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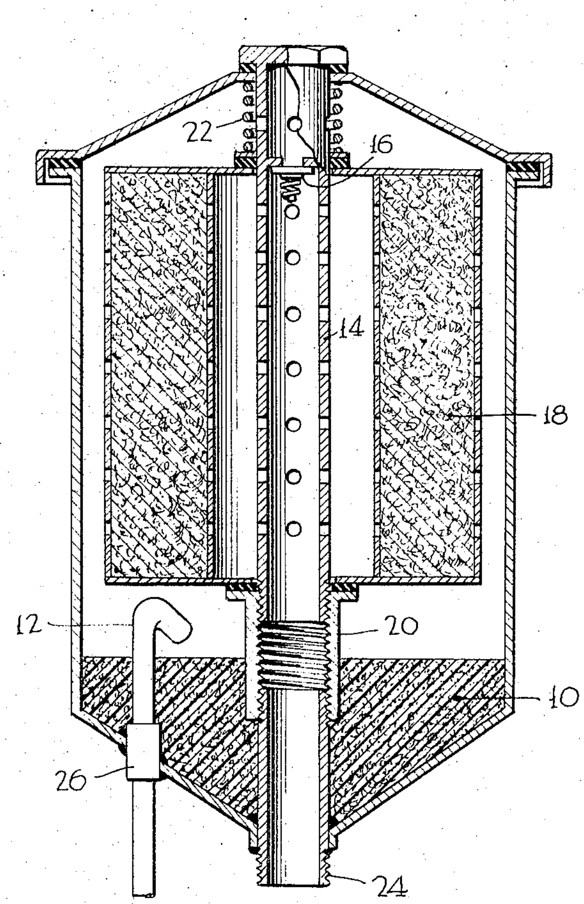

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METHOD AND MEANS FOR MAINTAINING AN EFFECTIVE CONCENTRATION OF ADDITIVES IN OIL

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This invention relates to a means for prolonging the useful life of libricating oil and more particularly, concerns a method, a composition, and a device for maintaining an effective concentration of additives in the oil during its use in a circulation system, such as, for example, the oil circulation system servicing an internal combustion engine.

Purchasers of recent automobile models have been advised by automobile manufacturers that the period between crankcase oil change as measured by distance traveled, be extended from about 2,000 miles to about 6,000 miles. Current quality crankcase oils contain adjuvants, commonly referred to as oil additives, which serve to improve and maintain the properties of the oil and thus prolong its useful life and concomitantly that of the engine. Examples of such additives are viscosity index improvers, oxidation inhibitors, detergents, sludge inhibitors, film strength agents and neutralizing agents.

Whereas the lubricity property of the oil, that is its ability to reduce friction between metal surfaces, is maintained over a long period of time, the additives which are employed to improve the oil's efficiency are relatively quickly depleted during engine use. In this connection, studies have shown that additives which are incorporated into oil to neutralize acids as they are formed (thereby reducing ring and cylinder bore wear during low temperature operation) are depleted exponentially, that is one-half of the remaining additive is lost every several hundred miles. Other additives similarly are depleted rapidly during use of the oil.

It is undesirable to initially incorporate larger concentrations of the additives in the oil. For example, an excess amount of polymeric viscosity index improver added originally to the oil detrimentally affects cold cranking when the oil is new and excess concentrations of oxidation inhibitors may deleteriously affect the performance of detergent additives present in the oil. Also the cost of the oil is increased as the concentrations of additives is increased. Moreover, this additional cost is not justified because the increase in the life of the additive in the oil due to the presence of more additive is negligible when compared to the increase in the cost of the oil.

Various methods for continuously replenishing the additives as they are depleted from the oil have been previously suggested. For instance, U.S. Patent No. 2,302,552 discloses that a lubricating oil may be replenished with additives consumed during use by passing the oil from the engine crankcase through a chamber provided with a porous absorbent medium impregnated with a relatively oil insoluble additive which slowly dissolves into the oil. Preferably the oil is filtered first. U.S. Patent No. 2,310,305 discloses that a chamber within the oil may be loaded with a slightly oil soluble additive which can be in solid or fine particle form or the filter material or elements may be impregnated with the additive. Other methods for incorporating the additives into the oil filter have also been suggested. For instance, the additives in particle form may either be commingled with the filter elements or alternatively, the additive in granular form may be bonded to the filter element by means of a porous thermo-setting adhesive.

