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RESEARCH ARTICLE

ELEMENTAL ANALYSIS OF AEROSOL SAMPLES USING PIXE TECHNIQUE

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Abstract

In this work a complex study of the capabilities Particle Induced X-Ray emission (PIXE) technique for the determination of minor constituents of aerosol samples has been done. The PIXE experiments were carried out at Cyclotron at Department of Physics, Panjab University Chandigarh using ~2.7 MeV proton beam. The X-rays were detected with the help of low energy HPGE detector. Total fifteen samples were collected from various locations in Chandigarh. The minor elements identified in the aerosol samples were S, Cl, K, Ca, Ti, Cr, Mn, Fe, Ni, Zn, V, Br and Pb. The data analysis was done using GUPIX software to extract the quantity of the trace elements.

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Introduction:-

Air pollution is one of the major concern around the world. Apart from trace gases, the atmosphere contains a variety of liquids and solids that exist as dispersed phases in air. They are collectively called aerosols. Aerosols result from both natural and anthropogenic sources. Natural sources include soil particles from ground surfaces, gases from volcanoes, forest fires, biological respiration, whereas anthropogenic sources are actually man made sources and include gases from industries, automobiles, refuse burning etc. Since aerosols carry most of the toxic metals, acids and nitrates of the atmosphere, dry or wet aerosol deposition may produce soil and water contamination and damages to buildings and vegetation [2]. In addition, aerosol inhalation by human beings and animals may produce deleterious health effects. Particulates with aerodynamic diameter 2.5 to 10 μm are referred to as coarse particles and the particles less than 2.5 μm as fine particles. These particles are generated due to industrial and vehicular activities and responsible for respiratory and cardiovascular problems as these particles can reach deeply into lungs. It is required to study the pollutant elemental levels which can be done by PIXE technique.

This method is based on the fact that the bombardment of the sample charged particles causes the ionization of the atomic inner shells followed by the subsequent of the characteristic x-rays. When the x-rays spectrum is detected by the high resolution detector, the well-known Z dependence of the x-rays energies as well as the intensities of the individual x-ray lines, allow a straight forward determination of elements present in the target. The properties of PIXE can be summarized as high sensitivity in small samples, high speed, surface analysis, genuinely multi-elemental and quantitative, partially non-destructive, possible to combine simultaneously with other ion beam techniques and microprobes. Negi et al⁶. have reported the urban aerosol composition for both major and trace elements in 4 cities of India i.e. Bombay, Bangalore, Nagpur and Jaipur. Bandhu et al⁷. have reported the elemental composition of air pollution in Chandigarh city in the yr.2000. In the present work, the elemental composition of aerosol samples has been done from the city Chandigarh in India which has been determined by PIXE technique.

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Experimental Set Up :**1. Sample Collection:-**

Samples were collected from four different sites of Chandigarh, Physics department, Panjab University (low traffic intensity and a relatively cleaner zone); Gate No. 2, Panjab University; Inter State Bus Terminus (ISBT) in sector 17; Inter State Bus Terminus (ISBT) in sector 43. At all the sites, sampler was placed at a height of about 8-9 feet from the ground level. The sampling period for all these samples was related to be the most acute one, viz. 10:00 hrs to 18:00 hrs. Samples were collected on 37mm Nucleopore polycarbonate filter paper with the help of model 200 Personal Environmental Monitor PEM. It has a single stage impactor with an after filter. The impactor stage removes particles of aerodynamics diameter greater than $10\mu\text{m}$ (PM_{10}) are collected on the after filter. The particles on the impactor are discarded and the particles on the after filter are analyzed.

Cyclotron:-

The cyclotron is a device for accelerating charged particles to high energies. The particles are held to a spiral trajectory by a static magnetic field and accelerated by a varying (radio frequency) electric field. Cyclotrons accelerate charged particle beams using a high frequency alternating voltage which is applied between two D shaped electrodes (also called Dees) [4]. These circular Dees are lying in a horizontal plane with a small gap separating them. A source of ions is placed in the middle point of the gap between the dees.

But in this we use single Dee cyclotron with a variable frequency in the range of 10-20 MHz. Single Dee placed inside a vacuum chamber between the poles of an electromagnet.

