PIXE (Proton Induced X-Ray Emission): determining concentration of samples

A. Rodriguez Manso, M. McCarthy, Y. Pajouhafsar, A.B. McIntosh, K. Hagel,
A. Jedele, A. Zarrella, A. Wakhle, and S.J. Yennello

Introduction

We used Proton Induced X-ray Emission (PIXE) [1] as an analysis technique to determine the composition of samples, in particular, its constituents and concentrations. Each sample is bombarded with protons (or alphas), inducing characteristic x-rays that serve as "fingerprints" for each element. Therefore, the x-rays produced can offer insight to the elemental composition of samples. This process is shown in Fig. 1.
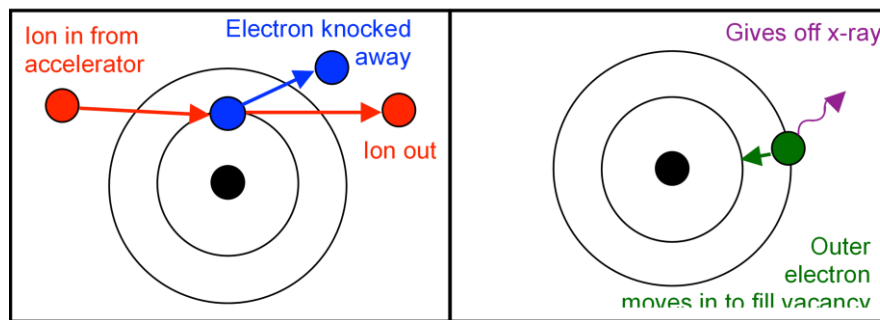

FIG. 1. Induced X-Ray Process. The left panel shows the bombardment of a lower energy level electron, and the right panel shows the displacement of a higher energy level electron and the induced x-ray.

The proton beam is produced and accelerated by the K150 at the Cyclotron Institute at Texas A&M University (TAMU). The setup includes a PX2T-CR Power Supply and Amplifier for the XR-100/CR SiPIN and a Mesytec ADC. The peaks of the spectrum are analyzed using GUPIXwin software tool [2] to determine the concentration of the known elements of each specific sample. The goals of this work are to implement a PIXE experimental set up at the Cyclotron Institute and to determine the concentration of thin films, given by our Chemical Engineering Department.

Experimental setup and measurement

The K150 delivered a 3.6 MeV proton beam over a three-day run. The intensity of the beam ranged from 0.5 - 3 nA. The detector setup consisted of a XR-100T/CR SiPIN detector and preamplifier, connected to its corresponding power-supply and shaping-amplifier. The experimental set up included the detector, a faraday cup, and a target ladder mechanism. These were placed in a vacuum chamber, as shown in Fig. 2, where the bombardment of samples with protons took place. The five-positioned target ladder mechanism, connected to a rod that goes through a slit, enabled to changes the samples within the chamber without breaking vacuum. Each run was approximately 15 minutes long. The target ladder had to be changed out frequently to test different samples and standards. After each target ladder change, an electron suppressed faraday cup was used to measure the beam. When the beam hits the cup, the carbon

backing's electrons are induced and create a signal. This signal gives a measurement of the beam. Throughout the run, there are fluctuations of the beam, and the faraday cup allowed us to precisely measure and record the beam current periodically. Faraday cup measurements lasted around 2 minutes.
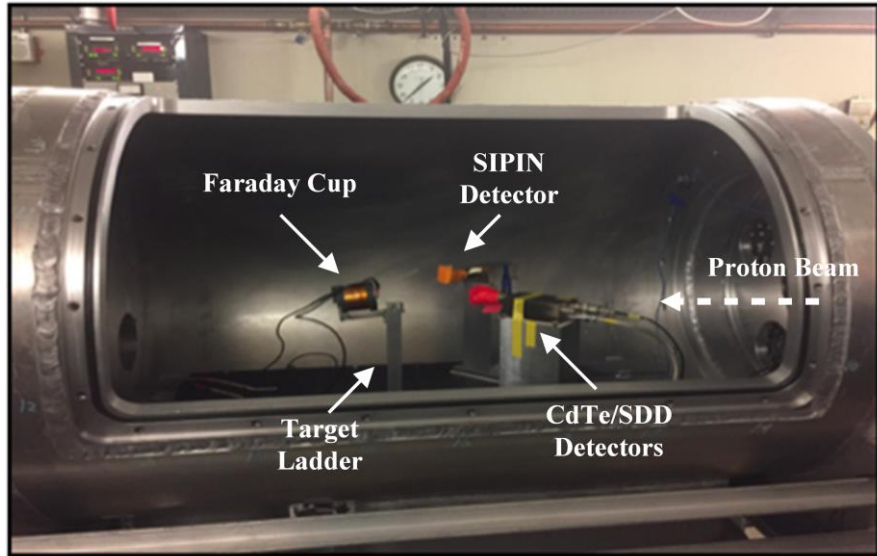

FIG. 2. Vacuum chamber where the experiment was conducted. The CdTe/SDD detectors shown, were used to validate results from the SiPIN detector, as well as the faraday cup. The white dashed line indicates the beam path through the vacuum chamber.

