Power System Technology
ISSN:1000-3673

Received: 16-03-2025 Revised: 05-04-2025 Accepted: 02-05-2025

Experimental Analysis of Low Viscosity Industrial Oily Waste Energetic Recovery

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Abstract: This paper presents an experimental investigation on industrial lubricants transformation to synthetic fuels and their use in a direct diesel diesel engine. Catalytic extraction was achieved using methanol and potassium hydroxide. Industrial oils are mainly pumps lubricant Tiska 32, compressors lubricant Torba 32 and turbines lubricant Torada 32. Biofuel extracted from waste cooking oil and conventional diesel fuel are taken for comparison. Density, viscosity, acidity and flash point of biofuel, synthetic fuels and their blends are measured and discussed. Gas chromatography associated to mass spectroscopy have been done to determine synthetic fuels and the biofuel chemical composition. The use of blends with diesel fuel seems to be required. Three ratios of 15%, 30% and 45% of synthetic fuels and biofuel are tested on a direct-injection diesel engine in order to compare performances, fuel consumption and emissions obtained when the engine is powered with conventionnel diesel fuel, biofuel and synthetic fuels.

Keywords: *Diesel engine, Industrial waste oil, Biofuel, Tiska32, Torba 32, Torada32, BSFC, NOx.*

1. Introduction

Nowadays, internal compression engines, conventional fuels and biofuels are at the center of critical issues and constitute a thin interface between interdisciplinary fields. Indeed, researchers, experts in politics, economics and health are involved together in the urgency of finding engineering processes, strategies but also adequate legislations and policies leading to sustainable, economical and clean energy sources for the internal combustion engines. Biodiesel is an oxygenated fuel considered as an alternative to conventional diesel for use in compression ignition engines without any modification to the engine structure or injection system. Biodiesels can be extracted from edible and inedible plant oil wastes [1-5]. However, the acidity, viscosity and density of biodiesels are high compared to those of conventional diesel fuel and engine exhaust contains higher levels of NOx when fueled by biofuels [6-8]. As a result, research is opening up on different topics such as the prediction and optimization of

Received: 16-03-2025 Revised: 05-04-2025 Accepted: 02-05-2025

the physical properties of biofuels and the engineering of extraction processes [9-11], the experimental use on engine benches and the analysis of NOX, CO, unburned hydrocarbons and particulate emissions [12, 13]. Despite of this, it can be noted that the process of extracting biodiesels and synthetic fuels from various sources is currently expensive and more focused on waste oil recovery. Furthermore, their use is highly recommended for static diesel engines operating at limited operating speeds and loads which simplifies their optimization issue [14-24]. The present paper focuses on energetical recovery of some industrial waste lubricants by the mean of catalytic transesterification. Thus, synthetic fuels are extracted from Tiska 32, Torba 32 and Torada 32 lubricants. in a former author's work. In a previous work, the author's present the process description of a synthetic fuel extraction from cooling lubricant waste and its use in a direct injection diesel engine [11, 25, 26]. First, the work focuses on thermal evolutions of biodiesel and synthetic fuels liquid densities and viscosities, their total acidity number and flash point measurements. The chemical composition of the biodiesel and the synthetic fuels are provided by gaseous chromatography coupled to mass spectroscopy. Synthetic fuels properties are compared to those of biodiesel and conventionnel diesel fuel ones. Experimental results obtained on an engine bench when a single cylinder diesel engine is filled by biodiesel and the three synthetic fuels blends with the same ratios of 15%, 30% and 45%. Specific carbon monoxides, nitrous oxides in addition to brake fuel consumption are compared.

2. Catalytic cracking process

A multitude of experiments aiming to biodiesel and synthetic fuels production from different waste oils presented in Table 1. It should be noted that the alcohol used is the methanol and the catalyst used is the potassium hydroxide. The present work focuses on results obtained with three industrial lubricants namely Tiska 32, Torba 32 and Torada 32 chosen for their low viscosities.

Table 1. Lubricants list.

Lubricant	Equipment	Use or Conditions
Tiska 32	Small centrifugal pumps	Temperature < 80°C
Torba 32	Volumetric compressors	Low Speeds 2800-3500 RPM
Torada 32	Volumetric compressors	Lubrication

Transesterification goes through several stages after volume measurements and weighing. Of which they are demonstrated as follows [11, 25, 26]:

Received: 16-03-2025 Revised: 05-04-2025 Accepted: 02-05-2025

Volume measurement and preheating at 60°C. Dissolution of the catalyst in methanol. Transesterification on a hot plate above 55 °C under 65°C at high stirring speed. Separation after 24 hours in a decanting ampoule. Washing with water heated at 40°C. Drying after 24 hours at 100°C. The quality of the biofuels extracted from different vegetable sources can be influenced by several factors that can be reflected in its chemical and physical properties [11]. Some physical and chemical specifications of the three industrial oils studied are presented in Table 2. As expected, except Tiska 32, the Torba 32 and Torada 32 industrial oils used in this study are not corrosive since they have a low acidity number and no heavy metals except a small amount of Sulphur and Phosphorus for Torba 32.

Table 2. Lubricants properties.

	Tiska 32	**Torba 32**	**Torada 32**
Viscosity @ 40°C [mm^2/s]	28.8-35.2	28.8-35.2	28.8-35.2
Acidity mg KOH/kg	0.45	0.45	Not indicated
S %	0.048-0.058	0.01-0.014	0
N %	0	0.013-0.018	0
P %	0.022-0.028	0	0
Ca %	0.0033-0.004	0	0
Zn %	0.028-0.036	0	0

Table 3 lists the transesterification reaction yield for each type of oil. The transesterification of used cooking oil seems to be more efficient than that of Torba 32 and Torada 32 oils but less than Tiska 32 oil. However, acceptable values are observed since a minimum value obtained for Torba 32 is above 60%. The Tiska 32 offers the highest efficiency value, followed respectively by the Torada 32 and Torba 32. The European Committee for Standardization (ISO) [27] and the American Energies Society for Testing and Materials (ASTM) [28] request quality specifications for biodiesel as reported in Table 4.

Table 3. Transesterification reactions efficiencies.

Designation	**Reaction Efficiency**
Tiska 32	83%
WCO	78%
Torada 32	68%
Torba 32	62%

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Table 4. EN 14214 [27] & ASTM D6751 [28] Biodiesel Specifications.

Properties	EN 14214	ASTM D6751
Density @ 15 °C (kg/m3)	860–900	880
Viscosity @ 40 °C (mm²/s)	3.5–5.0	1.9–6.0
Acidity Number (mg KOH/g)	>0.50	>0.50
Flash Point (°C)	<101	<130
Phosphorus content (mg/kg)	>4.0	>10
Sulphated ash (% (m/m))	>0.02	>0.02
Sulphur content (mg/kg)	>10.0	S15 >15 S500 >500

3. Physiochemical properties measurement

The liquid density and the liquid kinematic viscosity of the biofuel and synthetic fuels were measured at wide temperature intervals. Biofuel and synthetic fuels were blended at 15%, 30% and 45% by volume, respectively, with commercial diesel fuel. Continuous measurements can be made giving changes in the liquid densities and liquid kinematic viscosities versus temperature.

3. 1. Liquid Density Measurement

The liquid density of the fuels extracted and their blends is measured. The liquid density has been measured by Anton Paar DMA 35 densimeter with following settings: measuring ranges from 0 to 3 g/cm3 and an accuracy of 0.001 g/cm3 at 0.2 °C. Resolution of density and temperature are on the order of 0.0001 g/cm3 and 0.1 °C respectively [26]. Temperature of the sample varied from 0 to 100°C. The thermal liquid density evolution related to the biofuel as well as synthetic fuels blends and compared to the thermal liquid density of the conventional diesel fuel in as represented by Figures 1 to 4.

Received: 16-03-2025 *Revised: 05-04-2025* *Accepted: 02-05-2025*

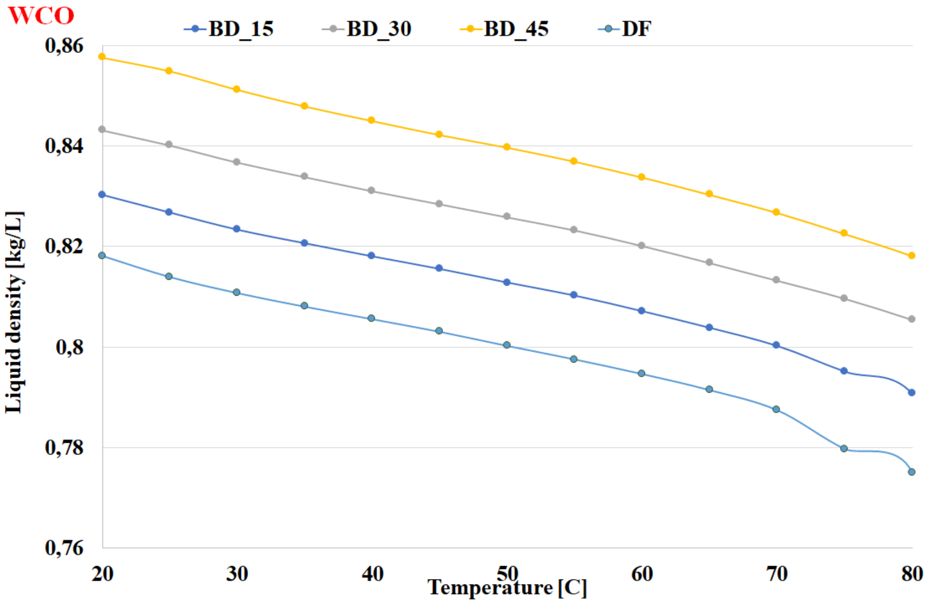

Fig. 1. Biofuel blends liquid density thermal evolutions.

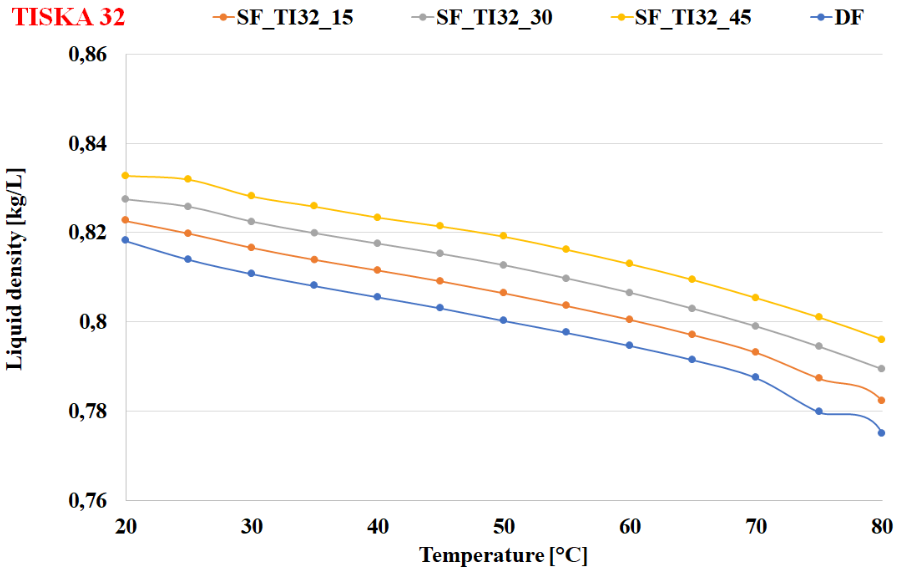

Fig. 2. Tiska32 blends liquid density thermal evolutions.

