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ORIGINAL PAPER

Qualitative Assessment of Biodiesel Produced from Primary Edible Oil Wastewater Sludge

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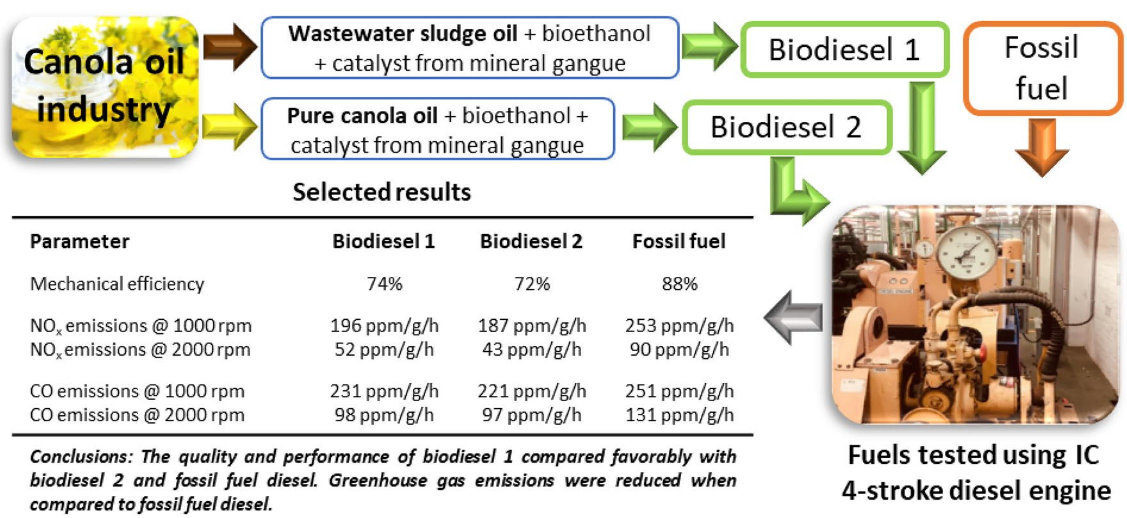
Abstract

Biodiesel is a suitable substitute for fossil fuels and is more environmentally friendly. However, the use of dedicated crops to produce biodiesel threatens food security and alternative feedstocks are therefore being evaluated. This study compared the performance of biodiesel produced from primary wastewater sludge from the edible oil industry with conventional biodiesel produced from pure canola oil, and commercial diesel from fossil fuel (50 ppm sulfur). The fuels were tested using a single-cylinder four-stroke diesel engine operated under steady state conditions at full load. The energetic performance parameters were computed and evaluated. The biodiesel produced from the wastewater sludge performed well. The thermal efficiency was 30.75% (compared to 28.4% and 26.95 for conventional biodiesel and petroleum-based commercial diesel, respectively). The fuel consumption was higher than commercial diesel at a maximum brake power of 12.8 kW (0.15 and 0.31 kg/kW.h for commercial diesel and wastewater sludge biodiesel respectively) but it was more environmentally friendly in terms of gaseous emissions–both fuels showed a linear decrease in emissions proportionate to the increase in engine speed, reaching averages of 51.5 ppm/g/h and 89.9 ppm/g/h nitrous oxides, respectively at 2000 rpm, and 249.3 ppm/g/h and 310.4 ppm/g/h carbon monoxide, respectively at 800 rpm. From the engine evaluation, it was concluded that the energy generation from the wastewater sludge biodiesel and commercial diesel was similar, but that the former was more environmentally friendly in terms of greenhouse gas emissions.

Graphic Abstract



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Extended author information available on the last page of the article

Keywords Edible oil · Wastewater sludge · Biodiesel · Canola oil · Internal combustion engine · Emissions

Statement of Novelty

The quality, engine efficiency and engine emissions of novel biodiesel made from edible oil wastewater sludge were compared with biodiesel made from food grade canola oil and fossil fuel.

