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Improved fuel composition for multi-port fuel injection systems, and use thereof.

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Description

BACKGROUND OF THE INVENTION

This invention is directed to an anti-fouling fuel composition and to a method for using same. More specifically, the present invention is directed at a fuel composition having particular applicability in minimizing and/or preventing injector fouling in gasoline engines equipped with electronically controlled multiport fuel injectors.

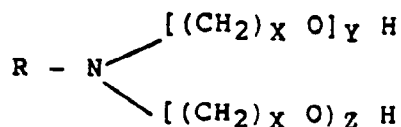
Over the past several years, improvements have been made in the performance of internal combustion engines. One of the most significant improvements which has been made has been the widespread use of fuel injection to improve the performance and fuel economy of internal combustion engines. While carburetor-equipped internal combustion engines admix the air and fuel for distribution through a manifold to all of the cylinders, in a fuel injected engine the fuel is injected into the manifold close to the intake valve of each cylinder for combustion. Fuel injection systems are of two basic types, mechanically controlled and electronically controlled. The early fuel injected engines were controlled mechanically, i.e., the operation of each injector was controlled by pressure. Recently, however, the use of electronically controlled fuel injection engines has become increasingly widespread. In an electronically controlled fuel injection system sensors disposed in the exhaust are employed to maintain the air to fuel ratio within narrow limits. Electronically controlled fuel injection systems offer the same performance and fuel economy benefits that would be achieved with mechanically controlled fuel injection systems and also serve to more closely regulate fuel-air mixtures to thereby enable the catalytic converter to oxidize carbon monoxide and hydrocarbons to carbon dioxide and simultaneously to reduce nitrogen oxides and thus meet emissions control legislation. Such legislation imposing as it did strict control of exhaust pollutants ultimately led to the development and widespread application of new technologies such as electronic fuel injection.

It has been found that the electronically controlled fuel injector systems have small port openings which are prone to fouling by deposits. These deposits are believed to occur, at least in part, by gasoline and oil vapor, which is present in close proximity to the injector tip, becoming baked onto the hot surfaces of the injector pintle and on the surfaces of the annulus surrounding the pintle when the engine is shut off. These deposits restrict the fuel flow to that particular cylinder. This, in turn, causes a sensor disposed in the exhaust to detect a higher than desired oxygen to fuel ratio. The sensor will attempt to correct this condition by increasing the amount of fuel injected into all of the cylinders. This, in turn, will result in a richer than desired fuel to air ratio in the exhaust. The sensor then will attempt to correct this by decreasing the amount of fuel injected into each cylinder. This cyclical adjustment of the fuel to air ratio ranging between too lean a mixture and too rich a mixture can at times result in poor operating performance of the vehicle. In addition, close tolerances in this new type of injector and concurrently higher underhood temperature also tend to enhance deposit formation resulting in poor vehicle driveability and exceeding exhaust pollutant levels set by emissions control legislation.

It has been found that conventional gasoline detergents, which have proven effective in preventing and/or eliminating carburetor deposits are not particularly effective in removing and/or preventing deposit build-up that may occur in electronically controlled fuel injection systems. Presently available methods for removing deposits from fuel injector orifices typically comprise either mechanically cleaning the injectors or the addition to the fuel of relatively large quantities of particular additives. Mechanical cleaning, which may involve either the complete removal of the injector for manual deposit removal or the use of polar solvents for flushing the deposits free, is not desired because of the relatively high cost and inconvenience. Currently available additives are not particularly desirable because product recommendations indicate they must be used at relatively high concentrations, i.e. about one to about two tons (1016 to 2032 kg) per thousand barrels (159 m³) of fuel.

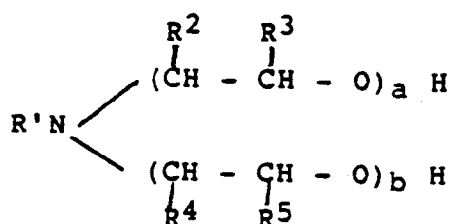
To be useful commercially a gasoline additive for reducing and/or preventing injector port fouling must be effective at low concentration, must not significantly affect the combustion characteristics of the fuel and must not foul the catalytic converter catalyst.

Additives have been added to gasoline to improve certain properties of the fuel. U.S. Patent US-A-3,115,400 discloses the use of compounds of the structure



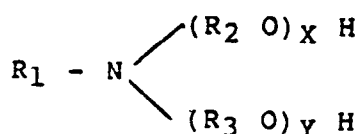
where R is a C₆-C₂₂ aliphatic hydrocarbon radical, X is an integer from 2 to 4, Y is an integer of at least 1, and Z is an integer of at least 1, for use in motor fuel to prevent or reduce carburetor icing.

U.S. Patent US-A-4,409,000 discloses combination of hydroxy amines and hydrocarbon-soluble carboxylic dispersants as engine and carburetor detergents for normally liquid fuels. Among the hydroxy amines disclosed are compounds of the formula



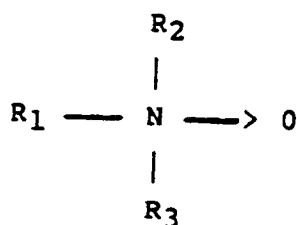
where R' may be an alkyl radical containing from 8 to 30 carbon atoms, where R², R³, R⁴ and R⁵ each may be hydrogen and where a and b may be integers from 1 to 75.

U.S. Patent US-A-4,231,883 discloses the use of a compound of the formula



where R₁ is a C₁₂-C₃₆ aliphatic hydrocarbon group, R₂ and R₃ are divalent hydrocarbon radicals containing 2-4 carbon atoms and X and Y are integers from 1-4, for friction reduction in lube oils. Preferred compounds comprise N, N-bis (2-hydroxyethyl) hydrocarbylamines.

U.S. Patent US-A-3,387,953 is directed at the use of organo-substituted nitrogen oxides, particularly amine oxides for rust inhibition and as anti-icing agents in gasoline. Several representative formulas for amine oxides are given including the following:



where: R₁ is C₆-C₂₄ alkyl, aryl cycloaliphatic, heterocyclic, substituted alkyl or substituted aryl; and R₂ and R₃ are the same or different and are C₁-C₂₄ alkyl, aryl, substituted alkyl or aryl, cycloaliphatic or heterocyclic. R₂ and R₃ preferably comprise hydroxy substituted alkyls. These compounds typically are added to gasoline in a concentration within the range of 2.0 to 100 pounds (0.91 to 45.4 kg) of amine oxide per 1,000 barrels (159 m³) of gasoline (ptb). Among the most preferred additives is bis(2-hydroxy ethyl) cocoamine oxide.

