CONVERSION OF DISTILLATION RESIDUES TO USEFUL METAL WORKING LUBRICANTS

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Field of Search .............. 72/42; 252/56 R, 56 S

References Cited
UNITED STATES PATENTS
3,702,301 11/1972 Baldwin...................... 252/56 S


Primary Examiner—Delbert E. Gantz
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Attorney, Agent, or Firm—Gerald A. Baracka; John D. Rice

ABSTRACT

Residues resulting from the distillation of fatty acids obtained from fat-splitting processes are converted by the process of this invention to useful metal working lubricants. The present process utilizes distillation residues, which are typically viscous oils ranging in color from dark brown to black and which heretofore had little or no commercial value and were burned or otherwise disposed of, to produce lubricants having performance characteristics which make them suitable for use in continuous metal-casting and other metal working operations.

10 Claims, No Drawings
CONVERSION OF DISTILLATION RESIDUES TO USEFUL METAL WORKING LUBRICANTS

BACKGROUND OF THE INVENTION

The present invention is directed to a process for converting waste residues obtained from the distillation of fatty acids produced in fat-splitting operations wherein fats and fatty oils are hydrolyzed into fatty acids and glycerol by subjecting the fatty materials to high temperatures and pressures in the presence of water. Fat-splitting processes are well-known and widely used industrially to obtain fatty acids and glycerine.

The fatty acids obtained from such splitting operations, in addition to being gross mixtures containing numerous fatty acids, typically have poor color and odor due to the presence of a variety of impurities resulting from the severe processing conditions and impurities in the fatty material being treated. In view of this, it is customary to distill the resulting fatty acid mixtures for purification purposes and/or to separate the various fatty acid components. This is accomplished using conventional distillation equipment and procedures. At the completion of the distillation there is a residue consisting primarily of oxidative and polymeric by-products including color bodies, odor bodies, modified glycerides and unhydrolyzed or partially hydrolyzed fatty materials. This residue, in some instances is subjected to further pressure splitting, however, even when one or more additional splitting steps are employed a final distillation residue will still be obtained.

These oily malodorous distillation residues are highly colored, ranging in color from deep brown to totally black. In the past, it has been the usual practice in industrial operations to dispose of these residues by burning, burying, etc., because of their low fuel value and for ecological reasons this is becoming increasingly more difficult and costly. These residues have also been suggested for use as extenders in tar products but there is limited use in this area.

SUMMARY OF THE INVENTION

We have now discovered a process whereby waste distillation residues are converted to useful metal working lubricants. The process simply involves contacting the residue with a hydroxyl compound containing at least one hydroxyl group and a least four carbon atoms at a temperature above 100°C, preferably between 150° and 250°C, while removing water from the reaction mixture. The reaction is continued until the acid value is less than 10, and more preferably, 5 or below. The equivalent ratio of the hydroxyl compound to the residue will be 1:1 or higher, based on the acid value of the residue. Residues employed are obtained when fatty acids produced in fat-splitting processes are distilled and typically are brown to black liquids having 180°F kinematic viscosities in the range 40-300 centistokes with iodine values from about 50 to 100, acid values between about 50 and 100 and saponification values between about 100 and 200. Hydroxylic compounds useful in the process are aliphatic, cycloaliphatic and aromatic and mono-, di- and polyhydric alcohols containing 6 to 20 carbon atoms and polyoxyalkylene glycols having molecular weights from about 100 to 4000. The products of this invention may be used as such or filtered with diatomaceous earth. While these products may vary in composition and lubrication properties, they preferably have an acid value of 5 or less, flash points above 500°C and fire points greater than 540°C. The products of this invention are useful in a variety of lubrication applications, however, they find particular utility as lubricants for the continuous casting of metals such as steel or steel alloys.

DETAILED DESCRIPTION

This invention provides a process for converting waste residues resulting from the distillation of certain fatty acids to useful products, namely, metal working lubricants. The distillation residues employed herein result from fat-splitting processes wherein fats and fatty oils are hydrolyzed into fatty acids and glycerol at high temperatures and pressures in the presence of water. The so-produced fatty acids upon distillation yield the waste residue.

