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ORIGINAL ARTICLE

Evaluation of physicochemical properties of biodiesel-diesel blends in the context of fuel quality

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ABSTRACT
The present study is aimed to investigate the physicochemical properties of biodiesel from waste cooking oil and its blends with fossil diesel and their effects on the fuel quality. The physicochemical properties of B5-B20 blends were within the limits of ASTM D7467. The B50 which is not included in the standards, proved to have desirable properties, as they were also within the limits of the ASTM D7467. A higher flash point is preferable as it ensures more safety and better handling. Results showed a significant increase of this parameter in samples containing min. 50 % biodiesel. Increasing the biodiesel fraction led to an increase in contamination, water and linolenic acid content, which makes the fuel more prone to oxidative degradation and operational problems. In contrast, a lower sulphur content due to a decrease in the biodiesel fraction is favourable for the environment. Similarly, blends containing less than 20 % biodiesel had a higher heat of combustion and therefore suggest a lower fuel consumption of the engine. The cetane number of fossil diesel is increased upon blending with biodiesel, however all blends met the ASTM D7467 requirement which implicates satisfactory combustion and engine efficiency. The presented data is useful in predicting the biodiesel blend quality according to physicochemical properties for any percentage of biodiesel obtained from waste cooking oil.
Keywords: *biodiesel, blend, quality, physicochemical parameters*

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INTRODUCTION

The chemical process by which biodiesel is prepared is known as the transesterification reaction, which involves a triacylglycerol (TAG) reaction with a short-chain monohydric alcohol normally in the presence of a catalyst at elevated temperature to form fatty acid alkyl esters (FAAE) and glycerol [1]. The major focal point for biodiesel high quality is the adherence to biodiesel standard specifications. These standard specifications could either be the American standards for testing materials (ASTM D6751) or the European Union standards (EN 14214) for biodiesel fuel. The purity and quality of biodiesel fuel can be significantly influenced by numerous factors, which amongst others include: the quality of feedstock, fatty acid composition of the vegetable oils, animal fats and waste oils, type of production and refining process employed and post-production parameters [2]. The ASTM D6751 identifies the parameters the pure biodiesel (B100) must meet before being used as a pure fuel or being blended with petroleum-based diesel fuel. Biodiesel is pure or 100%, biodiesel fuel. It is referred to as B100 or ''neat" fuel. A biodiesel blend is pure biodiesel blended with fossil diesel [3].

Biodiesel is currently the most widely accepted alternative fuel for diesel engines, due to its technical, environmental and strategic advantages. Compared to fossil diesel, it has improved lubricity, lower toxicity, higher flash point (FP), and biodegradability [4-6]. Previous studies have demonstrated that the addition of biodiesel to diesel leads to higher fuel consumption and higher NOx emission, lower CO, unburnt hydrocarbon and particulate matter emissions, while there is little change in the brake thermal efficiency [7-8]. However, the high viscosity, low volatility and poor cold-flow properties at low temperature of biodiesel affect its combustion quality [9-11]. High viscosity and low volatility have adverse effects on fuel atomization while poor cold-flow properties lead to the solidification of fatty acid compounds and the formation of crystals at low temperature [12]. The aim of this work was to evaluate physicochemical properties of blends containing biodiesel produced from waste cooking oil (WCO) and fossil diesel and determine their quality according to the ASTM D7467. The study included the evaluation of a B50 blend which is not encompassed in the current European and American standards.

MATERIAL AND METHODS

Chemicals: Toluene, 99.5% was purchased from Carlo Erba, Italy; n-Heptane, 99.93% from Fisher Chemical, Great Britain and Gasses (nitrogen, oxygen, hydrogen standard) from Messer, Germany. The following chemicals were purchased from Sigma-Aldrich, Germany: Methyl heptadecanoate ≥99%, H4515-1g; Karl Fischer Solution; Hydranal-Water Standard 1.0 (0.1%) H_2O; Chloroform X and Methanol X. Sulphur standard was purchased from NORMA R, Germany. All chemicals were of analytical reagent grade.

All experimental work has been performed in the laboratory of a quality control institution HERKON Ltd. in Mostar, Bosnia and Herzegovina, in 2015.

