AL-BUTANA JOURNAL Of APPLIED SCIENCE
http// : ojs.abutana.edu.sd, ISSN: 1858-6616
Issue (17), December, 2025, Page NO. 54-65

# Determination of Some Heavy Metals in Basaltic Rocks at Red Sea Hills, Eastern Sudan using Wet and ash Digestion Methods

**Eiman Ibrahim Balla Ibrahim**

*Department of Chemistry, Faculty of Science, Red Sea University, Sudan*

Email:Eimanballa5@gmail.com

## Abstract

*Rocks are an important source of a variety of metals, particularly heavy metals and may serve as an enriched source for these metals. Igneous rocks, basalt and granite, are widely distributed in Red Sea Hills. The present study aimed to measure the concentration of six heavy metals: Iron, Cobalt, Copper, Cadmium, Lead and Silver in basaltic rocks from Gebeit Mine (Gebeit Ma'aden) and MhamdQol, northern Red Sea Hills. Collected rocks samples were treated using wet and dry digestion by mineral acids-* $HNO_3$, $H_2SO_4$, *HCl,* $HClO_4$ *and HF. The analyses were performed using Atomic Absorption Spectrophotometry (AAS). Average concentration (mg/kg) of heavy metals obtained by AAS by wet digestion method was found as follows: iron (38.61), cobalt (6.44), copper (17.17), cadmium (1.33), lead (1.34) and silver (1.8), where the average concentration (mg/kg) by ash digestion method was found: iron (121.03), cobalt (9.32), copper (21.13), cadmium (2.04), lead (2.67) and silver (4.07). The study demonstrated that, heavy metals concentrations obtained by ash digestion method were highest in basaltic rocks samples. The results obtained from the study encourages to study of commercial production of these metals in Red Sea Hills.*

**Key Words**: *heavy metals, wet digestion, ash digestion, Red Sea Hills*

## Introduction:

Heavy metals occur in rocks as minerals or forming organic complexes. The occurrence of elevated concentrations of heavy metals in rocks and sediments can be a good indicator of man-induced pollution rather than natural enrichment of the sediment by geological weathering. The minerals in these rocks are predominantly silicates, which dominate the characteristics of most soils (Harrison, 2007). The chemical analysis of rocks and sediments is performed for

**Eiman Ibrahim Balla Ibrahim. (2025). Determination of Some Heavy Metals in Basaltic Rocks at Red Sea Hills, Eastern Sudan using Wet and ash Digestion Methods . *Al-Butana Journal of Applied Science* (17): 54-65.**

a variety of environmental and petrological purposes. For example, chemical data are used in the identification of various rock types; also, sediments contain significantly higher concentrations of many metals than are found in the underlying water; as such, they must be analyzed for pollutant contributions to the environment. The importance of these types of data is well established. Silicate dissolution usually involves either a fusion, with subsequent dissolution of the wet digestion employing mineral acids (Horowitz and Elrick, 1985).

## Heavy metals in rocks and soil:

The natural concentration of heavy metals in soils depends primarily on geological parent material composition. The chemical composition of parent material and weathering processes naturally conditions the concentration of different heavy metals in soils. In principle, these heavy metals constitute the trace elements found in the minerals of igneous rocks at the time they crystallize. In sedimentary rocks, formed by the compactation and compression of rocky fragments, primary or secondary minerals like clays or chemical precipitates like $CaCO_3$, the quantity of these trace elements depends on the properties of the sedimented material, the matrix and the concentrations of metals in water when sediments were deposited. In general, concentrations of heavy metals are much higher in igneous rocks. Nonetheless, these ranges vary widely, which implies that the natural concentration of heavy metals in soil will also vary widely (Nanos and Martin).

