ANALYSIS OF FUEL PROPERTIES OF BIODIESELS PREPARED FROM SELECTED VEGETABLE OILS (VIRGIN OILS AND THEIR WASTE)

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ABSTRACT

IThe impact of different frying times on the fuel properties of biodiesel produced from selected vegetables oils were investigated. Palm olein, soybean, groundnut and palm kernel oils were used for frying for 1, 3, 5 and 7 hours at a constant temperature of 185°C. The physico-chemical properties of the virgin oils were determined and compared with standards for edible oils. The oils and their different waste oils were used for biodiesel production via transesterification reaction. The fuel properties of the biodiesel produced were determined and compared with standard. The peroxide, iodine, acid, free fatty acid and saponification values of the vegetable oils ranged from 0.01 to 29.00 meq/kg, 0.92 to 76.30 gI₂/100g, 11.14 to 22.30 mg/KOH/g, 5.61 to 11.23% and 61.89 to 67.60 mg/KOH/g respectively. Biodiesel yield from palm olein, soybean, groundnut and palm kernel oils obtained were found to be 83.65, 87.46, 81.87 and 79.12% respectively. The frying times had very high significant effect on the viscosity of the biodiesels at 40°C and had insignificant effects on the cloud point, pour point, flash point and specific gravity of the biodiesels. The viscosity of the oils were found to be above the standard viscosity for biodiesel at 40°C. Some of cloud point values were outside the standard range. However the pour point, flash point and specific gravity of the oils were within acceptable limit. The vegetable oils and their waste derivatives produced using different frying times can be employed in diesel formulations with additives to enhance their quality.

Keywords: Vegetable oils, waste oils, frying times, transesterification, fuel properties

INTRODUCTION

The need for renewable fuels has been motivated by population growth, environmental concerns, and energy security concerns (Uthman and Abdulkareem, 2014). Considering the world's rapidly growing population, one of the major concerns is global traffic, which is reported to be caused by transportation. In recent years, the United Nations has repeatedly urged for the abandoning of fossil fuels in favour of cleaner and renewable energy fuels such as hydro, biomass, wind, solar, geothermal, hydrogen, and nuclear (Hansen et al., 2019). Alternative innovative and renewable fuels have the potential solve today's socioeconomic problems to (Stancin et al., 2020). However, biodiesel has been widely regarded as a alternative energy source for addressing these difficulties and as a viable replacement to diesel fuel (Adekanbi et al., 2021). Biodiesel is so named because it is made from biological products and functions similarly to petrodiesel. Biodiesel is a form of biofuel with significant promise for meeting heavy energy demands in a net-zero economy. It is a renewable and biodegradable fuel that may be produced from any oil-containing seed or animal fat (Ayoub et al., 2021). It is manufactured by chemically reacting lipids with an alcohol in the presence of a catalyst to form fatty acid methyl esters (Efavi et al., 2018).

Vegetable oil is one of the renewable fuels with appealing environmental benefits due to the fact that it is derived from renewable resources, but the high cost of pure vegetable oils, which accounts for 70 to 80% of total production costs, poses an economic issue. Importantly, vegetable oils have recently acquired appeal due to their environmental benefits and production from renewable resources (Demirbas. 2002). Vegetable oils utilized in biodiesel production include rapeseed, palm, sunflower, and soybean oils. However, due to competition with edible oil markets and subsequent price increases, their availability for use in biodiesel synthesis is limited (Kansedo et al., 2009). Furthermore, biodiesel production from edible oil has a negative environmental impact since it takes a substantial amount of arable farmland (Ahmed et al., 2011). As a result, it is vital to look for new sources of biodiesel production, such as free waste frying oil. When food crops such as palm oil are diverted to energy, feedstock selection can have a significant impact not only on the production process but also on food prices (Morais et al., 2010; Akhihiero et al., 2021). Every day, several litres of waste vegetable oils generated in homes. are our eating establishments, and restaurants; many are re-used without consideration for negative health repercussions, while other litres are discarded,

