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TREATMENT OF WOOD TAR DISTILLATE

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This invention relates to the treatment of wood tar distillate. More particularly it concerns a new and improved inhibitor oil and a method of preparing the same.

In the destructive distillation of wood, there is obtained, in addition to pyroligneous acid, an insoluble viscous material commonly termed "settled tar," which is usually separated into various fractions by distillation at reduced pressure. One of these fractions, having an atmospheric boiling range of roughly 220°–320° C., is known as "inhibitor oil," and finds extensive use as a stabilizing agent for preventing gum formation in gasolines and like petroleum products. Unfortunately, however, inhibitor oil as ordinarily prepared develops after standing an appreciable proportion of dark reddish-brown coloring material of unknown constitution, which lessens its desirability as an inhibitor.

In view of this disadvantage, an object of the present invention is to provide a method of treating inhibitor oil to remove therefrom much of the objectionable dark reddish-brown coloring material.

This object is obtained by subjecting the inhibitor oil to the action of an oxidizing agent and thereafter separating the substantially unoxidized fraction from the oxidized material, suitably by distillation. The product so obtained is a straw-colored oil which has an inhibiting power at least equal to that of ordinary inhibitor oil, and, even after long standing, develops very little reddish-brown coloring material. In addition, it is completely soluble in gasoline, as determined by the usual method. That is: three milliliters of the oil in a centrifuge tube are diluted to 100 milliliters with reference gasoline, and the sample is shaken thoroughly for ten minutes, and then centrifuged for ten minutes, after which the volume of the oil layer remaining is measured to determine the percentage of insoluble material.

The exact nature of the chemical reaction occurring during the oxidative treatment according to the invention is not understood. However, the oxidizing agent appears to attack preferentially the dark reddish-brown coloring materials and gasoline-insoluble bodies in the inhibitor oil, and to convert them to non-volatile tar-like substances from which the inhibitor oil can be separated by distillation or other means.

In so far as we have been able to determine, any oxidizing agent is operable in the invention. However, inorganic oxidizing agents, and particularly gaseous or volatile liquid inorganic agents, such as oxygen, air, and hydrogen peroxide, are to be preferred because of the fact they leave no residue in the treated oil.

In a preferred mode of operating, the inhibitor oil and a small proportion, usually 0.5 to 2.0 per cent, of the oxidizing agent are mixed and the mixture is stirred at a temperature of 20°–50° C. for several hours, after which it is heated to a temperature of 80°–120° C., for one or two hours. However, the oxidizing agent and oil may be allowed to react under other conditions of time and temperature, preferably within the range 20°–120° C. without departing from the invention. After the oxidation treatment, the resulting mixture is distilled, preferably though not necessarily at reduced pressure, to recover the purified inhibitor oil as a yellow liquid, which dissolves completely in cracked gasoline (according to the solubility test previously outlined), and when present therein in a small proportion, e. g. 0.01 to 0.1 per cent by weight, effectively retards gum formation.

Treatment according to the invention may also be accomplished by exposing the inhibitor oil in the vapor state to the action of the oxidizing agent, as during the distillation operation.

The following examples will illustrate the invention but are not to be construed as limiting its scope.

Example 1

A sample of inhibitor oil prepared by the vacuum distillation of hardwood "settled tar", having an atmospheric boiling range of 230° C.–297° C., a specific gravity of 1.093 at 15.6° C./15.6° C., and a color drop value of 22 (A. S. T. M. D–156–34T), was treated according to the invention. A sample of the oil was placed in a closed iron pressure vessel and oxygen passed into the container until a gauge pressure of 100 pounds per square inch was reached. The gas inlet was then closed and the vessel heated at 100° C. for three hours, during which time the gas pressure rose to 130 pounds and then gradually dropped to 100 pounds. The resultant treated oil was removed and distilled at reduced pressure, the distillate being a yellow-colored inhibitor oil having a color drop value of 30, which did not change appreciably even on long standing. A small proportion of tarry residue was left in the still.

Example 2

The untreated inhibitor oil of Example 1 was heated at a temperature of 100° C. for one hour

under oxygen maintained at a constant pressure of 20 pounds gauge, and was then vacuum distilled. A light-yellow inhibitor oil was obtained.

Example 3

A mixture of ordinary inhibitor oil and 1 per cent by volume of a 30 per cent (100 volume) aqueous solution of hydrogen peroxide was heated with agitation at about 40° C. for 10 hours, and was then heated at about 80° C. for a short period to drive off any unreacted peroxide. The resulting product was distilled, there being obtained a light-colored inhibitor oil which had excellent color stability and was completely miscible with gasoline.