## Digestion Methods:

Many metal analyses are carried out using atomic spectroscopic methods these methods commonly require the sample to be presented as a dilute aqueous solution, usually in acid. ICP-mass spectrometry requires similar preparation. Other samples may be analyzed in solid form. For x-ray fluorescence, the solid sample may require dilution with a solid buffer material to produce less variation between samples and standards, reducing matrix effects. A solid sample is also preferred for neutron activation analyses and may be obtained from dilute aqueous samples by precipitation methods (Mitra, 2003). Several wet digestion and dry ashing methods were compared for the precise and accurate determination of some trace elements in biological and environmental materials. The wet digestion methods were generally faster than the dry ashing methods, but required the use of large amounts of reagents and, therefore, gave higher blank contributions for some elements. The main advantages of the dry ashing method were the lower blank levels, improved (lower) background current and its ability to handle considerably larger amounts of sample. However, careful dissolution of the sample ash in a suitable reagent was necessary (Adeloju, 1989). Under suitable conditions, both decomposition methods allowed the reliable voltammetric determination of trace elements in biological and environmental materials with relative standard deviations of between 1 and 3%. The ultimate choice of decomposition method was influenced by the amount of sample available, the nature of the sample, the sample matrix and the analysis time available.

## Materials and method:

### Sampling sites Collection:

Igneous basalt rocks, are the common species of rocks at Red Sea area, basaltic rock samples were taken from various locations at the study area, Figure (1), placed in plastic bags, transferred to laboratory of Earth Science College, Red Sea University, and classified to their subgroups. Samples were dried and ground to a fine powder.

