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Metamorphic Confirmation through Spectroscopic Analysis of Elements and Minerals Present in the Rock Crystals of Velimalai Hills (Western Ghats)

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Abstract:- Rocks and Minerals are inseparable. The natural sources of most of the minerals are Rocks. Due to artificial exploration or natural degradations, the hidden minerals are exposed. The exposed Rocks are in the form of Rock Crystals. The Rock Crystals are subjected to proper investigation to identify the elements and minerals. The area of investigation is Velimalai Hills, which is located at the southern horn of India. Velimalai Hills is the part of Western Ghats. Western Ghats is a long continuous range of Rocks. The Rocks are mainly Metamorphic and Sedimentary in nature. Two thirds of the year, the rocks are exposed to rain. Severe and moderate rain are possible due to the South-West monsoon and North-East monsoon. The natural disintegration or degradation of the Rocks and exposure of minerals are mainly due to rain and flow of high speed water. This flow of water brings minerals from the Rocks to the ground or land. The Velimalai Hills is the Storage spot of many metallic, non-metallic and radioactive elements and minerals. But, No great mineralogical studies or surveys are done so far along this Hill range. Two different samples were taken and the spectroscopic analysis were done. The powdered Rocks samples were subjected to X- ray diffraction, Fourier Transform Infra Red and Electron Dispersive Spectroscopic Studies. The XRD analysis, FTIR analysis and EDS analysis collectively showed the presence various elements and minerals in the samples along with adsorbed water. The presence of various elements and minerals were identified and confined. The unequal presence of different Elements and Minerals in the two samples reveal that the rock system is not identical, regular or isotropic which is a major character of a Metamorphic Rock System.

Index Terms:- XRD;FTIR;SEM;EDS

I. INTRODUCTION

The analysis of physical characteristics and chemical characteristics enables us to identify elements and minerals present in materials. Various spectroscopic methods such as X-Ray Diffraction, X-Ray Fluorescence, Fourier Transform Infra-Red, Near Infra-Red, Transmission Electron Microscope, Scanning Electron Microscope, Energy Dispersive Spectroscopy are nowadays used as effective and alternate tools in the Elemental identification arena.

II. EXPERIMENTAL DETAILS

A. Sample preparation

The Rock Samples were collected from the top of the rock surface. They were brushed, cleaned with water and with distilled water. The cleaned samples were dried in sun light. The rock samples were crushed into small stones. The stones were ground into fine powder using a hand mortar, made of porcelain. Thus finely powdered rocks were taken as samples. The samples were separately collected and designated as Sample A and Sample B.

B. Elemental Identification

X-Ray Powder Diffractometry is a most powerful and established technique for elemental and mineral identification and characterization. The X-ray diffraction(XRD) patterns of the powdered sample were obtained by using Bruker AXS D8 Advance in the range of 3^o to 135^o with CuKα radiation of Wavelength 1.5406A°. The output gave several peaks for each sample and they were analyzed. From the output very promising and prominent values of d(A^o) with corresponding 2ɵ are noted out. The I/Io values are calculated and compared with the experimental values.

The Fourier Transform Infrared Spectroscopy(FTIR) of finely crushed powder of the samples in KBr matrix were obtained in the range of $4000cm^{-1}$ to $400cm^{-1}$ with the FTIR Spectrometer Thermo Nicolet, Avatar 370.

The Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) combination was used to carry out the Quantitative and Qualitative Analysis of the samples by X-ray line scans and mapping. The combination used SEM:JEOL model JSM-6390L and EDS:JEOL Model JED-2300.

Fig.1 **Sample A**

Fig.2 **Sample B**

III. RESULT AND DISCUSSION

A. X-Ray Diffraction Spectrum Analysis

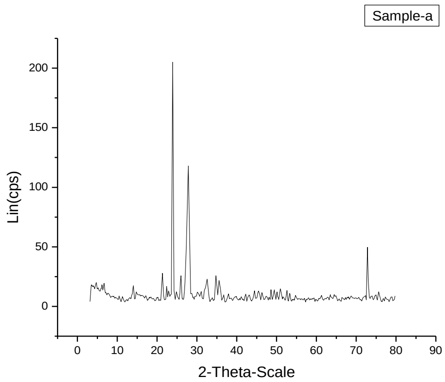

Fig.3 **XRD spectrum of Sample A**

Table-1 **X-ray powder diffraction data**

d(A^{o})	2θ	I/Io (%)
1.0500	39.404	10
1.9300	21.168	05
1.1500	35.909	05
1.6800	24.363	30
1.7872	51.063	142
2.5682	34.907	70
3.2600	27.333	100
1.2838	73.740	27
2.7426	32.624	55
3.1455	28.351	291