Introduction

The transport sector contributes directly to greenhouse gas (GHG) emissions through the combustion of fossil fuels [1], generating mainly carbon dioxide (CO_2), small amounts of methane (CH_4), nitrous oxide (N_2O) and carbon monoxide (CO) [1, 2]. The South African GHG inventory, an official submission to the United Nations Framework Convention on Climate Change (IEA, 2017) suggests that energy used in transportation contributes to approximately 8.8% of South Africa's GHG emissions (excluding emissions from agriculture, forestry and land use) [3]. Biodiesel has physical and chemical properties similar to petroleum-based commercial diesel (C-diesel) [4, 5]. The mass calorific power of biodiesel is around 13% lower than C-diesel, but because it has a slightly higher density, this does not lead to a notable difference in engine power [6]. The major advantages of biodiesel are that: (i) It is renewable [4]; (ii) Direct carbon emissions from combustion are minimal relative to fossil fuels [7]; and (iii) It can improve energy security in countries with surplus agricultural land [8]. The major disadvantages of biodiesel are that: (i) Over-utilisation of cash crops as feedstocks can threaten food security [1]; (ii) The high viscosity of biodiesel can lead to engine fouling [9]; and (iii) Due to the cost of the oilseed (70–85% of total production costs), biodiesel is more expensive than C-diesel [10, 11].

In order to replace C-diesel more widely, the quality of biodiesel needs to be improved and less expensive, readily available, non-edible feedstocks need to be sourced, such as oil-or fat-containing wastes [12, 13]. The quality of biodiesel is dependent on the composition of the feed oil and the choice of technology used for production [14]. Conventional biodiesel is typically produced by transesterification using homogeneous catalysts (i.e., acid or alkali solutions) in the presence of an alcohol (methanol or ethanol) [10]. The properties used to evaluate biodiesel are divided into two groups: (i) The general physicochemical properties, for example, density, viscosity, flash point, % sulfur, carbon residue, % sulfate, cetane number, and acid number [15] and (ii) The composition and purity of fatty esters. These parameters impact on engine efficiency and the quality and quantity of harmful emissions [16]. In South Africa, the parameter ranges and test methods for biodiesel and related petroleum fuels are stipulated by South African National Standards (SANS), specifically SANS 1598 and SANS 342:2016 [17]. These are based on the American Society for Testing and Materials (ASTM) standards (Table 1), and focus on the viscosity, flash point, high heating value (HHV), and ester content of the fuel. The main differences between biodiesel and petroleum diesel are that the former usually has a higher density, viscosity, cloud point and cetane number, but lower volatility, gross calorific value and sulfur content. The cetane number is inversely proportional to the ignition delay, and can be predicted once other parameters are known [17].

The guidelines have been formulated based on experience and are performance-based and feedstock neutral. As vehicle technology improves, new feedstocks are identified and/or legislation for environmental performance becomes more stringent, they will require revision to reflect the new status quo [18].

Table 1 Benchmark specifications for diesel as per the *ASTM Biodiesel Standard D 6751*[a] [17]

Properties	Test method	Limits	Units
Flash point (closed cup)	D 93	130.0 min	°C
Water and sediment content	D 2709	0.050 max	% volume
Kinematic viscosity	D 445	1.9 – 6.0	mm^2/s
Sulfated ash content	D 874	0.020 max	% mass
Sulfur content	D 5453	0.0015[a] max. (S15)/0.05 max. (S500)	% mass
Copper strip corrosion test	D 130	No. 3 max	ppm
Cetane number	D 613	47 min	NA
Cloud point	D 2500	Report	°C
Carbon residue	D 4530	0.050 max	% mass
Acid number	D 664	0.50 max	mg KOH/g
Free glycerin content	D 6584	0.020	% mass
Total glycerin content	D 6584	0.240	% mass
Phosphorus content	D 4951	0.001 max	% mass
Sodium/Potassium content	UOP 391	5 max. combined	ppm
Distillation temperature[b]	D 1160	360 max	°C