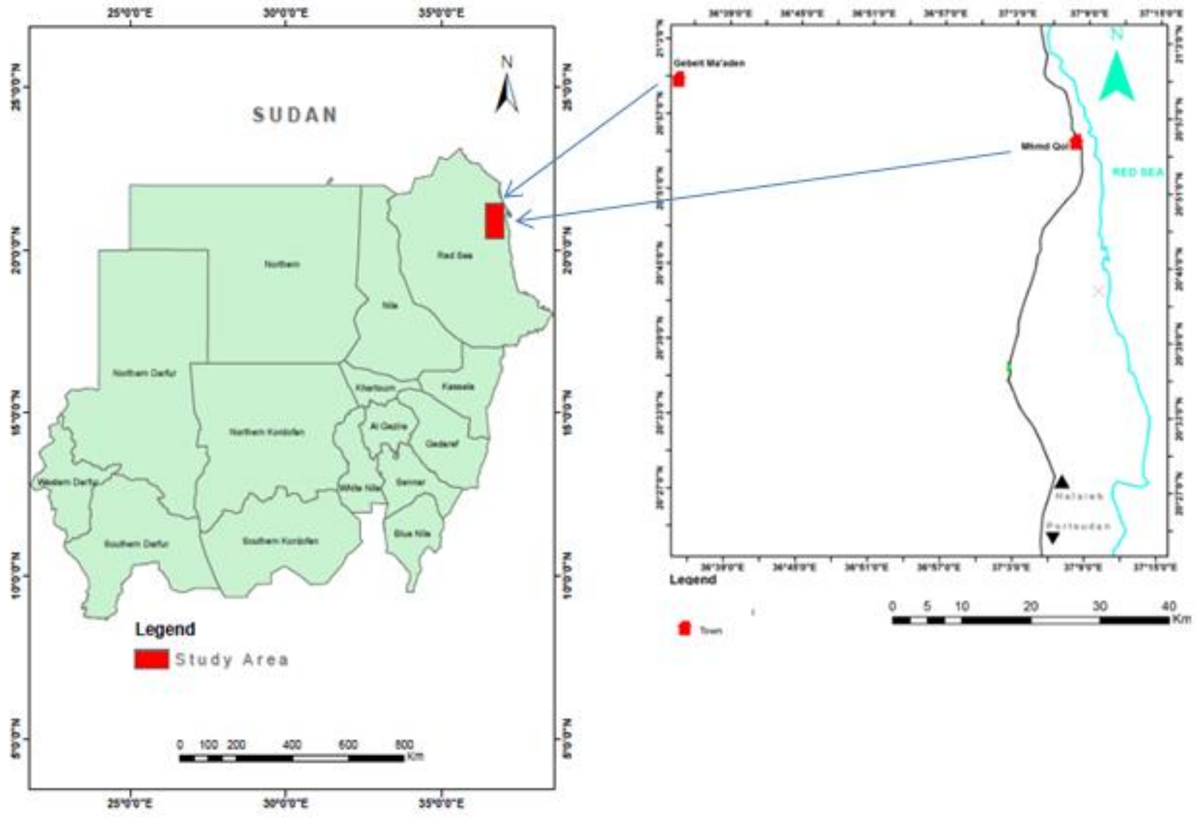


Fig. (1) Samples sites in eastern Sudan

## Preparation of the samples for (AAS) analysis:

### Wet digestion:

5 g of dried homogenous rock sample was weighted and placed in 100-ml Teflon beaker, 10 ml of concentrated nitric acid were added, the mixture was allowed to stand at 350 ℃ for 2 hours and heated carefully on a hot plate, beaker was cooled and added small amount (2-4ml) of 98% H2SO4. The mixture was heated again and allowed to evaporate to small volume, the completely digested sub samples were allowed to cool at room temperature, and the undigested portion of the rock was filtered off through a Whatmann filter paper and transferred the filtrate to a 100 ml flask and diluted to volume with deionized water. The obtained solution was used in the analysis by atomic absorption spectroscopy (Baker, 1971).

## Ash digestion:

5 g of dried rock sample was ignited in a porcelain crucible at 600 °C for 18 hours and transferred to a 100-ml Teflon beaker; dissolved by adding 5 ml of $H_2SO_4$ and 10 ml of HCl and evaporated to dryness. The addition of $H_2SO_4$ and HCl was repeated and evaporated to dryness. 5 ml of HF was added and evaporated to dryness. The residue was dissolved the in a minimum amount of $HNO_3$ and transferred the solution to a100-ml volumetric flask, and diluted to volume with deionized water. (Abollino et al., 2002). The obtained solution was used in the analysis by atomic absorption spectroscopy.

## Instrumentation:

The instrument used for metal measurement was atomic absorption spectrometer Atom (AAS), (A.A-6800, A.A.S. SHIMADZU). The general method involves atomization of samples by thermal sources and the absorption of a specific wavelength by the atomic source as it is excited. The quantity of the same element absorbed by the atomic vapor is proportional to the concentration of the atoms in the ground state (Perkin-Elmer, 1996). Instrumental settings used for the AAS analyses of the metals are shown in the Table (1).

**Table (1) AAS settings for the analyzed metals**

| Element | Lamp current (mA) | Wavelength (nm) | Slit Width | Flame Gases |
|---|---|---|---|---|
| Fe | 12 | 248.3 | 0.2 | Air-Acetylene |
| Co | 12 | 240.7 | 0.2 | Air-Acetylene |
| Cu | 6 | 324.8 | 0.7 | Air-Acetylene |
| Cd | 8 | 228.8 | 0.7 | Air-Acetylene |
| Pb | 10 | 283.3 | 0.7 | Air-Acetylene |
| Ag | 4 | 328.1 | 0.7 | Air-Acetylene |

## Statistical analysis:

The relative standard deviation (R.S.D) gives the precision as a percentage of the mean (average). The values of the relative standard deviation (R.S.D) of the triplicate measurements of the basalt rocks (wet and ash) samples showed acceptable repeatability. The data presentations, the principle component analyses were performed using a software package programme of Statistical Package for Social Studies (SPSS/PC+) (Gupta, 1999). Results of the heavy metal analyses were subjected to a one-way Analysis of variance (ANOVA) to test for significant differences ($p<0.05$) in the concentrations of heavy metals in different samples of the rocks.

