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(54) **Coning oil lubricant compositions.**

(57) Low-sling coning oils are achieved by the use of a composition comprising (a) a pituitive copolymer of a butene and a C_{5-20}alpha-olefin and (b) a lubricant comprising a mineral oil and/or a fatty ester.

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Coning oil lubricant compositions

This invention relates to lubricant compositions which are useful as coning oils.

Fibers which are used to manufacture garments are subjected during the manufacture and treatment of the fiber to rigorous processing conditions. Among the conditions to which a fiber will be exposed are high speed winding, knitting and weaving operations. To ensure that the fiber is not damaged during these operations a lubricant is added to reduce friction and transfer heat away from the fiber.

For ease of application to the yarn fibers, the lubricant or coning oil is used in a liquid state. The coning oil is introduced onto the yarn fiber by means of a kiss roll. The origin of the term kiss roll is that the yarn makes very minimal contact with a roll which is usually of stainless steel construction. The kiss roll rotates in a bath containing the lubricant which does not completely cover the kiss roll. The rotation of the kiss roll carries sufficient lubricant to the thread which contacts the roll at the top to impart the coning oil. Typically a kiss roll will operate at from 2 to 20 revolutions per minute.

For practical reasons, the kiss roll is usually an exposed piece of machinery and when excess coning oil is transferred to the yarn, there will be a considerable amount of sling off at or near the site of pick-up and at any point where the yarn fiber changes direction, e.g. at the cone.

As the coning oil is typically an organic substance a safety concern is encountered when substantial quantities of the coning oil are slung off. It has been observed that as much as 25% of a conventional coning oil (even with current oil sling additives) may be lost due to sling off.

There is thus not only a safety hazard presented but the yarn processor is not obtaining the most for his money as the coning oil cannot be easily recovered and recycled. Moreover, in some situations the coning oil is necessary for further operations such as in rewinding prior to the dyeing of yarns. In such a situation the manufacturer of the fiber sells the yarn to a dyeing company which rewinds the yarn at a high speed which is facilitated by having a lubricant on the yarn. Thus the required amount of lubricant must be on the yarn prior to the rewinding and any sling off loss during the original take-up is critical. The yarn may be retreated by the dyer with a lubricant and wound again at a high speed. The second application of lubricant is because the dyer must remove the lubricant prior to the dyeing operation.

It has been suggested in United States Patent 3,977,979, issued August 31, 1976 to Crossfield and in United States Patent 4,105,569, issued August 8, 1978 to Crossfield et al. to include a viscosity index improver such as a polyisobutylene. These materials while exhibiting some low sling capability are not sufficiently pituitive to substantially reduce the sling off.

British Patent 2,074,175 to Motier filed April 16, 1981 describes certain co-polymers which are useful within the present invention as a highly pituitive low sling coning oil additive. The use of the co-polymers of Motier in a coning oil are described in the August 19, 1982 issue of Industrial Chemical News which lists its address as P.O. Box 1409, Riverton, New Jersey 08077, USA.

It is thus an objective of the present invention to provide a low-sling off lubricant composition for use as a coning oil.

In one aspect, the present invention thus provides a coning oil lubricant composition comprising:

(a) a co-polymer of

(i) a monomer selected from 1-butene, 2-butene and isobutene and mixtures thereof, and

(ii) a C_{5-20} alpha-olefin and mixtures of two or more thereof; and

(b) at least one lubricant selected from

(i) mineral oil, and

(ii) a fatty ester containing a total of at least 6 carbon atoms and mixtures of two or more thereof.

In a further aspect the invention provides a process for treating a yarn with a coning oil lubricant containing a low sling additive to reduce lubricant loss from the yarn, said process incuding the steps of contacting the yarn prior to take-up of the yarn with a lubricant composition according to the invention, and taking up the yarn on take-up means therefor.

