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(54) **Title:** FUEL FORMULATIONS

(57) **Abstract:** The use of at least one C₇-C₃₀ alkane in a diesel fuel formulation for the purpose of reducing fuel injector deposits, in particular fuel internal injector deposits, and/or for the purpose of reducing filter deposits, and/or for the purpose of increasing fuel economy and/or for the purpose of reducing power loss and/or for the purpose of improving driveability.



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FUEL FORMULATIONS

Field of the Invention

This invention relates to diesel fuel formulations, and in particular to diesel fuel formulations having reduced injector deposits.

5 Background to the Invention

In the interests of the environment, and to comply with increasingly stringent regulatory demands, it is necessary to provide automotive fuels which have low emissions and excellent fuel economy. In a compression
10 ignition engine these two properties are dependent, among other things, on the efficient working of the fuel injection system. Any deposits which build up on the fuel injectors can cause the injectors to become less efficient and can lead to a negative effect upon emission
15 levels, power output and fuel economy.

It is known that degradation of diesel fuel can be caused by a process known as "cavitation". Cavitation is the formation and then implosion of cavities in a liquid - i.e. small liquid-free zones ("bubbles") - that are the
20 consequence of forces acting upon the liquid. It usually occurs when a liquid is subjected to rapid changes of pressure that cause the formation of cavities where the pressure is relatively low.

Whilst not wishing to be limited by theory, this
25 process of cavitation in the high pressure environment of the fuel injection equipment can ultimately lead to the degradation of diesel fuel and an undesirable build-up of deposits on fuel injectors and filters, in particular on the internal surfaces of the fuel injectors. These
30 deposits, sometimes known as Internal Injector Deposits

(IIDs) or Internal Diesel Injector Deposits (IDIDs) can be detrimental to the controlled delivery of the fuel and hence can cause driveability and maintenance issues, increased emissions, reduced fuel economy, and loss of power.

It would therefore be desirable to formulate a diesel fuel which reduces the severity of fuel degradation due to cavitation, and therefore which reduces the amount of fuel derived deposits.

Summary of the Invention

According to the present invention there is provided the use of at least one C₇-C₃₀ alkane in a diesel fuel composition for reducing fuel injector deposits, in particular internal injector deposits.

As used herein, the terms "internal injector deposits" and "internal diesel injector deposits" are used interchangeably.

According to another aspect of the present invention there is provided the use of at least one C₇-C₃₀ alkane in a diesel fuel composition for reducing filter deposits.

According to yet a further aspect of the present invention there is provided the use of at least one C₇-C₃₀ alkane in a diesel fuel composition for improving fuel economy.

According to yet a further aspect of the present invention there is provided the use of at least one C₇-C₃₀ alkane in a diesel fuel composition for reducing emissions.

According to yet a further aspect of the present invention there is provided the use of at least one C₇-C₃₀ alkane in a diesel fuel composition for reducing power loss.

According to yet a further aspect of the present

invention there is provided the use of at least one C₇-C₃₀ alkane in a diesel fuel composition for improving driveability.

Detailed Description of the Invention

5 An essential component of the diesel fuel composition herein is at least one C₇-C₃₀ alkane, preferably at least one C₈-C₁₈ alkane, more preferably at least one C₈-C₁₂ alkane, especially at least one C₁₀-C₁₂ alkane. The term "at least one C₇-C₃₀ alkane" means one
10 C₇-C₃₀ alkane or a mixtures of C₇-C₃₀ alkanes.

 Suitable C₇-C₃₀ alkanes for use herein include octane, decane, dodecane, tetradecane, hexadecane and mixtures thereof. Preferred C₇-C₃₀ alkanes for use herein are octane, decane and dodecane, and mixtures thereof.

15 The C₇-C₃₀ alkanes for use herein may be derived from a variety of sources, included, but not limited to, crude oil derived, Fischer-Tropsch derived (so-called "Gas-to-liquid" or "GTL" fuel), derived from a biological source or made by the hydrotreatment of vegetable oils, animal
20 fats and algal oils. The total concentration of C₇-C₃₀ alkane, in a diesel fuel formulation according to the invention, is preferably 0.1% v/v or greater and up to 90% v/v, or up to 10% or 5% v/v.