The manner in which the fat-splitting process and fatty acid distillation are conducted does not form a part of this invention but serve only as a source for the material to be processed. In other words, the present process is useful with any distillation residue without regard to the conditions employed during the fat-splitting operation and subsequent distillation. Known fat-splitting procedures, such as those described in U.S. Pat. Nos. 2,156,863 and RE 22,006, and various modifications thereof used by industry can be employed. A typical industrial fat-splitting operation, for example, involves countercurrent contact of fat and water in a column. This is accomplished by introducing the fatty material into the column under high pressure through a sparge ring which breaks the fat into small droplets. As the fat droplets are heated they pass upward through the column where the temperature is increased by the introduction of steam under high pressure. Water is charged under pressure through a sparge ring at the top of the column. The rate of flow of fat and water are carefully controlled to permit maximum contact with each other. The column is completely filled with liquids and vaporization of water is prevented be maintaining the pressure within the system in excess of the vapor pressure of water at the operating temperature. Generally, the pressure will range between about 150 and 600 psi while maintaining a temperature of at least 350°F and, more preferably, between about 365°F and 600°F. Sweet water (aqueous glycerine) is collected at the bottom of the column and pumped to a glycerine concentrator. Fatty acids are discharged from the top of the tower.

Fatty acids originating from the fat-splitting operation are highly colored and contain a variety of impurities. It is customary therefore to distill the fatty acids either solely for the purpose of purification distillation or additionally to separate the various acids of different chain lengths and boiling points. The distillation can be conducted either as a batch process or continuously. Continuous distillation is preferred since losses are minimized and it is possible to increase the yield and quality of the distilled products and decrease the time of exposure of the acids to high temperatures. Typically, a continuous commercial fatty acid distillation process involves initially stripping the fatty acids to remove “light ends” and odor bodies and then pumping the bottoms from the stripper column into the main fractionation column fitted with a plurality of bubble-cap or other suitable trays. The residue (bottoms) of the main column may be used directly in the process of this invention or may be fed to another stripping tower.
wherein very high boiling fatty acids are removed and the residue remaining after this step used in this process. Solutions containing the residue obtained after the so-called residues to a second splitting and distillation operation, following the same general procedure as outlined above, and to convert the residue to useful metal working lubricants in accordance with the invention. By subjecting the residue to additional splitting it is possible to improve the yield of fatty acids and glycerine. It is not generally practical, however, to have more than two splitting operations in the treatment of fatty materials since only marginal improvements are possible after two splittings. In all cases, however, a residue will be obtained from the distillation of the fatty acids which quite unexpectedly is converted by the process of this invention to a useful metal working lubricant. Similarly, if a batch distillation procedure is employed the pot residue remaining after removal of the fatty acids can be utilized for the process of this invention.

Residues useful for the present process are those obtained by splitting any of the commonly employed materials followed by distillation in the above manner. Common fatty materials which are split to obtain fatty acids and glycerine include vegetable and animal oils, such as tallow and other related greases, coconut oil, soybean oil, corn oil, peanut oil, cottonseed oil and the like. In some instances it may be advantageous to hydrolyze the fatty material prior to splitting.

The waste distillation residues which are converted to useful lubricant products by this process are mixtures containing numerous oxidative and polymeric by-products including modified glycrides, unhydrolyzed and partially hydrolyzed fatty materials and color and odor bodies and the precise composition is difficult and, in most instances impossible, to completely define. While the chemical makeup will vary depending on the fatty material used, splitting conditions and distillation procedure, the residues are in all instances highly colored (dark brown - black) liquids having 180°F kinematic viscosities ranging from 40 to 300 centistokes. The residues typically have iodine values from about 50 to 100, acid values between 50 and 100 and saponification values from 100 to 200. While the content of unsaponifiable materials in the residue can range up to about 50% by weight it is more usually less than about 25%.

