United States Patent Office

2,703,783

Patented Mar. 8, 1955

2,703,783

PROCESS FOR THE CLARIFICATION OF LUBRICATING OIL ADDITIVE CONCENTRATES

Alexander H. Popkin, New York, N. Y., assignor to Esso Research and Engineering Company, a corporation of Delaware

No Drawing. Application April 28, 1950, Serial No. 158,931

5 Claims. (Cl. 252—29)

This invention relates to lubricating oil additives and to the process of their preparation. Particularly, the invention relates to a process for the preparation of concentrates of lubricating oil additives. More particularly, the invention relates to a process for the clarification of oil concentrates of high molecular weight polymers and copolymers useful as viscosity index improvers, pour point depressants and the like.

With the increase in demand for specialized industrial lubricants has come an increase in the over-all quality of lubricating compositions designed to meet those demands. The art of lubricant manufacture is constantly searching for new methods, new processes, etc. to further raise the quality of their products.

Of particular rapid increase has been the over-all quality standards of lubricating oils such as gear oils, turbine oils, automotive engine oils, hydraulic transmission fluids and the like, those oils designed to meet the stringent and exacting requirements of high torque, high speed lubrication. In this particular phase of the lubricant art the development of new methods of refining and processing of petroleum fractions has been aided by the development of lubricating oil additives. These lubricating oil additive materials, when combined with the desired lubricating oil bases, further enhance the desirable characteristics of the lubricant composition. It is toward this particular segment of the art of lubricant manufacture, that is, the field of lubricant additives, that this invention is directed.

Among the long list of characteristics of lubricating compositions that are improved by addition agents may be mentioned the rate of change of the lubricant viscosity with temperature, the temperature at which the lubricant loses its property of free flow, the lubricant's resistance to oxidation, its resistance to shear breakdown, its ability to remain between moving metal parts at high temperatures and pressures, its lack of metal corroding properties, and many others. These improvements are brought about by the addition to the lubricant base, either natural occurring oil stocks or synthetic oils, of small proportions of additive materials which have been especially developed to improve one or more of these desired characteristics.

The additive materials or agents with which this invention is particularly concerned are those additives which are added to the lubricant base in the form of an oil concentrate containing as little as 5% or as much as 60% of the additive material. Thus, viscosity index improvers, pour point improvers, and the like are of primary concern in the instant invention.

Among the lubricant additive materials that have been developed to improve the viscosity index and pour points of lubricating oils may be mentioned polymers of unsaturated hydrocarbons, such as isobutylene, butadiene, vinyl aromatics, and the like, polymers of acrylate and methacrylate esters containing from 6 to 30 carbon atoms in the ester contributed portion of the molecule, copolymers of unsaturated hydrocarbons such as isobutylene and isoprene or butadiene, styrene and isobutylene, styrene and butadiene, naphthalene and chlorinated hydrocarbons, and the like. These additive materials are ordinarily prepared as oil concentrates and as such are added to the lubricant base.

In the preparation of the oil concentrates of the above-mentioned additive materials, considerable difficulty has been experienced in some instances in that the additive concentrates have been found to be cloudy or semi-turbid in appearance. This cloudiness has been found to be the

result of small extremely finely divided particles which may not be removed by any of the known processes of filtration ordinarily used in the art.

Copolymers of isobutylene and isoprene or butadiene, prepared in accordance with U. S. Patent 2,356,128 and known to the art as Butyl rubber, and polymers of isobutylene, prepared in accordance with known procedures and having a molecular weight of between about 35,000 to 50,000, are ordinarily increased in resistance to shear breakdown by a process of milling or extruding the prepared materials from a higher to a lower molecular weight. These processes are described in detail in U. S. Patents 2,239,501 and 2,466,300 for polyisobutylene and Butyl rubber, respectively. In the case of the milled polybutene the materials which cause the cloudiness of the oil concentrates have been found to be extremely small discrete particles of carbon black, grease, metal filings and the like, which have contaminated the polymer in the milling process. These contaminants have become dispersed in the milled polybutene and are so small and so finely divided that they are not removable by any known method of filtration, with or without the use of filter aids.