To the best of my knowledge it has not heretofore been possible to effectuate a controlled release of highly oil soluble additives into an oil circulation system. Such additives, when contacted with the oil in accordance with known methods are immediately dissolved into the oil only to be depleted quickly. According to prior art teachings different methods must be utilized to contact the oil with additives that are in different physical states, that is, in liquid or solid state.

It is therefore a primary object of this invention to provide an improved means for prolonging the useful life of an additive-containing lubricating oil.

It is another object of this invention to provide an improved process for continuously adding to the oil in controlled amounts materials which impart beneficial properties to the oil during its use in an oil circulation system.

It is a further object of this invention to provide a composition which is useful for maintaining an effective concentration of a highly oil soluble additive within the oil during its use in an oil circulation system.

It is still a further object of this invention to provide an improved device for prolonging the useful life of an additive containing fluid lubricant.

The foregoing objects may be attained in acordance with the present invention by the process for incorporating an additive into lubricating oil circulated through an oil circulation system including the step of contacting at least a portion of the oil with a solid thermoplastic polymer having the additive compounded therein, the polymer having a low rate of dissolution in the oil. This readily may be achieved by depositing a composition comprised of a solid thermoplastic polymer having compounded therein the additive, the polymer having a low rate of dissolution in the oil, this composition hereinafter referred to for convenience as the polymer-additive composition, in a device comprised of a chamber having oil inlet means and oil outlet means and circulating at least a portion of the oil through the chamber for contact with the polymer-additive composition. In oil circulation systems where it is expedient or mandatory that the oil be filtered during use, as in the oil circulation system which services an automobile engine, it is preferred to deposit the polymer-additive composition in the filter such as one shown in the accompanying drawing which is a cross-sectional view of a filter and which will be more fully described hereinafter.

As the oil contacts the polymer-additive composition, the polymer having a low rate of dissolution in the oil, is slowly dissolved and/or dispersed therein. If the additive which is compounded in the polymer is oil soluble it will dissolve slowly into the oil, that is, at the same rate as the polymer-additive composition. On the other hand, if the additive is oil insoluble, it, when exposed to the oil, will be carried along with the oil to perform its function. The polymer thus serves two purposes. It is the carrying medium for the additive and it also protects the additive from immediate contact with the oil thereby insuring that the additive is added continuously to the oil over a relatively long period of time. Accordingly, the polymer must be a thermoplastic polymer having a low rate of dissolution in oil, and be of a sufficiently high molecular weight so that the polymer and consequently, the polymer-additive composition be solid at the temperature of oil contact, i.e., range in viscosity from a plastic-solid to solid. Polymers which have a low rate of dissolution in oil include polymers which are relatively oil insoluble or relatively nondispersible in oil and polymers which although highly oil soluble or highly oil dispersible go into solution with difficulty, e.g. it is necessary that they be agitated in the presence of the oil for a relatively long period of time before going into the solution.

Virtually any polymer that has these properties may be used in the practice of this invention such as, for example, ethylene-propylene copolymers ranging in molecular weight from 200,000 to 300,000; ethylene-ethylacrylate copolymers ranging in molecular weight from 200,000 to 300,000; polypropylene oxide having a molecular weight of about 500,000; and ethylene-vinyl acetate copolymer ranging in molecular weight from 200,000 to 300,000. It should be understood that any polymer utilized will improve to some extent the viscosity properties of the oil as the polymer goes into solution. It is preferred to utilize a polymer that has optimum oil viscosity improving properties or one that imparts other beneficial properties to the oil. For instance, many polymeric materials are utilized as detergents, oxidation inhibitors, extreme pressure additives, etc. Use of these type polymers obviates the incorporation of an additional additive into the polymer-additive composition. Exemplary polymers of this type are the polymethacrylates and the polyalkylmethacrylates wherein the alkyl group contains from 1 to 20 carbon atoms. These polymers range in molecular weight from 400,000 to 1,200,000. Polyalkylacrylates wherein the alkyl side chain has a carbon content in excess of 9 and which have a molecular weight of about 1,000,000 are also suitable. In addition copolymers obtained by polymerizing a C_{10}–C_{20} alkyl methacrylate such as tetradecyl methacrylate with a vinylpyridine such as 2-vinylpyridine can also be used. Suitable copolymers range in molecular weight from 200,000 to about 800,000. Polystyrene ranging in molecular weight from 30,000 to 50,000 and copolymers obtained by polymerizing propylene with a C_{10} to C_{24} monoolefin and ranging in molecular weight from 50,000 to 150,000 are further examples of suitable polymers. Polyisobutylene ranging in molecular weight from 81,000 to 135,000 is still another example of a preferred polymer.