NA not applicable

[a]The limits are for Grade S15 and Grade S500 biodiesel, with S15 and S500 referring to maximum sulfur (in ppm)

[b]Atmospheric equivalent temperature method (90% recovered)

Food grade Canola oil has been identified as an ideal feedstock for biodiesel production [19]. Apart from the favourable fat content, it has a low cloud point, so it gels at lower temperatures than many other feedstocks [10]. In this study, an alternative feedstock was used for the production of biodiesel i.e., oil extracted from primary wastewater sludge from a Canola oil processing plant [edible oil wastewater sludge (EOWWS)]. The EOWWS is typically disposed to landfill, but it still contains high concentrations of oil. It was therefore hypothesised that EOWWS may be a suitable feedstock for biodiesel production. To assess the quality and performance, the EOWWS-biodiesel was evaluated against biodiesel produced from pure Canola oil (PCO-biodiesel) and C-diesel. The fuels were characterised in terms of viscosity, density, higher heating value (HHV) and flash point, and were then tested in a direct injection diesel engine.

Materials and Methods

In this study, the catalyst was prepared from cupriferous mineral processing wastes (dolomite based). It was synthesised using the sol–gel method supported by zero valent iron nanoparticles (ZVINPs). An in-depth study on the optimisation of the synthesis of the catalyst and production of biodiesel has been submitted for publication elsewhere ([20], unpublished data). Briefly, the process was carried out in a 500 mL flask using a 1:6 oil/ethanol molar ratio for a period of 120 min at 75 °C.

The PCO-biodiesel was prepared by the transesterification of pure Canola oil and methanol by a conventional method used for biodiesel production using KOH as a catalyst. The reaction was conducted for 2 h in a 500 mL flask at 50 °C with a catalyst dose of 2.5 wt.% and an oil to methanol ratio of 1:9. The C-diesel was purchased at a local petrol station. To assess the quality of the prepared biodiesel, the products were tested in accordance with the ASTM methods and the European normalisation [21].

Higher Heating Value

The higher heating value (HHV) is a measurement of the energy content in a fuel. The HHV of each fuel was measured in a bomb calorimeter (Model 1341 Plain Jacket Calorimeter, Parr Instruments Company, Moline, US) according to ASTM D2015 standard method using an oxygen bomb at 3 Mpa pressure. The bomb was fired automatically after the jacket and the bucket temperature equilibrated to the acceptable accuracy of each other as per the manufacturers' recommendations.

Viscosity and Density

The afflux time of ≈ 50 mL of each fuel was determined in a digital viscometer with stand and probe (Model BROOKFIELD LVDV-I Prime C-Digital, Middleboro, US) after heating each sample to 40 °C for 30 min in a viscosity tube and recording the flow rate as per the manufacturers' instructions. The kinematic viscosity (v) was calculated according to Eq. 1, adapted from Felder and Rousseau [22]. The viscosity indices were determined using the VI-ASTM D2270 [17].

$$V = \mathrm{c} * t \tag{1}$$

where v Viscosity in (mm^2/s or cSt), t time in seconds, c Viscosity tube constant.

Flash Point

The flash point of each fuels was measured by a flash point tester (Model Pensky-Martens-PM5, Rigana Manufacturing, Westmead, South Africa) according to manufacturers' instructions.

Cetane Number

Cetane has excellent ignition properties, and the cetane number is theorectically related to the proportion of cetane (n-hexadecane, $C_{16}H_{34}$) and isocetane (heptamethylnonane, $C_{12}H_{34}$) in a fuel mix, with cetane being assigned a value of 100 (Eq. 2).

$$\text{CN of fuel} = (\%\text{ of n} - \text{cetane}) + 0.15 \times (\%\text{ of isocetane}). \tag{2}$$

The parameter can be experimentally determined by comparing the ignition delay of the fuel to the ignition delay in a Cooperative Fuel Research (CFR) engine [23]. In this study, the cetane numbers of the biodiesels were determined empirically using equations formulated by Giakoumis and Sarakatsanis [23], with modifications based on the composition of the fatty ethyl esters in the canola oil and edible oil wastewater sludge [24].

