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LUBRICATING COMPOSITION

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ABSTRACT OF THE DISCLOSURE

A ternary lubricating composition having suppressed foaming properties comprising a condensate of a fatty acylating substance having an average of about 12 to 20 carbons in the acyl group and an alkylolamine having at least one acylatable hydrogen atom in the amino group, an alkylolamine soap of a fatty acid and polypropylene glycol having a molecular weight of between about 1,000 and 10,000 is disclosed. The compositions are useful in the drawing of copper metal so that the drawing may be accomplished with substantially no insoluble copper soap formation.

This invention relates to an improved lubricant and coolant for use in metal working. More particularly, the invention has to do with the preparation of an improved lubricant having superior lubricating properties and a greatly reduced tendency to foam.

It is common in the drawing of copper tubing and wire to use a blend of soap and fat to act as a lubricant at the point of extrusion. This lubricant is generally applied in an aqueous solution, i.e., an oil-in-water emulsion and at concentrations of 5% total solids or less. The resultant emulsion coats out on the copper wire or tubing or copper coated wire or tubing and provides lubrication at the draw point.

Blends of soaps and fats have provided adequate lubricity, yet they are not completely without drawbacks since there is a great tendency for them to form insoluble copper soaps. These soaps eventually precipitate to form large quantities of flocculent material having essentially no value in the process. In industrial practice it has been found necessary to construct holding tanks for the soap-fat emulsion in such a manner so that they are adequately baffled to permit the copper soaps to form an undisturbed sediment. This sediment is then manually removed from the tanks on a periodic basis.

Various attempts to improve upon the soap precipitation situation led to the use of alkanolamine condensates and/or salts. While some of these prior art compounds have overcome the problem of soap precipitation, they are not the most desirable substances due to their limited lubricity and their great tendency to form copius quantities of foam. The great majority of operations wherein copper wire or copper tubing is drawn involve a recirculating system where the lubricant solution is allowed to fall into the storage tank or it is circulated by pumps that can imbibe air into the liquid. Thus the majority of industrial installations are unable to take advantage of these products.

Another associated area is in the cold deformation of metals, such as the area of cold rolling steel sheet metal. In the cold rolling of steel sheet, a lubricant is applied to the sheet material to be reduced as it enters the first stand or bite of the rolling mill. The lubricant is generally a quasi stable emulsion of palm oil, tallow or some other fatty material. Generally speaking, the run-off and application of emulsified lubricant runs into the sewer and is discarded. There are several disadvantages to this procedure. As part of a program to eliminate potential sources of pollution, steel mills are switching to a recirculating type system. Here again, however, the inherent foaminess present in most of the prior art lubricants tend to preclude their use.

Another disadvantage of the fat-type emulsion is that the lubricant emulsion actually heats up the mill. Application of heat is now necessary to keep the internal phase of the emulsion liquid in the case of palm oil and tallow emulsions. Thus, if the lubricants could be applied at ambient or reduced temperatures, they would result in substantial increases in mill efficiency.

It is accordingly an object of this invention to provide an improved lubricant for metal working.

It is also an object of this invention to provide a metal working lubricant which will permit high speed rolling of steel or other metal plate.

It is a further object of this invention to provide an improved lubricant for metal working that has a greatly reduced foaming tendency.

It is a still further object of this invention to provide a lubricant which will permit the drawing of copper wire, copper coated wire and tubing without the formation of water insoluble copper soaps.

An additional object of this invention is to provide a base formulation for preparing paste type lubricants useful for the bright drawing of copper tubing and wire.

Additional objects, if not specifically set forth herein, will be readily apparent to those skilled in the art from the detailed description of the invention which follows.

In general, the novel lubricating compositions of this invention comprise a ternary mixture of an alkylolamine soap of fatty acids having an average of between about 12 and about 20 carbons in the acyl portion, a condensation product of an alkylolamine having at least one acylatable hydrogen atom in the amino group and a fatty acylating substance having an average of between about 12 and about 20 carbons in the fatty acid group, and polypropylene glycol having a molecular weight of between about 1,000 and about 10,000. The condensate may be further defined as comprising essentially fatty acid amides of the alkylolamine with a non-performance affecting amount of fatty acid monoester of the alkylolamine as well as small amounts of glycerine and free amine. While not preferred, the percentage of the ester, based on the weight of the condensate, can vary from 10 to 40% without substantially affecting the lubricating performance. However, in general, the condensate when first formed, will have an amide:ester ratio of about 7:3. In the presence of an amine soap, the condensate is aged so that it will end up with a low ester content. This low ester content of the condensate can be defined as less than about 10%, based on the weight of the condensate.

