

## Production and utilization performance of a glycerol derived additive for diesel engines

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### ABSTRACT

Oxygenated additives have been the subject of much research because they notably improve the fuel characteristics and combustion performance. Moreover, there is a tendency to use oxygenated additives derived from biomass. In the case of glycerol, previous studies have shown that oxygenated compounds are generated by dehydration, decomposition and isomerization reactions from glycerol and its intermediaries, where the selected route of these reactions depends on the type of catalyst used.

In this work, the liquid phase obtained during the catalytic glycerol decomposition at 400 °C using a basic catalyst was characterized by GC and GC–MS. This phase is constituted mostly by highly oxygenated compounds of known energetic use. After a drying process, the effect of the glycerol condensates as an additive in diesel–biodiesel (B5) engines at the 0.2% (v/v) concentration was evaluated. The physical properties of the fuel and the mechanic, thermodynamic, and environmental performance of the stationary diesel engine were analyzed in the current study. The presence of the additive decreased the pour point of diesel and the amount of particulate matter generated during combustion.

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### 1. Introduction

Vehicular traffic is currently one of the principal sources of polluting gases and particulates that are emitted to the atmosphere. Therefore, the standard specifications for fuels are becoming more demanding in relation to the established limits for this type of emissions which are highly harmful to health [1,2].

The use of diesel engines has dramatically increased in Europe as well as in some Latin American countries [3]. Therefore, it is necessary to search new technologies in order to improve the combustion performance of these types of vehicles. Diesel fuel is known to produce significant emissions of pollutants, specially NO<sub>x</sub> and particulate matter (PM) [4]. Two of the most employed alternatives to improve the diesel quality are blending it with products generated from biomass (i.e. diesel–bioethanol and diesel–biodiesel blends) and the use of additives to enhance specific properties of diesel [5–7]. Oxygenated additives have been the subject of much research because they notably improve the combustion process and therefore the engine performance [5]. Some of the most widely used oxygenated compounds are ethanol, acetoacetic and dicarboxylic acid esters, dimethyl ether, methyl tert-butyl ether, dimethylcarbonate, dimethoxy methane, dimethoxy ethane, dimethoxy propane, N-octyl nitramine [6]. Nowadays, there is a tendency to use oxygenated additives derived from biomass such as

ethanol, biodiesel, and some glycerol derivatives like diethylene glycol, propylene glycol, diethylene glycol, dimethyl ether, among others [6,8].

Currently there is a need to find alternatives for using the large amounts of glycerol obtained during biodiesel production. It is necessary to develop processes which generate higher added value products derived from glycerol. One of the promising applications is the chemical transformation of glycerol to obtain derivatives with energetic potential that can compete with those traditionally produced from petroleum such as tertiary alkyl ethers (MTBE and TBE) which currently present some controversy [8]. There are reports about additives generated from glycerol which are able to improve the viscosity of liquid fuels, the octane and/or cetane number according to the used engine type, also decrease the cloud point, and reducing polluting emissions, among others [9–11].

The transformation of glycerol has focused mainly on esterification routes (specifically on the glycerol acetylation) and the glycerol–ether or glycerol–glycerol etherification [12,13]. Previous studies have shown that oxygenated compounds are generated by dehydration, decomposition and isomerization reactions from glycerol and its intermediates. The selected route of these reactions depends on the type of catalyst used. Acid catalysts have been the most widely used because they favor the formation of high-value products such as acrolein [14,15]. On the other hand, the use of basic catalysts has not been much explored until this moment with glycerol reactions, but it is known that the basic sites should play a role in the catalytic activity for the dehydration of alcohols [16,17].

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The purpose of this study was to obtain an additive with high oxygen content from the catalytic decomposition reaction of the glycerol using a basic catalyst and to evaluate the effect of its addition to diesel in a stationary diesel engine.

## 2. Material and methods

### 2.1. Production of the additive

The additive was obtained from the catalytic glycerol decomposition. This reaction was carried out in a fixed bed reactor at atmospheric pressure. The experimental setup is shown in Fig. 1. The catalyst  $\text{Ni/La}_2\text{O}_3$  was prepared through the in situ reduction of the  $\text{LaNiO}_3$  perovskite following the described methodology in previous works reported [18,19].

