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LUBRICATING OIL

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5 Claims. (Cl. 87—9)

This invention relates to the manufacture of lubricating oil characterized by a low pour test, and to novel pour point depressants and to methods of producing the same.

The invention contemplates the preparation of mineral lubricating oil having a reduced pour test and other desired qualities from lubricating oil stock, such as those derived from mixed base and paraffin base crudes and the like, by the addition to the lubricating oil of a pour point depressant material or wax crystal inhibitor. The invention particularly contemplates the addition to a lubricating oil of glycerol tri-esters of higher saturated fatty acids, such as glycerol tri-stearate.

Lubricating oil stocks derived from paraffin and mixed base crudes contain appreciable amounts of wax and, therefore, have a relatively high pour test unless a substantial amount of this wax is removed. The removal of this wax is usually accomplished by cold settling, filtration or centrifuging. However, lubricating oil stock, after dewaxing by these processes, may still retain some wax, and may have a pour test above 15° F. and up to about 40° F. The removal of further quantities of wax from these stocks in order to further reduce the pour test necessitates expensive further processing.

On the other hand, the presence of a certain amount of wax in a lubricating oil may be beneficial from the standpoint of providing a comparatively flat temperature-viscosity curve. That is, there is a relatively small change in the viscosity of the oil between temperatures of say 100° F. and 210° F. Consequently, it is of advantage to add to the oil a material which will have the effect of reducing the pour test or cold test to the desired extent without the necessity of entirely removing the remaining wax.

I have discovered that a glycerol tri-ester of a higher saturated fatty acid, such as glycerol tri-stearate or glycerol tri-palmitate, constitutes a highly effective pour depressant material for purposes of the present invention. The ester may be prepared from chemically pure stearic acid and glycerine; or commercial stearic acid, or other relatively impure stearic acid such as that recovered from soap plants, grease plants and the like, may be used. The esterification may be carried out in conventional manner, such as that suggested by Bellucci (J. Chem. Soc. 1911, A. i. 416). For example, the theoretical amounts of glycerine and fatty acid to form the tri-ester are mixed, and the esterification carried out under high vacuum while gradually heating the reactants to a temperature of about 260° C. and maintaining them at this temperature for about 30 minutes. Following the esterification, any free fatty acid may be removed by dissolving the reaction product in ether, washing out with dilute caustic soda solution followed by distilled water, and then evaporating the ether to obtain the purified synthetic ester. A purified material consisting essentially of a glycol tri-ester having the properties of the above described synthetic esters may be separated from natural fats or fatty oils, such as oleo-stearin, by a separation and purification treatment, as by repeated pressing at high temperatures, or hydrogenation to reduce the olein content, followed by removal of free fatty acid in the manner outlined above.

The proportion in which the glycerol ester is added to a lubricating oil is critical in obtaining the desired lowering of pour point with a commercially practical quantity of the ester. Thus, it is found that increase in the proportion of glycerol ester up to a certain amount for the particular ester, and for a particular wax bearing oil, gives increased lowering of pour point; but increase beyond this amount gives no further beneficial effect, and in fact, may result in raising of the pour point from the low value obtained with the optimum proportions. Ordinarily, a proportion of the order of less than 1½ g. of the glycerol ester per 100 cc. of oil is employed; and as little as ¼ g. or less per 100 cc. of oil effects pronounced lowering of the pour point. Optimum results with glycerol tri-stearate are secured by the use of approximately ¾% to 1% by weight of the depressant.

By way of example, the following results were obtained by the addition of the specified amounts of the listed depressants to a partially dewaxed paraffin base lubricating oil having a Saybolt viscosity at 100° F. of 300, and a Saybolt viscosity at 210° F. of 50, with a normal pour point of +20° F.

Pour point depressant	Pour point in °F. of sample containing designated weight in grams of depressant per 100 cc. of oil				Maximum lowering of pour point, °F
	¼	½	¾	1	
Glycerol ester made from (commercial) stearic acid M. P. 52.2° C	−5	−10	−15	−10	35
Glycerol ester made from grease plant stearic acid M. P. 53.3° C	+5	0	------	−5	25
Glycerol tri-stearate C. P.	------	−5	------	------	25

I am aware that mineral lubricating oils have been blended with natural fats or fatty oils, usually in higher proportions, to produce compounded oils for various uses. My invention, on the other hand, is connected with a function not previously recognized or attained by the above practice. This is the reduction in pour point, and is distinguished by the use of a particular character of partially dewaxed paraffin base lubricating oil normally having a pour point in

excess of 15° F., and by the addition thereto of a very small proportion of a particular synthetic glycerol tri-ester of a higher saturated fatty acid, or a purified material consisting essentially of such an ester and having the properties of such a synthetic ester. I have found that natural fats or fatty oils do not possess the pour reducing qualities of the synthetic or purified esters mentioned above, and are commercially ineffective or undesirable for this purpose, particularly in connection with motor oils adapted for crank case lubrication of internal combustion engines.