While amounts of the ingredients of the ternary blend may vary somewhat, synergistic lubricating properties are present when the condensate is present in amounts of between about 44% and about 77%, the amine soap present in amounts of about 3% to about 16% and the polypropylene glycol in amounts of about 20–40%. Slightly more specific blends include those compositions wherein the condensate is present in amounts of about 47% to about 71%, the soap is present in amounts of between about 6% and about 13%, and the polypropylene glycol present in amounts of about 20% to 40%. Specific blends as to the polypropylene glycol are more preferred in some instances and these comprise blends of about 28% to 32% polypropylene glycol, about 44% to about 77% condensate and about 3% to about 16% amine soap. Most preferred compositons having synergistic lubricating characteristics yet possessing extremely low foaming properties and being capable of forming homogenous solutions not subject to hard water incompatability comprise those compositions containing about 28% to 32% polypropylene glycol, about 9% to about

13% amine soap and about 57% to 61% condensate. The above percentages are all weight percents.

More in detail as to the description of the various components, the polypropylene glycol may be formed by the polymerization of propylene oxide with propylene glycol. The terminal oxyalkylene group may be a butylene oxide or an ethylene oxide, as well as propylene oxide. Of special importance are the polypropylene glycols having molecular weights varying between about 1,000 and about 10,000. The preferred results are produced using polypropylene glycol having a molecular weight of approximately 2,000.

In preparing the amine soap and condensate, it is preferred to make mixtures of the condensate and amine soap and combine them with condensate containing some free amine. That is, in producing the amide, equal parts by weight of a fatty acylating agent and alkylolamine may be heated at temperatures varying between about 140° C. and 200° C. for ½ to 6 hours. The resulting mixture may then be cooled and to three parts of the said mixture one part of a fatty acid averaging between about 12 and 20 carbon atoms is added. That free fatty acid forms the soap with the unreacted alkylolamine from the initial condensation. That resulting blend consists of about 50 parts condensate, 35 parts amine soap, and a total of 15 parts free amine and glycerin. Perhaps it should be stated at this point that the free amine, while desirable, is not essential and that generally speaking the free amine and glycerin have substantially no effect on the superior lubricating properties of the final composition. Approximately 30% of the resulting mixture is then blended with 40% of a product formed by the reaction of about 3 parts of fatty acid with one part of alkylolamine at temperatures of around 300° F. under vacuum to form a mixture of about 90 parts condensate and about 10 parts free amine. The entire mass can then be blend with about 30 parts of polypropylene glycol to arrive at a final composition having about 33% polypropylene glycol, about 57% condensate and about 11% amine soap.

A specific example of preparing an amine soap and condensate is as follows:

The amide of coconut oil is produced by heating equal parts of coconut oil and diethanolamine to a temperature of about 150° C. for a period of 3–4 hours. The resulting mixture is then cooled and three parts of said mixture are blended with one part of oleic acid. The oleic acid forms the soap with the free or unreacted diethanolamines from the initial condensation. The resultant blend consists of glycerine, free or unreacted diethanolamine, diethanolamide of coconut fatty acids, and related by-products in the diethanolamine soap of oleic acid.

A solution containing a predominant amount of condensate is prepared by reacting 73 parts of oleic acid with 27 parts of diethanolamine. The two ingredients are caused to react at a temperature of approximately 300° F. under vacuum of about 27–29 inches of mercury. Generally about 30% by weight of the first-described product is combined with 40% by weight of the condensate product and with 30% of a polypropylene glycol. Such a composition, even though containing small amounts of glycerine and free amine, will exhibit excellent lubricating properties. The amine, while not essential, is preferred in some instances where bright drawing of copper metals is contemplated.

In the above-described reactions, diethanolamine or diisopropanolamine will generally be used. However, monoalkanolamines such as monoethanolamine or monoisopropanolamine may be used either alone or in mixtures with diethanolamine or diisopropanolamine to form the condensate. Mixtures containing mono-, di-, or trialkanolamines may be also used with the trialkanolamine forming the soap component. The acylating substance is preferably a monocarboxylic acid or derivative thereof having an average of 12 to 20 carbons in the acyl portion. By acylating substance is meant any substance which is capable of the formation or introduction of an acyl radical in or into the alkylolamine and includes carboxylic acids, carboxylic acid halides, carboxylic acid esters and anhydrides. In regard to the esters, the term is meant to cover both esters of monohydric alcohols and esters of polyhydric alcohols such as the glycerol esters.