The present invention will find applicability in any oil circulation system where it is desirable to add additives to the oil and particularly in one wherein the lubricity qualities of the oil are maintained over a long period of time whereas the additives are depleted in a relatively short period of time. For example, the oil circulation systems which service diesel engines, gasoline engines, steam turbines and the like are all benefited by the use of the present invention.

It should be understood of course that polymer selection will depend on the system in which it is used because of the different operating temperatures of different engines. As stated hereinbefore the polymer must be a plastic solid or solid mass and have a low rate of dissolution in oil at the temperatures of oil contact. Consequently there would be selected a higher molecular weight polymer having a lower rate of dissolution in oil for a polymer-additive composition utilized in the oil circulation system servicing a steam turbine than would be selected for utilization in a system that services an automobile engine which operates at lower average temperatures than a steam turbine.

The proportion of polymer that comprises the polymer-additive composition will depend on the rate at which it is desired to have the additives fed into the oil. The higher the proportion of polymer in the polymer-additive composition, the slower the rate of additive addition.

Additives suitable for admixing or compounding with the polymer can be in liquid or solid form; they can be oil insoluble, or they can range in oil solubility from partially to completely soluble. Mixtures of these various types of additives can be used conveniently and effectively.

It should be understood that "additive" when used herein and as a part of the term "polymer-additive composition" includes all materials which can be compounded or admixed with the polymer and which in any way impart beneficial properties to the oil circulation system or the apparatus that is lubricated. For example, the term

"additive" in addition to the various types of additives already mentioned would also include materials which function to increase the particle removal rate of the filtering media in oil circulation systems where filters are utilized.

The particular additives to be admixed with the polymer will of course, depend on the type of oil circulation system utilized and the problems connected therewith. For instance, a quality crankcase lubricant contains, as mentioned hereinbefore, a variety of additives. For example, detergent additives such as the metal sulfonates, metal phenates, metal phosphenates, derivatives of alkenyl succinimides and the like may be admixed with the polymer. Suitable oxidation inhibitors that can be included in the polymer-additive composition are the metal dithiophosphates, metal dithiocarbamates, hindered phenols aromatic amines, etc. Extreme pressure and oiliness additives such as sulfur, metal naphthenates, phosphate esters, and sulfurized hydrocarbons, etc., may also be admixed with the polymer. The polymer-additive composition may also contain alkaline agents such as overbased metal sulfonates or colloidal inorganic salts such as alkaline earth carbonates, calcium hydroxide, etc. "Multi-purpose oil additives," such as the sulfurized metal phenates and polyolefin substituted succinimides, also may be used. The above list is not meant to be all-inclusive, but is merely exemplary of some of the types and kinds of additives that can be used.

The polymer-additive composition may be prepared in any suitable way. It can be very conveniently prepared by heating and stirring the components in suitable mixing equipment where shear action is employed such as in a Banbury mixer, Sigma Blade mixer, and/or a laboratory mill. The mixing speeds, cycle times, and temperature will, of course, vary depending upon the particular components which are being blended. If the additives are liquid and/or powdery solids and the polymeric material is rubbery, it is preferred that the ingredients be compounded first on a laboratory mill prior to further compounding in the shear mixing device. If on the other hand, the additives are in liquid or powdery form and the polymer is in powdery form, it is preferred that the ingredients be premixed in a dry mixing device such as a Hobart mixer or a Henschel mixer prior to further shear compounding. After compounding in the shear mixing equipment is completed, the polymer-additive composition can be molded into the desired shape or form. If the composition is a hard rather than a rubbery solid it can be sheeted off of a laboratory mill to approximately ¼" sheets and fed to a suitable dicing apparatus for reduction to pellets of the order of ⅛" to ¼" in dimension. The pellets may then be fed to an injection molding or compression molding machine for conversion into the desired final shape.

Utilizing the above described methods, there also can be prepared a polymer-additive composition comprised of two or more layers of polymer-additive compositions which vary in additive concentration and/or rate of oil dissolution.

The polymer-additive composition can be incorporated into the oil circulation system for contact with the oil in any suitable manner. For instance, the polymer-additive composition may be deposited in a chamber that is provided with means for allowing the oil to flow in and out of the chamber. It may be desirable to contact only a portion of the oil with the polymer-additive composition. As stated hereinabove, if the oil is filtered, it is preferred that the polymer-additive composition be deposited within the filter.