ignoring the opportunity of converting them into usable goods. These waste oils have enormous potential as biodiesel feedstock. Deep fat frying is one of the oldest and most widely used food processing processes, involving briefly immersing a product in heated oil (150-200°C) until it achieves an acceptable minimum internal temperature (Sahasrabudhe et al., 2017). It is a low-cost method in which temperature and heat interact to affect the organoleptic aspects of food such as flavour, texture, and colour (Bouchon and Pyle, 2005; Saguy and Dana, 2003). Frying is a simultaneous heat and mass transfer method that induces product dehydration due to high oil temperature, resulting in a product with a porous and crispy surface and a soft and moist interior (Dana and Saguy, 2006). Starch gelatinization, protein denaturation, aromatizing and colouring via Maillard reactions, quick cooking, and texture and flavour development are the primary reactions involved in this process (Choe and Min, 2007; Ziaiifar et al., 2008). By hydrolysis, oxidation, and polymerization, frying alters the flavour and stability of oils (Xu et al., 2015). Long frying stability, fluidity, bland flavour, low propensity to froth or produce smoke, low tendency to polymerize, oxidative stability of the oil in the fried meal during storage, and cost are all factors in oil selection for frying (Aydar et al., 2016).

The transesterification approach is currently the most prevalent method for producing biodiesel. This is because the physical properties of fatty acid esters (biodiesel) are quite similar to those of diesel fuel and the procedure is reasonably straightforward, which appears to be the best option. Furthermore, fatty acid methyl or ethyl esters can be burnt directly in unmodified diesel engines with very little deposit formation. In the transesterification technique, animal fat is transesterified with alcohols such as methanol or ethanol in the presence of a catalyst to produce biodiesel. The transesterification process can be performed under а variety of reaction circumstances. including molar ratios. temperatures, reaction time, and catalyst concentration. Therefore, this study is aimed at investigating the effects of different frying times at 185°C on the yield and fuel properties of biodiesels produced from selected vegetable oils.

METHODOLOGY

Sample collection:

Palm olein oil (POO), soybean oil (SO), groundnut oil (GO) and palm kernel oil (PKO) were purchased from a local market in Benin City, Edo State, Nigeria. Analytical grade methanol and other chemicals used were purchased from Fluka Chemie, Germany and were used without further purification. **Determination of peroxide value** The method for determination of peroxide concentration is based on the reaction of the hydroperoxide group with KI or Fe²⁺ as described by Ali et al. (2022). The result of the iodometric titration is expressed as the peroxide value. This method determines all substance in terms of milliequivelents of peroxide per 1000g of sample. 5g of each oil were weight into 250ml Erlenmeyer flask, and 30ml of the 3:2 acetic acid-chloroform solution was added, 0.5ml of saturated potassium iodide solution was also added. The solution was allowed to stand with occasional shacking for exactly 1min, and 30ml of distilled water and drops of starch solution was added. The solution was titrated against 0.1N sodium thiosulfate $(Na_2S_2O_3)$ until end point. The peroxide value was calculated using Equation (1).

$$PV(Eq.) = \frac{N \times V \times M_{wt} \times 1000}{W_t}$$
(1)

Where:

 $N = normality of Na_2SO_3, V = volume of Na_2SO_3$ consumed in the titration, M.Wt = molecularweight of Na_2SO_3, Wt = weight of sample.

Determination of iodine value

The iodine value was determined according to methods by Kyari (2008), 0.13g of oils were weighed into in a glass weighing scoop and placed in a 250ml conical flask. 15 ml of carbon

tetrachloride was added to dissolve the oil followed by the addition of 25 ml wijs reagent. A stopper was then inserted and the content of the flask was shaken gently. The flask was then placed in the dark for 30mins. At the end of this period, 20ml of 10% aqueous potassium iodide and 150ml of distilled water were added using a measuring cylinder. The content was titrated with 0.1M sodium thiosulphate solution until the yellow color almost disappeared. A few drops of starch indicator were added and the titration continued by adding sodium thiosulphate dropwise until blue coloration disappeared after vigorous shaking. The above process was repeated with a blank under the same conditions. The iodine value (IV) is given by the expression:

$$IV = \frac{12.69 \times T \times (V_1 - V_2)}{m}$$
(2)

Where $V_1 = V$ olume of sodium thiosulphate used for the blank, $V_2 = V$ olume of sodium thiosulphate used for the test portion, T =Normality of sodium thiosulphate used, m =Mass in g, of the test portion.

