feb. 28, 1939</td>
</tr>
<tr>
<td>2,193,321</td>
<td>Liethe</td>
<td>Mar. 12, 1940</td>
</tr>
<tr>
<td>2,287,128</td>
<td>Pirkle</td>
<td>June 23, 1942</td>
</tr>
<tr>
<td>2,421,040</td>
<td>Shields et al.</td>
<td>May 27, 1947</td>
</tr>
</tbody>
</table>