The cloudiness of the oil concentrates of Butyl rubber which has been reduced from a higher to a lower molecular weight by the process of extrusion described in U. S. Patent 2,466,301 has been found to be due to a finely divided surface coating agent which is added to the Butyl rubber prior to extrusion to prevent agglomeration of small particles and aid in ease of handling in subsequent processing steps. This surface coating agent may be selected from a large number of known agents among which are the metallic stearates, zinc stearate being the most common and most widely used. When the extruded copolymer is admixed with the desired oil to form the oil concentrates this intimately dispersed zinc stearate, occurring in percentages of about 0.1% to 2.0% by weight, causes the oil concentrate to become cloudy or semi-turbid. As was stated above, this finely divided agent may not be removed from the oil concentrate by any known method of filtration.

In the case of copolymers of styrene and isobutylene, which are useful as viscosity index or pour point improvers, it is desired that copolymers having a styrene content of 25% or less be used. In the manufacture of this copolymer there is a certain amount of a higher styrene content formed, for example, copolymers containing from about 30% to 50% styrene. The cloudiness of the oil concentrates of these copolymers is due to the presence of this high styrene content copolymer.

Polymers and copolymers of acrylate and methacrylate esters made with methyl methacrylate as one component normally contain small percentages of a polymer of methyl methacrylate. This methyl methacrylate polymer is believed to be the cloud former in the oil concentrates of the acrylate or methacrylate copolymer addition agents.

It has now been found, and forms the subject of this invention, that these cloud forming contaminants may be removed from oil concentrations of these addition agents by an additional processing step prior to the ordinary filtration step.

In general, this added step is accomplished in the following manner.

The oil concentrate of the addition agent containing from 5% to 80% of the additive material is heated to a temperature within a range of from about 210° F. to 350° F. While at this temperature a gaseous aquophylic substance is passed through the oil concentrate for a period of time ranging from about 5 minutes to one hour. Although the exact mechanism of this processing step is not known, it is believed that the action of the aquophylic gaseous substance is such that coagulation or agglomeration of the small particles of contaminants occurs, the agglomerated particles being of sufficient size as to be removable by filtration.

As the gaseous aquophylic substance, it is preferred to use steam. However, other substances such as hydrogen chloride gas, ammonia gas, carbon dioxide, or mixtures of the above in any proportion may be used.

After the oil concentrate has been subjected to the action of the aquophylic gas for such time as is necessary to effect the desired clarification, the treated oil blend

which, at this stage is cloudy, both due to the agglomerated, insoluble contaminants and to the presence of condensed moisture from the steam, may be dried by (1) blowing an inert gas through the heated oil blend, or (2) by adding a suitable drying agent-filter aid, such as those diatomaceous earths sold under trades names of HyFlo, Celite, Filter-cel, etc., familiar in the art. The resulting oil blend may then be filtered with the aid of the filter aid present through a suitable filtering medium. In some instances it may be necessary to subject the filtered oil blend to a second treatment with the aquophylic gas although, in general, this can be avoided by sufficient treatment in the first instance.

The invention will be more clearly explained by a reference to the following examples wherein steam is used as the aquophylic gas.

EXAMPLE I

500 gr. of a 20 weight percent concentrate in a phenol treated Mid-Continent distillate having a viscosity index of 108 and a viscosity at 210° F. of 36 S. U. S. of a copolymer of isobutylene and isoprene, prepared in accordance with U. S. Patent 2,356,128 and reduced in molecular weight in accordance with U. S. Patent 2,466,300, was divided into two portions. One portion was filtered through HyFlo and the other portion was raised to a temperature of about 250° F. and blown with steam for 15 minutes followed by filtration through a filter aid known commercially as HyFlo. Heliger turbidimeter and visual observation readings were taken on the two samples and are reported in Table I below.