The rate at which the additives are fed into the oil also can be controlled by the manner in which the oil is contacted with the polymer-additive composition. For instance, and with reference to the drawing, the rate of addition of the polymer-additive composition **10**, to the oil may be controlled simply by changing the angle of the oil feed tube **12**. The feed tube as pictured allows the oil to strike the polymer-additive composition at approximately a 45° angle. This results in much oil agitation at the surface of the polymer-additive composition thereby increasing its rate of dissolution. On the other hand, the oil feed tube could be inserted vertical to the polymer-additive composition or horizontal to and a distance from the polymer-additive composition so that the oil would enter the filter without agitating the surface of the polymer-additive composition. Consequently the rate of dissolution of the polymer-additive composition would be slower.

In order that those skilled in the art may better understand how the present invention may be practiced, the following examples are given by way of illustration and not by way of limitation.

Example I

There was prepared a polymer-additive composition comprised of the following materials:

	Weight percent
Vistanex L–100	25.0
Oronite 1200	57.0
Bryton Hybase (calcium C–300)	14.3
Lubrizol 1095	3.7

Vistanex L–100, a viscosity index improver, is a polyisobutylene having an average molecular weight ranging from 81,000 to 99,000 as measured by the Staudinger formula. Oronite 1200, a commercial liquid detergent additive, is a highly oil-soluble alkenyl succinimide derivative. Bryton Hybase (calcium C–300), a commercial liquid alkaline agent, is a highly oil-soluble over-based calcium sulfonate. Lubrizol 1095, a commercial liquid oxidation inhibitor and film strength improver, is a highly oil-soluble zinc dialkyl dithiophosphate. The polymer-additive composition was prepared as follows. The Vistanex L–100 was cut into ¼″ cubes and deposited in a grease kettle which was fitted with counter-rotating stirrers. The Oronite 1200 and Bryton Hybase were added to the kettle. The contents of the kettle were continuously stirred while their temperature was maintained between 200° F. and 290° F. After a number of hours a homogeneous mass was obtained. The Lubrizol 1095 was then admixed with this mass at a temperature of 250° F. A homogenous mass was obtained after one hour of stirring. (This additive was not added sooner because it decomposes upon heating for long periods of time.) The contents of the kettle were then allowed to cool to room temperature and there was obtained a plastic-solid mass. Re-

ferring now more particularly to the drawing, a 265 gram portion **10** of this plastic-solid mass was deposited in the bottom of the filter. The filter was a standard AC Type S–2 oil filter commonly known as a large by-pass type filter and utilized in the oil circulation system of a truck engine. The filter was modified by replacing the central cylindrical pipe of the filter with a shorter pipe **14** having an eight pound differential by-pass valve **16**. The filtering element of the oil filter also was replaced by a conventional full flow oil filter cartridge **18** of the same diameter as the original element but shorter in height. The use of the shorter cartridge provided the necessary space for the polymer-additive composition. The central cylindrical pipe **14** was connected to a flanged coupling **20** that supported the filter cartridge **18** at its base. The cartridge **18** was held in place against the flanged coupling **20** by a helically wound spring **22**. The oil outlet pipe **24** was connected to the bottom of the flanged coupling **20**. The filter housing also was modified in that the original oil inlet pipe was plugged and the oil drain plug was replaced with a pipe socket **26**, which accommodated the oil inlet pipe **12**.

To demonstrate the effectiveness of the polymer-additive composition, the filter was connected to the flow lines of the oil circulation system of an automobile engine and a modified Sequence V test was run. This test simulates the operation of an automobile engine running at low temperatures, such as that experienced in stop-and-go driving, and is used to evaluate the effectiveness of the crankcase oil. The test was modified in that a 1960 Ford engine was used and the oil was filtered. As the selected filter did not fit the filter flange of this engine, an adapter plate was made for the filter flange and the oil lines from the filter were connected to the adapter plate. The filter was operated as a full-flow filter. The crankcase oil utilized was a standard CRC reference oil, REO 132–60, which is a fully compounded oil having a mild detergency level. The test was run for 192 hours at which time all of the polymer-additive composition had been taken up by the oil. At various times during the test and at the end of the test the oil alkalinity was measured. At the end of the test the oil was analyzed for viscosity, viscosity index, film strength properties, and dispersed insolubles. Another Sequence V test, modified as described above, also was run, but in this test run there was no polymer-additive composition placed in the oil filter. The same oil properties as mentioned above were measured at the end of the test. The results of these tests are included in Table I below

