

A New Trend in Diesel Adulteration Emerging in South Africa – A Look at Biodiesel, Commercial Diesel and Adulterated Diesel

F.B. Waanders, R. J. Venter and R Bekker and L. Hattingh

Abstract—A comparative analysis of biodiesel produced, that aligns with the SANS 1935:2011 standard, with commercial petroleum diesel samples indicated that some of the commercial diesel samples met the SANS 342:2016 standard quality, while some diesel samples contained high paraffin levels, indicative of contamination. Diesel adulteration with paraffin additions raises concerns about combustion efficiency, engine reliability, and possible environmental impacts.

The biodiesel samples were prepared by using waste cooking oil (WCO) through transesterification, using alkaline (KOH) and acid (H₂SO₄) catalysts, where the effects of catalyst loading and catalyst type, on the yield and the density, flashpoint, high heating values (HHV), GC-MS measurements were used to determine optimal biodiesel production conditions.

The results demonstrate that biodiesel quality and yield depend significantly on catalyst type and loading with KOH producing biodiesel that aligns with the SANS standards, while H₂SO₄ show less consistent compliance.

Four commercial diesel samples and a pure paraffin sample were sources from fuel stations in the area around Potchefstroom, South Africa. Two samples showed a paraffin content of up to 60%, much higher than the allowed standard of 5%, resulting in the lowering of the density, flashpoint, HHV and cetane number of the diesel.

Laboratory prepared mixtures of 1:0.1 up to a 1:1 diesel-paraffin ratios were made to determine whether an easy way could be found to determine the paraffin content of any commercially available diesel. At a ratio of 1:0.6 diesel-paraffin the flashpoint dropped significantly from 60°C to less than 40°C, whilst the density showed a steady decrease from 0.83 kg.m⁻³ to 0.81 kg.m⁻³. The HHV stayed relatively constant. The correlation of diesel-paraffin ratios with n-alkane peak areas shows a clear and predictable increase in the n-alkane peak area and the ratio of n-alkane peak area to the internal standard area as the paraffin concentration increases, with a linear relationship between diesel-paraffin mixture ratio and n-alkane/internal standard area ratio, making it easy to determine the paraffin content in any adulterated diesel sample.

Keywords— Adulterated Diesel, Biodiesel, Commercial Diesel, Contaminants.

I. INTRODUCTION

Diesel fuel is an essential fuel to drive critical societal and economic functions. A disadvantage is the fact that high unwanted emissions occur, contributing significantly to both air pollution and the accumulation of greenhouse gases in the atmosphere [1]. Particulate matter (PM), sulphur oxides (SO_x), nitrogen oxides (NO_x), and carbon dioxide (CO₂) are typically released during the combustion of standard diesel fuel [2], which represent significant risks to the environment and human health.

Due to the drastic increase in diesel prices, some institutions started, as far back as 2019, to add much cheaper unregulated illuminating paraffin to commercially available diesel and the occurrence of this illegal practice has surged, with an estimated loss of about R3 billion in annual tax revenues for the government [3]. The SANS 342:2016 standard [4] is being used in South Africa to ensure diesel sold at fuel outlets adhere to it. Increasing diesel supplies with more volatile contaminants such as paraffin, may increase sales and revenue, but the degradation of the diesel quality can negatively affect engine performance, longevity, and emissions. With the increased occurrence of adulteration of commercially available diesel, the fuel industry in South Africa is now calling on government to urgently take necessary action on the issue of diesel adulteration with illuminating paraffin [3].

Biodiesel produced from WCO represents a promising alternative source of energy and not only emphasises eco-friendliness but also promotes the reuse and reduction of waste [4]. Various biodiesel production routes are followed, but in this article only biodiesel, produced from waste cooking oil (WCO), will be discussed, where the SANS 1935:2011 standard [5] is adhered to. Vegetable oils cannot be used directly as a fuel, due to their high viscosity and the fact that they are edible and thus in high demand as a food source [6]. To address these issues, non-edible oils, such as WCO, are more viable feedstock options. Utilising WCO oil for renewable diesel production offers substantial benefits by converting a typically discarded material into fuel, thus ensuring less environmental pollution [7]. The biodiesel is made, through transesterification, from the mono-alkyl esters

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of the vegetable oils or animal fats, usually involving methanol and a catalyst under mild conditions [8]. Both homogeneous and heterogeneous catalysts can be used, but in this investigation only homogeneous catalysts were utilised.

