United States Patent

Rosenwald

[15] **3,653,853**

[45] **Apr. 4, 1972**

[54] SYNERGISTIC ANTI-ICING COMPOSITION

[72] Inventor: **Robert H. Rosenwald,** Western Springs, Ill.

[73] Assignee: **Universal Oil Products Company,** Des Plaines, Ill.

[22] Filed: **Nov. 14, 1969**

[21] Appl. No.: **877,011**

[52] **U.S. Cl.** **44/66,** 44/71, 44/77, 44/DIG. 1

[51] **Int. Cl.** **C10l 1/18,** C10l 1/22

[58] **Field of Search** ..... 44/56 D, 66, 71, 77

[56] **References Cited**

UNITED STATES PATENTS

2,936,223	5/1960	Lovett et al.	44/77 X
2,974,022	3/1961	Lindstrom et al.	44/56 D
3,402,030	9/1968	Morrison	44/56 D
3,416,902	12/1968	Anderson et al.	44/56 D
3,436,195	4/1969	Rosenwald et al.	44/77 X

Primary Examiner—Daniel E. Wyman
Assistant Examiner—W. J. Shine
Attorney—James R. Hoatson, Jr. and Bernard L. Kramer

[57] **ABSTRACT**

Synergistic anti-icing composition of (1) polyhydroxy alcohol and (2) C_8–C_{20} amide of N—C_8–C_{20}-alkylene-diamine.

8 Claims, No Drawings

SYNERGISTIC ANTI-ICING COMPOSITION

BACKGROUND OF THE INVENTION

A serious problem in the operation of automobiles is stalling of the engine due to the formation of ice in the carburetor throttle body and on the throttle plate. As is well known, at temperatures ranging from about 30° to about 60° F. and at periods of relatively high humidities, such stalling has been encountered under idling or low load conditions. This is caused by the air-borne moisture undergoing freezing due to the refrigerating effect encountered in the normal fuel vaporization within the carburetor. The ice formed on the throttle plate and adjacent carburetor walls restricts the narrow air opening and causes engine stalling.

The icing problem is of increasing importance because of the design of newer automobiles. For example, present cars do not have a manual throttle and therefore the operator of the car is no longer able to increase the idle speed during the warm up period to prevent such stalling. Furthermore, the increasing use of automatic transmissions adds to this problem because the idle speed must be kept low to avoid creeping and, accordingly, the idle speed is not sufficiently fast to avoid stalling due to icing. Still another development which appears to add to this problem is the increased volatility of commercial gasolines, because more frequent stalling is encountered with the more volatile fuels.

Various methods have been proposed to eliminate the stalling of automobile engines, including the use of additives. In one method, an alcohol is used but this has the objection of requiring large concentrations of the alcohol in order to obtain reasonably satisfactory anti-icing. In another method various salts have been proposed.

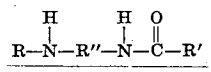
DESCRIPTION OF THE INVENTION

It now has been found that the use of certain amides in admixture with polyhydroxy alcohols produces a synergistic composition of improved potency for use as an anti-icing additive.

In one embodiment the present invention relates to a synergistic anti-icing composition of (1) polyhydroxy alcohol and (2) a C_8–C_{20} amide of N—C_8–C_{20}-alkylenediamine.

In another embodiment the present invention relates to gasoline containing an anti-icing concentration of the synergistic composition herein set forth.

As hereinbefore set forth, one component of the synergistic composition is a particular amide. The amide may be illustrated by the following formula:



where R and R′ each is an aliphatic group of eight to 20 carbon atoms and R″ is alkylene of two to six carbon atoms.

Referring to the above formula, R may be an alkyl group and thus is selected from octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, nonadecyl and eicosyl. In another embodiment, R is an alkenyl group and is selected from octenyl, nonenyl, decenyl, undecenyl, dodecenyl, tridecenyl, tetradecenyl, pentadecenyl, hexadecenyl, heptadecenyl, octadecenyl, nonadecenyl and eicosenyl.