TABLE I

Physical and chemical properties of oil	No. of hrs. engine had run when properties were measured	Oil filter with polymer-additive composition	Oil filter conventional
Alkalinity (ASTM D664–58):			
Total Base No.	0	0.24	0.24
Do.	16	1.52	------
Do.	32	0.65	<0.1
Do.	40	0.53	<0.1
Do.	80	<0.1	<0.1
Do.	192	<0.1	<0.1
Viscosity:			
Viscosity, SUS/100° F. (ASTM D445–61).	0	340.7	340.7
	192	335.6	282.4
Viscosity, SUS/210° F. (ASTM D445–61)	0	63.2	63.2
	192	64.0	54.8
Viscosity Index (ASTM D567–53)	0	133.5	133.5
	192	137.0	121.0
Sulfated ash content, wt. percent (ASTM D874–63)	192	1.04	0.20
Oil insolubles (ASTM D893–60T):			
Pentane insolubles, wt. percent	192	1.43	0.41
Benzene insolubles, wt. percent	192	1.08	0.21
Oil insolubles:*			
Pentane insolubles, wt. percent	192	3.54	0.38
Benzene insolubles, wt. percent	192	1.31	0.13
Film Strength: Falex Seizure Test, lbs.	0	1,830	1,830
	192	4,416	1,166

*(Test described in SAE Paper No. 262C, 11/60).

It is evident from the above table that the used oil which had been contacted with the polymer-additive composition had better overall properties at the end of the test than the oil not so contacted. The oil into which the additives were fed maintained its alkalinity for a longer period of time, and had higher viscosity, viscosity index, and film strength properties at the end of the test. It is also evident from the above table that the oil that was contacted with the polymer-additive composition had better detergent properties as evidenced by its containing a higher per cent of dispersed insolubles.

To further evaluate the effectiveness of the polymer-additive composition, cleanliness ratings of the engine parts were made. At the end of each test the engine was dismantled, inspected, and the standard CRC engine sludge and varnish rating scales (10-clean; 0-dirty) were used to determine the amount of varnish and sludge deposited on various engine parts. These measurements are tabulated in Table II below.

TABLE II

Evaluation of condition of engine parts	Deposition ratings	
	Oil filter with polymer-additive composition	Oil filter conventional
Varnish:		
Piston	8.2	6.0
Rubbing surfaces*	7.0	5.2
Overall average	6.0	5.7
Sludge:		
Overall average	8.0	6.3
Percent oil screen plugging	0	6.0
Percent ring plugging	0	4.0

*Cylinder walls, valve stems, rocker arms, valve lifters.

It is evident from Table II above that the polymer-additive composition functions to prolong engine life. This is evidenced by the significant improvement in the over-all sludge and varnish ratings of the engine serviced by the oil circulation system that contained the polymer-additive composition. It also will be noted that there was no oil screen plugging in the test run that utilized the polymer-additive composition.

The following example is included to demonstrate how the rate of addition of an additive to the oil may be controlled by utilizing polymers having different rates of dissolution in oil.

Example II

There was set up an oil filter flow bench. This was comprised essentially of an oil reservoir, a variac for heating the oil in the reservoir, a gear pump, the filter described in Example I and illustrated in the accompanying drawing, and pipes connecting the filter with the reservoir. The inlet pipe **12** of the filter was connected to the bottom of the oil reservoir by means of a pipe which was fitted with a gear pump, by-pass valve and pressure gauge. The outlet pipe of the filter **24** was connected to a pipe which ran to the top of the reservoir. This pipe was fitted with a pressure gauge and an 0.07 inch orifice. The oil was pumped from the reservoir through the filter and returned to the reservoir. The variac and by-pass valve were adjusted to simulate temperature and flow rates of an automobile oil system. The temperature of the oil in the reservoir was maintained at 200° F. and the oil was pumped through the system at the rate of 0.6 gallon per minute during operation. In one run there was deposited in the filter a polymer-additive composition comprised of 25 weight percent Vistanex L–100 (average molecular weight 81,000 to 99,000) and 75 weight percent Oronite 1200. In another run there was deposited in the filter a polymer-additive composition comprised of 25 weight percent Vistanex L–140 (also a polyisobutylene polymer, average molecular weight 117,000 to 135,000) and 75 weight percent Oronite 1200. The amount of Oronite 1200 contained in each of the polymer-additive compositions would comprise 4.3 volume percent of the oil upon complete dissolution into the oil. Each run was operated for 48 hours. At the end of each run the oil was analyzed. It was found that the oil which was contacted with the polymer-additive composition that contained the lower molecular weight polymer, that is the polymer having a higher rate of dissolution in oil, contained 3.9 volume percent of Oronite 1200. On the other hand, the oil which was contacted with the polymer-additive composition that contained the higher molecular weight polymer, that is the polymer having a lower rate of dissolution in oil, was found to contain 2.8 volume percent Oronite 1200. Thus it is evident that under the same operating conditions the rate of addition of additives to the oil may be varied by utilizing polymers having different rates of dissolution in oil.

