The major portion of sugar cane oil thus consists of saturated and unsaturated fatty acids, and these acids occur both as glycerol esters and free acids. The sugar cane oils are further characterized by the following typical analyses:

<table>
<thead>
<tr>
<th>Sample A</th>
<th>Sample B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Saponification No.</td>
<td>147</td>
</tr>
<tr>
<td>Acid No.</td>
<td>96</td>
</tr>
<tr>
<td>Ester No.</td>
<td>128</td>
</tr>
<tr>
<td>Hydroxyl Value</td>
<td>2.1</td>
</tr>
</tbody>
</table>

In accordance with my invention, mixtures of high molecular weight alcohols are prepared from such sugar cane oils by simultaneously treating the oil with alkali metal and reducing alcohol, followed by hydrolysis of the reduced mixture. The chemical reactions involved can be depicted as follows:

\[ R{-}(\text{CH}_2)_n{-}(\text{OH}) + \text{H}_2\text{O} + 12\text{M} \rightarrow 2\text{R}{-}\text{CH}_2{=}\text{OM} + 6\text{R}{-}\text{OM} + \text{CH}_3{-}\text{OM} \]

where \( R \) is a carbon chain having from 10 to 30 carbon atoms, \( R' \) is a lower alkyl radical, and \( M \) is an alkali metal. In carrying out the above reactions to prepare the alcohol mixtures, a preferred method utilizes alkali metal in the form of subdivided particles. Alkali metal dispersions, which are well known to the art, are therefore admirably suited for this purpose. Such dispersions are prepared by fusing alkali metal in an inert hydrocarbon and subjecting the entire mixture to vigorous agitation. The temperature of the mixture is maintained at above the melting point of the alkali metal during the agitation, and the resulting product is a dispersion or suspension of finely divided particles of alkali metal. A variety of inert liquids are suitable for use as dispersing media in preparing such dispersions. Examples of typical dispersing media include toluene, xylene, benzene, petroleum fractions, heavy alkylate, and the like. Still other dispersing media can be employed, the above merely representing a list of those most commonly used. The ratio of alkali metal to dispersing medium employed in forming dispersions can vary, and dispersions having metallic concentrations from trace amounts up to 60 per cent by weight are common. For use in my invention, however, it is preferred to utilize a dispersion having a metal concentration of between 20 and 50 per cent by weight.

In carrying out the reduction reaction to prepare high molecular weight alcohol mixtures from sugar cane oil, it is preferred to utilize about 5 per cent excess of alkali metal over that theoretically required to accomplish the reduction. Although any of the alkali metals such as potassium, sodium, lithium, and the like can be used, I prefer sodium. The quantity of alkali metal required to
give a 5 per cent stoichiometric excess can readily be calculated and is a sum of the following three quantities:

\[(A) \times (1.05) \times (4M) \times (W) = \text{lbs. metal for ester} \]
\[(D) \times (10)^{10}\]

\[(B) \times (1.05) \times (M) \times (W) = \text{lbs. metal for free acid} \]
\[(D) \times (10)^{10}\]

\[(C) \times (1.05) \times (M) \times (W) = \text{lbs. metal for hydroxyl compounds} \]
\[(D) \times (10)^{10}\]

where

\[A = \text{Ester number of oil} \]
\[B = \text{Acid number of oil} \]
\[C = \text{Hydroxyl value of oil} \]
\[M = \text{Atomic weight of alkali metal} \]
\[W = \text{Weight of oil used (lbs.)} \]
\[D = \text{Molecular weight KOH} \]
\[E = \text{Molecular weight OH} \]

As previously indicated, the reduction reaction is carried out using alkali metal and a reducing or hydrolytic alcohol. In general, the lower aliphatic secondary and tertiary alcohols are best suited for this purpose and include such alcohols as tertiary or secondary butyl alcohol, methyl isobutyl carboline, and the like, these alcohols merely representing those mostly commonly used. The amount of hydrolytic alcohol employed in my reaction is preferably 5 per cent in excess over the stoichiometric amount, and this amount can be calculated as follows:

\[(A) \times (1.05) \times (2P) \times (W) = \text{lbs. reducing alcohol} \]
\[(D) \times (10)^{10}\]

where \(F = \text{molecular weight of reducing alcohol, and } A, W, \text{ and } D \) are as designated above.