The above-described residues, which as such have little or no practical commercial value, are converted to useful lubricants in accordance with the process of the invention by contacting with hydroxyl compounds at elevated temperatures until a low acid value is obtained. The process is conducted at temperatures above 100°C in order that the water formed during the reaction can be removed from the system. Best results are obtained at temperatures between about 150° and 250°C and, more preferably, between about 180° and 230°C. It is not necessary that a catalyst be employed to promote the condensation, however, known esterification catalysts such as sulfuric acid, alkyl and aryl sulfonic acids such as p-toluene sulfonic acid, sodium sulfate, phosphoric acid, stannous oxalate and metal butyl halides, the latter being used. Also, it is possible in carrying out the reaction to employ an organic diluent which is inert to the reaction conditions employed but which preferably will form an azetrop with water. Xylene and toluene are suitable diluent/carriers for this purpose. The reaction is continued until the reaction mixture has a low acid value, that is, the acid value has been lowered to about 10 or below. Excellent results are obtained when the acid value is 5 or less.

Hydroxyl compounds useful in the present process contain one or more free hydroxyl groups and at least four carbon atoms. Suitable hydroxyl compounds include the aliphatic, cycloaliphatic, aromatic and polyoxyether alcohols, including mono-, di and polyhydric alcohols and mixtures thereof. Especially useful hydroxyl compounds for this invention are aliphatic, cycloaliphatic and aromatic mono-, di and polyhydric alcohols containing six to 20 carbon atoms and polyoxyalkylene glycols having molecular weights from about 100 to 4000. Useful monoalcohols, include n-butyl alcohol, amyl alcohol, isomyl alcohol, n-hexyl alcohol, 2-ethylbutyl alcohol, n-octyl alcohol, 2-ethylhexyl alcohol, decyl alcohol, lauryl alcohol, oleyl alcohol, ethylene glycol mono-n-butyl ether, diethylene glycol mono-n-butyl ether, diethylene glycol mono-2-ethylhexyl ether, propylene glycol mono-n-butyl ether, diethylene glycol mono-methyl ether, diethylene glycol mono-n-butyl ether, tripropylene glycol mono-methyl ether, triethylene glycol mono-isopropyl ether, cyclohexanol, benzyl alcohol, oxo alcohols, such as isocetyl, isodecyl and tridecyl alcohols, and the like. Di- and polyhydric alcohols, including polyoxyether alcohols, which can be used include 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 1,8-octanediol, 1,10-decanediol, 1,4-cyclohexanediol, glycerol, sorbitol, pentaerythritol, trimethylolethane, trimethyl propane, diethylene glycol, dipropylene glycol, tripropylene glycol and similar materials having higher degrees of polymerization such as polyethylene glycols, polypropylene glycols, polybutylene glycol and poly(ethylene-propylene) glycols.

In carrying out the process a stoichiometric amount (1:1 equivalents ratio) of hydroxyl compound, based on the acid value of the residue, can be used or the hydroxyl material can be present in excess. The only requirement as to the amount of mono-, di or polyhydric alcohol used for the process is that sufficient hydroxyl groups be present for reaction (to react with the acid groups) to reduce the acid value of the product to about 10 or below. As a practical matter when using hydroxyl materials which are not readily removable from the reaction mixture at the end of the reaction, that is, having a boiling point above about 180°C at 1 mm Hg., the stoichiometry is strictly observed and no more than about 5% excess of the alcohol should be charged. With the lower boiling and more volatile alcohols (such as monohydric alcohols) where it is possible to readily strip the materials from the reaction mixture, an excess can be employed which helps drive the reaction to completion. Some unreacted alcohol may be present in the final product without unduly detracting from the desirable properties.