U.S. Patent US-A-3,594,139 is directed at a rust-inhibitor concentrate that can be blended with gasoline year-round. This patent also discloses the use of amine oxides having the aforementioned formula for use as gasoline additives for rust prevention. This patent also discloses a particularly preferred concentrate comprising bis(2-hydroxy ethyl) cocoamine oxide.

The amine oxides described above have been typically used to inhibit rust and carburetor icing, although these amines also were known as carburetor detergents.

It has been discovered that use of hydroxy substituted amine oxides can result in additive losses because of high water solubility and adsorption on polar surfaces.

Accordingly, it would be desirable to provide an additive package for gasoline which will be effective in reducing and/or eliminating fouling without appreciable additive losses.

It also would be desirable to provide an additive package having a demulsifying agent which is effective in the presence of both neutral and basic waters.

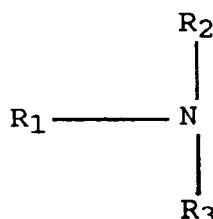
Accordingly, it would be desirable to provide a gasoline additive package which is relatively inexpensive and effective at low concentrations to reduce and/or eliminate injector fouling.

It also would be desirable to provide a gasoline additive package which is non-corrosive, non-deleterious to the catalyst, and does not affect the combustion characteristics of the fuel.

It also would be desirable to provide a gasoline additive package which could be easily added to the finished gasoline at any point during the storage and/or distribution system.

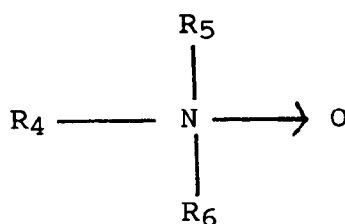
The present invention provides a fuel composition for an internal combustion engine, said fuel composition comprising:

- (a) fuel (e.g. gasoline);
- (b) an antifouling agent having the formula



wherein: R₁ is C₆ to C₂₄ alkyl, aryl, cycloaliphatic, heterocyclic, substituted alkyl or substituted aryl; R₂ and R₃ independently are C₁ to C₂₄ substituted alkyl, aryl, cycloaliphatic or heterocyclic; and

(c) an amine oxide antifouling agent having the following structural formula:



where R₄ is selected from C₆₋₂₄ alkyl, aryl, cycloaliphatic, heterocyclic, substituted alkyl, substituted aryl; R₅ and R₆ are independently selected from C₁₋₂₄ alkyl, aryl, substituted alkyl, substituted aryl, cycloaliphatic, heterocyclic and mixtures thereof.

R₁ may be C₆ to C₂₀ alkyl (e.g., C₆₋₁₈ substituents derived from fatty acid), or alkylated aryl; and, R₂ and R₃ independently may be hydroxy-substituted C₁ to C₁₂ alkyl.

The anti-fouling agent (b) may be selected from the group consisting of bis(2-hydroxy ethyl) cocoamine, bis(2-hydroxy ethyl) tallow amine, bis(2-hydroxy ethyl) stearylamine, bis(2-hydroxy ethyl) oleyl amine and mixtures thereof.

The concentration of the anti-fouling agent (b) in the fuel may be in the range of from 2 to 200 wppm (e.g. from 16 to 100 wppm).

The amine oxide (c) may be selected from bis(2-hydroxy ethyl) cocoamine oxide; bis(2-hydroxy ethyl) stearylamine oxide; dimethylcocoamine oxide; dimethyl hydrogenated tallow amine oxide; dimethylhexadecylamine oxide, and mixtures thereof.

The concentration of the amine oxide may be in the range of from 2 to 80 ppm (e.g., from 4 to 40 ppm).

The fuel composition may comprise a demulsifying agent (d) selected from the group consisting of:

- (i) acylated polyglycols;
 - (ii) alkylarylsulfonates, polyglycols, oxygenated alkylphenol-formaldehyde resins;
 - (iii) oxyalkylated alkylphenol-formaldehyde resins and polyglycols;
 - (iv) alkylphenol-formaldehyde resins and polyglycols;
 - (v) oxyalkylated alkylphenol-formaldehyde resins;
- and mixtures thereof.

The concentration of the demulsifying agent (d) may be in the range of from 0.1 to 20 wppm (e.g. from 1.0 to 8.0 wppm).

The invention also provides a method of making a fuel composition as described, which in one case, case (a), comprises adding to a fuel (e.g., gasoline) an additive concentrate comprising :

- (i) from 5 to 60 wt.% bis(2-hydroxy ethyl) cocoamine;

(ii) from 0.25 to 20 wt. % of a demulsifying agent selected from : acylated polyglycols, or alkylarylsulfonates; polyglycols; oxyalkylated alkylphenol-formaldehyde resins; or oxyalkalated alkylphenol-formaldehyde resins and polyglycols; or oxyalkylated alkylphenol-formaldehyde resins; or mixtures thereof; and
 (iii) from 40 to 95 wt. % solvent; and in another case, case (b), comprises adding to the fuel an additive

concentrate as for case (a) and, in addition, from 1 to 15 wt. % bis(2-hydroxy ethyl) cocoamine oxide.

The method may comprise adding to a fuel (e.g. gasoline) a concentrate comprising :

- . from 8 to 32 wt. % of an amine anti-fouling agent (b);
- . from 2 to 8 wt. % of an amine oxide antifouling agent (c); and
- . from 40 to 95 wt. % solvent.

The concentrate may comprise from 1 to 4 wt. % of a demulsifying agent as specified.

The invention further provides a method of minimizing and/or preventing fuel-injector fouling in a multiport fuel-injected engine which comprises delivering to the fuel-injection system of the engine either a fuel composition as described herein or a fuel composition made by the method described herein.

The fuel-injection system may be electronically controlled.

The fuel-injection system may have sensor means disposed in the engine exhaust system to regulate the air to fuel ratio of the fuel and air supplied to the engine.

A fuel composition according to the invention may comprise a fuel, such as gasoline, and :

A. from 2 to 200 ppm bis(2-hydroxy ethyl) cocoamine; and

B. from 0.1 to 20 ppm of a demulsifying agent selected from the group consisting of :

- i. acylated polyglycols;
 - ii. alkyl aryl sulfonates, polyglycols, oxyalkylated alkylphenol-formaldehyde resins;
 - iii. oxyalkylated alkylphenol-formaldehyde resins and polyglycols; and,
 - iv. oxyalkylated alkylphenol-formaldehyde resins;
- and mixtures thereof.

A fuel composition of the invention more preferably comprises :

A. from 20 to 120 ppm bis(2-hydroxy ethyl) cocoamine; and

B. from 1 to 12 ppm of a demulsifying agent selected from the group consisting of :

- i. acylated polyglycols;
 - ii. alkyl aryl sulfonates, polyglycols, oxyalkylated alkylphenol-formaldehyde resins;
 - iii. oxyalkylated alkylphenol-formaldehyde resins and polyglycols; and
 - iv. oxyalkylated alkylphenol-formaldehyde resins;
- and mixtures thereof.

