Characterization of Materials Using Accelerators

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Abstract. Mexico has three accelerators used for scientific research, one is located at the Autonomous University of Mexico (UNAM) and the other two at the Mexican Nuclear Research Institute (ININ). In the field of nuclear physics recent efforts have been focused in the areas of characterization of exotic decay modes of some nuclear excited states and the consequences on the paring interaction and the shell model, determination of the strength of the gravity interaction at very small distances, nuclear astrophysics and nuclear structure studies. For applications in Materials Science these accelerators have been applying mainly the PIXE and PIGE techniques to analyze the elemental composition of environmental, industrial and archaeological materials. Airborne particles collected in Mexico City were studied using PIXE to determine their elemental composition. Archaeological objects made of metal and ceramic were analyzed with PIXE to determine the manufacturing techniques.

1. Introduction

There are three accelerator facilities in Mexico used for scientific research, one is a 3 MV Pelletron located at the Institute for Physics Research at the National Autonomous University of Mexico (IFUNAM) another two a Tandem Van de Graff, nominal 6 MV accelerator and a Tandem Van de Graff 2 MV (Tandetron) at the Mexican Nuclear Research Institute (ININ).

1.1 Nuclear Physics Fundamental Studies

In the area of fundamental studies the main areas of research are**:**

- **a)** Characterization of exotic decay modes of some nuclear excited states and its consequences on the paring interaction and the shell model. Excited states of specific Beryllium and Carbon isotopes are prepared using (d,p) reactions. Some of the quantum numbers of these states are not well known and can be measured. Their decay through the emission of a two neutron cluster (dineutron), unobserved yet for any nuclear state can be studied.

- **b)** Nuclear Astrophysics with radioactive beams. Low intensity radioactive species (^{11}C, ^{25}Al, etc.) are produced as secondary beams through (d,n) reactions. The neutron detection serves as a tag for the radioactive particle that is directed towards a secondary target to scatter on protons (elastic, inelastic and capture reactions). Low energy cross sections, and particularly resonant scattering, on unknown or not well known states can be studied.

- **c)** Determination of the strength of the gravity interaction at very small distances. This is done through the precise determination of angular distributions in elastic scattering of neutrons on heavy targets.

- **d)** Nuclear Astrophysics. Using traditional charged particle detectors and the technique of the inverse kinematics on a thick target, using a gas (He) filled chamber. A complete excitation function is measured down to very low energies in one single measurement.

e) Nuclear structure. Precise measurement of the quantum numbers of known resonant states at sub-barrier energies. Thin targets, high quality beams and gamma detection are used to achieve absolute cross section determination with small error bars.

f) Nuclear reaction mechanisms of borromean nuclei. The study of these particular kind of very slightly bound nuclei requires previous solid knowledge of the characteristics of the reaction mechanisms. The fusion reaction mechanism of ^{6}Li on ^{58}Ni (isotopic pure target) can be studied through the detection of the angular distribution of the fusion products in charged particle telescopes.

In the area of materials characterization two main areas of research have been undertaken in the past few years.

1.2 Analysis of airborne particles using PIXE

Mexico City being one of the most populous city in the world has a serious problem with atmospheric pollution, for this reason the local government has started an atmospheric monitoring of airborne particles 2.5 μm (PM2.5). The aim of the project is to establish a network of sample collection stations in the most polluted areas.

The Mexican Nuclear Research Institute has installed a PIXE system in a new accelerator and is carrying elemental analysis on samples collected in EPA filters. With the information obtained on each of the reliable trace element concentration, a data base will be created.

The information on the concentration of certain elements such us S, V, Mn, Zn as well as some heavy metals will be used to determine the effects on health and to what degree..

The PIXE technique is a powerful method of multi element analysis and has been successfully applied to analyze major and trace components in environmental samples. The main advantage is that it is a fast and highly accurate non destructive testing and is more suitable for analysing heavier elements.

1.3 Analysis of archaeological materials using PIXE

Through elemental analysis composition of archaeological materials the location of material from which artefacts were made and possibly the place of manufacture can be deduced.
Using the PIXE technique obsidians, ceramics and metal archaeological materials can be studied. Only a small sample is required and a multi elemental composition can be obtained at once. The samples require almost no preparation except to be reduced to a standard geometry to be compared with a standard reference source.

At ININ the PIXE technique using a Tandem Van de Graff accelerator has been used for many years to study samples from our archaeological sites.

The base material as well as the pigments and glassy minerals have been studied in coloured obsidians obtained from sites in Central Mexico.

Metal objects found in ancient burial sites in the western part of Mexico were analyzed to understand the local manufacturing technology.

2. Elemental composition of PM 2.5 particles collected in Mexico City

Mexico City is amongst the most densely populated cities in the world, this normally brings high atmospheric pollution affecting public health. High levels of several pollutants such as particles PM 2.5 and sulphates have been continuously deposited in large areas of the city affecting most of the inhabitants children being the most vulnerable group. Since 1990 the Mexico City government has taken measures to improve the air quality and stringent regulations have been enforced.

