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STUDY OF THE QUALITY OF DIESEL OIL FROM DAKAR STATIONS: DETERMINATION OF THE SULFUR CONTENT, WATER CONTENT AND VOLUMETRIC MASS

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ABSTRACT

At a time when environmental sciences and the concept of sustainable development are becoming important references in our societies, it appears necessary to have the means to fight against pollution. The pollution of hydrocarbons by sulphur represents an important risk of public health because of the various pathologies which can generate this element. In the framework of this study, we are interested in the study of the quality of diesel in Senegal by verifying the contents of sulfur and water as well as the volumetric mass.

The determination of the sulfur content is done with the EPSILON sulfur analyzer and the HORIBA sulfur analyzer. The water content and volumetry mass were also determined using the Karl Fischer apparatus and the DMA 4500 densimeter respectively.

Keywords: content, sulfur, water, energy, gasoil, station

1. INTRODUCTION

In this work, we determined the contents of sulfur and water of four stations of the most representative groups in terms of distribution of light petroleum products as well as the volumetric mass to see the energetic content of samples of diesel oil.

Since the 1950s, the petrol has become the main source of energy in the world and it satisfies more than 30% of the energy needs, but it contains impurities such as sulfur, metals and others.

The use of diesel fuel justifies the importance that desulfurization must occupy in the treatment of these types of fuels because they are rich in hydrogen sulfide (H_2S). Indeed, a high sulphur content causes sulphuric corrosion in vehicle equipment without hiding its environmental impact creating acid rain with SO_X and hydrogen sulfide. The sulfur content is the total amount of sulfur contained in the product. The European directive 98/70/EC regulates the composition of fuels delivered to the pump throughout the EU. The maximum permitted sulphur content is one of the parameters of this fuel composition. Gasoil may contain a maximum of 350 ppm (parts per million or mg per kg) [1-5].

The industrial development leading to a proliferation of diesel vehicles favors the use of diesel whose density remains an important parameter. The density is a parameter that allows us to appreciate the energy content of a fuel, in this case diesel. The volumetric mass [6-8] is the weight of the product for a given volume. It depends on the temperature of the product. At 15°C, it is on average 830 kg/m³ for

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diesel. The higher the density, the higher the energy content of the product. The energy content is the gross (primary) energy required for the life cycle of a product.

We also note the presence of water in petroleum products, particularly diesel. This accumulated water oxidizes the metal parts and is the cause of the proliferation of micro-organisms. Water sucked into engines can also cause breakdowns.

The water content [9] is the quantity of dissolved water contained in liquid products (gasoline, diesel,). Created by condensation, infiltration or runoff, water can stagnate at the bottom of the tank. It is therefore important to regularly check for the absence of water using a water detecting paste. Diesel fuel can contain 90 ppm of dissolved water when it is hot, but only 60 ppm when it cools down due to colder weather.

In this paper, the objective is to determine the sulfur content in diesel samples taken at four stations of the most representative groups in terms of distribution of light petroleum products in order to reduce upstream the sulfur content as hydrogen sulfide in diesel.

2. MATERIAL AND METHODS

II.1 Determination of sulfur content

II.1.1. Principle

A hydrocarbon sample [3, 4] is either injected directly or placed in a sample pod. Then the sample enters a high-temperature combustion tube (1000°C to 1100°C) where the sulfur is oxidized to sulfur dioxide (SO_2) in an oxygen-rich atmosphere. The water produced during the combustion of the sample is extracted and the combustion gases from the sample are exposed to ultraviolet (UV) radiation. Sulfur dioxide (SO_2) absorbs the energy released by the UV radiation and thus changes into the state of excited sulfur dioxide (SO_2*). The fluorescence emitted during the return to the fundamental state of the excited sulfur dioxide (SO_2*) is detected by a photomultiplier tube and the signal obtained represents a measure of the sulfur content present in the sample.

II.1.2. Choice of samples

We chose four gasoline stations in Dakar corresponding to the most representative groups in the distribution of diesel in Senegal to determine the sulfur content. These sampling points are confidential gasoline stations in Dakar:
Sample A from station A
Sample B from station B
Sample C from station C
Sample D from station D

These gas stations are the most appropriate sampling points because they are the most frequented by users. The environmental study is more interesting considering the large number of vehicles. The majority of vehicle users buy diesel or gasoline at these stations, particularly in Dakar. These gas stations belong to the largest fuel distribution groups in Senegal.