Again referring to the above formula, R′ may be an alkyl group and will be selected from those hereinbefore set forth. Similarly, R′ may be an alkenyl group and likewise will be selected from those hereinbefore set forth.

Conveniently the R and R′ groups in the above formula are derived from fatty alcohols or fatty acids. In this embodiment, R and R′ are independently selected from capryl, n-decanyl derived from capric alcohol, lauryl, myristyl, palmityl, stearyl and arachidyl as representative of the alkyl radicals. When R and R′ are unsaturated radicals, they are independently selected from decylenyl, dodecylenyl, palmitoleyl, oleyl, ricinoleyl, petroselinyl, vaccenyl, linoleyl, linolenyl,

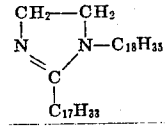
eleostearyl, licanyl, parinaryl, gadoleyl and arachidonyl. A number of mixed N-alkyl-1,3-diaminopropanes containing from eight to 20 carbon atoms are available commercially and may be used in preparing the amide for use in the present invention. One such mixture is available commercially as "Duomeen T" in which the alkyl groups are said to contain from about 12 to 20 carbon atoms per group and mostly 16 to 18 carbon atoms per group.

Referring again to the above formula, R″ is alkylene of two to six carbon atoms and thus is selected from ethylene, propylene, butylene, pentylene and hexylene. When R″ contains three or more carbon atoms, the alkylene group may be straight or branched chain. However, the nitrogen atoms should be attached to different carbon atoms of the alkylene group.

A particularly preferred compound for this component of the synergistic composition is oleoyl amide of N-oleyl-1,3-diaminopropane. It will be noted that the acyl group is named as an alkenoyl radical and, when R′ is a saturated radical, the acyl group may be named as alkanoyl. Other illustrative but not limiting compounds for this component of the composition include capryloyl amide of N-capryl-1,3-diaminopropane, lauroyl amide of N-lauryl-1,3-diaminopropane, myristoyl amide of N-myristyl-1,3-diaminopropane, palmitoleoyl amide of N-palmitolyl-1,3-diaminopropane, linoleoyl amide of N-linoleyl-1,3-diaminopropane, linolenoyl amide of N-linolenyl-1,3-diaminopropane, etc. In the preferred compounds the R and R′ radicals are the same. However, it is understood that these may be different but each will be selected from those hereinbefore set forth.

In still another embodiment R″ contains two carbon atoms and this moiety of the amide will comprise 1,2-diaminoethane. In still another embodiment the R″ moiety may comprise a butene, pentene or hexene group. As hereinbefore set forth the nitrogen atoms will be attached to different carbon atoms and R and R′ will be selected from those hereinbefore specifically set forth. However, it is understood that the different amides are not necessarily equivalent and the particular amide will be selected with reference to the particular polyhydroxy alcohol and also with the particular gasoline in which the mixture is to be used.

The amide may be obtained from any suitable source or prepared in any suitable manner. For example, the oleoyl amide and N-oleyl-1,3-diaminopropane is available commercially under the tradename of RD 3314. It may be prepared by reacting oleyl amine with acrylonitrile, then reduced to the diamine and subsequently reacted with oleic acid to form the amide. The commercial material is available as a paste at room temperature and may be contaminated with cyclic compound of the following formula:



As another advantage to the present invention, the impure material may be used satisfactorily and thus avoids the added expense of removing the cyclic compounds.

