ZIBELINE INTERNATIONAL™ PUBLISHING
ISSN: 2576-6724 (Print)
ISSN: 2576-6732 (Online)
CODEN: ACMCCG

Acta Chemica Malaysia (ACMY)

DOI: http://doi.org/10.26480/acmy.02.2024.114.119

RESEARCH ARTICLE

COMPARISON OF WET CHEMISTRY AND DRY COMBUSTION METHODS FOR ORGANIC CARBON AND SULFUR DETERMINATION IN SOILS OF NIGERIA'S SAVANNA

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ARTICLE DETAILS

Article History:

Received 10 October 2024
Revised 13 December 2024
Accepted 29 December 2024
Available online 18 January 2025

ABSTRACT

The study of physicochemical properties of soil is essential for evaluating nutrient status and other characteristics that regulate plant growth. Soil scientists in Nigeria commonly use wet chemistry and dry combustion methods to determine the organic carbon and sulfur content of soil. However, there is insufficient information on the efficiency and limitations of these methods for academic purposes. A laboratory experiment was conducted in 2019, using 60 topsoil samples from the Sudan savanna and northern Guinea savanna agroecology of Nigeria, to compare the results of wet and dry methods for determining organic carbon and sulfur content. The results showed that the CHNS/O analyzer provided higher accuracy and mean values for organic carbon determination, while wet chemistry revealed higher mean and R2 values for sulfur determination. The use of the CHNS/O analyzer is recommended due to its accuracy and safety. Future studies are needed to further evaluate the relationship between these two methods.

KEYWORDS

Organic carbon, sulfur, wet chemistry, dry combustion

1. INTRODUCTION

Several research on soil physical and chemical examinations across multiple fields have been undertaken to identify the textural classes, nutrient status, distribution, and fluctuation at different sites to assess their impact on crop yield in Nigeria's savannah agro-ecologies (Mustapha et al., 2021). Most soil investigations employ wet chemistry and dry combustion to determine soil chemical parameters such as organic carbon and sulfur concentration.

Soil and sediments may contain three fundamental types of carbon. These included (1) elemental carbon, (2) inorganic carbon, and (3) organic carbon (OC). According to Brian in 2002, the quality of organic matter in soil and sediments is crucial for the partitioning and bioavailability of pollutants associated with soil and sedimentation. However, naturally occurring organic carbon (OC) sources include plant and animal biodegradation (Paul, 2014). In nutrient management research, determining soil (SOC) to analyze the C:N balance is especially important because it directly or indirectly affects the mineralization of complex organic matter to release N and anions such as SO_3^-, SO_4^-, and CO_3^-, which is influenced by the availability of soil microbes such as bacteria, fungi, blue-green algae, and actinomycetes (Sinha, 1991). Anthropogenic activities also contribute to SOC pollution. Spills or releases of pollutants from both point and non-point sources into the environment raise the total carbon content of the soil or sediment (Angelova et al., 2013; Brian, 2002).

Three methods for soil SOC determination are available in the literature: (i) wet oxidation (also known as wet combustion), which consists of partial oxidation associated with a more labile soil SOC pool, addition of diluted potassium dichromate, and concentrated sulfuric acid with back titration using ferrous ammonium sulfate; (ii) wet oxidation and carbon dioxide (CO2) collection and determination in a trapping solution; and (iii) dry oxidation, popularly known as dry The dry combustion (DC) process oxidizes soil organic carbon at high temperatures while reducing combustion gasses to CO_2 at low temperatures. Researchers advocated total soil OC oxidation with external heating of soil at 150oC under reflux for only 30 minutes (Nelson and Sommers, 1982). The accurate and quick detection of sulfur is critical in soil and plant research, as well as agricultural and environmental monitoring programs (Tabatabai, 1982).

Several approaches have been developed for determining total sulfur in soil and plants (Schimel and Schaeffer, 2012). Tabatabai, examined a variety of techniques, including gravimetry, turbidimetry, and nephelometry, titrimetry, colorimetry, inductively coupled plasma atomic emission spectroscopy (ICP-AES), and dry combustion chromatography (Tabatabai, 1982).

