(54) Titre : COMPOSITION DE CARBURANT DIESEL A BASE DE DIETHYL ETHER

(55) Title: DIESEL FUEL COMPOSITION BASED ON DIETHYL ETHER

(57) Abrégé/Abstract:
A compression ignition fuel composition comprising diethyl ether (DEE) and water, in amounts that correspond to the reaction product of dehydration of anhydrous or hydrous ethanol (EtOH) with water content between 0 and 30 wt%, and further
characterized by: a) a diethyl ether content in wt% that is larger than or equal to 50, and a water content in wt% that is lower than or equal to 50 minus concentration of ethanol in wt% for mixtures containing 0 to 20 wt% ethanol; or b) a diethyl ether content in wt% that is larger than or equal to 54 minus 0.2 times the ethanol concentration in wt%, and a water content in wt% that is lower than or equal to 46 minus the 0.8 times concentration of ethanol in wt% for mixtures containing 20 to 30 wt% ethanol; or c) a diethyl ether content in wt% that is larger than or equal to 61.5 minus 0.45 times the ethanol concentration in wt%, and a water content in wt% that is lower than or equal to 38.5 minus the 0.55 times concentration of ethanol in wt% for mixtures containing 30 to 70 wt% ethanol.
Title: DIESEL FUEL COMPOSITION BASED ON DIETHYL ETHER

Abstract: A compression ignition fuel composition comprising diethyl ether (DEE) and water, in amounts that correspond to the reaction product of dehydration of anhydrous or hydrous ethanol (EtOH) with water content between 0 and 30 wt%, and further characterized by: a) a diethyl ether content in wt% that is larger than or equal to 50, and a water content in wt% that is lower than or equal to 50 minus concentration of ethanol in wt% for mixtures containing 0 to 20 wt% ethanol; or b) a diethyl ether content in wt% that is larger than or equal to 54 minus 0.2 times the ethanol concentration in wt%, and a water content in wt% that is lower than or equal to 46 minus the 0.8 times concentration of ethanol in wt% for mixtures containing 20 to 30 wt% ethanol; or c) a diethyl ether content in wt% that is larger than or equal to 61.5 minus 0.45 times the ethanol concentration in wt%, and a water content in wt% that is lower than or equal to 38.5 minus the 0.55 times concentration of ethanol in wt% for mixtures containing 30 to 70 wt% ethanol.
— of inventorship (Rule 4.17(iv))

Published:
— with international search report (Art. 21(3))
Title: Diesel Fuel Composition based on Diethyl Ether

The present invention relates to a compression ignition fuel composition. In particular the invention concerns a compression ignition fuel composition comprising diethyl ether, ethanol and water each in amounts being useful as fuel in conventional standard diesel engines.

Ether comprising blends are known to be excellent compression ignition engine fuel. Use of lower ethers prepared by dehydration of alcohols as compression ignition fuel has been described in number of publications, e.g. US Patent Nos. 4,892,561, 5,906,664 and 7,449,034.

WO08100721 discloses a universal liquid fuel consisting essentially of the following constituents:

a) from 1% to 71% by volume of one or more primary, secondary or tertiary monohydric aliphatic alcohols containing 1 to 8 carbon atoms, benzyl alcohol or mixtures thereof;
b) from 0.5% to 10% by volume of water;
c) from 1% to 90% by volume of one or more vegetable oils or mixtures thereof; and
d) from 10% to 80% of one or more ethers.

A fuel blend comprising a homogeneous liquid phase of at least about 80 percent by weight of an alcohol and about 1-10 percent by volume of ether such as dimethyl ether or diethyl ether and about 0.008-0.02 percent by volume of distilled organic oil is mentioned in US Patent Appln. 008244960 A.
US patent No. 7722688 mentions fuel water blended compositions consisting of a normally liquid hydrocarbon fuel, which comprises ethanol and diethyl ether, water, a nitrogen free surfactant, and optionally an acid having a pKa of up to about 6 with a water content of 0.5 to 50 percent by weight.

A technique for ethanol fuelled compression ignition engines make use of ignition enhancers such as 2-ethylhexyl nitrate (EHN), polyalkylene glycol compounds (EP403516), polyol having 3-10 hydroxyl 5 groups and ethylene oxide and/or propylene oxide (WO9505437). These compounds, however, are expensive and require also major adjustments to the engine. The engine has typically to be run at about twice the compression compared to conventional diesel fuel.

We have found that mixtures comprising diethyl ether, ethanol and a high content of water blended in predetermined mixture ratios can be used as fuel in a standard compression ignition engine fuel. Application of such a fuel does not require major adjustments in a conventional diesel-fueled compression ignition engine fuel. A particular advantage of such fuel mixtures is that they can be produced by dehydration of anhydrous or hydrous ethanol, enabling the use of ethanol as a fuel for compression-ignition engines.

Pursuant to the above finding, the invention provides a compression ignition fuel composition comprising diethyl ether (DEE) and water, in amounts that correspond to the reaction product of dehydration of anhydrous or hydrous
ethanol (EtOH) with water content between 0 and 30 wt%, and further characterized by:

a) a diethyl ether content in weight percent that is larger than or equal to 50, and a water content in weight percent that is lower than or equal to 50 minus concentration of ethanol in weight percent for mixtures containing 0 to 20 wt% ethanol; or

b) a diethyl ether content in weight percent that is larger than or equal to 54 minus 0.2 times the ethanol concentration in weight percent, and a water content in weight percent that is lower than or equal to 46 minus the 0.8 times concentration of ethanol in weight percent for mixtures containing 20 to 30 wt% ethanol; or

c) a diethyl ether content in weight percent that is larger than or equal to 61.5 minus 0.45 times the ethanol concentration in weight percent, and a water content in weight percent that is lower than or equal to 38.5 minus the 0.55 times concentration of ethanol in weight percent for mixtures containing 30 to 70 wt% ethanol.

Fuel mixtures without ethanol corresponding to a complete conversion of the anhydrous or hydrous ethanol with a water content between 0 and 30 percent by weight are a part of the invention.

The hatched area in Fig. 1 corresponds to the composition range of the diethyl ether/ethanol/water fuel mixtures of the invention.

With the present invention, it is possible to operate a compression ignition engine with a fuel based on ethanol/water mixtures without ignition enhancers, without
emulsifiers, and without making major adjustments to the engine compared to operation with conventional diesel fuel.

Fuel mixtures of the invention do not need further purification, such as removal of water, before use. Additional components such as lubricants, emulsifiers and antioxidants may be present in the fuel mixture but are not crucial.

The fuel mixtures can be produced by mixing water, diethyl ether and ethanol in the appropriate amounts, or by dehydration of anhydrous or hydrous ethanol with a water content between 0 and 30 percent by weight or by any other method that results in the fuel compositions according to the invention.

A process to produce the fuel may include steps aimed at obtaining diethyl ether yields that lie beyond the natural equilibrium, such as extraction, distillation, catalytic distillation, adsorption of water or diethyl ether, recirculation.

A preferred embodiment of the invention comprises ternary mixtures of ethanol, diethyl ether and water in amounts that correspond to the reaction product of dehydration of anhydrous or hydrous ethanol with a water content between 0 and 30 wt% further characterized by an ethanol content larger than or equal to 8 wt%. This corresponds to the fuel compositions indicated by the areas A and B in Fig. 1.

These fuel compositions can be produced by catalytic dehydration of anhydrous or hydrous ethanol with a water content between 0 and 30 percent by weight using acid cata-
lysts known in the art without separation processes to enhance the yield. After production of the fuel mixture, the product does not need further purification before use. This implies that in an on-board application of an ethanol/diethyl ether fuel neither removal of the water produced in the conversion of ethanol nor an enhancement of the diethyl ether concentration by other means is required.

