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WATER SOLUBLE LUBRICANT

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ABSTRACT OF THE DISCLOSURE

A water soluble composition comprising the product obtained by transesterifying a triglyceride (e.g. castor oil) with a polymeric alkylene oxide glycol until the reaction products are water-soluble and then esterifying the hydroxy compounds present with a carboxylic or dicarboxylic acid. These products are of value as lubricants in a wide variety of fields.

Metal working lubrication has been accomplished, as is well known, by the use of mineral and/or fatty oils. In the early technology, these fats and oils were used alone, but with the advent of improved technology, it was useful to prepare aqueous emulsions of mineral and/or fatty oils in order to reduce costs, improve cooling capacity, and give generally better performance. Until recent years almost all water containing lubricants were emulsions of fatty and/or mineral oils which might or might not contain various additives for particular applications. However, emulsion type products have several inherent problems, as, for example, emulsion stability, and for this reason and others they often find their utility limited. In recent years, water containing lubricants of the solution type have become available and in areas where cooling ability is the primary consideration the water soluble lubricant has shown great merit. However, where conditions require both a high degree of cooling plus a high degree of hydrodynamic or oil film lubricity characteristics, the water soluble lubricants have not been suitable because they have been unable to deposit a film containing sufficient lubricity for proper function under hydrodynamic conditions.

The water soluble lubricants appear to function by "plating out" the lubricant onto the surface to be lubricated during use. This appears to occur because heat of friction raises the temperature of the solution above its cloud point and causes the solution to change to a dispersion or emulsion, whereby the "plating out" phenomena occurs. In the past, however, the lubricating benefit derived from the film of "plate out" of these aqueous solution lubricants has been well below the degree of lubricity provided by the conventionally emulsified materials.

This invention now provides an improved water soluble lubricant which deposits a film on surfaces to be lubricated where the deposited film has greatly improved lubrication and anti-frictional qualities.

In one embodiment of the invention there is provided a water soluble product obtained by transesterifying a triglyceride such as castor oil or similar triglyceride with a polyoxyalkylene polyol until the reaction products are water soluble and then esterifying the hydroxy compounds present with a carboxylic or dicarboxylic acid.

Another embodiment of the invention involves an aqueous solution formulation useful as a metal working lubricant. Still another embodiment of the invention is the process for lubricating metal by providing on its surface a lubricating film of the water soluble lubricant composition above defined.

In making the compositions of the invention, a first step

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requires the transesterification of the triglyceride, preferably castor oil, with a polyoxyalkylenepolyol having a molecular weight of at least about 1000 until the reaction products are water soluble. As is well known, castor oil is a triglyceride which is predominantly the ricinoleic acid ester of glycerol, but which contains minor amounts of the oleic and linoleic esters. Thus, in the transesterification step, the polyoxyalkylene polyol will replace some or all of the glycerol OH groups and generate OH groups from the glycerol. The free OH groups in this water soluble product are then reacted with a carboxylic acid or a dicarboxylic acid to prepare the product which is subsequently formulated for use as will be discussed below.

In the transesterification procedure, the preferred amount of polyoxyalkylene polyol used will be on an approximate 1:1 mole equivalent weight ratio with the triglyceride. At higher content of polyoxyalkylene polyol there will be a decrease in the lubricity value afforded by the end product. At significantly lower content of polyoxyalkylene polyol, water solubility will not be achieved. The effective range for water solubility and good lubricity is 0.75 to 2.0 mole equivalents of the polyoxyalkylene polyol to 1.0 mole equivalent of the triglyceride.

In carrying out this transesterification step, it is preferred to remove air from the reaction medium and this is done preferably by sweeping nitrogen through the reaction equipment. The temperature of the transesterification process is on the order of 400° to 450° F. and the time of reaction will, of course, vary with reaction kettle size and other factors, but, in general, will be from about 10 to 20 hours. Completion of the reaction is determined when 5 g. of the product is completely soluble in 100 ml. of water at 100° F. The polyoxyalkylene polyol used in the reaction may be any one of a large number of such products which are commercially available, but for use in this invention, the glycol used will have a molecular weight of at least about 1000. If glycols of molecular weight less than this are used, the products are not water soluble and not useful in this invention. As a simple example of such materials that may be used reference is made to polyethylene glycol. Other polyoxyalkylenepolyols may be used such as the water soluble high molecular weight viscous liquid polyalkylene polyols which contain ethylene oxide and/or propylene oxide groups available as "Pluracol" V–5 and V–7 manufactured by Wyandotte Chemicals Corporation or the polyalkylene glycols known as "Ucons" available from Union Carbide Chemicals Company.

