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Total Sulfur Levels in Refined Petroleum Products of Southwestern Nigeria Using UV/VIS Spectrophotometer

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This study employed turbidimetric method using a UV/VIS spectrophotometer to determine the total sulfur levels of refined petroleum products in Southwestern Nigeria. The sulfur concentrations in the analyzed fuel samples were 0.0019–0.0178 wt% and 0.027–0.169 wt% for gasoline and diesel, respectively. Sulfur levels in the selected samples were below the Nigerian standard for gasoline and diesel while 89.9% of the samples exceeded the U.S. Environmental Protection Agency standard. The turbidimetric method showed better performance than the gravimetric method. The results gave insight into the adverse environmental impact the nonuniform quality of fuels imported could pose to the country.

Keywords: sulfur, gasoline, diesel, turbidimetric method, UV/VIS spectrophotometer

1. INTRODUCTION

Inadequate local petroleum refining capacities in Nigeria has resulted in importations of refined petroleum products with nonuniform quality. Some of these products are suspected to contain high sulfur levels despite the regulatory limits, thus resulting in the emissions of sulfur dioxide, which contribute to decreased air quality and have negative environmental and serious health effects. Sulfur and its compounds are present in virtually all petroleum products and lubricants from crude oils to the ultra-low sulfur fuels of the future. The sulfur levels in these products vary from the highest amounts in the crude oils and some lubricants to trace levels in the currently proposed road fuel. Sulfur determination is a primary step for the accurate prediction of sulfur dioxide forming potentials (Alam et al., 2012). Sulfur oxides formed during the combustion of fuels in internal combustion engines are undesirable because of the damage they do the engines and to the environment (Sonibare and Jimoda, 2009). Acids of sulfur oxides increase rusting and corrosion of engine parts, piston rings, and cylinder walls. In the atmosphere, sulfur oxides convert to sulfuric acid by reaction with moisture, and harm vegetation as well as aquatic, animal, and human life. Because of the deleterious effects of sulfur emissions from gasoline and diesel powered engines, a number of government regulatory agencies, principally in Africa, North America, Asia, and Europe, have been vigorously controlling and gradually reducing the sulfur content of the fuel used in automobiles, aviation, marine vessels, off-road vehicles, power generating utilities, and home heating (Nadkarni, 2004).

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The permitted sulfur levels are currently 0.003–0.008 wt% for gasoline and 0.05 wt% for diesel (U.S. Environmental Protection Agency, 2013).

There are numerous methods available for the determination of sulfur in diverse petroleum products. The most popular methods that are currently used for performing total sulfur analysis include energy-dispersive X-ray fluorescence (EDXRF), wavelength-dispersive X-ray fluorescence (WDXRF), UV fluorescence (Nadkarni, 2000), and sulfur chemiluminescence detection (SCD; Shearer et al., 1993). Gas chromatography (GC) coupled with an atomic emission detector (AED) is popular for the separation and quantitation of individual sulfur compounds because of its element-selective response, and also because of its low limit of detection and structure-independent elemental response (Quimby et al., 1992). One drawback to using GC–AED for the quantitative speciated sulfur analysis of petrochemical samples is that the length of time required to obtain an adequate separation of the components of interest is on the order of 30–50 min per sample (Link et al., 2002). Atar et al. (1991) reported a new generation of the flame photometric detector for total sulfur analysis called pulsed flame photometric detector (PFPD). In addition, gravimetric method of determining total sulfur content is a wet chemical method reported by Tijjani et al. (2012) for the determination of total sulfur of petroleum product in Nigeria. However, total sulfur determinations by turbidimetric method using a UV/VIS spectrophotometer is more efficient when compared with the typical analysis time of about 10–12 h of measurement, cost implication and low accuracy associated with gravimetric method. Online measurement is also another preferred choice of determining total sulfur content of fuel rather than waiting for the off-line laboratory analysis.

Many proposed online methods are extensions of the established laboratory test methods such as test method for sulfur in petroleum products by hydrogenolysis, rateometric colorimetry, and EDXRF spectrometry (Nadkarni, 2004). Other methods of determining total sulfur are Horiba sulfur-in-oil analyzer (Ibrahim et al., 2012) and general bomb method (Rand, 2003). This study describes a turbidimetric method of determining total levels of sulfur in fuels using a UV/VIS spectrophotometer. This instrument provides for the highest accuracy of measurement coupled with a very high level of stability with time. The operational principle has been described in earlier studies (Ademoroti, 1996; Skoog et al., 2007). Southwestern Nigeria receives the highest percentage of 41% out of the total volume of refined petroleum products distributed across the country annually (Nigerian National Petroleum Corporation, 2011). The impact of the nonuniformity in the quality of petroleum products consumed in the country is expected to be significant in this region.