As can be seen from the foregoing equations illustrating the chemical reactions involved, alcaloates are produced in the reduction reaction. In order that these alcaloates be kept in solution, the reaction should be carried out in an inert solvent medium; that is, a medium inert to the alkali metal. In general, aromatic hydrocarbons such as toluene and xylene are well suited for this purpose, although other materials such as petroleum fractions, dibutyl ether, and tertiary amine compounds and the like can be employed. The amount of inert solvent used can vary, although it is generally preferred to utilize a solvent-to-oil ratio from between about 1 to 1 and 5 to 1.

In accordance with one embodiment of my invention, the reactants are brought together by initially preparing in a suitable vessel an alkali metal dispersion of the type described above, utilizing the calculated amount of alkali metal. This dispersion is then heated sufficiently so as to maintain a vigorous reflux. The heating is then discontinued, and a solution of sugar cane oil, reducing alcohol, and solvent is added to the dispersion at a rate sufficient to maintain steady reflux. The addition is continued for a period of from one-half hour to two hours, after which time the mixture is heated for an additional period of from 15 minutes to an hour so as to insure complete reaction. This reduction mixture is then hydrolyzed by adding the mixture to a hydrolysis flask charged with a weight of water calculated to produce about a 20 per cent caustic solution. For example, when using sodium as the reducing metal, this amount of water is calculated as follows:

\[\text{Wt. } \text{H}_2\text{O} = 174 \times \text{Wt. Na} \times 0.08 \times \text{Wt. Na} \times 0.20\]

After a cook period of approximately one-half hour the agitation is stopped, and the hydrolyzed mixture separates into two phases, a caustic phase and an organic phase containing the alcohol products. The phases can then be separated by decantation and the organic layer containing the alcohols washed with a hot 0.5 per cent solution of sodium chloride. The solvent and reducing alcohol are then removed from the alcohol products by distillation.

The following examples, in which all parts and percentages are on weight basis, will serve to further illustrate my invention.

**Example I**

A reduction solution was prepared from 150 parts sugar cane oil (sample "A" described above), 72.8 parts methyl isobutyl carboline, and 476 parts toluene. A sodium dispersion was prepared in a reaction flask equipped with a stirring mechanism and reflux condenser by fusing 38.5 parts sodium in 173 parts toluene and subjecting the mixture to vigorous agitation for a period of about 30 minutes. After the dispersion was formed, heat was applied sufficient to establish a vigorous reflux. While maintaining reflux temperature, the reduction solution was added to the dispersion over a 30-minute period and the entire mixture cooked at reflux for a period of one hour. Following this cook period the mixture was hydrolyzed by addition to 272 parts water. Since the starting material contained an unusually high concentration of free fatty acid, an emulsion was encountered upon hydrolysis. In order to break this emulsion and effect a phase separation, the toluene-water azetropie was distilled off and the emulsion broken when a total of 595 parts toluene and 118 parts water was removed. During the azetroic distillation, 50 parts of water was added. When the emulsion was broken, the mixture separated into two clear liquid layers. The organic layer containing the product alcohols was decanted and washed with about 100 parts of a 0.5 per cent sodium chloride solution. The remaining toluene and methyl isobutyl carboline were then removed by heating to 130° C. at 15 mm. Approximately 140 parts product consisting essentially of a mixture of high molecular weight saturated and unsaturated alcohols was obtained representing a yield of approximately 59 per cent of theoretical.

As can be seen from the above example, in carrying out the process the presence of free fatty acids in the starting material somewhat complicates the hydrolysis step in that emulsions may be encountered. This can be minimized to a great extent by initially esterifying the free acid of the oil prior to reduction. This esterification can be conveniently accomplished by heating the oil under reflux conditions with a lower aliphatic alcohol such as methanol or ethanol so as to form the corresponding ester of the free acids. The following examples will serve to illustrate this embodiment of my invention.