The first aspect to be discussed in connection with the present invention are the types of yarns which most benefit from treatment with the improved low sling coning oil lubricant composition of the invention. The yarns are typically polyester which is used for knits and woven outerwear or nylon used in men's or women's hosiery. The yarns most benefitted are of the textured type as opposed to flat yarns which have little bulk. Textured yarns are twisted to increase the bulk of the fiber and to give a more natural (cotton-like) appearance. The textured yarns are generally utilised such that they contain from 1 to 60 individual filaments with 5-40 filament yarn being the norm.

The winding speeds at which the textured yarns or for that matter flat yarns are wound,

generally ranges from about 400 meters per minute to about 1000 meters per minute. At the higher speeds slightly higher levels of the coning oil composition are needed to ensure that the proper lubricating effect is obtained.

The level of the coning oil composition typically applied is at from about 1% to 10% by weight of the yarn, preferably from 2% to 6% and most preferably from 3% to 4% by weight of the yarn when the yarn is used for knitting. When the yarn is used for weaving the most preferred range will be in the 1% to 2% area as weaving does not require the same degree of lubricity.

The lubricant component of the composition of the present invention is the mineral oil or fatty ester as hereinbefore defined. The lubricant will typically be used in the formulation at from about 50% to 95% by weight of the composition, preferably 80% to 92% and most preferably at from 85% to 90% by weight. Preferred mineral oils are of two types, that is the white oil which is essentially a paraffinic oil which contains little or no aromatic content, and process oil which contains a mixture of paraffinic and aromatic oils. The primary reason for using a mineral oil of either the white oil or process type is to provide a low friction response of the lubricated yarn. The main concern in the use of the mineral oil is the viscosity. Thus, generally, as long as the viscosity of the mineral oil in the present invention is within the range of 40-400 Saybolt Universal Seconds at 37.7°C (100°F) the mineral oil will be useful in the present invention. Most preferably the mineral oils will have a 40-100, preferably 40-60 Saybolt Universal Seconds viscosity. The mineral oils may be blended for various reasons to obtain the desired viscosity level. As a general rule the lower the viscosity the better, however, without an adequate low sling

agent added to the mineral oil the lower viscosity also gives a greater ability to sling off.

The second type of lubricant which may be used in the compositions of the present invention alone or in combination with mineral oil are esters.

Typical esters which are useful in the present invention include monobasic esters, dibasic esters and polybasic esters. Generally, esters which may be used of the monobasic type have the formula RCOOR' where R is an alkyl, alkylaryl, or aryl radical containing from 5-22 carbon atoms and R' is an alkyl, alkylaryl, or aryl radical containing from 1 to 22 carbon atoms. the total number of carbon atoms in the monobasic ester is preferably from about 20-32 carbon atoms. The values of R and R' are such that they may be branched or straight chained, saturated or unsaturated and further include guerbet alcohols. Preferred monobasic esters are butyl stearate and 2-ethyl hexyl stearate.

The dibasic esters which are particularly useful within the present invention are general of the formula $R(C0_2)R"COOR'$ where R and R' are as described above for the monobasic esters while R" for the dibasic esters is represented as $(CH_2)_x$ where x is from 0-12. Most commonly the dibasic esters will be oxalic, adipic and sebacic. Esters of phthalic acid while not within the structural formula shown above may be employed.

The polybasic esters which may be used are suitably those prepared by condensing fatty acids of about 6-18 carbon atoms with one or more materials such as trimethyolpropane, trimethyolethane, pentaerythritol and neopentyl glycol.

Where a mixture of mineral oil and an ester are desired it will commonly be blended at a ratio of from 10 to 50 parts of the ester to 90 to 50 parts of the mineral oil, most preferrably from

20 to 40 parts of the ester to 80 to 60 parts of the mineral oil.

The next aspect of the present invention to be discussed is the co-polymer component (a) which accounts for the low sling capability of the yarn finish lubricant. The co-polymer has a highly pituitive nature. The pituitive nature or stringiness of the copolymer causes the coning oil, which would otherwise be slung off, to form elongated strings of the coning oil composition and therefore to drop back into the kiss roll take-up pan. The take-up pan is the original source of the coning oil and thus the product is inherently recycled. Using coning oil not containing the copolymer component (a), the coning oil will form droplets which are easily slung off.