An aqueous solution of glycerol at 50% (v/v) was employed during the reaction which was vaporized before entering the reactor. A flow of 20 mL/min of argon was used as carrier gas. The evaporator was maintained at 290 °C and the reactor at 400 °C throughout the whole reaction. Liquid phase products were recovered using a cooled trap of water-ice located at the reactor outlet. The products thus obtained were dehydrated in a vacuum desiccator over phosphorus pentoxide, which was used as drying material. Gas chromatography-GC – (Agilent 6890 N) and GC-MS (Agilent GC 7890A – MS Agilent 5975 C) techniques were used to separate and identify the principal compounds in this phase. The quantification of the generated products from this reaction will be presented somewhere else.

### 2.2. Engine test

To assess the effect of the additive in the diesel fuel, the mechanical, thermodynamic, and environmental performance of an engine were evaluated comparatively when using a fuel of reference (commercial diesel B5) and a blend of this fuel that contains the additive in a concentration of 0.2% (v/v). These tests were performed using an ISUZU 4JA1 diesel engine. It was operated in stationary mode. The engine speed and torque were 2420 rpm and 95 Nm, respectively. The main specifications of the engine are given in Table 1.

**Table 1**  
Automotive diesel engine characteristics.

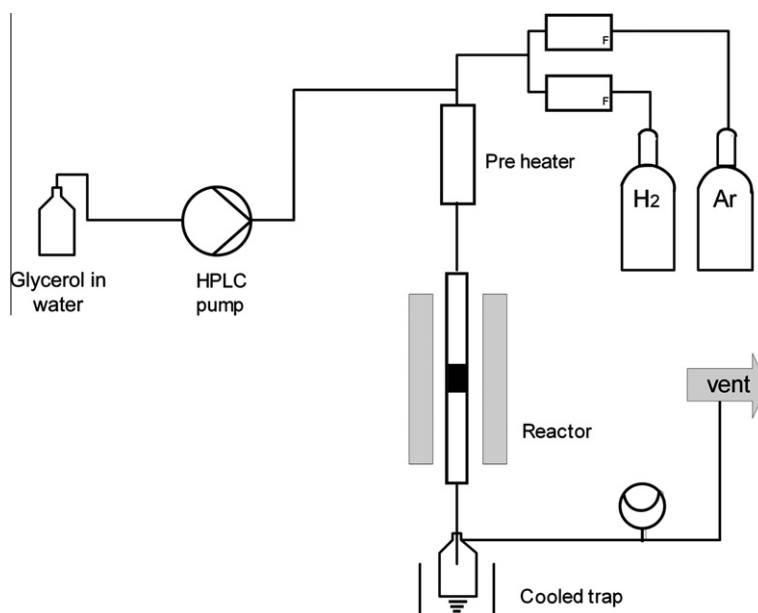
Item	Specification
Type	Direct injection
Swept volume	2499 cm <sup>3</sup>
Configuration	4 in line cylinders
Diameter × stroke	93 mm × 92 mm
Compression ratio	18.4:1
Rated power	59 kW (80 hp) at 4100 rpm
Maximum torque	170 Nm at 2300 rpm
Injection pump	Rotating pump

Specific consumption of fuel, effective thermal efficiency and power, and exhaust gases temperature were evaluated to determine the mechanical performance of the engine. Regarding environmental performance, CO, CO<sub>2</sub>, and NO<sub>x</sub> were measured by nondispersive infrared (AVL Dicom 4000), total hydrocarbons (TCH) was measured by flame ionization (thermoFID) and the smoke opacity was measured by a partial flow smoke opacimeter (optical opacimeter AVL Dicom 4000). Thermodynamic diagnosis of the combustion process was also performed. A general scheme of the configuration used during the engine tests is given in Fig. 2.

Particulate matter generated during the engine tests for both fuels was collected about the same time for further quantification and characterization by elemental analysis (Truspec CHNS Micro Leco), thermogravimetric analysis (TA Q 500) and infrared spectroscopy (PerkinElmer), in order to compare both samples.