**Example II**

Two hundred parts of sugar cane oil (sample "A") was mixed with 15 parts methanol and 0.5 part sulfuric acid and refluxed for a period of two hours. After treatment the excess methanol was removed by fractionation and the treated oil found to have the following analysis:

<table>
<thead>
<tr>
<th>Saponification No.</th>
<th>144</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid No.</td>
<td>5</td>
</tr>
<tr>
<td>Ester No.</td>
<td>139</td>
</tr>
<tr>
<td>Hydroxyl value</td>
<td>1.5</td>
</tr>
</tbody>
</table>

A reduction solution was then prepared by adding 112 parts methyl isobutyl carboline and 476 parts toluene to the methanol treated oil. This reduction solution was then added to 56.8 parts sodium dispersed in 215 parts toluene, and the reaction carried out as described in Example I. In this case, since the free acid had been largely esterified, the emulsion which formed upon hydrolysis was quickly broken by means of the azotropic distillation described in Example I. This process was worked up as in Example I, and approximately 181 parts of alcohol mixture was obtained, representing a 97 per cent yield.
The crude sugar cane oil obtained by conventional processes normally contains a residual amount of wax, generally of the order of 10 per cent by weight. Although it is not necessary in my process that this residual wax be removed prior to the treatment with alkali metal, this can be done by merely treating the oil with a wax solvent such as acetone and allowing the solution to stand for a period sufficient to permit complete extraction. The solution is then decanted from the wax. The following example will serve to further illustrate this embodiment of my invention.

**Example III**

Approximately 400 parts of sugar cane oil (sample "P") containing about 10 per cent wax was mixed with 1,280 parts of acetone and the mixture vigorously agitated. After standing for a period of 24 hours the acetone solution of the oil was decanted and the acetone removed by heating to 130° C. at 15 mm. pressure. By this process a yield of 341 parts of wax-free sugar cane oil was obtained. This wax-free oil was then treated with methanol as described in Example II. The reduction solution was then prepared utilizing this wax-free methanol-treated oil and 114.8 parts methyl isobutyl carbinol and 650 parts toluene. This reduction solution was reacted with 57.8 parts sodium dispersed in 215 parts toluene in the manner described in the preceding examples. The reduction mixture was then hydrolyzed and the product worked up in the manner identical to that described in Example I. A yield of approximately 181 parts alcohol mixture was obtained, representing a yield of 97 per cent theoretical.

The product obtained in each of the above three examples was a brown liquid when hot and had the appearance of a wax at room temperature. This product contains essentially high molecular weight alcohols, both saturated and unsaturated, having from about 10 to 30 carbon atoms. The steres are originally present in the sugar cane oil are substantially unaffected by the foregoing reduction process and are also present in the product mixture. Such a mixture can be utilized as such in the preparation of detergents, drying oils, and the like, although for some applications it may be desirable to effect a partial separation of the product into various fractions. For example, the steres can be separated from the product mixture if desired. Although in practice it may be convenient to cut out somewhat different fractions of alcohols from the crude product, I have found it convenient to separate the above products into two fractions, one consisting essentially of C6 to C9 alcohols and the other consisting essentially of C10 to C20 alcohols, including the steres. The C6 to C9 sterc fraction has an average molecular weight of 260, this weight being slightly higher than that of a C10 alcohol. The iodine number of this fraction indicates an average of about 1.5 double bonds per molecule. The C10 to C20 fraction has an average molecular weight of 400 (about that of a C10 alcohol) and an average of 2.9 double bonds per molecule.

The above separation of the crude product into the C6 to C9 and C10 to C20 fractions can be conveniently accomplished by distillation carried out at about 225° C. and 1 mm. pressure. The distillate will consist of the C6 to C9 fraction and the residue will contain the C10 to C20 fraction. This residue is then extracted with boiling acetonitrite, the extract decanted from the insoluble material, cooled to crystallize the C6 to C9 alcohols, and the C10 to C20 alcohols separated by filtration or some equivalent means.

The following examples will serve to illustrate this embodiment of my invention. The parts and proportions given are on weight basis.

