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Proton Induced X-ray Emission Studies Using Folded tandem Ion Accelerator (FOTIA) at Bhabha Atomic Research Centre (BARC), Trombay, Mumbai, India

Daisy Joseph*

Abstract

The Folded Tandem Ion Accelerator (FOTIA) at Van De Graaff was used to study PIXE (Particle-induced X-ray Emission) using protons of energy 3-5 MeV. It has been used for a variety of applications from studying intensity ratios, biological samples (blood), rare earth, materials (gold standards), geological samples (gemstones) pottery samples and forensic samples (ink). This article attempts to elucidate the preparation methods of the samples, the detectors used, the analysis and the findings therein for different applications.

Keywords: FOTIA, PIXE, Gemstones, Pottery Samples

1. Introduction

PIXE, as the name implies, is X-ray emission with particles (in this case it is protons as the excitation source). This technique needs to follow certain conditions for obtaining an excellent spectrum. The protons hitting the target should be of low energy, between 3-5MeV and possess a current of 10-20 nA so that it does not burn the samples and obtain reasonable counts per second (1-3). Further, the

* Nuclear Physics Division, Bhabha Atomic Research Centre, Trombay, Mumbai, India; djoseph@barc.gov.in

target should not be very thick so as to avoid matrix absorption and correction. In PIXE, due to charged particle beams, the vacancy production is achieved by two different processes. The primary process is the direct interaction of the incident charged particle with the atomic electron through Coulomb interaction. The advantage of excitation with protons is the low background obtained from the source itself and the almost limitless flux available. Characteristically, in PIXE the cross sections are large for low atomic numbers. PIXE, having a high cross-section for low Z elements, is excellent for biological applications. Figure 1 gives a schematic diagram of the PIXE set up at FOTIA.

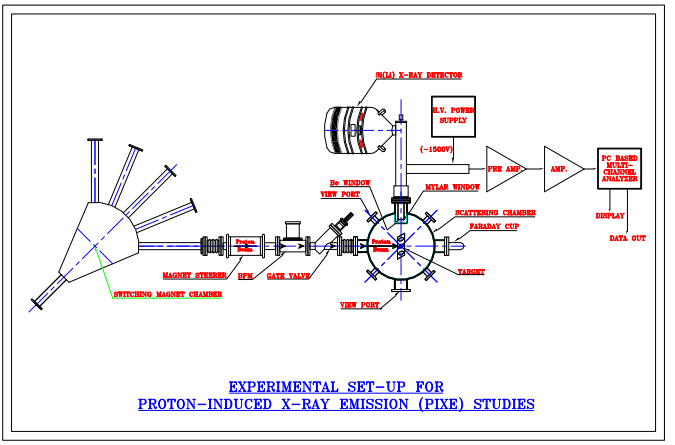

Figure 1: Experimental set up for PIXE

2. Experimental Methods

Generally, all samples are either mounted on the ladder or mixed with internal standard Y for normalising geometrical factors. The samples along with suitable standard and blank of diameter 12 mm are mounted on an aluminium holder, enclosed in a PIXE chamber. A graphite collimated beam (5 mm) of protons of energy 3-5 MeV and current 3-10 nA obtained from the Folded Tandem Ion Accelerator (FOTIA), Bhabha Atomic Research Centre, Trombay, Mumbai, India was directed on the targets placed at an angle of 45 degrees to the beam direction and at an angle of 135 to the detector.

The X-ray spectrometer consisted of a Si (Li) detector of energy resolution 170 eV for 5.9 keV Mn Kα X-rays and of 30 mm 2 × 3 mm size, with a Be window of 1 mil thickness. A thin (3 microns) mylar window separated the PIXE chamber from the detector. We have also used Peltier cooled detector for analysing pottery samples. The PIXE chamber was in the vacuum of the order of 10−6 Torr. The scattering chamber is shown in Fig. 2. The characteristic X-rays passed the Mylar window, 10 cm air gap and Be window of 1 mil thickness of the detector, mounted at an angle of 90^{o} to the beam direction (Fig. 2). All samples were counted for a counting time of 4000 seconds. The X-rays spectra were recorded in a PC-based MCA for further computations.