 The diesel fuel composition herein further comprises
25 an additional diesel fuel component (ii) in addition to the C₇-C₃₀ alkane (i). In the context of the present invention, "use" of at least one C₇-C₃₀ alkane in a diesel fuel formulation means incorporating the at least one C₇-C₃₀ alkane into the formulation typically as a blend
30 (i.e. a physical mixture) with the additional diesel fuel component (ii). It will be appreciated by a person skilled in the art that the additional fuel component (ii) may already contain some C₇-C₃₀ alkane components.

The concentration of the C₇-C₃₀ alkane referred to above means the concentration of C₇-C₃₀ alkanes which are added into the diesel fuel formulation as a blend with the additional fuel component, and does not include the concentration of any C₇-C₃₀ alkanes already present in the additional fuel component (ii).

The additional diesel fuel component (ii) may be any fuel component suitable for use in a diesel fuel formulation and therefore for combustion within a compression ignition (diesel) engine. It will typically be a liquid hydrocarbon middle distillate fuel, more typically a gas oil. It may be a kerosene fuel component. It may be petroleum derived. Alternatively it may be synthetic: for instance it may be the product of a Fischer-Tropsch condensation. It may be derived from a biological source (i.e. a biofuel).

Biofuels are combustible fuels, typically derived from biological sources, which result in a reduction in "well-to-wheels" (i.e. from source to combustion) greenhouse gas emissions. In diesel fuels for use in compression ignition engines, the most common biofuels are fatty acid alkyl esters (FAAEs), in particular fatty acid methyl esters (FAMEs) such as rapeseed methyl ester and palm oil methyl ester; these are used in blends with conventional diesel fuel components.

An additional fuel component (ii) will typically boil in the range from 150 or 180 to 360°C (ASTM D86 or EN ISO 3405). It will suitably have a measured cetane number (ASTM D613) of from 40 to 70 or from 40 to 65 or from 51 to 65 or 70.

A formulation for use herein may contain a mixture of two or more additional diesel fuel components (ii).

The concentration of the component(s) (ii) in the

formulation may suitably be 70% v/v or greater, or 75 or 80 or 85% v/v or greater, or 90 or 92 or 95% v/v or greater. It may be up to 98% v/v, or up to 95 or 92 or 90 or 85 or 80% v/v. In general, it will represent the major part of the fuel formulation. After inclusion of the C₇-C₃₀ alkane (i) and any optional fuel additives, the component (ii) will typically represent the balance to 100%.

A fuel formulation herein suitably has a CFPP (IP 309 or EN 116) of 5°C or lower, or of 0°C or lower, or of -5 or -10°C or lower. It may have a CFPP of -15°C or lower, or of -18°C or lower, or of -20 or -25 or -30 or -35 or -44°C or lower. It suitably has a cloud point (ASTM D5773) of 0°C or lower, or of -5 or -10°C or lower, or of -12 or -15 or -20 or -25 or -30°C or lower. It suitably has a flash point (ASTM D92 or D93) of 40°C or higher, or of 45 or 50 or 55°C or higher.

The formulation herein should be suitable for use in a compression ignition (diesel) internal combustion engine. Such an engine may be either heavy or light duty. The formulation may in particular be suitable for use as an automotive diesel fuel.

In an embodiment, the formulation is suitable and/or adapted for use as a "winter grade" automotive diesel fuel, for use in colder climates such as in northern Europe (particularly Scandinavia) or North America. It may be a so-called "arctic grade" fuel, for use in particularly extreme climates such as in northern Scandinavia.

In further embodiments the formulation may be suitable and/or adapted for use as an industrial gas oil, or as a domestic heating oil.