Preferably the co-polymer is one of 1-butene and the recited alpha olefins. These materials are more particularly described in British Patent 2,074,175 referred to above. The molecular weight of the polymers is conveniently from about 500,000 to 10,000,000, most preferably in the range of 1,000,000 to 5,000,000. It is noted, however, that the broadest useful range would generally be from about 100,000 to about 20,000,000 molecular weight.

The preferred co-polymers for use with 1-butene are 1-hexene, 1-octene, 1-decene, 1-dodecene and 1-tetradecene. Most preferably the co-polymer will be one of 1-butene and 1-decene.

In the co-polymer the butene and alpha-olefin components are preferably in a mole ratio to one another of from 90:10 to 10:90, most preferably of from about 70:30 to about 30:70. The copolymer is generally included in the coning oil at from about 0.01% to about 5.0%, preferably about 0.02% to about 4.0% by weight, and most preferably at from about 0.03% to about 2.0% by weight. The

compositions described herein are easily removable from the yarn which aids in dyeing applications.

Additional ingredients which may be included within the composition of the present invention include water, a polysiloxane, and an emulsifier to aid in removing the lubricant prior to the dyeing operation. Sugested emulsifiers are usually nonionic in nature, such as ethoxylated alcohols.

A further desired component in such compositions is an anti-static agent to prevent the build-up of static electricity during winding operations. The materials most typically used for anti-static agents are phosphate or sulfate esters of fatty alcohols and/or fatty alcohol ethoxylates. It is also noted that soap may be used as the anti-static agent.

In another aspect, the invention provides a process for the preparation of a coning oil lubricant composition which process comprises admixing components (a) and (b) defined above.

Throughout the present invention percentages and ratios are given by weight and temperatures as degrees Celsius unless otherwise indicated.

Example I

Into a two litre stainless steel reactor which has been purged with nitrogen and which is equipeed with a thermocouple, an agitator and a cooling jacket is charged 225 gms of kerosene which has been previously purified by passage through a bed of molecular sieve, 0.67 mole of dodecene-1, 13.7 mls of 25 weight percent solution of diethyl aluminum chloride in heptane and 1.5 gms of aluminum-activated titanium trichloride. Under a nitrogen blanket 0.67 mole of purified butene-1 is charged to the reactor. The reactor is sealed and the reactor pressure is adjusted to 20 $\pm$ 5 psig (137.9 $\pm$ 34.5 kPa gauge) with nitrogen and the reactor

temperature is reduced to 10°C. The reaction begins immediately upon addition of the reactants and catalyst. During the course of the rection the temperature is maintained at 10 ± 1°C, the pressure is autogenous and the reactor contents are agitated sufficiently to ensure a uniform temperature throughout the reaction mixture. One hour after the reaction begins, 164 gms of purified kerosene is added to the reactor to reduce the viscosity of the reaction mixture which increases as polymeric product is formed. After the kerosene addition the reaction is permitted to proceed for an additional four hours during which time no further viscosity reduction is necessary. The reaction is then terminated by the addition of sufficient alcoholic sodium hydroxide to completely inactivate the catalyst. The reaction product is stabilised by the addition of 200 ppm of a phenolic type antioxidant.

The conversion of monomer to polymer is 24%. The polymeric product contains 51 mole percent C_4 hydrocarbon units and 49 mole percent C_{12} hydrocarbon units. The polymeric product has a number average molecular weight of 1.14 million, a polydispersity of 4.5, and a weight average molecular weight of 5.13 million.

Example II

The procedure of Example I is repeated except that the reaction is conducted in the temperature range of -3 to -6°C for a period of twenty hours.

The conversion of monomer to polymer is 35%. The polymer in the product contains 52 mole percent of butene units and 48 mole percent of dodecene units and has a number average molecular weight 1.44 million, a polydispersity of 3.8 and a weight average molecular weight of 5.47 million.

Example III

The procedure of Example II is repeated except that the monomer charge comprises 0.89 mole of hexene-1 and 1.34 moles of butene-1, the amount of diethyl aluminum chloride is 9.1 mls of 25 weight percent solution and the reaction is carried out for eight hours.