## 3. Results and discussions

The decomposition of glycerol at 400 °C using a  $\text{Ni/La}_2\text{O}_3$  catalyst presented a 96% conversion products (liquid and gaseous). The condensed phase presented a yellowish appearance and a distinctive smell but not very intense. According to the elemental analysis, this phase has C 38.1%, H 8.4% and O 53.5%. This indicates the presence of highly oxygenated compounds, which suggests that this phase could be a good quality additive with possible positive effects on the diesel type fuel. Due to the complexity of the sample, liquid yield was calculated taking into account the carbon balance of the reaction. Liquid yield of 74.4% shows that there is a high selectivity towards liquids products at 400 °C.



**Fig. 1.** Experimental setup for the glycerol decomposition experiments at 400 °C.

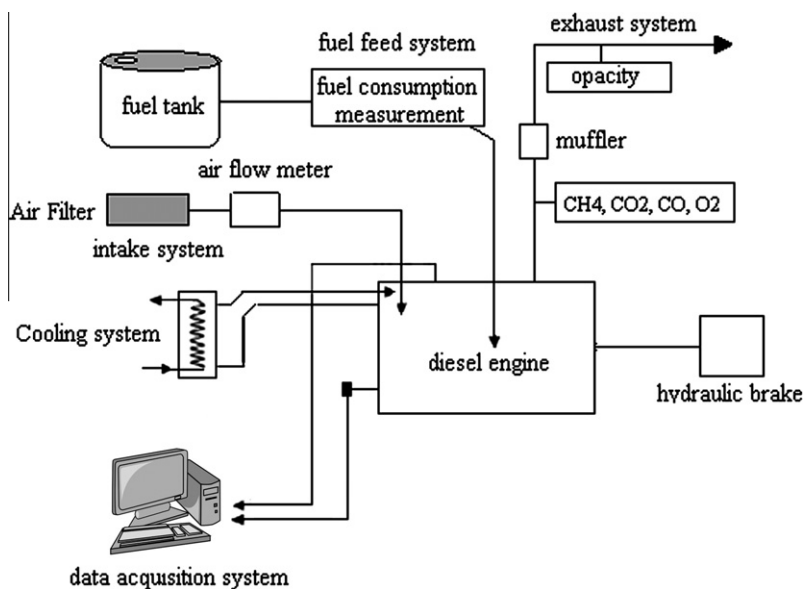


Fig. 2. Experimental setup used in the engine tests.

**Table 2**  
Compounds identified by GC–MS and verified with GC standards.

Name	Structure	Molecular weight (g/mol)
Acetaldehyde		44
Acetone		58
Acrolein		56
Methanol		32
Ethanol		46
Hydroxyacetone		74
Acetic acid		60
1,2 Propanediol		76
Glycerol		92

### 3.1. Product identification in glycerol condensates

In Table 2 gathers the different compounds that were identified by GC–MS in the liquid phase generated in the catalytic glycerol decomposition.

The partial decomposition of glycerol from dehydration reactions is one of the proposed routes to explain the formation of these products, as shown in Fig. 3. Reports in the literature suggest that the selectivity of these reactions is highly related to the acidity of the catalyst [15]. It has been found that the use of acid catalysts promotes dehydration of glycerol in the gas phase into the formation of acrolein.

In this study, Ni catalyst supported on lanthanum oxide was used, which has a strong basic character, while it has been reported that no significant acidic sites were observed in the TPD measurement using  $\text{NH}_3$  [16,20].

Given that previous studies has related the catalytic activity in the dehydration reaction of alcohols with the basic properties of rare earth oxides (REOs) [16,17], it could be suggest that Ni/ $\text{La}_2\text{O}_3$  favors the dehydration of glycerol path toward the formation of 1,2 propanediol, with the formation of acetol as an intermediate. The formation of carbonyl compounds via bond cleavage and isomerization of acetol explains the presence of ethanol, acetaldehyde, acetic acid, and acetone in the analyzed liquid phase, this would agree with the high activity of dissociation related to the strong basicity of  $\text{La}_2\text{O}_3$  [16]. The quantification of these products and the identification of possible oligomers of glycerol will be reported somewhere else.

### 3.2. Additive effect on the fuel characteristics

The diesel–additive blend and the diesel without additive (as reference) were characterized to determinate the effects of the additive obtained from the liquid phase produced during the catalytic glycerol decomposition. Table 3 lists the different evaluated properties.