2.1 Sampling Procedure

One of the most polluted areas in Mexico City was selected to be studied through a net work of monitors. A PM 2.5 Federal Reference Method manual sampler was used to collect 24h integrated samples to measure pollutants such as SO_2, NO_2, NOx and CO.

Samples were collected every third day on Teflon filters (47 mm diameter) during two sampling periods: dry-cold and rainy seasons as shown in Table I. The fine particle matter (FPM) mass concentrations were obtained by gravimetric analysis weighing the filters before and after taking the sample using a balance with a 0.1ug uncertainty. The filters were left for 48 hours under constant temperature and relative humidity conditions. During sample weighing an alpha source was used to eliminate static charges.

TABLE I: DESCRIPTIVE STATISTICS OF THE PARAMETERS INCLUDED IN THE CALCULATION.

	DRY-COLD SEASON				RAINY SEASON			
	N	**Min**	**Max**	**Mean**	**N**	**Min**	**Max**	**Mean**
SO_2	49	0.0016	0.0584	1.56E-02	48	0	0.02	8.90E-03
CO	47	0.7	3.2792	1.997434	49	0.6	2.6	1.4653
NO_X	51	0.0415	0.1799	0.100754	47	0.01	0.13	5.53E-02
NO_2	51	0.0205	0.082	4.81E-02	48	0.02	0.07	3.74E-02
FPM	51	7	59	30	49	11	40	21
S	51	252	4937	1598	49	418	2376	1132
C1	46	12	1022	152				
K	51	37	1824	287	49	46	750	135
Ca	51	38	289	122	49	40	927	113
Ti	48	3.7	26	9				
Mn	47	1.6	14	5	47	0	16	6
Fe	51	64	284	133	49	41	577	132
Cu	50	2.2	74	20	49	2	135	27
Zn	51	10	285	108	49	28	484	140
Pb	48	22	196	68	47	18	126	54

Number of appearances (N), minimum and maximum values detectd (min/max) in ng/m3 and mean value.

2.2 PIXE Characterization

The samples were bombarded by 2.5 MeV protons using beam currents of15nA in a 2MV Tandem Van de Graff (Tandetron) accelerator. The beam homogeneity was assured by using a diffuser foil. The characteristic X rays were detected using a Si(Li) detector located at 90 degrees from the beam looking into the irradiation chamber. The blank Teflon filters were systematically verified and the precision and reproducibility of the results checked to measure the consistency of the system. The element content of the FPM deposited in the membranes was identified and quantified. Elements such a as S, Cl, K Ca, Ti, Mn, Fe, Cu, Zn, and Pb were analyzed through the X ray spectra [1]. Elements such as V, Ni, Br, As, Se were also detected in some samples, however elements whose concentration was below the minimum detection limit were disregarded. [2,3,4]

The data was statistically treated using the method developed by Thurston and Spengler and the computation was carried using the statistical package SPSS.

2.3 Source identification

Four major sources were identified for the pollutants produced in the dry-cold season. The first was related to automotive exhaust fumes with elements such as Co, NOx, NO_2, K and Ca. The second source was related to road dust with associated elements such as Fe, Mn, Zn and Ti. The diesel combustion source was associated with sulphates S, SO_2. The four sources which included elements such as Cu and Pb were were small and can be associated with the operation of small scale industries in the area. These results are in agreement with results found elsewhere. [5,6]. Table II shows the results of the measurements and the contribution of each source.

TABLE II: MEAN SOURCE CONTRIBUTION TO EACH VARIABLE FOR THE DRY-COLD SEASON.

Variable	Automotive	Road Dust	Sulphates	Small Industry	Sumo of estimated mean contributions	Observed mean concentrations	Observed estimations r^2
SO_2	35.04	0.00	64.96	0.00	0.01	0.02	0.8883
CO	89.89	0.00	0.00	10.11	1.82	1.96	0.9257
NO_x	73.68	8.62	4.93	12.77	0.10	0.10	0.9724
NO_2	67.65	12.13	10.44	9.78	0.05	0.05	0.9690
FPM	44.88	24.45	30.68	0.00	30.81	30.33	1.0159
S	15.81	15.90	68.29	0.00	1454.59	1597.55	0.9105
Cl	0.00	109.85	55.00	-64.86	102.18	150.01	0.6811
K	127.16	30.79	0.00	-57.95	261.54	287.47	0.9098
Ca	59.84	30.63	9.54	0.00	116.36	121.78	0.9555
Ti	60.67	39.33	0.00	0.00	8.37	8.46	0.9888
Mn	22.21	62.17	0.00	15.62	4.41	5.01	0.8809
Fe	53.15	35.94	3.49	7.41	128.39	132.78	0.9669
Cu	0.00	0.00	0.00	100.0	17.44	19.79	0.8812
Zn	0.00	47.78	12.38	39.84	115.54	107.94	1.0704
Pb	7.31	31.72	19.78	41.19	52.18	68.04	0.7668