## Results and discussion:

### Wet digestion:

Table (2) and fig.(2), shows results of basalt rocks (mg/kg) by wet digestion. The concentrations of iron and copper in the samples showed the highest concentration (38.61±59.88) and 17.17±5.13) mg/kg respectively, where cadmium showed the lower concentration (1.33±0.41 mg/kg). Elements can be ranked according to their average concentration as follows: Fe > Cu > Co > Ag >Pb> Cd. According to One way ANOVA, (*P-value* 0.017), the metals were revealed that the concentration of metals were significantly varied with respect to different species ($P < 0.05$).

**Table (2) results of basalt rocks (mg/kg) by wet digestion**

| No. | Fe | Co | Cu | Cd | Pb | Ag |
|---|---|---|---|---|---|---|
| 1 | 5.86 | 6.234 | 15.9 | 1.78 | 2.95 | 1.96 |
| 2 | 107.72 | 6.928 | 22.82 | 1.00 | 0.068 | 1.78 |
| 3 | 2.24 | 6.156 | 12.8 | 1.20 | 0.996 | 1.66 |
| Mean | 38.61 | 6.44 | 17.17 | 1.33 | 1.34 | 1.80 |
| St.dv. | 59.88 | 0.42 | 5.13 | 0.41 | 1.46 | 0.15 |
| R.S.D % | 155.08 | 6.52 | 28.98 | 30.82 | 108.95 | 83.33 |