In general, most fuel properties are not affected by using the additive. Differences between the values found in these properties were not significant, i.e. cetane number and calorific value. However, the pour point (the lowest temperature at which the fluid motion is detected) decreased from  $-12\text{ }^\circ\text{C}$  until  $-18\text{ }^\circ\text{C}$  showing that the additive can improve some of the cold flow properties of diesel, see Table 3.

Table 3 also shows a remarkable flash point decrease (temperature at which the fuel will give off enough vapor to produce a flammable mixture) by adding the additive. This may be a conse-

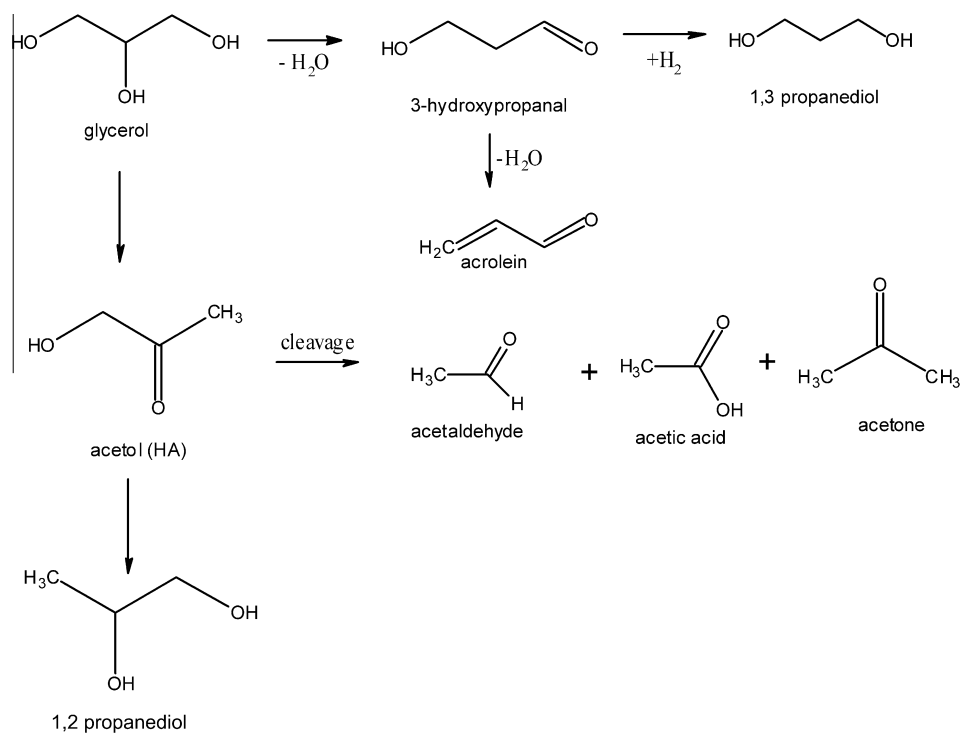


Fig. 3. Catalytic decomposition route of glycerol.

Table 3

Properties of the diesel and diesel–additive blend.

	Diesel	Diesel + additive
Heating power (MJ/kg)	45.02	45.11
API gravity (60 °F)	34.4	34.5
Kinematic viscosity 40 °C (cSt)	3.305	3.295
Flash point Pensky–Martens (°C)	76	66
Cloud point (°C)	–3	–2
Pour point (°C)	–12	–18
Calculated cetane index	49.31	49.47

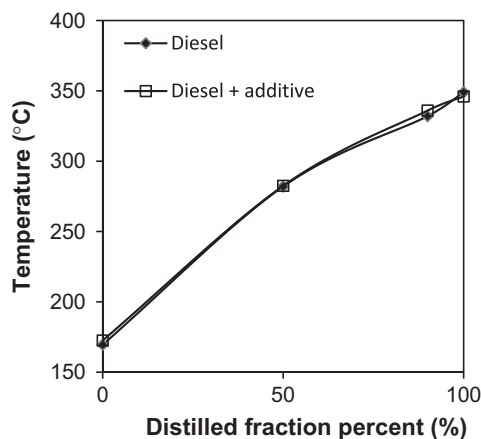


Fig. 4. ASTM D-86 distillation curves.

quence of the presence of alcohols and low molecular weight compounds in the additive [21]. Nevertheless, it is important to note that the diesel–additive blend has a flash point higher than the minimum allowed for fuel handling according to the minimum requirements (52 °C). In addition, fuel volatility is not affected by

the addition of the additive, as shown in the distillation curves in Fig. 4.