Set-Up and Operation of Internal Combustion Engine

A stationary and computerised Plint & Partners TTE95/5604 (Berkshire, Reading, England) 4-stroke, 5.2 kW, water cooled, single cylinder internal combustion (IC) diesel engine was used to evaluate the performance of diesel EOWSS-biodiesel, PCO-biodiesel and C-diesel in terms of mechanical and thermal efficiency and GHG emissions (Fig. 1). The fuel flow rates, engine speeds, exhaust gas

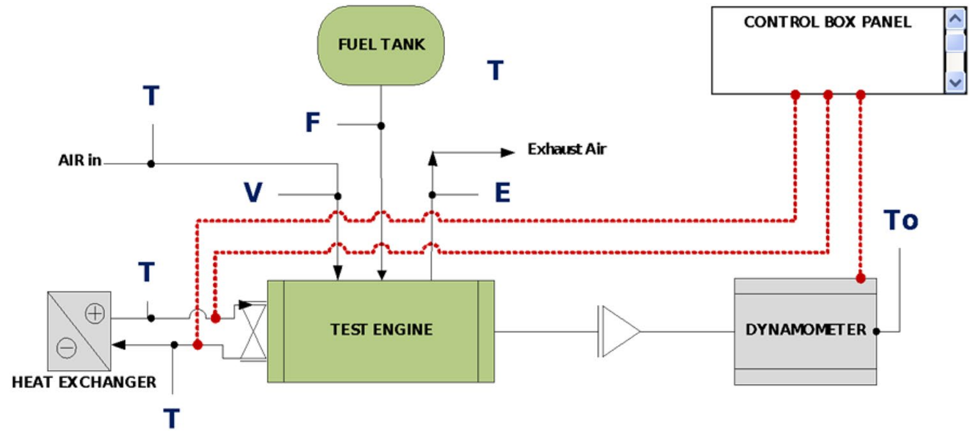

Fig. 1 Schematic diagram of experimental setup (T: Temperature; V: Velocity; Emissions (CO, NOx); F: Flow rate; T: Torque)

temperatures and gas flow rates were monitored on the IC engine control panels, and the exhaust gases were quantified using a QROTECH (Gyeonggi-do, South Korea) QRO-402 emission analyser at an engine speed range of 800 to 2000 rpm. For each experimental run, the engine was allowed to run for sufficient time to empty the contents of the fuel tank. The level of the lubricating oil in the sump was checked periodically. Constant water flow was maintained through the engine to prevent overheating. All the readings were taken under steady running conditions. All experiments were conducted using 5 L fuel and performed in duplicate for each fuel type.

The data obtained was used to calculate the thermal and mechanical efficiency of the fuels using energy balance equations and thermodynamic equations as previously described [22]. For referral, the equations and calculations have all been included as Supplementary material, together with relevant data.

Results and Discussion

Quality Evaluation of EOWWS-Biodiesel

Fuel properties for combustion analyses can be grouped conveniently into physical, chemical and thermal properties [25, 26]. For the selected thermo-physical parameters measured in this study, it was clear that both EOWWS-biodiesel and PCO-biodiesel were very similar to C-diesel, and parameters largely fell within the ranges specified by the relevant ASTM standards (Table 2). Fuels with parameters that fall within these standards can be used in most modern engines.