2. EXPERIMENTAL

2.1 Sample Collection and Storage

Gasoline and diesel fuel samples were collected directly from the nozzle of the bowser hose into 36 plastic bottles at three different filling stations from each of the six states in southwestern Nigeria and stored in the laboratory at room temperature before analysis. All fuel samples collected were given identity in alphanumeric character for easy identification in Table 1. The average density of all the gasoline samples collected for this study is 0.739 kg/L while the average density of all the diesel samples collected is 0.844 kg/L.

2.2 Analytical Methods

The sulfur level of the samples was determined using the turbidimetric method, which is based on the formation of barium sulfate in the colloidal form in the presence of acidified barium chloride. The absorbance of the colloidal solution was measured at wavelength of 425 nm on a UV/VIS

TABLE 1
Sample Identification

State	*Gasoline Sample*			*Diesel Sample*		
Osun	A_{11}	A_{12}	A_{13}	A_{21}	A_{22}	A_{23}
Oyo	B_{11}	B_{12}	B_{13}	B_{21}	B_{22}	B_{23}
Ogun	C_{11}	C_{12}	C_{13}	C_{21}	C_{22}	C_{23}
Lagos	D_{11}	D_{12}	D_{13}	D_{21}	D_{22}	D_{23}
Ekiti	E_{11}	E_{12}	E_{13}	E_{21}	E_{22}	E_{23}
Ondo	F_{11}	F_{12}	F_{13}	F_{21}	F_{22}	F_{23}

Gasoline = 1; Diesel = 2; Position of filling Station = 1, 2, or 3. A = Osun; State B = Oyo State; C = Ogun State; D = Lagos State; E = Ekiti State; F = Ondo State.

spectrophotometer. In order to evaluate the accuracy of the UV/VIS spectrophotometer for rapid determinations of total sulfur content, a wide variety of fuel samples were analyzed. Analysis of fuel samples was performed on Model CECIL CE 7400 Double Beam UV/VIS spectrophotometer. The spectrophotometer is a double beam optical system with a powerful integral control having wavelength range of 190–1000 nm and a 2 nm bandwidth. The fully symmetrical double beam system of the Aquarius instruments provides the highest accuracy of measurement coupled with very high stability with time. It is built for high-resolution applications with stability of ±0.0001A/H, scan rate of 1–4000 nm/min, wavelength precision of 0.1 nm, and stray light less than 0.02%.

In preparing the blank for sulfur standard calibration curve, 100 mL distilled water was measured into a 250 mL Erlenmeyer flask. A 5 mL conditioning reagent was added to the solution in the flask and mixed on the magnetic stirrer with the timing started immediately. While stirring the solution, a spoonful of barium chloride crystal was dissolved in 2 mL distilled water and added at the fourth minute. The stirring of the solution was done at a constant speed and timing stopped at the fifth minute. Some of the solution was poured into the cuvette and the absorbance was read at 425 nm in the spectrophotometer at the seventh minute. Standard solutions were prepared at 5 mg/L increments of 0–40 mg/L and at 100 mg/L increments of 0–1000 mg/L sulfate range using anhydrous Na_2SO_4 for gasoline and diesel calibration plots, respectively. Then 100 mL of each of the solutions was measured into a 250 mL Erlenmeyer flask. A 5 mL conditioning reagent was added to the solution in the flask and mixed on the magnetic stirrer with the timing started immediately. While stirring the solution, a spoonful of barium chloride crystal was dissolved in 2 mL distilled water and added at the fourth minute. The stirring of the solution was done at a constant speed and timing stopped at the fifth minute. Some of the solution was poured into the cuvette and the absorbance was read at 425 nm in the spectrophotometer at the seventh minute. The readings for all the various sulfate concentrations were taken and absorbance—a concentration graph was plotted.