I am also aware that it has been proposed to add in small proportion to mineral lubricating oils, such as motor oils, an ester selected from a large variety of synthetic organic esters, for the purpose of adding oiliness and increasing the bearing load capacity of the lubricants by reducing friction. My invention is distinguished from this suggestion by relating to the different function of reduction in pour point, which was not recognized or attained in the former suggestion, due to the use of different mineral lubricating oils of the naphthene base type or fully dewaxed type having initially low pour points, due to the use of a proportion range which lies mainly outside of the effective range of the present invention, and due to the use of a large variety of esters mainly of different classification, and of which substantially all are commercially ineffective for purposes of the present invention.

The depressants of the present invention are particularly valuable because they are colorless materials which have substantially no effect on the color of the oil, and therefore permit their use in connection with pale oils without lowering of color. The depressants are also free from metallic bases or soaps, which are objectionable in lubricating oils adapted for certain purposes such as motor oils. The small amount of the glycerol ester required to effect a lowering of pour point of as much as 25° to 35° F. has little effect upon other desirable properties of the lubricating oil, as is evident from the following results obtained by tests on a paraffin base lubricating oil before and after addition of 0.5 g. of glycerol ester, prepared from the commercial grade of stearic acid as outlined above, to 100 cc. of the oil.

	Paraffin base lubricating oil	Same oil containing 0.5 g. glycerol ester per 100 cc. of oil
Gravity	25.1 A. P. I.	25.1 A. P. I.
Flash	400–405° F.	400–405° F.
Fire	465–460° F.	460–460° F.
Visc. at 210° F	50–51	50–50
Color lovibond	150–6"	150–6 "
Pour, °F	+20° F.	—5° F.
Carbon residue	.09–0.10	0.11–0.09
Saponification No	2.2–1.8	3.1–2.7
Iodine No	21–21	20–20
Sligh oxidation No	14.5	14.1
Unsaturation	0.04%	0.04%
Demulsification at 130	13.9	13.1
Oiliness	0.075	0.075

Actual service tests of motor oils prepared in accordance with the present invention, in which the oils are used in the crank case of vehicle engines for the usual running period of 1,000 miles or more, indicate that oils containing the esters of the present invention in the small proportions disclosed herein, are free from objectionable bearing metal corrosion, sludge formation, or hydrolysis with resultant formation of free fatty acids.

Where the expression "paraffin base lubricating oil" is used in the accompanying description and claims, it is to be understood that this refers to an oil of the mixed base or Mid-Continent type as well as an oil of the paraffin base, or Pennsylvania type, unless the contrary appears from the text.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof, and therefore only such limitations should be imposed as are indicated in the appended claims.

I claim:

1. A lubricating oil having a pour point of approximately 5° F. or below, comprising a mixture of a partially dewaxed paraffin base lubricating oil normally having a pour point above 15° F., with a pour point depressant consisting essentially of a glycerol tri-ester of a higher saturated fatty acid in the proportion of from 0.25 g. to 1.50 g. per 100 cc. of oil, the depressant being substantially devoid of free fatty acid.

2. A lubricating oil having a pour point of approximately 5° F. or below, comprising a mixture of a partially dewaxed paraffin base lubricating oil normally having a pour point above 15° F., with a pour point depressant consisting essentially of glycerol tri-stearate in the proportion of from 0.25 g. to 1.00 g. per 100 cc. of oil, the depressant being substantially devoid of free fatty acid.

3. The method of preparing a lubricating oil of low pour point of approximately 5° F. or below, which comprises reacting glycerine and commercial stearic acid to form essentially glycerol esters of the higher fatty acids present therein, removing free fatty acid from the glycerol esters, and adding the purified glycerol esters in a proportion of from 0.25 g. to 1.50 g. to 100 cc. of a partially dewaxed paraffin base mineral lubricating oil normally having a pour point of above 15° F.

4. A lubricating oil having a pour point of approximately 0° F. or below, comprising a mixture of a partially dewaxed paraffin base lubricating oil normally having a pour point above 15° F., with a pour point depressant consisting essentially of glycerol esters prepared from commercial stearic acid in the proportion of from 0.25 g. to 1.00 g. per 100 cc. of oil, the depressant being substantially devoid of free fatty acid.

5. A lubricating oil having a lowered pour point comprising a mixture of a partially dewaxed paraffin base lubricating oil normally having a higher pour point with small proportion of the order of ¼ to 1½% of the glycerol ester prepared by reacting glycerin with commercial stearic acid, the ester being substantially devoid of free fatty acid.

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