The transesterification process is a three-step reaction that involves mixing the WCO with an alcohol in the presence of a catalyst where the triglycerides are converted into alkyl esters (biodiesel) and glycerol [7]. The alcohol is essential in biodiesel production, serving as acyl acceptors during the transesterification process. When methanol is used, the catalyst facilitates the transesterification reaction between the triglycerides and methanol to produce fatty acid methyl esters (FAME) and glycerol [9]. Alkaline catalysts such as KOH and NaOH are used due to their ability to provide high conversion rates of triglycerides to FAME. Acid catalysts, such as H₂SO₄, show high catalytic activity to esterify free fatty acids (FFAs) present in the feedstock, which can lead to soap formation and reduced biodiesel yield [10].

The properties of the specific paraffin and commercial diesel samples sourced are listed in table 1, indicating the lower density, flashpoint and HHV values of paraffin. Paraffin addition to diesel can thus reduce the overall fuel quality, leading to issues such as engine knocking and increased volatility [11]. The chemical compositions vary also in terms of the branched alkanes and n-alkanes which possibly could be an indication of contamination of diesel.

TABLE I: PARAFFIN AND DIESEL CHARACTERISTICS AND CHEMICAL CONTENT

Quality	Paraffin	Diesel
Acid value (mg KOH/g)	0.07	0.05
HHV (MJ/kg)	45.50	48.10
Flashpoint (°C)	52.70	76.80
Density (kg/m ³ @15 C)	0.79	0.83
Chemical functional groups	Content (%)	
	Paraffin	Diesel
Alkene	2.2	4.5
Branched Alkane	31.3	21.2
Cyclo-Alkane	3.3	3.9
n-Alkane	34.2	29.1
Poly Aromatic Hydrocarbon	6.9	3.8
Aromatics	6.2	3.0
Other	15.9	34.5

II. MATERIALS AND METHODS

A. Materials

The WCO was sourced from the local cafeteria at the North-West University, Potchefstroom, South Africa and the alkaline catalyst (KOH), acid catalyst (H₂SO₄) and methanol (CH₃OH) were obtained from Sigma Aldrich. Two unadulterated commercial diesel samples (DS1, 2) and two possible adulterated diesel samples (DS5, 6) and a paraffin sample were obtained from service stations in the Potchefstroom area, South Africa.

B. Methods

The WCO was initially filtered to remove impurities such as food particles and solid residues and transferred into a suitable container and placed in an oven set at 100°C, where it was left for 48 hours to effectively remove any water content. The properties of the WCO used as feedstock for biodiesel production are shown in table 2.

The transesterification process was conducted at 60 ± 1°C, slightly below the boiling point of methanol, and at atmospheric pressure. Each experiment was repeated three times, conducted in a fume cabinet, using a ball flask, condenser, stirrer set at a speed of 1500 rpm and hot plate, which is shown in Figure 1.

TABLE II: WCO PROPERTIES

Parameter	WCO
Moisture (ppm)	205.37
FFA (%)	0.63
Acid value mg KOH/g	1.26
HHV(MJ/kg)	39.6
Flashpoint (°C)	>200
Density (kg/m ³)	0.926

The homogeneous catalyst loading was varied between 0.25, 0.5, 1.0 wt.% for KOH; whilst for the H₂SO₄ it varied between 0.5, 1.0, 2.0 wt.% with the CH₃OH:Oil ratios for both catalysts being 5:1, 6:1, 7:1. The retention time (RT) varied between 60, 90 and 120 mins.

After the reaction was completed, the mixture cooled to room temperature and transferred to a separatory funnel where glycerol separated at the bottom and the biodiesel floated on top.

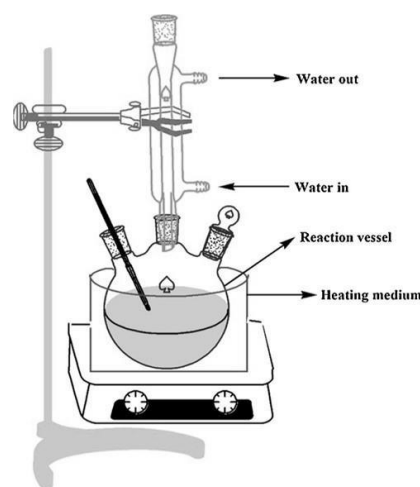


Fig. 1: Schematic of the biodiesel production set-up.