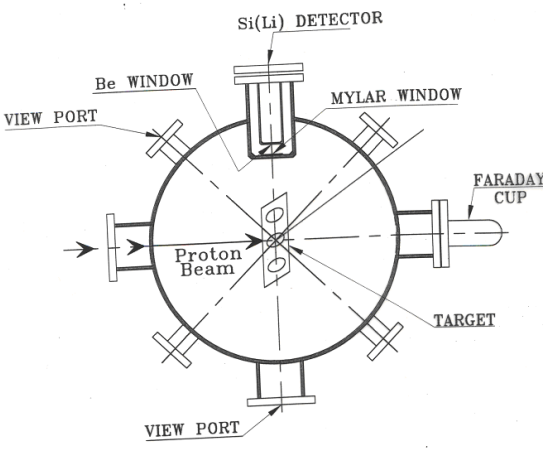

Figure 2: Scattering Chamber for PIXE

3. Application of PIXE in Forensic Science

Proton Induced X-ray Emission (PIXE) was used for elemental characterisation of offset printing ink tagged with rare-earth tangents (4-5). Tagging of offset printing ink with suitable taggant(s) is a unique method to come out with definitive inferences on the detection of forgery in printed documents. The offset printing ink was tagged with rare-earth (La, Pr, Nd, Sm, Eu and Gd) thenoyltrifluoroacetonate chelates at about 1000-ppm level

for each element separately. Small aliquots (approximately 20 mg) of tagged inks were coated on paper supports in the form of small circles having diameter 10−15 mm each and then analysed. A proton beam of energy 4 MeV was used to excite the samples. The PIXE analysis showed well-resolved rare-earth L X-rays rare earth. Fig. 3 (a) represents the PIXE spectrum of ink sample tagged with a mixture of rare-earth chelates while PIXE spectrum of ink sample without any rare-earth chelates (blank) is depicted in Fig. 3 (b). On comparing the spectrum, satisfactory results to identify and quantify the taggants were achieved.

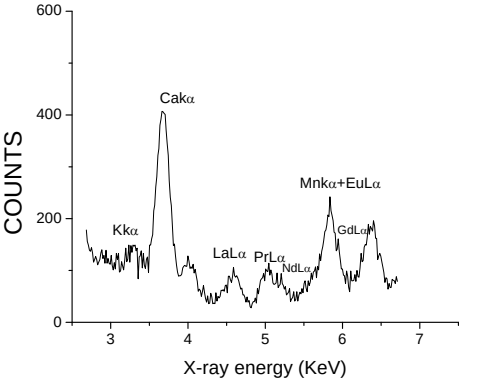

Figure 3a. PIXE Spectrum of Ink Sample Tagged

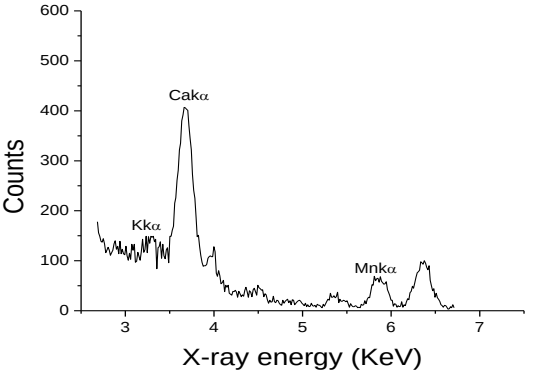

Figure 3b. PIXE Spectrum of Ink Sample without Any Rare-Earth Mixture

4. Application of PIXE in Biosciences

PIXE was used to study lead levels in blood samples of children from Dharavi slum areas (1-2). Pb was found to be high even in normal children due to vehicular exhaust containing lead in petrol. PIXE spectrum of a Pb poisoned child is presented in Figure 4.