II.1.3. Reader at the EPSILON sulfur analyzer

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Image 1: Sulfur analyzer EPSILON

The container is composed of three elements (a large capsule, a small capsule and a lid), the filter and the glass support (sample scoop)

Procedure

- Shake the diesel sample to be analyzed before sampling
- Pour a quantity of sample into the container
- Avoid touching the bottom of the container
- Open the EPSILON machine and place the container
- Close the EPSILON machine
- Choose the parameter to be measured (sulphur content)
- Name the sample to be analyzed
- Check the repetition factor (in our case 5)
- Start the measurement by pressing the measurement button
- Each measurement lasts 187 seconds

II.1.4. Readings with the HORIBA sulfur analyzer

Image 2: HORIBA Sulfur Analyzer

The container is composed of the polyethylene terephthalate (PET) cell, the filter, the male ring and the female ring

Operating mode

- Shake the diesel sample to be analyzed
- Pour a quantity of sample in the polyethylene terephthalate (PET) cell
- Place the male ring so that it fits inside the cell
- Cover the female ring with a filter
- Insert the filter and the female ring into the male ring
- Fix the male ring
- Open the cover and place the device in the device
- Close the cover and press the button and wait for the display to read the value

II.2 . Determination of water content by the Karl Fischer method

The Karl Fischer method uses a coulometric titration to determine the amount of water in a sample. It can measure water concentrations in the order of milligrams per liter. It is used to evaluate the amount of water in substances such as butter, sugar, cheese, paper and oil.

The reaction involves the conversion of solid diiodine to hydrogen iodide in the presence of sulfur dioxide and water. Methanol is the most commonly used solvent, but the reaction also works in ethylene glycol and diethylene glycol. Pyridine is also used to prevent the accumulation of sulfuric acid, although the use of imidazole and diethanolamine for this purpose is becoming more common. All reagents must be anhydrous for the analysis to be quantitative. The balanced equation, using methanol and pyridine, is:

$$[C_5H_5NH]SO_3CH_3 + I_2 + H_2O + 2C_5H_5N \longrightarrow [C_5H_5NH]SO_4CH_3 + 2[C_5H_5NH]I$$

Scheme 1

Image 3: Galvanic cell

The Karl Fischer method is a chemical method for measuring the water content of a sample by titration. It was invented in 1935 by the German chemist Karl Fischer.

It is particularly suitable for the determination of water in a liquid or the detection of traces of water, in the ppm range, in a sample.

The procedure is based on the oxidation of sulfur dioxide by diiodine in the presence of water, originally observed by Robert Bunsen.

$SO_2 + I_2 + 2\ H_2O \leftrightarrow H_2SO_4 + 2\ HI$

Scheme 2

The galvanic cell in which the reaction takes place has three electrodes: two platinum and one for the detection of the end point of the assay.

Procedure

- Take a quantity of sample to be analyzed up to the 10 ml mark
- Inject the sample by graduation into the instrument
- Press the Sample data button
- Name the sample and display the drift
- Press the green arrow and the Continue button
- Wait for the display Condition
- Take a reading

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II.3. Determination of volumetric mass by the densimeter DMA 4500M

The sample is introduced into a U-shaped borosilicate glass tube which oscillates at its characteristic frequency which is directly related to the volumetric mass of the sample. After reaching a stable oscillation, the excitation is switched off and the oscillation fades freely. This sequence of excitation and fading is repeated continuously (patented pulsed excitation method). By evaluating this pattern, very accurate volumetric mass results [6, 7] are obtained, viscosity effects are compensated and air bubbles or particles are detected.