Condensation of the product to a liquid phase may result in a phase separation in the liquid phase. Thus, additional components, such as emulsifiers, lubricants, antioxidants, may be present in the fuel mixture but are not crucial.

A further preferred embodiment of the invention comprises mixtures of ethanol, diethyl ether and water in amounts that correspond to the reaction product of dehydration of anhydrous or hydrous ethanol with a water content between 0 and 30 wt% further characterized by a diethyl ether content in weight percent is higher than or equal to 145 minus 4 times the ethanol content in weight percent. This corresponds to the fuel compositions indicated by the area B in Fig. 1 of the drawings.

Condensation of the above fuel composition to the liquid phase does not result in a phase separation.

In still a preferred embodiment, the fuel composition contains ethanol (EtOH), water and diethyl ether (DEE) in the amounts in wt% shown in Table 1 below.
**Table 1**

<table>
<thead>
<tr>
<th>DEE</th>
<th>E10H</th>
<th>water</th>
</tr>
</thead>
<tbody>
<tr>
<td>54.7</td>
<td>12</td>
<td>33.3</td>
</tr>
<tr>
<td>51.5</td>
<td>16</td>
<td>32.6</td>
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<td>61.5</td>
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<td>57.9</td>
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<td>18</td>
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<tr>
<td>49.7</td>
<td>33.3</td>
<td>17</td>
</tr>
<tr>
<td>45.8</td>
<td>38</td>
<td>16.2</td>
</tr>
</tbody>
</table>

The emissions from a fuel of the invention in a compression ignition engine are excellent. The emission without purification already complies with the EU-IV standard. The emission of soot particulates is several orders of magnitude lower compared to conventional diesel fuel. The emission of nitrogen oxides is reduced significantly as well. Therefore, the elaborate exhaust gas cleaning as applied today in diesel fuelled compression ignition engines can be significantly reduced or completely removed.

Fig. 1 in the drawings is a composition diagram of the fuel mixtures according to the present invention;

Fig. 2 shows diagrammatically results of fuel composition test in an Audi 1.9 liter turbo diesel engine. The engine runs on the fuel compositions indicated by the closed symbols and does not run on the fuel compositions indicated by the open symbols. The labels A, B, C, and D indicate the fuel compositions used in Example 8 below; and
Fig. 3 displays measured particle emission in engine exhaust gas running on a fuel composition according to the invention.

**Example 1**

This example shows that a fuel composition of 70 wt% diethyl ether, 10 wt% ethanol and 20% water corresponds to a reaction product of dehydration of anhydrous or hydrous ethanol with a water content between 0 and 30 wt%.

First, the amounts of diethyl ether, ethanol and water in weight percent are converted to mol %, as follows:

\[
C_{\text{mol},i} = \frac{100 C_{\text{weight},i}}{\sum_{i} C_{\text{weight},i}}
\]

(1)

where the index \( i \) indicates the three components, diethyl ether, ethanol and water; \( C_{\text{mol},i} \) and \( C_{\text{weight},i} \) are the concentrations in mol percent and weight percent, respectively, and \( M_i \) is the molar mass of the components, which is 74 for diethyl ether, 46 for ethanol and 18 for water. Thus, 70 wt% diethyl ether in the mixture a corresponds to

\[
\frac{100 \times 70}{70 + 10 + 20} = 41.59 \text{ mol } \%
\]

diethyl ether. Likewise, the molar concentrations of ethanol and water are 9.56 mol% and 48.85 mol%, respectively.