For the calibration of the SiPIN detector, 4 standards were used: CsBr, KCl, NaCl and InS. Fig. 3 corresponds to the spectra of the CsBr standard as an example. The x-ray emission lines are extracted from [3]. With reference to known energy peaks, centroid positions (channels) equivalences were found.
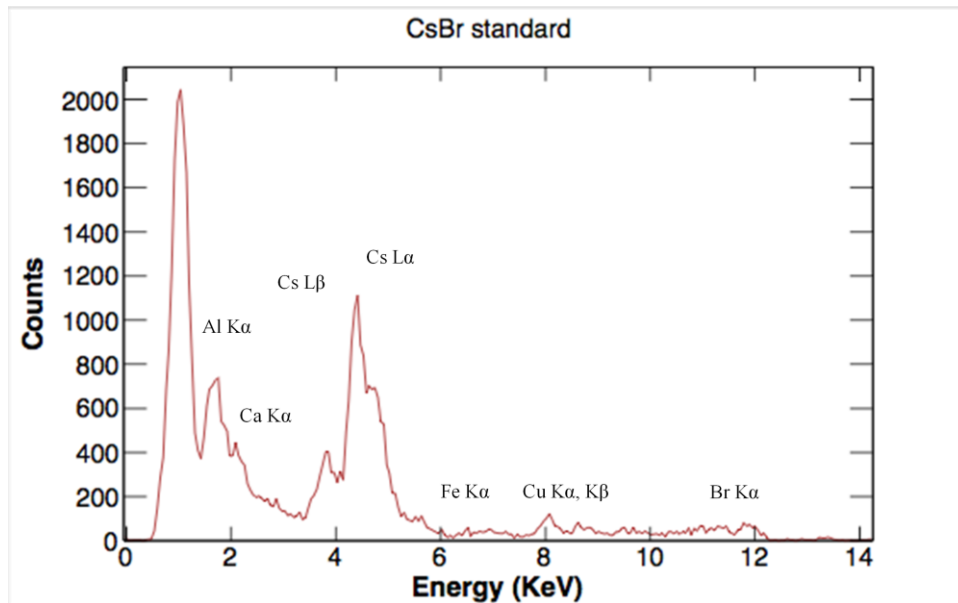

FIG. 3. CsBr standard which noticeable elements include Al, Cs, Ca, Fe, Cu and Br.

The selected standards provided a wide range of energies to form a linear regression by plotting against the energy value, creating a calibration curve, shown in Fig. 4.
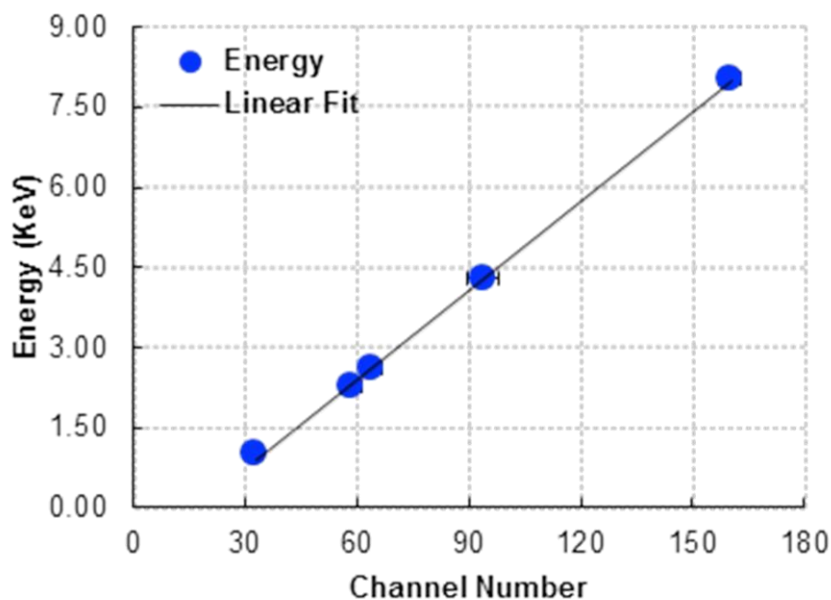

FIG. 4. SIPIN detector calibration curve. Various peaks from the CsBr, KCl, InS and NaCl standards were used.

Results

The samples given by the Chemical Engineering department were poly-dialyldimethyl-ammonium-chloride (a.k.a PDAC) and poly-styrene-sulfonate (a.k.a. PSS) that were treated either with KBr or NaCl. Fig. 5 shows the spectra of one of the KBr and NaCl treated polymers, in the left and right panels, respectively. Additional elements were found in the analysis such as S, Al, Cu, Ca and Fe, the last 4 from the target ladder.