Sampling and sample preparation

For the testing purposes, biodiesel was produced by transesterification from waste cooking oil (System EcologiaSrbac, Bosnia and Herzegovina). Commercially available diesel fuel from Bosnia and Herzegovina was used as well. Biodiesel was sampled in the production plant immediately after production. Sampling of biodiesel and fossil diesel was carried out in 1L bottles made of stainless steel, designed for obtaining samples of liquid petroleum fuels.

Blending process

The blends were prepared in the following (%, v/v) proportions: 5% (B5), 10% (B10), 15% (B15), 25% (B25) and 50% (B50). Samples were poured into a glass jar with a lid, labelled and stored until analysis.

Testing of physicochemical parameters

The following methods were used in testing of the default quality parameters: EN ISO 3675: 2004 for the determination of density; standard test method ASTM D93, Procedure A, ASTM 2012 for the FP; EN 116:1998 for predicting the Cold Filter Plugging Point (CFPP); EN ISO 4264:2009 for the calculation of the cetane index (CI); EN ISO 3104:2002 for the calculation of dynamic viscosity at 40°C; EN 12662: 2009 for the determination of contamination; ASTM D 86:2014 for the determination of distillation flow (DF); EN ISO 20846:2004 for the determination of the sulphur content; EN ISO 12937:2002 for the determination of the water content; EN ISO 6245:2003 for the determination of the ash content; ASTM D 4868:2003 for the determination of the heat of combustion; and EN ISO 14103:2011 for the determination of total content of fatty acid methyl esters (FAME) and linolenic acid (LA) contents.

RESULTS AND DISCUSSION

This paper examines the physicochemical properties of pure diesel fuel (D100), pure biodiesel (B100) and five blends of diesel and biodiesel. The following physicochemical properties were investigated as quality parameters: density, viscosity, CFPP, FP, sulphur content, CI, heat of combustion, total contamination amount, water content, ash content, FAME content and LA content. The specifications for B100 are given in standards EN14214 and ASTM D6751, however the quality of biodiesel blends is evaluated according to the standard ASTM D7467. The ASTM D7467 is designed so that if B100 meets D6751 and fossil diesel meets D975, B6 to B20 will meet its specification (Table 1).

Table 1. US and EU specifications for biodiesel and diesel

US and EU Biodiesel Specifications							Diesel Fuel Specifications	
Property	**ASTM D6751-12**		**ASTM D7467**		**EN 14214:2012**		**ASTM D 975**	
	Limits	Test	Limits	Test	Limits	Test	Limits	Test
Flash point, min	93°C	D93	52°C	D93	101°C	EN ISO 2719	52°C	D93
Water, max			0.05 vol %	D2709	500 mg/kg	EN ISO 12937	0.05 vol%	D2709
Total contamination, max					24 mg/kg	EN 12662		
Kinematic viscosity	1.9-6.0 mm^2/s	D445	1.9– 4.1 mm^2/s	D445	3.5-5.0 mm^2/s	EN ISO 3104	1.9-4.1 mm^2/s	D445

Density					860-900 kg/m^3	EN ISO 3675 EN ISO 12185		
Ester content					96.5% min	EN 14103		
Ash, max	0.02 % m/m		0.01 % m/m	D482	0.02 % m/m		0.01 % m/m	D482
Sulphur, max (by mass)	Two grades: S15 15 ppm S500 0.05%	D5453	Three grades: S15 15 ppm S500 0.05% S5000 0.5 %	D5453, D2622, D129	10.0 mg/kg	EN ISO 20846 EN ISO 20884 EN ISO 13032	Two grades: 0.5%; 0.05%	D129; D2622
Cetane number, min	47	D613	40	D613	51.0	EN ISO 5165	40	D613
CFPP					Location & season dependant	EN 116		
Linolenic acid methyl ester, max					12.0% wt	EN 14103		

The results (presented in Table 2) showed that B100 met all criteria outlined by EN14214 and ASTM D6751, except for the CI, while the diesel fuel D100 met all criteria of the standard ASTM D975. Accordingly, all blends B5-B20 should have physicochemical properties within the limits of ASTM D7467, which was confirmed by this work, despite the discrepancy regarding the CI. Further on, the B50 which is not included in the standards, proved to have desirable properties (FP, CI, viscosity, water content, sulphur content, ash content), as they were within the limits of the ASTM D7467. A detailed interpretation of results and the significance of the investigated parameters for the fuel quality are given below.

