Performance of PIXE Technique at Elemental Analysis of Halogens in Charnockite Matrix

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Abstract— Proton Induced X-ray Emission is applied to the determination of F, Cl, Br and I in high grade metamorphic igneous rocks of halogen rich Charnockite composition. In this study, Chlorine, and Bromine halogen contents are found to well correlate and were determined in high grade metamorphic rocks because of within the Si (Li) detector limits of PIXE as 3MeV. But for F and I in Charnockite, the fluorine with low X-ray energy and not detected in the present investigation due to detector limits, because the spectrum is started from element Chlorine. The next, iodine concentration is in the order of ppb range and resolution problem in matrix Charnockite composition by PIXE.

I. INTRODUCTION

The Charnockites near Visakhapatnam form a prominent hill, A 136', and are being extensively quarried for the extension of air port construction. Two kinds of Charnockites are recognized in the area [1]. Coexisting biotite, hastingsite and apatite in a granulitic Charnockite from Eastern Ghats, India, contain large amounts of halogen elements chlorine and fluorine. Halogens generally are enriched in rhyolites in the volcanic rock series while they are depleted in plutonic rock series, especially in granulites Chanockites. Halogens have been reported in some minerals of high grade metamorphic rocks like Charnockites [2]. Precambrian terrains appears to be critical as fluids containing halogens are recognized to have contributed significantly to the evolution of early crust with understanding of the occurrence and distribution of these Charnockite minerals. In the course of a petrochemical study of a suite of granulite facies rocks from Eastern Ghats, Southern India, and a sample with halogen rich minerals have been discovered. The halogen-bearing minerals include biotite, hastingsite and apatite, and this report deals primarily with their occurrence and composition.

The halogens (fluorine, chlorine, bromine and iodine) are key role elements and one of the most important in geochemical analysis of Charnockites [3]. Halogen Elements in geological rock samples are generally low, for fluorine and chlorine, ranging from μg/g or ppm to for bromine and iodine, ng/g or ppb and hence, non-destructive analytical methods like electron micro probe analysis and X-ray fluorescence or present PIXE are hard to apply to those samples for determining halogens except for some extreme cases. With destructive analyses of those solid Charnockite samples for trace halogens, serious analytical problems may occur when samples are decomposed by acid digestion; halogens (especially iodine and bromine) could be partly lost when heated in the atmosphere because of their volatility and samples could be subjected to halogens' contamination because of their ubiquitous presence in the environment.

When we observe the results obtained by PIXE [4] from high grade Charnockites rocks of earth sciences its applicability in the analysis of materials from geological sciences, include rocks, minerals and ores etc. During the discussion of these results it is observed that though the PIXE is giving results of geological materials for their wide range of elements. It has been an argument that some of the elements present in matrix composition of geological samples are purely determined or could not be determined at all by the PIXE. This may be due to various reasons including the materials like high grade metamorphic matrix rock and this challenge it is felt that a proper study needs to be designed and implemented to understand the limitation of the PIXE technique at certain elements of matrix composition like charnockite. For this investigation, high grade metamorphic rock belongs in to Eastern Ghats Visakhapatnam of A.P state, India has been chosen. The important factor for selecting this rock is based on the fact that the Charnockites of this area data are published in various scientific publications and are well investigated.

PIXE can be applied non-destructively for determining elements belongs to halogens in matrix composition. In particular, PIXE has been often used for the determination of trace elements of halogens in rock samples. Among halogens, chlorine, and bromine, can be determined by PIXE while fluorine and iodine is difficult to determine by PIXE for samples containing minor concentrations of fluorine and low concentration (trace) of Iodine. It is clear that the number of halogen data is generally very limited even for geological rocks. Especially, this is the case for Fluorine and iodine, which clearly suggests that halogens especially are among such elements whose contents, cannot be easily determined by PIXE for solid samples such as geological standard rocks.

The elements in the target of Charnockite composition through emission spectrum are identified by the energies of the characteristic X-ray peaks and the intensity of its X-rays [5]. PIXE techniques can provide accurate and precise data when correctly applied to a suitable type of Charnockite geological material. PIXE can be used to analyze geological materials for a continuous range of elements down to levels of a few ppms. PIXE gives higher signal to background ratio as compared to other techniques. Both matrix and trace elements, PIXE spectrum hundred times better sensitive and has higher resolution produce data for intermediate and high Z elements. The excitation and ionization cross-sections of various elements decreases with increasing atomic number in PIXE and PIXE is better at the elements whose Z nearly 50 which are relatively higher characteristic X-ray energies [6]. Although PIXE is superior to because the entire range of elements in a sample can be excited by high-energy protons without contributing a high background to the spectrum.