Image 3: Densimeter DMA 4500M

Operating mode

- Shake the diesel sample to be analyzed before sampling
- Rinse the tube and the syringe with the sample
- Pour a quantity of sample in the tube
- Take a quantity with the syringe
- Inject this quantity progressively into the DM 4500 M aspirator
- Inject the sample with the syringe into the aspirator
- Wait for the display
- Then collect the data on the screen

3. RESULTS AND DISCUSSION

III.1 Determination of the sulfur content

III.1.1. Sulfur reader with EPSILON analyzer

Table 1: Sulfur content of the four diesel samples by EPSILON

Appareil	GASOIL	Date	Measures	A in %	B in %	C in %	D in %
EPSILON	Sulfur content	2023/19/01	1	0,311	0,131	0,279	0,328
			2	0,311	0,131	0,279	0,327
			3	0,311	0,13	0,279	0,328
			4	0,311	0,131	0,278	0,327
			5	0,310	0,132	0,278	0,327
			Medium	**0,311**	**0,131**	**0,279**	**0,327**
			Deviation	0,0005	0,0007	0,0004	0,0003
			Norms in %	**0,3500**	**0,3500**	**0,3500**	**0,3500**

In Table 1 above, we report the results of the sulfur analyses of the four diesel samples taken on January 15, 2023 and stored at room temperature. The analyses were performed on January 19, 2023 using the EPSILON sulfur analyzer.

We took a repetition factor that is equal to five (05); meaning that the instrument will make five measurements and average them.

Table 2: Average sulphur content expressed in % of the four diesel samples

EPSILON	A in %	B in %	C in %	D in %
Medium	0,311	0,131	0,279	0,327
Norms in %	0,3500	0,3500	0,3500	0,3500

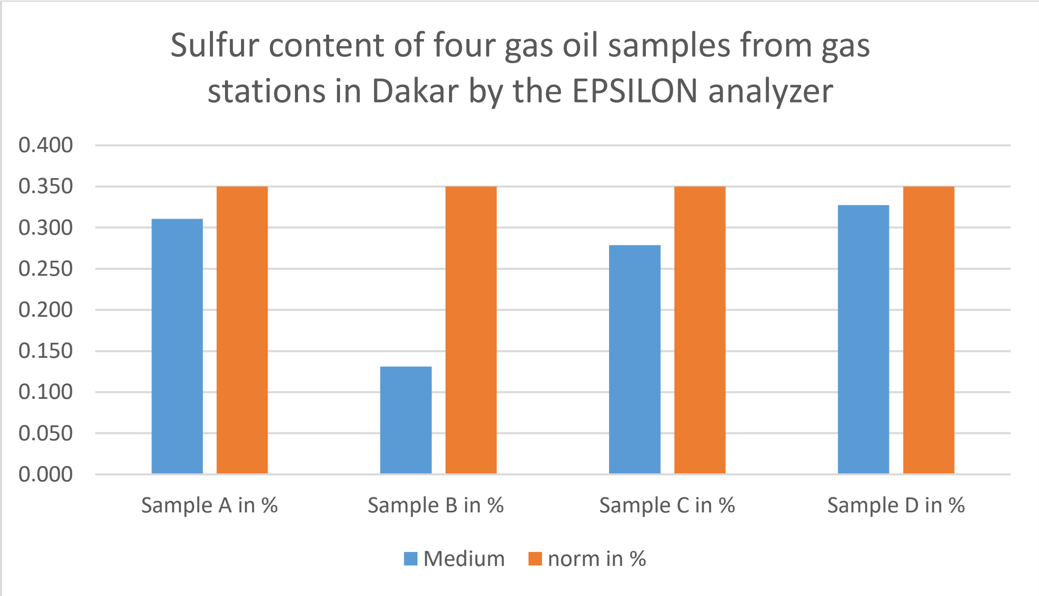

Figure 1: Average sulphur content expressed in % of the four diesel samples

This figure 1 shows that the sulphur content values of the four diesel oil samples B, C, A and D are lower than the standard set at 0.35%.

We note that sample B contains less sulphur with a content equal to 0.131%, i.e. about 2.67 times lower than the standard and presents the best quality. Samples C and A with sulfur contents of 0.279% and 0.311% respectively are lower than the standard of 0.35%.

Sample D has the highest sulphur content among the samples which is equal to 0.327% and the diesel contained in this sample is of lower quality compared to the other three samples.