Table I

Sample	Treatment	Visual Observation	Heliger Turbidimeter Reading
20% concentrate of copolymer.	clay filtration	very cloudy	100
Do	15 min. steaming followed by clay filtration.	clear	30

It is to be seen that the steaming treatment followed by filtration removes substantially all of the turbidity from the oil concentrate, the turbidimeter reading decreasing 70 units.

EXAMPLE II

About 300 gr. of a polymer of isobutylene having an average molecular weight of about 32,000 Staudinger, obtained by milling a copolymer of isobutylene having a molecular weight of about 70,000 Staudinger according to the process of U. S. Patent 2,239,501 was blended to a 20% concentration in a phenol extracted Mid-Continent distillate having a viscosity index of 108 and a viscosity at 210° F. of 36 S. U. S. This concentrate was divided into two portions. One portion was filtered through a filter aid, known commercially as HyFlo, and the other was heated to a temperature of 250° F., submitted to the action of steam for about 15 minutes and then filtered through activated clay. Turbidimeter readings are reported in Table II below.

Table II

Sample	Treatment	Visual Observation	Heliger Turbidimeter Reading
20% concentrate of polymer.	clay filtration	slightly turbid	22
Do	15 min. steaming followed by clay filtration.	very clear	6

The utility of the process of this invention is remarkedly apparent in this example since the slightly hazy concentrate after the filtration step showed the turbidimeter reading of 22 and this haze was almost completely removed by the process of this invention, the turbidimeter reading being reduced to 6.

To summarize briefly, this invention relates to a process for the clarification of oil concentrates of lubricating oil additives which are ordinarily turbid or cloudy which comprises the steps of subjecting the oil concentrate to the action of an aquophylic gas, preferably steam, at high temperatures followed by filtration through a filter aid. Although steam is the preferred gaseous treating medium, other aquophylic gases such as carbon dioxide, ammonia, hydrogen chloride gas, or mixtures of the above with steam are equally operable. The invention has particular advantage for removal of zinc stearate from oil concentrates of copolymers of isobutylene and isoprene, commonly known as Butyl rubber, and for removal of small proportions of impurities from oil concentrates of polyisobutylene which have been reduced from high molecular weight to a lower molecular weight by a milling or extruding process.

What is claimed is:

1. A process for the clarification of oil concentrates of a polymeric lubricating oil additive material selected from the class of polymers of iso-butylene and copolymers of isoprene, said oil concentrates being clouded due to the presence of a material of the class consisting of zinc stearate and carbon black, which comprises heating the oil concentrate to a temperature within a range of from about 210 to about 350° F., subjecting the heated concentrate to the action of steam for from about 5 minutes to about 1 hour and filtering said heated steamed concentrate to obtain a clear, unclouded solution.

2. A process according to claim 1 wherein said polymeric material is a copolymer of iso-butylene and isoprene and wherein said material of the class is zinc stearate.

3. A process according to claim 1 wherein said polymeric material is a polymer of iso-butylene and wherein said material of the class is zinc stearate.

4. A process according to claim 1 wherein said polymeric material is a copolymer of iso-butylene and isoprene and wherein said material of the class is carbon black.

5. A process according to claim 1 wherein said polymeric material is a polymer of iso-butylene and wherein said material of the class is carbon black.

References Cited in the file of this patent

UNITED STATES PATENTS

1,564,501	Weir	Dec. 8, 1925
1,603,174	Weir	Oct. 12, 1926
1,769,766	Bryant	July 1, 1930
1,856,934	Stafford	May 30, 1932
2,090,741	Zublin	Aug. 24, 1937
2,234,111	Doyle	Mar. 4, 1941

OTHER REFERENCES

"Reprinted from The Science of Petroleum," published by the Oxford University Press, around 1938; pages 1680, 1681, and 1685 pertinent.

"Chemical Refining of Petroleum," by Kalichensky et al.; Reinhold Pub. Co., 1942, page 244 pertinent.