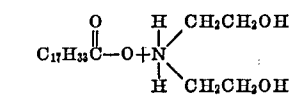
The ternary ingredients of the compositions of this invention were tested singly and in combination on the Baroid mud tester. This machine was designed to evaluate extreme pressure, lubricant additives to drilling mud and measures inch pounds of torque between zero and 600 pounds. Modification had to be made in the torque wrench portion of the apparatus in order to measure the readings at the optimum synergistic areas. The mud tester is essentially a takeoff on the Timken wear testing machine using a smaller cup and the same size block. The principle is that the rotating cup or ring is brought into contact with the block of metal while the entire system is immersed in the lubricant solution being tested. Torque is applied to the torque wrench and the value at which the amperage required by the motor takes a sharp jump is noted. This sharp jump indicates lubricant failure at the interface resulting in an increased load on the motor.

The individual values for polypropylene glycol having a molecular weight of about 2,000 averaged 150 inch pounds of torque at seizure. For the condensate, i.e., the amide with a low ester content, the value was 380 inch pounds of torque while with the amine soap, the value was about 320 inch pounds of torque at seizure. Accordingly, it can be seen that one would normally expect the value at seizure to be somewhat less than 380 inch pounds of torque when the three ingredients are combined in any given ratio. While it is true that torque values of less than 380 inch pounds are produced at various ratios of ingredients, it was unexpectably found that at certain levels, the ternary mixture produced clearly synergistic results. When using a ternary blend of ingredients in the ratios set forth in the earlier part of this specification, torque values well above 450 inch pounds were obtained. The lubricating value at the point described as the optimum blend was in excess of 635 inch pounds of torque at seizure. Such values were produced using a composition containing about 30% polypropylene glycol, about 60% condensate and about 10% amine soap.

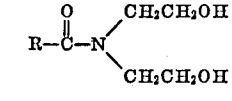
The following examples are presented to illustrate the invention. It will be understood that these examples are illustrative only and should not be taken in any manner as limiting the invention as defined by the appended claims.

EXAMPLE I

In the charts set forth in this example the amine soap is diethanolamine oleate having the structure:



The condensation product is essentially the diethanolamide of coconut oil and oleic acid with a low ester content. The amide may be represented by the formula:



wherein R is an alkyl or alkenyl radical averaging between about 12 and about 20 carbons. The lauric acid amide forms the major portion of the amide composition. The polypropylene glycol has a molecular weight of about 2,025.

In carrying out this example, the amine soap, condensate and polypropylene glycol were blended at various levels and used in the Baroid mud tester. The torque values are the average value in inch pounds of torque at seizure, i.e. the point at which the lubricant failed.

CHART NO. 1

	Condensation Product	Polypropylene Glycol	Torque
Amine Soap:			
80	0	20	440 in. lb.
25.6	54.4	20	308 in. lb.
22.4	57.6	20	190 in. lb.
19.2	60.8	20	308 in .lb.
16.0	64.0	20	403 in. lb.
12.8	67.2	20	532 in. lb.
9.6	70.4	20	515 in .lb.
6.4	73.6	20	362 in. lb.
3.2	76.8	20	434 in. lb.

CHART NO. 2

	Condensation Product	Polypropylene Glycol	Torque
Amine Soap:			
70	0	30	370 in. lb.
22.4	47.6	30	293 in. lb.
19.2	50.8	30	243 in. lb.
16.0	54.0	30	438 in. lb.
12.8	57.2	30	490 in .ln.
9.6	60.4	30	635 in. lb.
6.4	63.6	30	407 in. lb.
3.2	66.8	30	437 in. lb.
0	70.0	30	217 in .lb.

CHART NO. 3

	Condensation Product	Polypropylene Glycol	Torque
Amine Soap:			
60	0	40	370 in. lb.
19.2	40.8	40	340 in. lb.
16.0	44.0	40	479 in. lb.
12.8	47.2	40	548 in. lb.
9.6	50.4	40	461 in. lb.
6.4	53.6	40	520 in. lb.
3.2	56.8	40	468 in. lb.
0	60.0	40	294 in. lb.

CHART NO. 4

Condensation Product	Polypropylene Glycol	Torque
0	100	150 in. lb.
60	40	293 in. lb.
70	30	213 in. lb.
100	0	380 in. lb.