The preferred fuel composition also may comprise from 4 to 40 ppm of the bis(2-hydroxy ethyl) cocoamine oxide.

A preferred fuel additive concentrate for internal combustion engines comprises :

A. from 5 to 60 wt. % bis(2-hydroxy ethyl) cocoamine;

B. from 0.25 to 10 wt. % of a demulsifying agent selected from the group consisting of :

- i. acylated polyglycols;
 - ii. alkyl aryl sulfonates, polyglycols, oxyalkylated alkylphenol-formaldehyde resins;
 - iii. oxyalkylated alkylphenol-formaldehyde resins and polyglycols; and
 - iv. oxyalkylated alkylphenol-formaldehyde resins;
- and mixtures thereof.

C. from 40 to 95 wt. % solvent.

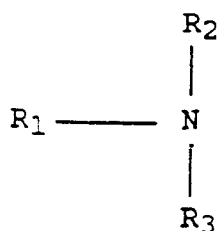
The fuel additive concentrate also may comprise from 1 to 15 wt. % of the bis(2-hydroxy ethyl) cocoamine oxide.

The solvent preferably comprises an alkyl aromatic hydrocarbon solvent, such as xylene, and a C₄+ alcohol, preferably a C₄-C₁₂ alcohol, more preferably a C₈ alcohol and most preferably a C₈ oxo alcohol. Where the ratio of the concentration of water relative to amine oxide exceeds about 0.05, a highly water and hydrocarbon soluble alcohol, preferably isopropanol, also should be added.

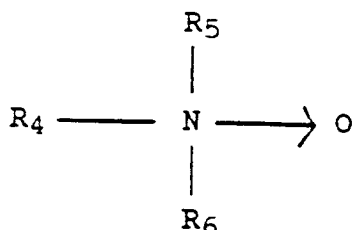
DETAILED DESCRIPTION OF THE INVENTION

The present invention is directed at a fuel composition, a fuel (e.g. gasoline) additive package, and a method for delivering the fuel composition to a fuel injection system in which the composition has been found to be particularly effective in reducing and/or eliminating injection fouling. The present invention is directed at a fuel comprising :

- (a) fuel (e.g. gasoline);
- (b) an antifouling agent having the formula



wherein: R_1 is C_6 to C_{24} alkyl, aryl, cycloaliphatic, heterocyclic, substituted alkyl or substituted aryl; R_2 and R_3 independently are C_1 to C_{24} substituted alkyl, aryl, cycloaliphatic or heterocyclic; and (c) an amine oxide antifouling agent having the following structural formula:



where R_4 is selected from C_{6-24} alkyl, aryl, cycloaliphatic, heterocyclic, substituted alkyl, substituted aryl; R_5 and R_6 are independently selected from C_{1-24} alkyl, aryl, substituted alkyl, substituted aryl, cycloaliphatic, heterocyclic and mixtures thereof.

The amine anti-fouling agent (b) preferably is selected from the group consisting of bis(2-hydroxy ethyl) cocoamine, bis(2-hydroxy ethyl) stearylamine, bis(2-hydroxy ethyl) oleyl amine and mixtures thereof. These additives are prepared in accordance with known techniques, such as disclosed in US-A-2,541,678. A particularly preferred anti-fouling agent is bis(2-hydroxy ethyl) cocoamine.

The amine oxide anti-fouling agents (c) also have been found to be effective as anti-fouling agents. While these compounds may be extracted to varying degrees into any water present, these compounds also provide anti-rust properties to the fuels.

The use of the amine oxide compounds (c) in combination with the previously described amines (b) may provide an effective anti-fouling composition also providing anti-rust properties. These amine oxides may be prepared by well-known techniques, such as disclosed in US-A-3,387,953.

In fuel compositions containing the said amine and amine oxide components in combination as the anti-fouling agent, the concentration of the amine typically will range between 2 and 200 ppm, preferably between 16 and 100 ppm, while the amine oxide concentration will range between 2 and 80 ppm, preferably between 4 and 40 ppm.

The amine oxide typically has water present from the manufacturing process. While it is possible to remove most of the water, removal of the water to relatively low levels, e.g. a ratio of 0.02 to 0.04 of water to amine oxide, adds complexity to the manufacturing process. Therefore, the amine oxide is commercially available as a solution comprising water and a solvent, which typically is isopropyl alcohol. It has been found that when a concentrate comprising the above amine oxide solution and a solvent containing demulsifiers was admixed with gasoline and terminal tank water bottoms a three phase system resulted, two organic phases and a water phase.

Formation of two organic layers is not desirable, since this was found to result in uneven distribution of the amine oxide between layers. In addition, the second organic layer, which has a much higher amine oxide concentration, tends to adhere to surfaces, resulting in additive loss and potential contamination of subsequent hydrocarbon products that might contact these surfaces. It has been found that replacement of a portion of the isopropanol by a higher alcohol, preferably a C_4 - C_{12} alcohol, more preferably a C_8 oxo alcohol, decreases the likelihood of forming a two organic layer system. While the admixture of the amine with the amine oxide may also decrease the formation of two organic phases, it is preferred that the solvent comprise a C_4 - C_{12} alcohol as described above to further decrease the possibility of two organic phase formation.

A concentrate utilizing both the amine and amine oxide typically also comprises 40 to 95 wt.% solvent. A

preferred composition range is as follows:

	<u>Component</u>	<u>Wt. % Range</u>
5	Amine	8-32
	Amine Oxide	2-8
10	Solvent	
	Xylene	30-80
	C₄-C₁₂ alcohol	2-20
	Isopropanol	2-16
15	Water	0.2-1.5
	Demulsifier	1-4

The following Comparative Examples and Examples demonstrate the utility of the anti-fouling agent in reducing and/or eliminating fuel injector fouling. In the following Comparative Examples and Examples, the octane rating of the fuel utilized is the posted octane rating which is defined as:

$$\frac{\text{Research Octane} + \text{Motor Octane}}{2}$$

25 COMPARATIVE EXAMPLE I

In this test three 1985 Oldsmobile 98's having electronically controlled, fuel injected, 3.8 liter, six cylinder engines were driven on a commercial, unleaded, 87 octane reference fuel having a detergent concentration of about 32 ppm by weight of the fuel for approximately 3500 miles (5632.6 km) under the following driving cycle: 0.5 hours city-type driving, 0.5 hour engine off, 0.5 hour highway driving, 0.5 hour engine off. Driveability on all four vehicles became poor to very poor. The vehicles then were driven for 300 mile (482.8 km) with a commercial premium grade 92 octane unleaded fuel containing 2.5 times the detergent used in the above reference fuel. Driveability remained unchanged. The data in Table I below show that there was still a marked reduction in fuel flow indicating that a high level of deposit was unaffected by the detergent even at the high treat rate. The percent fuel flow reduction was determined by measuring the volume of a mineral spirit that flowed through the injector under predetermined standardized conditions, including fuel pressure, pulse width and duty cycle. The percent reduction is calculated using the formula:

$$\% \text{ Reduction} = \frac{V_{\text{clean}} - V_{\text{dirty}}}{V_{\text{clean}}} \times 100\%$$

where V_{clean} and V_{dirty} are the measured volumes of mineral spirit passed through the clean and dirty fuel injectors.