The transesterification is carried out generally in the presence of a catalyst. The catalyst used is an important factor in achieving water solubility of the transesterification reaction product. Buffer catalysts such as alkali metal carbonates and acetates, (e.g. sodium carbonate, sodium acetate, and the like) are necessary. Acidic and basic catalysts produce a product with only partial water solubility.

When the transesterification reaction is complated as indicated by the above water solubility test, reaction of the first product is made to occur with the carboxylic or dicarboxylic acid and such acids may be any of the well-known long-chain fatty acids such as lauric, oleic, stearic acid, ricinoleic, linoleic and the like. Dicarboxylic acids which may be used are succinic, adipic, glutaric, azelaic, and sebacic. Also useful are aryl substituted fatty acids such as phenylstearic and the like.

In this second reaction step, a catalyst is also preferably used and p-toluenesulfonic acid or similar acidic catalyst (e.g. dodecylbenzenesulfonic acid, etc.) is employed. The temperature of the reaction may be from about 350° F. to 450° F. and reaction is continued by stirring the reactants at this temperature until completion of the reaction which is indicated by the acid number falling below to six.

The resulting product obtained by the above procedure is a viscous, amber fluid, soluble in 100° F. water with distinct cloud points below 180° F. and gives a pH in a 5% water solution of 7.0 to 7.7. This product is used by dilution with water and this formulation may also contain other additives as e.g. agents for rust protection, silicone polymers as defoamers, chlorinated phenol type bactericides and the like. Generally, the formulation of product in water will be such that the amount of lubricant will be on the order of 5% to 50% by weight and preferably 10% to 25%.

In order to further illustrate the invention the following examples are given:

EXAMPLE 1

A stainless steel reaction vessel is charged with 20 pounds of castor oil, 44.5 pounds of polyalkylene polyol containing ethylene oxide and propylene oxide groups and having a molecular weight of about 3000 (Pluracol V–5) and 0.15 pound of sodium acetate. The reaction vessel is swept with nitrogen at a rate of 5 to 10 cubic feet per hour and as the mixture is agitated the temperature is raised to 425° F. After 17 hours at this temperature a sample is taken and 5% by weight solution of the product in water at 100° F. is readily obtained. The temperature of the reaction mixture is then reduced to 375° F. and 5.5 pounds of phenylstearic acid (Armour Neofat LPS) and 0.18 pound of p-toluene sulfonic acid is added. The temperature is raised to 420° F. and after the acid number reaches 3.8 (about 24 hours) the heat is removed and when the temperature drops to about 200° F. the reaction mixture is discharged into containers for storage and subsequent use. The product is an amber fluid that has the following typical properties:

Acid number: 4.0
Hydroxyl number: 62
Saponification number: 68
Color [FAC]: 7
Flash point [COC]: 565° F.
Fire point [COC]: 615° F.
Specific gravity: 1.04 at 60°/60° F.
Viscosity at 100° F.: 2725 SUS
Viscosity at 210° F.: 410 SUS
Viscosity index: 132

EXAMPLE 2

A stainless steel reactor is charged with 21.1 pounds of castor oil and 45.3 pounds of Pluracol V–5 and 0.17 pound of sodium acetate. Nitrogen is swept through the reaction kettle at 5 to 10 cubic feet per hour and the temperature of the reactants is raised to 400° to 450° F. while maintaining agitation. This temperature is held for approximately 17 hours at which time a 5% by weight solution of the product in water at 100° F. is obtained as a clear solution. At this point the temperature of the reaction product is lowered to 380° F. and 3.3 pounds of monosodium azelate and 0.07 pound of dodecyl benzene sulfonic acid are added. The reaction mass is maintained at 380° to 420° F. with a nitrogen sweep being maintained for approximately 20 hours after which time an acid number of 3.5 is reached. The product is an amber fluid that has the following typical properties:

Acid number: 3.8
Hydroxyl number: 61.5
Saponification number: 71.2
Color [FAC]: 20
Flash point [COC]: 560° F.
Fire point [COC]: 610° F.
Specific gravity: 1.04 at 60°/60° F.
Viscosity at 100° F.: 3067 SUS
Viscosity at 210° F.: 436 SUS
Viscosity index: 129

EXAMPLE 3

Example 1 is repeated except that the phenylstearic

acid is replaced with stearic acid and the product obtained has the following properties:

Acid number: 3.21
Hydroxyl number: 61
Saponification number: 69.3
Color [FAC]: 2
Flash point [COC]: 625° F.
Fire point [COC]: 640° F.
Specific gravity: 1.03 at 60°/60° F.
Viscosity at 100° F.: 3059 SUS
Viscosity at 210° F.: 464 SUS
Viscosity index: 130

EXAMPLE 4

Example 1 is repeated except that the Pluracol V–5 is replaced with 47.2 pounds of polyethylene glycol of 2500 molecular weight (Ucon 75–H–1400). The fluid product obtained from this reaction has the following typical properties:

Acid number: 4.2
Hydroxyl number: 63
Saponification number: 57.6
Color [FAC]: 3
Flash point [COC]: 525° F.
Fire point [COC]: 620° F.
Specific gravity: 1.04 at 60°/60° F.
Viscosity at 100° F.: 1687 SUS
Viscosity at 210° F.: 252 SUS
Viscosity index: 133

As indicated, the above compositions are used primarily as lubricants in the metal working field. For this purpose the products are formulated with water and such formulations will contain from about 5 to about 50% by weight of the active lubricant. The following example illustrates the type of lubricant formulations which may be employed.

EXAMPLE 5

The lubricant composition of Example 1 is formulated for sale as a concentrate as follows:

	Parts by weight
Water	74.58
Antirust agent	0.30
Tri - sodium salt of ethylenediaminetetra acetic acid	0.10
Sodium salt of pentachlorophenol	0.02
Product of Example 1	25

The above formulation is very effective as a mold release agent in the die casting of aluminum.

In carrying out the mold release use for the product, a solution of 40 parts of water to 1 part of the above formulation was applied to the die by hand spray prior to closing the die and then injection of approximately 2 pounds of molten aluminum at 1200° F. was made. Casting time was six seconds with an approximate pre-injection die temperature of 450° F. The part cast was the body of a saber saw. When the die was open no sticking of the part to the die was experienced and the quality of the surface of the cast part was considered good. In a comparative test where the mold release agent was composed of a higher cost colloidal graphite in water, the graphite product gave good mold release at comparable dilution, but resulted in poorer surface of the cast part.

The products of the invention are also useful in the cold reduction of steel where the lubricant formulation is used at dilutions of about 8 to about 25 parts of water to one part of the formulated product. Likewise, in the hot reduction of steel, the lubricant may be used as an additive to the cooling water used on the mill at a concentration of from 0.01% to 0.5% or may be applied to the rolls in the mill from a separate system at a concentration in water of from 1% to 20% parts by weight.

In chip forming metal working, the formulations of the invention will be used as concentrations in water of from 1% to 20% of various cutting and grinding operations of ferrous and non-ferrous metals. Likewise, in wire drawing of ferrous and non-ferrous metals, the lubricant is useful at concentrations in the water of 5% to 15% to draw copper, steel, and copper coated steel wire. In metal deformation processes, the formulations may be used as concentrations of from 10% to 100% in water for lubrication of the deep drawing of metal. The lubricants are also useful in hot working and other procedures well known in the trade.

The compositions of the invention generally provide excellent cooling, and excellent uniform lubrication, resulting in a more uniform surface of the work metal, easier cleaning of the residual film of lubricant and a greater economy owing to the longer life of the lubricant solution due to the greater immunity to change of the operating parameters. By using the lubricants of the invention, good tool life, good finish on the machine part, and good lubrication in moving parts is generally achieved. The lubricant of the invention has excellent ability to deposit a film on a hot surface making it a particularly good lubricant for hot working of metals such as forging, hot rolling, and die casting.

The lubricants of the invention are also of value as a rubber parting agent in extrusion of crude rubber and steam curing, and are able to replace the conventional soap/silicone polymer mixtures used heretofore. In order to further illustrate the characteristics of the lubricants of the invention, the following test results are given in the table which follows:

hydroxy compounds present with phenylstearic acid until the acid number of the product is below six.