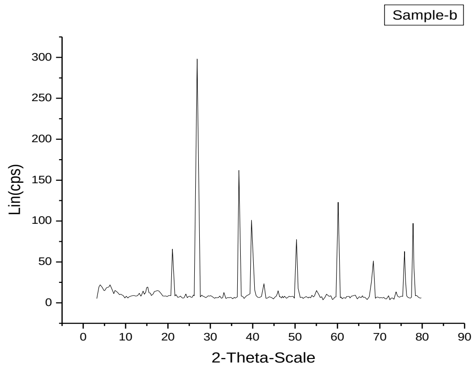

Fig.4 **XRD spectrum of Sample B**

Table-2 **X-ray powder diffraction data**

d(A^o)	2ɵ	I/Io(%)
1.930	21.68	80
1.0500	39.464	40
3.3420	26.652	100
1.8126	50.147	108
1.8174	50.153	110
1.5290	60.503	5
4.1338	21.479	99

From the XRD spectra (Fig.3and 4) the values of interfacial angle of different intersecting planes were calculated and compared with the experimental values. We could see a very good agreement between the two values. The values of I/Io were calculated theoretically and compared with the experimental values. The calculated and the experimental values were very closer as in Table-1and Table-2.

B. FTIR Spectrum Analysis

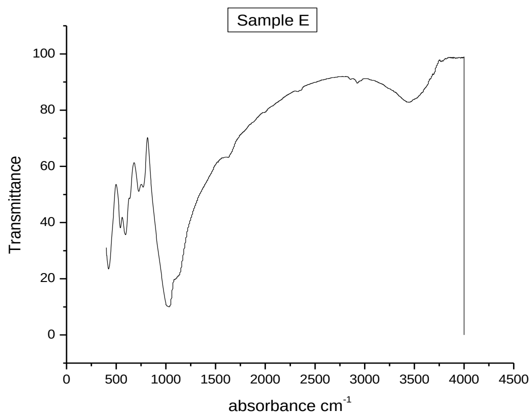

Fig.4 **FTIR spectrum of Sample A**

Table-3 **Absorption Data and Assignments**

Wave Number cm^{-1}	**Visual Intensity**	**Assignment**
423.45	Medium	Si
541.97	Weak	Si-O
593.41	Medium	Al-O
725.95	Medium	Si-O
771.85	Medium	Si-O
1032.96	Strong	Si-Al- Si
1634.05	Strong	OH
2925.08	V-Strong	O-H

Fig.5 **FTIR spectrum of the sample A**

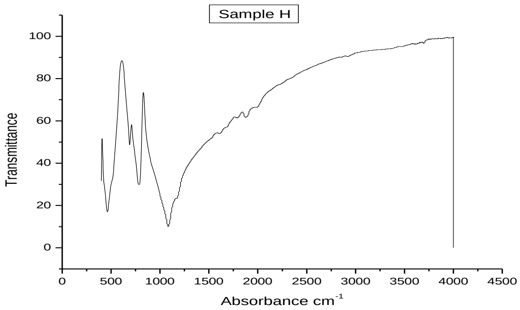

Fig 6 **FTIR spectrum of the sample B**

Table.4 **Absorption Assignments**

Wave number cm^{-1}	Visual Intensity	Assignment
461.55	Strong	Si-O
689.18	Medium	Si-O
784.91	Strong	Si-O
1084.09	Very Strong	Si-O
1878.36	Very Strong	Si-O

From the FTIR spectra(Fig.4 and 5) the peaks were analyzed for their absorption frequencies, visual intensities and their relevant tentative assignments were tabulated as in Table.3 and 4. Most of the peaks of either spectra were present near the absorption wavelength[14] of 400cm^{-1}. This revealed that the minerals present in the sample are metallic and non – volatile.