TABLE I

	<u>% FLOW REDUCTION THROUGH INJECTOR PORTS</u>						
	Cyl #	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
50	Car A	11	12	35	30	7	10
	Car B	7	9	12	38	9	14
	Car C	22	11	28	4	11	10
55	Typical New Injectors	2	2	0	0	2	1

From Table I it can be seen that this conventional, known carburetor detergent was ineffective in removing deposits from injector ports and in fact permitted deposits to form.

COMPARATIVE EXAMPLE II

A 1985 Chrysler LeBaron equipped with a 2.2 liter turbocharged engine having electronically controlled fuel injection was driven for 2858 miles (4599.4 km) on a mileage accumulation dynamometer using a typical regular grade, 87 octane, unleaded, detergent-free gasoline. The driving was based on repetition of the following cycle: 30 minutes city driving, 30 minutes engine off, 30 minutes highway driving, 30 minutes engine off. The driveability became very poor as typified by rough idle, severe hesitation, backfire and roughness during acceleration. The hydrocarbon emissions measured before the catalytic converter were 804 ppm at engine idle and 725 ppm at 2500 rpm. The injector fouling also was measured using a pressure differential test. In this test the fuel rail is pressurized to 49 psig (337.86 kPa) and an injector is pulsed for 0.5 seconds. The difference in the pressure drop between the injectors is a rough measure of the degree to which the injectors are obstructed, i.e. the greater the numerical difference between the highest and lowest values, the greater the injector fouling. A summary of the results at 2585 miles (4599.4 km) on the detergent-free fuel are set forth in Table II as the measurements at 0 miles (0 km) after HECA addition.

EXAMPLE I

Following the test set forth in Comparative Example II, the vehicle was refueled with the same fuel except that the fuel also contained 80 ppm of bis(2-hydroxy ethyl) cocoamine (HECA). The vehicle then was driven on the following cycle: 15 minutes city driving, 30 minutes highway driving, 15 minutes city driving, 2 hours engine off. This test continued until 308 miles (495.7 km) were accumulated on the vehicle. At the end of this test period the driveability was very good. The hydrocarbon emissions at idle before the catalytic converter were reduced to 65 ppm and to 16 ppm at 2500 rpm. The emissions before the catalytic converter at idle and at 2500, rpm and the pressure differentials measured at various intervals during the clean-up driving are summarized in Table II. The injector flow reduction measurements are summarized in Table III.

From the data of Example I and Tables II and III, it can be seen that the use of a relatively low concentration of HECA was able to produce a significant improvement in driveability. The idle emissions were significantly reduced and the pressure differential and percent flow reduction of the flow injectors were returned to "as new" conditions after a relatively few miles (or km) of driving.

TABLE II

km (MILES) AFTER ADDITION OF HECA	EMISSIONS BEFORE CATALYTIC CONVERTER		2500 RPM		CYL No.	Δ P LEAKDOWN, kPa (PSI)			
	HC, PPM	CO%	HC, PPM	CO%		1	2	3	4
0 (0)	804	0.68	725	0.31		117 (17)	124 (18)	137.9 (20)	144.8 (21)
117.5 (73)	813	1.78	188	1.99		117 (17)	117 (17)	131 (19)	151.7 (22)
225.3 (140)	375	0.95	35	1.10		120.7(17.5)	127.6(18.5)	131 (19)	141.3(20.5)
352.4 (219)	95	0.70	30	0.75		131 (19)	131 (19)	134.5(19.5)	172.4 (25)
495.7 (308)	65	0.67	16	0.69		158.6 (23)	165.5 (24)	165.5 (24)	172.4 (25)

TABLE III
PORT INJECTOR FLOW REDUCTION

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	km (MILES) DRIVEN AFTER HECA ADDITION	INJECTOR NO.	1	2	3	4
10	495.7 (308)	% FLOW REDUCTION	0	0	0	1

COMPARATIVE EXAMPLE III

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A second 1985 Chrysler LeBaron equipped with a 2.2 liter turbocharged engine was driven on a mileage accumulation dynamometer using a regular grade 87 octane unleaded, detergent-free gasoline from a different batch from that of Comparative Example II and Example I. The same driving cycle was used in this Comparative Example as was used in Comparative Example I. The engine was judged to be fouled and the driveability poor after 4016 miles (6462.9 km).

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The emissions before the catalytic converter and the pressure differential across each injector were measured and are presented in Table IV as the measurements at 0 miles (0 km) after HECA addition.

EXAMPLE II

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Approximately 60 ppm of bis(2-hydroxyethyl) cocoamine was added to the fuel of Comparative Example III and the vehicle of Comparative Example III was driven on the same driving cycle described in Example I. Measurements of the emissions before the catalytic converter and the pressure differential across each injector were measured as previously described. These results are presented in Table IV. Driveability was judged to be good after only 357 miles (574.5 km) of driving. At the termination of the test the injectors were removed and flow tested as previously described. These results are presented in Table V.

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TABLE IV

km (MILES) AFTER ADDITION OF HECA	EMMISSIONS BEFORE CATALYTIC CONVERTER				CYL No.	ΔP LEAKDOWN, kPa (PSI)			
	IDLE		2500 RPM			1	2	3	4
	HC, PPM	CO%	HC, PPM	CO%					
0	148	2.17	126	2.21	75.8(11.0)	68.9(10.0)	86.2(12.5)	68.9(10.0)	
302.5(188)	190	2.6	125	3.2	103.4(15.0)	110.3(16.0)	131.0(19.0)	144.8(21.0)	
577.7 (359)	112	0.94	53	1.12	106.9(15.5)	110.3(16.0)	110.3(16.0)	113.8(16.5)	

TABLE V
PORT INJECTOR FLOW REDUCTION

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km (MILES) DRIVEN AFTER HECA ADDITION	INJECTOR NO.	1	2	3	4
577.7 (359)	% FLOW REDUCTION	5	2	1	0

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15 From a review of Tables II-V it can be seen that the use of relatively low concentrations of HECA was able to reduce the injector tip deposits in a relatively short period of time. By comparison, the use of a conventional carburetor detergent was unable to prevent a relatively rapid deposit buildup of injector tip deposits.

