JABAL AL NOOR (MAKKAH) SAUDI-ARABIA, A ROCK OF PRECIOUS METALS

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ABSTRACT: The purpose of study was to assess concentration of some selected heavy metals along with selected alkali and alkaline earth metals in the sample of Jabal-al-Noor Makkah. Various parameters such as pH, EC, TDS, %age organic matter (OM), %age Lime, %age Sesquioxide and %age Silica were also studied by physical and chemical analysis. The alkali and alkaline earth metals like Li^{+} ,Na^{+} ,K^{+} ,Ca^{+2} ,Ba^{+2} were analyzed by flame photometer and transition metals like Cr^{+3} , Mn^{+2} ,Fe^{+2}, Co^{+2}, Ni^{+2},Cu^{+2}, Ag^{+} and Cd^{+2} were determined by Atomic Absorption Spectrometer. X-ray diffraction Analysis was used to asses the presence of precious metals (Au, Pt, and U). Non metals like Cl^{-}, P, and CO_3^{-2}, were also determined by chemical analysis. The observed ranges of various physical parameters were as pH (7.15), EC (2.6mS/cm), % age Organic Matter (1.95), % age Lime (1.46), % age Sesquioxide (50.05) and % age Silica (41.45). The average level of metals was found in the following order :
$Ba^{+2}>Ca^{+2}>Fe^{+2}>Mg^{+2}>Na^{+}>K^{+}>Li^{+}>Cr^{+3}>Ni^{+2}>Co^{+2}>Cu^{+2}>Mn^{+2}>Cd^{+2}>Ag^{+}$.
XRD analysis confirmed that Gold (Au) and Platinum (Pt) are present in alloy form (Au_3Pt) with tetrahedral units.

Key Words*:* Jabal-al-Noor, Precious Metals, Rock, XRD analysis, Heavy Metals, Makkah

1. INTRODUCTION

Jabal-al-Nour (also 'Jabal an Nur' or 'Jabal Nur'), meaning "The Mountain of light ", is a mountain in Saudi Arabia. It houses the Ghar- E- Hira (Cave of Hira). The Cave, the sanctum of the Holy Prophet Muhammad (Peace Be upon Him), the place of His devotions and mediations and the sacred spot where the Holy Quran began to reveal. The Cave is situated on mount *Al- Noor* on way to *Mina* near Makkah and its peak is visible from a great distance. Muhammad (P.B.U.H) had just stepped into the forty first year of his life, when during a night in the month of Ramadan the first 5 verses of the Sura Al- Alaque were revealed to him from GOD through the angel Gabriel.

It is said that this mountain contains precious metals. So the present study was conducted to determine these metals along with some common transition metals and non metals. For this purpose flame photometer was used for the determination of Li^{+} ,Na^{+} ,K^{+} ,Ca^{+2} ,$Ba^{+},^{2}$ and atomic absorption spectrophotometer was used to determined transition elements like Cr^{+3} , Mn^{+2} ,Fe^{+2}, Co^{+2}, Ni^{+2},Cu^{+2},Ag^{+} *and* Cd^{2+} .Precious metals like Pt and Au were determined by X- ray Diffraction (XRD) analysis.

2. EXPERIMENTAL WORK

pH was measured with the pH meter (PHS-3B). It was calibrated by using standard buffer solutions of pH 4.0, pH 7.0, and pH 9.2[1]. Electrical conductivity was measured with the help of digital EC meter (DDS- 11A) and further calculations were done as reported by [2].