Researchers frequently utilize their preferred method without first assessing the inherent limits of such a strategy. However, a thorough comparison of wet chemistry and dry combustion methods is required to gain a proper understanding of the principles underlying their mode of operation, result accuracy, advantages, and limitations, which will guide their applicability in a variety of situations in Nigerian savanna soil analysis. The purpose of this study was to evaluate wet and dry techniques of determining organic carbon and sulfur in Nigerian savanna soils. This endeavor will give a foundation for making educated judgments about the appropriate application of either approach.

2. MATERIALS AND METHODS

2.1 Experimental site

The experiment was conducted at the Soil Science Laboratory of the

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Cite the Article: Habib, D. W, Idris F. B, Mustapha A. A. and Abdurrahman B. L (2024). Comparison Of Wet Chemistry and Dry Combustion Methods for Organic Carbon And Sulfur Determination in Soils of Nigeria's Savanna. *Acta Chemica Malaysia, 8(2): 114-119.*

Department of Soil Science, Bayero University Kano (BUK) in 2019. BUK is located within latitude (11°58'N), longitude, and (826E) meters above sea level. Composite soil samples were collected from farmers' fields across the Sudan Savanna (SS) and Northern Guinea Savanna (NGS) agro-ecology.

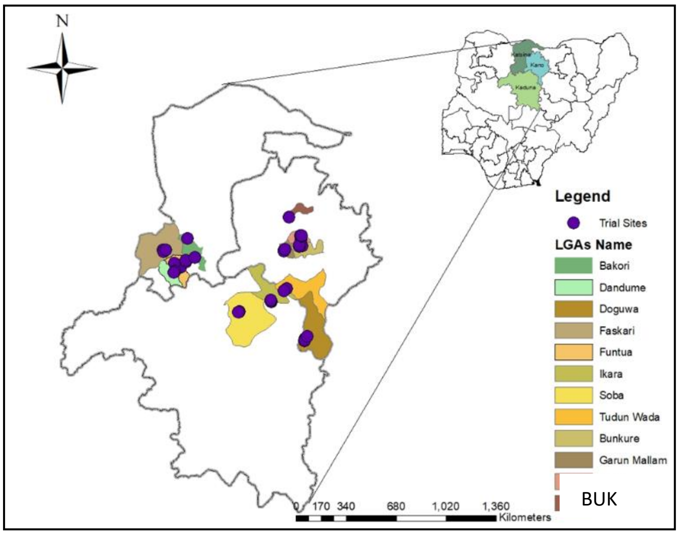

Figure 1: Map showing the study areas where the composite soil samples were collected.

2.2 Data collection

2.2.1 Soil sampling technique

Sixty (60) composite soil samples were collected from a depth of 0 to 20 cm. The samples were strategically gathered from five (5) points in each farmer's field, arranged in a "W" shape sampling framework. The five (5) samples were then combined to form a single composite sample, which was air-dried, crushed, and filtered through a 2-mm screen for laboratory examination. Some soil samples were brought to the laboratory to test particular parameters such as organic carbon and sulfur using the wet technique, while others were transferred to the CDA laboratory to test the same parameters (OC and S) using the dry method.

2.2.2 Soil sample preparation

The composite samples collected were air-dried, crushed, and sieved using a 2-mm sieve and taken to the Soil Science Laboratory and CDA Laboratory at BUK for organic carbon (OC) and sulfur(S) content analyses.

2.3 Experimental methods and materials

Two experimental methods were used in this study. These are wet chemistry and dry combustion methods. For wet chemistry, a modified Walkley-Black method for OC and a turbidimetric method for sulfate content determination were used, and secondary data from previously conducted analyses using a CHNS/O analyzer (CHNS/O 2400 Series II Perkin Elmer Inc., Waltham, MA, USA) were used for the dry combustion method to compare the results of both OC and sulfur content in the sample.

