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MANUFACTURE OF LITHIUM BASE GREASES

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13 Claims. (Cl. 252—41)

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This invention relates to the manufacture of lubricants containing lithium soaps such as liquid lubricants and greases.

In the usual processes for the manufacture of lithium soap greases, i. e., greases consisting of lithium soaps such as lithium stearate and a mineral oil, the temperature employed reaches 200° C., a temperature which introduces difficulties in the case of greases which have to function under low temperature conditions in which the mineral oil base is necessarily of low viscosity and relatively high volatility. However, a high temperature process is necessary when the lithium is added to the oil in the form of a lithium soap or when it is mixed in as an aqueous solution of lithium hydroxide which is combined with a fatty acid in the presence of the oil.

It is an object of the present invention to avoid the difficulties arising from the use of temperatures of 200° C. or thereabouts and with this object in view the present invention provides in a process for the manufacture of lubricants containing lithium soaps the improvement comprising intimately contacting a quantity of a free soap-forming acid containing at least sixteen carbon atoms in the molecule in solution in a lubricating oil with an approximately equivalent quantity of a saponifying agent of the group consisting of lithium oxide, anhydrous lithium hydroxide, lithium hydroxide and its hydrates at a temperature preferably between 35 and 45° C. for a time sufficient to react at least 90% by weight of one of said lithium compounds and said acid to form a soap.

The water of reaction is subsequently removed from the lubricant by heating, preferably to a temperature not exceeding 110° C. It is undesirable, however, to employ a higher temperature than is necessary as this increases losses due to vaporisation of the oil base and a temperature of 105 to 110° C. at atmospheric pressure has been found quite suitable. Naturally, lower temperatures can be employed if the operation is carried out under reduced pressure.

Preferably the removal of the water of reaction is effected by heating in thin layers for example on a rotary film drier such as a Kestner double drum film drier.

The lithium hydroxide or oxide should preferably be fine enough to pass a 100 mesh B. S. S. sieve.

It is preferred to use stearic acid as the soap forming acid but other fatty acids containing at least sixteen carbon atoms in the molecule may be employed for example palmitic acid, arachic

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acid, hydroxy-stearic acids, oleic acid, ricinoleic acid, mixed fatty acids such as may be obtained from various fats such as tallow, lard and various seed oils, wool fat acids, naphthenic acids such as may be derived from petroleum oils, rosin acids, tall oil acids, acids produced by the oxidation of paraffin wax or produced by the controlled oxidation of hydrocarbons and acids from montan wax and beeswax.

Mineral oils suitable for making the greases may be paraffinic or naphthenic preferably having a low content of aromatics and an aniline point of at least 80° C. with a difference in aniline point before and after sulphation not exceeding 15° C. Oils which do not have such aniline points yield greases which are soft and bleed badly or do not yield greases at all. Therefore best results are obtained with solvent-extracted oils, i. e., oils from which the bulk of aromatics has been removed for example by extraction with liquid SO_2, phenol, furfural, nitrobenzene, aniline, sulpholane, etc. In general, the oils fall within the range of lubricating oils.

If the grease is to be used for low temperature operation, it is of course essential that the oil have both a very low viscosity and a very low pour-point. In this event a relatively high viscosity index is also desirable, but not essential.

Examples of suitable oils are Edeleanised transformer oils after acid and earth treatments, finished transformer oils, a light distillate prepared from a naphthenic crude which has been Edeleanised and finally treated with activated earth and lime, having a viscosity of Redwood I at 70° F. of about 60 seconds, a technical white oil of viscosity about 70 seconds Redwood I at 100° F., and a light paraffinous distillate of the mineral Colza type having a viscosity of 45 seconds Redwood I at 70° F.

The finished lubricants should contain between 7 and 40% preferably 10% to 20% by weight of lithium soap for greases and between 0.25 and 7% by weight of lithium soap for liquid lubricants.

The lithium oxide and the soap-forming acid are brought together in approximately stoichiometric quantities, and it is preferable to have a slight exceess of the acid over the lithium oxide. The reaction may be carried out continuously or in batch.

The following example, in which the parts are by weight, illustrates how the process of the invention may be carried into effect:

A suspension of 1.25 parts of anhydrous lithium hydroxide passing a 100 mesh B. S. S. sieve in 5.0 parts of a mineral oil prepared by solvent extrac-

tion from a gas oil fraction having the following characteristics:

S. G. at 60/60° F.	0.899
Viscosity Redwood I at 70° F. ____secs__	61
Flash point Clsd. P. M ____°F__	275
Pour Point ____°F__	—70
Refractive index at 50° C	1.4845

A. S. T. M. distillation:

	°C.
I. B. P.	282.5
30% distils to	300
50% distils to	306
70% distils to	313
90% distils to	324
Maximum temperature	340

said mineral oil having the following characteristics:

S. G. at 60/60° F.	0.8625
Viscosity Redwood I at 70° F. ____secs__	61
Flash Point P. M.:	
(Closed) ____° F__	275
(Open) ____° F__	285
Pour Point	—75
Refractive index at 50° C	1.4600
Viscosity Redwood I at 100° F. ____secs__	44

Distillation

	°C.
I. B. P.	282.5
10% recovered at	293
20% recovered at	296.5
30% recovered at	300
40% recovered at	303
50% recovered at	306
70% recovered at	313
80% recovered at	317.5
90% recovered at	324
Maximum temperature	340

Recovery

	Per cent
Distillate	98.0
Residue	1.5
Loss	0.5

was added with stirring to a solution of 14.0 parts of stearic acid in 79.75 parts of the aforesaid mineral oil at a temperature of 40° C. If higher temperatures are employed at this stage a softer and less smooth product is obtained. Reaction between the lithium hydroxide and stearic acid proceeds rapidly under such conditions and appreciable thickening occurs within 1–2 minutes of mixing the components. The water of reaction is subsequently evaporated by heating the product to a temperature of 105–110° C. in thin layers on a rotary film drier.

