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LUBRICANT

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This invention relates to lubricants and the treatment of the same to improve the lubricating characteristics thereof, and is particularly applicable to mineral lubricating oils. Heretofore it has been customary to build up the oiliness and film strength characteristics of lubricating oils by the addition to such oils of minor proportions of various addition agents, of which may be mentioned the friction reducing agents such as fatty acids, fatty acid esters, castor oil, rape seed oil, Alox compounds (disclosed for example in U. S. patent to Burwell No. 1,863,004), and the acids, esters, aldehydes and ketones of organic acids and mixtures thereof, nitriles and ketones, and the halogen and sulphur film strength increasing agents of which there are many on the market.

I have discovered that the lubricating characteristics of a lubricating oil may be improved by oxygenating or air treating the lubricating oil at a temperature substantially above normal room temperature. This oxygenation or air treatment may be accomplished by aerating the oil, for example. This aeration may be accomplished by any of a number of different methods, such as the rapid beating or agitation of the oil with a mechanical beater and mixer, operating on the principle of the common household or culinary electric beater and mixer, or by spraying the oil into air, or by bubbling air through the oil. The intimate and thorough contact between the heated oil and the air at a temperature well above room temperature appears to add oxygen to the oil in a manner to change its characteristics, and by tests I have definitely established that such air treatment or aeration at temperatures above room temperatures will increase the lubricating characteristics of the oil, and particularly the film strength thereof. By treating the oil in this manner, the necessity of adding oiliness or film strength increasing agents is greatly reduced or eliminated, and oils having the desired lubricating characteristics can in most cases be produced directly.

In accordance with this invention, I aerate the oil or cause an intimate contact or mixture of the oil and air, while at a temperature well above room temperature, for a period of time which will produce approximately maximum increase in the lubricating characteristics of the oil. The increase in lubricating characteristics can be conveniently measured by measuring the increase in film strength as determined by the testing of samples of the oxygenated or aerated oil on a simple lubricant tester. As soon as the treated oil shows that it possesses approximately the maximum film strength obtainable by air treatment, the aeration is stopped and the oil cooled back to approximately room temperature, which stops further oxygenation of the oil. Other oiliness and film strength increasing agents may also be added to the oil if desired, and if the air treatment does not give the desired results.

In further explanation of the nature of the invention, a number of practical examples, based on tests, will be given for illustrative purposes. The oil used in one series of tests, except as otherwise noted, was an S. A. E. No. 90 gear oil made by blending 65% of No. 8 Pennsylvania Bright Stock with 35% of 230 viscosity (at 100° F.), zero pour, 2½ color Pennsylvania Neutral oil. The first group of tests on this blend of oils was made on the blend itself, and the second group of tests was made on the same blend of oil after air treatment or oxygenation in accordance with this invention, the aeration or air treatment occurring at a temperature of approximately 150° C. and while the oil was being beaten or stirred at full speed with a double impeller, "Sunbeam Mix Master" or similar culinary beater for 2½ minutes. The sample so mixed was a 2000 gram sample in an 8¼ inch

kettle. This stirring or beating of the
d heavy foaming thereof, and tended to
into the body of the oil.
sts which were made of the various oil
herein described, were all run on a Tim-
cant tester, the test block being exam-
he end of 1 minute, 3 minutes, and 10
an interval of 10 seconds being allowed
ase to start the machine, another inter-
seconds in which to load the machine
uniformly as possible, but this loading
10 seconds is, of course, included each
he total time under load. The results
ests are indicated in the following table:

TABLE II

Lever load, lbs.	Time, end of—	Naphthenic S. A. E. 50 motor oil as rec'd		Same, heated to 150 and vigorously stirred for 1 min. (air treated)	
		Scarf width Brin. div.	Timken film strength	Scarf width Brin. div.	Timken film strength
	Minutes				
12	1	5.67	10,600	5.33	11,300
	3	7.00	8,640	6.00	10,000
	10	7.00	8,640	6.67	9,080
15	1	Failed 42 sec. after loading.		5.33	14,100
	3			6.00	12,600
	10			Failed 215 sec. after initial loading.	