Table 2. Physicochemical parameters of the analysed samples

	D100	**B5**	**B10**	**B15**	**B20**	**B50**	**B100**
Density at 15 °C (kg/m^3)	842.7	845.0	845.3	850.2	854.4	861.6	888.2
Flash point (°C)	57.5	58.5	59.0	60.0	60.5	71.0	178.0
CFPP (°C)	-13	-12	-12	-12	-12	-10	-2
Kinematic viscosity at40 °C (mm^2/s)	2.58	2.88	2.92	2.97	3.05	3.53	4.01
Cetane number	47.95	47.88	47.76	47.64	47.58	47.02	46.06
Water (mg/kg)	52.92	67.39	86.87	105.90	122.40	246.10	374.40
Sulphur (mg/kg)	8.96	8.68	8.40	8.21	7.38	6.17	4.92
Heat of combustion (MJ/kg)	42.86	42.84	42.83	42.78	42.73	42.63	42.30
Total contamination (mg/kg)	4.2	4.6	4.9	5.1	5.3	6.1	8.3
Ash %(m/m)	0.01	0.01	0.01	0.01	0.01	0.01	0.01
FAME % (m/m)	3.20	6.95	11.99	16.56	21.06	49.89	96.70
Linolenic acid methyl esther % (m/m)	4.01	4.68	5.12	5.54	5.89	6.52	6.88

Density

The purpose of the density specification in EN 14214 is to exclude extraneous material as biodiesel feedstock [13]. The obtained results show a constant increase in density with an increase in biodiesel content in relation to diesel. The density increase is continuous, but not linear. In order to determine the effects of temperature on density, the measurement of each of the seven samples was carried out at three different temperatures: 12.0°C, 17.0°C and 22.0°C. The fuel density increased with the increase in biodiesel content and a decrease in temperature.

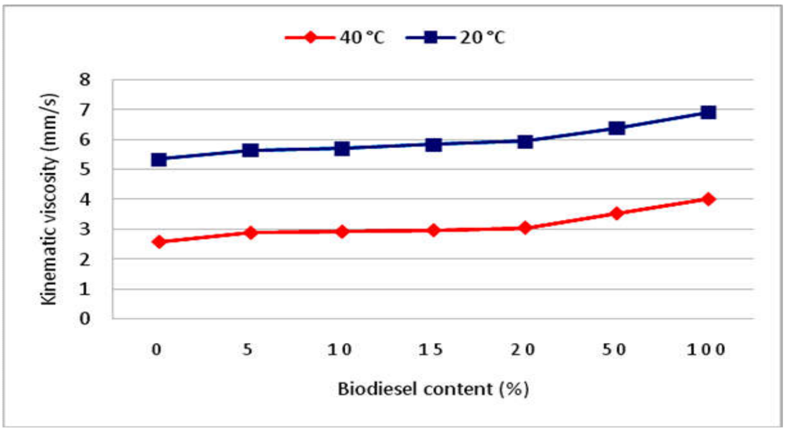

Figure 1. The influence of temperature on fuel viscosity

Kinematic viscosity

Vegetable oils and animal fats have a high kinematic viscosity and cause operational problems, which is why biodiesel is used as an alternative fuel [14]. In contrast, the kinematic viscosity of biodiesel is slightly higher than petrodiesel. This parameter is determined by a variety of structural features. Longer chain length and larger ester head group lead to an increase in kinematic viscosity, as opposed to a higher degree of unsaturation which will decrease kinematic viscosity. As for the double bond orientation, *cis* double bonds contribute to a lower kinematic viscosity [1]. According to the obtained experimental values, the kinematic viscosity of fuel samples at 40 °C showed a slow but gradual increase with the increase of the volume fraction of biodiesel in the fossil diesel. A higher viscosity value due to the biodiesel makes it possible to improve the lubricant properties of the fuel. However, the lower the viscosity of the oil, the easier it is to pump and atomize and achieve finer droplets [15]. The viscosity values of all samples were in the range between 2.58 and 4.01 mm^2/s and within limits of their respective standards. The effect of the fatty acid profile on the viscosity has practical importance. The viscosity can be adjusted to the required values by blending biodiesel from different sources [16]. In order to investigate the effect of temperature on the rheology, the viscosity of samples was investigated at a lower temperature, at 20°C. The results showed that the viscosity increased by increasing the content of biodiesel in the fuel with the same progression for both temperatures. However, it was clearly noticable that viscosity at 20 °C is higher by 50-60 % for all biodiesel and diesel ratios and above the prescribed upper limits.