Using the stoichiometry of the dehydration reaction from ethanol to ether: \( 2 \text{ ethanol} = \text{diethyl ether} + \text{water} \), the ethanol and water content of the original anhydrous or hydrous ethanol in mol percent are given by:
\[ C^{0}_{\text{ethanol}} = C_{\text{mol, ethanol}} + 2 C_{\text{mol, diethyl ether}} \]
\[ C^{0}_{\text{water}} = C_{\text{mol, water}} - C_{\text{mol, diethyl ether}} \]

In the present example we find that the concentration of ethanol becomes \(9.56 + 2 \times 41.59 = 92.74\) mol\% and the concentration of water becomes \(48.85 - 41.59 = 7.26\) mol\%. These concentrations are then converted to weight percent as follows:

\[ C^{0}_{\text{weight},i} = \frac{100 \cdot M_i C^{0}_{\text{mol},i}}{\sum_i M_i C^{0}_{\text{mol},i}} \]

where the index \(i\) indicates water and ethanol. The content of ethanol in the anhydrous or hydrous ethanol then becomes \(\frac{100 \cdot 46 \cdot 92.74}{46 \cdot 92.74 + 18 \cdot 7.26} = 97.03\) wt\% ethanol; likewise we find for the water content 2.97 wt\%. This means that a fuel composition of 70 wt\% diethyl ether, 10 wt\% ethanol, and 20\% water corresponds to a reaction product of a dehydration of a hydrous ethanol that contains 2.97 wt\% water. This water content is between 0 and 30 wt\%.

**Example 2**

This example shows that a fuel composition of 70 wt\% diethyl ether, 10 wt\% ethanol, and 20\% water is included in the invention. In Example 1 it is shown that the composition corresponds to a reaction product of dehydration of anhydrous or hydrous ethanol with a water content between 0 and 30 wt\%. The ethanol content is 10 wt\%, and the diethyl ether content is 70\% which is larger than 50 and therefore this fuel composition is included in the invention.
**Example 3**

This example shows that a fuel composition of 70 wt% diethyl ether, 10 wt% ethanol, and 20% water lies in area A in Fig. 1.

Area A in Fig. 1 is characterized by a fuel composition that is included in the invention and further characterized by

1. an ethanol content larger than or equal to 8 wt%, and
2. a diethyl ether content that is smaller than or equal to 145 minus 4 times the ethanol content in weight percent - if it were larger than or equal this amount, it would lie in area B in Fig. 1.

The ethanol content is 10 wt%, which is above 8 wt%, and therefore condition (1) is satisfied.

Test of condition 2: The diethyl ether content in wt% should be lower than 145 - 4*10 = 105 to fulfil this condition. The diethyl ether concentration is 70% and therefore this condition is fulfilled. It is noted here that the conditions used are merely mathematical tests, which may produce unphysical concentration values (negative values or values above 100%).

**Example 4**

This example shows that a fuel composition of 70 wt% diethyl ether, 20 wt% ethanol and 10% water does not corre-
spond to a reaction product of dehydration of anhydrous or
hydrous ethanol with a water content between 0 and 30 wt%.

The composition in mol percent of the above fuel is, ac-
cording to Eq. (1), 48.85 mol% diethyl ether, 22.45 mol%
ethanol and 28.70 mol% water. According to Eq. (2), an
equivalent molar content of 2*48.85 + 22.45 = 120.16 mol%
ethanol and 28.70 - 48.85 = -20.15 mol% water is found.
Converting to wt% according to Eq. (3) gives a composition
of 107.03 wt% ethanol and -7.03 wt% water. The (unphysical)
water content is not between 0 and 30 wt%, and therefore
the fuel composition of 70 wt% diethyl ether, 20 wt% etha-
nol and 10% water does not correspond to a reaction product
of dehydration of anhydrous or hydrous ethanol with a water
content between 0 and 30 wt%.

Example 5

This example shows that a fuel composition of 40 wt% di-
ethyl ether, 10 weight percent of ethanol and 50 weight
percent of water does not correspond to a reaction product
of dehydration of anhydrous or hydrous ethanol with a water
content between 0 and 30 wt%.