As hereinbefore set forth, the particular amide is used in admixture with a polyhydroxy alcohol. A preferred polyhydroxy alcohol is glycerol (glycerin or glycerine). Other polyhydroxy alcohols include ethyleneglycol, propyleneglycol, butyleneglycol, pentyleneglycol, hexyleneglycol including 2,4-dihydroxy-2-methylpentane, 1,6-dihydroxyhexane and 2,6-dihydroxyhexane, heptyleneglycol, octyleneglycol, etc., diethyleneglycol, dipropyleneglycol, dibutyleneglycol, tributyleneglycol, etc., dihydroxycyclohexane as, for example, 1,4-dihydroxycyclohexane, 1,3-dihydroxycyclohexane, 1,2-dihydroxycyclohexane, etc., 1,2,3-butanetriol, pentanetriol, hexanetriol, heptanetriol, erthrytol, etc. In a preferred em-

bodiment the polyhydroxy alcohol is a dihydroxy alcohol containing from two to eight carbon atoms and may be of straight or branched chain. However, it is understood that the polyhydroxy alcohol may contain three or four hydroxy groups, as well as being of aliphatic or cyclic configuration.

In another embodiment, the polyhydroxy alcohol is an alkylene oxide addition product of a polyol. For example, a commercially available polyhydroxy alcohol is marketed as "-NIAX" polyol LT 550 and is believed to be the ethylene oxide addition product to hexane triol. This material is said to have a hydroxyl number of about 500, an acid number of 0.2 maximum and a specific gravity 20/20 of 1.0910.

When desired, a mixture of polyhydroxy alcohols is employed. Illustrative mixtures include a mixture comprising from 85 to 95 percent by weight of hexylene glycol and 5 to 15 percent by weight of glycerol, a mixture of 75 to 95 percent by weight of ethylene glycol and 5 to 25 percent by weight of erythritol, etc. Also when desired, a mixture of the particularly substituted amides may be employed. These will be selected from those hereinbefore set forth.

The mixture of amide and polyhydroxy alcohol will be used in any suitable proportion, which will be determined by the solubility of the polyhydroxy alcohol in the amide. Accordingly, the polyhydroxy alcohol may be used in a concentration of from about 0.5 to about 5 weight percent or more of the amide but, as mentioned above, preferably is to the extent that the polyhydroxy alcohol is soluble in the amide. It may be considered as surprising that such a small amount of alcohol has the comparatively large effect in improving the anti-icing properties of the amide.

The amount of total anti-icing composition to be added to the gasoline will be sufficient to effect improved de-icing. For economic reasons, the concentration should be as low as practicable and may range from 0.0001 to 0.05 percent by weight and preferably within the range of from about 0.002 percent to about 0.01 percent by weight of the fuel, based on the mixed amide and polyhydroxy alcohol exclusive of solvent when employed. While each of the amide and the polyhydroxy alcohol may be added separately to the fuel, it generally is preferred to prepare a composition of the amide and polyhydroxy alcohol in the proper concentrations and to add this composition to the fuel in the desired amount. When desired, the mixture of amide and polyhydroxy alcohol may be prepared as a solution in a suitable solvent such as a paraffinic, aromatic and/or naphthenic naphtha or gasoline. When desired, the solvent may comprise an aromatic or paraffinic hydrocarbon, including benzene, toluene, xylene, ethylbenzene, etc., pentane, hexane, heptane, octane, etc. When a solvent is used, the amide and hexylene glycol will comprise from about 10 percent to about 90 percent and preferably from about 25 to about 75 percent of the solution.

The synergistic composition of the present invention may be used in any gasoline. Commercial gasolines generally comprise a mixture of two or more of cracked gasoline, hydrocracked gasoline, reformed gasoline, alkylate, isoparaffins, aromatics, etc. and in some cases, may contain straight run gasoline, coker distillate, etc. It is understood that the synergistic composition of the present invention may be used along with other additives incorporated in gasoline. These include antioxidant, metal deactivator, tetra-alkyl lead, detergent, dye, etc. When desired, one or more of these additional additives may be admixed with the composition of the present invention and marketed and used in this manner.

The following examples are introduced to illustrate further the novelty and utility of the present invention but not with the intention of unduly limiting the same.

EXAMPLE I

The synergistic composition of this example comprises a mixture of the oleyl amide of N-oleyl-1,3-diaminopropane and glycerol.