The formulation will suitably comply with applicable

current standard diesel fuel specification(s) such as for example EN 590 (for Europe) or ASTM D975 (for the USA). By way of example, the overall formulation may have a density from 820 to 845 kg/m³ at 15°C (ASTM D4052 or EN ISO 3675); a T95 boiling point (ASTM D86 or EN ISO 3405) of 360°C or less; a measured cetane number (ASTM D613) of 51 or greater; a kinematic viscosity at 40°C (ASTM D445 or EN ISO 3104) from 2 to 4.5 centistokes; a sulphur content (ASTM D2622 or EN ISO 20846) of 50 mg/kg or less; and/or a polycyclic aromatic hydrocarbons (PAH) content (IP 391(mod)) of less than 11% w/w. Relevant specifications may however differ from country to country and from year to year, and may depend on the intended use of the formulation. Moreover a formulation according to the invention may contain fuel components with properties outside of these ranges, since the properties of an overall blend may differ, often significantly, from those of its individual constituents.

The relative concentrations of the components (i) and (ii) may be chosen to achieve desired properties for the formulation as a whole, for example a desired maximum CFPP and/or cloud point, and/or a desired minimum flash point. Thus the relative concentrations will also depend on the physicochemical properties of the individual components.

A fuel formulation for use herein may contain standard fuel or refinery additives which are suitable for use in diesel fuels. Many such additives are known and commercially available.

The diesel fuel formulation described herein is prepared by a process which involves blending together (i) at least one C₇-C₃₀ alkane and (ii) one or more additional diesel fuel components, optionally with one or

more fuel additives. The process may be used to produce at least 1,000 litres of the fuel formulation, or at least 5,000 or 10,000 or 25,000 litres, or at least 50,000 or 75,000 or 100,000 litres.

5 The diesel fuel formulation described herein is used in a method of operating an internal combustion engine, and/or a vehicle which is driven by an internal combustion engine, which method involves introducing into a combustion chamber of the engine a diesel fuel
10 formulation as described herein. The engine is suitably a compression ignition (diesel) engine. Such a diesel engine may be of the direct injection type, for example of the rotary pump, in-line pump, unit pump, electronic unit injector or common rail type, or of the indirect
15 injection type. It may be a heavy or a light duty diesel engine.

 According to the present invention, there is provided the use of at least one C₇-C₃₀ alkane, in a diesel fuel formulation, for the purpose of reducing fuel
20 injector deposits, in particular internal injector deposits (IIDs) or internal diesel injector deposits (IDIDs). In the context of this aspect of the invention, the term "reducing" embraces any degree of reduction, including reduction to zero. The reduction may for
25 instance be 10% or more, or 25 or 50 or 75 or 90% or more of the fuel injector deposits produced by an analogous diesel fuel formulation comprising (ii) an additional fuel component for use in an analogous context, prior to adding at least one C₇-C₃₀ alkane to it in accordance with
30 the present invention.

 According to the present invention, there is provided the use of at least one C₇-C₃₀ alkane, in a diesel fuel formulation, for the purpose of reducing

filter deposits. In the context of this aspect of the invention, the term "reducing" embraces any degree of reduction, including reduction to zero. The reduction may for instance be 10% or more, or 25 or 50 or 75 or 90% or more of the filter deposits produced by an analagous diesel fuel formulation comprising (ii) an additional fuel component for use in an analogous context, prior to adding at least one C₇-C₃₀ alkane to it in accordance with the present invention.

According to another aspect, the invention provides the use of at least one C₇-C₃₀ alkane, in a diesel fuel formulation, for the purpose of increasing fuel economy. In the context of this aspect of the invention, the term "increasing" embraces any degree of increase. The increase may for instance be 10% or more, or 25 or 50 or 75 or 90% or more of the fuel economy produced by an analagous diesel fuel formulation comprising (ii) an additional fuel component for use in an analogous context, prior to adding at least one C₇-C₃₀ alkane to it in accordance with the present invention.

According to yet a further aspect of the present invention there is provided the use of at least one C₇-C₃₀ alkane in a diesel fuel composition for reducing emissions. As used herein the term "emissions" means tailpipe emissions such as CO, NO_x, hydrocarbons and particulates. In the context of this aspect of the invention, the term "reducing" embraces any degree of reduction, including reduction to zero. The reduction may for instance be 10% or more, or 25 or 50 or 75 or 90% or more of the emissions produced by an analagous diesel fuel formulation comprising (ii) an additional fuel component for use in an analogous context, prior to adding a C₇-C₃₀ alkane to it in accordance with the

present invention.

