FUDMA Journal of Sciences (FJS)
ISSN online: 2616-1370
ISSN print: 2645 - 2944
Vol. 10 No. 9, May, 2026, pp 240 – 244
DOI: https://doi.org/10.33003/fjs-2026-1009-5202

Determination of Elemental Composition of Treated Sewage Compost Used as Fertilizer in Zaria Metropolis, Nigeria, Using Instrumental Neutron Activation Analysis

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ABSTRACT

The use of treated sewage compost in agriculture promotes nutrient recycling and sustainable waste management. However, the presence of potentially toxic elements (heavy metals) necessitates detailed knowledge of elemental composition before large-scale land application. In this work, we determined the elemental composition of treated sewage compost used by farmers in Zaria metropolis, Nigeria, using instrumental neutron activation analysis (INAA). Ten samples were collected from ten dump sites as well as and two reference soil samples from uncontaminated locations. The samples were analysed at the Nigeria Research Reactor-1 (NIRR-1) laboratory, located at the Centre for Energy Research and Training (CERT), Ahmadu Bello University, Zaria, where they were irradiated in a thermal neutron flux of 5.0×10^{11} $n \cdot cm^{-2} \cdot s^{-1}$. Gamma-ray spectrometry was performed using a high-purity germanium detector. A total of 32 elements Mg, Al, Ca, Ti, V, Mn, Dy, Na, K, As, Br, La, Sm, U, Sc, Cr, Fe, Co, Zn, Rb, Sr, Cs, Ba, Nd, Eu, Tb, Yb, Lu, Hf, Ta, Sb, and Th were detected. Comparative evaluation with reference soils revealed higher concentration of elements in the compost. Although most concentrations were within internationally recommended agricultural limits, however, long-term accumulation may pose environmental risks under continuous application. The study provides baseline data for regulatory assessment in Nigeria.

Received: 12 May 2026

Accepted: 17 June 2026

Published: 26 June 2026

Keywords: Sewage compost, Heavy metals, Instrumental neutron activation analysis, Environmental monitoring, Soil contamination, Nigeria

INTRODUCTION

The use of treated sewage sludge (biosolids) in agriculture has become a widely adopted strategy for nutrient recycling and sustainable waste management (Pereira *et al*., 2020; Bertanza *et al*., 2016). Sewage compost is rich in organic matter and essential plant nutrients such as nitrogen, phosphorus, potassium, and micronutrients. These properties improve soil structure, water retention, and microbial activity, enhancing crop productivity.

Despite these agronomic benefits, sewage compost may contain elevated concentrations of potentially toxic elements (PTEs) such as cadmium, chromium, arsenic, lead, and mercury (Islam *et al*., 2017; Lamastra *et al*., 2018). These elements are non-biodegradable and may accumulate in soils over time, entering the food chain and posing risks to human and ecological health (Bolan *et al*., 2013). Continuous monitoring of compost quality is therefore, essential for environmental protection and regulatory compliance.

Advanced analytical techniques are required for accurate multi-element determination in complex environmental matrices. Instrumental neutron activation analysis (INAA) is a highly sensitive and non-destructive technique capable of determining major, minor, and trace elements simultaneously without chemical digestion (Glascock & Neff 2003; IAEA 2017). Its high accuracy and minimal matrix effects make it particularly suitable for environmental monitoring applications (Smith et al., 2018).

NAA is particularly advantageous due to its high sensitivity, ability to detect trace elements at parts-per-million or parts-per-billion levels, and minimal sample preparation requirements. The non-destructive nature of NAA makes it suitable for analyzing complex materials such as sewage compost without altering the sample matrix (Smith et al., 2018).

Smith et al., (2018) analyzed sewage compost samples from urban wastewater treatment plants using neutron activation analysis. Their findings indicated elevated levels of cadmium and lead, suggesting potential risks of soil contamination if such composts are applied continuously over long periods. The study emphasized the importance of routine monitoring of compost quality.

Jones et al., (2020) compared the elemental composition of sewage compost obtained from different geographical regions using NAA. Significant regional variations were observed in both nutrient and potentially toxic element concentrations. The study highlighted the necessity of developing region-specific guidelines for sewage compost application in agriculture.

Ozge Hanay et al., (2008) evaluated the agricultural suitability of municipal sewage sludge through the speciation of heavy metals using sequential extraction methods. Their results showed that iron, zinc, and manganese were the most abundant metals in the sludge. When compared with Turkish, European, and United States Environmental Protection Agency (US EPA) regulatory limits, most metals were below permissible levels, except for cadmium. The study concluded that the high mobility of cadmium and nickel posed a risk of phytotoxicity, rendering the sludge unsuitable for direct agricultural application.

Although several studies have evaluated sewage sludge composition globally, limited data exist for Nigeria. This study aims to determine the elemental composition of treated sewage compost used in Zaria metropolis.

MATERIALS AND METHODS

Study Area

Zaria is located in Kaduna State, Nigeria at Latitude 11.04°N and Longitude 7.71°E. The region is characterized by intensive agricultural activity, hence the use of compost and the importance of this study. Compost samples were collected from the following towns: Shika, Bomo, Samaru, Saye, and Biye, all within Zaria metropolis.

Sample Collection and Preparation

Ten treated sewage compost samples (Z1–Z10), two from each location, were collected from the 5 different agricultural locations earlier mentioned. Two untreated compost-free soil samples (C1 and C2) were collected as reference (controls). All the samples were collected using hand gloves, plastic containers and collecting spoon to avoid contamination. The samples were properly labelled at the points of collection to avoid mix-up as well as their locations of collection which were tagged as site l, site ll ...to site X. The Samples were labelled as sample (l) or Z(1), sample(ll) or Z(2)... to sample(X) or Z(10) respectively. The samples were then transported to the Centre for Energy Research and Training (CERT), Ahmadu Bello University, Zaria, for analysis. At CERT laboratory the samples were left open to ensure they were completely dried. They were then crushed into fine powder using mortar and pistol and then passed through a mesh sieve of size 2 mm to obtain a homogeneous matrix. Each sample was poured into a small transparent polythene leather and weighed using an electronic balance to achieve a desired weight of 0.15 g. Each of the samples, weighing 0.15 g, was then sealed using a hand dryer, and then finally placed inside a small vial and sealed for irradiation.

Irradiation and Gamma Spectrometry

The irradiation was carried out at the Nigeria Research Reactor-1 (NIRR-1) located at the Centre for Energy Research and Training (CERT), Ahmadu Bello University, Zaria. The thermal neutron flux in the irradiation position is 5.0×10^{11} n·cm^{-2}·s^{-1} and the samples were irradiated as follows. For the short-lived radionuclides, the samples were irradiated for 1 minute and 6 hours for long-lived radionuclides.

After irradiation, the samples were taken to the laboratory for gamma spectroscopy. The spectrometer consists of a high purity germanium detector (HPGe) connected to a PC- based multichannel analyser equipped with Maestro software.

For short lived radioisotopes, the first counting was performed immediately for 600 seconds. It was then followed by a second counting after 2 hours for 600 seconds.

For the long-lived radioisotopes, the counting was done after 3 days, which is referred to as first long counting and is done for 30 minutes. The sample was then allowed to cool further for another 7 days after which the second-long counting was performed for 1 hour.

Gamma-ray spectra were acquired using the high-purity germanium (HPGe) detector coupled to a multichannel analyzer. Energy and efficiency calibrations were performed using certified standard sources (Americium-241, Cobalt-57, and Caesium-137) from the International Atomic Energy Agency (IAEA). Elemental concentrations were then calculated using the comparative method of Glascock and Neff (Glascock and Neff 2003).

Calculation of Concentration

In order to convert measured activities of the samples (Bq) to concentrations (ppm), we used the comparator method given by Glascock (Glascock, 2003). In this case, the mass of an element present in the unknown sample relative to the comparator standard of unknown concentration is given by (Glascock & Neff 2003)

$$\frac{A_{sam}}{A_{std}} = \frac{m_{sam}(e^{-\lambda T})_{sam}}{m_{std}(e^{-\lambda T})_{std}} \quad (1)$$

where A_{sam} = activity of the sample (sam), and
A_{std} is the activity of the standard (std),
m_{sam} = mass of element in the sample,
m_{std} = mass of the element in the standard
λ = decay constant sample (sam) and standard (std), and
T = decay time.

In a steady-state neutron flux, the irradiation time, decay time and counting time are usually fixed for all samples and standards such that time dependent factors will cancel out. Thus, equation 1 simplifies to

$$C_{sam} = \frac{W_{std}A_{sam}}{W_{sam}A_{std}} C_{std} \quad (2)$$

where C_{sam} = concentration of the element in the sample and C_{std} that of the standard,
W_{sam} = weight of samples,
W_{std} = weight of the standard,
A_{sam} = activity of the sample,
A_{std} = activity of the standard.

RESULTS AND DISCUSSION

A total of 32 elements Mg, Al, Ca, Ti, V, Mn, Dy, Na, K, As, Br, La, Sm, U, Sc, Cr, Fe, Co, Zn, Rb, Sr, Cs, Ba, Nd, Eu, Tb, Yb, Lu, Hf, Ta, Sb, and Th, were detected across all samples. Tables 1 and 2 show respectively the concentrations of the 32 elements detected in the compost and reference samples. All the results are in ppm (mg/kg). In some of the composts, as well as in the reference samples, some elements were below detection limit indicated as BDL in the Tables. The results show that these composts contain seven (7) out of seventeen (17) essential elements needed by plants for growth. These essential elements, which are often classified as macronutrients (essential elements needed in large quantities), micronutrients (elements needed in trace amounts), and non-mineral, are as follows. The non-minerals are carbon, hydrogen, and oxygen while the primary macronutrients are nitrogen, phosphorus, and potassium. Others are the secondary macronutrients which are calcium and magnesium as well as the micronutrients which are iron, boron, chlorine, manganese, zinc, copper, molybdenum and nickel. They are required to complete a plant's life cycle, providing structure, energy transfer, chlorophyll production and enzyme activation. The essential macronutrients (major nutrients) detected in the compost are potassium (K), calcium (Ca) and Magnesium (Mg), while the essential micronutrients (trace elements) are iron (Fe), Manganese (Mn), Cobalt (Co) and Zinc (Zn). Potassium is essential for enzyme activation, osmotic regulation and photosynthesis. Calcium contributes to cell wall development and membrane stability. Magnesium serves as the central atom in chrolophll molecules and plays a key role in photosynthesis processes. Iron, manganese and zinc are important for enzymes reaction involved in plant metabolism and growth. We also detected some beneficial non-essential elements which can enhance plant growth or resistance such as sodium (Na), cobalt (Co), and aluminium (Al). These elements can contribute positively to plant growth under specific condition. For instance, sodium may partially substitute for potassium in some physiological functions whereas aluminum can enhance growth in certain plants specie adapted to acidic soil. Thus, these composts can effectively be used as fertilizer.

Table 1 also shows that these composts contain some heavy elements such as arsenic (As) and chromium (Cr). It is important to mention that the micronutrient (trace elements), including Fe, Co, Mn and Zn which indeed are heavy metals, are essential for plants growth at low concentration. However, excessive accumulation in the soil over time may pose risk to plants, animals and human through the food chain. These heavy metals are non-biodegradable and can accumulate in the soil following repeated compost application. So, long-term accumulation remains a concern, particularly for non-biodegradable metals such as chromium and cobalt. Continuous monitoring is therefore recommended to prevent ecological risk (Islam *et al*., 2017).

Table 1a: Concentration of Elements in the Compost (ppm)

ELEMENT	Z1	Z2	Z3	Z4	Z5
Mg	2920 ± 444	7490 ± 577	4410 ± 406	6893 ± 583	9303 ± 539
Al	28620 ± 458	101800 ± 1018	26740 ± 615	46550 ± 512	50830 ± 254
Ca	16580 ± 2089	22550 ± 2548	6562 ± 1430	48230 ± 3472	BDL
Ti	2576 ± 301	4205 ± 408	2437 ± 295	6175 ± 371	2990 ± 239
V	25.1 ± 3.0	55.7 ± 5.4	22.2 ± 3.8	42.0 ± 3.6	36.5 ± 2.8
Mn	211 ± 2	271 ± 2	246 ± 2	654 ± 3	316 ± 2.5
Dy	3.24 ± 0.27	9.23 ± 0.4	3.40 ± 0.31	7.93 ± 0.36	7.57 ± 0.33
Na	1280 ± 4	3390 ± 10	20740 ± 42	28840 ± 58	21880 ± 44
K	5520 ± 112	20900 ± 417	15690 ± 988	55130 ± 1433	85300 ± 1535
As	BDL	0.73 ± 0.16	BDL	BDL	BDL
Br	2.05 ± 0.09	4.35 ± 0.26	BDL	BDL	BDL
La	15.4 ± 0.08	32.4 ± 0.2	6.26 ± 0.18	4.20 ± 0.18	0.66 ± 012
Sm	2.34 ± 0.01	4.53 ± 0.02	0.90 ± 0.02	0.72 ± 0.02	0.086 ±0.015
U	2.12 ± 0.07	3.27 ± 0.18	BDL	BDL	BDL
Sc	4.56 ± 0.07	5.09 ± 0.08	26.6 ± 0.2	6.24 ± 0.09	1.30 ± 0.05
Cr	11.0 ± 1.9	18.8 ± 2.5	42.3 ±6	8.47 ± 2.14	11.9 ± 2.2
Fe	12790 ± 371	11250 ± 349	92400 ± 738	17350 ± 382	3201 ± 22
Co	4.85 ± 0.48	4.11 ± 0.50	57.4 ± 0.92	4.49 ±0.46	0.71
Zn	47.3 ± 12.2	104 ± 15	77.9 ±14.8	42.6 ± 14.3	BDL
Rb	77.1 ± 8.2	60.6 ± 8.9	19.7	138 ± 10	185 ± 9
Sr	601 ± 126	462	1219 ± 178	335	252
Cs	2.28 ± 0.29	4.30 ±0.50	3.14 ± 0.47	1.97 ±0.33	1.66 ± 0.23
Ba	401 ± 40	191 ± 40	231 ± 47	948 ± 54	714 ± 24
Nd	59.7	240 ± 28	185 ± 26	120 ± 22	BDL
Eu	0.90 ± 0.18	2.48 ± 0.26	0.91 ± 0.27	1.39 ± 0.18	0.48 ± 0.13
Tb	0.29	4.27 ± 0.25	0.72 ± 0.21	BDL	0.29
Yb	1.10 ± 0.15	15.4 ± 0.33	2.60 ± 0.24	2.74 ± 0.19	1.84 ± 0.15
Lu	0.16 ± 0.02	5.87 ± 0.13	BDL	BDL	0.47 ± 0.04
Hf	5.72 ± 0.36	8.04 ± 0.41	5.80 ± 0.41	5.40 ± 0.33	1.26 ± 0.23
Ta	1.05 ± 0.23	BDL	1.70 ± 0.25	BDL	BDL
Sb	0.25	0.28 ± 0.09	BDL	BDL	666
Th	9.90 ± 0.21	6.46 ± 0.19	2.26 ± 0.16	3.83 ± 0.16	1.08 ± 0.09

Table 1b: Concentration of Elements in the Compost (ppm)

ELEMENT	Z6	Z7	Z8	Z9	Z10
Mg	6555 ± 393	431 ± 37	2886 ± 214	9370 ± 48	2944 ± 344
Al	36340 ± 363	32840 ± 494	32590 ± 242	26970 ± 378	32710 ± 491
Ca	21320 ± 2217	6711 ± 1342	14620 ± 2003	8147 ± 1409	18290± 2209
Ti	4090 ± 303	3303 ± 310	2381 ± 257	2482 ± 263	4515 ± 321
V	32.1 ± 3.1	36.0 ± 2.9	33.8 ± 2.7	24.9 ± 2.6	25.4 ± 3.1
Mn	371 ± 2.6	287 ± 2.3	283 ± 2	342 ± 3	300 ± 2
Dy	3.84 ± 0.29	3.75 ± 0.25	4.09 ± 0.29	4.10 ± 0.27	4.52 ± 0.31
Na	21600 ± 43	20170 ± 40	8766 ± 88	6090 ± 12	4490 ± 45
K	66260 ± 1524	87880 ± 1670	63700 ± 573	40680 ± 448	17800 ± 178
As	BDL	BDL	1.93 ± 0.22	1.36 ± 0.17	0.50 ± 0.09
Br	BDL	BDL	7.82 ± 0.41	2.77 ± 0.30	7.42 ± 0.33
La	15.1 ± 0.2	3.02 ± 0.15	20.5 ± 0.1	30.3 ± 0.21	55.2 ± 0.2
Sm	1.39 ± 0.02	0.38 ± 0.01	9.98 ± 0.03	4.63 ± 0.02	6.57 ± 0.03
U	BDL	1.27 ± 0.25	5.93 ± 0.27	3 59 ± 0.24	4.65 ± 0.26
Sc	2.13 ± 0.06	4.80 ± 0.07	3.56 ± 0.07	3.07 ± 0.06	3.01 ± 0.06
Cr	BDL	29.4 ± 2.4	42.7 ± 2.8	28.0 ± 2.6	55.2 ± 3.0
Fe	5971 ± 29	12330 ± 272	17730 ± 337	11280 ± 271	12420 ± 286

ELEMENT	Z6	Z7	Z8	Z9	Z10
Co	1.47	4.82 ± 0.30	6.51 ± 0.36	3.76± 0.32	4.19 ± 0.32
Zn	BDL	73.3 ± 7.5	288 ± 0.90	379 ± 132	231 ± 12
Rb	184 ± 9	65.9 ± 4.7	82.0 ± 5.9	67.8 ± 5.1	68.7 ± 5.9
Sr	BDL	336	BDL	724 ± 108	BDL
Cs	3.00 ± 0.16	0.36 ± 0.07	2.14 ± 0.32	2.99 ± 0.26	1.07 ± 0.20
Ba	899 ± 49	1.98 ± 0.27	506 ± 39	511 ± 38	552 ± 40
Nd	144 ± 23	405 ± 42.9	BDL	BDL	BDL
Eu	1.29 ± 0.25	0.86 ± 0.12	0.88 ± 0.14	0.68 ± 0.12	0.78 ± 0.21
Tb	0.95 ± 0.17	0.60 ± 0.12	1.57 ± 0.15	0.37 ± 0.10	0.72 ± 0.13
Yb	1.53 ± 0.18	1.55 ± 0.15	3.71 ± 0.21	2.29 ± 0.17	2.13 ± 0.19
Lu	0.027 ± 0.005	0.47 ± 0.04	BDL	0.85 ± 0.07	BDL
Hf	4.64 ± 0.32	22.6 ± 0.5	22.2 ± 0.5	10.4 ± 0.37	17.3 ± 0.5
Ta	BDL	1.60 ± 0.14	0.89 ± 0.14	0.72 ± 0.14	1.08 ± 0.14
Sb	BDL	0.36 ± 0.07	7.51 ± 0.17	2.34 ± 0.11	1.05 ± 0.15
Th	37.8 ± 0.4	9.06 ± 0.19	24.0 ± 0.3	6.66 ± 0.18	27.5 ± 0.3

Table 2: Concentration of Elements in the Reference Samples (ppm)

ELEMENT	C1	C2
Mg	3200 ± 531	1714 ± 223
Al	53370 ± 800	6228 ± 193
Ca	BDL	BDL
Ti	5063 ± 440	2400 ± 293
V	57.2 ± 4.6	18.9 ± 1.6
Mn	224 ± 2	218 ± 2
Dy	6.55 ± 0.36	9.60 ± 0.39
Na	647 ± 4	554 ± 4
K	12260 ± 257	10550 ± 243
As	1.62 ± 0.11	1.10 ± 0.10
Br	3.79 ± 0.19	2.16 ± 1.16
La	33.4 ± 0.2	39.7 ± 0.2
Sm	5.89 ± 0.02	7.14 ± 0.03
U	4.36 ± 0.21	6.95 ± 0.24
Sc	7.77 ± 0.15	6.04 ± 0.08
Cr	35.4 ± 5.5	48.2 ± 3.6
Fe	19760 ± 751	13020 ± 299
Co	4.67 ± 1.01	3.24 ± 0.33
Zn	BDL	37.3 ± 8.1
Rb	68.7 ± 16.5	56.8 ± 6.4
Sr	169	95.4
Cs	2.29	2.92 ± 0.37
Ba	204	226 ± 36.5
Nd	221 ± 55	201 ± 21
Eu	1.98 ± 0.51	1.54 ± 0.15
Tb	2.84	3.27 ± 0.45
Yb	17.3 ± 0.6	11.3 ± 0.3
Lu	1.80 ± 1.0	1.65 ± 0.04
Hf	28.8 ± 1.0	48.8 ± 0.6
Ta	2.92 ± 0.83	2.59 ± 0.20
Sb	0.83 ± 0.12	0.11 ± 0.03
Th	21.9 ± 0.7	26.1 ± 0.4

CONCLUSION

In this work, we determined the elemental composition of treated sewage compost used in Zaria, Nigeria. Thirty-two elements were identified as follows: major elements include Mg, Al, Ca, Na, and K, trace and rare earth elements such as La, Sm, Eu, Yb and Th, and then environmentally sensitive heavy metals such as Fe, Mn, Zn, Cr, Co and As are also detected. Out of these 32 elements detected, 7 of them are beneficial for plants growth. The essential macronutrients (major nutrients) detected are potassium (K), calcium (Ca) and magnesium (Mg), while the essential micronutrients (trace elements) are iron (Fe), Manganese (Mn), Cobalt (Co) and Zinc (Zn). We also detected some beneficial but non-essential elements such as sodium (Na) and aluminium (Al) which can enhance plant growth or resistance. Thus, these composts can effectively be used as fertilizer by farmers.

However, for the heavy elements, care must be taken as high concentration over time can lead to soil contamination, and thus, become harmful to man or even affect plant performance. Although concentrations were largely within recommended limits (EPA, 2016). However, enrichment relative to reference soils indicates the need for periodic monitoring. The findings contribute valuable baseline data for environmental regulation and support sustainable waste-to-agriculture practices in Nigeria. It also provides the first comprehensive INAA-based assessment of treated sewage compost used in Zaria, Nigeria.

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