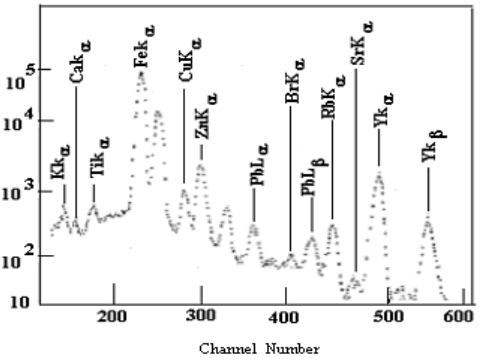

Figure 4: PIXE Spectrum of the Blood of a Lead Poisoned Child

5. Application of PIXE in Geology

Proton Induced X-ray Emission (PIXE) using a Peltier cooled X-ray detector was used to determine concentrations of 10 elements in 20 clay pottery samples collected from excavated Buddhist sites of four districts of Andhra Pradesh, India (Ref 6). Sample mass of 400 mg was mixed with cellulose (binder) in 1:1 ratio along with yttrium oxide (1200 ppm of Y). The International Atomic Energy Agency (IAEA) certified reference material (CRM) SL-3 (Lake Sediment) was used as a multielement comparator. Samples along with the multielement comparator were irradiated in different batches. The control samples, CRMs SL-1 and Soil-7 were prepared and irradiated along with multielement comparator in a similar way to that of the samples. Soil samples from corresponding sites with known origin were analysed for the validation of provenance methodology adopted in this study. For determining the accuracy of the PIXE method, two certified reference materials (CRMs) namely SL-1 and Soil-7 obtained from IAEA were analysed. Using PIXE, concentrations of seven elements namely K, Ca, Ti, V, Cr,

Mn, Fe, Rb, and Zr were determined. The results of PIXE were found to be in good agreement with the certified/information values. Fig. 5 shows the PIXE spectrum of a pottery sample.

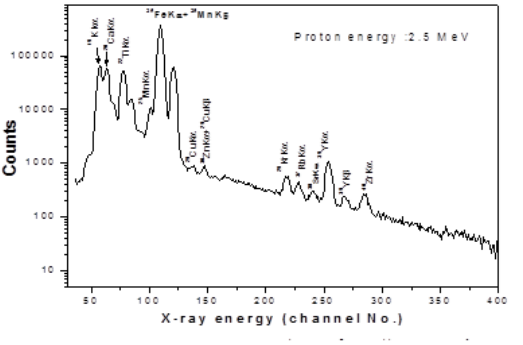

Figure 5: PIXE X-Ray Spectrum of a Pottery Sample

5.1 Analysis of Gemstones (External PIXE)

Gemstones such as Labradorite Feldspar, Moonstone Feldspar, Almandine Garnet, Tsavorite Garnet, Apatite, Natural Spinel, Natural Zircon, Spessartine Garnet, Natural Ruby (lead filled), Natural Ruby were characterised for their elemental profile to see the differences in composition besides the main matrix differences (7). Elements such as Ca, Ti, V, Cr, Mn, Fe, Cu, Zn, Sr, Y, and Zr were detected. Fig 6 shows a PIXE spectrum of a gemstone.

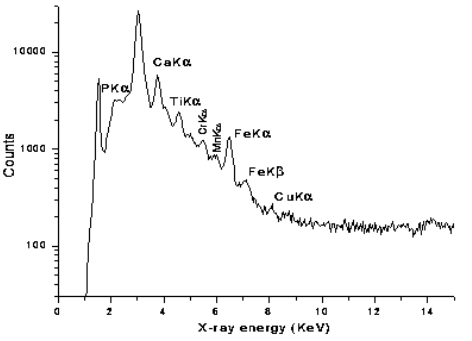

Figure 6: PIXE Spectrum of a Gemstone

6. Conclusion

It is seen that a large variety of applications in Forensic Science, BioSciences and Geology can be studied using PIXE. FOTIA can provide the desired beam. Samples in liquid, solid and metals can be analysed with no damage to the samples making it a novel technique.

Acknowledgements

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