The concentrations of the samples were found using the GUPIXwin software as analysis tool. The software allows the user to enter certain parameters such as beam charge, H-value standardization, starting channel, and the elements contained in the sample, all of which can be tuned to obtain concentration values for the samples, using the standards. The concentrations are calculated using Eq. (1) as follows:

$$Concentration = \frac{I_Z}{I_{Z,0} * Eff * H * Q} \qquad (1)$$

where *Eff* is the efficiency of the detector at a particular energy, Iz is the x-ray intensity, $I_{z,o}$ is the theoretical x-ray intensity, Q is the amount of beam charge hitting the sample and H is a ratio of measured to computed x-rays.
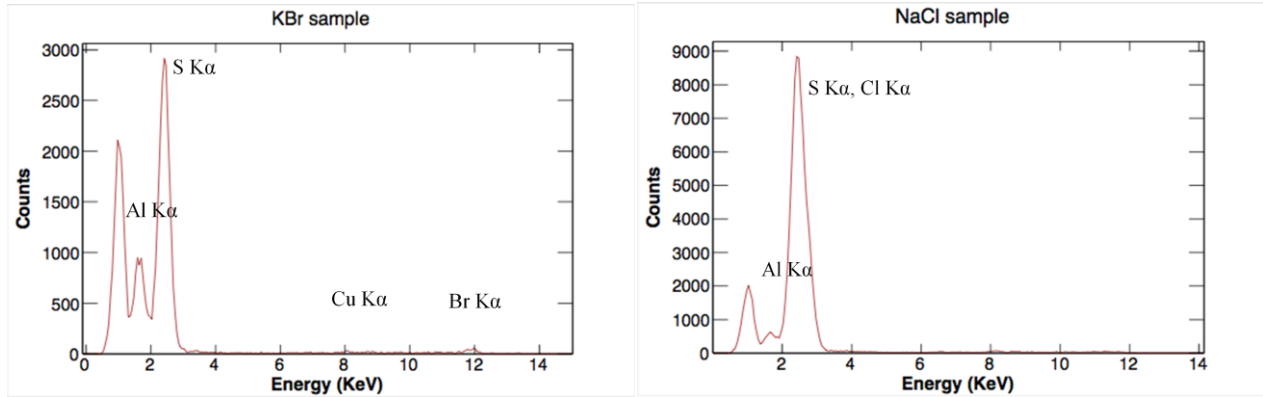

FIG. 5. KBr and NaCl samples in the left and right panels respectively. Both the KBr and the NaCl samples consistently showed a considerable high concentration of S.

With GUPIXwin we analyzed 9 KBr samples and 5 NaCl samples. We found K, Br and Cl concentrations as well as high concentrations of S. The preliminary results are shown in Fig. 6 left and right panels for the KBr and NaCl samples, respectively. The elements found in the samples are consistent with the expectations from the Chemical Engineering Department: the concentration of K is the lowest of all and the appearance of high concentrations of S coming from the sample's production process. The work is still in progress.
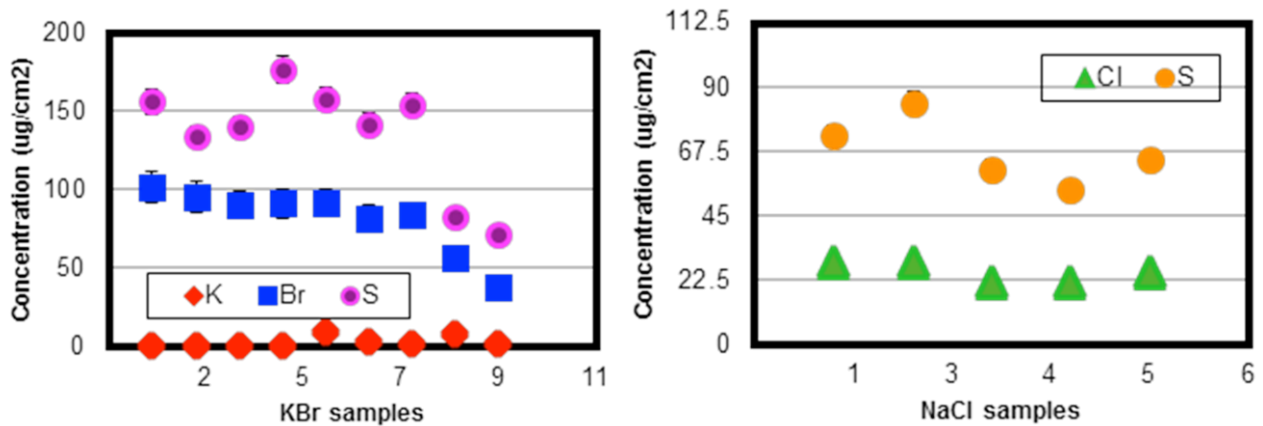

FIG. 6. KBr (left panel) and NaCl (right panel) concentrations. To find the concentration of the samples, the parameters of the standards were calculated and then applied to the samples in GUPIXwin. The 10% error accounts for the uncertainty in the beam current calculation.

In conclusion, the implementation of the PIXE experiment with the K150 cyclotron proton beam was successful. Not only can a spectra's composition be determined through PIXE, but also the concentrations can be calculated.

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[2] J.A. Maxwell, J.L. Campbell, and W.J. Teesdale, Nucl. Instrum. Methods Phys. Res. **B43**, 218 (1989).
[3] J.A. Bearden, *X-Ray Wavelengths*, XRay Emission Lines, Review of Physics, Jan.1967.