In preparing the blank for sulfur in gasoline analysis, 100 mL iso-octane was measured into a 250 mL Erlenmeyer flask. 5 mL conditioning reagent was added to the solution in the flask and mixed on the magnetic stirrer with the timing started immediately. While stirring the solution, a spoonful of barium chloride crystal was dissolved in 2 mL distilled water and added at the fourth minute. The stirring of the solution was done at a constant speed and timing stopped at the fifth minute. Some of the solution was poured into the cuvette and the absorbance was read at 425 nm in the spectrophotometer at the seventh minute. To prepare the blank for sulfur in diesel analysis, 100 mL heavy distillate for petroleum testing was measured into a 250 mL Erlenmeyer flask. To this solution was added 5 mL conditioning reagent in the flask and mixed on the magnetic stirrer with the timing started immediately. While stirring the solution, a spoonful of barium chloride crystal was dissolved in 2 mL distilled water and added at the fourth minute. The stirring of the solution was done at a constant speed and timing stopped at the fifth minute. Some of the solution was poured into the cuvette and the absorbance was read at 425 nm in the spectrophotometer at the seventh minute.

In fuel sample analysis, 100 mL fuel sample was measured into a 250 mL Erlenmeyer flask with 5 mL conditioning reagent added to the solution in the flask and mixed on the magnetic stirrer. The timing started immediately and while stirring the solution, a spoonful of barium chloride crystal was dissolved in 2 mL distilled water and added at the fourth minute. The stirring of the solution was done at a constant speed and timing stopped at the fifth minute. Some of the solution was poured into the cuvette and the absorbance was read at 425 nm in the spectrophotometer at the seventh minute. The absorbance was measured and the corresponding sulfate concentration was determined by extrapolation from the calibration data. The blank was used to adjust the instrument to zero between each sample measurement. The sulfate and sulfur concentrations were calculated using Eqs. (1) and (2).

$$SO_4^{2-}\left(\frac{mg}{l}\right) = \frac{mass\ SO_4^{2-} calculated\ from\ the\ curve\ equation\left(\frac{mg}{l}\right)}{sample\,(ml)} \times 1000\,(ml) \tag{1}$$

The mass of $SO_4{}^{2-}$ for a sample is calculated by extrapolation from standard calibration plot using the corresponding absorbance of the sample.

$$S\left(\frac{mg}{l}\right) = \frac{molar\ mass\ of\ sulphur\,(S)}{molar\ mass\ of\ sulphate\,\left(SO_4^{2-}\right)} \times SO_4^{2-}\left(\frac{mg}{l}\right) \tag{2}$$

3. RESULTS AND DISCUSSION

Figure 1 shows a standard calibration plot obtained from prepared standard solution for sulfate concentration in gasoline. The calibration showed that the fit gave a good measure of regression with an R^2 value of .9965. The validity of the plot was verified with the closeness of this value to unity. All the absorbance measurements for gasoline samples obtained were within the range of the standard sulfate solutions prepared. The sulfate concentrations in gasoline samples were therefore

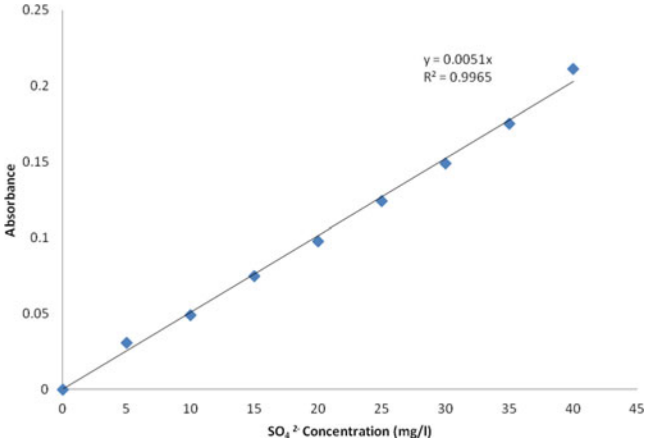

FIGURE 1 Calibration plot for sulfate in gasoline.

