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WIREDRAWING LUBRICATION

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ABSTRACT OF THE DISCLOSURE

A concentrate for use in forming an aqueous wiredrawing lubricant comprising a mixture of sulphated sperm oil and sulphated rapeseed oil. The concentrate may be mechanically dispersed in the water to form the lubricant or a surfactant may be included in the concentrate to aid dispersion.

The present invention relates to the lubrication of wires which are subjected to wiredrawing processes.

Metal wire is made by drawing a rod or wire through a die or series of dies of durable material which reduce the diameter of the wire while elongating the wire and sometimes producing changes in the quality of the metal of the wire.

The frictional forces generated during wiredrawing are considerable and it is desirable to lubricate the passage of the wire through the die(s) in order to mitigate the frictional forces, avoid the generation of excessive heat in the die(s) which would adversely affect the die(s) and the properties of the wire, and to ameliorate the degree of attrition of the wire and the die(s).

Usually the die(s) and the wire are immersed in a sump or bath of lubricant so that lubricant is available to the critical regions of each die and so that heat is quickly dissipated. Wires formed wholly or mainly of copper or materials having similar drawing characteristics are usually drawn through dies at relatively high speeds (for example, 3000 feet per minute): consequently, the need to dissipate heat from the die is important and water-based lubricants are used in order to make use of the high specific heat of water.

The water-based lubricants which have previously been used have included an emulsion of a fatty oil such as rapeseed oil. These prior lubricants were subject to a number of disadvantages such as poor emulsion stability, despite the presence of emulsifiers, and excessive formation of metallic soaps which trapped metal fines abraded from the wire causing clogging or excessive wear of the die and producing a deleterious change in the composition of the emulsion. In addition, the metallic soaps and fines contaminated the lubricating emulsion forming a scum which impaired the properties of the emulsion and deposited on the sides of the sump, necessitating frequent cleaning of the sump with consequent loss of through-put. Some previously-known aqueous emulsions emulsions employed as wiredrawing lubricants also have been known to attack ferrous metal parts of the wiredrawing machine, causing corrosion.

According to the present invention, a concentrate for making an aqueous wiredrawing lubricant comprises sulphated rapeseed oil and sulphated sperm oil.

By "sulphated oil" is meant the product obtained by treating the oil with relatively concentrated sulphuric acid for several hours at temperatures of from 0° C. to 40° C. to cause sulphation of the oils to take place at double bonds to form true sulphates rather than sulphonates. The SO_3 content of such sulphates may range from 0.5 to 10 wt. percent depending on the strength of the sulphuric

acid, the nature of the oil and the conditions of treatment.

After sulphation, the oils are water-washed and then treated with a base such as ammonia or caustic soda solution until the pH is 6.5 to 10.0, preferably 7.0 to 9.5.

The compositions of suitable sulphated oils (as determined by the methods of analysis prescribed in "Standard Methods of Analysis" published by the Society of Leather Trade Chemists, The National Leather Sellers College, 176 Tower Bridge Road, London, SE. 1.) are as follows, expressed in weight percentages on a basis of 100 wt. percent total fatty matter by extraction:

TABLE I

	Sulphated sperm oil	Sulphated rapeseed oil
Total fatty matter by extraction	100	100
Combined SO_3	1.1–2.3	2.3–4.1
Neutral oil	74–85	70–82
Free fatty acids, as oleic acid	1.1–6	1.1–6
Unsaponifiable matter	30–33	1.2–2.3
Total alkali, expressed as NaOH	0.2–1.2	0.2–1.8

Generally speaking, the sulphated oils will comprise a certain amount of water, and typical ranges of compositions of each of the sulphated oils, expressed in weight percentages on a basis of 100 wt. percent sulphated oil are as follows:

TABLE II

	Sulphated sperm oil	Sulphated rapeseed oil
Total fatty matter by extraction	85–90	84–88
Combined SO_3	1.0–2.0	2.0–3.5
Neutral Oil	65–75	60–70
Free fatty acids, expressed as oleic acid	1.0–5.0	1.0–5.0
Unsaponifiable matter	26.5–29.0	1.0–2.0
Total alkali, as NaOH	0.2–100	0.2–1.5
Inorganic salts	0.5–1.5	0.5–1.5
Water	10.5–5.5	10.5–5.5

The pH of 10 vol. percent emulsions in water of both sulphated oils is from 7.5 to 9.5 in each case.