C. Energy dispersive spectroscopy(EDS)

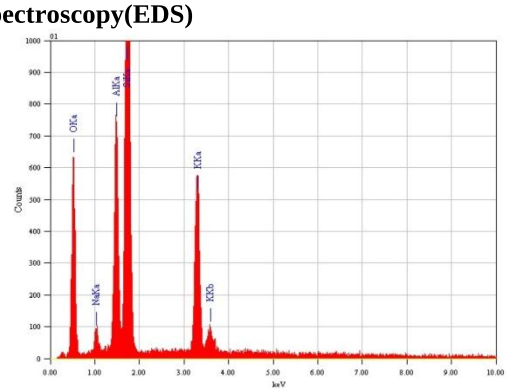

Fig.7 **EDS spectrum of the sample A**

***Table.5*.EDS data of the sample A**

Element	(keV)	Mass%	Atom%
O K	0.525	54.17	68.2
Na K	1.041	2.48	2.18
Al K	1.486	10.22	7.63
Si K	1.739	24.37	17.48
K K	3.312	8.76	4.51
Total		100	100

The EDS spectrum(Fig.7) was taken along with SEM. The spectrum showed all possible elements present in the sample with the % of mass of individual element as in Table.5

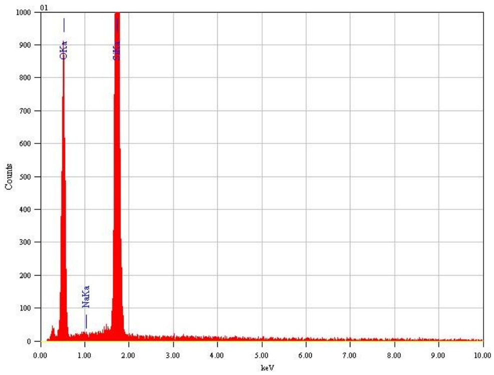

***Fig.8* EDS spectrum of the sample B**

***Table.6*.EDS data of the sample B**

Element	(keV)	Mass%	Atom%
O K	0.525	68.72	79.41
Si K	1.739	31.28	20.59
Total		100	100

IV. RESULTS AND DISCUSSION

The three XRD analysis, FTIR analysis and EDS analysis collectively showed the presence of following elements and minerals in the **sample A** along with adsorbed water.The **XRD** showed that the peak of 2θ = 28.707 and d(A) = 1.4300 of intensity 90 and hkl (200) represented the element Aluminium(Al)[2][8]. The peak of 2θ=24.363 and d(A°) = 1.6800 of intensity 30 and hkl (200) represented the element Potassium(K)[8]. The peak of 2θ = 35.909 and d(A°) = 1.1500 of intensity 05 and hkl (200) represented the element Sodium(Na)[8]. The peak of 2θ = 21.168 and d(A°) = 1.1.9300 of intensity 05 and hkl (220) represented the mineral Silicon(Si)[6] [8]. The peak of 2θ = 39.404 and d(A°)=1.0500 of intensity 10 and hkl (511) also represented the mineral Silicon(Si)[6][8]. The peak of 2θ = 28.351 and d(A°) = 3.1455 of pdf# 831658 represented the mineral Anarthoclase ((Na)[$AlSiO_3O_8$])[8]. The peak of 2θ = 34.707 and d(A°) = 3.1455 of pdf# 760831 represented the mineral Microcline ((K)[$AlSiO_3O_8$])[8].The peak of 2θ = 32.624 and d(A°) = 2.7426 of pdf# 830971 represented the mineral Kaolinite [$Al_4Si_4O_{10}(OH_8)$)][6] [8]. The following Table 7 shows the summary of elements and minerals present in the sample.