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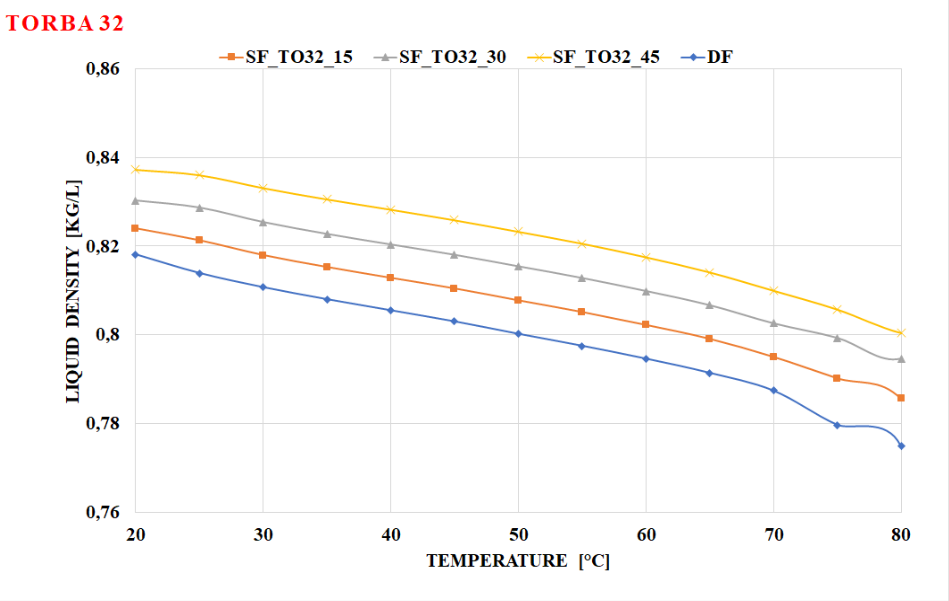

Fig. 3. Torba 32 blends liquid density thermal evolutions.

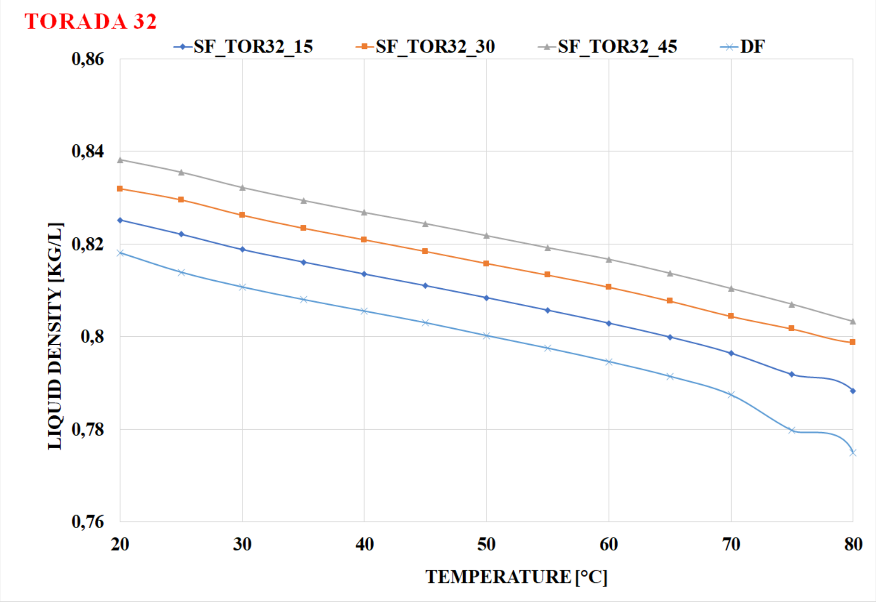

Fig. 4. Torada 32 blends liquid density thermal evolutions.

3. 2. Total Acidity Number

It should be noted that the total acidity number was measured twice for the biofuel and for synthetic fuels. The measurement interval is six months for the same samples. The total measured acidity number related to the biofuel extracted from waste cooking oil and oxygenated fuels extracted from Tiska 32, Torba 32, Torada 32 are gathered in Table 5. Total acidity number measurement is achieved by TitroLine 6000 following ASTM 2896 standard. Considering the standards and limitations, it can be concluded that for used household oils, the biofuel, although due to its low viscosity, cannot be stored beyond 6 months, on the contrary it must be consumed as soon as possible. In contrast to this, the oxygenated fuels extracted from Tiska 32, Torba 32, Torada 32 cannot be used without being blended with commercial diesel fuel, these oxygenated fuels have the merit of being stored for periods of up to 6 months after their production.

Table 5. Synthetic fuels and biofuel total acidity number. (the same samples measured at 6 months interval).

Designation	TAN 1	TAN 2
SF_TI32	0.09	0.13
SF_TO32	0.04	0.31
SF_TOR32	0.04	0.08
Biofuel	0.16	0.58

3. 3. Liquid Viscosity Measurement

The liquid dynamic viscosity of the biofuel and synthetic fuels as well as their blends are measured using a rheometer. The liquid dynamic viscosity of the biofuel and synthetic fuels as well as their blends are measured using RheolabQC with the following specifications: Speed from 0,01 1/min to 1 200 1/min, Torque from 0,20 m Nm to 75 m Nm, Sheer stress from 0,5 Pa to 3 x 104 Pa, Viscosity measuring range from 1 mPa.s to 109 mPa.s, Temperature range from -20 °C to +180 °C. The thermal viscosity evolution as a function of the temperature variation associated with commercial diesel fuel and biofuel obtained from used cooking oils, as well as the variation in liquid viscosities associated with oxygenated fuel extracted from Tiska 32, Torba 32 and Torada 32 oils, are assembled in Figure 5. Table 6 lists the liquid kinematic viscosities of blends at 40 °C for biofuel as well as the oxygenated fuels extracted from Tiska 32, Torba 32 and Torada 32 respectively.

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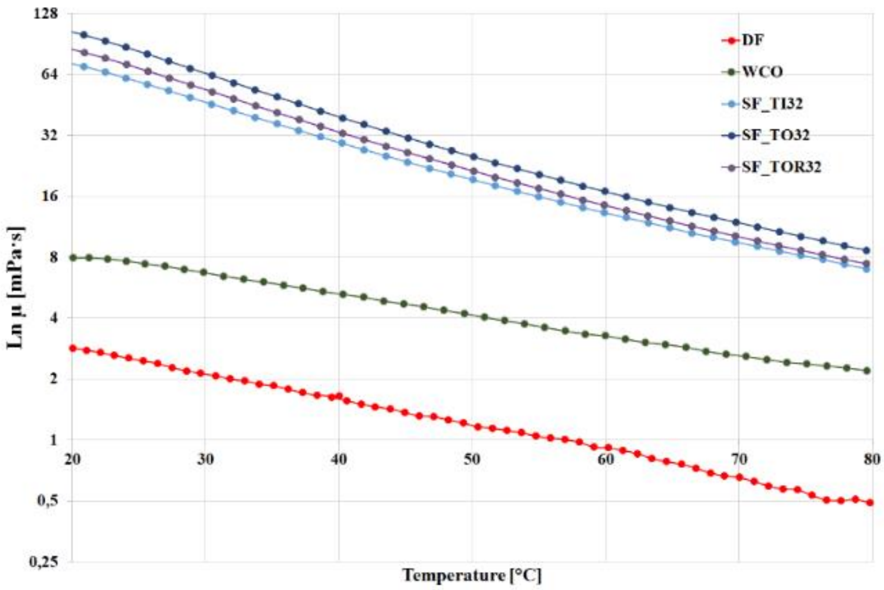

Fig. 5. Studied fuels logarithmic liquid dynamic viscosity thermal evolutions.