II.   RELATED WORK

A proton induced X-ray emission (PIXE) method- a new tool in geochemistry has been developed for determination of the concentrations of elements in geological samples. In this procedure proton bombardment is utilized to induce characteristic X-rays in all elements present in samples to be analyzed. The method involves observation and counting of these X-rays thereby providing a quantitative measurement of the concentration of each element (with $Z > 12$) in the samples [7]. Complete analysis can be obtained in about 30 min, only mg amounts of material are required, samples are not destroyed, the concentrations of as many as 75 elements are obtained in one operation and the procedure is fully automated. Concentrations of a few ppm, in some favorable cases 1 ppb, can be quantitatively determined. Advantages and limitations of the method are discussed. Isotope dilution mass spectrometry and neutron activation analysis are applied to the determination of Cl, Br and I in igneous standard rocks. The Cl contents range from 10 to 1000 ppm, Br from 0.05 to 2.5 ppm, and I from 5 to 100 ppb. The halogen contents are found to be well correlated for Cl and Br, but less clearly for I. Halogens generally are enriched in rhyolites in the volcanic rock series while they are depleted in plutonic rock series, especially in granite [8].

The application of PIXE technique for trace element analysis of, geological samples is discussed [9]. PIXE is a nuclear analytical technique, which is very much suitable for quantitative elemental characterization in a wide range of complex materials, especially in the range of middle Z elements. Not only are exploratory investigations of PIXE's capabilities presented but also synopses of studies aimed at answering geological questions [10]. Particle induced X-ray emission analysis (PIXE) [11] have performed on the nuclear microprobe was used to provide quantitative information about the major and trace element content of the samples on a micrometer scale. Ion beam analytical methods, including micro-PIXE has many advantages in the characterization of mineral samples with complex structures. Proton-Induced X-ray Emission (PIXE) [12] technique has been employed for the determination of major, minor and trace constituents of some multi-elemental samples. PIXE analyses have been carried out using a 3 MeV proton beam generated using GUPIX software. In the investigated environmental samples like soils, PIXE technique allowed determination of: S, Cl, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu, Zn, As, Sr, Se, Br, Hg and Pb. The elements identified in the l samples using PIXE were: K, Ca, V, Cr, Mn, Fe, Co, Cu, Ni, Zn, W, Ga, As, Pb, Mo, Rb, In, Rh, Zr, Pd, Nb, Sn and Sb. A comparison of the matrix effects in PIXE analysis of thick and thin targets has been made. PIXE is a sensitive, multi-element analytical technique used for the determination of minor and trace elements in a variety of samples, with sensitivities in the range of mg kg-1. The chemical analysis of geological samples by PIXE [13], results of chemical analysis of powder samples of Japanese rock standards by PIXE were slightly different from certified values by the Geological Survey of Japan because of self-absorption of characteristic X-rays in measurements. Calibration factors were calculated on the basis of the measured values and certified values. The use of these calibration factors enables accurate results to be obtained for small amounts of powder samples of rocks and ore.

III.   MATERIALS AND METHODS

The investigations were carried out with a 3MV accelerator at the ion beam laboratory, Institute of Physics, Bhubaneswar, India. For the measurements, 3MeV protons deflected by a 60° analyzing magnet was used. The energy calibration of the accelerator has already been arranged. With respect to the incident proton beam, samples were placed at the centre of a scattering chamber at an angle of 45°. The X-rays are detected with a Si (Li) detector, which has an area of about 80 square mm and a resolution of 160 eV at 5.9 keV and is located about 15 mm away from the target, outside the vacuum. The detector is mounted at 90° with respect to the proton beam and is connected to an amplifier with a computer controlled. The X-rays pass through a Be window. The Si (Li) detection output is completed to data acquisition system which records the X-

ray spectrum. The spectrum of each sample is recorded for a sufficiently long time so as to ensure good statistics.