To simulate the contamination of diesel with paraffin, ten 50 ml bottles were used where 10 grams of diesel was poured into each and then paraffin was added to create 10% to 100% contamination levels and the content of the bottles mixed thoroughly.

The analytical techniques used in the present investigation to determine the properties of the samples, included: An

Agilent 7890 gas chromatograph equipped with an Agilent 5975 triple axis mass-selective detector for composition analyses. The GC-MS was fitted with a 30 m x 0.25 mm x 0.1 μm J&W HP-5ms column. The methyl ester quantification was done with an Agilent 7820A GC-FID, fitted with a 5m x 0.535mm x 0.15μm DB-HT-SimDist column curves; the determination of the boiling range distribution was done by following a modified version of the ASTM D7213 Standard Test Method [13]; an Eraflash model EREF01 apparatus was used for flashpoint determinations, following the ASTM D6450 standard test method; an Anton Paar DSA 5000 M instrument was used to measure the density; the moisture content of the samples was determined using a Karl Fischer titration method, by means of a Metrohm 831 KF coulometer; the free fatty acid and total acid number were determined by using the Metrohm 848 Titrino plus apparatus; an IK C5003 bomb calorimeter was used to determine the HHV of all diesels samples.

III. RESULTS AND DISCUSSION

A. Biodiesel production with KOH catalyst

In biodiesel production, the goal is to convert triglycerides in the feedstock into methyl esters. With the biodiesel produced with KOH as the catalyst at three different weight percent loadings, compared to the SANS biodiesel standard, as shown in table 3, the 0.5 wt.% catalyst loading yielded the highest methyl ester content and thus this catalyst loading was used in subsequent experiments.

The next step was to determine the optimal methanol to oil molar ratio as well as the ideal residence time for the reaction to take place. From table 4 it is clear that a 5:1 methanol to oil ratio and a residence time of 90 minutes yielded the optimal diesel yield with a methyl ester content of 96.4%, in accordance with the SANS 1935:2011 standard.

The results all indicate successful transesterification where the majority of triglycerides were converted to esters leaving minimal residual acid in the fuel.

TABLE III: COMPARISON OF THE BIODIESEL PROPERTIES PRODUCED WITH DIFFERENT KOH CATALYST LOADINGS AGAINST THE SANS BIODIESEL STANDARD*

Property	Biodiesel	KOH catalyst loading for biodiesel produced		
	SANS standard	0.25 wt. %	0.5 wt. %	1.0 wt. %
Methyl ester Content %	> 96.5	72	94	77
Density at 15 °C, g/cm ³	0.860-0.900	0.898	0.891	0.888
Flash point, °C, min.	101	165.6	168.5	>200
HHV, MJ/kg	36-41	39.7	39.8	39.8
Moisture Content, ppm, max.	500	138	131	25
Free Fatty Acid, %	0.5	0.136	0.080	0.066
Acid value, mg KOH/g	0.5	0.270	0.166	0.130

*The HHV and FFA not explicitly specified in the standard, but indirectly represented by the acid value

TABLE IV: COMPARISON OF THE BIODIESEL PROPERTIES PRODUCED WITH DIFFERENT METHANOL TO OIL RATIOS AT DIFFERENT TIMES AGAINST THE SANS BIODIESEL STANDARD

Quality	Standard	5:1 methanol to oil molar ratio			6:1 methanol to oil molar ratio			7:1 methanol to oil molar ratio		
		Time (minutes)			Time (minutes)			Time (minutes)		
		60	90	120	60	90	120	60	90	120
Methyl ester content %	>96.5	86.0	96.5	89.0	92.0	90.5	88.0	90.0	93.5	91.0
Density (kg/m ³ @15°C)	860-900	889	889	889	888	888	889	888	888	888
Flash point (°C)	101, min	>200	169.5	170.5	170.5	>200	172.5	169.7	169.5	172.6
HHV (MJ/kg)	36-41	43.5	43.5	42.8	44.6	45.7	42.1	45.1	44.1	45.9
Ave. moisture (ppm)	10-110	109	12	49	79	95	99	30	20	64
FFA (%)	0.5	0.097	0.066	0.078	0.074	0.094	0.057	0.084	0.1	0.107
Acid value (mg KOH/g)	0.1-0.25	0.194	0.14	0.155	0.148	0.188	0.133	0.166	0.2	0.212