On the other hand, the kinematic viscosity decreased in 0.01 cSt which is insignificant comparing the diesel–additive blend with the reference diesel. It could be related to the small amount of free glycerol present in the additive (less than 4%) determined by a GC analysis.

### 3.3. Additive effect on engine performance

#### 3.3.1. Engine mechanical performance

Table 4 shows the mechanical performance of the engine when using the reference diesel and diesel containing the additive. It is worth to note that the use of the additive generated a slight increase in specific fuel consumption, but taking into account the standard deviation of collected data, this increase was not statistically significant at 95% confidence. In general, mechanical parameters of the engine performance were not affected by the use of the additive.

#### 3.3.2. Thermodynamic performance engine

Table 5 shows the results for the analysis of the thermodynamic combustion engine; it is observed that the burning time decreased with the use of the additive.

The maximum combustion pressure in the cylinder was not affected by the use of the additive; a value of 77 bars was obtained for both fuels (see Fig. 5). Maximum pressure combustion was achieved at the same angle for both fuel and corresponds to 5° after top dead center (TCD).

On the other hand, there are differences between the maximum combustion temperatures of gases generated in the combustion chamber, this parameter decreased with the use of the additive as shown in Fig. 6, which could be related to a slight decrease in the time of the premix before combustion. The angle at which the maximum combustion temperature was obtained does not change with the additive, reaching 8° after top dead center (TDC).

**Table 4**  
Comparison of engine mechanical performance.

	Diesel	Diesel + additive
Engine speed (rpm)	2421 (5.91)	2423 (5.44)
Torque (Nm)	9454 (0.87)	94.64 (0.46)
Effective pressure	23.97 (0.17)	24.02 (0.11)
Mean effective pressure (bar)	1.21 (0.01)	1.21 (0.01)
Fuel consumption (mg/s)	1601.47 (23.37)	1617.71 (9.42)
Air consumption (g/s)	62.83 (1.30)	65.29 (0.50)
Equivalence ratio	0.38 (0.004)	0.37 (0.005)
Specific fuel consumption (g/kW h)	240.55 (3.41)	242.50 (2.03)
Exhaust temperature	669.59 (4.11)	670.79 (2.49)

Values in parentheses indicate the standard deviation value calculated among five measurements made during the essay. Test was performed at 2420 rpm and 95 Nm.

**Table 5**  
Comparison of combustion analysis.

	Diesel	Diesel + additive
Maximum combustion pressure (bar)	77.01	77.05
Maximum pressure gradient (bar/°C A)	3.99	4.07
Combustion duration (crank angle, °C A)	30.04	29.85
Maximum combustion temperature (K)	1337	1275

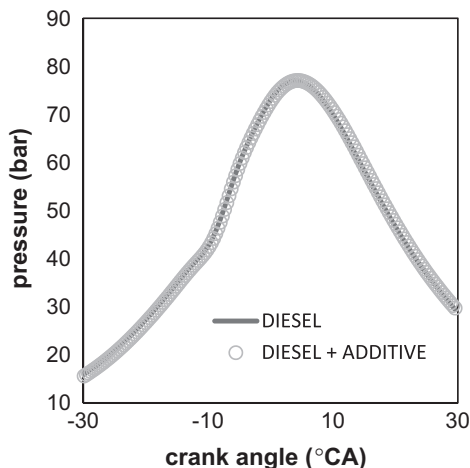
Test was performed at 2420 rpm and 95 Nm.

The analysis of heat release rate is shown Fig. 7. It is a typical profile for diesel engines with mechanical injection control, and suggests that the combustion duration is not changed with the use of the additive, indicating that its presence did not affect the different stages of combustion.