While the data presented above has demonstrated the utility of the anti-fouling agent in gasoline, the anti-fouling agent also may be of utility in other fuels, such as diesel fuel.

20 While the presently described anti-fouling agent may be used alone, it also may be desirable to utilize the present invention in combination with a demulsifying agent to facilitate the separation of the gasoline from any foreign substances which may be present in the distribution system, such as water and sediment.

The water, if any, typically has a pH ranging from 7 to 13. Thus, a demulsifying agent for use with the anti-fouling agent preferably should be effective over this pH range. The following Comparative Examples and Examples demonstrate the utility of various demulsifying agents.

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COMPARATIVE EXAMPLE IV

In this Comparative Example the effectiveness of various commercially available demulsifying agents were tested in a 90 wt.% fuel - 10 wt.% water system. The fuel contained an additive package comprising approximately 60 ppm HECA and 2 ppm of the various additives noted below. The effectiveness of the various demulsifying agents was determined using a modified Multiple Contact Emulsion Test. In this test 10 ml of terminal water bottoms having a pH of approximately 10 was added to separate half-pint (0.237 l) bottles. To each bottle was added 100 ml of gasoline. The bottles were capped, placed on their sides in a mechanical shaker and agitated at approximately 180 cycles per minute for ten minutes. The bottles then were placed upright and allowed to stand for 1 hour. The mixture then was rated considering the gasoline layer, the water layer and the interface using the rating scale set forth in Table VI below. After the ratings were completed, the gasoline level was sucked down to a level about 1/4 inch (0.635 cm) above the interface or emulsion layer without disturbing the interface or water layer. The withdrawn fuel was discarded and 100 ml of fresh gasoline was added to each bottle. The mixture was then shaken and the test repeated for the indicated number of times with the worst rating noted. The trade names of the commercially available additives utilized, the worst ratings of each mixture and the number of times the test was run are set forth in Table VII below.

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TABLE VIRATING SCALE FOR REPORTING EMULSION TEST RESULTS

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<u>RATING</u>	<u>DESCRIPTION OF EMULSION</u>
0	No skin or interface
10	1 Slight skin on interface - not completely continuous
15	2 Thicker skin on interface - usually completely continuous
	3 Incipient emulsion 1/8 as thick as water layer
20	4 Emulsion 1/4 as thick as water layer
	5 Emulsion 3/8 as thick as water layer
25	6 Emulsion 1/2 as thick as water layer
	7 Emulsion 5/8 as thick as water layer
	8 Emulsion 3/4 as thick as water layer
30	9 Emulsion 7/8 as thick as water layer
	10 Emulsion completely filling water layer
	Emulsion of maximum severity

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TABLE VII
EMULSION TEST RESULTS

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	<u>DEMULSIFIER DESCRIPTION</u>	<u>WORST RATING</u>	<u>NO. OF TIMES TEST RUN</u>
10	Tolad T-292	3	2
	Tolad T-347	3	2
15	Tolad T-370	4	1
	Nalco 5450	4	1
	Nalco 5451	3	4
20	Nalco 5452	3-4	4
	Nalco 5453	4	1
25	Nalco 85BD-194	4	1
	Nalco 3BD-829	4	1

30 **EXAMPLE III**

A 100 ml gasoline sample containing 60 ppm of HECA was admixed with 10 ml of the terminal water bottoms of Comparative Example IV. However, in place of the demulsifiers listed in Table VII the following demulsifiers were utilized individually: Tolad T-500; Tolad T-284; Tolad T-286; Tolad T-326; and Nalco 5455. The modified Multiple Contact Emulsion Test previously described was utilized to determine the effectiveness of each demulsifier. These test results are summarized in Table VIII below. A description of each additive is presented in Table IX below.

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TABLE VIII
EMULSION TEST RESULTS

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	<u>DEMULSIFIER DESCRIPTION</u>	<u>WORST RATING</u>	<u>NO. OF TIMES TEST RUN</u>
50	Tolad T-284	2	4
	Tolad T-286	1-2	4
	Tolad T-326	2	2
55	Tolad T-500	2	4
	Nalco 5455	2	4

TABLE IX
DEMULSIFIER DESCRIPTIONS

<u>Demulsifier</u>	<u>Description</u>
Tolad T-284*	Solution of acylated polyglycols in aromatic hydrocarbons
Tolad T-286*	Alkyl aryl sulfonates, polyglycols, oxyalkylated alkylphenol-formaldehyde resins in aromatic hydrocarbons and isopropyl alcohol
Tolad T-326*	Oxyalkylated alkylphenol-formaldehyde resins and polyglycols in aromatic naphtha
Tolad T-500*	Oxyalkylated alkylphenol-formaldehyde resins in aromatic hydrocarbons and alkanols
Nalco 5455**	Oxyalkylated alkyl phenol-formaldehyde resin in aromatic solvent
* Manufactured by Tretolite Division of Petrolite Corporation, St. Louis, Missouri. ** Manufactured by Nalco Chemical Company, Oak Brook, Illinois.	

COMPARATIVE EXAMPLE V

A 1985 Chrysler LeBaron equipped with a 2.2 liter turbocharged engine was driven on a mileage (i.e., distance) accumulation dynamometer using a regular grade 87 octane unleaded detergent-free fuel. The driving cycle to foul the injectors was 30 minutes city-type driving, 30 minutes soak, 30 minutes highway driving, 30 minutes soak. The engine was judged to be fouled after 2,300 miles (3701.4 km).

The emissions before the catalytic converter and the pressure differential across each injector were measured and are presented in Table X as the measurement at 0 miles (0 km) after additive addition.

EXAMPLE IV

This Example demonstrates the utility of using an additive comprising the combination of an amine and an amine oxide in cleaning up fouled injectors in the vehicle of Comparative Example V. The fuel utilized was similar to that of Comparative Example V, but further comprised 80 ppm of bis(2-hydroxy ethyl) cocoamine and 10 ppm of bis(2-hydroxy ethyl) cocoamine oxide. The driving cycle was the same as that of Example I. After 301 miles (484.4 km) of driving the driveability went from very poor to good.

The measurements of the emissions before the catalytic converter and the pressure differential across each injector also were measured as previously described. These results also are presented in Table X. At the termination of the test the injectors were removed and flow tested as previously described. These results are presented in Table XI.