Other variations in the process of this invention are possible as will be recognized by those skilled in the art. For example, it is possible to modify the lubricant properties, as well as other properties, of the resulting product by including other carboxyl materials in the reaction. Mono- and dihydric alcohols such as saturated and unsaturated monobasic acids and saturated and unsaturated di- and tricarboxylic acids, are useful for this purpose. For example, acids which can be employed include valeric acid, capric acid, caprylic acid, pelargonic acid, lauric acid, palmitic acid, stearic acid, oleic...
acid, azelaic acid, sebacic acid, succinic acid, glutaric acid, malonic acid, fumaric acid, and dimer and trimer acids containing 32 to 54 carbon atoms, which are the polymerization products of unsaturated monocarboxylic acids. The addition of dimer acid is particularly useful to enhance the lubricant properties of the product whereas with azelaic acid and sebacic acids the solubility characteristics can be markedly altered, particularly when the hydroxylic compound is polyoxymethylene glycol.

The product obtained from the above-described process can be used directly if stoichiometric amounts of the hydroxylic compound have been employed, otherwise the excess mono-, di or polyhydric alcohol will be stripped from the product at the termination of the reaction. This is accomplished by heating while the pressure is lowered to about 1 mm Hg. While it is not necessary, it is often advantageous to filter the product at the termination of the reaction using diatomaceous earth. Any of the commonly used and commercially available diatomaceous earths (often referred to as diatomite or kieselguhr) obtained from natural deposits and comprised primarily of silica and alumina are suitable for this purpose. By such filtration it is possible to obtain more uniform and consistent products which are easily applied to metals.

Acid values of the products will be about 10 or below. Superior results are obtained when the acid value is 5 or below. The products are further characterized as having a flash point above about 500°C and fire points greater than about 540°C. The products have lubricities, measured using the Falex Test Method (ASTM D 2670), comparable to soybean oil, crumbe oil and rapeseed oil without having certain undesirable properties generally attributed to these natural oils.

The following Examples serve to illustrate the invention more fully, however, they are intended solely for the purpose of illustration and not as a limitation on the scope of the invention. In these examples all parts and percentages are given on a weight basis unless otherwise indicated. Viscosities are kinematic viscosities obtained in accordance with ASTM D 445-65. Acid values and hydroxyl values reported as mg. KOH/gram are determined by ASTM D 1980-67 and ASTM D 1957-63, respectively.

**EXAMPLE I**

A glass reactor equipped with a stirrer, thermometer, nitrogen inlet and water trap topped with a cooled condenser was charged with 411 parts of the black waste residue obtained from the distillation of fatty acids produced from the pressure splitting of tallow. The residue had an acid value of about 75° and 180°F kinematic viscosity of 95. Eighty-nine parts 2-ethylhexanol (25% excess based on the equivalent weight of the distillation residue as determined from the acid value) were then added and the reaction mixture heated under an atmosphere of nitrogen with continuous stirring at about 220°C for approximately 4 hours during which time about 9 mls of water were removed. When the acid value of the reaction mixture was less than 10 a vacuum of about 1 torr was applied to remove any additional water of reaction and the excess 2-ethylhexanol. The resulting dark product had a viscosity of 26.2 centistokes at 180°F and other properties making it useful as a metal working lubricant.

**EXAMPLE II**

Using a procedure similar to that described in Example I, the residue obtained when a first fatty acid distillation residue is subjected to a second splitting operation and fractional distillation was reacted with benzyl alcohol. The residue had the following properties:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid value</td>
<td>80</td>
</tr>
<tr>
<td>Saponification value</td>
<td>164</td>
</tr>
<tr>
<td>Iodine value</td>
<td>76.9</td>
</tr>
<tr>
<td>% Unsat. 180°F viscosities</td>
<td>5.6</td>
</tr>
<tr>
<td>180°F viscosities</td>
<td>122 centistokes</td>
</tr>
</tbody>
</table>

Benzyl alcohol was employed in 25% excess based on a calculated equivalent weight of 750 for the residue. The reaction mixture was maintained between 200° and 220°C for about 10 hours during which time the theoretical amount of water was removed. A vacuum was then applied to the system and heating continued at 225°C to remove the final traces of water and strip off the unreacted (excess) benzyl alcohol. About 0.5 wt. % Dicalite (a commercially available diatomaceous earth filter aid) was added and the reaction mixture filtered. The final product had an acid value of about 8 and a viscosity of 26.0 centistokes at 180°F.