The viscosity of biodiesel is considered to be one of the most important parameters for engine performance, and studies [12, 27] have shown that it has a direct influence on combustion properties such as atomisation and spray characteristics. Fuels with lower viscosities are more volatile, which enhances ignition without having any negative effect on cold flow properties [27]. Viscosity increases with a decrease in temperature, but high viscosities at any temperature negatively affects fuel flow and causes incomplete combustion. However, if the viscosity is too low, the lubrication properties of the fuel are negatively affected, leading to engine wear and leakage [14]. In the performance evaluation of a diesel engine fuelled with biodiesel produced from soybean oil, other researchers reported that a decrease in the combustion quality and higher exhaust emissions may be related to the formation of larger droplets during injection associated with higher viscosities [28]. In this study, viscosities of 3.7×10^{-6} m^2/s, 3.9×10^{-6} m^2/s and 3×10^{-6} m^2/s for EOWWS-diesel, PCO-biodiesel, and C-diesel, respectively

Table 2 Selected fuel parameters and specified standards

Properties	Unit	Method	Standards [17]	C-diesel*	EOWWS-biodiesel	PCO-biodiesel
Viscosity at 40 °C	m^2/s	ASTM D445	2.5–4.2 $\times 10^{-6}$	3.0 $\times 10^{-6}$	3.7 $\pm$ 0.38 $\times 10^{-6}$	3.9 $\pm$ 0.78 $\times 10^{-6}$
Density at 15 °C	kg/m^3	ASTM D941	860–890	830	833 $\pm$ 0.81	841 $\pm$ 1.59
HHV	MJ/kg	ASTM D2015	44.6–49.5	48.1	45.7 $\pm$ 0.73	46.4 $\pm$ 0.64
Flash point	°C	ASTM D93	52–96	58.6	61.3 $\pm$ 1.34	50.4 $\pm$ 0.86
Cetane number	–	ASTM D613	47 min	45	48.09 $\pm$ 1.92**	49.13 $\pm$ 1.76**

*Parameters obtained from source. **Predicted

Results expressed as averages $\pm$ standard deviation ($n = 3$)

were recorded, which were in line with the relevant ASTM D445 specifications [19].

The fuel densities at 15 °C all fell slightly below the specified range (Table 2). However, the density of the EOWWS-biodiesel and the C-diesel in particular, were very similar (830 kg/m^3 and 833 kg/m^3, respectively). These results also compare with those of previous researchers investigating biodiesel properties, where densities ranged from 800 to 856 kg/m [16].

The HHV is dependent on the chemical nature of a fuel and is defined as the amount of heat energy released from the complete combustion of a fuel under standard temperature (25 °C) and pressure (1.0 atm.) [29]. In this study, the HHV was 46 MJ/kg for both biodiesels and 48 MJ/kg for C-diesel, which were in line with literature values [10].

Of note was the fact that the predicted cetane number of both PCO-biodiesel and EOWWS-biodiesel were above the minimum ASTM specifications, while the cetane number provided by the manufacturer of the C-diesel was slightly lower (Table 2).

The flash point is the lowest temperature at which a fuel can spontaneously ignite in air without the presence of a flame or spark, and is therefore a key parameter to consider for safe handling, storage and transport of fuels [30]. The flashpoint may reduce spontaneously during storage, but can be increased by adding antioxidants [6]. Ideally, diesel should have a high flash point, but because compression engines do not have spark plugs, it should have a low auto-ignition temperature [31]. Biodiesel typically has a lower flash point than C-diesel [16], which was the case in this study with PCO-biodiesel (50.4 °C v/s 58.6 °C for C-diesel). In contrast, although still close to the value for C-diesel, the highest flash point was determined for EOWWS-biodiesel (61.3 °C).

Computation of the Fuel Thermodynamic Data

The mechanical efficiency of a diesel engine is particularly dependent on the brake power within the system, which is a product of the mass flow rate of air and the inverse of the specific fuel consumption for a given fuel air ratio. The mechanical efficiencies of the EOWWS-biodiesel, PCO-biodiesel, and C-diesel attained in the test engine were 73.8%, 72.1% and 87.5%, respectively (results not shown).