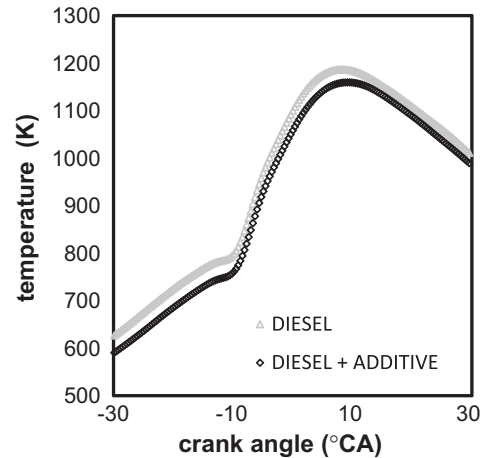
### 3.3.3. Environmental performance of the engine

In order to evaluate the environmental performance of the engine working with the diesel–additive blend, polluting emissions were measured at engine exhaust as shown in Table 6. CO, CO<sub>2</sub> and THC emissions did not vary significantly with the additive addition. On the other hand, smoke opacity decreased by 7% and NO<sub>x</sub> emissions decreased by 2%, which is explained by the drop in the maximum combustion temperature, which affects the thermal mechanism (Zeldovich), through which it generates most of this pollutant.

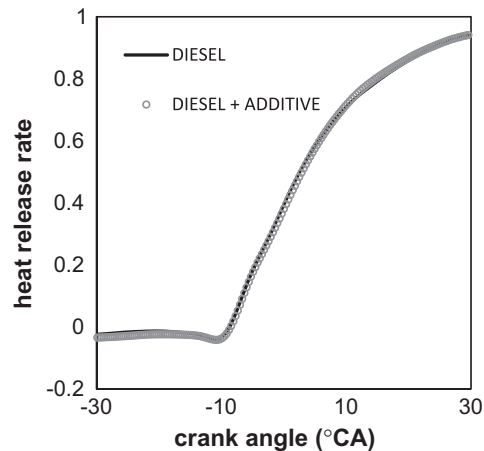
In relation to the amount of particulate matter generated during engine combustion, the additive decreased by 23% the production of soot. This effect may be related to the additive nature, which



**Fig. 5.** Instantaneous cylinder pressure.



**Fig. 6.** Average temperature in the combustion chamber.



**Fig. 7.** Accumulated heat release rate.

**Table 6**  
Comparison of the polluting emissions of the motor.

	Diesel	Diesel + additive
% CO	0.01 (0)	0.01 (0)
% CO <sub>2</sub>	5.60 (0.07)	5.68 (0.04)
NO <sub>x</sub> (ppm)	1574.2 (16.19)	1542 (23.69)
THC propane (ppm)	14.55 (0.76)	14.87 (1.55)
Smoke opacity	3.54 (0.28)	3.30 (0.27)
PM (mg)	1.8 (0.05)	1.38 (0.15)

Values in parentheses indicate the standard deviation value calculated among five measurements made during the essay. Test was performed at 2420 rpm and 95 Nm. Particulate matter samples were collected at 15 min intervals throughout the test.

contains large amounts of oxygenated compounds that promote the combustion process.

### 3.4. Characterization of particulate matter

#### 3.4.1. Thermogravimetric analysis

Thermogravimetric analysis of PM samples produced during the combustion process of diesel and the diesel–additive blend shows three main events: the first one is related to the loss of moisture and volatile compounds adsorbed on the soot which evolve until 120 °C, the second event corresponds to the desorption of higher molecular weight compounds (primarily polycyclic aromatic hydrocarbons, PAHS) which evolve between 120 °C and 800 °C in

inert atmosphere. Finally, the third event is the oxidation of carbon remaining inside the structure, related to existing graphitic domains, this process occurs at 800 °C when the oxidizing atmosphere takes place. In general, PM from diesel engine consists of mainly three components: Soot generated during combustion, heavy HC condensed or absorbed on the soot, and sulfates [22].

On the other hand, a comparison of both profiles suggests that the evolved volatiles are different. For example, the increase of the fixed carbon percentage in soot (see Table 7) indicates an enhancement of the graphitization degree of the particulate material when the additive is added to the diesel. The residue value found for both samples is possibly related to the sulfates generated during the combustion of a diesel engine [22].

