



# Engine testing of novel diesel fuel detergent–dispersant additives<sup>☆</sup>



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## HIGHLIGHTS

- Novel thermally and chemically modified succinimide-amides had been synthesized.
- The effectiveness of novel and commercial fuel detergent additives has been compared.
- The engine bench tests had been performed for additivated fuels.

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## ABSTRACT

HPCR (High Pressure Common Rail) design features of modern fuel injection systems and engine working conditions force the use of fuels with good thermooxidative properties, very low solids and metallic elements content, satisfactory resistance to microbial contamination and, above all, very effective detergent performance to prevent deposit formation and its accumulation in modern HSDI (High Speed Direct Injection) diesel engines. The paper presents the state of testing of novel detergent additives for diesel fuels, succinimide-amide type, synthesized at INiG compared to commercial additives and base diesel fuel results. The dynamometer engine tests were performed on the Peugeot XUD 9A/L and on HSDI (Ford Duratorq 2.0i 16V TdCi and Fiat 2.0 JTD) tests engines, equipped with indirect injection (the first one), and with HPCR fuel injection systems (the last two).

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## 1. Introduction

Changes introduced in recent years in diesel engines construction concerned to commonly used in direct combustion systems, high pressure common rail design fuel injection and significant structural changes of the fuel injection systems (multi-hole injectors with reduced diameters and hole as well as injection orifice entry geometry and precise control split dose injected under high pressure fuel). As a result, it improved both quantitative and qualitative fuel atomization in the engine combustion chambers which resulted in a reduction of fuel consumption and lower tail pipe emission. Construction of injection equipment operating at pressures reaching higher than 2000 bar, resulted in an increase in the temperature of the fuel injection system of some elements (e.g. injector tips) up to approximately 250–350 °C.

High, variable temperature of fuel flowing through the injection apparatus can accelerate its degradation, and enhance the tendency to form deposits both external, causing nozzle coking of fuel injector tips and internal, Internal Diesel Injector Deposits (IDID)

[1–3]. Coke or polymeric deposits on the surfaces of the tapered ends of the nozzle slots and injector tips lead to leaks and so called leaking nozzles which in combination with external sediment accumulating on the ends of nozzles, around the fuel spray orifices, adversely affects fuel atomization characteristics, extent and shape of the spray jet and fuel spray droplet size. The tubular nozzle deposits interfere with the flow of the fuel sprayed at high pressure (sometimes above 2000 bar), change the size of the dose, and significantly degrades the quality of the spray.

The end result of the above adverse effects may decrease engine power, increase the unwanted harmful emissions, fuel consumption and, in extreme cases, cause engine failure. Operation conditions and the above-described design features of modern engine injection systems HPCR type, enforce the use of modern diesel fuel with high oxidation and temperature stability, resistant to microbial contamination, low impurities and metals traces content and, above all, containing very effective detergent additives to prevent external and internal deposits formation. Adverse phenomena associated with the exploitation of the fuel can be prevented by various methods, including the use of detergent active substances. The use of such substances (additives) does not interfere with the above mentioned chemical processes, but it prevents the accumulation of deposits produced as a result thereof as well as removes

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the already accumulated (these are processes called “keep clean” and “clean up”).

The role of these substances is to produce a protective layer on the metal surface, preventing the formation of precursors which initiate the formation of aforementioned deposits. The chemical compounds used as detergent active substances are organic compounds of defined structure usually containing nitrogen atoms. Due to their structure, they are strongly polar, and this property provides them with on one hand detergent properties, and on the other, allows adhesion to metal surfaces. The mechanism of action of the above-mentioned substances is complex and does not depend only on the polarity or nitrogen content. Fig. 1 presents the most commonly used substances having detergent–dispersant properties.

The main feature of organic compounds used as active ingredients (additives) with detergent properties, as mentioned, is the presence of nitrogen. It is important both as the total nitrogen content (expressed in %) as well as the so-called basic nitrogen content expressed in mg KOH/g. Strong correlation between these two quantities is the essence of detergency action. Information on the expected effectiveness of a given substance can also give polarity and polarizability. The average molecular weight of detergent substances should range from 500 to 5000 daltons. Besides the above-mentioned physicochemical properties, assuming satisfactory detergency, it is necessary to obtain adequate thermal properties [17–19], and analysis of the progress curves of thermal decomposition temperature and heat flow curves, with respect to the temperatures prevailing in the critical engine elements of CI engines. Bench engine tests are the most comprehensive, and credible way for the definitive assessment of these fuel properties, under the same conditions as close as possible to that of real operation. The current knowledge of the chemical nature of the active substances allows to limit research to a relatively small group of chemical compounds as well as their internal modifications, which is extremely important in achieving highly efficient fuel detergents.