The specific fuel consumption is effectively a measure of the engines' ability to convert the chemical energy to mechanical energy [13]. The thermal efficiency that can be attained with a particular fuel is highly dependent on its viscosity as well as other parameters, such as the amount of oxygen present in the ethyl esters, with 14 wt.% being ideal [6, 13].

The specific fuel consumption of C-diesel was lower than that of PCO-biodiesel and EOWWS-biodiesel, within

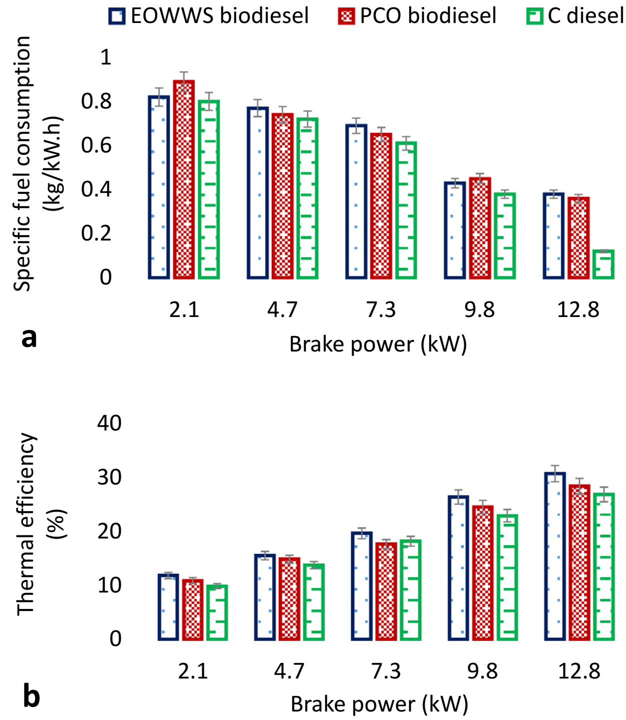

Fig. 2 Relationship between the brake power and the specific fuel consumption (**a**) and thermal efficiency (**b**) of EOWWS-biodiesel, PCO-biodiesel and C-diesel [Error bars represent the standard deviation from the mean (n = 3)]

the range of brake power outputs applied (2.1 to 12.8 kW). The C-diesel performed notably better at higher brake power (Fig. 2a). The thermal efficiency of the EOWSS-biodiesel was highest of all three fuels over the range of brake power outputs (Fig. 2b). The average thermal efficiencies (Fig. 2b) and viscosity (Table 2) were both lowest for the C-diesel, while the HHV (Table 2) was highest. In comparison, at the lowest brake power output (2.11 Kw), a higher thermal efficiency (34.2%), but a lower specific fuel consumption (0.56 kg/kW h) was obtained by other researchers when operating an IC engine with biodiesel produced from waste cooking oil [32].

Although the biodiesels themselves were not blended, the differences in engine performance parameters between the biodiesels and the C-diesel are supported by previous studies comparing the performance of engines operated with petroleum diesel before and after blending with biodiesel made from rapeseed oil [33, 34]. One group of researchers [34] showed that the thermal efficiency of jet fuel increased when it was blended with biodiesel, while another [33] found that the ignition delay and heat release rate decreased, and the injection and combustion duration increased with an increase in the ratio of biodiesel to petroleum diesel. The authors ascribed the increase in combustion duration to an advanced ignition delay and lower HHV due to the addition of biodiesel [31, 32].

At maximum combustion temperature, the exhaust gas temperature and the engine load are related to fuel consumption. Hence, the combustion temperature increases with an increase in load because of increased fuel supply [35]. In a conventional diesel engine cycle, the gas is not exhausted at ambient temperature, so a major part of the energy is lost with the exhaust gases [35, 36]. The diesel engine used in this study was fully insulated, but not equipped with any heat loss regulators to retrieve the heat lost in the exhaust gas [37].