Table 6. Kinematic viscosities of blends at 40 °C (unit mm²/s).

Blending ratio	BD	SF_TO32	SF_TO32	SF_TOR32
100	5.66	26.61	45.60	65.07
75	5.01	7.32	7.85	25.91
50	4.37	4.24	1.66	2.09

The liquid kinematic has been measured by KV-6 Viscometer bath with the following specifications: Temperature range from Ambient to 160 °C, Temperature stability at 40°C is ±0.002 °C, Temperature uniformity at 40°C is ±0.003 °C. Figure 6 shows the variation of the synthetic fuels kinematic viscosities measured at 40 °C as a function of blending percentage. The standard viscosity limit level of 5 mm²/s can be achieved at 60%, 75% and 55% for oxygenated fuels extracted from Tiska 32, Torba 32 and Torada 32 respectively. Therefore, a set of three blending percentages was chosen for oxygenated fuels extracted from industrial oils. Blending rates of 15%, 30% and 45% will be prepared and used to be explored on the experimental diesel engine test bench. Table 7 regroup regression law formula which subsequently give an estimated value of the kinematic viscosities of each synthetic fuel blends at 40 °C as function of the blending ratio.

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ISSN:1000-3673

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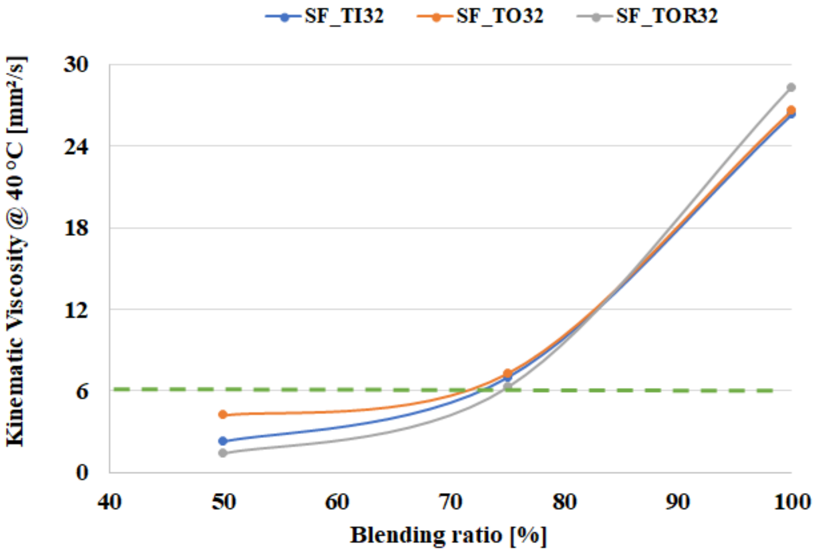

Fig. 6. kinematic viscosities of synthetic fuels function of blending percentage.

Table 7. Kinematic viscosities of synthetic fuels blends at 40 °C regression laws.

Designation	**Power law regression formula**	**R²**
SF_TI32	$\nu_{40} = 2 * 10^{-9} * (BR)^{5.1209}$	0.99
SF_TO32	$\nu_{40} = 3 * 10^{-9} * (BR)^{5.0809}$	0.99
SF_TOR32	$\nu_{40} = 2 * 10^{-9} * (BR)^{5.1272}$	0.99

3. 4. GC-MS characterization

The samples were prepared by injecting into the upper phase a mixture consisting of 1g of substances with 10 ml of heptane and 500 µl of methanoic KOH. The operative conditions can be summarized as follows [25, 26]:

Injector: Temperature: 280°C. Injected volume: 01 µl.

Column: Type: HP-5MS.

Dimensions: long 30 m * D int 0.25 mm * film thickness 0.25 µm.

Stationary phase: 5% Phenyl 95% dimethylpolysiloxane. Oven temperature: 42°C for 05 min, 10°C/min up to 300°C; isothermal 20 min; Analysis time: 50 min

Carrier gas: pure helium 6.0

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Flow rate: 02 ml/min

Mass detector: Solvent delay: 3.5 min. Interface temperature: 270°C.

Type of mass analyzer: Quadrupoles. Spring temperature: 230°C.

The spectra resulting from the chromatographic characterization of the biofuel and the oxygenated fuel extracted from Torba 32 and Torada 32 are shown in Figures 7 to 9.

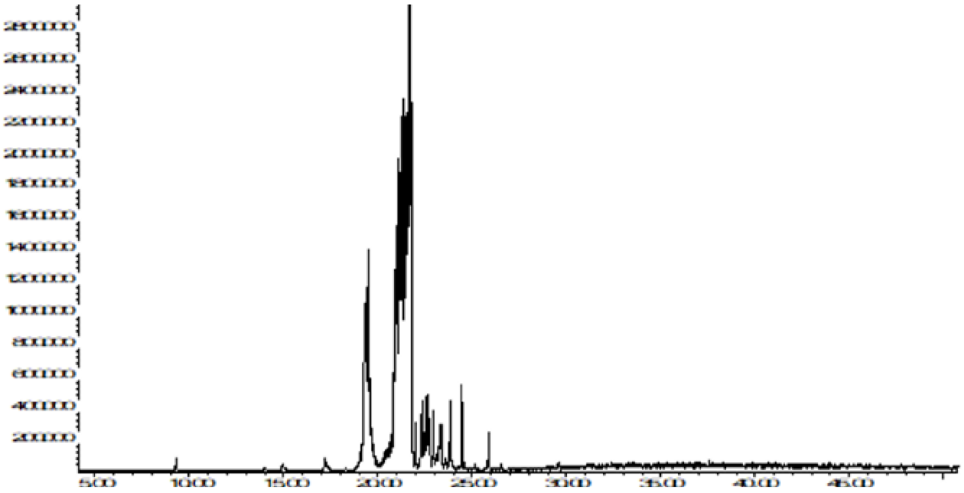

Fig. 7. WCO biofuel chromatography spectrum.