2.3.1 Wet method for organic carbon determination

Materials: One gram of soil sample, a conical flask measuring (500 ml),beaker, weighing balance, 1–10 phenonthrolein (indicator), concentrated tetraoxosulfate six acid (H_2SO_4), distilled water, a burette (500 ml), 1N $K_2Cr_2O_7$ and 0.5N $FeSO_4$.

2.3.2 Wet method for sulfate determination

Materials: Five grams of soil, volumetric flask (25 ml), plastic bottles, spectrometer, beaker, measuring cylinder, KH_2PO_4 (500ppm), centrifuge machine, Geltin Bacl, distilled water, and one mechanical shaker.

2.4 Laboratory procedure

2.4.1 Organic carbon determination using the wet method (Walkley-Black)

1 g of the prepared soil sample was weighed and placed in a conical flask, followed by the addition of 5 ml of $K_2Cr_2O_7$ and then swirled gently to allow for thorough formation of the mixtures. This was accompanied by the addition of 10 ml of H_2SO_4 acid and swirled gently, and the reagents were allowed to settle for 30 min. Then, 100 ml of distilled water and 3-4 drops of phenonthrolein indicator were added, followed by back-titration with 0.5 N FeSO4. The following formula was used to calculate the percentage (%) of organic carbon:

$$\frac{(B-T)\times N\times 0.003\times 1.33}{sampleweight\ (g)}\times\ 100$$

Where,

B = blank

T = titer value

N = normality

Constant = 0.003

Correction factor =1.33

2.4.2 Wet method for sulfate determination (Turbidimetric)

25 ml of KH_2PO_4 solution was added to 5 g of soil sample, shaken thoroughly for 30 min, and centrifuged for 15 min at 2000 rpm. A total of 10 ml was sampled from the mixture, and 10 ml of distilled water was added, followed by the addition of 1 ml of Geltin Bacl. A substantial proportion of the sample was inserted into a spectrophotometer at a wavelength of 400 nm (wavelength), and the readings were recorded.

2.4.3 CHNS/O Determination of organic carbon and sulfur using the dry method

In this set of experiments, a CHNS/O elemental analyzer was used to determine the organic carbon and sulfur contents. Although CHNS/O was desired to simultaneously determine the amounts of carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O) present in the sample, only OC and S were used in this study. The data for OC and S were already obtained in another study; therefore, a comparison was made between the two methods for accuracy and limitations.

2.4.4 Data analysis

The generated data were subjected to simple linear regression analysis using JMP version 14 pro to determine the model fitness based on the coefficient of determination (R^2) difference between the results obtained from organic carbon and sulfur content using both wet and dry methods.

3. Results and discussion

3.1 Results

3.1.1 Comparison between the wet and dry methods for organic carbon determination

3.1.1.1 Comparison of methods

The effectiveness of the models used in assessing organic carbon levels is demonstrated in Figure 2. Despite this, when analyzing the data based on wet and dry methods, the model's coefficient of determination value of 0.001 (R^2) suggested that there was no significant fit between the two methods, with a relatively high root mean square error (RMSE) of 2.64.

3.1.1.2 Comparison of agroecologies

When the data were assessed using agroecology as the baseline, there was a slight improvement over the results of the compared methods, with an R^2 value of 0.021 and an RMSE of 2.80 for the northern Guinea savanna. In contrast, the Sudan savanna agroecology showed lower values of 0.218 and an RMSE of 1.91, indicating a slightly higher level of agreement. However, these values were still far from the ideal value of 1.

3.1.1.3 Comparison of communities

Similarly, when analyzing the data for the various methods used to determine OC content across 12 communities, the results did not show complete agreement, indicating that the model was more effective than the earlier analyses used. Among the communities, only Bunkure in the Sudan savannah showed a significant value of 0.565, indicating that more than half of the model's results were reliable in that community.