Cold filter plugging point

Biodiesel has generally a higher CFPP than petrodiesel and therefore poorer performance properties in cold weather [17]. This is important as in cold temperate countries, a high CFPP will clog up vehicle engines more easily. The cold flow properties of biodiesel are dictated by the length of the hydrocarbon chains and the presence of unsaturated fatty acids in biodiesel [18]. Figure 2 clearly shows the linear growth of the CFPP by increasing the biodiesel content from 20 % to 100 %, while increasing the biodiesel content from 0 % to 20 % does not have such an effect on the CFPP. To provide options for different climates, the EN14214 specifies individual requirements for different countries. The CFPP of the analysed samples ranged between -13 °C and – 2 °C. The CFPP of B5 to B20 was -12 °C.

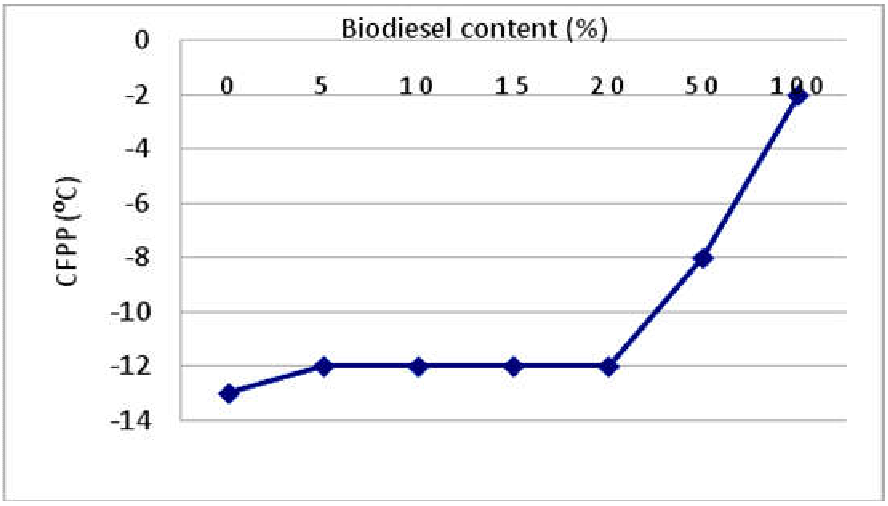

Figure 2. The influence of biodiesel content on the CFPP

Flash point

Generally, biodiesel possesses a higher FP than petrodiesel. The FP specification is largely a reflection of the boiling points of the individual components. The branched and lower molecular weight components in petrodiesel lead to a reduction of the FP. However, alcohol remains in the finished product influences the FP [19]. The initial boiling point (IBP) can also indicate the purity of the biodiesel sample, since the presence of compounds such as glycerin and/or solvents may alter the IBP and even the boiling point range [16]. According to the obtained experimental values, the increase of the FP is insignificant all the while the biodiesel content is below 50% (increase by 14.5 °C), while for pure biodiesel B100, the FP is significantly higher if compared to diesel and B50 blend (increase by 107°C). The FP of B50 is 71°C and while the ASTM D7467 does not include blends containing 50 % (v/v) biodiesel, the FP is above the minimum temperature outlined in that standard. In contrast, B50 can not fit the requirements for pure biodiesel with respect to this parameter.