Based on these results, it can be seen that the use of an additive comprising the amine and amine oxide in combination cleaned fouled injectors. Additional tests were run on other test vehicles. In almost all cases it

has been found that this combination of amine and amine oxide cleaned fouled injectors in a relatively short period.

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TABLE X

km (MILES) AFTER ADDITIVE ADDITION	EMISSIONS BEFORE CATALYTIC CONVERTER				CYL No.	Δ P LEAKDOWN, kPa (PSI)				MAX Δ P DIFFERENCE kPa (PSI)	Driveability
	IDLE		2500 RPM			1	2	3	4		
	HC, PPM	CO%	HC, PPM	CO%							
0 (0)	491	1.66	175	1.32		96.5 (14)	110.3 (16)	144.8 (21)	131.0 (19)	34.5 (5)	Very Poor
243.0 (151)	1,112	2.22	140	2.43		117.2 (17)	131.0 (19)	131.0 (19)	165.5 (24)	48.3 (7)	Very Poor
484.4 (301)	128	0.77	61	1.06		124.1 (18)	131.0 (19)	124.1 (18)	131.0 (19)	6.9 (1)	Good

TABLE XI
PORT INJECTOR FLOW REDUCTION

5	km (MILES) AFTER ADDITIVE ADDITION	INJECTOR NO.	1	2	3	4
10	484.4 (301)	% FLOW REDUCTION	2	1	4	2

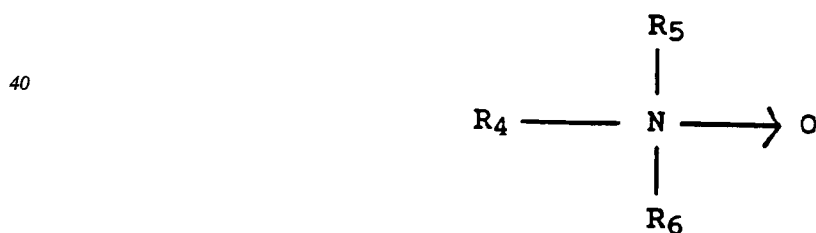
Where the presently described invention is used as a gasoline additive, the additive package may be added to the gasoline at any point after the gasoline has been refined, i.e., the additive package can be added at the refinery or in the distribution system.

Claims

1. A fuel composition for an internal combustion engine, said fuel composition comprising:
(a) fuel (e.g. gasoline);
(b) an antifouling agent having the formula



wherein: R_1 is C_6 to C_{24} alkyl, aryl, cycloaliphatic, heterocyclic, substituted alkyl or substituted aryl; R_2 and R_3 independently are C_1 to C_{24} substituted alkyl, aryl, cycloaliphatic or heterocyclic; and
(c) an amine oxide antifouling agent having the following structural formula:



where R_4 is selected from C_{6-24} alkyl, aryl, cycloaliphatic, heterocyclic, substituted alkyl, substituted aryl; R_5 and R_6 are independently selected from C_{1-24} alkyl, aryl, substituted alkyl, substituted aryl, cycloaliphatic, heterocyclic and mixtures thereof.

2. The fuel composition of claim 1 wherein R_1 is C_6 to C_{20} alkyl (e.g., C_{6-18} substituents derived from fatty acid), or alkylated aryl; and, R_2 and R_3 independently are hydroxy substituted C_1 to C_{12} alkyl.

3. The fuel composition of claim 1 or claim 2 wherein the anti-fouling agent (b) is selected from the group consisting of bis(2-hydroxy ethyl) cocoamine, bis(2-hydroxy ethyl) tallow amine, bis(2-hydroxy ethyl) stearylamine, bis(2-hydroxy ethyl) oleyl amine and mixtures thereof.

4. The fuel composition of any one of claims 1 to 3 wherein the concentration of the anti-fouling agent (b) in the fuel is in the range of from 2 to 200 wppm (e.g. from 16 to 100 wppm).

5. The composition of any one of claims 1 to 4 wherein the amine oxide (c) is selected from bis(2-hyd-

roxyethyl) cocoamine oxide; bis(2-hydroxyethyl) stearylamine oxide; dimethylcocoamine oxide; dimethyl hydrogenated tallow amine oxide; dimethylhexadecylamine oxide, and mixtures thereof.

6. The composition of any one of claims 1 to 5 wherein the concentration of the amine oxide is in the range of from 2 to 80 ppm (e.g., from 4 to 40 ppm).

7. The composition of any one of claims 1 to 6 comprising a demulsifying agent (d) selected from the group consisting of:

- (i) acylated polyglycols;
 - (ii) alkylarylsulfonates, polyglycols, oxygenated alkylphenol-formaldehyde resins;
 - (iii) oxyalkylated alkylphenol-formaldehyde resins and polyglycols;
 - (iv) alkylphenol-formaldehyde resins and polyglycols;
 - (v) oxyalkylated alkylphenol-formaldehyde resins;
- and mixtures thereof.

8. The fuel composition of claim 7 wherein the concentration of the demulsifying agent (d) is in the range of from 0.1 to 20 wppm (e.g. from 1.0 to 8.0 wppm).

9. A method of making a fuel composition according to any one of claims 1 to 5 (case (a)), or any one of claims 6 to 8 (case (b)), which in case (a), comprises adding to a fuel (e.g., gasoline) an additive concentrate comprising :

- (i) from 5 to 60 w% bis(2-hydroxyethyl) cocoamine;
- (ii) from 0.25 to 10 w% of a demulsifying agent selected from : acylated polyglycols, or alkylarylsulfonates; polyglycols; oxyalkylated alkylphenol-formaldehyde resins; or oxyalkylated alkylphenol-formaldehyde resins and polyglycols; or oxyalkylated alkylphenol-formaldehyde resins; or mixtures thereof; and
- (iii) from 40 to 95 w% solvent; and in case (b) comprises adding to the fuel an additive concentrate as for case (a) and, in addition, from 1 to 15 w% bis(2-hydroxyethyl) cocoamine oxide.

10. A method of making a fuel composition according to any one of claims 1 to 8 comprising adding to a fuel (e.g., gasoline) a concentrate comprising :

- . from 8 to 32 w% of an amine anti-fouling agent (b);
- . from 2 to 8 w% of an amine oxide anti-fouling agent (c); and
- . from 40 to 95 w% solvent.

11. A method as in claim 9 wherein the concentrate comprises from 1 to 4 w% of a demulsifying agent as specified in claim 7.

12. A method of minimizing and/or preventing fuel-injector fouling in a multiport fuel-injected engine which comprises delivering to the fuel-injection system of the engine either a fuel composition in accordance with any one of claims 1 to 8 or a fuel composition made by the method of claim 9 or claim 10.