The conversion of monomer to polymer is 15%. The polymer in the product contains 56 mole percent C_4 hydrocarbon units and 44 mole percent C_6 hydrocarbon units and will have a number average molecular weight above 1 million.

Example IV

To 1 part of a white mineral oil (50 SUS viscosity) is added 3 parts of the polymer solution of Example III. This resulting solution containing 3.0 weight percent copolymer is used to prepare the coning oils described in Examples V to VIII.

Example V

A coning oil formulation is prepared using the co-polymer solution obtained from Example IV.

To 80 parts of a white mineral oil of 50 SUS viscosity is added 0.8 parts of the copolymer solution of Example IV. To this mixture is added 15 parts of a nonionic emulsifier and 4 parts of a sulfated alcohol ether and 0.2 parts water. The product is tested by running a yarn through a standard kiss roll applicator to a cone. The product is tested at a take-up speed of 600 meters per minute. To test the sling reducing capability of the co-polymer, a cardboard disk is placed immediately beneath the cone. The amount of oil sling is determined by the amount of oil staining of the cardboard by the oil that is slung off the yarn. When compared to a coning oil containing the state-of-the-art low sling additive, the low sling coning oil of

the present invention exhibits approximately 50-75% reduction in oil sling.

Example VI

A further coning oil formulation is made as according to Example V utilising a blend of a paraffinic mineral oil and process oil at a respective ratio of 35:65. The total mineral oil content of 85 parts has added thereto 1 part of the co-polymer as described in Example IV. As additional ingredients in the formulation a nonionic ethoxylated alcohol emulsifier is present at 8 parts and an anti-static agent is utilised at 4 parts.

Water, perfume and additional materials are also added. It is noted with the above formulation that the speed of the kiss roll may be decreased to attain the same lubricant add-on exhibited by coning oils without the co-polymer additive. This is particularly valuable in that at higher speeds the kiss roll tends to deliver too much lubricant to the yarn and the lubricant does not have sufficient time to wick into the yarn. In such a situation the coning oil stays on the outside of the yarn and is more subject to throw-off. Therefore as an additional benefit to the present invention the coning oil is easier to introduce to the yarn from the kiss roll and as an energy savings the kiss roll can be run at a slower speed.

Example VII

A coning oil is prepared as in Example VI using as the base lubricant butyl stearate. The co-polymer is added at about 1.5%. This particular formulation exhibits an excellent take-up and low sling capability.

This example may be further modified by utilising a mixture of one of the mineral oils to the butyl stearate in a ratio of from about 10:90 to about

90:10. The product may be modified in Examples VI and VII by substituting as the copolymer a polymerised mixture of but-1-ene and dec-1-ene.

CLAIMS FOR THE CONTACTING STATES: BE, FR, DE, IT, NL, LU, SE, CH/LI, GB:

1. A coning oil lubricant composition comprising:
   (a) a co-polymer of
      (i) a monomer selected from 1-butene, 2-butene and isobutene and mixtures thereof, and
      (ii) a C_{5-20} alpha-olefin and mixtures of two or more thereof; and
   (b) at least one lubricant selected from
      (i) mineral oil, and
      (ii) a fatty ester containing a total of at least 6 carbon atoms and mixtures of two or more thereof.

2. A composition as claimed in claim 1 wherein said component (b) comprises as a mineral oil white oil.

3. A composition as claimed in claim 1 wherein said component (b) comprises as a mineral oil process grade mineral oil.

4. A composition as claimed in any one of claims 1 to 3 wherein component (b) comprises a mixture of a mineral oil and a fatty ester.

5. A composition as claimed in claim 4 wherein components (b)(i) and (b)(ii) are employed in a weight ratio of from 90:10 to 50:50.

6. A composition as claimed in any one of claims 1 to 5 wherein components (i) and (ii) of copolymer (a) are present therein in a mole ratio of from 90:10 to 10:90.