The concentrate may comprise between 25 vol. percent and 75 vol. percent of sulphated rapeseed oil, the remainder being sulphated sperm oil.

Particularly preferred concentrates according to the invention comprise about 50 vol. percent sulphated rapeseed oil, the remainder being sulphated sperm oil.

Preferably, the free fatty acid content of the concentrate is below 3.0 wt. percent expressed as oleic acid, and the wiredrawing properties of the aqueous emulsion of the concentrate are most satisfactory when the free fatty acid of the concentrate is initially from 2.5 to 3.0 wt. percent expressed as oleic acid. The amount of free fatty acid in the concentrate is determinable by the degree of sulphation and neutralisation during manufacture. The concentration of free fatty acid may change during the wiredrawing operation, but it has been found that the fatty acid content of the concentrate before addition to the water is important in obtaining the most advantageous results in the wiredrawing process.

The concentrates according to the invention are found to give aqueous wiredrawing lubricants having extremely good emulsion stability and long working life without excessive formation of copper soaps. The scum and metal fines deposition of previously-known lubricants are substantially absent, or mitigated to an unprecedented extent. Another benefit of the wiredrawing lubricant made from the concentrate from the operatives point of view is that substantially no unpleasant odour is produced even under arduous working conditions.

The invention also includes an aqueous wiredrawing lubricant comprising a major proportion of water and a minor proportion, such as 1 to 20 vol. percent, of the concentrate described in the preceding paragraphs. Usually, the lubricant will comprise 2 to 9 vol. percent of the concentrate and 5 to 7 vol. percent is a preferred range of

compositions. It is to be understood, however, that during the use of the wiredrawing lubricant, the concentrations of the sulphated rapeseeed and sperm oils will change and additions of concentrate to the lubricant to restore the preferred range of compositions will be desirable.

The aqueous wiredrawing lubricant may be made simply by adding the concentrate to water in the desired proportions, and stirring or agitating until satisfactory dispersion is effected: generally speaking, the better the degree of dispersion, the greater will be the stability of the emulsion, and emulsion stability is an important factor in ensuring satisfactory lubrication.

The stirring of the concentrate in the water may be performed manually by means of a paddle, or mechanically by means of an industrial stirrer of any type. In the case of manual stirring, the degree of dispersion depends on the conscientiousness of the personnel to a great extent: where the operatives cannot be relied upon to give the concentrate a suitable degree of dispersion, it may be more attractive in a wiredrawing plant to rely on physico-chemical means of dispersion rather than to employ a mechanical stirrer which occupies floor space, and due to intermittent usage, would represent uneconomically deployed capital. To this end a surfactant may be incorporated in the concentrate. A suitable surfactant is the sulphonated castor oil product commercially available under the name Turkey Red oil (hereinafter designated TRO). TRO mixes readily with the sulphated oils of the concentrate, and the concentrate of the invention may comprise 3 to 7 volume percent of TRO or any other suitable surfactant: a preferred concentration of the surfactant would be about 5 vol. percent.

The invention further includes a method of drawing wire in which a wire lubricant with an aqueous wiredrawing lubricant formed from the concentrate hereinabove described is drawn through one or more dies.

In addition, the invention includes wire drawn by the method of the preceding paragraph.

The invention will now be further illustrated by reference to some tests carried out on a number of aqueous wiredrawing lubricants:

Sulphated rapeseed and sperm oils were prepared by treating the oils either separately or in combination with 96% sulphuric acid for several hours at between 0° C. and 40° C. in agitated tanks. The contents of the tanks were allowed to separate into an upper oily layer and a lower acid layer, the latter being run off. The oily layer was then washed with water, and caustic soda solution added until the pH of a 10 vol. percent emulsion in water was from 6.5 to 9.5.