The composition in mol% of the above fuel is according to
Eq. (1) 15.29 mol% diethyl ether, 6.15 mol% ethanol and
78.56 mol% water. According to Eq. (2), an equivalent molar
content of 2*15.29 + 6.15 = 36.73 mol% ethanol and 78.56 -
15.29 = 63.27 mol% water is found. Converting to wt% ac-
cording to Eq. (3) gives a composition of 59.73 wt% ethanol
and 40.27 wt% water. The water content is not between 0 and
30 wt%, and therefore the fuel composition of 40 wt% di-
ethyl ether, 10 wt% ethanol and 50 wt% water does not cor-
respond to a reaction product of dehydration of anhydrous or hydrous ethanol with a water content between 0 and 30 wt%.

Example 6
This example shows that a fuel composition of 45.8 wt% di-ethyl ether, 38 wt% ethanol and 16.2 wt% water lies in area B in Fig. 1.

The composition in mol% of the above fuel is according to Eq. (1) 26.39 mol% diethyl ether, 35.23 mol% ethanol and 38.38 mol% water. According to Eq. (2), an equivalent molar content of $2 \times 26.39 + 35.23 = 88.01$ mol% ethanol and 38.38 - 26.39 = 11.99 mol% water is found. Converting to weight percent according to Eq. (3) gives a composition of 94.94 wt % ethanol and 5.06 wt% water. The water content is between 0 and 30 wt%, and therefore this mixture corresponds to a reaction product of the dehydration of anhydrous or hydrous ethanol with a water content between 0 and 30 wt%.

The ethanol content in the mixture is 38 wt%. To be included in the invention, the diethyl ether content of 45.8 wt% must be larger than $61.5 - 0.45 \times 38$ (the ethanol content) = 44.4 wt%. This is the case, and therefore this composition is included in the invention.

Area B in Fig. 1, a preferred embodiment is characterized by a fuel composition that is included in the invention, and further characterized by

1. an ethanol content larger than or equal to 8 weight %,
(2) a diethyl ether content that is larger than or equal to 145 minus 4 times the ethanol content in weight percent.

The ethanol content in the fuel mixture is 38 wt%, which is larger than 8 wt% and therefore condition 1 is fulfilled. The diethyl ether content of 45.8 wt% is larger than 145 - 4*38 = -7, and therefore condition (2) is fulfilled as well. This means that the fuel composition is a preferred embodiment of the invention.

Example 7
A number of fuel mixtures obtained by mixing the appropriate amounts of dry diethyl ether, ethanol and water have been used as a fuel for an Audi 1.9 liter turbo diesel engine. About 2 w% of two-stroke oil was added to the fuel for lubrication in the engine. No emulsifier was added. The filled symbols in Fig. 2 represent the fuel compositions with which it is possible to run the engine; the open symbols represent fuel compositions with which it is not possible to run the engine. This shows that the conventional Audi compression ignition engine can run on the fuel compositions that lie in the area above the line in Fig. 1.

The three fuel compositions in this example with which it is not possible to run the engine are 31.7 wt% water and 68.3 wt% hydrocarbon fuel, 21.4 wt% water and 78.6 wt% hydrocarbon fuel, and 15.2 wt% water and 84.8 wt% hydrocarbon fuel. These three compositions all lie within the boundaries of the embodiments of the fuel compositions of the invention disclosed in patent US patent No. 7722688. This
shows that those embodiments not always result in a useful fuel for compression-ignition engines.

**Example 8**

The performance of a compression ignition engine with the fuel mixture of the invention is compared to the performance using a standard diesel fuel. The tests were performed on a standard Audi 1.9 liter turbo diesel engine. The tests were performed by switching the fuel from diesel to the fuel mixtures containing ethanol, diethyl ether and water as disclosed here, without further adjustments of the engine. The fuel mixtures contained about 2 wt% 2 stroke oil for lubrication. The fuel did not contain emulsifiers.

Table 2 shows thermal efficiency in an Audi 1.9 l Diesel Engine together with nitrogen oxides (NOx) emission, and particle emission using conventional diesel fuel and four mixtures of diethyl ether, ethanol and water corresponding to the points A, B, C, and D in Fig. 2, at two different loads of the engine. The NOx and particle emissions are key parameters for emission norms, e.g. the Euro standard.