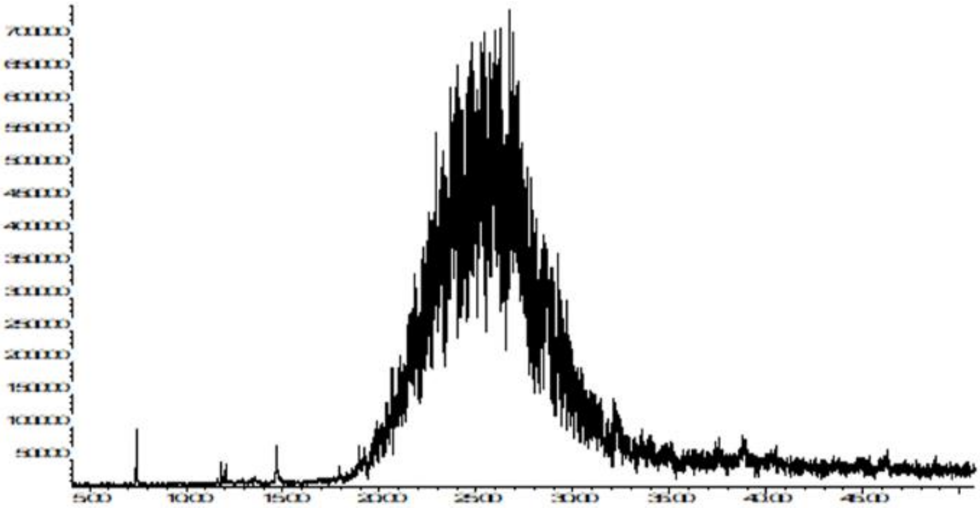

Fig. 8. SF_TO32 chromatography spectrum.

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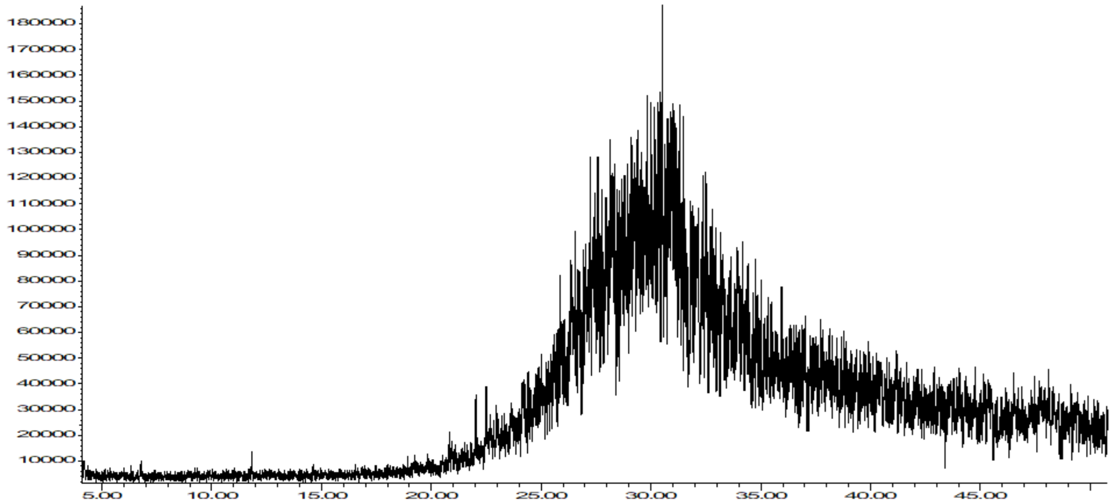

Fig. 9. SF_TOR32 chromatography spectrum

The chemical species involved in the composition of the substances analyzed are presented in Table 8.

Table 8. Biodiesel and synthetic fuels composition.

Source	Components
Biofuel	$C_9H_{18}O_2$ $C_{10}H_{20}O_2$ $C_{15}H_{30}O_2$ $C_{16}H_{32}O_2$
Torba 32	$C_8H_{18}O$ $C_{11}H_{10}$ $C_{10}H_{10}O_4$
Torada 32	$C_{18}H_{36}O_2$

3. 5. Flash Point

Measured flash points related to oxygenated fuels extracted from Tiska 32, Torba 32 and Torada 32 and their blends are illustrated in Figure 10. It can be easily demonstrated that all values are above the limits of ASTM D6751 and EN 14214.

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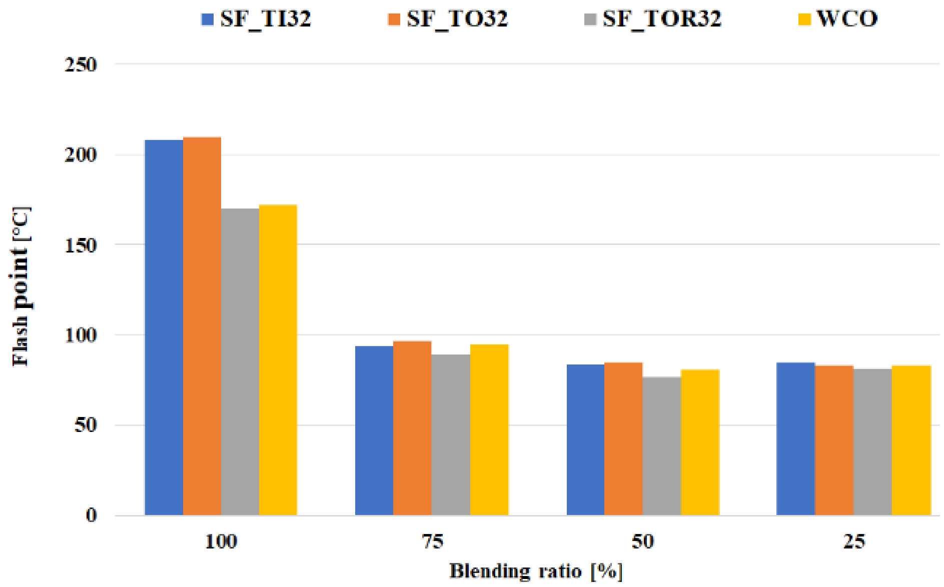

Fig. 10. Measured flash point of biofuel and synthetic fuels blends.