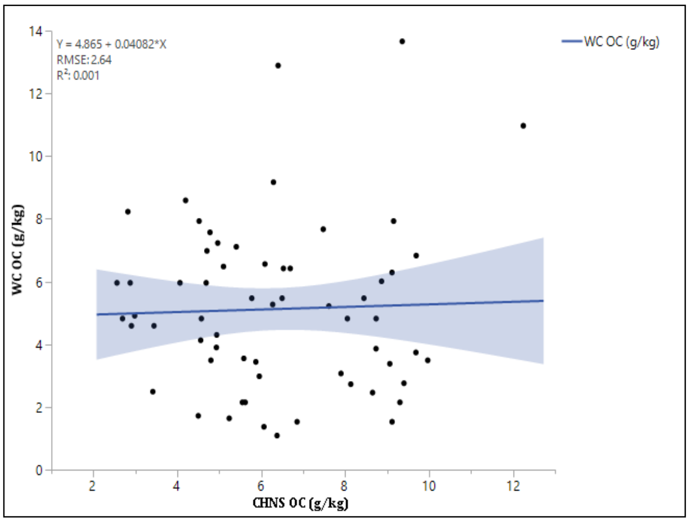

Figure 2: Comparison of the wet and dry methods for OC determination

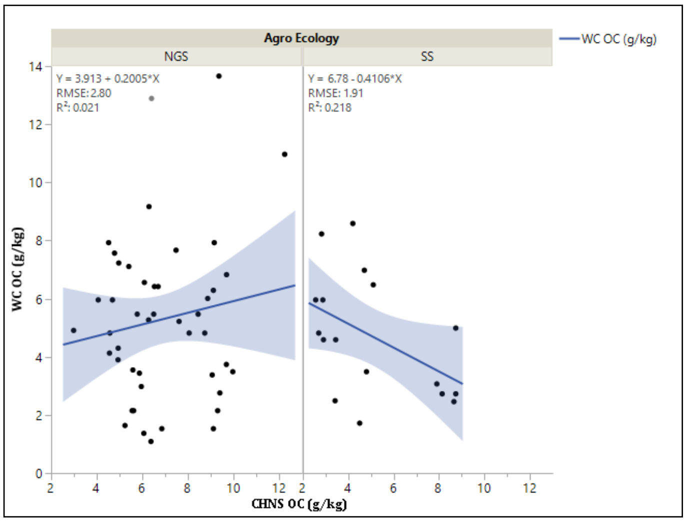

Figure 3: Comparison of wet and dry methods for OC determination based on agroecology.

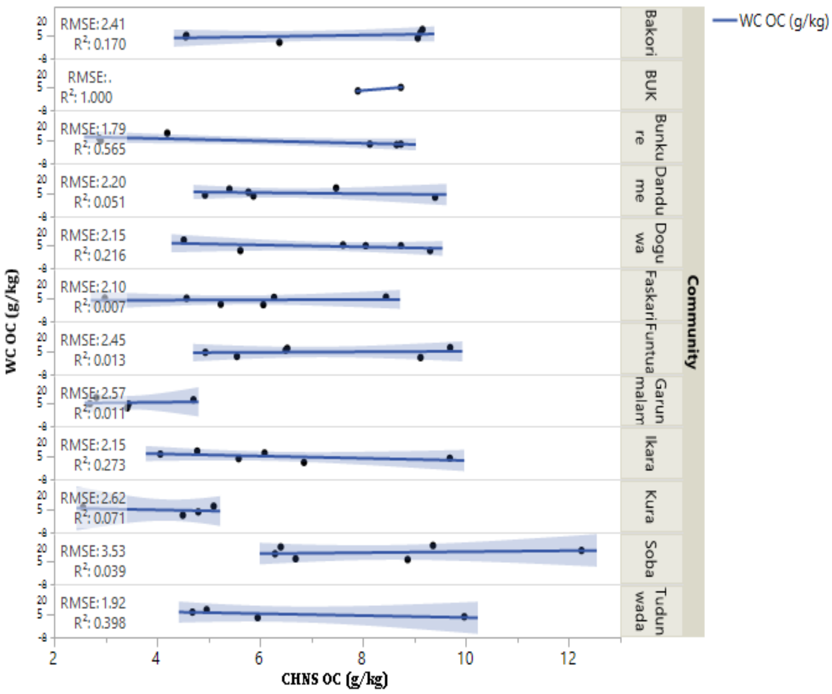

Figure 4: Comparison of wet and dry methods for OC determination at the community level.