Characteristics of Cyclotron:

1. Main Magnet: It has pole pieces of 27" diameter with 6" between the pole pieces fixed to the horizontal base of the yoke. It provides not only the magnetic field but also maintain the vacuum. It has four current carrying coils, two on each side, each carrying a maximum current of 100 Ampere at 100 V.
2. The Oscillator: A 10' long and 26" outer cavity with Dee stem of 3" diameter forms the tank circuit of the oscillator tube, which is coupled to the tube by filament leads through coupling cylinder of 10" diameter.
3. Ion Source: Ions are produced in the center of the cyclotron by employing a biased hot filament. The thorium Tungsten filament is placed inside a ceramic boat. To generate ionization of hydrogen, the filament is at (-) potential w.r.t ground and heated to white hot and showered with hydrogen gas, electrons emitted from the filament ionize the hydrogen gas and the ions are pulled out by an electric field generating ion currents of the order of mA.
4. The Deflector: It deflects the ion beam out of the orbit with a provision of max. DC voltage of about 70kV. The blade of deflector is made of Cu with the oil cooling arrangement and the septum is made of very thin tungsten plate.
5. Steering Magnet: This centralizes the proton beam.
6. Analyzer Magnet: It turn the beam to an angle 45° and filter the unwanted particles.

Scattering Chamber:

It encloses the evacuated area where the ion beam strikes the target. It is made up of steel provided with windows and is connected to the beam lines by means of a gate valve so that the vacuum in the beam line and scattering chamber could be maintained separately. The targets are placed at 45 degree to the beam direction. A Faraday cup is housed behind the target at the end of the chamber window. The thin targets are mounted on the target ladder placed at the center of the chamber. The HPGe detector is placed at 45 degree to the beam direction to collect the characteristic X-rays of the target.

Hpgc Detector:

In order to detect x-rays we used low energy high pure Ge Detector. This detector fabricated mainly in two configurations.

1. Planar
2. Co-axial.

Here we use coaxial configuration.

HPGe detectors commonly use Li Diffusion to make an n^+ ohmic contact and boron implantation to make a p^+ contact [3]. Co-axial detectors with a central n^+ contact are referred to as n type detector while p type detectors have p^+ central contact. The thickness of these contacts represents a dead layer around the surface of the crystal. Typical

dead layer thickness are several hundred of micrometers for a Li diffusion layer and a few tenths of micrometer for a Boron implantation layer.

HPGe must be cooled to liquid nitrogen temperature. At higher temperature, the electrons can easily cross the band gap in the crystal and reach the conduction band where they are free to respond to the electric field, producing too much electronic noise. Cooling to liquid nitrogen temperature (77K) reduces thermal excitations of valance electrons [5]. HPGe detectors is supplied along with its preamplifier fitted on its top of a liquid nitrogen vessel.

Data analysis:

GUPIX

The Guelph PIXE code GUPIX was employed for data analysis .The quantitative estimation was done using thin target option of GUPIX code. The GUPIX software utilizes the fundamental parameter method for quantitative analysis [1]. For a known experimental geometry the sample composition can be calculated from the measured intensities of the X-ray lines using known physical parameters like X –ray cross section, mass attenuation coefficient and fluorescence yields.

GUPIX code uses H-value standardization method for the calibration of experimental set up. The spectrum of standard target was processed through GUPIX, by inserting a reasonable guess at the value of H on the set up screen and arrived at a concentration which was wrong, but whose ratio to the true concentration then provided the factor needed to normalize the guesstimate value of H to get the real H-value. This software determines the intensities of characteristic X-ray peaks in a PIXE spectrum by fitting a theoretical spectrum to the measured spectrum using a non linear least – square techniques. The theoretical spectrum is generated by using data base of K,L and M X-ray energies ,fluorescence and coster –kronig probabilities and relative X-ray intensities.

Results And Discussion:-

Caliberation of Experimental Set up:

We used nickel as standard element for energy calibration .It was irradiated with proton beam. The two peaks in the spectra are shown below



Figure1:- Calibrated Nickel Spectra.

The two peaks were calibrated with energy $k\alpha = 7.46952\text{keV}$ and $k\beta = 8.26466\text{keV}$.

Table 1:- Concentration (ng/cm^2) of the elements present in the different samples.