The data in Table 2 show that the power and efficiency of the engine using the fuel mixtures disclosed in the invention is close to that obtained with conventional diesel fuel. However, the emission of NOx and particles can be considerably reduced, possibly enabling to meet emission standards for diesel engines without further treatment of the exhaust gas.

A large part of the particle emission is due to the presence of the 2 stroke lubrication oil. This is illustrated
in Fig. 3, which displays the measured particle emission during a run in which a fuel mixture containing 28.5 wt % ethanol, 53.5 wt % diethyl ether and 18.0 wt% water containing the 2 stroke oil was replaced by the fuel mixture without the 2 stroke oil. In the period where the engine was operated without the 2 stroke oil, the particle number dropped from about $2.8 \times 10^6$ per cm$^3$ to a level of about $4.5 \times 10^5$ per cm$^3$, corresponding to a reduction of 84%. This shows that application of the fuel mixtures as disclosed here are capable of reducing particle emission to very low levels.

**Table 2**

<table>
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<th></th>
<th>Low power</th>
<th></th>
<th></th>
<th></th>
<th></th>
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<th>High power</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>diesel</td>
<td>A</td>
<td>B</td>
<td>C</td>
<td>D</td>
<td>diesel</td>
<td>A</td>
<td>B</td>
<td>C</td>
<td>D</td>
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<td>Comp (wt%)</td>
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<td></td>
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<tr>
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<td>0.21</td>
<td>0.15</td>
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<tr>
<td>NOx emiss. g/kWh</td>
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<td>2.56</td>
<td>1.13</td>
<td>0.92</td>
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<td>1.65</td>
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<td>Particle</td>
<td>5.41</td>
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<td>1.57</td>
<td>0.90</td>
<td>1.98</td>
<td>7.78</td>
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</tbody>
</table>
CLAIMS:

1. A compression ignition fuel composition comprising diethyl ether (DEE) and water in amounts that correspond to the reaction product of dehydration of anhydrous or hydrous ethanol (EtOH) with water content between 0 and 30 wt% and further characterized by:
   a) a diethyl ether content in wt% that is larger than or equal to 50, and a water content in wt% that is lower than or equal to 50 minus concentration of ethanol in wt% for mixtures containing 0 to 20 wt% ethanol; or
   b) a diethyl ether content in wt% that is larger than or equal to 54 minus 0.2 times the ethanol concentration in wt%, and a water content in wt% that is lower than or equal to 46 minus the 0.8 times concentration of ethanol in wt% for mixtures containing 20 to 30 wt% ethanol; or
   c) a diethyl ether content in wt% that is larger than or equal to 61.5 minus 0.45 times the ethanol concentration in weight percent, and a water content in wt% that is lower than or equal to 38.5 minus the 0.55 times concentration of ethanol in wt% for mixtures containing 30 to 70 wt% ethanol.

2. The compression ignition fuel of claim 1, wherein the ethanol content is higher than or equal to 8 wt%.

3. The compression ignition fuel composition of claim 1 or 2, wherein the diethyl ether content in wt% is higher than or equal to 145 minus 4 times the ethanol content in weight percent for mixtures containing ethanol.
4. The compression ignition fuel composition of claim 1, having a composition in wt% as shown in the following Table:

<table>
<thead>
<tr>
<th>DEE</th>
<th>E1OH</th>
<th>water</th>
</tr>
</thead>
<tbody>
<tr>
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<td>12</td>
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<tr>
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<tr>
<td>61.5</td>
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<tr>
<td>57.9</td>
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<tr>
<td>45.8</td>
<td>38</td>
<td>16.2</td>
</tr>
</tbody>
</table>

5. The compression ignition fuel according to anyone of the preceding claims further comprising lubricants, emulsifiers and/or antioxidants.
Without 2 stroke oil
FIG 1

Diethyl ether

Water

Ethanol