***Table 7.* Summary-1**

2θ	Element	Mineral
28.707	Aluminium	
24.363	Potassium	
35.909	Sodium	
21.168	Silicon	
28.351		Anarthoclase
34.707		Microcline
32.624		Kaolinite

The **FTIR** spectroscopy,gave very interesting absorption peaks. The prominent absorption peaks were assigned tentatively and the minerals were identified as follows, The absorption peak 2925.08cm^{-1} corresponded to the mineral Kaolinite [$Al_4Si_4O_{10}(OH_8)$)][5]. The absorption peak 3444cm^{-1} corresponded to the mineral Kaolinite[$Al_4Si_4O_{10}(OH_8)$)][5]. The absorption peak 1032.96cm^{-1} corresponded to the mineral Albite [$NaAlSi_3O_8$][11][4]. The absorption peak 725.95cm^{-1} corresponded to the mineral Dolomite [CaMg(Co_3)[16]. The absorption peak 423.25cm^{-1} corresponded to the mineral Quartz [SiO_2][5][6]. The absorption peak 771.85cm^{-1} corresponded to the mineral Quartz[SiO_2] [5][6]. The absorption peak 541.97 cm^{-1} corresponded to the mineral Sodium Feldspar [K($AlSiO_3O_8$)][18]. The absorption peak 1634.05cm^{-1} corresponded to the absorbed water[4] [5][16]. The following Table 8 shows the summary of minerals present in the sample.

Table 8. Summary-2

Absorption peak cm^{-1}	**Mineral**
2925.08, 3444	Kaolinite
1032.96	Albite
725.95	Dolomite
423.25	Quartz
541.97	Sodium Feldspar

EDS analysis of sample A revealed the presence of Na, Al, Si and K.

The three XRD analysis, FTIR analysis and EDS analysis collectively showed the presence of the following elements and minerals in the **sample B** along with adsorbed water.

The **XRD** spectroscopic analysis revealed the following results. The peak of 2ɵ = 21.68 and d(A) = 1.9300 of intensity 80 and hkl (220) represened the mineral Silicon(Si)[6][8]. The peak of 2ɵ = 39.464 and d(A) = 1.0500 of intensity 40 and hkl (511) represented the mineral Silicon(Si)[10][18]. The peak of 2ɵ = 50.153 and d(A) = 1.8174 of intensity 110 and hkl (110) of pdf#782315 represented the mineral Quartz(SiO_2) [6] [8]. The peak of 2ɵ = 50.147 and d(A°) = 3.3420 of pdf# 850930 represented the mineral SiO_2[8]. The peak of 2ɵ = 50.153 and d(A°) = 1.8174 of pdf# 78235 represented the mineral Quartz[SiO_2][8]. The peak of 2ɵ = 21.479 and d(A°) = 4.1338 of pdf# 850621 represented the mineral Cristobalite [SiO_2] [8]. The following Table 9 shows the summary of elements and minerals present in the sample.

Table.9 **Summary - 3**

2ɵ	**Element**	**Mineral**
21.68	Silicon	
50.153		Quartz
21.479		Cristobalite

The sample was subjected to **FTIR** spectroscopy.. The prominent absorption peaks were assigned tentatively and the minerals were identified as follows,The absorption peak 1084.09cm^{-1} corresponded to white clay[18] [10].The absorption peak 784.91cm^{-1} corresponded to the mineral Quartz[SiO_2][27][15]. The absorption peak 1878.36cm^{-1} corresponded to the mineral Quartz[SiO_2][18]. The absorption peak 461.56 cm^{-1} corresponded to the mineral Quartz[SiO_2][10]. The absorption peak 689.16cm^{-1} corresponded to the mineral Quartz(Si-O)[10] . The following Table 10 shows the summary of elements and minerals present in the sample.

Table.10 **Summary- 4**

Absorption peak cm^{-1}	Element	Mineral
1084.09		white clay
784.91		Quartz
461.56	Silicon	

The **EDS** study of the sample B revealed the presence of the element Si predominantly.

V. CONCLUSION

From the above, we can come to conclusion that the Spectroscopic route of Analysis is highly reliable and precious. No element or minerals is left unidentified and calculated.

Fig-7

Fig.8

The Fig.7 and Fig.8 reveals that the two samples collected are distinct and they represent different places of the area of study. The different proportions of mass percentage of Silicon in the two samples reveal that the mineral assemblage is out of equilibrium due to temperature which results a Metamorphic Rock system[25]. The unequal presence of different Elements and Minerals in the two samples reveal that the rock system is not identical, regular or isotropic which is a major character of a Metamorphic Rock System.

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