III.1.2. Reading with the HORIBA sulfur analyzer

Table 3: Sulfur contents expressed in % of the four diesel samples by HORIBA

Appareil	GASOIL	Date	Measures	A in %	B in %	C in %	D in %
HORIBA	Sulfur	2023/19/01	1	0,3595	0,1332	0,2804	0,3405

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	content		2	0,3595	0,1332	0,2804	0,3405
			3	0,3595	0,1332	0,2804	0,3405
			Medium	**0,360**	**0,133**	**0,280**	**0,341**
			Norms in %	**0,3500**	**0,3500**	**0,3500**	**0,3500**

On the table above, we report the results of the analysis of the sulfur content of the four samples of diesel taken on January 15, 2023 and kept at room temperature and the analyses were made on January 19, 2023.

We have taken a repetition factor that is equal to three (03); this means that the apparatus will perform three measurements and make the average.

Table 4: Average sulphur content expressed in % of the four diesel samples

HORIBA	**A in %**	**B in %**	**C in %**	**D in %**
Measures	0,360	0,133	0,280	0,341
Norms in %	0,3500	0,3500	0,3500	0,3500

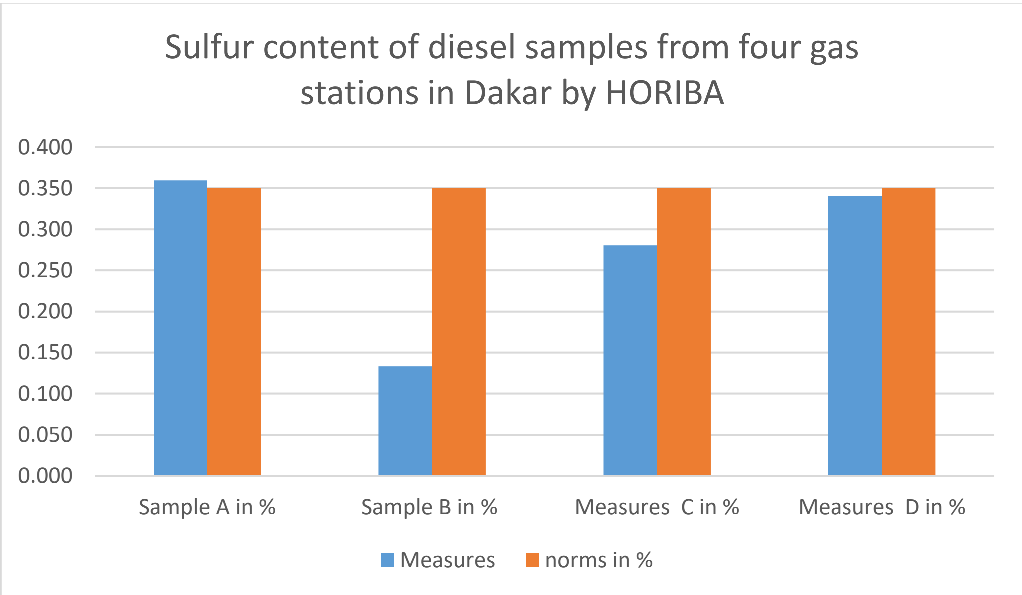

Figure 2: Average sulphur content expressed in % of the four diesel samples

This figure 2 shows that the sulphur content values of the three diesel samples B, C and D are lower than the standard set at 0.35%. Only diesel sample A has a sulfur content of 0.360% to slightly above the 0.35% standard.

Sample B contains less sulphur with a content equal to 0.133% or about 2.63 times lower than the standard and presents the best quality and is followed by sample C whose sulphur content equal to

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0.280% lower than 0.35%. Sample D has a sulfur content equal to 0.341% below the standard which is 0.35%.

III.2 Determination of water content

Table 5: Water content expressed in ppm of the four diesel samples

Appareil	GASOIL	Date	Measures	A in ppm	B in ppm	C in ppm	D in ppm
Karl Fischer	Sulfur content	2023/02/02	1	110	31	50	72
			2	70	14	52	9
			3	74	31	28	6
			4	105	13	43	15
			5	18	28	27	15
			6	18	27	39	28
			7	15	24	47	13
			8	12	28	38	6
			9	16	21	40	14
			Measures	**18**	**31**	**50**	**15**
			Limits in ppm	**60**	**60**	**60**	**60**

In the table above, we report the results of the water content analyses of the four diesel samples taken on January 15, 2023 and kept at room temperature. The analyses were carried out on February 02, 2023.