4. Engine test bench

For the tests, a single-cylinder diesel engine of the Kipor 178FWX brand was made available to us, air-cooled, with a power of 4.5 kW running at 3600 rpm. The basic data for this engine are shown in Table 9.

Table 9. Engine test bench specifications.

Designation	Value
Model	Kipor 178FWX
Type	1 cylinder, 4-stroke, air-cooled, direct injection
Bore	78 mm
Stroke	62 mm
Compression ratio	20:1
Volumetric Capacity	296 cm3
Power Output	4.5 kW at 3600 RPM
Fuel Temperature	40 °C
Fuel injection timing	18 CAD BTDC

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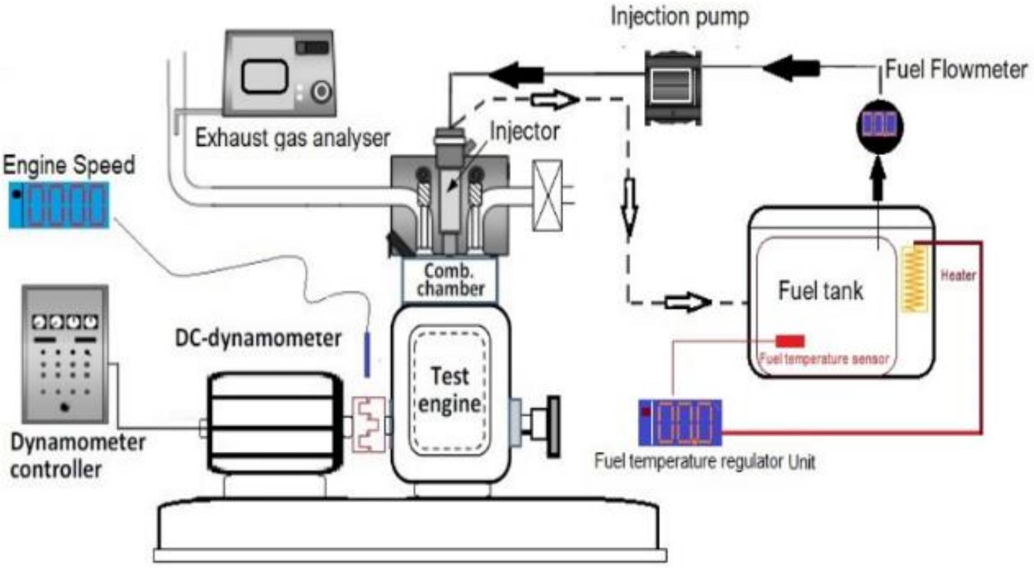

Fig. 11. Experimental set-up.

The experimental engine test bench is illustrated in Figure 11, which includes a single-cylinder diesel engine with direct injection, an electric generator as engine load and the exhaust gas analyzer used for NO_X, CO and exhaust temperature measurements. A measurement chain is used to note the current and voltage generated by the electric generator to calculate the power absorbed, torque and specific consumption. Exhaust gas analyzer device used has the following measurement ranges: O_2: 0 to 21%, CO: 0 to 10000 ppm, NOx: 0 to 1000 ppm and the maximal exhaust gases temperature is 800 °C.

5. Results and discussion

A set of measurements on the engine test bench was performed. In order to better understand engine performance and emissions, the results obtained by biodiesel and synthetic fuel blends must be compared with those obtained by diesel oil. The experimental results are obtained when the diesel engine is operating at 1600 rpm from the engine brake at full load. Particular attention was paid to the concentrations of CO and NOx emitted by the engine as well as its brake-specific fuel consumption when the engine was operated with biodiesel and blends of 15%, 30% and 45% BD15, BD30 and BD45 for biodiesel and SF15, SF30 and SF45 for synthetic fuels. Figures 12, 13, and 14 show the variation in BSFC at 1600 rpm as a function of BMEP for biodiesel, synthetic fuel blends, and diesel fuel.

Received: 16-03-2025 Revised: 05-04-2025 Accepted: 02-05-2025

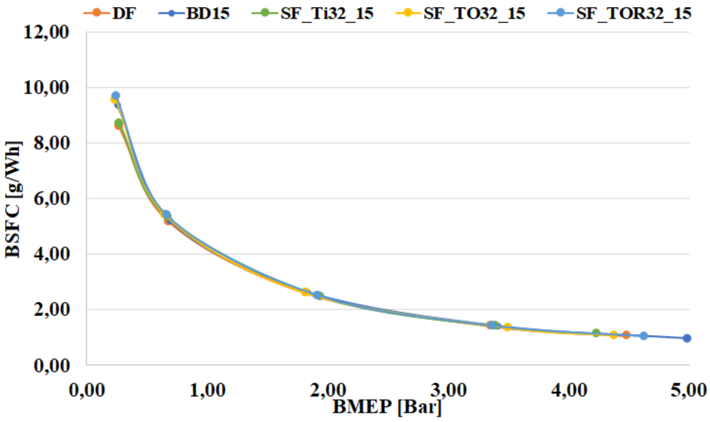

Fig. 12. 15 % blends BSFC evolutions at 1600 RPM.