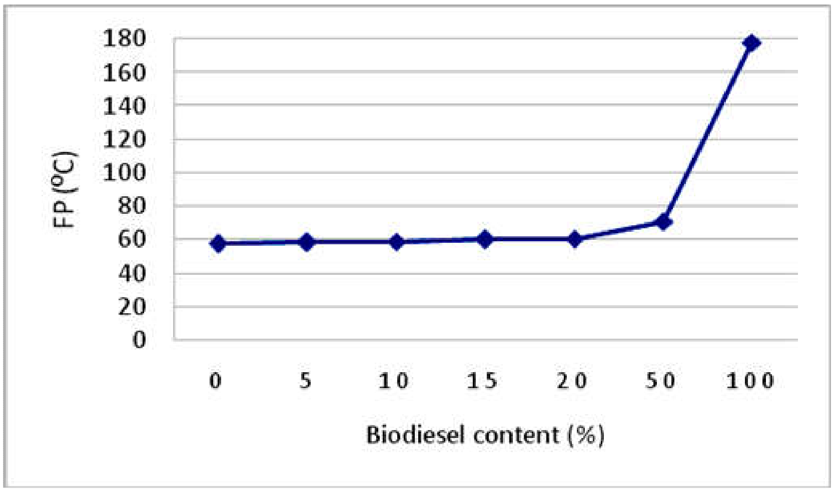

Figure 3. The influence of biodiesel content on the FP

Distillation flow

One of the most important and informative parameters that is measured for complex fluid mixtures is the distillation curve [20]. Distillation curves are useful for measuring the overall volatility and driveability of a fuel, and have served as a basis for the modelling and development of gasoline-like fuel mixtures with desirable volatility properties [21].

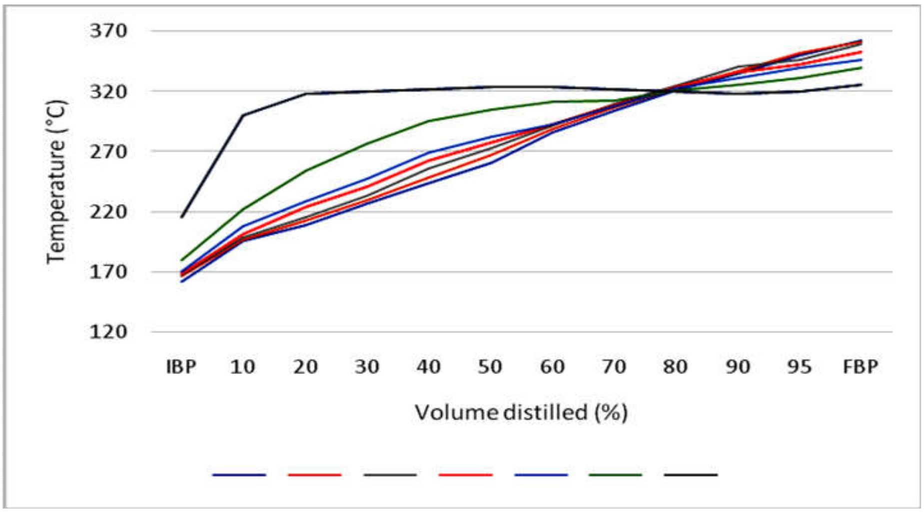

Figure 4. Graphic representation of the distillation flow of the tested fuel samples

The IBP in biodiesel produced from WCO is 216 °C and of the fossil diesel is 165 °C. This indicates a possible cold start of the engine when using biodiesel. The 50 % distillation temperature is significant for the warm-up period of the engine and it is desirable to be as low as possible so that the engine achieves the operating temperature faster. The 50 % distillation temperature for pure biodiesel is 324 °C, and for fossil diesel is 261 °C. Blends containing a low biodiesel volume fraction (B5; B10) have a slight difference in the temperature of 50% of distillation compared to D100. The final boiling point (FBP) is another parameter important for fuel characterization. The FBP for the biodiesel sample was 326 °C, while for the diesel sample it was 362 °C. The obtained data show that the combustion of biodiesel is more complete than the combustion of diesel, which makes the emission of exhaust gases considerably smaller. However, a smaller FBP for biodiesel may also be an indicator that the double bond of unsaturated alkyl chains could be polymerized at high temperatures. This obstructs the evaporation of the liquid at the end of the distillation process, which can lead to the formation of rubber, sediment in the engine injectors or on the surface of the piston, causing corrosion.