The properties of the sulphated oils were approximately as follows:

TABLE III

	Sulphated sperm oil	Sulphated rapeseed oil
Total fatty matter by extraction wt. percent	87.5	86.0
Free fatty acids (wt. percent as oleic acid)	2.0–3.0	2.0–3.0
Total alkali (as NaOH) wt percent	0.5	0.2
Water, wt. percent	8.0	8.3
Combined SO_3, wt. percent	1.5	3.2
Unsaponifiable matter, wt. percent	27.7	
Neutral oil content, wt. percent		70.0

Tests were carried out in which a high conductivity copper wire comprising substantially pure copper was drawn through 15 successive dies to reduce its diameter from 0.104 to 0.018 inch, the final speed of the wire being 3000 feet per minute. Different wiredrawing lubricants (300 gallons per test) were used in each of the tests as follows:

TEST 1

The lubricant was a 6 volume percent emulsion of the above-specified sulphated sperm oil in water, similar to known commercially-used wiredrawing lubricants.

After a short operating time, a scum of copper fines appeared on the surface of the lubricant and a build-up

of copper soaps and copper fines was apparent on the dies and the sides of the tank containing the lubricant, rendering the lubricant unsuitable for further use. In addition, the sulphated sperm oil began to de-emulsify, thus impairing its remaining lubricating properties.

TEST 2

The wire was lubricated with a 6 volume percent emulsion of the sulphated rapeseed oil in water. The emulsion maintained its stability even though during the test the concentration of the rapeseed oil fell to about 2%. Less copper soaps and deposits were produced than in test 1, but a build-up on the dies and the sides of the tank was observed. An unpleasant odour arose during the wiredrawing process. The sulphated rapeseed oil is somewhat disadvantageous from a cost point of view compared with the sulphated sperm oil.

TEST 3

Wiredrawing was undertaken using a lubricant comprising 6 volume percent of an emulsion in water of a concentrate comprising 75 wt. percent of the sulphated sperm oil and 25 wt. percent of the sulphated rapeseed oil and having a free fatty acid content of between 2.5 and 3.0 wt. percent, expressed as oleic acid.

The emulsion was very stable even when its concentration was allowed to fall to 4 vol. percent. Copper soap formation was markedly decreased compared to the lubricants used in tests 1 and 2 and the amount of copper fines, scum and deposit was smaller.

TEST 4

The wire was lubricated with a lubricant comprising a 6 vol. percent emulsion in water of a concentrate comprising 50 vol. percent of the sulphated sperm oil and 50 vol. percent of the sulphated rapeseed oil, the free fatty acid content of the concentrate being 2.5 to 3.0 wt. percent on an oleic acid basis.

Copper fines, scum and deposit were virtually absent and the surface of the lubricant was almost wholly devoid of discolouring from this cause. The emulsion maintained its stability (i.e. there were no substantial signs of emulsion splitting) over 500 hours of operation with very little copper soap formation and the copper wire leaving the last die had a bright, smooth surface. There was no noticeable unpleasant odour and the general performance was considered to be slightly superior to that of the lubricant used in test 3. In addition, the lubricant was cheaper than the lubricant of test 3.

TEST 5

An emulsion for the lubrication of drawing of high conductivity copper wire in a heavy rod drawing machine was prepared. The essential features of the machine were the provision of only nine dies to effect a reduction in the wire diameter from 0.285 inch to 0.167 inch, corresponding to a reduction in cross-sectional area of the wire by 21% in each die. The speed of the wire drawn from the last die was 2500 feet per minute, and the amount of lubricant employed in the sump of the machine was about 800 gallons. The conditions of operation at the dies for this thicker copper wire were rather more severe than at the dies used for the thinner copper wire of tests 1 to 4. Accordingly, the tendency to form copper soaps and copper fines in the dies and the machine generally, and to cause consequential emulsion splitting, would be expected to be greater than in tests 1 to 4.

The emulsion consisted of 94 vol. percent water and 6 vol. percent of the concentrate of 50 vol. percent sulphated sperm oil and 50 vol. percent of sulphated rapeseed oil as employed in test 4, the concentrate having been dispersed by manual stirring into the water.