7. A composition as claimed in any one of claims 1 to 6 wherein component (a)(i) comprises 1-butene.

8. A composition as claimed in any one of claims 1 to 7 wherein component (a)(ii) comprises 1-decene.

9. A composition as claimed in any one of claims 1 to 8 wherein the molecular weight of co-polymer (a) is from about 500,000 to about 10,000,000.

10. A composition as claimed in any one of claims 1 to 9 wherein component (a)(i) is 1-butene and component (a)(ii) is 1-decene.

11. A composition as claimed in any one of claims 1 to 10 further containing a polysiloxane and/or an emulsifier.

12. A composition as claimed in any one of claims 1 to 11 wherein said copolymer (a) is present at from 0.01 to 5% by weight.

13. A composition as claimed in any one of claims 1 to 12 wherein said lubricant (b) is present at from 50 to 95% by weight.

14. A process for treating a yarn with a coning oil lubricant containing a low sling additive to reduce lubricant loss from the yarn, said process including the steps of contacting the yarn prior to take-up of the yarn with a lubricant composition as claimed in any one of claims 1 to 12, and taking up the yarn on take-up means therefor.

CLAIMS FOR THE CONTRACTING STATE: AT:

1. A process for treating a yarn with a coning oil lubricant containing a low sling additive to reduce lubricant loss from the yarn, said process including the steps of contacting the yarn prior to take-up of the yarn with a lubricant composition comprising:

(a) a co-polymer of

(i) a monomer selected from 1-butene, 2-butene and isobutene and mixtures thereof, and

(ii) a C_{5-20} alpha-olefin and mixtures of two or more thereof; and

(b) at least one lubricant selected from

(i) mineral oil, and

(ii) a fatty ester containing a total of at least 6 carbon atoms and mixtures of two or more thereof,

and taking up the yarn on take-up means therefor.

2. A process as claimed in claim 1 wherein there is used as said composition a composition wherein said component (b) satisfies one or more of the following conditions:

(i) component (b) comprises as a mineral oil white oil;

(ii) component (b) comprises as a mineral oil process grade mineral oil;

(iii) component (b) comprises a mixture of a mineral oil and a fatty ester;

(iv) component (b) comprises components (b)(i) and (b)(ii) in a weight ratio of from 90:10 to 50:50; and

(v) component (b) is present in said composition at from 50 to 95% by weight.

3. A process as claimed in either of claims 1 and 2 wherein there is used as said composition

a composition wherein said component (a) satisfies one or more of the following conditions:

(α) component (a)(i) thereof comprises 1-butene;

(β) component (a)(ii) thereof comprises 1-decene;

(γ) component (a) is a copolymer of 1-butene and 1-decene;

(ζ) component (a) has a molecular weight of from about 500,000 to 10,000,00; and

(ε) component (a) is present in said composition at from 0.01 to 5% by weight.

4. A process as claimed in any one of claims 1 to 3 wherein there is used as said composition a composition wherein components (i) and (ii) of copolymer (a) are present therein in a mole ratio of from 90:10 to 10:90.

5. A process as claimed in any one of claims 1 to 4 wherein said composition further comprises one or more of the following: water; a polysiloxane; and an emulsifier.

6. A process for the preparation of a coning oil lubricant which process comprises admixing

(a) a copolymer of

(i) a monomer selected from 1-butene, 2-butene and isobutene and mixtures thereof, and

(ii) a C_{5-20} alpha-olefin and mixtures of two or more thereof; and

(b) at least one lubricant selected from

(i) mineral oil, and

(ii) a fatty ester containing a total of at least 6 carbon atoms and mixtures of two or more thereof.

7. A process as claimed in claim 6 wherein components (a) and (b) and the concentrations thereof are selected to satisfy one or more of conditions (i) to (v) and (α) to (ε) defined in claims 2 and 3 above.

8. A process as claimed in either of claims 6 and 7 wherein components (i) and (ii) of copolymer (a) are present therein in a mole ratio of from 90:10 to 10:10.

9. A process as claimed in either of claims 6 and 7 wherein there is further admixed with components (a) and (b) one or more of the following: water; a polysiloxane; and an emulsifier.