According to yet a further aspect of the present invention there is provided the use of at least one C₇-C₃₀ alkane in a diesel fuel composition for reducing power loss. In the context of this aspect of the invention, the term "reducing" embraces any degree of reduction, including reduction to zero. The reduction may for instance be 10% or more, or 25 or 50 or 75 or 90% or more of the power loss produced by an analogous diesel fuel formulation comprising (ii) an additional fuel component for use in an analogous context, prior to adding at least one C₇-C₃₀ alkane to it in accordance with the present invention.

According to yet a further aspect of the present invention there is provided the use of at least one C₇-C₃₀ alkane in a diesel fuel composition for improving driveability. As used herein the term "driveability" means, for example, the ability to start-up and/or the tendency to stall and/or the tendency for smooth running. "Poor driveability" in this context means, for example, poor start-up and/or rough running and/or engine stalls. In the context of this aspect of the invention, the term "improving" embraces any degree of improvement in driveability. An improvement in driveability means, for example, quicker start-up and/or smoother running and/or fewer engine stalls. The improvement may for instance be 10% or more, or 25 or 50 or 75 or 90% or more of the driveability produced by an analogous diesel fuel formulation comprising (ii) an additional fuel component for use in an analogous context, prior to adding C₇-C₃₀ alkanes to it in accordance with the present invention.

In the context of the present invention, "use" of at least one C₇-C₃₀ alkane in a diesel fuel formulation means

incorporating at least one C₇-C₃₀ alkane into the formulation typically as a blend (ie a physical mixture) with one or more other diesel fuel components. The C₇-C₃₀ alkane will conveniently be incorporated into an engine or other system which is to be run on the formulation. Instead or in addition, the use of C₇-C₃₀ alkanes may involve running a fuel-consuming system, typically an internal combustion engine, on a diesel fuel formulation containing the C₇-C₃₀ alkanes, typically by introducing the formulation into a combustion chamber of an engine.

"Use" of C₇-C₃₀ alkanes in the ways described above may also embrace supplying the C₇-C₃₀ alkanes together with instructions for its use in a diesel fuel formulation, to achieve the purpose(s) of any of the aspects of the invention, for instance to reduce fuel injector deposits or to increase fuel economy.

Throughout the description and claims of this specification, the words "comprise" and "contain" and variations of the words, for example "comprising" and "comprises", mean "including but not limited to", and do not exclude other moieties, additives, components, integers or steps. Moreover the singular encompasses the plural unless the context otherwise requires: in particular, where the indefinite article is used, the specification is to be understood as contemplating plurality as well as singularity, unless the context requires otherwise.

Preferred features of each aspect of the invention may be as described in connection with any of the other aspects. Other features of the invention will become apparent from the following examples. Generally speaking the invention extends to any novel one, or any novel combination, of the features disclosed in this

specification (including any accompanying claims and drawings). Thus features, integers, characteristics, compounds, chemical moieties or groups described in conjunction with a particular aspect, embodiment or example of the invention are to be understood to be applicable to any other aspect, embodiment or example described herein unless incompatible therewith. Moreover unless stated otherwise, any feature disclosed herein may be replaced by an alternative feature serving the same or a similar purpose.

The present invention will now be further described with reference to the following non-limiting examples.

Example 1

Laboratory experiments involving the acoustic cavitation of diesel fuels were used to develop insight into how the collapse of micro-bubbles in the fuel injection equipment of compression ignition engines generates small soot-like particles. These particles are believed to be related to the problems of blocked fuel filters and internal injector deposits.

For some time, the area of sonochemistry has used ultrasound to acoustically generate cavities in fluids and drive chemical reactions. This approach has been adopted in the present examples to simulate the manner in which diesel fuel is degraded during hydrodynamic cavitation in the high pressure fuel injection equipment of compression ignition vehicles.

Cavitation is the formation of gas bubbles in a region where the pressure of a liquid falls below its vapour pressure. This can occur when a diesel fuel experiences a pressure drop across an orifice or when fast moving flows turn sharp corners. When the bubbles that are generated return to high ambient pressure they

collapse and produce localized areas of extreme temperature and pressure.