Elements	Mean concentration A2(ng/cm^2)	Mean concentration A4(ng/cm^2)	Mean concentration A5(ng/cm^2)	Mean concentration C1(ng/cm^2)
S	7024.275	2632.275	4208.225	8514.805
Cl	804.875	529.225	690.975	1246.879
K	1330.625	933.875	1745.400	4080.000
Ca	2012.075	1494.600	3067.975	6433.600
Ti	113.425	125.225	202.800	613.201
V	4.175	1.550	5.925	7.301
Cr	118.725	104.970	80.325	212.712
Mn	67.175	29.450	56.800	137.825
Fe	1087.375	998.900	2145.25	5735.500
Ni	34.350	21.600	22.275	43.233
Zn	537.200	200.675	189.875	865.510
Br	25.575	14.300	46.025	26.200
Pb	112.325	11.200	137.075	12.700

Where A2 samples from sector -17 (ISBT)
 A4 samples from Cyclotron Physics Department
 A5 samples from Gate no.-2 PU
 C1 sample from Sector – 43(ISBT)

The X-ray spectra for different samples are shown in figures 2-5. It is found that elements K, Ca, Ti, V, Mn, Fe, Zn and Br are mainly soil related elements. The presence of Zn and Br in soil may be due to vehicular traffic and refuse burning. The presence of Cl is due to wood combustion or coal combustion[3]. The presence of Ti and S is due to the road dust. Cr and Ni shows the contamination of soil due to oil combustion and V may be due to the wood combustion and refuse burning in the nearby areas.

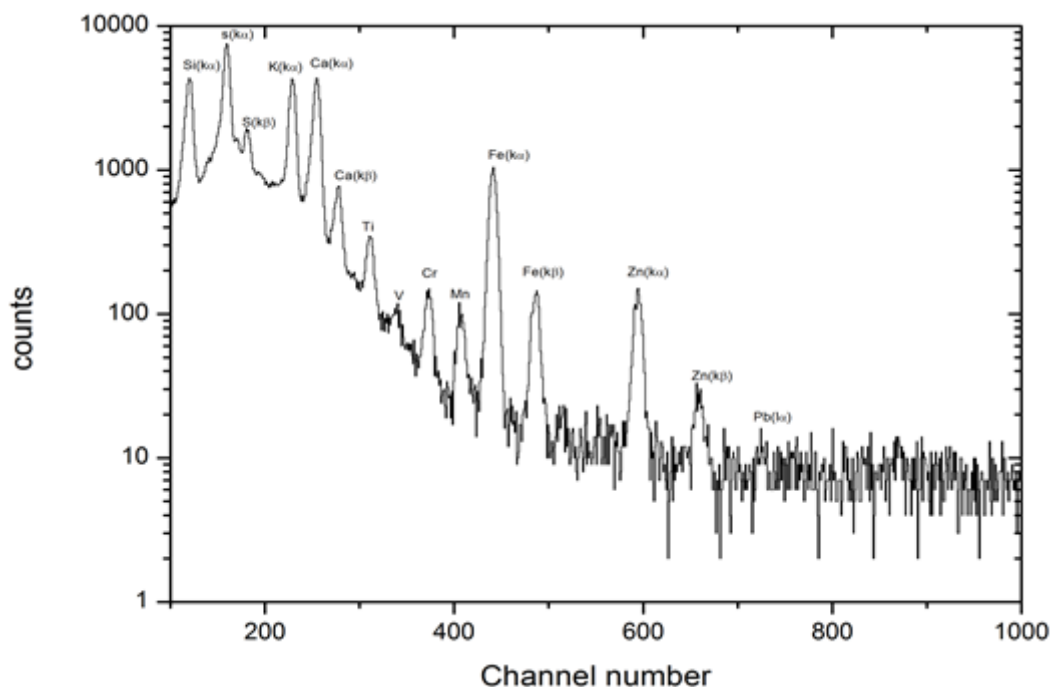


Figure 2:- X-ray spectrum of aerosol sample from Sec-17(ISBT).