Determination of acid value

Acid value is an important indicator of vegetable oil quality. The acid value of the virgin vegetable oils were determined according to method reported by Abdullah and Salimon (2009). 2g of the oil was dissolved in 25ml of a mixture of neutral ethylether and ethanol (1:1 ratio). The mixture was titrated with 0.1N potassium hydroxide using a phenolphthalein indicator to end point with constant shaking for which a permanent pink colour was observed and the volume of 0.1N potassium hydroxide was noted. The acid value is given by the expression:

Acid value =
$$56.1 \times V \times M/W_s$$

(3)

Where Ws = weight of sample, V = volume of potassium hydroxide solution titrated, M =molarity of potassium hydroxide.

Determination of free fatty acid

The acid value is equivalent to the percent free fatty acid multiplied by the factor of 1.986, when the percent free fatty acid is based upon oleic acid as reported by (Adefarati, 1986).

Determination of saponification value

The saponification value of the oils were determined using method described by Salimon

et al. (2012a). A known quantity of oils were refluxed with an excess amount of alcoholic KOH. After saponification, remaining KOH was estimated by titrating against standard acid. 2 g of oil was taken in the flask and 25 ml alcoholic KOH was added until the oil dissolved completely. The flask was connected to air condenser and boiled for 30 minutes on boiling water bath. Then the mixture was cooled and 3 drops of phenolphthalein was added as an indicator. The mixture was then titrated against standard 0.5N HCI until the pink colour disappeared. Similarly the blank was titrated in the absence of oil. The saponification value was determined using Equation (4).

Saponification value =
$$\frac{(Blank-titre) \times 28.06}{weight of oil(g)}$$
(4)

Preparation and transesterification of the

vegetable oil

Virgin POO, SO, GO and PKO and their waste fried derivatives were heated to 185°C to remove moisture. The waste fried derivatives of POO, SO, GO and PKO were obtained by using the oils to prepare beans cake after 1, 3, 5 and 7 hours of frying. A total of 20 oils were obtained and used for the transesterification reaction with methanol and catalyst after preparation. The virgin oils were used for biodiesel production because of their poor physico-chemical properties and hence considered not edible. 200 ml of methanol was poured into the glass blender pitcher at room temperature and 3.5 g sodium hydroxide was added slowly to the blender as catalyst. The mixture was properly swirled for about 2 minutes. The reaction gave sodium methoxide. In the different transesterification reactions, 1 litre of the individual virgin and waste frying oils were added into methoxide mixture and blended for 20 - 30 minutes. The mixture was poured into a separating funnel to separate into layers and left for 24 hours after the reaction time. The top layer which is the biodiesel was collected in a PET bottle (). The biodiesel of the different oils were assigned as BPOO, BSO, BGO and BPKO.

Yield and Fuel Properties of Biodiesels

Determination of Yield:

The percentage yield of BPOO, BSO, BGO and BPKO was calculated using the Equation (5).

Biodiesel yield (%) = $\frac{mass \ of \ biodiesel}{mass \ of \ oil} \times 100\%$ (5)

Determination of Flash Point

The flash points of the biodiesel samples were measured using the Cleveland Open Cup method. The biodiesel samples were heated to a specified level and the correct size of the test flame was periodically directed to the vapor of the sample. The temperature of the biodiesel samples in which the steam explodes was marked as a flash point.

Determination of Kinematic Viscosity

Here the viscosity of the biodiesel samples were measured at 40°C using ASTM D445. A clean dry calibrated viscometer was selected and the bath was allowed to attain its set temperature. The viscometer was charged and immersed in the bath with the holder suspending it. The time required for the sample to pass from the first timing mark to the second mark was taken and calculated. Two measurements were taken and the average was calculated as the kinematic viscosity at 40°C when the bath set temperatures was 40°C.

Determination of Pour Point

Pour point can be defined as the lowest temperature, expressed as a multiple of 3°C at which the were observed to flow when cooled and examined under prescribed conditions (ASTM D 97–66) described by Salimon *et al.* (2012b). About 10 ml each of the different biodiesels were poured into a U-tube and then

covered with aluminium foil at the top of that tube. Then, the sample in the U-tube was placed overnight in the freezer (-80°C). After a night, sample was taken out and the cover was removed. The tube was turned down while the temperature recorded when the sample had started to flow.