From table 5 it is clear that the methyl oleate component (C18:1), remained the most abundant ester, with concentrations around 60% for all methanol-to-oil ratios. The similar levels of methyl oleate across the different ratios suggest that by 90 minutes, the available unsaturated fatty acids have been largely converted, regardless of the specific methanol-to-oil ratio.

TABLE V: FATTY ACID COMPOSITION OF BIODIESEL AT DIFFERENT METHANOL TO OIL MOLAR RATIOS AT 90 MINUTES RESIDENCE TIME

Methyl ester	Methanol to oil molar ratio		
	5:1	6:1	7:1
methyl palmitate C16:0 (%)	10.1	10.4	10.2
methyl oleate C18:1 (%)	60.2	60.1	60.2
methyl linoleate C18:2 (%)	32.1	32.0	32.2
methyl stearate C18:0 (%)	3.9	4.2	4.0
Total	96.5	90.5	93.5

The elemental analysis of the biodiesel produced at different methanol-to-oil ratios and residence times is shown in table 6. The data indicates that a residence time of 90 minutes yields biodiesel with the highest carbon and hydrogen content across all methanol-to-oil ratios. The carbon content reached a peak of 78.2% at the 5:1 ratio, while the hydrogen content was 12.1%, both of which represent improvements over the 60-minute results. This suggests a more complete transesterification process at the 90 minutes residence time, consistent with the biodiesel properties at the 90 minutes residence time with a 5:1 methanol to oil ration, as listed in table 4. Extending the reaction time to 120 minutes did not result in much improvement.

TABLE VI: BIODIESEL ELEMENTAL ANALYSIS

Residence time (minutes)	Methanol to oil ratio	Elemental			
		%C	%H	%N	%R
60	5:1	77.16	12.04	0.38	10.42
	6:1	77.58	12.10	0.38	9.96
	7:1	76.05	11.78	0.38	11.79
90	5:1	78.21	12.08	0.42	9.31
	6:1	77.26	11.71	0.42	10.30
	7:1	76.52	11.77	0	12.03
120	5:1	75.77	11.73	0.45	12.06
	6:1	75.14	11.66	0.40	12.8
	7:1	78.77	11.93	0.51	8.79

The trend in %R-values (remaining elements such as oxygen and sulphur) shows that a lower R-value correlates with a better biodiesel quality, as the oxygen content decreases when triglycerides are converted to methyl esters. Since the sulphur content in biodiesel is minimal, the R-value essentially tracks the changes in oxygen

B Biodiesel production with H_2SO_4 catalyst

With the biodiesel produced with H_2SO_4 as the catalyst at three different weight percent loadings, compared to the SANS biodiesel standard, as shown in table 7, the 2.0 wt.% catalyst loading yielded the highest methyl ester content, but still with a lower methyl ester yield than the required SANS biodiesel standard.

TABLE VII: COMPARISON OF THE BIODIESEL PROPERTIES PRODUCED WITH DIFFERENT H_2SO_4 CATALYST LOADINGS AGAINST THE SANS BIODIESEL STANDARD

Property	Biodiesel	H_2SO_4 catalyst loading for biodiesel produced		
	SANS standard	0.5 wt. %	1.0 wt. %	2.0 wt. %
Methyl ester Content %	96.5	64	46	87
Density at 15 °C, g/cm ³	0.860-0.900	0.911	0.916	0.907
Flash point, °C, min.	101	180.6	181.6	179.6
HHV, MJ/kg	36-41	19.4	18.9	24.0
Moisture Content, ppm, max.	500	204	260	359
Free Fatty Acid, %	0.5	0.406	0.517	0.714
Acid value, mg KOH/g	0.5	0.41	0.52	0.71

The highest triglyceride conversion and biodiesel quality were achieved at 2.0 wt.% H_2SO_4 catalyst loading but the quality was low and thus no further optimisation was done for the H_2SO_4 catalyst.