Example 4

A charge of ordinary inhibitor oil was heated at a temperature of 70° to 90° C. for 24 hours, during which time air was blown continuously through the hot oil. The product was then vacuum distilled, the distillate being a light-yellow inhibitor oil as in the preceding examples.

Example 5

In a series of tests, ordinary inhibitor oil was mixed with a small proportion of each of the following oxidizing agents: nitric acid, aqueous potassium permanganate, manganese dioxide, yellow mercuric oxide, mercuric acetate, potassium chlorate, sulfuric acid, and saturated bromine water. Each test sample was heated at a temperature of 40° C. for 15 to 20 hours followed by a further heating at 100° to 120° C. for two hours, and was then distilled at reduced pressure. In every instance, the distillate was a stable light-colored inhibitor oil having gasoline gum-inhibiting properties at least equal to those of ordinary inhibitor oil.

It is to be understood that the process of the invention is applicable to the treatment of inhibitor oil as prepared from "settled tar" from the destruction distillation of any ligno-cellulosic substance, particularly hardwoods, such as birch and maple, as well as softer woods such as hemlock, and other like materials, including peat and rotten wood. The process is ordinarily applied to the inhibitor oil itself, or to a mixture of all the fractions obtained by distilling "settled tar" under reduced pressure, excluding the lowest boiling fraction which essentially comprises methanol products, water, and acetic acid. However, it is also within the scope of the invention to treat "settled tar" or other substance containing the inhibitor oil with an oxidizing agent, and thereafter to distill the treated product to separate the improved light-colored stable inhibitor oil of the invention.

Other modes of applying the principle of the invention may be employed instead of those explained, change being made as regards the details disclosed, provided the method or product stated in any of the following claims, or the equivalent thereof, be employed.

We claim:

1. The method which comprises subjecting the inhibitor oil derived from the tarry products of wood distillation to the action of an oxidizing agent, and separating the substantially unoxidized fraction from the tarry oxidation products, the product so obtained being light-colored, miscible with gasoline, and effective as an inhibitor in preventing gum formation in gasoline and like petroleum products.

2. The method of treating the inhibitor oil derived by distillation from the tarry products of wood distillation, which comprises subjecting the said oil to the action of a small proportion of an oxidizing agent, and distilling the treated product, to recover as distillate a light-colored oil, miscible with gasoline, and effective as an inhibitor in preventing gum formation in gasoline and like petroleum products.

3. The method of treating the inhibitor oil derived by distillation from the tarry products of wood distillation, which comprises subjecting the said oil to the action of a small proportion of an inorganic oxidizing agent and distilling the resulting product, to recover as distillate a light-colored oil, miscible with gasoline, and effective as an inhibitor in preventing gum formation in gasoline and like petroleum products.

4. The method of treating the inhibitor oil derived by distillation from the tarry products of wood distillation, which comprises subjecting the said oil to the action of a small proportion of a gaseous inorganic oxidizing agent, and distilling the resulting product, to recover as distillate a light-colored oil, miscible with gasoline, and effective as an inhibitor in preventing gum formation in gasoline and like petroleum products.

5. The method of treating the inhibitor oil derived by distillation from the tarry products of wood distillation, which comprises subjecting the said oil to the action of a small proportion of a volatile liquid inorganic oxidizing agent, and distilling the resulting product, to recover as distillate a light-colored oil, miscible with gasoline, and effective as an inhibitor in preventing gum formation in gasoline and like petroleum products.

6. The method of treating the inhibitor oil derived by distillation from the tarry products of wood distillation which comprises heating the said oil at a temperature between 70° and 90° C. and passing air therethrough, and thereafter distilling the treated oil to recover as distillate a light-colored oil, miscible with gasoline, and effective as an inhibitor in preventing gum formation in gasoline and like petroleum products.

7. In a method of treating the mixture of all the fractions obtained by distilling "settled tar" under reduced pressure except the lowest boiling fraction essentially comprising methanol products, water, and acetic acid, said mixture of fractions containing inhibitor oil, the steps which comprise subjecting the said mixture to the action of an oxidizing agent, and thereafter fractionally distilling the treated mixture to recover the fraction having an atmospheric boiling range of 220° C. to 320° C., this fraction being a light-colored oil, miscible with gasoline, and effective as an inhibitor for preventing gum formation in gasoline and like petroleum products.

8. The method of treating the tarry mixture derived from the destructive distillation of wood and known as "settled tar", said mixture containing inhibitor oil, which comprises subjecting the said mixture to the action of an oxidizing agent, and thereafter fractionally distilling the treated mixture to recover the fraction having an atmospheric boiling range of 220° to 320° C., the said fraction being a light-colored oil, miscible with gasoline, and effective as an inhibitor for preventing gum formation in gasoline and like petroleum products.

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