Determination of specific gravity

The density of the biodiesel were determined using a specific gravity bottle based on standard ASTM D792 at 23°C (Hariram *et al.*, 2018). Firstly, the empty 50 ml specific gravity bottle mass (m_1) was obtained using a digital weighing balance. Then the mass (m_2) of the specific gravity bottle filled to the brim with the oil was recorded and adopted using Equations (6) and (7).

Density
$$\left(\frac{kg}{ml}\right) = \frac{m_2 - m_1}{v}$$
 (6)

Where:

m₁ = mass of empty specific gravity bottle withthe lid on it (kg)

 $m_2 = mass$ of specific gravity bottle filled with the biodiesel (kg)

v = volumetric capacity of the bottle for specific gravity which is 50ml

Density
$$\left(\frac{kg}{m^3}\right) = specific \ gravity \times$$

997.6

(7)

STATISTICS AND DATA PROCESSING

The effect of the different frying times on the yield and fuel properties of the biodiesel produced from the different oils under study was examined using analysis of variance (ANOVA) at a significant level of 5% (i.e., a confidence level of 95%). ANOVA is a statistical technique for evaluating the significance of factors on responses at a probability level.

RESULTS AND DISCUSSIONS

Physico-chemical properties of virgin vegetable oils

purchased locally is given in Table 1 in comparison with SON/NIS (1992) standards for edible oils.

The physico-chemical properties of the virgin vegetable oils (POO, SO, GO and PKO)

Physico-chemical properties	POO	SO	GO	РКО	Standard
Peroxide value (meq/kgoil)	0.01	0.01	18.00	29.00	< 10.00
Iodine value (gI ₂ /100g)	1.67	0.92	76.31	70.30	45 - 53
Acid value (mg/KOH/g)	11.46	14.93	22.30	11.14	< 0.60
Free fatty acid (%)	5.77	7.52	11.23	5.61	< 3.50
Saponification value (mg/KOH/g)	61.89	61.91	65.92	67.60	195 - 205

Table 1: Physico-chemical properties of virgin vegetable oils

From Table 1, the peroxide value of POO, SO, GO and PKO were found to be 0.01, 0.01, 18.00 and 29.00 meq/kg respectively. The peroxide values of POO and SO were lower than standard of 10.00 meq/kg while that of GO and PKO were higher. The high peroxide values of GO and PKO may be due to the continuous exposure of the oils to oxygen which causes continuous oxidative rancidification of oils during the period of storage. The iodine values of POO, SO, GO and PKO are 1.67, 0.92, 76.31 and 70.30 gI₂/100g. The iodine value could be used to quantify the amount of double bond present in the oil which reflects the susceptibility of the oil to oxidation. Oils with iodine value less than 100

 $gI_2/100g$ of oil non-drying oils; are correspondingly, Aremu et al. (2006) reported that the lower the iodine value the lesser the number of unsaturated bonds; thus the lower the susceptibility of such oil to oxidative rancidity. The iodine values of GO and PKO were above the SON/NIS standard of 45 - 53 gI₂/100g for edible oils. The free fatty acids of POO, SO, GO and PKO are 5.77, 7.52, 11.23 and 5.61% respectively and were all above the recommended standard of less than 3.5% for edible oils. The acid values of POO, SO, GO and PKO are 11.46, 14.93, 22.30 and 11.14 mg/KOH/g respectively. The acid value of the oils were significantly higher than the recommended minimum of 0.6 mg/KOH/g. The lower the acid value of oil, the fewer fatty acid it contains which makes it less exposed to rancidity (Obasi *et al.*, 2012). Also, high acid value of the oils can be attributed to increase in the inhibition of enzymes activity on the oils (Alajtal *et al.*, 2018), during the period of storage before purchase. The saponification value of POO, SO, GO and PKO are 61.89, 61.91, 65.92 and 67.60 mg/KOH/g respectively and were all below the standard of 195.00 - 205.00 mg/KOH/g. The properties of the oils did not completely meet the SON/NIS 1992 requirements for edible oil which may be as a result of duration of storage, processing and storage conditions of the oils before purchase. The oils were hence used for biodiesel production via the transesterification reaction with methanol and catalyst.