C Comparative analysis of commercial diesel and adulterated diesel

A chemical characterisation, to obtain a semi-quantitative comparison of the chemical content in the diesel and paraffin samples that were obtained at local fuel station, was determined by using GC-MS analysis and the results are shown in Figure 2.

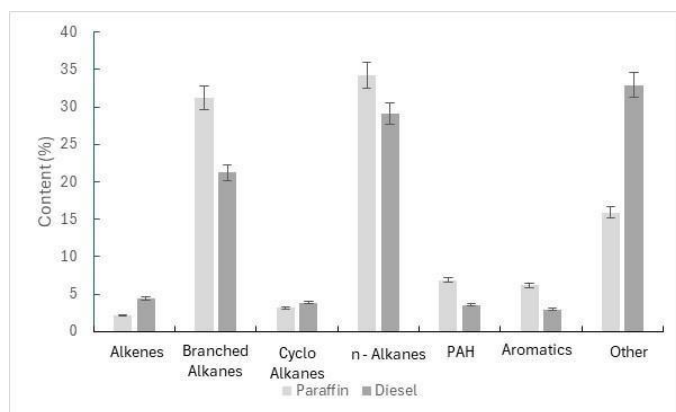


Fig. 2: Semi quantitative comparison of chemical functional groups in paraffin with chemical functional groups in diesel.

Diesel shows a higher content of alkenes and less branched alkanes, which contribute to improved combustibility and energy efficiency. On the other hand, paraffin shows a greater percentage of n-alkanes, which provide stability but result in

lower energy efficiency compared to diesel. Poly aromatic hydrocarbons (PAH) were found in both samples. Mixtures of diesel and paraffin will thus show intermediate conditions, depending on the amount of adulteration of the diesel.

In Figure 3 the total C9-C13 n-alkanes concentration for the commercial diesel (DS1 and DS2), suspect commercial diesel samples (DS5 and DS6) and the paraffin are shown, with samples DS5 and DS6 having characteristics in between the unadulterated diesel and the paraffin, indicating the possible extent of adulteration in the suspect diesel samples.

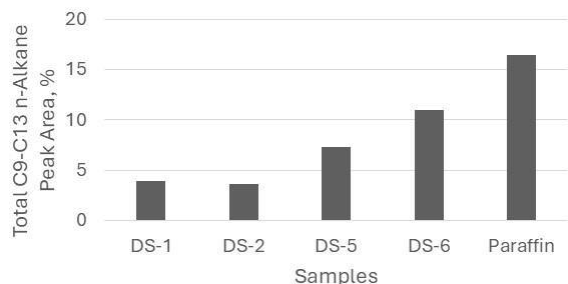


Fig 3: Comparison of the total C9-13 n-alkane peak areas for standard diesel, adulterated diesel and paraffin.

The hydrocarbon composition can be used to determine which diesel samples are under suspicion, as shown in Figures 2 and 3, but the boiling ranges of different components present in the fuel can also be used to quantify the extent of adulteration that took place by constructing the simulated distillation (SimDist) curves, with the results for sample DS1 and DS6 shown in Figure 4. To classify the boiling ranges of these samples, horizontal lines were drawn on the curve to