Cetane index

The CI is a measure of the ignition quality of diesel fuels, and a high cetane number (CN) implies short ignition delay. The longer the fatty acid carbon chains and the more saturated the molecules, the higher the CN [22]. It should be emphasized that the higher the CN, the better will be the combustion. Residual methanol in biodiesel is responsible for a decrease in the CN [16]. The increase of the volume fraction of biodiesel resulted in a constant linear decrease of the CN, which is in line with other studies that showed that the CN of petrodiesel is increased upon blending with biodiesel [23]. The CN of the B100 sample was slightly lower (46.06) than prescribed in ASTM D6751 and EN14214, while D100 and the blend samples met their respective requirements.

Heat of combustion

The heat of combustion is the thermal energy that is liberated upon combustion, so it is commonly referred to as energy content. The addition of biofuels decreases the heat of combustion of fossil diesel [24-26]. Our results were in line with that. The decrease was more expressive in the samples containing more than 20 % biodiesel. The heat of combustion of biodiesel (B100 - 38.3 MJ/kg) was 11% lower than that of diesel (D100 - 42.86 MJ/kg). Although the standards referenced in this work do not prescribe requirements for the heat of combustion of fuels, its values are important in planning fuel supplies and predicting technological process capacities. This reduction in heat of combustion would result in increased fuel consumption when burning in the engines.

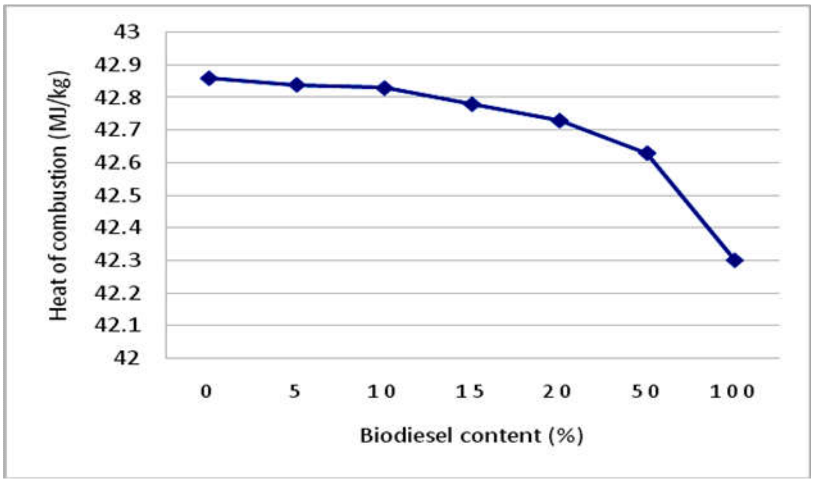

Figure 5. The influence of biodiesel content on the heat of combustion of the fuel

Content of sulphur, contaminants, ash, FAME and linolenic acid

The content of sulphur, contaminants, ash, FAME and linolenic acid in the investigated samples are presented in Figure 6. The obtained experimental data showed that the content of contamination, FAME and linolenic acid increased linearly with the increase in the volume fraction of biodiesel in the fossil diesel. The ash content remained the same in all samples. Only the sulphur content linearly decreased with the increase of the biodiesel volume fraction.