TABLE I.—*Penna. S. A. E. 90 gear base oil*

me, of—	Oil as received		Same, air treated		Oil and .5% Alox 150 ketone	
	Scarf width Brin. div.	Timken film strength	Scarf width Brin. div.	Timken film strength	Scarf width Brin. div.	Timken film strength
utes						
1	5.00	12,100	4.33	13,900	5.00	12,100
3	6.00	10,100	5.33	11,300	5.00	12,100
10	7.33	8,260	6.00	10,100	6.67	9,070
1	5.33	14,200	4.67	16,200	5.67	13,300
3	8.00	9,460	6.67	11,300	7.67	9,870
10	11.00	6,880	7.00	10,800	7.67	9,870
1	Would not carry 18# lever load		5.67	16,000	5.67	16,000
3			6.67	13,600	8.33	10,800
10			*7.33 (7 min.)	12,300	9.00	10,000

carf last loading.

sults of these tests given above in Table
indicate that the oil blend has increased
nd film strength from the air treatment
nation, which is comparable in many
he results obtained by the addition to a
end of oils of 0.5% of a special oiliness
agent which was made by the controlled
of Pennsylvania Neutral Oil, followed
mination of acidic material, such as by
tion and washing, and of which Alox
tone is an example. In Table I, the
divisions" refer to the average of the
ths taken at each end and the center
ith a Brinell microscope, the scale of
70 divisions, of which 63 divisions equal
The Timken film strength referred to
is the film strength calculated from the
formula:

$$\text{strength, lbs. per sq. in.} = \frac{\text{lever load} \times 10}{\text{scarf width in Brin. div.} \times K}$$

formula, K is a constant, equal to
1984, which is the factor employed for
the average scarf width in a Brinell
½ inch scarf length) into square inches.
en machine on which the tests were run
iven from a 5 horsepower, alternating
onstant speed motor. The motor may
n the tests herein referred to was, of
size in comparison to the power actu-
which makes speed variations during
erations virtually negligible. For all
purposes, the 5 H. P. motor on such
as a constant speed. Hence the speed
ractically constant at about 810 R. P.
Timken tester for all ranges of load up
nds.
thod of improving the lubricating char-
of an oil is not limited to one par-
e of oil, as is demonstrated by the
results of which are given below in
wherein the oil tested was an S. A. E.
cosity, solventized naphthenic motor

Where the oil is used for lubricating purposes at approximately room temperature or only slightly thereabove, any additional aeration or air treatment of the oil during use does not materially increase the oxygenation of the oil, because the air treatment must be at a temperature materially above room temperature, if the lubricating characteristics of the oil are to be changed materially. The higher the temperature of the oil above room temperature during air treatment, the more rapid the oxygenation of or change in the oil, and the period of time requisite for the desired air treatment of the oil will depend upon the temperature at which the aeration or contact with the air takes place. Excellent results are obtained when the temperature of the oil during aeration is approximately 150° C.

Oils treated in this manner are particularly valuable in the manufacture of maximum film strength, gear lubricants. A lubricant made up of 95% of the Pennsylvania No. 90 gear oil base mentioned above, and 5% of chlorinated paraffine (approximately 40% chlorine) heated to 150° C. and surface flashed but without such stirring as would incorporate much air into the oil, gave the tests shown under "Oil 1" in Table III below; the same oil heated to 150° C., and stirred with a beater vigorously for 2½ minutes (i. e. air treated), gave the test results under "Oil 2" below; the same oil reheated several times to 150° C. and air treated for a total of 30 minutes while at 150° C. gave the test results listed under "Oil 3" below; 75% of an oil of the same composition as "Oil 1," plus 25% of "Oil 3" blended together, gave the test results listed under "Oil 4" in Table III below; while an oil of exactly the same composition as "Oil 4" but after air treatment or aeration at 150° C. by means of an air jet for 20 seconds rather than high speed stirring, gave the test results listed under "Oil 5" in Table III below. "Oil 6," referred to in Table III below, was made up of 95% of the air treated or aerated oil of Table I, blended at 90° C. with 5% of the

same chlorinated paraffine. The results of tests on these various samples are listed in the following table:

TABLE III

Lever load, pounds	Time, end of—	Oil 1 Not "air treated" Timken film str.	Oil 2 Air treated 2½ min. Timken film str.	Oil 3 Air treated 30 min. Timken film str.
	Minutes			
90#	1	56,700#	59,300#	45,400#.
	3	42,500#	46,900#	36,700#.
	10	Failed 340 sec.	30,200#	27,200#.

Lever load, pounds	Time, end of—	Oil 4 75% like "Oil 1" 25% of "Oil 3" Timken film str.	Oil 5 Air jet "air treated" oil Timken film str.	Oil 6 Oil air treated, then 5% el. pffe. blended: Timken film str.
	Minutes			
90#	1	59,300#	59,300#	56,700#.
	3	45,300#	50,400#	45,300#.
	10	30,900#	30,900#	31,600#.

Lever load, pounds	Time, end of—	Oil 7 Large batch, air treated 6 min. Timken film str.	Oil 8 Air treated 15 min. additional Timken film str.	Oil 9 10 mg./100 gm. benzoic acid, air treated 15 min. Timken film str.
	Minutes			
90#	1	61,900#	59,300#	56,700#.
	3	45,900#	43,900#	46,900#.
	10	31,600#	Failed 390 sec.	31,600#.

Lever load, pounds	Time, end of—	Oil 10 10 mg./100 gm. di iso amyl amine, then air treated 15 min. Timken film str.	Oil 11 10 mg./100 gm. pyrogallol—then air treated 15 min. Timken film str.
	Minutes		
90#	1	59,300#	45,300#.
	3	48,700#	38,800#.
	10	Failed 300 sec.	Failed 390 sec.

These tests demonstrate that air treatment or aeration at temperatures well above room temperatures increases the film strength and other lubricating characteristics of an oil or gear lubricant, for example, to desirable limits. A comparison of the tests for "Oils 1 and 2" above clearly establishes this fact, but it will be noted from the tests listed under "Oil 3," that this air treatment or aeration can be carried or continued to such an extent that some of the desirable properties are lost, that is, the final results are not as satisfactory as they are when the air treatment period is shorter. I have found, however, that if the oil is air treated for too long a period such as is illustrated under "Oil 3," for example, the desired maximum lubricating characteristics may be restored to that overtreated oil by blending or incorporating therewith a proper quantity of non-air treated oil, as indicated under the results of tests on "Oil 4" in Table III. It is believed that oxygen will add on chemically to some parts or constituents of lubricating oils, particularly mineral oils, and it is my conclusion that air treatment of lubricating oil is a function of the air, presumably the oxygen of the air, that is chemically combined in the oil as shown by the results of the tests on "Oil 5" listed in Table III. That the air treatment increases the property of the oil itself, and not that of any film strengthening agent which may be added, is demonstrated by the test results listed under "Oil 6" in Table III, where the film strengthening agent received no air treatment whatever, and this indicates clearly that the air treatment or aeration of oil may be used on oil that also has added thereto, either before or after air treatment, any other addition agent for increasing the lubricating characteristics of the oil, such as any suitable or desired film strengthening or friction reducing agents.

As a matter of convenience, the addition of a film strengthening agent of known properties to a small sample of a batch of air treated oil, followed by the testing of the blend, is an easy method of developing and testing an oil for maximum air treated properties in a large batch, which properties are rather difficult to determine in any other well known laboratory method. Of course it is preferable to air treat the oil approximately to the maximum film strength or increase in lubricating characteristics, rather than beyond that point, but free of other film strengthening agents, yet the maximum point is hard to determine by any other ordinary method.