We performed nine one-milliliter injections of diesel fuel from each of the four samples in order to have a reliable measurement of water content. For reliability, we used the criteria of repeatability and reproducibility to select the water content measurement.

The repeatability noted r consists in verifying that the difference between two test results obtained by the same operator with the same equipment, under identical operating conditions and on the same product, by correctly and normally applying the test method, should not, in the long term, exceed more than one time in twenty the following value:

$$\mathbf{r = 0{,}01874 * X^{0{,}5}}$$

X is the average of the compared values of the test

- The reproducibility noted R consists in verifying that the difference between two single and independent test results, obtained by different operators working in different laboratories on identical products, by correctly and normally applying the test method, should not, in the long term, exceed more than one time out of twenty the following value :

$\mathbf{R = 0{,}06877 * X^{0{,}5}}$

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X is the average of the comparative values of the test

Table 6: Average water content expressed in ppm of the four diesel samples

Karl Fischer	A in ppm	B in ppm	C in ppm	D in ppm
Measures	18	31	50	15
Limits en ppm	60	60	60	60

The water content values of the four diesel samples are all below the 60 ppm limit.

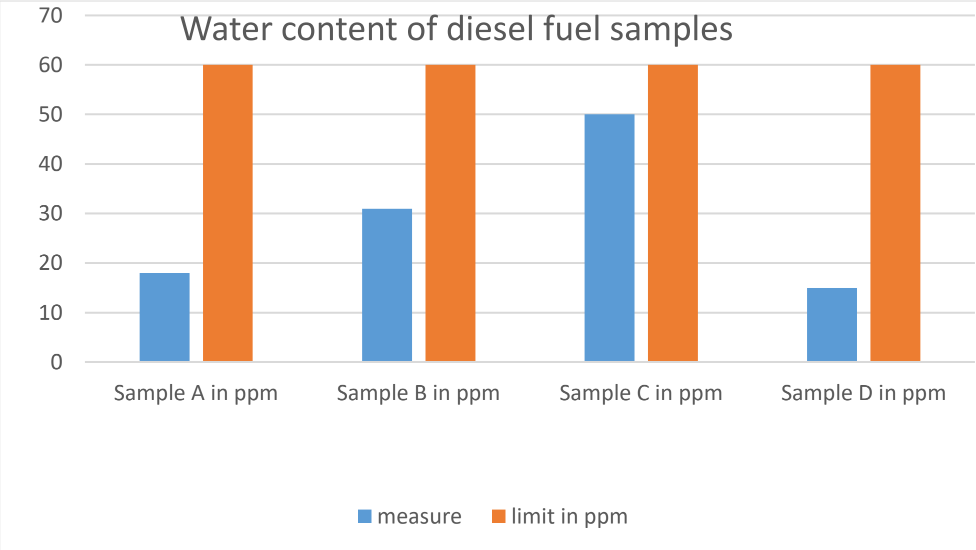

Figure 3: Average water content expressed in ppm of the four diesel samples

This figure 3 shows that the water content values of the four diesel samples A, B, C and D are lower than the threshold set at 60 ppm. Diesel oil sample D has the lowest water content, equal to 15 ppm, well below the threshold. Its content represents 25% of the threshold value. Sample A is in second position with a water content equal to 18 ppm, i.e. 30% of the threshold value. Sample B has a water content equal to 31 ppm below the threshold which is 60 ppm or about 51.67% of the threshold value. Sample C has a high water content equal to 50 ppm but below the threshold which is 60 ppm or about 83.33% of the threshold value.

The gasoline station D presents the best quality of diesel with a water content of 15 ppm very low compared to the threshold which is 60 ppm. It is followed by gas station A with a water content of 18 ppm representing 30% of the threshold value of 60 ppm.

To avoid contamination of the fuel system, clogging of the fuel filter and running out of fuel with a full tank, it is advisable to buy diesel from gas stations D and A with low water content.