3. Characterization of archaeological objects using PIXE

3.1 Object from an ancient burial site

The first Mexican group with suitable technology to manufacture metallic objects were the Purepechas in western Mexico [7,8,9]. In 1957 a tomb of a noble Purepechan Indian and his wife was discovered in the City of Uruapan Michoacan. Amongst the several objects recovered from the tomb was an axe handle, an elaborate collar made of golden beads, copper rattles and bezotes made of obsidian gold, zoomorphic earrings, copper and gold rings. Other objects found were boring tools, pliers, needles, hooks, an axe and artefacts made of pearl, turquoise, rock crystal, green stone, serpentine, amber and amazonite. His wife was buried with objects such as a polychrome clay vessel, rattle bracelets made of copper, frog figures, an elaborate vessel and copper needles. Table III shows the different objects studied in this work.

TABLE III ARCHAEOLOGICAL OBJECTS UNDER STUDY.

Sample	Weight (g)	Height, wide, thickness (cm).
Ax	800	18, 8.5, 1.0
Coiled wire	0.3	1.0, 2.5, 0.01
Belt (3 CH)	0.6	6.0, 0.04, 0.09
Belt (3 G)	7.4	4.0, 2.0, 0.05
Belt (4)	1.4	2.5, 1.0, 0.03
Anthropomorphic figure	6.5	3.0, 8.5, 4.0
Metal ring or copper band	11.6	---, ---, 4.0, diameter 4.6
Flat Disk	1.6	---, ---, 0.01, diameter 2.2
Pliers Fragments (5 G)	6.3	---, ----, ---
Pliers Fragments (5 CH)	5.6	---, ----, ---
Punch with 2 extremes(U12)	6.5	7.3, ---, 0.04
Punch with 2 extremes(U13)	7.5	7.0, ---, 0.05
Needle (U14)	4.7	12.0, 0.04,0.02

3.2 Experimental Technique using PIXE

The objects were cleared of all corrosion using a tungsten drill to avoid contamination of the samples and to obtain a polished and clean surface.
The objects were irradiated using an external beam facility at the accelerator Tandem Van de Graff from the Mexican Nuclear Research Institute and the PIXE technique was used to measure the elemental composition.

The beam spot from the accelerator was 2 mm diameter and the incident angle was 90 degrees. A 16 µm thick aluminium foil was used as a window for the proton beam spot on the samples positioned 1 cm ahead. The collimator connected to a current integrator served to monitor the proton current which ranged between 10 and 50 nA. The detection system was composed of a Si Li detector with a resolution of 200 eV at a 6.4 keV X ray energy. The detector was placed at a distance of 4.5 cm from the target at an angle of 52.5 degrees. A counting rate of about 1000 seconds is desirable in order to avoid pulse pile up. The mean proton energy was 2.6 MeV with a 4mm diameter.

For each of the experiments a standard reference material SRM (commercial bronze NIST) was used to calibrate the system. Measured X ray spectra were processed by GUPIX [10]

3.3 Results from the analysis of some objects

The main component of the metal object was Cu and to smaller extent Fe, As and Sn. An Cu-Sn alloy was identified as the main component of the axe and the boring tool. The bell was found to be made of Cu-As, the big bell was made of copper and the decoration figure made of Cu-Sn alloy. The tweezers and the boring tool were made of copper and As. The ring, the flat disc and the needle were also made of copper. Figure 1 shows a typical spectrum from the X ray analysis.

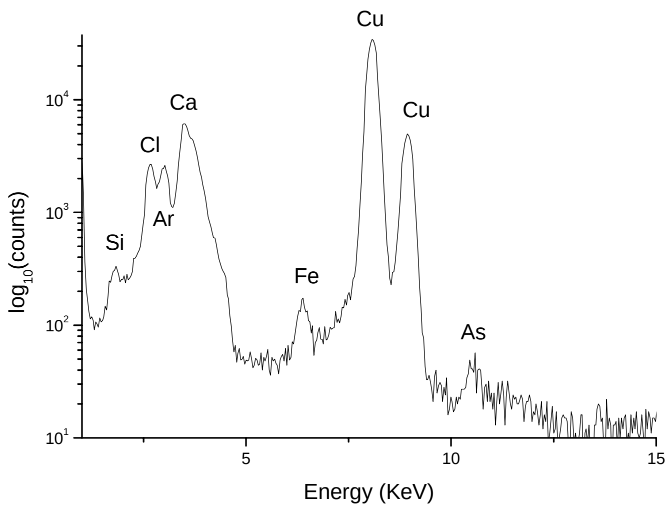

FIG.1 Typical X-ray spectrum obtained with a 2.6 proton bombardment of a metallic object.

4. Conclusions

The Mexican research accelerators are used for nuclear physics studies and for the characterization of different materials using PIGE and PIXE. In this work we present the analysis of airborne particle matter and archaeological objects using the PIXE technique. This analytical technique is most suitable for determining the elemental composition of materials and the results obtained are in good agreement with the data available in the literature.

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