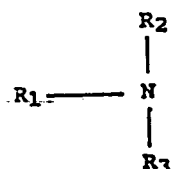
13. A method as in claim 12 wherein the fuel-injection system is electronically controlled.

14. A method as in claim 12 or claim 13 wherein the fuel-injection system has sensor means disposed in the engine exhaust system to regulate the air to fuel ratio of the fuel and air supplied to the engine.

Patentansprüche

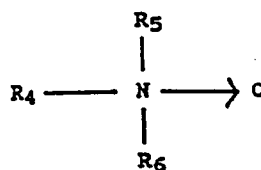
1. Treibstoffzusammensetzung für eine Verbrennungskraftmaschine, wobei diese Treibstoffzusammensetzung umfaßt:

- a) Treibstoff (z.B. Benzin);
- b) ein Antifoulingmittel mit der Formel



worin R₁ ein C₆-C₂₄ Alkyl-, Aryl-, cycloaliphatischer, heterocyclischer, substituierter Alkyl- oder substituierter Arylrest ist; R₂ und R₃ unabhängig C₁-C₂₄ substituierte Alkyl-, Aryl-, cycloaliphatische oder heterocyclische Reste sind; und

c) ein Aminoxyd-Antifoulingmittel mit der folgenden Strukturformel



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worin R_4 ausgewählt wird aus C_{6-24} Alkyl-, Aryl-, cycloaliphatischen, heterocyclischen, substituierten Alkyl-, substituierten Arylresten; R_5 und R_6 unabhängig ausgewählt werden aus C_{1-24} Alkyl-, Aryl-substituierten Alkyl-, substituierten Aryl-, cycloaliphatischen, heterocyclischen Resten und Mischungen davon.

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2. Treibstoffzusammensetzung nach Anspruch 1, worin R_1 C_6 - C_{20} Alkyl (z.B. C_{6-18} Substituenten, abgeleitet von Fettsäure) oder alkyliertes Aryl ist und R_2 und R_3 unabhängig Hydroxy-substituiertes C_1 - C_{12} Alkyl sind.

3. Treibstoffzusammensetzung nach Anspruch 1 oder 2, worin das Antifoulingmittel (b) ausgewählt wird aus der Gruppe bestehend aus Bis (2-hydroxyethyl)cocosamin, Bis (2-hydroxyethyl)-Talg-amin, Bis (2-hydroxyethyl)-stearylamin, Bis (2-hydroxyethyl)oleylamin und deren Mischungen.

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4. Treibstoffzusammensetzung nach irgendeinem der Ansprüche 1 bis 3, worin die Konzentration des Antifoulingmittels (b) in dem Treibstoff im Bereich von 2 bis 200 wppm (z.B. 16 bis 100 wppm) liegt.

5. Zusammensetzung nach irgendeinem der Ansprüche 1 bis 4, worin das Aminoxid (c) ausgewählt wird aus Bis (2-hydroxyethyl) - stearylaminoxid; Dimethylcocosaminoxid; Dimethyl-hydrierter Talg-aminoxid; Dimethylhexadecylaminoxid und deren Mischungen.

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6. Zusammensetzung nach irgendeinem der Ansprüche 1 bis 5, worin die Konzentration des Aminoxids im Bereich von 2 bis 80 ppm liegt (z.B. 4 bis 40 ppm).

7. Zusammensetzung nach irgendeinem der Ansprüche 1 bis 6, enthaltend einen Demulgator (d), der ausgewählt wird aus der Gruppe bestehend aus:

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- (i) acylierten Polyglycolen;
- (ii) Alkylarylsulfonaten, Polyglycolen, oxygenierten Alkylphenol-Formaldehyd-Harzen;
- (iii) oxalkylierten Alkylphenol-Formaldehyd-Harzen und Polyglycolen;
- (iv) Alkylphenol-Formaldehyd-Harzen und Polyglycolen;
- (v) oxalkylierten Alkylphenol-Formaldehyd-Harzen; und Mischungen davon.

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8. Treibstoffzusammensetzung nach Anspruch 7, worin die Konzentration des Demulgators (d) im Bereich von 0,1 bis 20 wppm (z.B. 1,0 bis 8,0 wppm) liegt.

9. Verfahren zur Herstellung einer Treibstoffzusammensetzung nach irgendeinem der Ansprüche 1 bis 5 (Fall (a)) oder irgendeinem der Ansprüche 6 bis 8 (Fall (b)), welches Verfahren im Falle (a) das Zusetzen eines Additivkonzentrats enthaltend:

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- (i) 5 bis 60 Gew.-% Bis(2-hydroxyethyl)cocosamin;
- (ii) 0,25 bis 10 Gew.-W% eines Demulgators, der ausgewählt wird aus: acylierten Polyglycolen oder Alkylarylsulfonaten; Polyglycolen; oxalkylierten Alkylphenol-Formaldehyd Harzen; oder oxalkylierten Alkylphenol-Formaldehyd-Harzen und Polyglycolen oder oxalkylierten Alkylphenol-Formaldehyd-Harzen; oder Mischungen davon; und
- (iii) 40 bis 95 Gew.-% Lösungsmittel; zu einem Treibstoff (z.B. Benzin) und im Falle (b) das Zusetzen eines Additivkonzentrats wie im Fall (a) und zusätzlich 1 bis 15 Gew.-% Bis(2-hydroxyethyl)cocosaminoxid zu dem Treibstoff umfaßt.

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10. Verfahren zur Herstellung einer Treibstoffzusammensetzung nach irgendeinem der Ansprüche 1 bis 8, umfassend das Zusetzen eines Konzentrats enthaltend:

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- . 8 bis 32 Gew.-W% eines Amin-Antifoulingmittels (b);
- . 2 bis 8 Gew.-% eines Aminoxid-Antifoulingmittels (c); und
- . 40 bis 95 Gew.-Wo Lösungsmittel zu einem Treibstoff (z.B. Benzin).

11. Verfahren nach Anspruch 9, worin das Konzentrat 1 bis 4 Gew.-W% eines Demulgators nach Anspruch 7 enthält.

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12. Verfahren zum Minimieren und/oder Verhindern des Verschmutzens der Treibstoffeinspritzvorrichtung in einem Motor mit Mehrlocheinspritzung, umfassend das Zuliefern einer Treibstoffzusammensetzung nach einem der Ansprüche 1 bis 8 oder einer nach dem Verfahren von Anspruch 9 oder 10 hergestellten Treibstoffzusammensetzung an das Treibstoffeinspritzsystem des Motors.