Yield of biodiesels

The yield of biodiesel prepared from virgin POO, SO, GO and PKO is given in Figure 3.1.



Figure 3.1: Yield of biodiesels from selected vegetable oils

The yield of biodiesel prepared from virgin POO, SO, GO and PKO are 83.65, 87.46, 81.87 and 79.12% respectively. From Figure 3.1, it can be seen that the yield of biodiesels increased according to the order: PKO < GO < POO < SO. This indicate that highest yield was observed

with SO, while lowest yield was observed with PKO.

Viscosity of biodiesels

The kinematic viscosity of BPOO, BSO, BGO and BPKO obtained at 40 is given in Table 2.

S/N	Frying times		P-value			
D/11	(hr)	BPOO	BSO	BGO	ВРКО	
1	0.0	7.86	7.86	7.78	7.80	
2	1.0	7.14	7.64	7.12	7.62	
3	3.0	7.60	8.06	7.70	7.98	< 0.0001
4	5.0	11.78	10.03	10.62	10.62	
5	7.0	6.48	6.99	7.00	7.60	
ASTM D6751			1.90 - 6.00	mm ² /s		

Table 2: Viscosity of biodiesels at 40°C

The viscosities at 40°C of POO, SO, GO and PKO ranged from 6.48 to 11.78 mm²/s, 6.99 -10.03 mm²/s, 7.00 - 10.62 mm²/s and 7.60 - 10.62 mm^2/s respectively for 0 to 7 hrs of frying time (Table 2). The viscosity of all biodiesels produced at 40°C were above the ASTM D445 standard of 1.9 - 6.0 mm²/s. High values of viscosity causes blockage in the nozzles, leading to spraying, combustion, carbon accumulation on the piston rings, decreases the engine power and degradation of engine oil (Oguz, 2004). Maximum viscosity of the biodiesels were achieved at 40°C after 5 hrs of frying. This indicate that more alkyl bonds of the biodiesel were formed at 5 hrs of frying before the degradation in the viscosity values due to breakage in bonds (Anekwe and Ajiwe, 2015). Analysis of variance showed that the different frying times had very high significant effects on the viscosity of the biodiesels with p-values < 0.0001 at 40°C.

Cloud point of biodiesels

The cloud points of BPOO, BSO, BGO and BPKO are given in Table 3 for the virgin vegetable oil and oils obtained at different times in comparison with ASTM D6751 standard of cloud point of biodiesels.

S/N	Frying times					
(hr)	(hr)	BPOO	BSO	BGO	ВРКО	P-value
1	0	21.00	24.00	21.00	23.00	
2	1	5.00	12.00	9.00	12.00	
3	3	22.00	8.00	20.00	18.00	0.0636
4	5	11.00	22.00	13.00	22.00	
5	7	20.00	7.00	20.00	20.00	
ASTM D6751						

Table 3: Cloud Points of biodiesels

From Table 3, the cloud points of BPOO, BSO, BGO and BPKO ranged from 5.00 to 22.00°C, 7.00 to 24.00°C, 9.00 to 21.00°C and 12.00 to 23.00°C respectively for 0 to 7 hrs of frying time. Most of the cloud points obtained for the biodiesels were above the ASTM D6751 standard of -3.00 to 12.00°C. Cloud points above the standard indicate that wax appeared in the biodiesels at higher temperatures. This indicate

Pour point of biodiesels

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that the biodiesels can only be used in cold weather countries with cloud point depressers as additives. From the analysis of variance done on the effects of the different frying times on the cloud points of the biodiesels at 5% significant level showed that the effects of the different frying times with p-value of 0.0636 is slightly insignificant on the cloud point of BPOO, BSO, BGO and BPKO.