2.1 Determination of %age Lime Contents: Percentage lime contents were determined by back titration method with the standards solution of alkali (NaOH). 5ml of sample was taken, 50ml of 0.5N HCl was added into it. Refluxed it for few minutes and filtered and then titrated against 0.5N NaOH with 2-3 ml of phenolphthalein as an indicator. The calculations were done by following formula [3].

$$\% \text{ age Lime} = \frac{[(\text{ml of HCl xN}) - (\text{ml of NaOH xN})]}{\text{Wt of Soil Sample (g)}} \text{X0.05X100}$$

Where
N = Normalities of HCl & NaOH solutions

2.2 Determination of %age Organic Matter: The organic matter in the sample was determined by back titration. 1g of soil sample is taken in the titration flask along with 10ml of 1N Potassium dichromate, 20 ml of sulphuric acid (98.5%) and allowed it to cool for one hour. After adding 150-200ml deionized water, 10ml of orthophosphoric acid and 10-20 drops of barium biphenyl amine sulphate as indicator, titrated it against 0.2N Ammonium Ferrous Sulphate having a sharp green end point **[**4-5**]**. The calculations were done by using the formula.

$$\%\text{age Org. Matter} = \frac{(\text{meq of reagent B/Wt of Soil sample}) \text{ X0.69}}{[(\text{ml of B for blank}) - (\text{ml of B for sample})]}$$
XNX0.69

Wt. of soil sample (g)%age Organic Carbon= %age Organic Matter/ 1.742

Where B = H_2SO_4, (1N), sp.g:1.84

2.3 Determination of TDS: Transferred 10g sample into 100 ml beaker and transferred 30ml of water and refluxed it for 30 minutes. Cooled the sample and TDS was determined by using TDS meter (CD-302) [6]. It was calculated by multiplying EC by factor of 0.64

TDS (mg/L) = EC (us cm^{-1}) x 0.64

2.4 Determination of %age Silica (SiO_2): Taken 10g of sample and added 30ml of aqua regia and refluxed it for 30 minutes. After cooling, filtered with vacuum pump. Dried

the filter paper and residue in oven and ignited the residue on a strong flame for and hour. Cooled the residue and weighed [7].

$$\% \text{ age } SiO_2 = \frac{[\text{mass of ignited residue} - \text{mass of filter paper ash}]}{\text{Wt. of soil Sample}} \times 100$$

2.5 Determination of %age Sesquioxide (Fe_2O_3): Taken 10ml of sample and added 30ml of conc HCl and refluxed for 30minutes. Filtered it and marked upto 100ml in flask. Taken 50ml of extract in beaker and added 2ml of conc HNO_3, boiled the contents to convert Fe^{+2} to Fe^{+3}. Cooled inside desiccators
and weighed accurately [8]. The residue composed of sesquioxide + P_2O_5 + filter paper ash.

$$\%\text{age Sesquioxide} + P_2O_5 = \frac{(S\text{-}B) \times T \times 100}{A \times W}$$

Where
S is the mass of %age Sesquioxide +P_2O_5 +ash
B is the mass of ash
T is volume of extracts
A is extract used for precipitation
W is mass of soil in gm
%age Sesquioxide = % age Sesquioxide + P_2O_5 – %age Sesquioxide in Sample

2.6 Determination of Alkali and Alkaline Earth Metals by Flame Photometer: The amount of alkali and alkaline earth metals (Li^+, K^+, Na^+, Ca^{+2}, Ba^{+2}) in the sample extract was determined by Janway PFP-7 Flame Photometer [9]. Flame emission spectroscopy uses quantitative measurement of the optical emission from excited atoms to determined analyte concentration [10].

2.7 Determination of Heavy metals By Atomic Absorption Spectrophotometer: Metals such as Chromium (Cr), Manganese (Mn), Iron (Fe), Cobalt (Co), Nickel (Ni), Copper (Cu), Cadmium (Cd), Silver (Ag) were determined by Atomic Absorption spectrophotometer [11]. The Atomic Absorption Spectrophotometer is mainly employed for the determination of metals whose resonance lines lie in the ultraviolet and visible region of the spectrum [12].