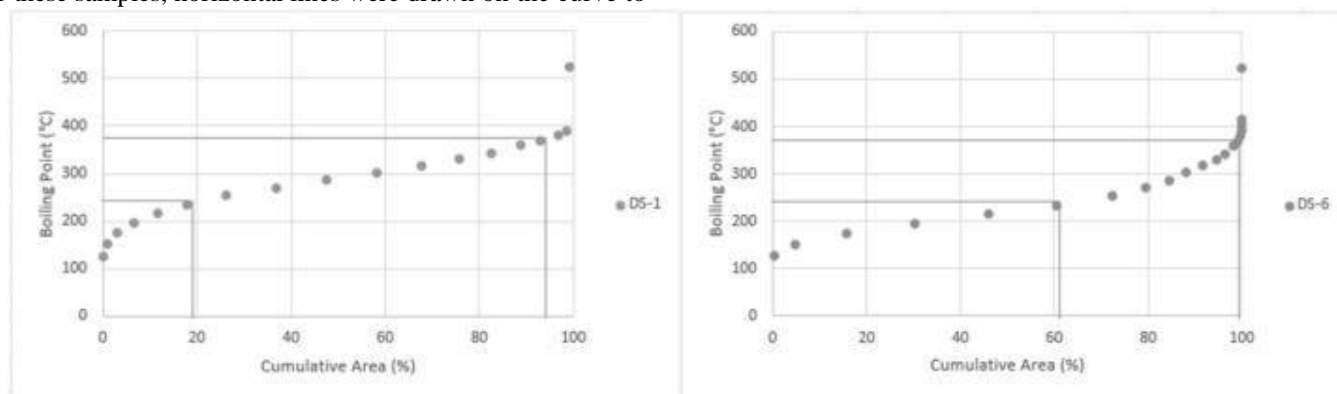


Fig. 4: SimDist curve of unadulterated diesel (DS1) as compared to an adulterated diesels sample (DS6).

Lastly, to obtain a possible general deduction method of diesel adulteration, diesel and paraffin were mixed in the ratios as listed in table 8.

mark the boiling point ranges for paraffin (150°C-240°C) and diesel (240°C-370°C). Vertical lines were added to show the proportions of each component in each sample. Where these horizontal and vertical lines intersect, they indicate the percentages of paraffin and diesel in each diesel sample. These boiling curves give a good indication of the quality of a diesel sample from a boiling range point of view.

For sample DS 1 the paraffin content is less than 20%, while the diesel fraction is about 95%, indicating that sample DS1 meets the expected quality standards for diesel. Comparable results were obtained for sample DS2.

The results for the adulterated sample (DS-6) shows a significant difference compared to diesel sample DS1. For sample DS6 the paraffin content is approximately 60% and the diesel component less than 40%. The SimDist curve for sample DS5, yielded a paraffin content of approximately 40% and the diesel component being less than 60%.

The high paraffin levels in both samples (DS5 and DS6) suggest that they cannot be classified as standard diesel quality; instead, they are indicative of contaminated, or adulterated diesel. The substantial presence of paraffin, which falls within the boiling point range of 150°C to 240°C, is problematic because it can lead to several adverse effects. The adulterated diesel may cause operational issues such as poor combustion, increased emissions, and potential damage to engine components [14]. Additionally, the decreased diesel fraction, falling within the 240°C to 370°C boiling point range, indicates inadequate diesel quality, which compromises the energy value per Liter and engine performance and efficiency [14].

TABLE VIII: PROPERTIES OF THE DIESEL MIXTURES AND THE n-ALKANE PEAK AREAS (C9-C13) WITH INTERNAL STANDARD RATIOS FOR THE DIESEL-PARAFFIN MIXTURES

Diesel: Paraffin	HHV (MJ/kg)	Flashpoint (°C)	Density (kg.m ³)	Area n-alkane	Area internal standard (IS)	Area n-alkane/(IS)
1:0.1	46.00	60.40	0.83	2.9E+08	7.2E+07	4.0
1:0.2	47.40	60.40	0.82	3.1E+08	6.6E+07	4.8
1:0.3	47.80	60.40	0.82	3.4E+08	6.3E+07	5.4
1:0.4	44.50	60.40	0.82	4.4E+08	6.7E+07	6.5
1:0.5	46.90	60.40	0.82	4.7E+08	6.4E+07	7.3
1:0.6	45.80	40.40	0.82	4.8E+08	6.0E+07	8.0
1:0.7	46.00	38.40	0.81	5.0E+08	6.0E+07	8.3
1:0.8	46.60	38.40	0.81	4.9E+08	5.5E+07	8.9
1:0.9	45.70	38.40	0.81	5.1E+08	5.5E+07	9.4
1:1	47.20	36.40	0.81	5.8E+08	5.8E+07	10.1

The HHV value of the mixtures does not change much and stays approximately at 45-47 MJ/kg. The flash point initially stays constant, with mixtures at and above a 1:0.6 ratio falling below the standard and at 1:1 ratio, the flash point drops to 36.4°C. The density stays almost constant with a slight drop at 1:0.6 paraffin-to-diesel ratio.