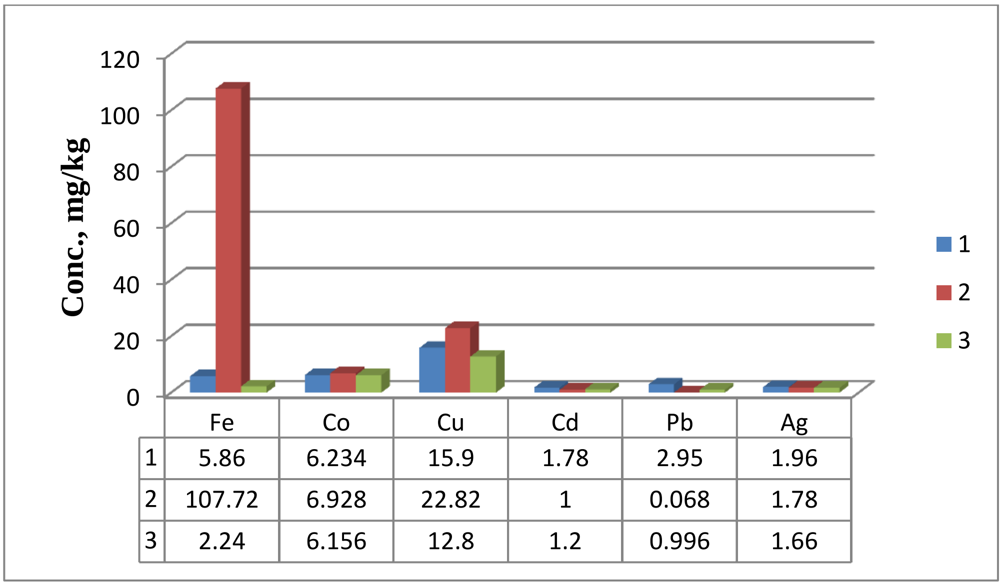


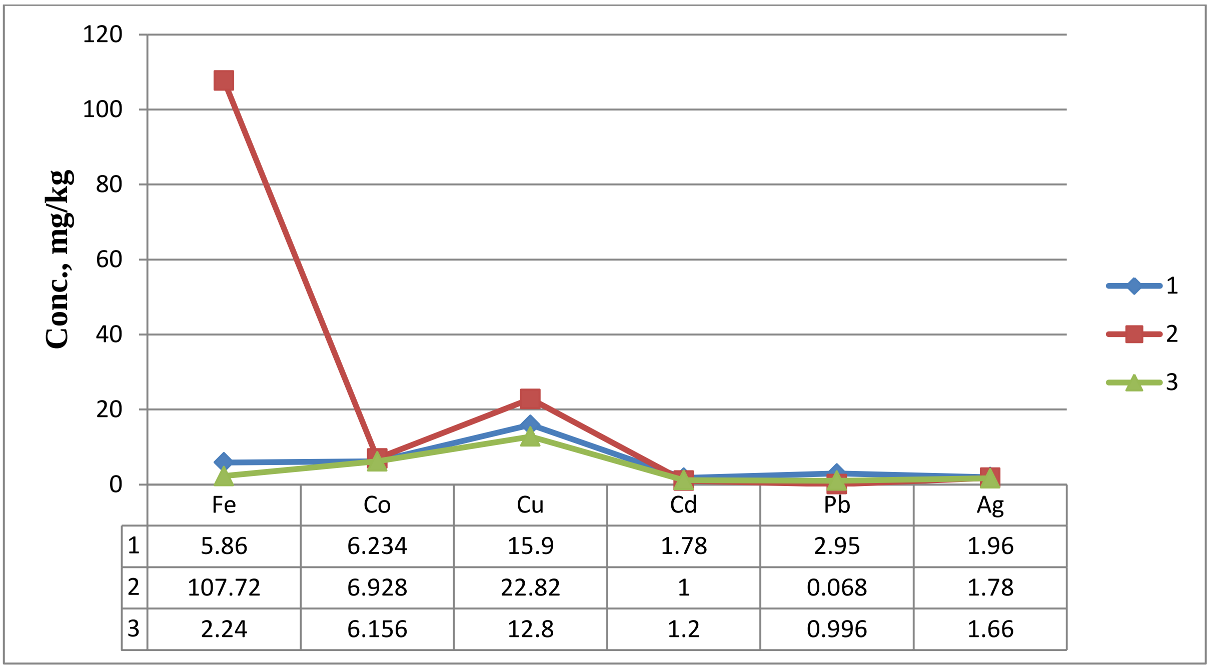
**Fig. (2) Results of basalt rocks (mg/kg) by wet digestion**

**Correlation coefficient between elements, basalt rocks, wet digestion:**

Correlation coefficient is significant at the 0.05 level, Table (3) and fig.(3), silver, Ag shows strongest correlation with all elements except with Cd and Pb (strong), the other strong correlation as follows: Fe-Cd, Fe-Pb, Co-Cd, Co-Pb, where weak correlation was observed between the elements; Fe- Co, Fe-Cu, Co-Cu, Cd-Pb,

Table (3) Correlation coefficient between elements in basalt rocks (wet digestion)

| | Fe | Co | Cu | Cd | Pb | Ag |
|---|---|---|---|---|---|---|
| Fe | 1 | | | | | |
| Co | 0.039 | 1 | | | | |
| Cu | 0.176 | 0.137 | 1 | | | |
| Cd | 0.527 | 0.566 | 0.703 | 1 | | |
| Pb | 0.482 | 0.521 | 0.658 | 0.046 | 1 | |
| Ag | 0.946 | 0.985 | 0.878 | 0.419 | 0.464 | 1 |



|   | Fe | Co | Cu | Cd | Pb | Ag |
|---|---|---|---|---|---|---|
| 1 | 5.86 | 6.234 | 15.9 | 1.78 | 2.95 | 1.96 |
| 2 | 107.72 | 6.928 | 22.82 | 1 | 0.068 | 1.78 |
| 3 | 2.24 | 6.156 | 12.8 | 1.2 | 0.996 | 1.66 |