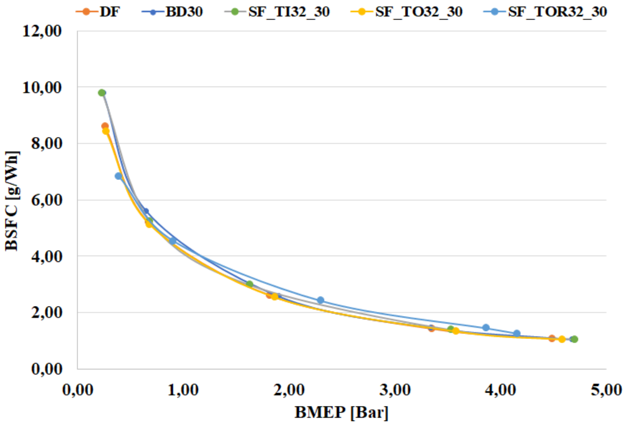

Fig. 13. 30 % blends BSFC evolutions at 1600 RPM.

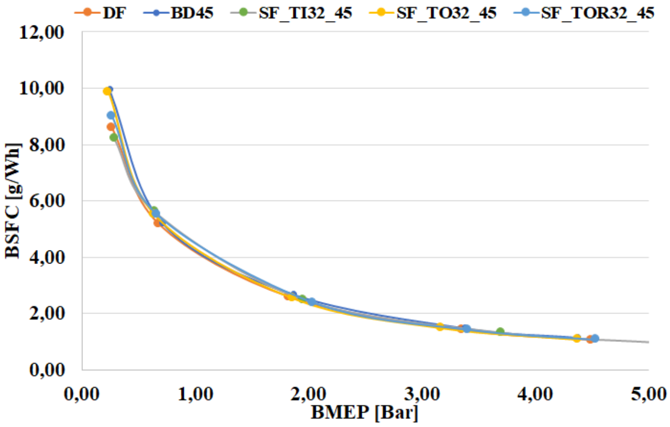

Fig. 14. 45 % blends BSFC evolutions at 1600 RPM.

Received: 16-03-2025 Revised: 05-04-2025 Accepted: 02-05-2025

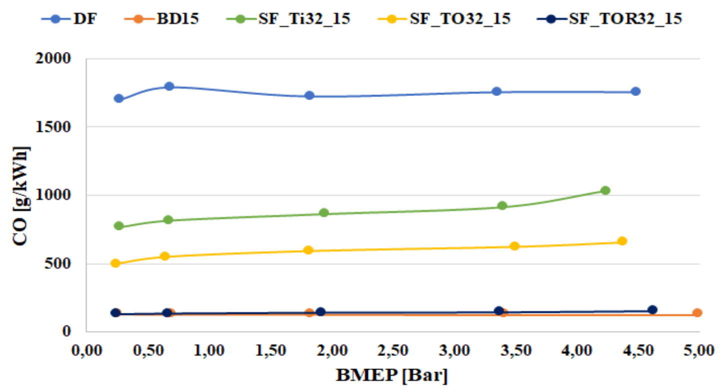

Fig. 15. 15 % blends specific CO emission evolutions at 1600 RPM.

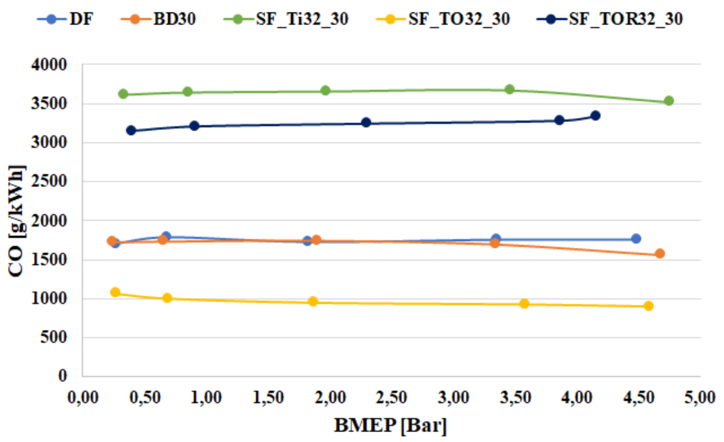

Fig. 16. 30 % blends specific CO emission evolutions at 1600 RPM.

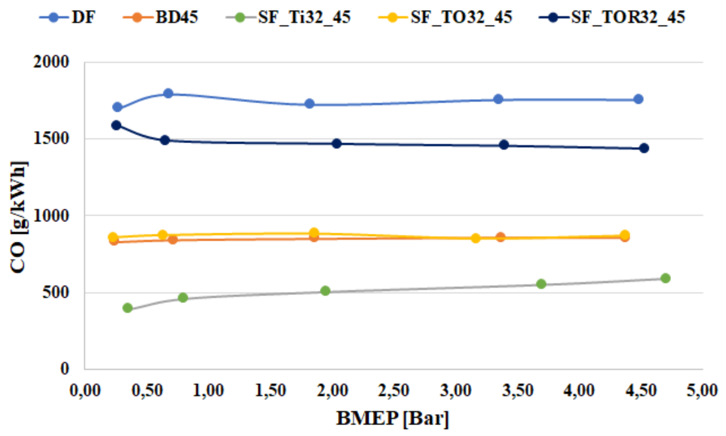

Fig. 17. 45 % blends specific CO emission evolutions at 1600 RPM.

Received: 16-03-2025 Revised: 05-04-2025 Accepted: 02-05-2025

It can be shown that the BSFC is excessive at low loads and the engine seems to consume more when powered by both biodiesel and synthetic fuel oil. Figures 15, 16, and 17 show the variation in specific carbon monoxide emissions as a function of the average effective pressure for biodiesel and synthetic fuel blends at 1600 rpm. Figures 18, 19, and 20 show the variation in specific nitrogen oxide emissions as a function of BMEP for biodiesel, synthetic fuel blends and diesel fuel at 1600 rpm.

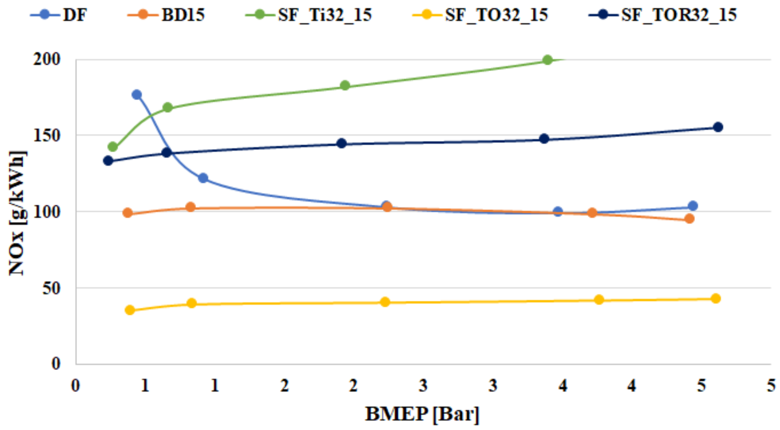

Fig. 18. 15 % blends specific NO emission evolutions at 1600 RPM.