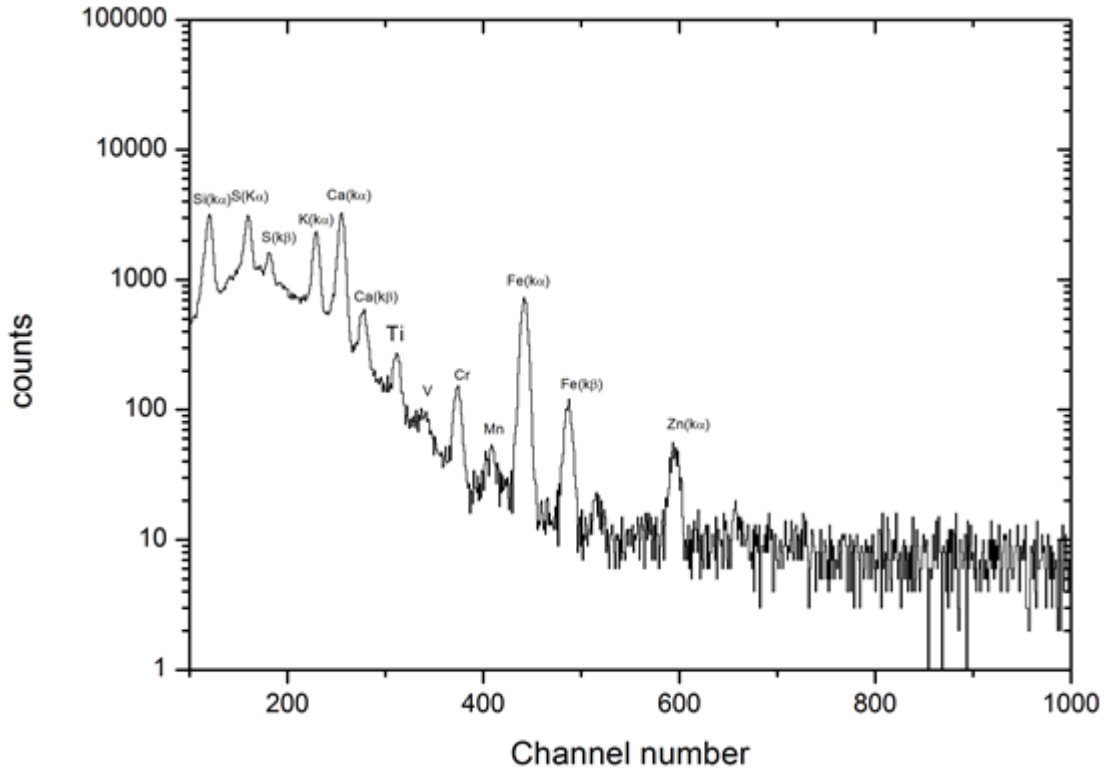


Figure 3:- X-ray spectrum of aerosol sample from Cyclotron Physics Department.

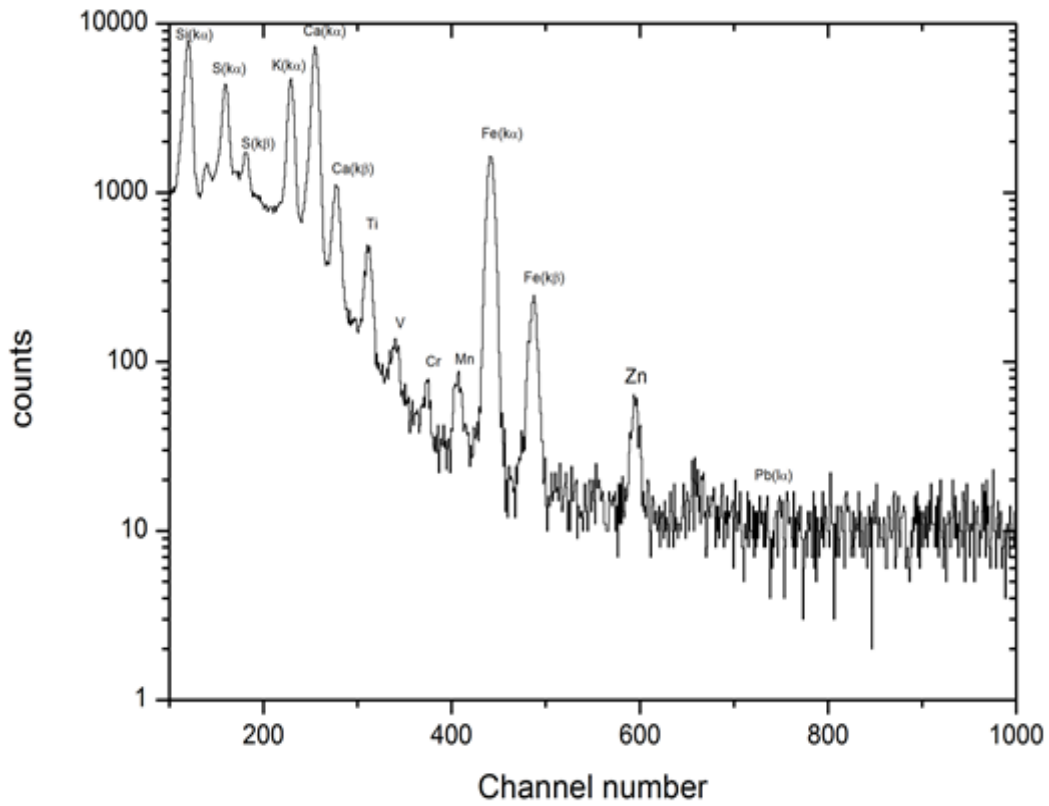


Figure 4:- X-ray spectrum of aerosol sample from Gate no.-2 PU.