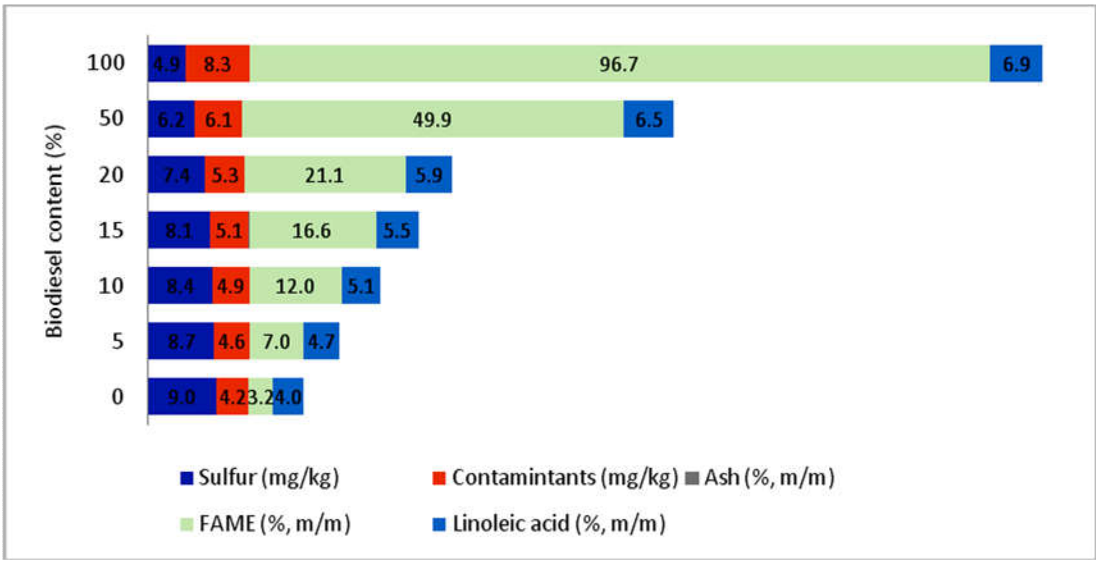

Figure 6. Content of sulphur, contaminants, ash, FAME and linolenic acid depending on the biodiesel content

Contaminants content

Contaminants such as partial glycerols, unreacted TAG as well as unseparated glycerol, free fatty acids (FFA) and residual alcohol can lead to severe operational problems when using biodiesel [13]. As previously stated, if biodiesel is contaminated with methanol, it will fail to meet the minimum FP specified in relevant fuel standards [1]. The upper limit for contaminants is only presribed for B100 but not for D100 and the biodiesel-diesel blends. The contamination content was the highest in B100 (8.3 mg/kg), almost twice as much than in D100 (4.2 mg/kg), but still within the EN14214 limit.

Sulphur content

The sulphur content in the B100 sample (3.92 mg/kg) is 57.25% less than sulphur content in D100 (8.96 mg/kg), which confirms the ecological significance of the biodiesel produced from WCO, but also of biofuels in general. All other samples met the prescribed criteria for the sulphur content (<10.0 mg/kg), as well.

FAME content

One of the specifications that ensure safe and satisfactory engine operation of biodiesel in its pure form or as blending stock is the content of fatty acid methyl esters (FAME) [27]. Contrary to the assumption that the commercially obtained sample D100 is pure, our research revealed that it contains 3.20 % biodiesel. The standard EN 14214:2012 prescribes the minimum FAME content of 96.5%. The maximum value is not prescribed. In our case, the FAME content in the biodiesel sample (B100) is 96.70%, which is satisfactory.

Linolenic acid content

Establishing the relative proportions of oleic, linoleic, linolenic and other fatty acids is helpful for a better characterization of biodiesel's propensity to oxidize [28]. The autoxidation of unsaturated fatty compounds proceeds with different rates depending on the number and position of double bonds [29-30]. Most biodiesel fuels contain significant amounts of esters of oleic, linoleic or linolenic acids; influencing the oxidation stability of the fuel. Small amounts of more highly unsaturated fatty compounds have a disproportionately strong effect in reducing oxidation stability [14]. The LA content of the B100 sample was within limits of the standard EN 14214:2012 (max. 12%). In sample D100 the LA content was 4.01 %, whereas in sample B100 it reached 6,88 %, which means a higher volume fraction of biodiesel increases oxidative degradibility of the fuel.

Ash content

The ash content of the fuel is essential for the choice of the appropriate combustion and gas-cleaning technologies [31]. The analysed samples met the requirements regarding this parameter. As a general rule, a lower ash content is preferable.