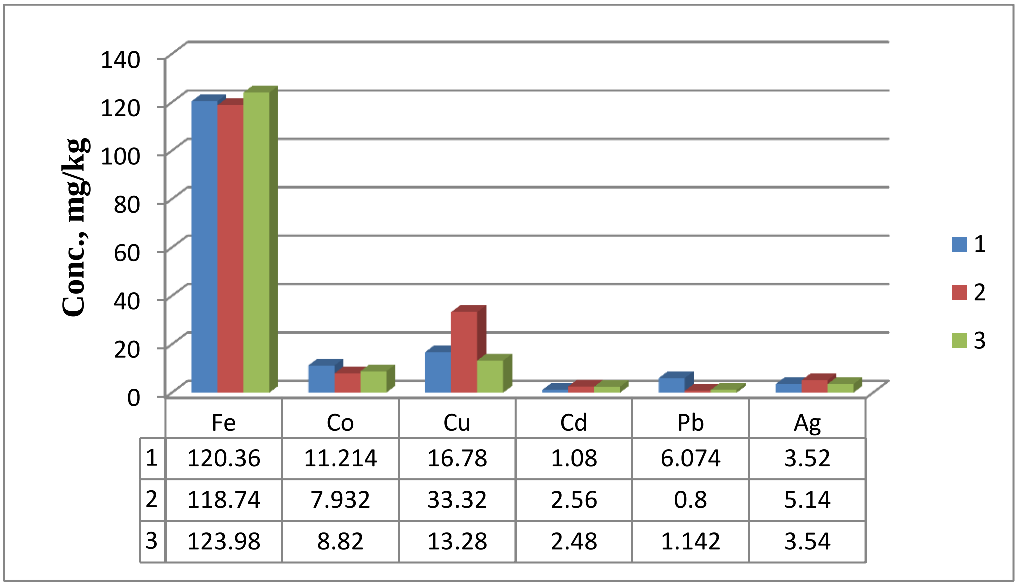
**Fg. (3) Correlation coefficient between elements in basalt rocks (wet digestion)**

## Ash digestion:

Table (4) and Fig.(4) presented the result of heavy metals by ash digestion; iron concentration shows the highest concentration (121.03±2.68 mg/kg), the lower concentration was cadmium (2.04±0.83 mg/kg). The ranking of the elements by this method were in the following order: Fe > Cu > Co > Ag >Pb> Cd. One-way analysis of variance ANOVA, (*P-value* 0.078) showed no significant differences between elements ($P < 0.05$) with respect to different species.

**Table (4) Results of basalt rocks (mg/kg) by ash digestion**

| No. | Fe | Co | Cu | Cd | Pb | Ag |
|---|---|---|---|---|---|---|
| 1 | 120.36 | 11.214 | 16.78 | 1.08 | 6.074 | 3.52 |
| 2 | 118.74 | 7.932 | 33.32 | 2.56 | 0.8 | 5.14 |
| 3 | 123.98 | 8.82 | 13.28 | 2.48 | 1.142 | 3.54 |
| Mean | 121.03 | 9.32 | 21.13 | 2.04 | 2.67 | 4.07 |
| St.dv. | 2.68 | 1.69 | 10.70 | 0.83 | 2.95 | 0.93 |
| R.S.D % | 2.21 | 18.13 | 50.63 | 40.86 | 110.48 | 22.85 |



|   | Fe | Co | Cu | Cd | Pb | Ag |
|---|---|---|---|---|---|---|
| 1 | 120.36 | 11.214 | 16.78 | 1.08 | 6.074 | 3.52 |
| 2 | 118.74 | 7.932 | 33.32 | 2.56 | 0.8 | 5.14 |
| 3 | 123.98 | 8.82 | 13.28 | 2.48 | 1.142 | 3.54 |