To assure the various parameters and reliability of experimental system, in the same experimental conditions, the PIXE spectrum is recorded with NIST certified reference material and the relative concentrations of different elements are estimated using GUPIX software package [14]. The relative concentrations of different elements, thus obtained in the present experiment for the above standard samples are compared with the certified concentrations supplied by NIST [4].

To analyze the spectra utilizing a standard Marquardt non-linear least square fitting procedure, the Guelph PIXE (GUPIX) software package is used. Using this GUPIX software package the X-ray intensities of different elements are converted into the respective concentrations using a standardized technique involving fundamental parameters, pre-determined instrument constants and input parameters such as solid angle, charge collected etc. The reliability of the input parameters also is checked by comparing the concentrations of Yttrium obtained in the present work with the known concentration of Yttrium added to the sample.

IV. RESULTS AND DISCUSSIONS

The PIXE spectrum of the geological samples G1 to G7 locations collected from the interior of the Charnockite rock recorded by Si (Li) detector. The concentrations in ppm of these various elements in each sample were determined using the GUPIX software [14]. These concentrations are presented with errors in Table-1.

Since, the detector sensitive volume ranges from order of 10 mm in diameter, and order of 5 inch thickness depending on the derived application. Better energy resolution of elements at low X-ray energies achieved at smaller diameter detectors and if energies of the X-rays about 20 keV, the thicker detectors have higher detection efficiency. The X-ray enters the cryostat through a thin beryllium window to reach the detector. The beryllium windows of detectors are typically 8-25.4 pm thick. The thickness of the window sets the lower energy limit for photons that can be detected by the detector. The low Z elements can be measured only using the above window thickness if the X-ray of the order of 2 keV energies.

The element F with X-ray energy $K\alpha$=0.677 keV and not detected in present investigation due to above detector limits, but these elements analyses by previous another analytical methods. Actually the analysis is started from element S ($K\alpha$=2.309 keV) but not present in Charnockite composition. The spectrum in the above started from peak of Cl because $K\alpha$= 2.622 keV, since detector limits starts from Cl element and >2 keV. From the element Cl onwards all the elements within the detector limits, depend on characteristic X-rays of elements and all are detected in above spectrum [3] of Chanockite sample.

Therefore, L X-ray energy of fluorine is less than 2keV, not detected by above Si (Li) detector. The detection of Cl element is possible since the L X-ray energy of Cl is 2.66 keV Table-1. The concentration value of the Cl element in igneous rocks in the order of 500-1000ppm, from above Table-1, S.No-1, detected by in this PIXE investigation is around 500ppm in seven samples. But in this investigation, the escape peaks of the Ca and K in the spectrum, overlapped and correct for the exact value in ppm.

The next halogen element bromine contain $K\alpha$= 11.924, $L\alpha$ =1.481 keV energy X-rays and L X-ray energy not traced out due to detector limits <2keV and K X-ray detected by detector without resolution problem. Since Br contain very small in the order of 1-10 ppm in metamorphic igneous rocks, from the Table-1 S.No-13 in seven samples.

The last element in halogen group is iodine, which is containing in igneous rocks but its value is in the order of ppb. Since from above Table-1, the minimum detection limit of PIXE is 1 ppm. So iodine not detected by using present PIXE and also possible if iodine contain more than ppb limits, PIXE not suitable to detect because of $K\alpha$ =28.612 $L\alpha$= 3.938 keV energies. Since the K X-ray of iodine beyond detector limits>20keV X-ray energy and LX-ray energy is same as that of Sc K X-ray which is at 4.09 keV energy and also Ca and Ti are major concentration elements in the Charnockite composition whose peaks are dominated at 4 keV energy X-rays.

The important applications of techniques in geological materials Table-2 such as PIXE, PIGE, XRF, NAA [15] etc. and most of them already successfully implemented in the laboratories. To quantify elements in a sample besides, each method has its proper limitations, eventual complications obtained from compositional characteristics of a sample and the poorest results were obtained in some geological materials. So the obtained results must always be compared using other analytical technique. The elemental concentration of geological samples by using one technique and compared data with other nuclear technique and should be reviewed.

Because, the low K X-ray fluorescence yields are strongly attenuated by the absorption edge of higher atomic number elements present in the target [16], A weakness of PIXE is the general inaccessibility of light elements like F. Due to PIXE have limitation of analyzing elements with Z <11 and Si (Li) detector [17], while complimentary tools, help to fill this gap for elements providing [18] routine major element data down to at least Na. NAA is most favorably used for analysis of halogens, rare earth elements, uranium, and thorium and has applications. For PIGE, the use of protons at 4 MeV energy permits to the analysis of elements, ranging from light elements [19] like Li to up to Zn and is very sensitive for some low Z [20] elements, ranging from Li up to K [21].