TABLE 2
Reproducibility Check of Absorbances of Sulfate Concentration in Gasoline and Diesel

Gasoline Sample	*Average Absorbance*	*%RSD*	*Diesel Sample*	*Average Absorbance*	*%RSD*
A_{11}	0.085 ± 0.0057	6.70	A_{21}	1.372 ± 0.0866	6.31
A_{12}	0.099 ± 0.0905	9.62	A_{22}	1.056 ± 0.0019	0.18
A_{13}	0.027 ± 0.0016	6.10	A_{23}	1.455 ± 0.0164	1.13
B_{11}	0.026 ± 0.0015	5.79	B_{21}	0.580 ± 0.0254	4.38
B_{12}	0.207 ± 0.0020	0.97	B_{22}	1.826 ± 0.0062	0.34
B_{13}	0.206 ± 0.0016	0.79	B_{23}	0.963 ± 0.0077	0.80
C_{11}	0.090 ± 0.0056	6.18	C_{21}	0.431 ± 0.0072	1.67
C_{12}	0.093 ± 0.0025	2.80	C_{22}	1.832 ± 0.0146	0.80
C_{13}	0.102 ± 0.0051	5.00	C_{23}	1.344 ± 0.0040	0.30
D_{11}	0.177 ± 0.0038	2.38	D_{21}	1.283 ± 0.0400	4.22
D_{12}	0.101 ± 0.0031	3.07	D_{22}	1.215 ± 0.0312	2.57
D_{13}	0.203 ± 0.0048	2.38	D_{23}	1.326 ± 0.0108	0.81
E_{11}	0.207 ± 0.0031	1.49	E_{21}	1.834 ± 0.0044	0.23
E_{12}	0.076 ± 0.0037	4.86	E_{22}	0.969 ± 0.0409	4.22
E_{13}	0.178 ± 0.0042	2.33	E_{23}	0.978 ± 0.0029	0.29
F_{11}	0.045 ± 0.0038	8.26	F_{21}	1.593 ± 0.0216	1.36
F_{12}	0.130 ± 0.0027	2.11	F_{22}	1.518 ± 0.0091	0.60
F_{13}	0.085 ± 0.0048	5.71	F_{23}	0.387 ± 0.0074	1.91

%RSD = percentage relative standard deviation.

calculated by extrapolation using the corresponding average absorbances (Table 2) and the standard calibration curve (Figure 1).

The calibration plot for the concentration of sulfate in diesel shown in Figure 2 was obtained from the standard sulfate solution prepared. The calibration plot showed that the fit gave a good measure of regression with an R^2 value of .9935. The validity of the plot was verified with the closeness of this value to unity. All the absorbance measurements for diesel samples obtained were within the range of the standard sulfate solutions prepared. The sulfate concentrations in diesel samples were therefore calculated by extrapolation using the corresponding average absorbances in Table 3 and the standard calibration plot (Figure 2).

The analytical precision test results for the various absorbances of sulfate concentration in gasoline and diesel samples are presented in Table 2. The coefficient of variation in terms of standard deviations for the absorbances of sulfate concentration in gasoline and diesel samples indicates a minimal or negligible deviation from standards. This validates UV/VIS spectrophotometer as a good instrument for accurate determination of sulfur in fuels.

The fuel samples analyzed contained a wide variety of sulfur concentrations ranging from 0.0019–0.0178 wt% for gasoline and 0.027–0.169 wt% for diesel. In Table 3, the sulfur concentrations of all the gasoline samples tested are presented. The obtained percentage by weight of total sulfur level in all the gasoline samples analyzed were observed to fall below the range of the reported sulfur level of 0.05–0.30 wt% in Nigerian refined petroleum products as established by past studies (Farroha et al., 1984; Adegbite, 1989; Oderinde, 1989; Tijjani et al., 2012) and the sulfur level limit of 0.1 wt% for gasoline recommended by the Department of Petroleum Resources Nigeria (2010). However, it was also observed that sulfur level in 89.9% of gasoline samples exceeded the 0.003–0.008 wt% (30–80 ppm) limit specified by the U.S. Environmental Protection Agency (2013).

The obtained percentage by weight of total sulfur level in all the diesel samples analyzed was observed to fall below the range of the reported sulfur level of 0.05–0.30 wt% in Nigerian refined

