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PREPARATION OF OLEFIN LUBRICANT
HAVING A HIGH VISCOSITY

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The present invention is directed to a lubricating oil composition. More particularly, the invention is directed to a method of preparing a lubricating oil composition having a high viscosity and a low neutralization value.

In accordance with the present invention a high viscosity lubricating oil composition having a low neutralization value is prepared by forming a mixture of an olefinic lubricating oil polymer having lubricating oil characteristics and an amount in the range between 0.1% and 5% by weight of diphenylthiourea. The mixture is then subjected to oxidizing conditions at an elevated temperature for a time sufficient to form an oxidized product. The oxidized product is appreciated in viscosity by formation of oxidized bodies but the oxidized product has a relatively low neutralization value indicating that the oxidized bodies are not acidic in nature.

The diphenylthiourea employed in the practice of the present invention may be used, as stated, in an amount in the range between 0.1% and 5% by weight with a preferred range from 0.5% to 3% by weight. An amount which gives very satisfactory results is 1% by weight.

The olefinic lubricating oil polymer employed in the practice of the present invention may be obtained by polymerizing alpha olefins to produce polymers in the lubricating oil boiling range and having lubricating oil characteristics. Such polymers may be produced by polymerizing alpha olefins boiling in the range between 100°

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and 400° F. in the presence of a Friedel-Crafts catalyst at a temperature of about 75° to 200° F. Olefins which may be polymerized to produce lubricating oil polymers are the alpha olefins having from 5 to 10 carbon atoms in the molecule including pentene-1, hexene-1, heptene-1, octene-1, nonene-1, and decene-1. A convenient source of alpha olefins is the naphtha fraction produced in the Fischer-Tropsch synthesis when employing a promoted iron catalyst. The olefinic lubricating oil polymer will ordinarily be employed in an amount in the range between 95% and 99.9% by weight with a preferred range being from 97% to 99.5% by weight.

In oxidizing a mixture of olefinic lubricating oil polymer having lubricating oil characteristics and diphenylthiourea, the mixture may be oxidized at a temperature in the range from 300° to 600° F. The oxidation time may range from about 30 minutes up to about 16 hours. Air is a suitable source of oxygen but other sources may be used.

In order to illustrate the invention further, a number of compositions were made up in which various additives were admixed with an olefinic lubricating oil polymer. The admixtures were then oxidized at 375° F. and the oxidized mixture then tested for per cent increase in viscosity at 210° F., for neutralization value and for sludge formation. The results of these tests are presented in the following table:

Table

Oxidation Promoter	Tests on Oxidized Oils			
	Wt. Percent Promoter	Olefin Polymer ¹		
		Percent Increase Vis., 210° F.	Neut. Value	Sludge Formation
None.....		247	11.6	None.
Beta-Naphthol.....	1.0	440	16.2	Do.
Diethanol Sulfide.....	2.0	540	8.4	Moderate.
Hydroquinone.....	1.0	550	14.0	None.
Hydroquinone.....	2.0	6	8.4	Do.
Dibenzyl p-Amino Phenol.....	2.0	580	11.8	Heavy.
Sulfur Monochloride.....	1.0	680	7.7	None.
Diphenylthiourea.....	1.0	944	0.8	Moderate.
Lauryl Mercaptan.....	1.0	1,070	6.2	None.
Do.....	2.0	1,600	2.8	Do.
Commercial Metal Deactivator.....	1.0	1,560	20.2	Do.
p-Toluene Sulfonyl Amide.....	2.0	² Tar	1.5	Very Heavy.
Benzene Sulfonyl Amide.....	2.0	² Tar	0.9	Do.

¹ Prepared by AlCl₃ catalyzed polymerization of octene-1, 47.7 vis., 210° F. SSU, 120 viscosity index.

² Extremely viscous material, too viscous for SSU viscosity determination.

It will be seen from the foregoing results that of 10 additives added to the olefinic polymer only diphenylthiourea increased the viscosity while yet maintaining a low neutralization value. High neutralization values are indicative of high acid contents which are undesirable in lubricating oil. These data show that the viscosity of an olefinic polymer may be varied by varying the amount of the diphenyl thiourea to produce nearly any desired viscosity while yet allowing the production of a lubricating oil having a low neutralization value and in which is not formed any substantial amount of sludge.

The oxidation of the naphthenic mineral oil in the method of the present invention was carried out as follows in a 200 cc. glass three-necked round bottom flask. The central neck of this flask is 5 cm. long and has an inside diameter of 3 cm. The flask has two opposed side necks which are 6 cm. long and 1.5 cm. inside diameter, and are situated at a 30° angle with the central neck, perpendicular to the surface of the spherical flask. The stirrer is a glass rod 0.5 cm. in diameter, entering the flask through a glass tube bearing held in place by a rubber stopper in the central neck, with two glass blades 1.0 cm. long by 0.7 cm. wide welded to opposite sides of the bottom end of the glass rod at a 30° angle with the vertical. The stirrer is driven by a 1750 R. P. M. motor connected to the stirrer by a rubber tubing coupling. The blades of the stirrer are situated about 0.5 cm. from the bottom of the flask and spin in such a direction that their propeller-like action circulates the oil to the bottom of the flask, up along the sides of the flask, back to the center of the flask, and down to the stirrer blades again. A considerable amount of air is sucked down with the descending oil and intimately contacted with it by the action of the stirrer. A thermometer well 5 cm. long and 1.0 cm. inside diameter is situated in one side of the flask equidistant from the two side arms. During the oxidation the flask is about two-thirds immersed in an oil bath maintained at 375° F. The temperature of the oil in the flask is usually 5° to 10° F. below the temperature of the oil bath.

The oxidation operation is carried out in the specially constructed flask by stirring 100 cc. of the sample in the presence of air for sixteen hours at 375° F. At the end of that time the flask is removed from the oil bath and tests obtained on the oxidized oil.

While the oxidation operation was carried out for 16 hours at 375° F., the operation to appreciate the viscosity may be conducted at a lower temperature for a longer period of time or at a higher temperature for a shorter period of time. For example, the oxidation may be carried out at a temperature in the range from 300° to 600° F. and for a time ranging from about 30 minutes up to 16 hours.

The neutralization value which is used as a method of test is described in A. S. T. M. Standards for Petroleum Products and Lubricants prepared by A. S. T. M. Committee D-2, November, 1948 and designated "Tentative Method of Test for Neutralization Value (Acid and Base Numbers) by Color Indicator Titration," A. S. T. M. designation: 974-48T.

While the neutralization value of the oxidized product in accordance with the present invention is low it may be desirable to decrease the neutralization value even further. This may be done by contacting the oxidized product with an adsorbent material, such as a contact clay. Another way of lowering the neutralization value is to neutralize the organic acidic material with an alkaline material, such as a solution of sodium hydroxide or sodium carbonate.

The oxidized product of the present invention may suitably be used as a compounding oil and may find particular usage as a component of steam cylinder lubricant or as the hydrocarbon component in grease manufacture.

The nature and objects of the present invention having been completely described and illustrated, what I wish to claim as new and useful and to secure by Letters Patent is:

1. A method for increasing the viscosity of an ing oil characteristics which comprises oxidizing olefinic lubricating oil polymer having lubricated the polymer at a temperature in the range from 300° to 600° F. in the presence of a promotional amount no less than 0.1% by weight of diphenylthiourea to form an oxidized product having an increased viscosity and a low neutralization value and recovering said product.

2. A method for forming a high viscosity lubricating oil which comprises forming a mixture of a lubricating oil polymer of an alpha olefin having from 5 to 10 carbon atoms in the molecule and a promotional amount no less than 0.1% by weight of diphenylthiourea, oxidizing the mixture at a temperature in the range from 300° to 600° F. to form an oxidized product having an increased viscosity and a low neutralization value and recovering said product.

3. A method for forming a high viscosity lubricating oil which comprises forming a mixture of a lubricating oil polymer of octene-1 having lubricating oil characteristics and 1% by weight of diphenylthiourea, oxidizing the mixture at a temperature of about 375° F. for a time sufficient to form an oxidized product having an increased viscosity and a low neutralization value and recovering said product.

4. A method for producing a high viscosity lubricating oil which comprises forming a mixture of a lubricating oil polymer of an alpha olefin having from 5 to 10 carbon atoms in the molecule and a promotional amount in the range from 0.1% to 5% by weight of diphenylthiourea, oxidizing the mixture in the presence of air at a temperature in the range from 300° to 600° F. for a time in the range from 30 minutes to 16 hours to form an oxidized product having an increased viscosity and a low neutralization value, and recovering said product.

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