2.8 Determination of Phosphorous: Determination of Phosphorous was done by back titration, using 0.1N NaOH in excess and it was titrated against 0.1N H_2SO_4 using 0.5 % phenolphthalein as an indicator. Calculations were done as follows

$$\%\text{age Total P in sample} = \frac{(S - B) \times C \times T \times 100}{A \times W}$$

$$= \frac{(20 - 0.58) \times 0.001348 \times 125 \times 100}{50 \times 5}$$

$= 1.30$ Where
S= meq of NaOH used
B = meq of H_2SO_4 used for blank
(Titre used x normality = 5.8x0.1)
T= Volume of total HCl extract
A= extract used for P precipitation
W= mass of soil in gm
%age Total P_2O_5 = % Total P in soil x 2.29
= 1.30x 2.29
=2.97

2.9 Determination of Carbonates (CO_3^{-2}): Determination of Carbonate ions was done by titration. Taken 5ml of sample solution and added 50ml of 0.5N HCl in a beaker boiled for 5 minutes and then cooled. Then filter it. Then added 3 drops of phenolphthalein indicator and titrated against 0.25N NaOH until the colour changes from colorless to persistent pink [14].

$$\text{meq } CO_3^{-2}/100 \text{ gm soil} = \frac{(\text{meq HCl added} - \text{meq NaOH used})}{\text{Mass of sample (g)}} \times 100$$

2.10 Determination of Chloride (Cl^-): The concentration of chloride ion in the sample solution was done by Argentometric titration by using 50ml of sample solution and adjusting the pH to 8.2. The added 1ml K_2CrO_4 as indicator and titrated against 0.05N $AgNO_3$ and noted the readings. Similarly run a blank with the same volume of chlorine free distilled water [15].

$$\text{meq } Cl^-/\text{ litre} = \frac{(\text{ml of titre for S} - \text{ml of titre for B}) \times N \times 1000 \text{ ml}}{\text{of sample solution for titration}}$$

3. RESULTS AND DISCUSSIONS

Table-1: Concentration of Some Different Physical Parameter

Parameters	Values
pH	7.15
EC(mS/cm)	2.6
% lime	1.46
% Organic matter	1.95
TDS	-
% Silica	41.45
% Sesquioxides	50.05

Table-2: Concentration of Metals Obtained By Flame Photometer**:**

Sr. No	Name of Metal	Conc. from granh	Dilution factor	Resultant conc. in mg/10 gm	Concentration in mg/ kg	Concentration in g / kg
1	Lithium	27	10	270	2700	2.7
2	Sodium	71	10	710	7100	7.1
3	Potassium	56	10	560	5600	5.6
4	Calcium	70	100	7000	70000	70
5	Barium	88	100	8800	88000	88

Table-1 shows that the pH & EC of sample are moderate i.e., 7.5 & 2.6 (mS/cm) respectively. The % age of lime is very small but of organic matter is even higher than normal agricultural soils. Table-2 shows that the concentration of Calcium (Ca) & Barium (Ba) are much higher (70 & 88 mg/kg) as compare to the other selected members of group I-A & II-A. Among the selected transition metals the concentration of iron is highest i.e., 17.8 g/kg. All the other selected metals are in fractions.Table-4 shows that among the selected non-metals the concentration of phosphorous is higher i.e., 1.3%.

Table-3: Concentration of Metals obtained By Atomic Absorption Spectrophotometer**:**

Sr. no .	Name of Metal	Con. from graph (ppm)	Dilution factor	Resultant conc. In mg/10 gm	Conc. in mg/ kg	Conc. in g/kg
1	Magnesium	9	100	900	9000	9.00
2	Chromium	31	0	31	310	0.31
3	Manganese	11	0	11	110	0.11
4	Iron	17.8	100	1780	17800	17.8
5	Cobalt	6	0	6	60	0.06
6	Nickel	30	0	30	300	0.30
7	Copper	3	0	3	30	0.03
8	Cadmium	0.168	0	0.168	1.68	0.0017
9	Silver	0.12	0	0.12	1.2	0.0012

Table-4: Concentration of Some Non- metals obtained by Chemical Analysis.