Although PIXE is superior to XRF because the entire range of elements in a sample can be excited by high-energy protons without contributing a high background to the spectrum yet both have limitation of analyzing elements with $Z < 11$ due to Si (Li) detector. WDXRF can however, be employed for the detection of 9F. The WDXRF, however, is the better technique [6] for the detection of elements with low energy lines, especially in the range 1-4 keV and when bulk or thick samples must be analyzed. WDXRF with its superior spectral resolution at medium to low energies can provide information on the chemical state in a sample of a number of elements e.g., Br. Trace amounts of three halogens chlorine, bromine and iodine were determined using radio chemical neutron activation analysis (RNAA) [22][8] . Therefore another complementary technique [23] is required to in addition to the PIXE to obtain overall information of halogens presented in Charnockite matrix composition.

V. CONCLUSION AND FUTURE SCOPE

In the analysis of materials from geological sciences, include rocks, minerals and ores etc., PIXE is giving results of geological materials for their wide range of elements. PIXE can be applied non-destructively for determining halogens in high matrix composition like Charnockites. Among halogens, chlorine, and bromine, can be determined by PIXE while fluorine and iodine is difficult to determine by PIXE for samples containing high concentrations of fluorine and low concentration of iodine.

Since the thickness of the window sets the lower energy limit for photons that can be detected by the detector. The X-ray energies of the order of <2 keV energies of low Z elements cannot be measured. Therefore, L X-ray energy of fluorine is less than 2keV, not detected by above Si (Li) detector at PIXE with 3MeV. The spectrum in the above started from peak of Cl because $K\alpha = 2.622$ keV, since detector limits starts from Cl element and >2 keV. From the element Cl onwards, all the elements within the detector limits, depend on characteristic X-rays of elements.

Bromine L X-ray energy not traced out due to detector limits i.e, <2keV and K X-ray detected by detector without resolution problem. , the minimum detection limit of PIXE is 1 ppm. So iodine not detected by using present PIXE and also possible if iodine contain more than ppb limits, PIXE not suitable to detect because of $K\alpha = 28.612$ $L\alpha = 3.938$ keV energies. Since the K X-ray of iodine beyond detector limits>20keV X-ray energy and LX-ray energy is same as that of Sc K X-ray which is at 4.09 keV energy.

A weakness of PIXE is the general inaccessibility of light elements i.e, limitation of analyzing elements with 3MeV PIXE and Si (Li) detector. For PIGE, the use of protons at 4 MeV energy permits to the analysis of elements, ranging from light elements like Fluorine. NAA is most favorably used for analysis of chlorine, bromine and iodine even though present in minute amounts. Because the entire range of elements in a sample can be excited by high-energy protons without contributing a high background to the spectrum in PIXE, When compare to XRF, but both have limitation of analyzing elements with $Z < 11$ due to Si (Li) detector. The WDXRF, however, is the better technique for the detection of elements with low energy lines in the range 1-4 keV and superior spectral resolution at medium to low energies can provide information on the chemical state in a sample of a number of elements.

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Tabl-1; Analytical results of geological samples with all elements (PIXE) [3]