What I claim is:

1. In a process of manufacturing a lubricant containing a lithium soap, the improvement consisting essentially of intimately contacting a quantity of a free soap-forming acid containing at least sixteen carbon atoms in the molecule in solution in a lubricating oil with an approximately equivalent quantity of a saponifying agent of the group consisting of lithium oxide, anhydrous lithium hydroxide, lithium hydroxide and its hydrates at a temperature of between about 35° and 45° C. for a time sufficient to react at least 90% by weight of one of said lithium compounds and said acid to form a soap.

2. The process of claim 1 wherein the soap-forming acid is stearic acid.

3. The process of claim 1 wherein the lubricating oil has an aniline point of at least 80° C. and a difference in aniline point before and after sulphation of not more than 15° C.

4. The process of claim 1 wherein the saponifying agent is lithium oxide.

5. The process of claim 1 wherein said saponifying agent is anhydrous lithium hydroxide.

6. In the process of manufacturing a lubricant containing a lithium soap, the improvement consisting essentially of intimately contacting a quantity of a free soap-forming acid containing at least sixteen carbon atoms in the molecule in solution in a lubricating oil with an approximately equivalent amount of a saponifying agent of the group consisting of lithium oxide, anhydrous lithium hydroxide, lithium hydroxide and its hydrates at a temperature between about 35° and 45° C. for a time sufficient to react at least 90% by weight of one of said lithium compounds and said soap-forming acid to form a soap, and thereafter removing water from the resulting mixture by heating it to a temperature not above about 110° C.

7. In the process of manufacturing a lithium soap grease, the improvement consisting essentially of intimately contacting a quantity of a free soap-forming acid containing at least sixteen carbon atoms in the molecule in solution in a lubricating oil with an approximately equivalent quantity of a saponifying agent of the group consisting of lithium oxide, anhydrous lithium hydroxide, lithium hydroxide and its hydrates at a temperature between about 35° and 45° C., for a time sufficient to cause one of said lithium compounds and acid to react and to form a soap, thereby causing the grease to set, said quantities being such that the reacted mixture contains between about 7 and 40% of said soap.

8. In a process of manufacturing a lubricant containing a lithium soap, the improvement consisting essentially of initially contacting a quantity of free soap-forming acid containing at least 16 carbon atoms in the molecule in solution in a lubricating oil with an approximately equivalent quantity of a saponifying agent selected from the class consisting of lithium oxide, anhydrous lithium hydroxide, lithium hydroxide, and its hydrates at a temperature of about 40° C. for a time sufficient to react at least 90% by weight of one of said lithium compounds and said acid to form a soap, and thereafter removing water from the resulting mixture by heating the product to a temperature between about 105–110° C. in thin layers.

9. In a process of manufacturing a lubricant containing a lithium soap, the improvement consisting essentially of intimately contacting a quantity of stearic acid in solution in a lubricating oil with an approximately equivalent amount of anhydrous lithium hydroxide at a temperature of about 40° C. for a time sufficient to react at least 90% by weight of said anhydrous lithium hydroxide and said stearic acid to form the soap.

10. In a process of manufacturing a lubricant containing a lithium soap, the improvement consisting essentially of intimately contacting a suspension of about 1.25 parts by weight of anhydrous lithium hydroxide in about 5 parts by weight of a lubricating oil with about 14 parts of weight of stearic acid in about 79.95 parts by weight of foresaid lubricating oil at a temperature of about 40° C. to form the soap and thereafter heating the resulting product to a temperature between about 105° to 110° C. in thin layers.

11. In a process of manufacturing a lubricant containing a lithium soap, the improvement consisting essentially of intimately contacting a

quantity of free soap-forming acid containing at least 16 carbon atoms in the molecule in solution in a lubricating oil with an approximately equivalent quantity of a saponifying agent of the group consisting of lithium oxide, anhydrous lithium hydroxide, lithium hydroxide and its hydrate at a temperature of between about 35° and 45° C. for a time sufficient to react at least 90% by weight of one of said lithium compounds and said acid to form a soap and dehydrating said grease in thin layers.

12. In a process of manufacturing a lubricant containing a lithium soap, the improvement consisting essentially of intimately contacting a quantity of stearic acid in solution in a lubricating oil with an approximately equivalent amount of anhydrous lithium hydroxide at a temperature of about 40° C. for a time sufficient to react at least 90% by weight of said anhydrous lithium hydroxide and said stearic acid to form the soap and dehydrating said grease in thin layers.

13. In a process of manufacturing a lubricant containing a lithium soap, the improvement comprising intimately contacting a suspension of about 1.25 parts by weight of anhydrous lithium hydroxide in about 5 parts by weight of a lubricating oil with about 14 parts by weight of stearic acid in about 79.95 parts by weight of foresaid lubricating oil at a temperature of about 40° C. to form the soap and dehydrating said grease in thin layers.

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