3.1.1.4 Comparison of wet and dry methods based on community data

In addition to this, the data revealed substantial outcomes when the central tendency was measured, and the site values were grouped into mean, median, and coefficient of variation (CV). Nonetheless, the results for individual communities showed inconsistent mean, median, and CV values when both wet and dry methods were compared simultaneously.

When comparing the mean values of OC for Bakori using the wet chemistry method, the results indicated that the CHNS/O method was more effective in determining the quantity of organic carbon content within the same sample volume tested under similar laboratory conditions. The mean, median, and CV values for the wet method in Bakori were 4.6, 4.5, and 51.4 (g kg^{-1}), while the CHNS/O values were 7.1, 7.7, and 31.7 (g kg^{-1}).

In BUK, the mean, median, and CV values for the wet method were 4.0, 4.0, and 33.7 (g kg^{-1}), respectively, while the CHNS/O values were 8.3, 8.3, and 7.1 (g kg^{-1}), respectively. At Bunkure, the mean, median, and CV values for the wet method were 4.5, 3.7, and 5.9 (g kg^{-1}), and for the dry method, they were 5.9, 6.2, and 48.7 (g kg^{-1}). Similarly, in Dandume, the mean, median, and CV values for the wet method were 5.1, 4.7, and 40.0 (g kg^{-1}), and for the dry method, they were 6.5, 5.8, and 25.8 (g kg^{-1}).

In Doguwa, there was a significant variation in the mean, median, and CV values when comparing the wet and dry methods, with values of 4.5, 4.8, 48.0 (g kg^{-1}) for the wet method and 7.3, 7.8, and 25 (g kg^{-1}) for the dry method. Finally, in Faskari, the mean, median, and CV values for the wet method were 3.9, 4.9, and 48.0 (g kg^{-1}), while the CHNS/O values were 5.6, 5.7, and 32.8 (g kg^{-1}), and CV (g kg^{-1}), respectively. Invariably, the mean values for Funtua were 4.5, 4.9 median, and 49.7 (g kg^{-1}) CV for the wet technique, whereas those for the dry method were 7.1, 6.5, and 27.3, respectively, mean, median, and CV (g kg^{-1}). The mean, median, and CV for the wet approach were 5.4, 4.8, and 41.2 (g kg^{-1}), respectively, whereas the dry method yielded values of 3.4, 3.4, and 23.3 (g kg^{-1}). The figures for the wet technique in Ikara were 4.8, 4.9, and 46.9 (g kg-1), whereas those for the dry method were 6.2, 5.8, and 32.0 (g kg-1). Furthermore, the mean results for Kura were 4.4, 4.7, and 50.3 (g kg^{-1}) CV, whereas the dry technique yielded 4.2, 4.7, and 27.0 g kg^{-1}. Similarly, the results further revealed that the mean values for the wet chemistry in Soba were 9.9, median 10.1, and CV 32.7 (g kg^{-1}) compared with 8.3, 7.8, and 28.1, respectively. In Tudun Wada, the values were 4.9, 4.7, 41.1 g kg^{-1}) for the wet method compared to the mean, median, and CV for the dry method, which were 6.4, 5.5, and 38.2, respectively.

Table 1: Comparison between wet and dry methods based on mean, median, and coefficient of variation (CV) of soil sample communities.

Community	Agro Ecology	Mean (WC OC) (g kg-1)	Median(WC OC) (g kg-1)	CV(WC OC (g kg-1)	Mean(CHNS OC (g kg-1)	Median(CHNS OC (g kg-1)	CV(CHNS OC (g kg-1)
Bakori	NGS	4.6	4.5	51.4	7.1	7.7	31.7
BUK	SS	4.0	4.0	33.7	8.3	8.3	7.1
Bunkure	SS	4.5	3.7	53.7	5.9	6.2	48.7
Dandume	NGS	5.1	4.7	40.0	6.5	5.8	25.8
Doguwa	NGS	4.5	4.8	48.0	7.3	7.8	25.5
Faskari	NGS	3.9	4.9	48.1	5.6	5.7	32.8
Funtua	NGS	4.5	4.9	49.7	7.1	6.5	27.3
Garun Malam	SS	5.4	4.8	41.2	3.4	3.4	23.3
Ikara	NGS	4.8	4.9	46.9	6.2	5.8	32.0
Kura	SS	4.4	4.7	50.3	4.2	4.7	27.0
Soba	NGS	9.9	10.1	32.7	8.3	7.8	28.1
Tudun Wada	NGS	4.9	4.7	41.1	6.4	5.5	38.2

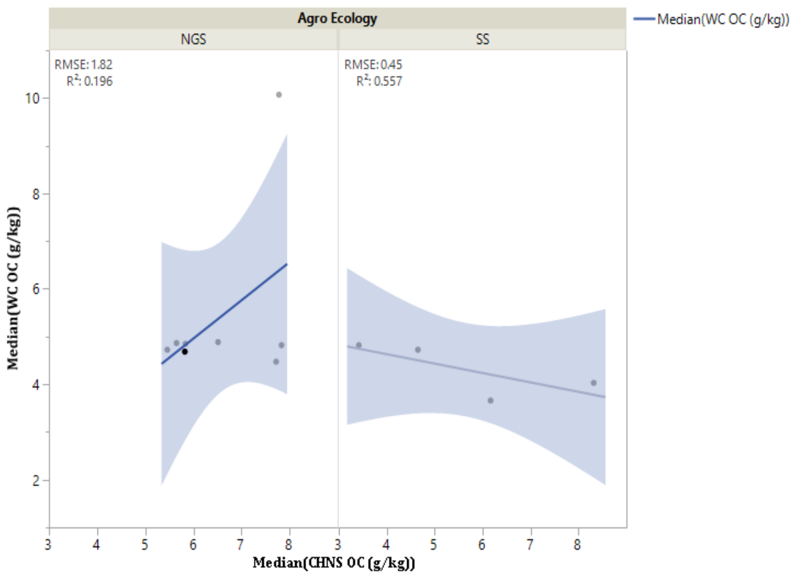

Figure 5: Comparison of wet versus dry methods based on agroecology using mean and median values.

3.1.2 Comparison of communities based on data distribution for sulfur determination

In this scenario, the data sets were examined using the mean, median, and coefficient of variation, followed by a chart to build a link between the compared sulfur techniques based on site values.

In Bakori, the mean, median, and coefficient of variation (CV) for wet chemistry were 46.56, 52.58, and 26.16, respectively, compared to values of 5.90, 5.76, and 8.64 (g kg-1) for the dry method; however, for BUK, they were 46.13, 46.13, and 48.19 (g kg-1) for the wet method, compared to 5.87, 5.87, and 20.72 (g kg-1). In Bunkure, however, the mean, median, and CV for the wet approach were 99.48, 57.73, and 111.83 (g kg-1), respectively, as opposed to values for the dry method of 4.81, 4.83, and 13.84 (g kg-1).

Dandume's mean, median, and CV for the wet approach were 65.81, 62.11, and 18.84 (g kg-1), respectively, whereas the dry method yielded 6.56, 6.53, and 5.35 (g kg-1). In Doguwa, the figures for the wet technique were 61.00, 55.67, and 26.30 (g kg-1), whereas the dry method had 7.53, 7.54, and 13.71 (g kg-1) mean, median, and CV, respectively.

The values for the wet technique in Faskari were 130.24, 76.80, and 116.33, respectively, compared to values of 7.01, 7.14, and 8.26 mean, median, and CV for the dry method. In Funtua, the wet chemistry values were 66.32, 60.31, and 40.66 (g kg-1), whereas the dry method values were 7.36, 7.51, and 12.23. Furthermore, the mean values for wet chemistry in Garun Malam were 114.12, 72.68 median 105.18 CV, as opposed to 6.45, 6.81, and 12.16 for the dry technique. When comparing the wet and dry methods in Ikara, the dataset revealed varying mean, median, and CV values.

The wet approach yielded 64.52, 49.48, and 57.68 (g kg-1), whereas the dry method yielded 6.63, 6.70, and 27.05 (g kg-1). In Kura, the mean was 72.04, the median was 75.26, and the CV was 11.69 (g kg-1). The wet and dry methods were 4.04, 4.04, and 0.89 (g kg-1). The wet method yielded mean values of 100.52, 101.80 median, and 21.58 CV (g kg-1), whereas the dry method yielded 7.46, 7.34, and 10.73, respectively. In another comparison, Tudun Wada had a mean of 186.34, a median of 93.04, and a CV of 103.77 (g kg^{-1}), compared to 9.09, 9.71, and 15.36 (g kg^{-1}).

3.1.2.1 Comparison based on agroecology for sulfur

When the dataset was aggregated and evaluated as mean values based on agroecology and shown in a histogram graphic (Figure 6), the findings revealed a remarkable degree of variance. However, the mean result for the northern Guinea savanna using wet chemistry was significantly greater (87 g kg^{-1}) than the dry combustion approach (8 g kg^{-1}). Similarly, in Sudan savanna agroecology, the mean value for wet chemistry was significantly higher (90 g kg^{-1}) compared to just (5 g kg^{-1}).

Table 2: Comparison between wet and dry methods based on mean, median, and coefficient of variation (CV) of soil sample communities.

Community	Mean(WC S (g/kg))	CV(WC S (g/kg))	Median(WC S (g/kg))	Mean(CHNS S (g/kg))	CV(CHNS S (g/kg))	Median(CHNS S (g/kg))
Bakori	46.56	26.16	52.58	5.90	8.64	5.76
BUK	46.13	48.19	46.13	5.87	20.72	5.87
Bunkure	99.48	111.83	57.73	4.81	13.84	4.83
Dandume	65.81	18.84	62.11	6.56	5.35	6.53
Doguwa	61.00	26.30	55.67	7.53	13.71	7.54
Faskari	130.24	116.33	76.80	7.01	8.26	7.14
Funtua	66.32	40.66	60.31	7.36	12.23	7.51
Garun Malam	114.12	105.18	72.68	6.45	12.16	6.81
Ikara	64.52	57.68	49.48	6.63	27.05	6.70
Kura	72.04	11.69	75.26	4.04	0.89	4.04
Soba	100.52	21.58	101.80	7.46	10.73	7.34
Tudun Wada	186.34	103.77	93.04	9.09	15.36	9.71

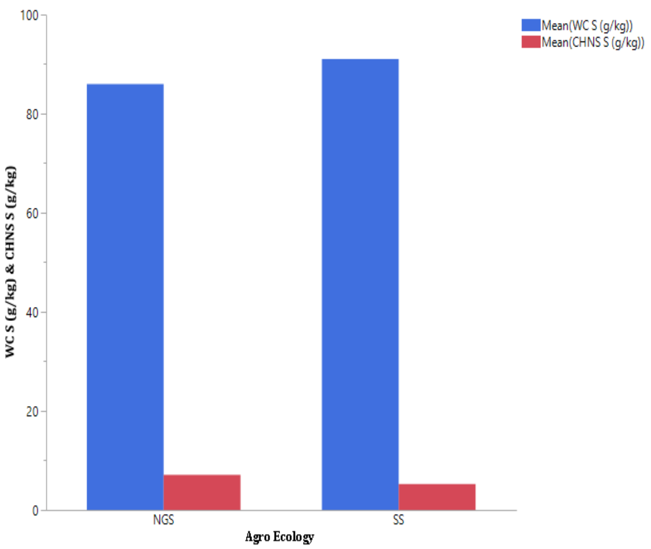

Figure 6: A histograph showing the mean values of the wet chemistry and dry combustion methods for sulfur determination based on agroecologies.