### 3.4.2. Soot infrared characterization

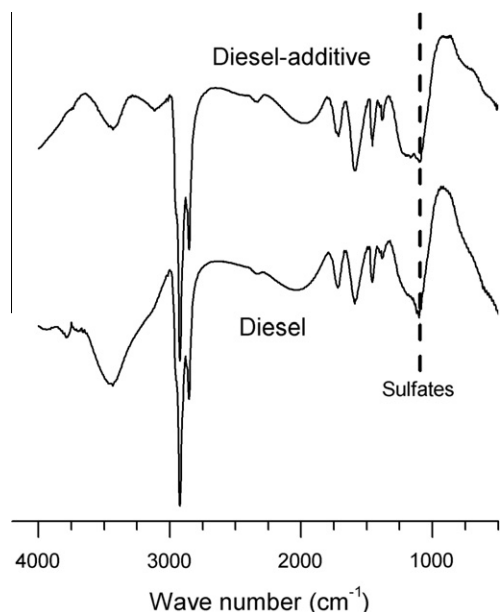
The infrared spectra shown in Fig. 8, contain typical functional groups of the soot samples, finding characteristic signals of C–H stretching (3300 cm<sup>-1</sup>), symmetric–asymmetric CH<sub>3</sub> and CH<sub>2</sub> stretching (2975 cm<sup>-1</sup>, 2925 cm<sup>-1</sup>, 2850 cm<sup>-1</sup>), C=O carbonyl stretching (1720 cm<sup>-1</sup>), C=C stretching of aromatic and alkenes (1595 cm<sup>-1</sup>), CH<sub>2</sub> and CH<sub>3</sub> symmetric–asymmetric deformation (1450 cm<sup>-1</sup>, 1380 cm<sup>-1</sup>), C–C and C–H deformations, and C–O–C stretching (1000 cm<sup>-1</sup>, 1300 cm<sup>-1</sup>) [23]. The signal observed at 1100 cm<sup>-1</sup> is characteristic of sulfate groups. Both spectra do not present significant changes.

### 3.4.3. Elemental analysis

The elemental analyses for the PM samples obtained during the combustion process of diesel and the diesel–additive blend are presented in Table 8. It is observed that in the presence of the additive

**Table 7**  
TGA analysis of the diesel and diesel–additive blend.

	Temperature range	PM (diesel)	PM (diesel + additive)
% Moisture	20–120 °C (N <sub>2</sub> )	2.01	1.99
% Volatiles	120–800 °C (N <sub>2</sub> )	32.93	27.74
% Fixed carbon	800 °C (air)	62.31	66.13
% Residue	800 °C (air)	2.75	4.14



**Fig. 8.** IR spectra for PM samples obtained during the combustion process of diesel and the diesel–additive blend.

**Table 8**  
Elemental analysis of the diesel and diesel–additive blend.

	PM (diesel)	PM (diesel + additive)
% C	85.99	92.40
% H	2.99	2.79
% N	0.84	0.80
% O	10.18	4.01
(C/H) total	2.39	2.75

the carbon percentage increases and the hydrogen and oxygen percentages decrease which is in agreement with the thermogravimetric analysis where an enhancement of the fix carbon percentage as well as a decrease of the volatiles were observed. Moreover, Table 8 shows that the total (C/H) ratio increases with the addition of additive, which implies an increase in the aromatic character of the PM.

Additionally, elemental analysis of sulfur for the sample of PM obtained during the combustion of diesel corroborates the presence of sulfur (1.5%) as it can be inferred from the Infrared analysis. On the other hand, considering the remarkable similarity between both spectra in Fig. 8, specifically the signal around 1100 cm<sup>-1</sup> corresponding to sulfates, it could be possible to confirm the presence of sulfur also in the diesel–additive blend although for this latter the elemental analysis could not be carried out.

## 4. Conclusions

The production and performance of a glycerol derived additive for use with diesel fuel has been described. The liquid phase obtained during the catalytic glycerol decomposition contains large amount of oxygenated compounds with known energetic use. This is the case of acetol and 1.2 propanediol, which were used as diesel fuel additives to improve the environmental performance of the engine. It was shown that the additive significantly decreases the amount of particulate matter, which is the major pollutant emissions generated by using a diesel fuel, and reduces the pour point of fuel to improve its flow properties cold.

In the other hand, it could be suggest that use of the additive affects the chemical nature of particulate matter, increasing its aromatic character.

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