The present invention has numerous advantages over heretofore known methods for incorporating an additive into lubricating oil circulated through an oil circulation system. The rate of addition of the additive to the oil is controlled by the polymer and therefore, is not dependent on the solubility of the particular additive utilized. Consequently, the use of the additives is not limited to those which are relatively oil insoluble. This is extremely important in view of the fact that a good many of the best additives are highly or completely soluble in oil. According to prior art methods a liquid additive could be utilized only by impregnating the filter elements, or some other porous absorbent medium which came into contact with the oil, with the liquid additive. As pointed out hereinbefore, highly or completely oil soluble additives previously could not be utilized with any degree of success because they immediately dissolved into the oil and, consequently, were quickly depleted. According to the instant invention, these additives may be used successfully because they are protected by the polymer from immediate contact with the oil. Another advantage of the instant invention is that a mixture of additives which are in the same or different physical states can be prepared conveniently in a one-step process, i.e., they simply are compounded into the polymer.

I claim:

1. A method for maintaining in an oil circulation system an effective concentration of oil additives which tend to become depleted over a period of service, comprising contacting the oil with an oil soluble, solid thermoplastic polymer having the oil additive compounded therein, said polymer having a low rate of dissolution in oil which controls the rate of release of the oil additive into the system.

2. The process of claim **1** wherein said thermoplastic polymer is a polyolefinic material.

3. The process of claim **2** wherein said polyolefinic material is polyisobutylene.

4. The process of claim **1** wherein said additive is highly oil soluble.

5. A composition for use in incorporating an additive into a lubricating oil circulated through an oil circulation system which comprises an oil soluble solid thermoplastic poymer having an oil additive compounded therein, said polymer having a low rate of dissolution in said oil.

6. The composition according to claim **5** wherein said thermoplastic polymer is a polyolefinic material.

7. The composition according to claim **6** wherein said polyolefinic material is polyisobutylene.

8. The composition according to claim **5** wherein said additive is highly oil soluble.

9. A device for incorporating an oil additive in a lubricating oil circulated through an oil circulation system which comprises a chamber having oil inlet means and oil outlet means, and a deposit in said chamber for direct contact with at least a portion of the oil as it passes through said chamber, said deposit comprised of an oil soluble solid thermoplastic polymer having an oil additive compounded therein, said polymer having a low rate of dissolution in said oil.

10. An oil filter for incorporating an oil additive in a lubricating oil circulated through an oil circulation system having deposited therein a composition comprised of an oil soluble solid thermoplastic polymer having an oil additive compounded therein, said polymer having a low rate of dissolution in said oil.

11. The oil filter of claim **10** wherein said thermoplastic polymer is polyisobutylene and said additive is a highly oil soluble material.

12. A composition according to claim **7** wherein the polyisobutylene has a molecular weight ranging from 81,000 to 135,000.

13. A composition according to claim **5** comprising a solid thermoplastic polymeric viscosity index improver having a detergent additive, an alkaline agent, and an oxidation inhibitor compounded therein, said viscosity index improver having a low rate of dissolution in oil.

14. A composition according to claim **5** comprising a polyisobutylene having a molecular weight ranging from 81,000 to 99,000 having compounded therein an alkenyl succinimide, calcium sulfonate and zinc dialkyl dithiophosphate.

15. An oil filter according to claim **11** wherein the polyisobutylene has a molecular weight ranging from 81,000 to 135,000.

16. A device according to claim **9** wherein said chamber has

(a) a filter cartridge located in the upper portion thereof,

(b) wherein said oil soluble thermoplastic polymer having said oil additive compounded therein is located in the lower portion of said chamber, and

(c) wherein said oil feed inlet is positioned in such a manner as to allow the incoming oil to strike the surface of said thermoplastic polymer.

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