Name	Concentration
Chlorine (Cl)	0.056%
Phosphorous (P)	1.3%
P_2O_5	2.97%
Carbonate (CO_3^{-2})	1.15 meq/ 100g soil

Table-5: XRD data for Gold (Au), Platinum (Pt), and Uranium (U)

Metal	In Sample availability	Crystal System	Density (g/cm^3)	Cell Vol. ($10^6 pm^3$)
Gold	Au_3Pt	Tetrahedral	21.78	59.93
Platinum	Au_3Pt	Tetrahedral	21.78	59.93

The XRD method was adopted to assess the selective precious metals like Gold (Au) and Platinum (Pt). The XRD graph (Fig-1) and XRD data (Fig-2) show that the gold and platinum are Present in alloy form (Au_3Pt) having density 21.78 g/cm^3 and tetrahedral crystal structure. The volume of the unit cell was found out to be 59.93, 10^6 pm^3. The unit cell volume was found out to be 4692.12, 10^6 pm^3 which is much higher than that of alloy of gold and platinum.

4. CONCLUSIONS

The results obtained showed that sample is enriched with different elements and it also has precious metals like Au and Pt. The presence of precious metals like Gold and Platinum was confirmed by X-ray diffraction method. The concentration of different metals lies in the following order.
$Ba^{+2}>Ca^{+2}>Fe^{+2}>Mg^{+2}>Na^{+}>K^{+}>Li^{+}>Cr^{+3}>Ni^{+2}>Co^{+2}>Cu^{+2}>Mn^{+2}>Cd^{+2}>Ag^{+}$.

Additional Information: The XRD graphs (Fig. 1 and Fig 2) and related details have been submitted to the journal additionally.

REFERENCES

1. Jackson, M.L. " *Soil Chemical Analysis*", Prentice Hall of India, Private Limited, New Delhi(1973)
2. Wilcox. L.V. Electrical Conductivity. Amer. *Water Works Assoc. J*., **42** : 775- 776(1950)
3. Black. C.A (ed). *Method of Soil Analysis*. Vol. 1 Am. Soc. Argon. Madison. Wisconsin USA(1965)
4. Walkley, A. Critical examination of rapid method for determining organic matter in soils, effect of variation in digestion conditions and of Inorganic soil constitutuents. *Soil*, pp632-651(1947)
5. G. Saeed Khan and Muhammad Rafiq Government of Pakistan Ministry of Food and Agriculture, Soil Survey of Pakistan, Lahore. Technical Guide for Chemical Analysis of Soil and Water, Bulletin no 14(1980)
6. United States Salinity Laboratory Staff. "Diagnosis and improvement of Saline and Alkali Soils. USDA Hand book No 60". Govt Printing office, Washington D.C(1954)
7. A.O.A.C. Official and Tentative method of Analysis of the Association of Agricultural chemists 7th editon.pp 910 Washington D.C (1950).
8. Muthuval, P.C .Udaya soorian, R Nateson, and P.P.Ramaswami. Introduction to Soil Analysis, Tamil Nadu Agricultural University, Coimbatore 64 1002.(1992)
9. Stanford, S and L. English. Use of Flame Photometer in rapid soil tests for K and Ca. *Agron J*., **4**: pp 446-447.(1949)
10. Gray D, Christian "Analytical Chemistry" John Willey and Sons , 5th Edition.
11. Thomas, D; Cheston D "Department of Chemistry". San Houston University Hunstrille, Texas, 77341, Copyright (2000).
12. Robinson. J.W. Atomic Absorption Spectroscopy. Marcel Dekker Inc, New York, USA.
13. A.O.A.C. Official and Tentative method of Analysis of Association of Agricultural Chemists 7th edition. pp 910. Washington D.C.(1950)
14. Schollenberger C.J., 1945 Determination of carbonates in soil. *Soil Sci.,*.**59**: 57-63(1945).
15. Eaton.F.M.Toxicity and accumulation of chloride and sulfate salts in plants *J Agric* Res., **64** pp 357- 399 (1942).