In this study, a simple mass-balance for heat losses was conducted (Fig. 3). In comparison to both biodiesels, a greater and lower fraction of heat energy was transferred to exhaust gases (15.7%) and cooling water (18.8%), respectively for the C-diesel (Fig. 3). This was attributed to biodiesel containing constituents of poor volatility, which burn only late in the combustion phase [33]. Unaccounted $heat_{out}$ was virtually the same (≈40%) for both biodiesels tested (PCO-biodiesel and EOWWS-biodiesel), which was higher than for C-diesel (35.9%). The unaccounted heat is the fraction that could not be accurately measured and was calculated as the difference between the heat supplied and the sum of the heat transferred to the precisely measured fractions (brake power, cooling water and exhaust gases). In this instance, the 'unaccounted' heat energy was lost by convection, radiation, leakage of gases, and mechanical energy from the engine used to run accessories such as the lubricating pump and water circulating pump [9, 33].

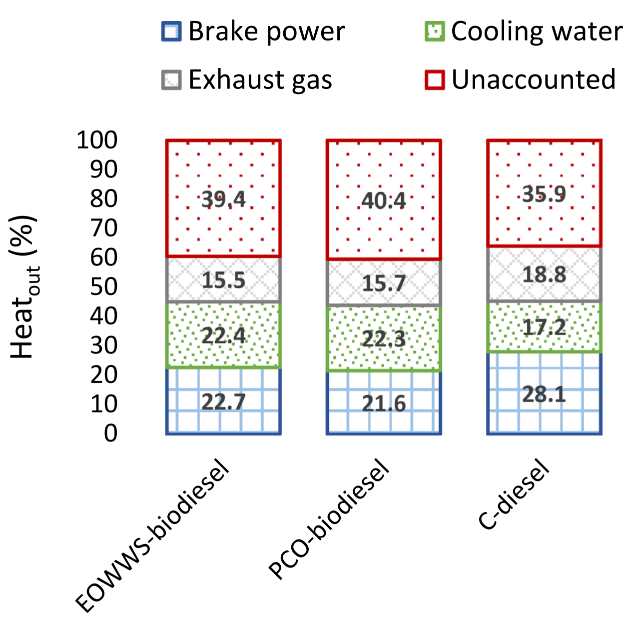

Fig. 3 Results of overall heat balances for the fuels tested in an internal combustion engine

Evaluation of Emissions

As engine speed increases, fuel combustion improves due to more efficient mixing of fuel and air. In addition, when engines are operated under higher loads or for longer periods, the in-cylinder temperatures increase, which results in improved fuel atomization, evaporation, and air–fuel mixing [34, 37]. In this study, the engine was operated for the same duration and under the same load for each of the fuels tested, so emission changes were assessed using the engine speed, which was varied. Only carbon monoxide (CO) and nitrogen oxides (NOx) were analysed in this study (Fig. 4) because they are the most potentially harmful emissions from diesel engines [33]. Nitrogen dioxide (NO_2), for example has a global warming potential 250 times that of CO_2 due to its strong capacity to absorb infrared rays [38].

In comparison to petroleum diesel, combustion of biodiesel typically results in high NOx emissions, a factor that may be reversed by increasing the cetane number of the biodiesel and/or increasing the engine speed [34, 39–42]. For example, less NOx was emitted from an IC engine operated with high cetane biodiesel (960 ppm) than with C-diesel (1093 ppm), and (ii) the NOx emissions from biodiesel decreased slightly under full load conditions [40, 41]. The latter may be explained by an increase in engine speed leading to a decrease of residence time at maximum combustion temperature, furthermore, the release of NOx emissions is more likely to be thermally controlled in the high-temperature flame region [40, 42]. In this study, the cetane number of both biodiesels were above the minimum ASTM specification of 47, while that of the C-diesel was slightly lower (45). The measured emissions for all fuels decreased almost linearly with an increase in engine speed within the range tested (Fig. 4a–f). The test engine emitted notably lower NOx when operated with either biodiesel type than with C-diesel. Lower CO was also emitted when the engine was operated with EOWWS-biodiesel than with C-diesel, but in some instances (engine speed of 1200 and 1400), the CO emissions were slightly higher with PCO-biodiesel (Fig. 4c) than C-diesel (Fig. 4e).