**EXAMPLE III - VI**

A series of experiments was conducted in accordance with the process of this invention wherein the distillation residue of Example II was reacted with various polyhydric alcohols. The alcohols employed were neopentyl glycol, trimethylolpropane, trimethylolethane and pentaerythritol. In all these experiments the equivalents ratio of alcohol to residue was 1:0.1:0.1 and the reaction mixtures were heated (200°-220°C) until the acid value was less than 9. Reaction times ranged from 6 to 10 hours. The following table lists the polyhydric glycol used for each of the preparations, the amount (grams) of the alcohol and the residue charge, and the viscosities and pour points of the resulting products.

<table>
<thead>
<tr>
<th>Example No.</th>
<th>III</th>
<th>IV</th>
<th>V</th>
<th>VI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alcohol</td>
<td>neopentyl glycol</td>
<td>trimethylolpropane</td>
<td>trimethylol-ethane</td>
<td>pentaerythritol</td>
</tr>
<tr>
<td>Reactant charge (alcohol/residue)</td>
<td>64/8/93/5</td>
<td>56/3/943.7</td>
<td>48/900</td>
<td>43.4/956.6</td>
</tr>
<tr>
<td>180°F Viscosity (centistokes)</td>
<td>118</td>
<td>259</td>
<td>264</td>
<td>489</td>
</tr>
<tr>
<td>Pour point (°F) (ASTM D 97-57)</td>
<td>0°</td>
<td>20°</td>
<td>15°</td>
<td>35°</td>
</tr>
</tbody>
</table>

All of the above products were filtered with a diatomaceous earth filter aid and were useful lubricants.

The above examples clearly demonstrate that a variety of useful products are obtained by treating residues resulting from the distillation of fatty acids produced by pressure splitting fatty materials. It is truly surprising that waste distillation residues, heretofore, considered
3,923,702

to have little or no practical or commercial value, are converted by the process of this invention to products as lubricants for continuous casting operations and other metal working applications. That acceptable lubrication can be obtained from these undesirable appearing wastes is by itself truly surprising; however, it is even more remarkable when one considers that the products of this process can be used without further purification.

To demonstrate the effectiveness of the products of this invention as lubricants they were evaluated with a Falex machine. This machine provides a convenient and reliable means of determining the film strength or load carrying properties of materials as extreme pressures are applied. Falex testing is recognized throughout the industry as a means of measuring the relative effectiveness of various lubricants. For the Falex wear test (ASTM D 2670-67) a 60 gram sample of the product is placed in the cup and positioned in the machine so that the steel pin and blocks are completely immersed in the sample. The loading device is attached, the machine started and the load increased to 350 pounds and run for 5 minutes. After this time the load is further increased to 800 pounds and maintained for 15 minutes. Readings are taken at the beginning and end of the 15 minute period and the difference in the readings reported as the units of wear. The following table sets forth the test results obtained when the products of Examples I-VI were evaluated in the Falex test. In addition to the wear tests data, other significant lubricant properties including viscosities of the product at 100°F and 210°F and the flash and fire points are provided.

### PRODUCT OF EXAMPLES

<table>
<thead>
<tr>
<th>I</th>
<th>II</th>
<th>III</th>
<th>IV</th>
<th>V</th>
<th>VI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Units of Wear</td>
<td>20</td>
<td>1</td>
<td>76</td>
<td>49</td>
<td>1</td>
</tr>
<tr>
<td>Viscosity (cSt)</td>
<td>129</td>
<td>130</td>
<td>858</td>
<td>2025</td>
<td>2170</td>
</tr>
<tr>
<td>100°F</td>
<td>173</td>
<td>175</td>
<td>68.5</td>
<td>133</td>
<td>142</td>
</tr>
<tr>
<td>210°F</td>
<td>510</td>
<td>525</td>
<td>575</td>
<td>575</td>
<td>585</td>
</tr>
<tr>
<td>Flash point (°F)</td>
<td>555</td>
<td>550</td>
<td>590</td>
<td>605</td>
<td>600</td>
</tr>
<tr>
<td>Fire point (°F)</td>
<td>555</td>
<td>550</td>
<td>590</td>
<td>605</td>
<td>600</td>
</tr>
</tbody>
</table>

Refined rapeseed oil and soybean oil gave 46 and 33 units wear, respectively, in the Falex machine under identical test conditions. The product of Example I was also evaluated using a four-ball extreme pressure lubricant tester in accordance with ASTM D 2783. The test was conducted for 1 hour (167°F) at a load of 40kg. and 1,200 rpm. The average wear spot diameter was only 0.70 mm.