The anti-icing properties were determined in a carburetor icing demonstrating apparatus consisting of a vacuum pump equipped so that cool moisture-saturated air from an ice tower is drawn through a simulated carburetor. The gasoline sample passes from a fuel reservoir through a flow meter into the carburetor at a rate of 1.4 lb./hr. The air from the ice tower is passed at a flow rate of 14.4 lb./hr. at a temperature of 40° F. The manifold vacuum is 9.5 in Hg at the start and 12.5 in Hg at the end of the test. Evaporation of the gasoline in the carburetor further cools the cold moist air, with resulting ice formation on the throttle plate. The time in seconds is measured until a drop of 3 in Hg vacuum occurs, which indicates stalling conditions.

The fuel used in this example is a commercial gasoline which, without anti-icing additive, reached stalling conditions within 7.4 seconds.

When the oleyl amide of N-oleyl-1,3-diaminopropane was added to the gasoline in a concentration of 25 p.p.m. (parts per million), the gasoline reached stalling conditions within 14.8 seconds. When a polyhydroxy alcohol in concentrations up to 1 p.p.m. is added to the gasoline, there is substantially no change in stalling conditions as compared to the blank or control sample of the gasoline.

In contrast to the above, when the oleoyl amide of N-oleyl-1,3-diaminopropane at a concentration of 25 parts per million was used in admixture with 0.25 p.p.m. of glycerol, the stalling conditions were increased to 18 seconds. It will be noted that the glycerol was used in a concentration of 1 percent by weight of the amide and, as hereinbefore set forth, it is surprising that such a small amount of alcohol was effective in increasing the stalling time more than 20 percent.

EXAMPLE II

Another run was made in the same manner as described in Example I except that the polyhydroxy alcohol in this example was ethylene glycol and it was used in a concentration of 0.5 p.p.m. which is 2 percent by weight of the oleoyl amide of N-oleyl-1,3-diaminopropane. This small amount of ethylene glycol in admixture with the amide increased the stalling time from 14.8 to 16.7 seconds, which is an increase of more than 10 percent.

EXAMPLE III

The synergistic mixture of this example is the lauroyl amide of N-lauryl-1,2-diaminoethane and hexylene glycol, the latter being in a concentration of 2 percent by weight of the former. This mixture is used in a concentration of 35 p.p.m. in commercial gasoline and serves to increase the time before the engine reaches stalling conditions.

EXAMPLE IV

The synergistic composition of this example is the stearoyl amide of N-stearyl-1,3-diaminopropane and dipropylene glycol in a concentration of 1 percent by weight of the amide. This mixture is incorporated in a commercial gasoline and serves to prevent stalling.

EXAMPLE V

The synergistic composition of this example comprises the oleoyl amide of N-stearyl-1,4-diaminobutane. The synergistic mixture contains 1.5 percent by weight of dipropylene glycol. The mixture is incorporated in a concentration of 100 p.p.m. in commercial gasoline and serves to reduce stalling.

I claim as my invention:

1. Synergistic anti-icing composition of (1) polyhydroxy aliphatic alcohol containing from two to eight carbon atoms and from two to four hydroxy groups and (2) C_8–C_{20} aliphatic amide of N—C_8–C_{20} alkylenediamine, said polyhydroxy aliphatic alcohol being present in a concentration within the range of from about 0.5 percent to about 5 percent by weight of said amide.

2. The composition of claim 1 in which said polyhydroxy aliphatic alcohol is glycerol.

3. The composition of claim 1 in which said polyhydroxy aliphatic alcohol is ethylene glycol.

4. The composition of claim 1 in which said polyhydroxy aliphatic alcohol is hexylene glycol.

5. The composition of claim 1 in which said amide is the oleoyl amide of N-oleyl-1,3-diaminopropane.

6. The composition of claim 1 in which said amide is the stearoyl amide of N-stearyl-1,3-diaminopropane.

7. The composition of claim 1 incorporated in gasoline to prevent stalling due to icing of an internal combustion engine.

8. The composition of claim 7 incorporated in a concentration of from about 0.0001 percent to about 0.05 percent by weight of the gasoline.

* * * * *