III.3 Determination of the volumetric mass

Table 7: Volumetric mass by the DMA 4500 M densimeter

Parameters	A	B	C	D
Volumetric mass kg/m³ à 15 °C	844,66	854,52	855,12	843,24

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Density	0,84113	0,85100	0,85161	0,83972
Volumetric mass g/mL à 20 °C	**0,84141**	**0,85098**	**0,85159**	**0,83969**
Norm kg/m³ à 15 °C	**830**	**830**	**830**	**830**

In Table 7 above, we report the results of the volumetric mass measurements of the four diesel oil samples taken on January 15, 2023 and stored at room temperature. The measurements were made on January 24, 2023 using the DMA 4500 M density meter. We took a repetition factor that is equal to three (05); this means that the device will make five measurements and average them.

Table 8: Volumetric mass of the four samples in diesel

Parameters	**A**	**B**	**C**	**D**
Volumetric mass kg/m³ à 15 °C	**844,66**	**854,52**	**855,12**	**843,24**
Norm kg/m³ à 15 °C	**830**	**830**	**830**	**830**

All four samples have volumetric mass values above the standard set at 830 kg/m³ at 15 °C [12] [16]. which means that the contents of all four samples are significant.

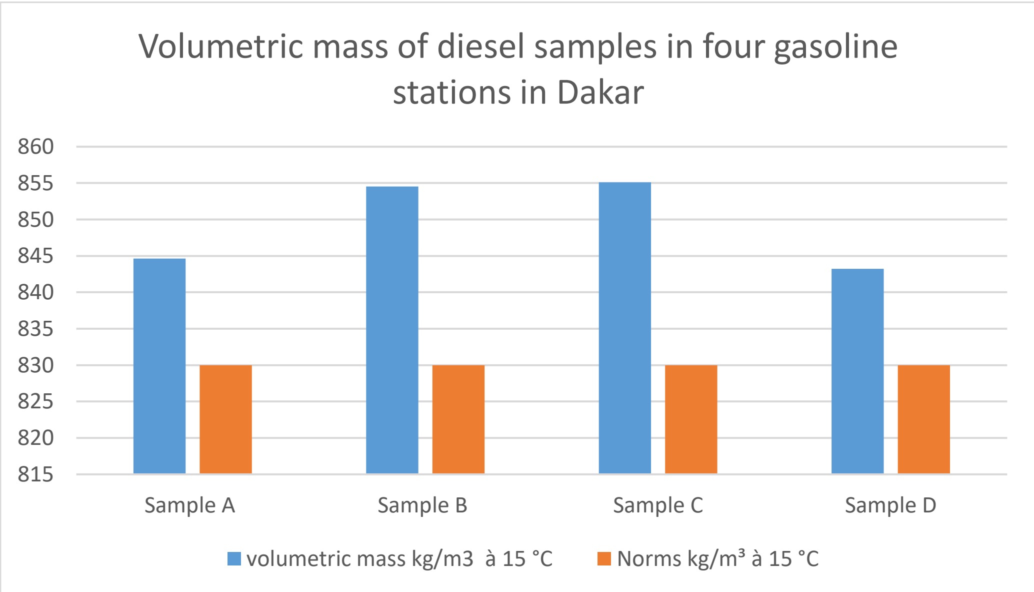

Figure 4: volumetric mass of the four diesel samples

This figure 1 shows that the values of the densities of the four diesel oil samples A, B, C and D are higher than the standard set at 830 kg/m³ at 15 °C [10] [11] [12]. Station C has the gas oil with the highest volumetric mass of 855.12 kg/m³ at 15 °C. Its gas oil has the highest energy content. It is followed by station B with a volumetric mass of 854.52 kg/m³ and then A and C with volumetric mass of 844.66 kg/m³ at 15 °C and 843.24 kg/m³ at 15 °C respectively.

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4. CONCLUSION

Station B has the best quality of diesel followed by station C with very low sulfur content compared to the standard. The density results also show that for a long autonomy of the motorized system, it is best to buy the gas oil in the gas stations C and B whose gas oils have the highest energy contents.
The diesel samples from gas stations A and D are of lower quality with sulfur contents of 0.311/ 0.327% by EPSILON and 0.360/ 0.341% by HORIBA.

The most sensible choice is to refuel at gas stations B and C in order to preserve the condition of the various vehicle equipment and to mitigate SOx pollution.

The results also showed that the water content values of the four diesel samples A, B, C and D are below the threshold of 60 ppm.

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