Conclusions

Overall, in terms of engine performance assessed using thermo-mechanical parameters and emissions, EOWWS-biodiesel performed well. The emission measurements were comparable to those from PCO-biodiesel, which were notably lower than with C-diesel. EOWWS, perhaps in conjunction with reject oilseeds, has the potential to be used as a biodiesel feedstock at edible oil facilities where oil extraction facilities are available in-situ. The use of biodiesel,

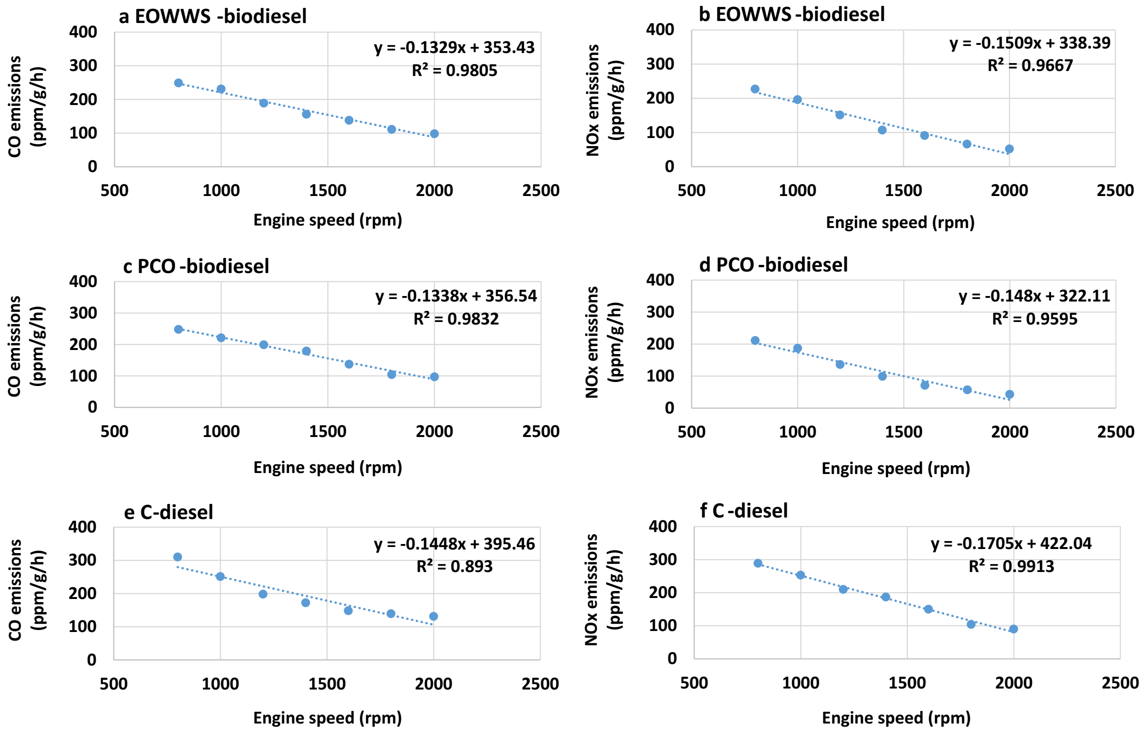

Fig. 4 Engine speed effect on CO and NOx emissions for selected fuels: EOWWS-biodiesel (**a**, **b**) PCO-biodiesel (**c**, **d**) C-diesel (**e**, **f**) [R^2 regression values correlating to the standard deviation from the mean (n = 3)]

either alone or blended with C-diesel could have both positive financial and environmental impacts for this industry.

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