The correlation of the n-alkane peak areas, as presented in table 8, shows a clear and predictable increase in the n-alkane peak area. The n-alkane peaks considered, include C9 - C13, which provides a representative profile of the alkanes within the diesel-paraffin mixtures. By monitoring the combined peak areas of these specific n-alkanes, it is possible to trace the influence of the paraffin content on the fuel composition. The internal standard serves as a reference point, allowing the observed n-alkane areas to be evaluated relative to a stable baseline. This approach compensates for potential fluctuations in the instrument, ensuring that variations in peak areas reflect actual changes in sample composition rather than equipment variability. Figure 5 represents the linear relationship of the area of the n-alkane peak area to the internal standard between the diesel to paraffin ratios and thus suggests a reliable method for estimating the paraffin content in unknown diesel samples. By using a GC-MS to measure the area of the n-alkane / area IS of an unknown sample and applying the equation in the graph, it is possible to calculate the approximate ratio in terms of mass.

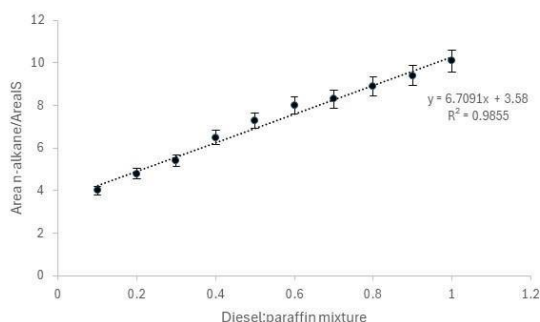


Fig. 5: Linear relationship between the n-alkane / internal standard area to the paraffin-to-diesel ratios plotted on the x-axis

D. Comparative analysis of biodiesel, commercial diesel and adulterated diesel

The densities of the standard commercial diesel samples range from 0.82 to 0.83 g/cm³, indicating consistency among them. Slightly lower densities for the adulterated diesel samples were observed, but still in acceptable ranges. In contrast, the densities of biodiesel produced with KOH and H₂SO₄, are notably higher, ranging from 0.89 to 0.93 g/cm³. Overall, the data shows that while biodiesel produced from different catalysts have higher densities than commercial diesel, the use of KOH as catalyst, results in the least dense biodiesel.

The flash points of the commercial diesel samples range from 70°C to 78°C, which fall within acceptable standards for diesel, suggesting the diesel samples are free from

contamination with paraffin, as lighter components are more abundant in paraffin compared to diesel. Excessively low flash points of about 36°C were observed for the adulterated diesel samples, which indicates the presence of more volatile components. Once the flashpoint falls below the SANS 342 minimum of 55°C, the fuel becomes more volatile and poses a higher risk of ignition in storage and use., where incremental paraffin additions lead to a direct reduction in flashpoint, potentially causing safety issues. In comparison, the flash points of the biodiesel produced with KOH and H₂SO₄ are significantly higher. For KOH, the flash points range from 166°C to 200°C and for H₂SO₄ from 180°C to 182°C. These high flash points indicate that the biodiesel samples are properly washed, with no significant residual methanol present as the methanol contamination would lower the flash point, which was not observed and resulted in biodiesel with good ignition safety. The biodiesel's flashpoint exceeds 100°C, surpassing that of both pure and contaminated diesel, which is required for safe handling and storage. Biodiesel, by contrast, shows stability in flashpoint values, suggesting that it has a safety advantage over contaminated diesel

The HHV of the diesel samples have a HHV range from 45 to 46 MJ/kg, whilst the biodiesel samples show values around 40 MJ/kg, regardless of the catalyst used and thus consistently lower than the commercial diesel samples. Biodiesel in principle has lower energy values compared to the petroleum diesel which can be attributed to the oxygen content in the biodiesel. Oxygen atoms do not contribute to the fuel's combustibility but still take up mass and volume. In contrast, paraffin contamination does not reduce the diesel HHV as the paraffin ratios increase. This difference in HHV suggests that biodiesel could provide more consistent energy output compared to diesel with varying paraffin-to-diesel ratios

IV. CONCLUSION

Biodiesel, adhering to the biodiesel standard was successfully produced with KOH as catalyst. Adulterated diesel on the other hand does not comply with the standards required for commercial diesel and the practice of adulteration should be stopped to ensure longer engine live of engines using diesel. A simple method of determining the paraffin content in adulterated diesel was developed to assist in identifying this malpractice.

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