TABLE 3
Concentrations of Sulfur in Gasoline and Diesel

Gasoline Sample	*Conc., mg/L*	*Conc., ppm*	*Conc.,%wt*	*Diesel Sample*	*Conc., mg/L*	*Conc., ppm*	*Conc.,%wt*
A_{11}	57.25	77.46	0.0077	A_{21}	901.21	1067.8	0.107
A_{12}	67.31	91.08	0.0091	A_{22}	694.87	823.31	0.082
A_{13}	14.52	19.64	0.0020	A_{23}	955.40	1131.9	0.113
B_{11}	13.98	18.92	0.0019	B_{21}	346.64	410.72	0.041
B_{12}	131.5	177.9	0.0178	B_{22}	1303.3	1544.2	0.154
B_{13}	131.1	177.3	0.0177	B_{23}	629.84	746.26	0.075
C_{11}	60.87	82.37	0.0082	C_{21}	255.64	302.89	0.030
C_{12}	63.04	85.31	0.0085	C_{22}	1309.3	1551.3	0.155
C_{13}	69.23	93.68	0.0094	C_{23}	882.93	1046.1	0.105
D_{11}	117.6	159.1	0.0159	D_{21}	843.10	998.93	0.099
D_{12}	68.59	92.81	0.0093	D_{22}	798.69	946.32	0.095
D_{13}	129.6	175.4	0.0175	D_{23}	871.17	1032.2	0.103
E_{11}	131.5	177.9	0.0178	E_{21}	1430.0	1694.3	0.169
E_{12}	50.72	68.59	0.0069	E_{22}	634.28	751.52	0.075
E_{13}	118.1	159.8	0.0160	E_{23}	640.94	759.40	0.076
F_{11}	29.63	40.09	0.0040	F_{21}	1069.8	1267.6	0.127
F_{12}	87.33	118.2	0.0118	F_{22}	996.54	1180.7	0.118
F_{13}	57.25	77.46	0.0077	F_{23}	229.54	271.96	0.027

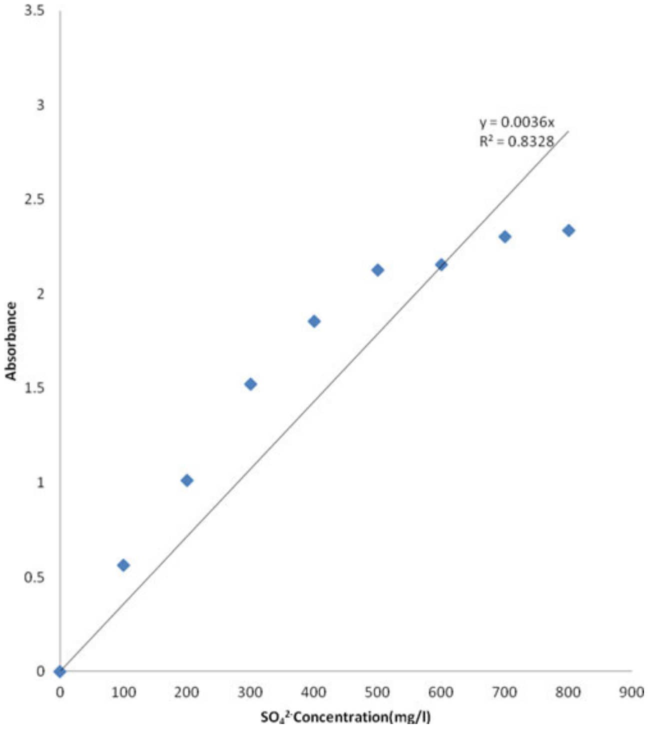

FIGURE 2 Calibration plot for sulfate in diesel.

petroleum products as established by earlier studies (Farroha et al., 1984; Adegbite, 1989; Oderinde, 1989; Tijjani et al., 2012) and the sulfur level limit of 0.3wt% in diesel recommended by DPR. It was also observed that sulfur levels in 94.44% of diesel samples were above 0.05 wt% (500 ppm) limit specified by the U.S. Environmental Protection Agency. The nonuniformity in the obtained sulfur levels of petroleum products collected from indigenous and multinational filling stations could be as a result of storage of petroleum products from various sources over a long period of time, fuel adulteration and use of contaminated tankers.

4. CONCLUSIONS

The UV/VIS spectrophotometer has demonstrated good performance for low-level and high-level sulfur determination compared with gravimetric method. Using the UV/VIS spectrophotometer reduces analysis times, minimizes integration differences, and yields more accurate results, especially for complex mixtures that contain many different sulfur-containing compounds at trace levels It is recommended that low or near-zero sulfur fuel must be enforced by the Nigerian regulatory body to ensure total compliance to the sulfur limit recommended by International regulatory body. The turbidimetric method has established that the UV/VIS spectrophotometer can be used for rapid determinations of total sulfur in comparison with the time scale of other methods, such as WRXRF and GC–AED. The reproducibility check carried out in the form of standard deviations showed a minimal or negligible deviation of the absorbances of sulfate concentration in gasoline and diesel samples from standards. This validates the UV/VIS spectrophotometer as a good instrument for accurate determination of sulfur in fuels.

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