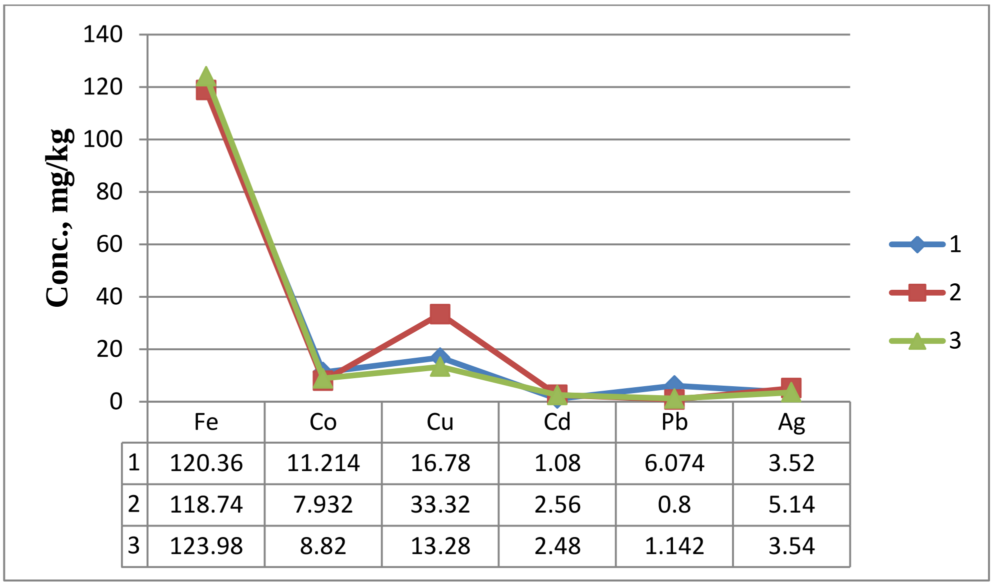
**Fig. (4) Results of basalt rocks (mg/kg) by ash digestion**

### Correlation coefficient between elements, basalt rocks, ash digestion:

Correlation coefficient between metals in basalt rocks, ash digestion are presented in table (5) and fig.(5), the strongest correlation was obtained from Fe with Co, Cd , Pb and Cu with Cd and Pb, strong correlation; Fe-Ag, Co-Cu, Co-Ag, Cd-Ag and Pb-Ag while the weak correlation was obtained from Fe-Cu, Co-Cd, Co-Pb, Cu-Ag and Cd-Pb.

**Table (5) Correlation between elements in basalt rocks (ash digestion)**

|   | Fe | Co | Cu | Cd | Pb | Ag |
|---|---|---|---|---|---|---|
| Fe | 1 |  |  |  |  |  |
| Co | 0.970 | 1 |  |  |  |  |
| Cu | 0.367 | 0.603 | 1 |  |  |  |
| Cd | 0.893 | 0.138 | 0.741 | 1 |  |  |
| Pb | 0.899 | 0.132 | 0.734 | 0.006 | 1 |  |
| Ag | 0.478 | 0.491 | 0.111 | 0.629 | 0.623 | 1 |



| | Fe | Co | Cu | Cd | Pb | Ag |
|---|---|---|---|---|---|---|
| 1 | 120.36 | 11.214 | 16.78 | 1.08 | 6.074 | 3.52 |
| 2 | 118.74 | 7.932 | 33.32 | 2.56 | 0.8 | 5.14 |
| 3 | 123.98 | 8.82 | 13.28 | 2.48 | 1.142 | 3.54 |

**Fg. (5) Correlation coefficient between elements in basalt rocks (ash digestion)**

## Comparison of the results between digestion methods (wet and ash):

Comparison of heavy metals contents (mean concentrations) between wet and ash digestion in basaltic rock were shown in Figure (6), significant differences in concentrations of all heavy metals concentration were found between wet and ash digestion, where ash digestion showed high concentration of all studied elements.