### EXAMPLE VII

To further demonstrate the versatility of the present process and the ability to obtain useful lubricant products using mixed alcohols an experiment was conducted wherein 407 parts of the residue of Example II, 4.2 parts neopentyl glycol and 88.3 parts 2-ethylhexanol (25% excess) were reacted. Stannous oxide (0.03%) was used to promote the reaction. After about 14 hours (220°C) an acid value of 3.8 was obtained. At this point the vacuum was further reduced and about 18 parts 2-ethylhexanol stripped from the reaction mixture. The final product (acid value 4.0) was recovered by filtration with a diatomaceous earth filtering agent. The product had 100°F and 210°F viscosities of 104 centistokes and 14.5 centistokes, respectively, a flash point of 500°F and a fire point of 535°F. In the Falex test, following the procedure set forth above, there was no measurable wear using the product of this Example.

### EXAMPLE VIII

That additional advantages are possible employing the process of this invention is evident from the following example wherein a lubricant product, emulsifiable with water without the addition of external emulsifying agents, was prepared from the distillation residue of Example II. To prepare this self-emulsifiable product polyoxymethylene glycol (average molecular weight of 400) and 2-ethylhexanol were reacted with the residue at an equivalent ratio of 2.0:8:1.0, respectively. 0.03 Percent condensed butyl titanate was employed to catalyze the reaction which was carried out at 210°-220°C for about 30 hours, until an acid value of 3.4 was obtained. The recovered product had a 100°F viscosity of 118 centistokes, 210°F viscosity of 17.0 centistokes, 315°F smoke point, 495°F flash point and 545°F fire point. In the Falex wear test, there was no measurable wear when the product of this Example was used as the neat oil and only 2 units wear when a 5% aqueous emulsion product was tested. In addition to the excellent wear test properties obtained with the emulsion, the emulsions were quite stable and very readily formed. A 5% aqueous emulsion was also prepared from the product of Example I using ethoxylated nonylphenol as the emulsifier and gave 22 units wear in the Falex test.

The above products are useful as lubricants in a variety of metal working operations including, for example, forging, hot pressing, blanking, bending, stamping, drawing, cutting, punching, drilling, and the like, however, they find particular utility as lubricants for continuous casting of molten metals. These lubricants greatly improve metal working operations in that they prevent sticking of metal pieces, decrease wear of dies and cutting bits and lower the power requirements for the operation by reducing friction. The lubricants are readily substitutable for, or can be used in combination with, any of the commonly used industrial lubricants including vegetable, animal, mineral and petroleum oils and, in most cases, provide distinct advantages over the aforementioned oils.

The products of this invention are suitable for use with both ferrous and non-ferrous metals. They can be used with steel and various alloys, such as brass and bronze, aluminum, copper, titanium and other metals. They are especially useful with steel and steel alloys, particularly, in continuous casting operations involving these metals. In this type of operation molten steel is poured into a suitable mold and continuously cast into billets or slabs. Lubricants are necessary to facilitate heat transfer and prevent sticking of the metal to the mold to assure continuous and uniform flow of the metal. While mineral oils and various natural oils including rapeseed oil, crambe oil, castor oil, cottonseed oil and soybean oil have typically been used by the industry, there are certain disadvantages associated with the use of these oils. Mineral oils tend to burn too readily and produce excessive amounts of smoke which makes it impossible to maintain visual contact with the mold. The vegetable oils, which all suffer common drawbacks of limited supply and rising prices, have been the most widely used lubricants for this purpose. Rapeseed oil and crambe oil are particularly effective and in some cases are mixed with a higher viscosity mineral oil. The vegetable oils, however, upon decom-
position produce excessive amounts of acrolein, which has a very disagreeable choking odor, causes considerable irritation to the eyes, and in sufficient concentration is potentially dangerous to mill personnel. Most of these oils have the added disadvantage of being susceptible to oxidation and form a coating (varnish) on the mold which requires that the mold be periodically cleaned resulting in considerable and costly downtime.