It is recognized that even properly prepared gear lubricants apparently wear out in use, that is, they lose some of their lubricating characteristics, which loss is believed to be due to deterioration caused by exposure of the lubricant to pressure, temperatures above room temperature, entrained air, and metallic surfaces and particles, and other foreign matter introduced into the lubricant during use. A study of that problem of loss of desirable lubricating characteristics, particularly in ordinary gear lubricants during use, has led me to believe that the chief cause of the trouble or loss of lubricating characteristics was due to over-oxidation of the lubricant, which is similar to the effects pointed

out in Table III above with reference to "Oil 3."

I have discovered that oxygen stabilizers added to the oil prevent such deterioration of the lubricants after maximum properties have been developed in the lubricant. Apparently any of the agents which inhibit or stabilize oxidation may be used for this purpose, and I have indicated in Table III the results obtained when such an oxygen stabilizer was added to the air treated oil after maximum results have been obtained. Benzoic acid and di iso amyl amine are examples of oxygen stabilizers which have been successfully used for this purpose, and the results of tests using such stabilizers are listed under "Oils 9 and 10" in Table III. To demonstrate this effect more forcibly, I have listed under "Oil 11" in Table III the results obtained after addition to the air treated oil of a minor proportion of pyrogallol which either accelerates oxygenation or deterioration of the oxygenated effect, that is, deterioration of the oxygenation of the oil, or adds an excess of oxygen material to the oil.

The tests, the results of which are tabulated in the foregoing tables, except for "Oil 7," were upon samples ranging in size from 1600 to 2000 grams and were all treated in 8½ inch diameter kettles. For "Oil 7," however, I used a 20 pound sample composed of 19 pounds of No. 90 gear base oil into which I blended 1 pound of the same chlorinated paraffine, and this mixture was air treated in a 13¼ inch diameter kettle for a total of 6 minutes at 150° C. "Oil 7" in Table III refers to this oil at this point. The results given in Table III for "Oil 8" were obtained after air treating 2000 grams of this same "Oil 7" 15 minutes longer at 115° C. in 8¼ inch diameter kettle. The results given for "Oil 9" on Table III were obtained by adding 10 mgs. of benzoic acid to 100 grams of this "Oil 7" material, then air treating exactly like "Oil 8." The results listed in Table III for "Oil 10" were obtained by adding 10 mgs. of di iso amyl amine to 100 grams of the same "Oil 7" material, then air treating the same exactly like "Oil 8."

The results given in Table III for "Oil 11" were obtained by adding 10 mgs. of pyrogallol to 100 grams of "Oil 7" material, then air treating exactly like "Oil 8." These tests clearly indicate marked retention of desired and better lubricating properties in the case of the benzoic acid sample. Similar results were obtained in the case of di iso amyl amine, although not quite so marked, and an acceleration of deterioration resulted when the pyrogallol was used. For the purposes of demonstration, equal amounts of the oxygen stabilizing materials and pyrogallol were used in these tests, but it will be understood that the invention is not limited to such amounts, because experience has demonstrated that the amount of one material required usually differs from that required when another material is used. Benzoic acid and di iso amyl amine belong to that class of materials which permits development of copper dish corrosion in the case of unstable gasoline and similar distillates, while pyrogallol is one of the most powerful inhibitors or preventors of such copper dish corrosion of unstable gasoline.

I have found that various oiliness agents, such as fatty acids, fatty acid esters, castor oil, rape seed oil, Alox compounds, nitriles, ketones, and various other friction reducing agents may also be added to the air treated oil, either before or after air treatment, in order to lower the coefficient of friction of the blend and increase the lubricating characteristics of the product.

It will be understood that the hereinbefore described tests have been tabulated and described for the purpose of explaining the nature of the invention, and the manner in which it may be carried out, and that the invention contemplates considerable variation in the details, materials and proportions which have been herein described in connection with the listed examples, within the principle and scope of the invention, as expressed in the appended claims.