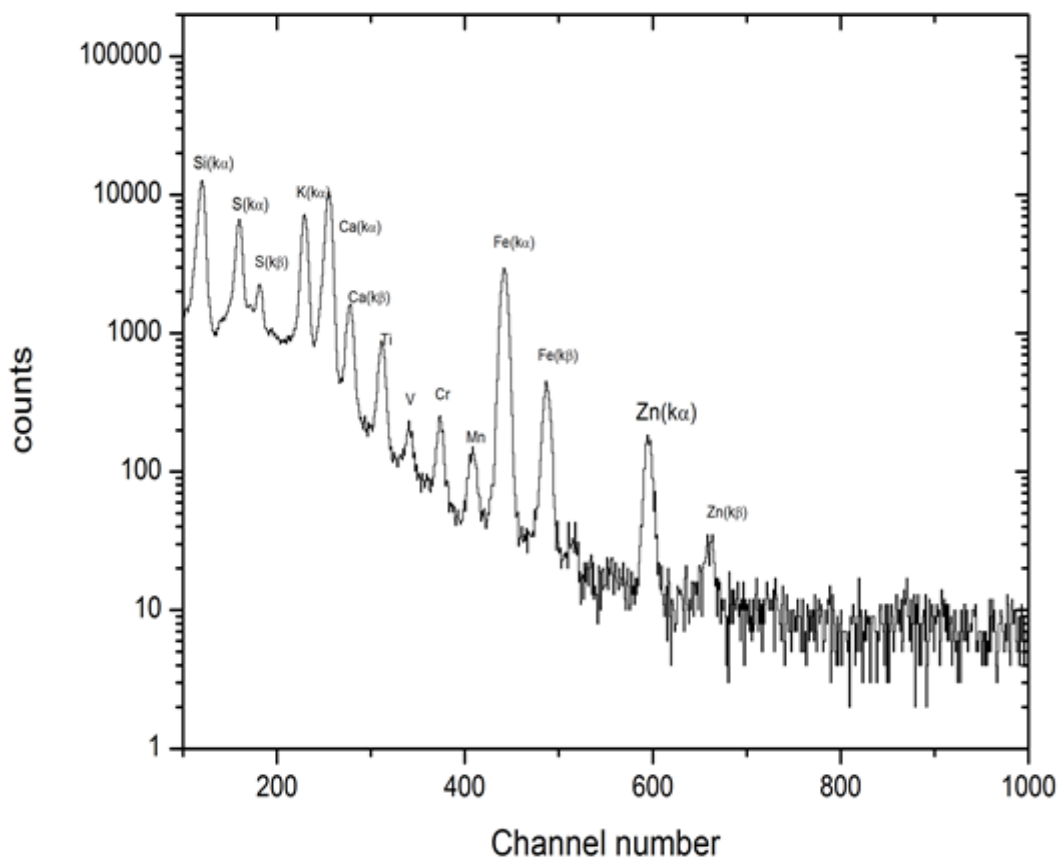


Figure 5:- X-ray spectrum of aerosol sample from Sector – 43 (ISBT).

From the Concentration levels one can say that the concentration levels at Sector-17(ISBT) , Sector- 43(ISBT) and Gate no.-2 PU are higher than those found from Physics Department in Panjab University.

Conclusion:-

From this work, it results that the PIXE technique can be successfully applied to examine quality of aerosol samples. The target preparation and the PIXE measurement are not time consuming. The targets are irradiated with 2.7 MeV proton beams for 3 to 10 minutes depending upon the intensities of X-rays in the spectra. The samples are analyzed using GUPIX code in which many kinds of elements can be detected simultaneously in the widespread concentrations due to the multi-elemental nature and wide dynamic range of detection of the PIXE analysis. By using PIXE Technique we can also find the elemental composition of various biological and vegetable samples.

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