While the products of this invention find utility in numerous applications where lubricants are required they are particularly useful as lubricants in the continuous casting of steel and eliminate many of the difficulties encountered with the heretofore used lubricants. The present products have sufficiently low viscosities so that they flow easily, are readily pumpable and can be applied using conventional application methods to form a uniform and continuous film between metal and mold, which is essential for efficient operation of the process and the obtainment of slabs and billets substantially free of surface defects. Another desirable feature of the products is their ability to burn without generating excessive amounts of smoke and soot and without depositing unduly large amounts of carbonaceous matter on the steel. Still more advantageous is the fact that with these products acrolein formation is minimized or completely eliminated. Also, these products exhibit excellent oxidative stability which eliminates problems generally associated with the storage and use of natural oils and minimizes varnish formation on the mold and surface of the metal.

Numerous modifications of the process and the products obtained thereby will be evident to those skilled in the art arc are within the scope of the invention. For example, the viscosity, fire point, flash point, pour point, etc. can be widely varied by judicious selection of the polyhydric compound or by the inclusion of other materials. Products obtained from this process may be mixed with other oils including mineral oil and other synthetic or natural lubricants. Additives may be incorporated into the product either be blending after the process is completed or by including in the process, if the additives do not interfere with the product forma-

We claim:

1. A process for converting residues, obtained when fatty acids produced from fat-splitting processes are distilled, to useful lubricants which comprises contacting the residue with a hydroxylic compound containing at least one hydroxyl group and at least four carbon atoms at a temperature above 100°C while removing water from the reaction mixture until the acid value of the product is about 10 or below, the equivalents ratio of said hydroxylic compound to said residue being 1:1 or higher, based on the acid value of the residue.

2. The process of claim 1 wherein the residue has an iodine value from about 50 to 100, an acid value between about 50 and 100 and a saponification value between 100 and 200.

3. The process of claim 1 wherein the temperature is maintained between about 150° and 250°C and the hydroxylic compound is selected from the group consisting of aliphatic, cycloaliphatic and aromatic, mono-, di- and polyhydric alcohols containing six to 20 carbon atoms and polyoxyalkylene glycols having molecular weights from about 100 to 4,000.

4. The process of claim 3 conducted to an acid value of 5 or below and wherein the temperature is between about 180° and 230°C.

5. The process of claim 3 wherein the reaction product is treated with diatomaceous earth and filtered.

6. The composition prepared by the process of claim 1.

7. The composition prepared by the process of claim 3.

8. The composition of claim 7 which is further characterized by having an acid value of 5 or below, a flash point above 500°C and a fire point greater than 540°C.

9. The composition of claim 8 wherein the hydroxylic compound is 2-ethylhexanol.

10. The composition of claim 8 wherein the hydroxylic compound is isodecyl alcohol.

"
UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,923,702 Dated December 2, 1975

Inventor(s) R. J. Sturwold, F. O. Barrett and W. E. Utz

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 37, "there" should read --- their ---.

Column 2, line 53, delete --- distillation ---.

Column 5, line 30, "°C" should read --- °F ---; Column 5, line 31, "°C" should read --- °F ---.

Column 6, line 4, "vaccum" should read --- vacuum ---; Column 6, line 22, "viscosities" should read --- viscosity ---;

Column 7, line 2, after products insert --- useful ---; Column 7, line 31, "tests" should read --- test ---.

Column 9, line 33, the first "are" should read --- and ---.

Signed and Sealed this first Day of June 1976

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks