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PROCESS OF IMPROVING LUBRICATING OILS

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6 Claims. (Cl. 208—28)

This invention relates to a process for the improvement of lubricating oils and, more particularly, to the treatment of lubricating oils with ethylene under pressure and elevated temperatures in the presence of free-radical initiators.

It is known that ethylene can be polymerized into high-grade lubricating oils by using aluminum chloride or organoaluminum compounds. It is further known that these ethylene lubricating oil polymers are miscible with mineral lubricating oils in any ratio and that the properties of these mixtures can be improved by an aftertreatment with aluminum chloride.

It has now been found that lubricating oils may be improved by treating mineral lubricating oils from any source with ethylene under pressure and at elevated temperature in the presence of free-radical initiators. The reaction of the invention is advantageously carried out at pressures from 50 to 300 atmospheres gage and at temperatures from 100°–300° C., preferably between 160° C. and 250° C.

Compounds which may be considered as free-radical initiators are peroxides, such as benzoyl peroxide, lauroyl peroxide, acetyl peroxide, diethyl peroxide, di-tert-butyl peroxide, cyclohexanone peroxide, acetone peroxide, methyl ethyl ketone peroxide, hydrogen peroxide, barium peroxide, ozone, persulfate, perborate, percarbonate, and molecular oxygen in concentrations below 0.01%; hydrazine, hydrazine salts, bibenzalazine, trialkylamino oxides, azo compounds, such as azodinitriles, metal alkyls, such as tetraethyllead; redox systems of peroxides and reducing agents, such as salts of acids of sulfur in the lower oxidation state, if necessary in the presence of amines or alcohols, or salts of metals of lower valences. The catalytic amounts are about 0.001 to 5% by weight, preferably 0.05 to 1%, of the oil to be improved.

Distillates and refined bright stock from any source may be considered as mineral lubricating oils. The treatment with ethylene can be carried out either before or after the dewaxing.

Since the treatment with ethylene may also lead to the formation of small amounts of high molecular polymers which are insoluble in the lubricating oil and which render the latter turbid, it may be advantageous to carry out the treatment with ethylene before the dewaxing so that these high molecular, oil-insoluble polymers can be removed simultaneously with the paraffin in the subsequent dewaxing.

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The properties of the lubricating oils obtained may be improved by subsequently treating with anhydrous aluminum chloride. The aftertreatment consists of agitation with anhydrous aluminum chloride (1 to 5% and preferably 2 to 3%) at elevated temperatures from 80° to 150° C., preferably from 110° to 130° C. The treatment should last from 1 to 2 hours. After the reaction is complete, the resulting aluminum chloride sludge is removed by decantation, after cooling to room temperature, and the oil which has been freed of the sludge is neutralized by agitation with quick lime and fuller's earth at elevated temperature. The aftertreatment with aluminum chloride can be carried out also by adding pure ethylene polymers, such as are obtained according to U.S. Patent No. 2,165,372 and German Patents No. 767,929 or No. 767,128.

Example 1

(*a*) A spindle oil distillate having a specific weight of 0.898 and a viscosity of 15.78 centistokes=2.4 E° at 50° C. was treated with ethylene under a pressure of 150 atmospheres gage and at 220° C. with the addition of 1.7% by weight of di-tert-butyl peroxide. 26.2% by weight of ethylene was taken up by the spindle oil. The lubricating oil obtained was then agitated for half an hour at 150° C. with 2% quick lime and 2% fuller's earth and then filtered.

(*b*) The lubricating oil obtained in accordance with (*a*) was brought into contact with 3% by weight of aluminum chloride (anhydrous) and agitated for 2 hours at 110° C. The oil was then cooled to room temperature, allowed to stand for 8 hours, and decanted from the aluminum chloride sludge which had formed and settled.

The lubricating oil obtained was then agitated for half an hour with 2% quick lime and 2% fuller's earth at 150° C. and then filtered. The lubricating oils obtained had the following properties:

	Sp. Gr. at 20° C.	Viscosity in Centistokes at—				Viscosity Index [1]	m-value [2]
		20° C.	37.8° C.	50° C.	99° C.		
Original oil	0.898	63.25	25.60	15.78	4.29	68	3.39
Treated according to 1–*a*	0.880	86.35	34.46	20.86	5.56	107	3.70
Treated according to 1–*b*	0.878	132.7	47.60	28.80	7.00	113	3.63

[1] Cf. Zerbe "Mineralöle" (Springer), p. 32.
[2] Cf. Zerbe "Mineralöle" (Springer), p. 29.

Example 2

A mineral bright stock having a specific gravity of 0.908 and a viscosity of 53 E_{50}° was treated with ethylene at 300 atmospheres gage and 220° C. with the addition of 0.75% di-tert-butyl peroxide. The reaction product obtained was then agitated for 30 minutes with 2% quick lime and 2% fuller's earth at 150° C. and then filtered. The greasy reaction product was diluted with a low viscosity spindle oil in a ratio of 30:70 and freed of oil-insoluble, high polymers by filtration. The same mix-

ture was prepared with the original bright stock (also 30:70). The two oils had the following properties:

	Sp. Gr. at 20° C.	Viscosity in Centistokes at—				Viscosity Index	m-value
		20° C.	37.8° C.	50° C.	99° C.		
Untreated bright stock and spindle oil 30:70	0.892	96.5	36.58	22.18	5.90	114	3.68
Treated bright stock and spindle oil 30:70	0.888	100.3	39.0	24.12	6.40	123	3.55

Example 3

A mineral spindle oil as in Example 1–*a* was treated at 220° C. under an ethylene pressure of 100 atmospheres gage with the addition of 0.75% di-tert-butyl peroxide. The reaction product was mixed 50:50 with a pure ethylene polymer obtained according to the above-mentioned patents, then agitated as in Example 1–*b* with 3% by weight of aluminum chloride (anhydrous) for 1 hour at 120° C., and cooled to room temperature. After the aluminum chloride sludge was allowed to settle, the oil was decanted and treated with fuller's earth. The properties of these oils were as follows:

	Sp. Gr. at 20° C.	Viscosity in Centistokes at—				Viscosity Index	m-value
		20° C.	37.8° C.	50° C.	99° C.		
(*a*) Original spindle oil	0.882	27.65	13.37	8.99	3.01	82	3.84
(*b*) Spindle oil treated with C_2H_4	0.881	65.53	26.00	16.02	4.82	120	3.71
(*c*) 50 parts of (*b*) +50 parts of the polyethylene	0.864	199.0	73.20	42.80	10.10	123	3.33
(*d*) Mixture (*c*) treated with aluminum chloride	0.860	266.1	93.54	53.95	12.09	122	3.27

What we claim and desire to protect by Letters Patent is:

1. A process for the preparation of an improved lubricant which comprises contacting a mineral oil lubricant with ethylene under superatmospheric pressure and elevated temperature in the presence of a free-radical polymerization initiator, whereby the viscosity index and lubricating properties of the lubricant are substantially improved.

2. A process for the preparation of an improved lubricant which comprises contacting a mineral lubricating oil with ethylene at a pressure of from about 50 atmospheres to about 300 atmospheres and a temperature of from about 100° C. to about 300° C. in the presence of free-radical polymerization initiator, whereby the viscosity index and lubricating properties of the mineral oil are substantially increased.

3. Process of claim 2 wherein the lubricating properties of the mineral oil are further improved by subsequent treatment of the reaction mixture by agitation with anhydrous aluminum chloride at a temperature of about 80–150° C.

4. Process of claim 3 wherein the lubricant is cooled following the aluminum chloride treatment, the aluminum chloride sludge is removed by decantation, and the oil is then neutralized.

5. Process of claim 2 wherein the ethylene-treated oil is then dewaxed, whereby oil-insoluble polymers formed in the ethylene treatment are removed simultaneously with the paraffin.

6. A process for the preparation of an improved lubricant which comprises contacting a mineral oil lubricant with ethylene under superatmospheric pressure and elevated temperature in the presence of a free-radical polymerization initiator, whereby the viscosity index and lubricating properties of the lubricant are substantially increased, and subsequently treating the reaction mixture with anhydrous aluminum chloride at an elevated temperature.

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UNITED STATES PATENT OFFICE

CERTIFICATION OF CORRECTION

Patent No. 2,939,831 June 7, 1960

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It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

In the heading to the printed specification, line 9, for "Claims priority, application Germany Oct. 17, 1955" read -- Claims priority, application Germany Oct. 5, 1955 --.

Signed and sealed this 25th day of April 1961.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents