Comparative Analysis of Some Digestion Methods Used in the Determination of Metals in Soil and Sediments

Innocent Joseph[1*], H. M. Maina[1], Pigweh Amos Isah[1] and J. E. Eneche[1]

[1]*Department of Chemistry, School of Pure and Applied Science, Modibbo Adama University of Technology, Yola, Nigeria.*

Authors' contributions

This work was carried out in collaboration between the authors. Author IJ designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscripts. Author HMM supervised the work. Authors PAI and JEE reviewed all the experimental design for the analyses and managed the literature searches. All authors read and approved the final manuscript.

Article Information

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ABSTRACT

Analytical study was carried in order to compare the accuracy of three (3) frequently used digestion methods, in the determination of metals in soil and sediments. Samples of soil and sediments were taken from three different locations namely; Bakalchi, Batare, and Anguwan Honna in Girei Local Government Area in Adamawa State Nigeria. Sample matrix was formed from the three samples and the three digestion methods was carried out on each of the sample matrix formed, the methods are, Method I: Roast before digestion using aqua regia as used by Ehi-Eromosele and coworkers [1], Method II: Air dried and digest using Aqua-regia similar to the work of Saxena [2], and Method III; Roast using ammonium chloride before digestion with aqua regia similar to the work of Voica et al [3]. The metal elements Calcium and Iron were chosen because of their relative abundance in natural soil and sediments. Results obtained shows that the soil and sediment samples contain the metal element calcium and Iron which were used to examine the frequently used digestion methods. Based on the result obtained from the analysis, method III (Roast with NH_4Cl and digest with aqua regia) appears to be the most promising, given the higher concentration of the elements in all the matrix samples examined.

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Corresponding author: E-mail: innocentjoseph1218@gmail.com;

1. INTRODUCTION

The accuracy of soil quality for agricultural purpose depends solely on the methods used to ascertain the various components of soil elements. The challenges of analytical complexity has been recognized by the soil community and consequence, acceptable precision and accuracy for digestion methods has been reported as typically less than 20% [3]. The nature of the soil has also provided an avenue for knowledge where soil provides vascular plants with medium for growth, store mineral and trace elements, also its supplies soil organism with most of it nutritional requirements in form of metal elements as component. To ascertain the level of different elemental components of soil the right method must be used. These components can exist in its various forms in soil and different forces keep them bound to soil particles [4].

The distribution of metals in soil and sediments is greatly influenced by activities of organisms, climate, topography, parent's materials and time [5]. But the methods used in determining the level of such elements in the soil are of great concern. Soil represents a matrix with inherent heterogeneity and variable minerals compositions leading to analytical complexity. The determination of metals in soil is challenging and requires the development of various specific methods. For this reason, three different methods have been compared in this study, the efficiency of digestion or dissociation of the metal elements in solution which depends on the procedures has been evaluated. It is essential to examine thoroughly some of these analytical methods since soil is of utmost importance to plants, human health, livestock and aquatic industries [6].

2. MATERIALS AND METHODS

2.1 Sample and Sampling Methods

Samples were taken from Girei Local Government in three specific areas: Batare (BT), Bakalchi (BK), and Anguwan Honna (AN). A matrix was formed by mixing 5 g of each soil from the three different locations to form a composite of soil samples or matrix. Sampling method carried out is similar to that described by [7]. Sampling involves the selection from the total population of subset of individuals upon which measurement will be made [7]. It is also seen as the method of obtaining fraction of bulk material which represents the whole material under analysis.

2.2 Sample Preparation

Three different (3) digestion methods of sample preparation process were carried-out on each separate portion of the sample matrix.

2.2.1 Method I; Roast before digestion using aqua regia

The required sample (1.0 g) of soil sample was transferred completely into crucible and was placed on heating mantle and roasted at about 105-110°C (the temperature was controlled using thermometer) for one hour and was allowed to cool for two hours in a desicator. The method employed was similar to that of Ehi-Eromosele [1]. An exact 1 g of the soil sample was transferred into kjeldahl digestive flask, 24 ml aqua regia solution at the ratio of 3:1 HCl to HNO_3 acid was added to the sample. The 24 ml aqua regia was added and swirled to wet the sample and it was left over night. Next day the flask was placed on hot plate and heated at 60°C to 70°C for 15 minute. The temperature was raise until it boiled for two minutes. The material was then cooled, distilled water was added and made up to 40 cm^3, the solution was filtered, the filter paper was then washed with 0.25M HNO_3 acid after which, 0.25 ml HNO_3 was added to mark.

2.2.2 Method II; Air dried and digest using aqua-regia;

The method carried out was similar to the work of Saxena [2]. Soil sample was stored in seal polyethylene container before arrival to the laboratory. Each of the soil sample collected was spread on a glass slid and air dried for 72 hrs before grinding with mortar and pistil, then sieved using 0.2 mm nylon sieve. The required sample (1.0 g) of soil sample was transferred on glass slid and air dried until a constant weight was obtained and the whole sample was transferred quantitatively into kjeldahl digestive flask and digested with 5 cm^3 aqua regia and the sample was allowed to stand all night. The next day the sample was swirled and made up to mark with distilled water to 40 cm^3 and then filtered.

2.2.3 Method III; Roast using ammonium chloride before digestion with aqua regia

The method carried out was similar to the work of Voica [3]. The required sample (1.0 g) of soil sample was weighed and transferred into a crucible. 5 cm^3 of ammonium chloride was added and mixed and the sample was placed on heating mantle and roasted at about 105℃ to 110℃ for 1 hour and allowed to cool for two hours in a desicator. Then the exact sample was transferred quantitatively into kjeldahl digestive flask and 24 cm^3 Aqua-regia was added. The sample was swirled in order to wet the entire sample and was allowed to stand overnight. The next day distilled water was added and made up to 40 cm^3 and then filtered.

All the filtrates were quantitatively transfered into sample bottle at the end of each filtration and the absorption of calcium and iron using AAS in each sample respectively and the concentration was traced from the calibration curve obtained.

3. ANALYTICAL TECHNIQUES

The analytical technique of the analysis of soil samples involves the use of atomic absorption spectrophotometer (AAS). For determination of essential metals calcium (Ca^{2+}) and heavy metals iron (Fe^{3+}). Analysis by AAS is based on absorption of electromagnetic radiation by atoms. The absorption of the radiation is proportion to the number of atoms or concentration of the element of interest in the sample. This follows Beer-Lambert Law.

$$A = Log\ I_o/I_t = abc$$

Where;

A = absorbance of radiation by atom
I = intensities of incidence and transmitted radiation respectively
a = absorptivity constant
b = path length
c = concentration of atomic specie of interest in the sample

Absorbance readings for various standard concentrations were obtained using the same instrument. Various standards were prepared and there absorption was used to plot calibration curve for each metal elements. The absorbance of the sample element of interest was obtained and the concentration was traced on the standard calibration curve.

The results obtained from the calibration curve have concentration in ppm which was converted to mg/Kg of soil sample using the formular,

$$[metal] = mg/Kg = ppm\ X\ (v/Wt)$$

Where;

ppm = concentration in part per million
v = volume of digested soil
Wt = weight of sample

Sample Mean and Standard deviation was calculated.

4. RESULTS AND DISCUSSION

4.1 Results

This section covers all the result of the study as well as the discussion and interpretation of the results obtained.

Table 1. Results for calcium

Sample site	Methods	Concentration (mgKg^{-1})
Matrix Sample	I	416±0.57
	II	444.57±0.94
	III	581.84±1.98

Table 2. Results for iron

Sample site	Method	Concentration (MgKg^{-1})
Matrix Sample	I	924.48±0.65
	II	852.48±1.68
	III	937.20±1.50

Method I; Roastbefore digestion using aqua regia
Method II; Air Dried and Digest Using Aqua-regia
Method III; Roast using ammonium chloride before digestion with aqua regia

4.2 Discussion

All the methods used detected the presence of the metals (Iron and Calcium) in the digested sample of the sample matrix of soil and sediments obtained from three different locations.

From the result obtained as shown in Table 1 which shows the different concentration of Calcium metal in mgKg^{-1} in the matrix sample; Method III revealed the highest concentration of the calcium metal. This revealed that the Method extracts the particular metal i.e. Calcium with highest effectiveness and efficiency than the others.

Table 2 shows the different concentration of Iron metal in mgKg^{-1} at different location sites; Method III shows the highest concentration of the Iron metal in the sample matrix.

In general, method III shows the highest concentration of the metals (both Calcium and Iron) in the different sample matrix of soil samples from different sites locations.

5. CONCLUSION

The results obtained revealed that for Calcium and Iron metal digestion, method III has the highest sensitivity which gives the highest concentration compare to other methods in the same sample matrix of soil and sediments, in method III the sample of soil have been roasted with an ammonium chloride salt (NH_4Cl). The heating process decomposes the ores of these mineral elements to their oxides, on heating the ammonium salt evaporate leaving mineral acid (HCl), the mineral acid dissolves the oxides of the elements and then treatment with aqua regia extracts the metals elements [8], as shown in Table 1; Methods II has the concentration of the Calcium metals higher than method I but lower than method III. This is probably, because the metals have been converted to their oxides by heating but they have not been digested with any minerals before extraction with aqua regia. The aqua regia function is to extract the metal elements in this case but not as effective and efficient as in the presence of mineral acids. Method I has the lowest concentration. The mineral elements have been extracted from its mineral ore with no pre-treated that decomposes the mineral to its oxides.

6. RECOMMENDATION

This study recommends that further study should be carried out to examine other elements with the same methods that have not been included in this study and other methods should be study as well.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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