The pour points of BPOO, BSO, BGO and BPKO obtained at different frying times is given in Table 4

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	S /NI	Frying times		Pou	r Point (°C)		- D voluo
	S/N (hr)	BPOO	BSO	BGO	ВРКО	r-value	
-	1	0	7.00	7.00	9.00	8.00	0 6 4 1 2
	2	1	-6.00	8.00	-4.00	9.00	0.0413

Table 4: Pour Point of biodiesels

3	3	9.00	-9.00	8.00	12.00	
4	5	-3.00	11.00	-5.00	-5.00	
5	7	6.00	-7.00	8.00	7.00	
ASTM	D6751					

The pour point values of BPOO, BSO, BGO and BPKO from Table 4 ranged from -6.00 to 9.00°C, -9.00 to 11.00°C, -5.00 to 9.00°C and -5.00 to 12.00°C respectively for 0 to 7 hrs of frying time. All values are within the ASTM D6751 standard of -15.00 to 16.00°C. Low pour point values may cause problems during cold weather hence oil having negative pour points can be applied during cold weather as a lubricant due to their ability to gel at low temperature. ANOVA to determine the effect of the frying times of the

S/N Frying times			P-value			
D/11	(hr)	BPOO	BSO	BGO	BPKO	i vulue
1	0	184.00	184.00	180.00	182.00	
2	1	189.00	192.00	186.00	188.00	
3	3	188.00	189.00	184.00	187.00	0.3595
4	5	190.00	180.00	187.00	180.00	
5	7	176.00	192.00	178.00	189.00	
AST	ГМ D6751		130.00°C	minimum		

I WOIC COLLUDIE DOINED OF DIOWICDO	Table	5:	Flash	points	of	biodiesel
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oils on the pour points gave p-value of 0.6413, which indicate that there was no significant effect on pour point values by the different frying times.

Flash point of biodiesels

The flash points of the different oil types and their frying times is given in Table 5 in comparison with ASTM D6751 standard of biodiesel.

From Table 5, the flash points of BPOO, BSO, BGO and BPKO ranged from 184.00 to

190.00°C, 180.00 to 192.00°C, 178.00 to 187.00°C and 182.00 to 189.00°C respectively for

thee different frying times. From the study, flash point was improved by frying from 1 to 5 hrs due to degradation of some impurities (Anekwe and Ajiwe, 2015). The flash points of all biodiesel samples are higher than the recommended ASTM D6751 standard minimum of range of 130°C. Biodiesel with lower flash point values burn easily (more flammable) and need more care in handling compared to oil with higher

Table 6: Specific Gravity of Biodiesel

flash points. The different frying times had insignificant effect on the flash points of the BPOO, BSO, BGO and BPKO with p-value of 0.3595.

Specific gravity of biodiesels

The specific gravity of the biodiesels if given in Table 6 in comparison with ASTM D6751 standard for biodiesel.

S/N	Frying times		D volue			
5/1	(hr)	BPOO	BSO	BGO	ВРКО	
1	0	0.90	0.89	0.88	0.89	
2	1	0.88	0.90	0.88	0.89	
3	3	0.88	0.90	0.88	0.90	0.1768
4	5	0.88	0.90	0.872	0.89	
5	7	0.88	0.84	0.87	0.89	
ASTM D6751			0.80	- 0.94		

The specific gravity ranged from 0.88 to 0.90, 0.84 to 0.90, 0.87 to 0.88 and 0.89 to 0.90 for BPOO, BSO, BGO and BPKO respectively as seen in Table 6. The specific gravity values obtained for BPOO, BSO, BGO and BPKO were all within ASTM D6751 acceptable standard of 0.80 - 0.94. Analysis of variance showed that the different frying times had insignificant effect on the specific gravity of the biodiesel oils with p-value of 0.1768 (> 0.05).

CONCLUSION

Palm olein, soybean, groundnut and palm kernel oils were used for frying for 1, 3, 5 and 7 hours at a constant temperature of 185°C. The virgin oils and their different waste oils were used for biodiesel production via transesterification reaction. The yield and fuel properties of the different biodiesel produced were determined and compared with standards. The yield of biodiesel of palm olein, soybean, groundnut and oils obtained palm kernel from their transesterification reactions were found to be 83.65, 87.46, 81.87 and 79.12% respectively. The different frying times had very high significant effect on the viscosity of the biodiesels at 40°C but of insignificant effect on the cloud point, pour point, flash point and specific gravity of the biodiesels. The viscosity of the oils were found to be above the standard viscosity value for biodiesel at 40°C. Some of cloud point values were outside the standard range. However the pour point, flash point and specific gravity of the oils were within the acceptable limit. It can be concluded that the vegetable oils and their waste derivatives obtained after frying for a period of time can be employed in diesel formulations with additives to enhance their performances.

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