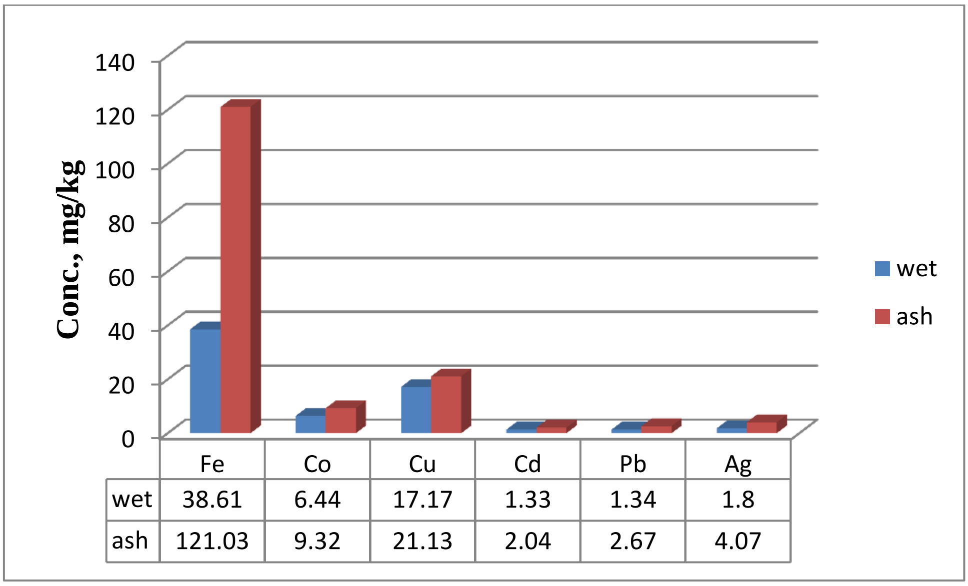


**Fig. (6) Comparison of mean concentrations between wet and ash digestion**

## Conclusion:

For the determination of heavy metals in basalt rocks samples, wet and ash digestion using mineral acids are used for this purpose. Concentrations of heavy metals Fe, Co, Cu, Cd, Pb, and Ag were measured in basaltic igneous rocks in Red Sea Hills. The results showed interspecies variations, the order of heavy metals abundances measured in basalt rocks samples were: Fe >Cu > Co > Ag >Pb>Cd for wet and ash digestion, where levels of heavy metals concentration obtained from ash digestion were shown highest heavy metals contents.

## References:

**Abollino O., Aceto M., Malandrino M., Mentasti E., Sarzanini C. and Petrella F.,** (2002). Heavy Metals in Agricultural Soils from Piedmont, Italy. Distribution, Speciation and Chemometric data Treatment., Chemosphere, 49, p. 545–557.

**Adeloju S.B.,** (1989). Comparison of Some Wet Digestion and DryAshing Methods for Voltammetric Trace Element Analysis, ANALYST. Vol. 114.

**Baker A. D., (1971).** Determination of Copper in Alfalfa, J. AOAC 54, 951

**Gupta V.,** (1999). SPSS for Beginners. VJ. Books Inc. Library of Congress.

**Harrison R. M.** (2007). Principles of Environmental Chemistry. The Royal Society of Chemistry. Cambridge, UK.

**Horowitz J. and Elrick K. A.** (1985). Multielement Analysis of Rocks and Sediments by Wet Digestion and Atomic Absorption Spectroscopy. Varian Atomic Absorption. No. AA-47.

**Mitra S.,** (2003). Sample Preparation Techniques in Analytical Chemistry, John Wiley and Sons, New Jersey.

**Nanos N., and Martin A.R.,** Using a Multi-Scale Geostatistical Method for the Source Identification of Heavy Metals in Soils. Geochemistry – Earth's System Processes; www.intechopen.com.

**Nanos N., and Martin A.R.,** Using a Multi-Scale Geostatistical Method for the Source Identification of Heavy Metals in Soils. Geochemistry – Earth's System Processes; www.intechopen.com.

**Perkin-Elmer Corporation.** (1996). Analytical Methods for Atomic Absorption Spectroscopy, U.S.A