3.2 Discussion

3.2.1 Comparison between the wet chemistry and dry combustion method for organic carbon

The results of the data analyses based on mean values, median, and coefficient of variation (CV) across the sites in both northern Guinea savanna and Sudan savanna agroecologies indicated that the CHNS/O approach used for determining organic carbon was more reliable, as the mean and median values were higher and with low CV in all the observed cases. However, when the dataset was later grouped and analyzed based on agroecology, an agreeable R^2 value of 0.565 with a low RMSE of 1.79 of mean was observed, indicating that up to 57% agreement was observed using both methods for OC determination and was observed in (Bunkure) of the Sudan savanna. The remaining 43% of variance could not be accounted for due to experimental effects that were outside the researchers' control. This might be ascribed in part to the underlying mechanisms that created the observed disparities in accuracy between both approaches, as well as unexplained experimental error, which could have impacted the total agreement between the two methods utilized.

Furthermore, the initial comparison of the data obtained using the two methodologies utilized in the analysis (Figure 2) revealed extremely low R^2 values (0.001). Similarly, when the data were evaluated first, measures of central tendency were employed to demonstrate the influence of agroecologies, but this revealed considerable values over the subsequent analyses, which were based on the methodologies utilized. In this situation, the R^2 values for (NGS) were 0.021 and 0.218 for (SS).

The observed variances in values utilizing different analytical processes in the research are consistent with the findings of Faina (2011), who obtained comparable results in Israel using the modified Walkley-Black titration for organic carbon quantification in organic-rich sedimentary rocks. Brye and Slaton (2003) found that the modified WB approach occasionally overstated OC content while the regular WB method underestimated it. Furthermore, the investigations in the Sudan savannah produced a higher R^2 value, indicating that the model fitness was more consistent than in the NGS.

3.2.2 Comparison between the Wet and Dry Methods for Sulfur Determination

In contrast, the results for sulfur under wet circumstances are more promising than those obtained using the dry combustion approach. For sulfur determination, the average of the different locations resulted in more accurate findings than the wet technique or the dry approach. For example, wet chemistry yielded around 87 g kg^{-1}, but the dry technique yielded just 8 g kg^{-1} in the NGS. A similar pattern was seen in SS, with levels of 90 g kg^{-1} using the wet technique and only 5 g kg^{-1} recorded using the dry approach. This is consistent with a conclusion published by Schimel and Schaeffer (2012).

This also validates the findings of Lange et al. (2015), who assessed OC and sulfur in soil using a high-temperature induction furnace. They stated that the quantity of sulfur detected in the dry technique was less than the theoretical amount collected; consequently, the discrepancy was most likely due to the loss of sulfate from the sample during the drying process.

However, it appears to produce consistent findings and is useful if sample loss during drying is avoided. Furthermore, it was found that the dry approach has the benefit of allowing for the relatively quick application of the dry combustion process to soil extract. Similarly, it can be applied to extracting solutions containing redox reagents, which are tedious to analyze using the wet method.

4. Conclusion

Based on these data, it is feasible to conclude that the dry combustion approach yielded higher amounts of organic carbon than the community's mean and median values. Furthermore, in a similar case, the mean results obtained using the wet chemistry approach revealed an extremely high sulfur concentration. As a result, the dry combustion approach should be utilized for organic carbon (OC), as it yielded higher values suggesting full oxidation of (OC), however the wet method is preferred over the dry method for sulfur, according to the current study.

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Cite the Article: Habib, D. W, Idris F. B, Mustapha A. A. and Abdurrahman B. L (2024). Comparison Of Wet Chemistry and Dry Combustion Methods for Organic Carbon And Sulfur Determination in Soils of Nigeria's Savanna. *Acta Chemica Malaysia, 8(2): 114-119.*