The copper wire leaving the last die was uniformly bright and smooth and there was virtually no scum of copper fines and only insignificant amounts of copper soap formation at the dies. However, some emulsion-

splitting was observed after 2 months use, involving an aggregated working period of 250 hours, and investigation established that the emulsion splitting could be avoided by ensuring a greater degree of dispersion of the concentrate in the water than could be achieved by manual stirring.

TEST 6

For reasons of convenience and economy, as explained above, it was preferred to derive a higher degree of dispersion of the concentrate by incorporating a surfactant in the concentrate rather than by using a mechanical stirrer.

The surfactant chosen was TRO, and a concentrate was formulated having the following composition:

Sulphated sperm oil—47.5 vol. percent
Sulphated rapeseed oil—47.5 vol. percent
TRO (sulphonated castor oil)—5.0 vol. percent
Free fatty acid (as oleic acid)—2.5–3.0 wt. percent.

The concentrate was manually dispersed in water to give a 6 vol. percent emulsion, and the wiredrawing was performed using the same machine and operating conditions as for test 5. Over a period of 4 months, aggregating 500 working hours, no scum of copper fines or build-up of copper soaps was observed, and there was no emulsion splitting. The quantity of concentrate which was added to the sump to maintain the 6 vol. percent concentration of the emulsion was only 30 gallons—a remarkably small quantity for such an extended period of service under relatively severe operating conditions.

The aqueous lubricant of the invention has been found not to attack ferrous metal parts of the wiredrawing machines even over substantially long periods of use, and this was confirmed by laboratory tests in which moist air was bubbled through the emulsions of the invention for 96 hours, while the emulsions were maintained at 60° C. and in contact with iron nails: no rusting of the nails was observed.

From the above, it will be seen that a concentrate of sulphated rapeseed oil and sulphated sperm oil (optionally with the inclusion of a minor proportion of a suitable surfactant, such as Turkey Red oil) gives a wiredrawing lubricant emulsion which is superior to either of the sulphated oils used alone, or to previously known wiredrawing lubricants.

We claim:

1. A concentrate for use in forming an aqueous wiredrawing lubricant comprising about 25–75 vol. percent of sulphated sperm oil and 75 to 25 vol. percent of sulphated rapeseed oil.

2. A concentrate according to claim 1 in which the properties of the sulphated sperm oil, on a non-aqueous basis, are substantially as follows:

	Percent
Total fatty matter by extraction	About 100
Combined SO_3	1.1–2.3
Neutral oil	74–85
Free fatty acids, as oleic acid	1.1–6.0
Unsaponifiable matter	30–33
Total alkali, as NaOH	0.2–1.2

3. A concentrate according to claim 1 in which the properties of the sulphated rapeseed oil, on a non-aqueous basis, are substantially as follows:

	Percent
Total fatty matter by extraction	About 100
Combined SO_3	2.3–4.1
Neutral oil	70–82
Free fatty acids, as oleic acid	1.1–6.0
Unsaponifiable matter	1.2–2.3
Total alkali, as NaOH	0.2–1.8

4. A concentrate according to claim 1 in which the sulphated oils are present in about equal proportions by volume.

5. A concentrate according to claim 1 in which the free fatty acids content, as oleic acid, is no more than about 3 wt. percent.

6. A concentrate according to claim 5 in which the free fatty acids content, expressed as oleic acid, is at least about 2.5 wt. percent.

7. A concentrate according to claim 1 comprising, in addition, effective amounts of a surfactant.

8. A concentrate according to claim 7 in which the surfactant is sulphonated castor oil.

9. A concentrate according to claim 7 in which the surfactant comprises about 3 to 7 vol. percent of the concentrate.

10. A concentrate according to claim 9 in which the surfactant comprises about 5 vol. percent of the concentrate.

11. A concentrate according to claim 1 comprising about equal proportions of sulphated rapeseed oil and sulphated sperm oil, and about 5 to 7 vol. percent of sulphonated castor oil, wherein the free fatty acid content, expressed as oleic acid, is from about 2.5 to about 3.0 wt. percent.

12. A wiredrawing lubricant comprising a major proportion of water and a minor lubricating proportion of a concentrate in accordance with claim 1.

13. A wiredrawing lubricant comprising water and 2 to 9 vol. percent of the concentrate of claim 11.

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