Two key parameters associated with fluid cavitation are:

1. the extent of cavity formation (that is the number of bubbles generated)
2. the intensity of the bubble collapse (which is related to the speed of collapse and the magnitude of the temperature and pressure generated).

Sonication experiments were carried out with a VCX 500 ultrasonics processor (ex. Sonics Materials Inc.) and a 13mm extender horn which delivers ultrasound to a 50mL sample of hydrocarbon contained within a jacketed glass beaker. Cold water (5°C) is passed through the jacket during sonication to keep the liquid hydrocarbon below its flash point. Dry air is blown over the surface of the fuel to ensure no condensation inside the reaction vessel and a PTFE lid prevents splashing. The whole apparatus is housed inside an insulated box (fitted with a safety cut-out mechanism) to reduce acoustic noise.

Ultrasound is produced at a frequency of 20 kHz and when the amplitude of the processor is set to 65% the transfer of energy to the hydrocarbon occurs at $6 \text{ kJ mL}^{-1} \text{ h}^{-1}$. The probe is made of titanium alloy (Ti 6Al-4V) and consists of 90% titanium, 6% aluminium and 4% vanadium. This material is susceptible to cavitation erosion and also becomes tarnished during sonication. The probe is polished on silicon carbide paper between each experiment to maintain the tip as a smooth and shiny surface.

All experiments were run with a 65% amplitude setting on the ultrasound processor, a cut-out temperature of 55°C and with dry air blown over the surface of the hydrocarbon being sonicated.

In a typical experiment 50 mL of 1-methylnaphthalene (95% purity ex. Aldrich) is sonicated for 1 hour, during which time the hydrocarbon darkens in colour due to the build up of a dispersion of soot-like carbon particles.

5 The 1-methylnaphthalene is mixed with 50mL of n-heptane and spun at 4,000rpm for 20 minutes, which leads to sedimentation of deposit material at the bottom of the centrifuge tube. Filtration of this sample through a 0.7 μ m paper then allows the cavity induced deposit mass to
10 be gravimetrically determined.

A series of hydrocarbons (as set out in Table 1) were sourced from Sigma-Aldrich and filtered through a 0.7 μ m paper in preparation for sonication. 1-methylnaphthalene was used as a reference fuel to
15 establish a baseline level of deposit mass generated under the following conditions:

50 mL fuel

1 hour sonication time

65% amplitude

20 55°C cut-off temperature

Dry air blown over surface of fuel

Table 1

Chemical	Formula	mp °C	bp °C	Flash Point °C	Density g/mL	MW g/mol
1-methylnaphthalene	C ₁₀ H ₁₁	-22	240	108	1.001	142.2
n-octane	C ₈ H ₁₈	-57	125	13	0.703	114.2
n-decane	C ₁₀ H ₂₂	-30	174	46	0.730	142.3
dodecane	C ₁₂ H ₂₆	-10	216	83	0.750	170.3
tetradecane	C ₁₄ H ₃₀	6	252	100	0.762	198.4
hexadecane	C ₁₆ H ₃₄	18	287	135	0.773	229.4
ethylbenzene	C ₈ H ₁₀	-95	136	15	0.867	105.2
n-propylbenzene	C ₉ H ₁₂	-99	156	42	0.862	120.2
Indan	C ₉ H ₁₀	-51	176	54	0.965	118.2
Indene	C ₉ H ₈	-5	181	58	0.996	116.2

A sample of 1-methylnaphthalene was then prepared with 1, 2 and 3%v of the hydrocarbons listed in Table 1. The fuels were sonicated in two blocks of testing:

Block 1: 1-methylnaphthalene and 1-methylnaphthalene with 1-3%v n-octane or n-decane

Block 2: 1-methylnaphthalene and 1-methylnaphthalene with 1-3%v n-dodecane, tetradecane, hexadecane, ethylbenzene, n-propylbenzene, indan or indene.