S.NO	Element	G1	G2	G3	G4	G5	G6	G7
1	F	ND	ND	ND	ND	ND	ND	ND
2	Cl	394.1±16.5	399.6±17.1	379±16.5	403.5±19.5	546.9±23.5	383.7±16.7	462.6±20.6
3	K	4080±28.2	4187±25.1	4148±26.1	4246±29.3	6699±40.2	5458±27.1	5393±33.4
4	Ca	2229±25.9	2281±22.8	2637±25.6	2754±28.1	4120±23.5	2544±26.2	3091±32.1
5	Ti	1394±11.6	1271±9.3	1109±9.3	13.67±11.3	1590±13.8	1044±9.1	1510±12.4
6	V	17.92±4.8	23.69±4.1	7.85±4.0	BDL	37.78±6.0	10.79±4.0	11.55±12.4
7	Cr	16.63±2.3	38.53±2.1	15.16±2.1	16.93±2.5	17.71±3.1	9.118±2.1	14.13±2.6
8	Mn	18.62 ±3.9	34.71±3.7	34.31±3.7	33.68±4.1	47.43±5.3	27.36±3.5	27.03±4.5
9	Fe	5200±20.3	6575±21.0	5649±19.8	5838±22.2	7325±26.4	4905±17.7	6238±23.1
10	Ni	10.96±3.7	11.43±3.7	10.06±3.39	8.94±3.92	28.29±5.02	10.11±3.1644	16.5±4.3692
11	Cu	BDL	BDL	6.1±2.7	BDL	BDL	BDL	8.717±3.47
12	Zn	9.147±3.3	18.21±3.5	14.23±3.0	14.41±3.5	11.96. ±4.6	24.73±2.9	4.29±2.0
13	Se	6.3±65±2.5	BDL	BDL	0.9499±1.7	BDL	BDL	BDL
14	Br	4±2.2	12.17±2.93	11.82±2.85	13.47±6.2	10.32±4.031	8.61±2.455	9.08±3.503
15	Rb	48.87±6.0	42.08±6.1	62.73±6.0	34.71±6.2	56.14 ±7.9	34.58±5.3	52.46±7.1
16	Sr	38.5±5.4	28.6±4.6	44.61±4.9	27.65±5.2	38.02±6.5	33.91±4.9	35.53±6.1
17	Y	BDL	12.64±4.5	BDL	18.13±5.0	BDL	BDL	BDL
18	Zr	95.91±9.8	20.86±6.0	23.6±6.1	63.7±8.7	11.44±7.0	12.16±5.0	77.82±9.3
19	Nb	7.035±3.1	9.812±3.9	BDL	6.09±3.29	BDL	BDL	BDL.
21	Mo	BDL	24.34±6.3	BDL	10.84±4.04	BDL	BDL	BDL
22	Ru	BDL	BDL	BDL	BDL	BDL	9.977±3.59	BDL
23	Ag	BDL	12.36±9.0	BDL	BDL	BDL	BDL	BDL
24	I	BDL	BDL	BDL	BDL	BDL	BDL	BDL
25	Pb	32.93±15.6	38.35±17.3	17.68±7.7	28.82±11.2	41.58±14.48	BDL	24.49±12.5

Table -2; Analytical results of complex geological samples with halogen elements (PIXE)

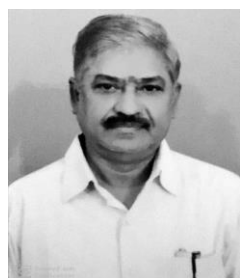| S.NO	Element	G1	G2	G3	G4	G5	G6	G7
1	F	ND	ND	ND	ND	ND	ND	ND
2	Cl	394.1±16.5	399.6±17.1	379±16.5	403.5±19.5	546.9±23.5	383.7±16.7	462.6±20.6
3	Br	4±2.2	12.17±2.93	11.82±2.85	13.47±6.2	10.32±4.031	8.61±2.455	9.08±3.503
4	I	BDL	BDL	BDL	BDL	BDL	BDL	BDL

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Dr. A. V. S. Satyanarayana, has been qualified with higher degrees M. Sc., M. Phil, and Ph.D. Physical Science from Andhra University. He has significant teaching and research contributions in different areas of Physical Sciences, Atomic and Nuclear Physics, Advances of Nuclear Techniques in Elemental Analysis, Material Science, Geo-Chemistry and Geo-Physics. He has 20 years of teaching experience and 15 years of research experience. He has published research papers in reputed national and international journals.

Prof. Mokka Jagannadha Rao has been qualified with higher degrees from India and abroad, working on various aspects of Geological research and having more than three decades of experience. He has significant teaching and research contributions in different earth science specializations including, Mineral Resources, Coastal Geology, Environmental Geology, Remote Sensing, Computer Applications, Geo-Chemistry, Regional Geology and related fields. He has published more than 150 research articles in various national and international journals. He has undertaken 10 funded projects and 2 consultancy projects funded by various funding agencies and industry. He filed a patent entitled "Innovative Model that explains the genesis of Bay of Bengal". Thirty students got their Ph.Ds' under his guidance including students from other disciplines like computer sciences, Geo-Physics, Geo-Engineering and Nuclear Physics. He has been a member of various professional bodies and recipient of many awards and recognitions.

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