The main purpose of this work was synthesis of effective modified detergent additives to preventing external and internal deposits formation and their effectiveness evaluation in engine tests.

## 2. Materials and methods

### 2.1. Materials used in the test

#### 2.1.1. Base diesel fuel

This base (not additivated) fuel (Table 1) has been labeled as DF 0.

#### 2.1.2. Additivated diesel fuels

At first is recommended the preparation of a pre-mixture to easy the fuel additivation e.g. significant difference between the masses of fuel and additive and their viscosities. Additivated diesel fuels were prepared by mixing at room temperature an appropriate amount of not additivated diesel fuel with a suitable amount of the pre-mixture (ratio fuel:additive in the pre-mixture was 9:1) in a reactor equipped with a mechanical stirrer during one hour. Dosage of tested additives was 100 mg/kg of fuel.

#### 2.1.3. Thermally modified succinimido-amide

Succinimido-amides are modified thermally by subjecting them to a conditioning process at a temperature range from 50 to 130 °C. Conditioning process is carried out until the stabilization of the intensity of the IR spectrum band varied from 1650 to 1694  $\text{cm}^{-1}$  and the ratio of the intensity of the IR spectrum band varied from 1650 to 1694  $\text{cm}^{-1}$  to the intensity of the IR spectrum band at 1705  $\text{cm}^{-1}$ , therefore the intensity ratio of amide to imide IR spectrum band from 1: 0.5 to 1: 1.5, preferably from 1: 0.7 to 1: 0.9. Probably during the thermal conditioning process a five-membered imide ring opens forming the reaction center on the carbonyl carbon atom. At this reaction center a second succinic anhydride molecule can be joined to form an amide bond between a primary or secondary nitrogen atom and a carbonyl group (Fig. 2).

Technology of thermally modified succinimido-amide is protected by patent law – Application No. P.398289 [20]. A process for obtaining thermally modified succinimido-amide (additive A) was carried out at 60 °C for 12 h until the ratio of IR spectrum band intensity of amide to imide 1:0.5 was achieved.

#### 2.1.4. Unmodified succinimido-amide

A method for obtaining unmodified succinimido-amide is well known and described [13,16]. Succinimido-amide for tests is obtained by acillating polyalkylenpolyamine alkenylsuccinic anhydride having a molecular weight from 750 to 2500 daltons. Depending on the molar ratio of polyamine to the anhydride of from 1:1 to 1:3 are obtained mono-, bis- or trissuccinimido-amides (Fig. 3).

The synthesis of unmodified monosuccinimido-amide (additive B) was carried out in a hydrocarbon solvent in the presence of an acid catalyst. The synthesis was carried out for 5 h at the reflux temperature of the solvent. The resulting product was subjected to azeotropic distillation to achieve water content less than 100 mg/kg. IR spectrum of unmodified succinimido-amide was

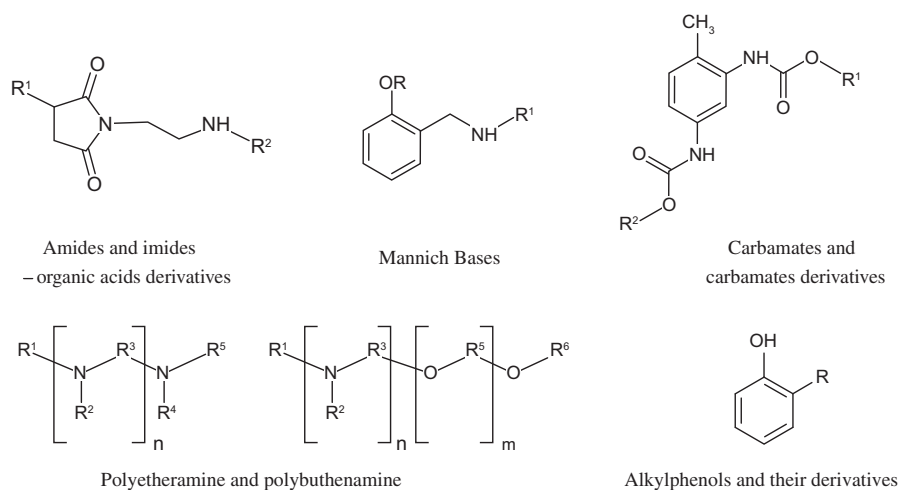


Fig. 1. The most commonly used substances having detergent–dispersant properties [4–16].

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