In each case several repeats of 1-methylnaphthalene were performed to establish baseline results. All fuels were run in a randomized test order in case of any drift in performance of the sonication probe, however energy delivery was consistent over the course of the experiments.

The deposit mass generated after cavitation was measured for each fuel sample. Details of the impact of cavitation in the form of the deposit mass produced by cavitation is shown in Table 2 and Table 3. Table 3 also shows details of the % change in deposit mass provided by each of the C₇-C₃₀ alkanes added (a negative number being a % reduction in deposit mass).

Table 2

1-methylnaphthalene	Deposit mass mg
Block 1	5.39
Block 1	5.18
Block 1	5.07
Block 2	4.95
Block 2	4.71
Block 2	5.10
Block 2	4.87
Block 2	5.11
Block 2	5.01
Block 2	4.93

Table 3

Fuel (1-methylnaphthalene w/ x %v hydrocarbon)	Deposit mass mg	Change in deposit mass %
1%v n-octane	4.41	-15.2
2%v n-octane	4.19	-19.5
3%v n-octane	3.50	-32.7
1%v decane	4.43	-14.9
2%v decane	4.22	-18.9
3%v decane	3.77	-27.5
1%v dodecane	4.38	-11.6
2%v dodecane	4.27	-13.8
3%v dodecane	4.58	-7.6
1%v tetradecane	4.38	-11.6
2%v tetradecane	4.21	-15.0
3%v tetradecane	4.28	-13.6
1%v hexadecane	4.88	-1.5
2%v hexadecane	4.55	-8.2
3%v hexadecane	4.47	-9.8
1%v ethylbenzene	5.11	3.1
2%v ethylbenzene	4.71	-4.9
3%v ethylbenzene	4.63	-6.5
1%v n-propylbenzene	4.92	-0.7
2%v n-propylbenzene	4.94	-0.3
3%v n-propylbenzene	4.40	-11.2
1%v indan	4.61	-6.9
2%v indan	4.95	-0.1
3%v indan	4.95	-0.1
1%v indene	4.86	-1.9
2%v indene	5.03	1.5
3%v indene	5.08	2.5

Discussion

5 The aromatic hydrocarbons (indan, indene, n-propylbenzene and ethylbenzene) have a limited impact on deposit mass. In contrast the paraffinic hydrocarbons reduce the amount of deposit, with this trend becoming more pronounced as the alkane chain length becomes shorter. Generally the suppression of deposit formation increases with the blend ratio of the alkane in 1-methylnaphthalene.

C L A I M S

1. Use of at least one C₇-C₃₀ alkane, in a diesel fuel formulation, for the purpose of reducing fuel injector deposits, in particular fuel internal injector deposits.
2. Use according to Claim 1 wherein the diesel fuel formulation comprises an additional diesel fuel component.
3. Use according to Claim 1 or 2 wherein the C₇-C₃₀ alkane is present in a concentration of 0.1% v/v or greater.
4. Use according to any of claims 1 to 3, wherein the C₇-C₃₀ alkane is present in a concentration of up to 90% v/v or greater.
5. Use according to any of claims 1 to 4 wherein the additional diesel fuel component is present in a concentration of 70% v/v or greater.
6. Use according to any of claims 1 to 5 wherein the additional diesel fuel component is present in a concentration of up to 98% v/v or greater.
7. Use according to any of Claims 1 to 6, wherein the C₇-C₃₀ alkane is a C₈-C₁₈ alkane.
8. Use according to any of Claims 1 to 7 wherein the C₇-C₃₀ alkane is a C₈-C₁₂ alkane.
9. Use according to any of Claims 1 to 7 wherein the C₇-C₃₀ alkane is a C₁₀-C₁₂ alkane.
10. Use of at least one C₇-C₃₀ alkane in a diesel fuel formulation for the purpose of reducing filter deposits.
11. Use of at least one C₇-C₃₀ alkane, in a diesel fuel formulation, for the purpose of increasing fuel economy.
12. Use of at least one C₇-C₃₀ alkane, in a diesel fuel

formulation, for the purpose of reducing power loss.

13. Use of at least one C₇-C₃₀ alkane, in a diesel fuel formulation, for the purpose of improving driveability.