I claim as my invention:

1. The method of increasing a lubricating characteristic of an oil without material thickening thereof, which comprises bringing said oil while substantially above room temperature into intimate contact with air for a period of time sufficient to obtain approximate maximum and possible film strength of a sample as determined by a lubricant tester, without material thickening of the oil, and then immediately stopping the intimate contact with air and cooling the oil.

2. The method of increasing a lubricating characteristic of an oil without material thickening thereof, which comprises bringing said oil while substantially above room temperature into intimate contact with air for a period of time dependent upon the temperature of the oil and the efficiency of the intimate contact of the air with the oil, and until samples thereof at successive intervals during the heating, when tested on a lubricant tester, show that approximately maximum film strength has been obtained, and before material thickening of the oil occurs, and then immediately cooling the oil.

3. The method of increasing a lubricating characteristic of an oil without material thickening thereof which comprises bringing the oil while at a temperature of approximately 150° C. into intimate contact with air for the minimum period of time which will produce desired and possible increased film strength, without material thickening of the oil, and then immediately stopping the intimate contact with air, and adding thereto a minor amount of an oxidation inhibiting agent.

4. The method of increasing a lubricating characteristic of an oil without material thickening thereof, which comprises aerating the entire body of the oil at a temperature above room temperature, for the minimum period of time, dependent upon the increased temperature and the efficiency of aeration, which will impart to the oil a desired and possible increased film strength without material thickening of the oil, and then adding thereto an oxidation stabilizing agent to halt further oxidation.

5. The method of increasing a lubricating characteristic of mineral lubricating oil without material thickening thereof which comprises aerating the oil under controlled conditions at a temperature well above room temperature until an increased film strength of the oil is obtained and before material thickening of the oil occurs, and then blending with said oil a minor proportion of an addition agent for stabilizing oxidation of the oil to halt further oxidation.

6. The method of directly increasing the film strength of a mineral lubricating oil without material thickening thereof which consists in aerating all of the oil to be so increased, under controlled conditions at a temperature substantially above room temperature merely until the oil has the desired and possible increased film strength,

then adding to the oil a minor proportion of an oxygen stabilizing agent to halt further oxidation of the oil.

7. The method of increasing a lubricating characteristic of a mineral oil without material thickening thereof which comprises directly aerating the oil to be so increased, under controlled conditions at a temperature substantially above room temperatures until the oil possesses a desired and possible increase in film strength as determined by the testing of a sample on a lubricant tester, and before material thickening of the oil has occurred, and then cooling the oil back to room temperature, and adding a minor amount of an oxidation inhibitor to halt further oxidation.

8. The method of increasing a lubricating characteristic of a mineral oil without material thickening thereof which comprises oxygenating a portion of the oil under controlled conditions at a temperature substantially above room temperature, until a possible and desired degree of oxidation is obtained, and before material thickening of the oil occurs, then adding another portion of the oil with a lesser degree of oxygenation until a desired and possible increase in film strength of the mixture of oils is obtained, and adding to the mixture a minor amount of an oxidation inhibiting agent to halt further oxidation.

9. An improved lubricant comprising a mineral lubricating oil which has been separately aerated under controlled conditions at a temperature substantially above room temperature until a desired and possible increase in film strength is obtained, and before material thickening of the oil has occurred, and an added minor proportion of an oxygen stabilizing agent.

10. An improved lubricant comprising a mineral lubricating oil which has been separately aerated at a temperature substantially above room temperature until a desired and possible increase in film strength is obtained and an added minor proportion of an oxidation stabilizing agent of the type of benzoic acid and di iso amyl amine.

11. An improved lubricant comprising a mineral lubricating oil which has been separately aerated at a temperature substantially above room temperature until a desired and possible increase in film strength is obtained, and before material thickening of the oil has been caused, an added minor proportion of a film strength increasing, addition agent, and an added minor proportion of an oxygen stabilizing agent.

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