**Example IV**

Approximately 123 parts of the product obtained in Example II above was distilled at a temperature of 225° C. and a pressure of 1 mm. Approximately 73 parts of distillate was obtained consisting essentially of C6 to C9 alcohols having the following analysis:

<table>
<thead>
<tr>
<th>Saponification No.</th>
<th>Acid No.</th>
<th>Ester No.</th>
<th>Hydroxy value</th>
<th>Iodine No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>0</td>
<td>0</td>
<td>6.3</td>
<td>145</td>
</tr>
</tbody>
</table>

Fifty-seven parts of the residue was extracted with 158 parts boiling acetonitrite, the extract cooled to below 70° C. and the C10 to C20 alcohols recovered by filtration. This extraction was repeated four times using the filtrate from the previous extraction, and a total of 26 parts of C10 to C20 alcohols was obtained. This product had the following analysis:

<table>
<thead>
<tr>
<th>Saponification No.</th>
<th>Acid No.</th>
<th>Ester No.</th>
<th>Hydroxy value</th>
<th>Iodine number</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>0</td>
<td>3</td>
<td>4.1</td>
<td>173</td>
</tr>
</tbody>
</table>

Melt point: 70-100° C.

**Example V**

One hundred forty parts of the product obtained in Example III above was treated in the manner described in Example IV. A yield of 81 parts of the C10 to C20 alcohol mixture was obtained having the following analysis:

<table>
<thead>
<tr>
<th>Saponification No.</th>
<th>Acid No.</th>
<th>Ester No.</th>
<th>Hydroxy value</th>
<th>Iodine No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>1</td>
<td>1</td>
<td>6.7</td>
<td>153</td>
</tr>
</tbody>
</table>

Twenty-two parts of C10 to C20 alcohols was obtained and had the following analysis:

<table>
<thead>
<tr>
<th>Saponification No.</th>
<th>Acid No.</th>
<th>Ester No.</th>
<th>Hydroxy value</th>
<th>Iodine number</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>1</td>
<td>6</td>
<td>4.5</td>
<td>194</td>
</tr>
</tbody>
</table>

It can therefore be seen that by my invention I have provided new and useful mixtures of high molecular weight alcohols characterized by a relatively high degree of unsaturation. I have also provided a novel process for obtaining these alcohols using a starting material sugar cane oil which has herefore been generally considered waste material. An important embodiment of my process is that the unsaturation present in the starting material is preserved, thereby rendering the resulting products highly useful in applications such as in formulating drying oils and resins which require high molecular weight unsaturated materials. The sugar can oil alcohols of my invention can also be used to prepare novel high molecular weight wax compositions by esterifying these alcohols with high molecular weight fatty acids. For example, one method of preparing such a wax consists of esterifying the sugar cane oil alcohols with sugar cane oil fatty acids.

It is to be understood that the above examples are given only by way of illustrating specific embodiments, and I intend by the appended claims to cover all modifications which fall within the spirit and scope of my invention.

I claim:

1. A new composition of matter comprising essentially a mixture of higher molecular weight alcohols derived from sugar cane oil by a process which comprises reducing sugar cane oil with an alkali metal and a hydrolytic alcohol and hydrotreating the resulting reduction mixture.

2. A new composition of matter comprising a mixture consisting essentially of steres derived from sugar cane oil and alcohols corresponding to the fatty acid radicals of sugar cane oil obtained by a process which comprises reducing sugar cane oil with an alkali metal and a
hydrolytic alcohol and hydrolyzing the resulting reduction mixture.

3. A process for the preparation of high molecular weight alcohols from sugar cane oil, comprising treating said oil with a lower aliphatic alcohol so as to esterify at least in part the free acids present in said oil, reducing the alcohol-treated sugar cane oil with alkali metal and a hydrolytic alcohol, hydrolyzing the reduced mixture thereby formed, and separating said high molecular weight alcohols from said mixture.

References Cited in the file of this patent

UNITED STATES PATENTS

868,252  Bouveault -------------- Oct. 15, 1907

1,971,742  Bertsch -------------- Aug. 28, 1934
1,971,743  Bertsch -------------- Aug. 28, 1934
2,019,022  Scott -------------- Oct. 29, 1935
2,070,597  Henke -------------- Feb. 16, 1937
2,381,420  Balch -------------- Aug. 7, 1945
2,460,969  Blinoff -------------- Feb. 8, 1949

OTHER REFERENCES