Water content

Another important aspect of fuel quality is the water content. It is a parameter that entirely depends on the process of production of biofuels and the quality of biofuels. The hygroscopic nature of the biodiesel can lead to an increase in soluble water content during the storage [32]. Biodiesel has the feature to absorb more moisture than petroleum diesel since FAMEs are hygroscopic compounds. Research has shown that, under constant relative humidity conditions, biodiesel absorbs 6.5 times more moisture than diesel [33]. Our experimental results confirmed that the addition of biodiesel in diesel increases the water holding capacity of the blend. The standard EN 14214:2012 sets 500 mg/kg as the maximum water content. Among the investigated samples, B100 had the highest value of water content (374.4 mg/kg) which was still below the allowed upper value. A high water content would lead to problems such as water accumulation and microbial growth during storage and transport of biodiesel and diesel [34].

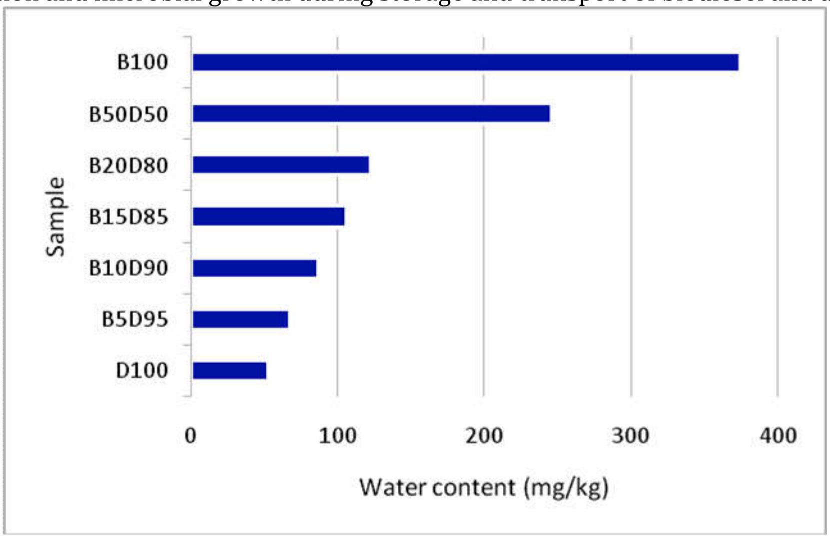

Figure 7. Water content depending on the biodiesel content

CONCLUSION

Prior to investigating the quality requirements of biodiesel-diesel blends (B5-B20) according to the standard ASTM D7467, the physicochemical parameters of B100 and D100 were evaluated according EN14214, ASTM D6751 and ASTM D975. It was found that all analysed parameters outlined by these standards were met, except for the CN in case of B100. Despite the deviation in the CN, as expected, all blends B5-B20 fulfilled the requirements of their physicochemical properties set by ASTM D7467. Additionally, the B50 which is not encompassed in the standards, proved to have desirable properties (FP, CI, viscosity, water content, sulphur content, ash content), as they were also within the limits of the ASTM D7467. The viscosity values of all samples were in the range between 2.58 and 4.01 mm^2/s and within limits of their respective standards. A high CFPP causes the engines to clog up more easily. The

CFPP of the analysed samples ranged between -13 °C and –2 °C. Results showed that the increase of the FP is insignificant all the while the biodiesel content is below 50%. A higher FP means safety during handling, transportation, and storage. The FP of pure biodiesel was significantly higher if compared to fossil diesel and the B50 blend. The distillation characteristics of the seven examined samples are such that they indicate a difficult cold start of the engine using biodiesel. The combustion and hence the engine motor efficiency depend on the CN. In our work, the increase of the volume fraction of biodiesel resulted in a constant linear decrease of the CN. The CN of the B100 sample was slightly lowerthan prescribed in ASTM D6751 and EN14214. By decreasing the heat of combustion, the fuel consumption is increased and according to the obtained data that is more evident in samples containing more than 20 % biodiesel. Further on, the content of contamination, FAME and linolenic acid increased linearly with the increase in the volume fraction of biodiesel. The D100 had a higher content of sulphur than B100, which confirms the ecological significance of biofuels in general. There was a linear increase of LA content in samples containing a higher biodiesel content, meaning that such samples are more prone to oxidative degradability. Fuels with low ash content are preferable. The same applies to the water content. The analysed samples met the requirements regarding these parameters.

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