CHART NO. 5

Amine Soap	Polypropylene Glycol	Torque
100	0	320 in. lb.
80	20	440 in. lb.
70	30	370 in. lb.
60	40	370 in. lb.
0	100	150 in. lb.

CHART NO. 6

Amine Soap	Condensation Product	Torque
100	0	320 in. lb.
35	65	235 in. lb.
0	100	380 in. lb.

EXAMPLE II

Various experimental runs were made using the lubricant of this invention dispersed in water of concentrations ranging from about 1/10 percent to about 10%. Copper tubing, copper wire, and copper coated steel wires were drawn through various dies lubricated with the compositions containing about 11% amine soap, 59% condensate and 30% polypropylene glycol. The ternary ingredients are those defined in Example I. Using this system, a very adequate die life was observed. Perhaps one of the outstanding properties of the ternary lubricating composition was the fact that essentially no flocculent copper soaps were formed during the drawing process. Accordingly, there was no problem of sludge formation.

In addition, the ternary lubricants can be the basis for formulating a paste-type lubricant used in the bright drawing of copper wire as shown by the following example.

EXAMPLE III

The ternary lubricant in an amount of 45%, by weight, was combined with 25%, by weight of water and about 30% diatomaceous earth. These materials were blended together in a Mixmueller crutcher designed to handle high viscosity pastes. This paste had a stable viscosity over a relatively wide temperature range, i.e., 80 to 120° F. Copper wire drawn through the paste had a very bright finish.

EXAMPLE IV

The improved lubricants of this invention may also be used for the working of ferrous materials such as steel. Using the optimum blends set forth in Example I as the lubricating composition in the cold-rolling of steel, it was found possible to roll at a speed of 3,500 feet per minute to reduce the thickness of the sheet from .05 inch to .017 inch in a 1 stand mill. The steel was adequately lubricated with no staining of the worked surfaces. One attractive feature of this system was that the lubricant was applied at ambient temperatures. This is in contrast to the normally used palm oil or tallow emulsions which must be heated. Using the instant lubricant at ambient temperatures naturally decreases the temperature of the mill.

While specific examples have been set forth above, it should be noted at this time that the improved lubricating compositions of this invention are adaptable for lubricating metals during various processing operations such as cutting, drilling, tapping, milling, drawing and the like. The lubricating compositions of this invention may be used at full strength, however, they are generally dispersed in water in amounts of 1/10 to 10%, preferably 2–4%. In some instances oil-in-water emulsions may be formed. However, for purposes of this specification, the term dispersing will include all types of colloidal systems including emulsions.

It is apparent that many modifications and variations of the invention may be made without departing from the spirit and scope thereof and, accordingly, only such limitations should be imposed as are indicated in the appended claims.

I claim:

1. A ternary lubricating composition having suppressed foaming properties comprising a condensate, in an amount of between about 44% and about 77%, which contains predominantly an amide prepared by the reaction of a fatty acylating substance having an average of about 12 to 20 carbon in the acyl group and an alkylolamine having at least one acylatable hydrogen atom in the amino group; an alkylolamine soap of a fatty acid in an amount of between about 9% and about 13%; and polypropylene glycol having a molecular weight of between about 1,000 and about 10,000 in an amount of between about 20% and about 40%.

2. The composition of claim 1 wherein the condensate is present in an amount of between about 57%, to about 61%, the amine soap is present in an amount of between about 9% and about 13% and the polypropylene glycol is present in an amount of between about 28% to about 32%.

3. The composition of claim 1 wherein the condensate comprises an amide of a member selected from the group consisting of diethanolamine and diisopropanolamine and the acylating agent is selected from the group consisting of coconut oil and oleic acid or mixtures thereof.

4. The composition of claim 1 wherein the amine soap is selected from the group consisting of monoethanolamine

oleate, monopropanolamine oleate, diethanolamineoleate, diisopropanolamine oleate, triethanolamine oleate, triisopropanolamine oleate and mixtures thereof.

References Cited

UNITED STATES PATENTS

2,079,803	5/1937	Holtzclaw et al.	252—49.5 XR
2,624,708	1/1953	Langer et al.	252—49.3 XR
2,668,146	1/1954	Cafcas et al.	252—33.4
3,177,144	4/1965	Reamer et al.	252—49.3 XR
3,233,442	2/1966	Zvanut	252—49.3 XR
3,244,638	4/1966	Foley et al.	252—49.5 XR
3,311,557	3/1967	Schiermeier	252—49.3 XR

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