13. Verfahren nach Anspruch 12, worin das Treibstoffeinspritzsystem elektronisch gesteuert wird.

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14. Verfahren nach Anspruch 12 oder 13, worin das Treibstoffeinspritzsystem Sensormittel hat, die im Abgassystem des Motors angebracht sind, um das Luftzu-Treibstoff-Verhältnis des Treibstoffes und der Luft, die dem Motor zugeführt werden, zu regeln.

Revendications

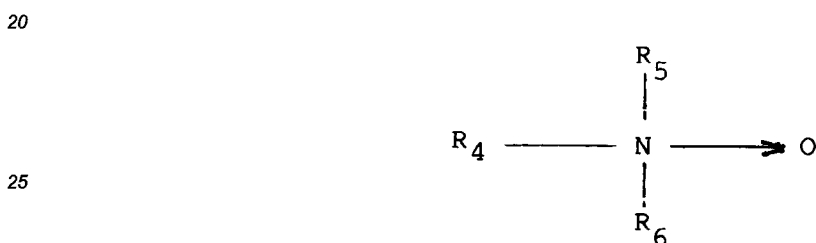
1. Une composition de carburant pour moteur à combustion interne, ladite composition de carburant comprenant :

- 5 a) un carburant (par ex. l'essence) ;
b) un agent de désencrassement répondant à la formule



15 dans laquelle R_1 est un alkyle en C_6 à C_{24} , un aryle, un reste cycloaliphatique, hétérocyclique, un alkyle substitué ou un aryle substitué ; R_2 et R_3 sont, indépendamment, un alkyle substitué en C_1 à C_{24} , un aryle, un reste cycloaliphatique ou hétérocyclique et

c) un agent de désencrassement à base d'oxyde d'amine possédant la formule de constitution suivante :



30 dans laquelle R_4 est choisi parmi un alkyle en C_6 à C_{24} , un aryle, un reste cycloaliphatique, hétérocyclique, un alkyle substitué, un aryle substitué ; R_5 et R_6 sont choisis, indépendamment, parmi un alkyle en C_1 à C_{24} , un aryle, un alkyle substitué, un aryle substitué, un reste cycloaliphatique, hétérocyclique et leurs mélanges.

2. La composition de carburant selon la revendication 1, dans laquelle R_1 est un alkyle en C_6 à C_{20} (par ex. des substituants en C_6 à C_{18} dérivés d'acides gras) ou un aryle alkylé et R_2 et R_3 sont, indépendamment, un alkyle en C_1 à C_{12} hydroxysubstitué.

3. La composition de carburant selon la revendication 1 ou la revendication 2, dans laquelle l'agent de désencrassement b) est choisi dans le groupe consistant en bis(2-hydroxyéthyl) cocoamine, bis(2-hydroxyéthyl) suif amine, bis (2-hydroxyéthyl) stéarylamine, bis(2-hydroxyéthyl) oléylamine et en leurs mélanges.

4. La composition de carburant selon l'une quelconque des revendications 1 à 3, dans laquelle la concentration de l'agent de désencrassement b) dans le carburant se situe dans la gamme de 2 à 200 ppm en poids (par ex. de 16 à 100 ppm en poids).

5. La composition selon l'une quelconque des revendications 1 à 4, dans laquelle l'oxyde d'amine c) est choisi parmi l'oxyde de bis(2-hydroxyéthyl) cocoamine, l'oxyde de bis(2-hydroxyéthyl) stéarylamine, l'oxyde de diméthylcocoamine, l'oxyde de diméthyl suif hydrogéné amine, l'oxyde de diméthylhexadécylamine et leurs mélanges.

6. La composition selon l'une quelconque des revendications 1 à 5, dans laquelle la concentration de l'oxyde d'amine se situe dans la gamme de 2 à 80 ppm (par ex. de 4 à 40 ppm).

7. La composition selon l'une quelconque des revendications 1 à 6 contenant un agent désémulsifiant d) choisi dans le groupe consistant en

- 50 i) polyglycols acylés ;
ii) alkylarylsulfonates, polyglycols, résines d'alkylphénol-formaldéhyde oxygénées ;
iii) résines d'alkylphénol-formaldéhyde oxyalkylées et polyglycols ;
iv) résines d'alkylphénol-formaldéhyde et polyglycols ;
v) résines d'alkylphénol-formaldéhyde oxyalkylées et en leurs mélanges.

8. La composition de carburant selon la revendication 7, dans laquelle la concentration de l'agent désémulsifiant d) se situe dans la gamme de 0,1 à 20 ppm en poids (par ex. 1,0 à 8,0 ppm en poids)

9. Un procédé de préparation d'une composition de carburant selon l'une quelconque des revendications 1 à 5 (cas (a)) ou selon l'une quelconque des revendications 6 à 8 (cas (b)) qui, dans le cas a), consiste à ajouter

à un carburant (par ex. l'essence) un concentré d'additif contenant

i) 5 à 60% en poids de bis(2-hydroxyéthyl)cocoamine ;

ii) 0,25 à 10% en poids d'un agent désémulsifiant choisi parmi : des polyglycols acylés ou des alkylaryl-sulfonates, des polyglycols, des résines d'alkylphénol-formaldéhyde oxyalkylées ou des résines d'alkylphénol-formaldéhyde oxyalkylées et des polyglycols ou des résines d'alkylphénol-formaldéhyde oxyalkylées ou leurs mélanges et

iii) 40 à 95% en poids de solvant et qui, dans le cas b), consiste à ajouter au carburant un concentré d'additif comme pour le cas (a) et, en plus, 1 à 15% en poids d'oxyde de bis (2-hydroxyéthyl)cocoamine.

10. Un procédé de préparation d'une composition de carburant selon l'une quelconque des revendications

1 à 8 consistant à ajouter à un carburant (par ex. l'essence) un concentré comprenant :

. 8 à 32% d'un agent de désencrassement à base d'amine (b) ;

. 2 à 8% en poids d'un agent de désencrassement à base d'oxyde d'amine (c) et

. 40 à 95% en poids de solvant.

11. Un procédé selon la revendication 9, dans lequel le concentré contient 1 à 4% en poids d'un agent désémulsifiant tel que spécifié dans la revendication 7.

12. Un procédé pour minimiser et/ou empêcher l'encrassement de l'injecteur de carburant dans un moteur à injection à orifices multiples qui consiste à fournir au système d'injection de carburant du moteur soit une composition selon l'une quelconque des revendications 1 à 8, soit une composition de carburant réalisée par le procédé de la revendication 9 ou ou la revendication 10.

13. Un procédé selon la revendication 12, dans lequel le système d'injection de carburant est à commande électronique.

14. Un procédé selon la revendication 12 ou la revendication 13, dans lequel le système d'injection de carburant comporte des sondes disposées dans le système d'échappement du moteur pour réguler le rapport air à carburant du carburant et de l'air alimentant le moteur.