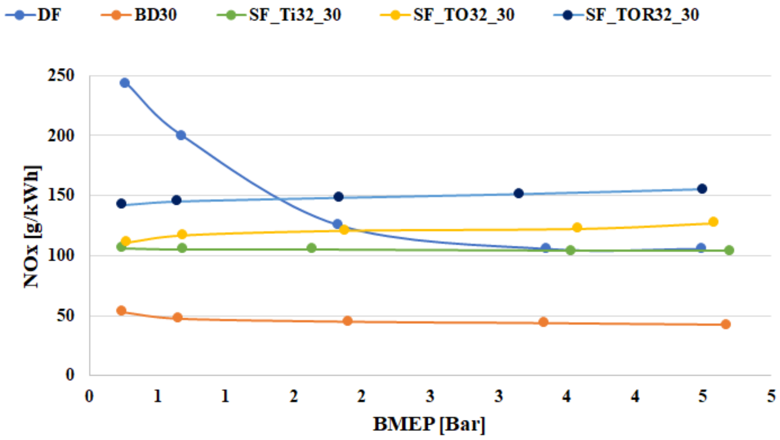

Fig. 19. 30 % blends specific NO emission evolutions at 1600 RPM.

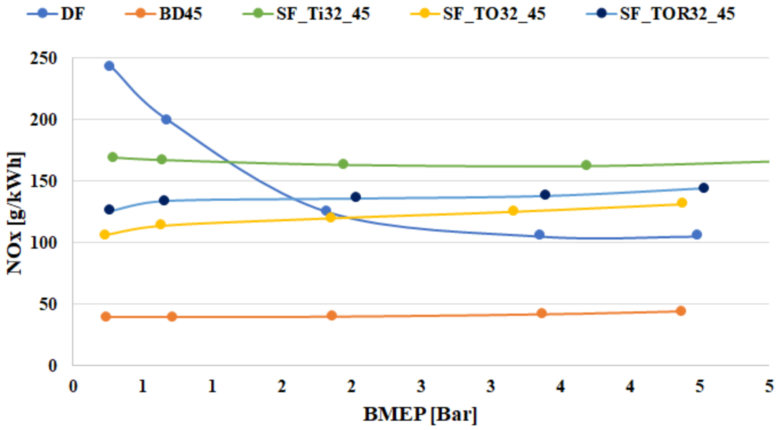

Fig. 20. 45 % blends specific NO emission evolutions at 1600 RPM.

It can be easily seen that whatever are the mixing ratios, the nitrogen oxides emitted are lower when the engine is fueled by synthetic fuels when the engine loads are low. In addition, nitrogen oxide emissions are lower when using biodiesel with a blending rate of 30% and 45%. Lower NOx levels are observed when the engine is fueled by synthetic fuel extracted from Torada 32. Nox levels for biodiesel and synthetic fuels does not seem sensitive to the engine load variation.

6. Conclusions

This article presents the complete methodology required for the extraction of biodiesel from Tiska 32, Torba 32 and Torada 32 lubricants and domestic waste oil. The paper focused on the main physicochemical properties of each oxygenated biofuel grade, namely the measurement of their respective kinematic densities and viscosities, the total acidity index, the flash point and characterization by gas chromatography and mass spectroscopy. It can be concluded that the transesterification of industrial and domestic oils leads to substances whose properties perfectly match the ASTM D6751 and EN 14214 standards and limitations. Because their high viscosities synthetic fuels extracted from Tiska 32, Torba 32 and Torada 32 cannot be used directly on the diesel engine without being blended with commercial diesel fuel. In contrast, synthetic fuels extracted offer a clear advantage of storage capacities since their acidities are low and remain low even after a long storage time. On the other hand, biodiesel extracted from domestic waste oil cannot be stored because of it very high acidity when stored but has a low viscosity and can therefore be used directly in diesel engines with or without blending. As mentioned earlier, blending rates of 15%, 30%, and 45% have been explored on an experimental test bench which is an air-cooled direct injection single-cylinder diesel engine fed with synthetic fuels and biodiesel extracted from Tiska 32, Torba 32 and Torada 32 oils and domestic waste oils. whatever are the blending ratios, the nitrogen oxides emitted are lower when the engine is fueled by synthetic fuels for lower engine loads. NOx levels are lower when using biodiesel for higher blending ratios. Lower NOx levels are observed when the engine is fueled by synthetic fuel extracted from Torada 32. Specific NOx levels for biodiesel and synthetic fuels does not seem to be sensitive to the engine load variation.

List of abbreviations

ASTM – American Testing and Materials Society
BD – Biodiesel
BR – Blending ratio
BSFC – Brake-Specific Fuel Consumption, [g/Wh]
BTDC – Before Top Dead Center, [CAD]
CAD – Crank Angle Degree
CO – Carbon monoxide
DF – Diesel Fuel
EN – European standards
ISO – European Committee for Standardization
NOx – Nitrous oxide
R^2 – Linear regression coefficient
RPM – Revolution per Minute
SF – Synthetic Fuel
TAN – Total Acidity Number
TI32 – Tiska 32
TO32 – Torba 32
TOR32 – Torada 32
WCO – Waste Cooking Oil
μ – Liquid dynamic viscosity, [mPa.s]

Acknowledgements

The authors confirm that there is no conflict of interest in this paper. The present study has not benefit of any funding resources. This paper is the result of a scientific cooperation between LCGE/USTO-MB laboratory, LTE/ENPO-MA laboratory and RA1Z/SONATRACH (Oran, Algeria) supervised by Development department of SOMIZ-SPA/SONATRACH (Oran, Algeria).

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