3. A lubricant formulation comprising an aqueous lubricating solution containing at least about 50% by weight of water and from about 5 to 50% by weight of a water soluble lubricant composition obtained by transesterifying at a temperature of from about 400° to 450° F. and in the presence of a buffer catalyst 1 mole of castor oil with 1 mole of polyoxyalkylene polyol having a molecular weight of at least about 1000 until the reaction products are water soluble and then esterifying the hydroxy compounds present with monosodium azelate until the acid number of the product is below six.

4. A lubricant formulation comprising an aqueous solution containing at least about 50% by weight of water and from about 5 to 50% by weight of a water soluble lubricant composition obtained by transesterifying at a temperature of from about 400° to 450° F. and in the presence of a sodium acetate catalyst, 1 mole of castor oil with 1 mole of a polyethyleneoxy, polypropylene oxy polyol of molecular weight of about 3000 until the reaction products are water soluble and then esterifying the hydroxy compounds present with stearic acid until the acid number of the product is below six.

5. A process for lubricating a metal surface in friction generating contact with another surface which comprises applying to the metal surface a lubricating film of the composition of claim 1.

6. A process for lubricating a metal surface in friction generating contact with another surface which comprises applying to the metal surface a lubricating film of the composition of claim 2.

7. A process for lubricating a metal surface in friction generating contact with another surface which comprises applying to the metal surface a lubricating film of the composition of claim 3.

8. A process for lubricating a metal surface in friction generating contact with another surface which comprises applying to the metal surface a lubricating film of the composition of claim 4.

TABLE.—TEST CHARACTERISTICS OF 2% BY WEIGHT AQUEOUS SOLUTION OF LUBRICANTS AT 100°–130° F.

	Product tested			Example			
	(A) Commercial rolling oil	(B) Commercial drawing oil	(C) Soluble cutting oil [3]	1	2	3	4
Timken maximum 10 min. pass load	8	8	12	20	10	20	20
Amsler, coef. of friction, steel on steel (4140)	.045	.047	.049	.031	.039	.046	.041
Falex torque at—							
500 lb. load	10.0	11.0	12.0	9.0	7.0	8.0	10.0
1000 lb. load	14.5	16.5	20.0	17.0	16.0	18.0	20.0
2000 lb. load	30.0	30.0	35.0	27.0	19.0	28.0	31.0
3000 lb. load	35.0	37.0	43.0	38.0	28.5	38.0	40.0
4000 lb. load	39.5	46.0	Fail	46.0	42.5	35.0	45.0
4500 lb. load	45.0	52.0	----	46.0	42.0	38.0	46.0

[1] 90% beef tallow; 8% tallow fatty acids; 2% emulsifier.
[2] 50% 200 S.U.S. at 100° F. mineral oil; 40% lard oil; 5% oleic acid; 5% emulsifier.
[3] 10% chlorinated paraffin 40%; 10% lard oil; 65% 200 S.U.S. at 100° F. mineral oil; 15% sodium sulfonate.

I claim:

1. A lubricant formulation comprising an aqueous lubricating solution containing at least about 50% by weight of water and from about 5 to 50% by weight of a water soluble lubricant composition obtained by transesterifying at a temperature of from about 400° to 450° F. and in the presence of a buffer catalyst 1 mole of castor oil with from 0.75 to 2.0 moles of polyoxyalkylene polyol having a molecular weight of at least about 1000 until the reaction products are water soluble and then esterifying the hydroxy compounds present with a member of the group consisting of high molecular weight fatty acids, phenyl substituted fatty acids and aliphatic dicarboxylic acids until the acid number of the product is below six.

2. A lubricant formulation comprising an aqueous lubricating solution containing at least about 50% by weight of water and from about 5 to 50% by weight of a water soluble lubricant composition obtained by transesterifying at a temperature of from about 400° to 450° F. and in the presence of a buffer catalyst 1 mole of castor oil with 1 mole of polyoxyalkylene polyol having a molecular weight of at least about 1000 until the reaction products are water soluble and then esterifying the

References Cited

UNITED STATES PATENTS

2,925,429	2/1960	Kirkpatrick et al.	260—404.8
2,971,923	2/1961	Walker et al.	252—342
3,202,607	8/1965	Koch	252—56 R

FOREIGN PATENTS

820,571	9/1959	Great Britain.

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