

NUCLEAR ANALYTICAL TECHNIQUES IN TANZANIA: A REVIEW

YIA Koleleni

Physics Department, University of Dar es Salaam, P.O.Box 35063 Dar es Salaam
ykoleleni@gmail.com

ABSTRACT

It has been a tradition to perceive that Nuclear/Atomic radiations are only related to Nuclear/Atomic bombs, heavy ammunitions and consequently applications for human and non-peaceful destructions. In this paper the questions on how the Nuclear/Atomic radiations may be used for peaceful applications and specifically to Tanzanian situations are presented. Methods like Energy Dispersive X-Ray fluorescence (EDXRF), Particle Induced X-Ray Emission (PIXE), Electron probe Micro-Mass Analysis (EPMA), Neutron Activation Analysis (NAA) for investigations of different types of samples in geology; environmental, medical, biological and chemical are discussed.

INTRODUCTION

The radiations referred in this article are those originating from atomic or nuclear process. They are categorized in to two groups of charged particles and uncharged radiation. The charged radiation includes fast electron and heavy charged particles like protons and alpha (α) particles. While uncharged radiations are electromagnetic radiation and neutrons (Knoll 2000). The fast electrons β^\pm are emitted as a result of nuclear decay and other process in the atomic transition. The heavy elements with mass of at least one atomic mass unit are energetic ions like α particle (^4He), protons, fission product or products of nuclear radiation. The electromagnetic radiations referred to in this case are x-rays from electronic transitions or γ - rays from nuclear transition (Knoll 2000). The other category includes neutrons from nuclear process depending on their speed fall under slow neutron and fast neutrons group (Knoll 2000).

Energy-dispersive x-ray fluorescence (EDXRF) analysis has been established at the University of Dar es Salaam, College of Natural and Applied Sciences (CONAS), Department of Physics since 1979 with support from International Atomic Energy Agency (IAEA) for peaceful applications of nuclear and nuclear related technology. Calibration was conducted using thin films from Macro matter for secondary target XRF (Koleleni 1987, Koleleni and Kondoro 2005).

Previous work has included measurements of photon cross-sections for different materials, (Mashauri 1983). The incident radiations to the selected elements were at energies 2.2, 59.6 and 88 keV. The total photo interaction cross-sections for the elements Al ($Z = 13$), Cu, Zn, Cd, and Sn at photon energies 22.2, 59.6, and 88.8 keV have been measured. Hyper pure germanium (HPGe) detector area = 0.5 cm^2 , Crystal thickness 5 cm and Be window was 2 mm thick, Resolution 188 eV at 5.9 keV, Mn K – line after background correction analysis

was done with great precision (Mashaury 1984).

Other work were on trace analysis of water for pollution studies (Mwilike 1985), aerosol trace analysis (Koleleni 1987), geological rock analysis looking for concentrations of tin (Mwambeule 1989) and serum analysis of malnourished children (Mwiruki 1990). Mean serum conc. 0.6, 1.0, 0.8 and 4.1 mg/l sample volume deposited on Mylar film suspended on the flow ring. The excitation condition for Mo, tube was 40 KV and 40mA 1000 seconds and results show Ca deficiency on malnutrition samples (Mwiruki 1991). Since that time, efforts have continuously been made to refine the method and improve the detection limits to cover as low a concentration as possible in different types of samples (Mwilike 1985, Koleleni 1987, Koleleni and Kondoro 2005). In Total Reflection X-Ray Fluorescence (TXRF), the passage of x-rays through slits and reflectors are conducted so as to obtain a total reflection condition. This is used to excite the sample without introducing a Bremsstrahlung background in the sample spectra (Koleleni 2001).

The EDXRF method is applied to analyze and quantify tin in geological samples. The method uses scattered radiation technique (Mwambeule 1988). Calibration for tin analysis is discussed and quantification established. The accuracy between 2% and 5% was made for standard reference rock sample and 3% to 8% for rocks from Karagwe tin fields and Lupa Gold mines (Mwambeule 1988). A number of precious stones and minerals in Tanzania are found either just on the ground surface or unexplored inside the earth (Schumann 1977). In several parts of the country such

information is completely unavailable and in many cases unexplored (Koleleni 2010). In order to provide this information to the decision makers and investors, Wavelength Dispersive X-ray Fluorescence (WDXRF) analysis has been used with objective of identifying the composition of some of these stone and rock samples (Koleleni 2010). The geological samples reported (Koleleni 2010) include: Iron ore, Green Aventurine, Amethyst, Red Garnet, Sapphire nuts, Green Opal, Blue Kymete, Black Tourmaline, White opal, Aquamarine and Granite. The samples were prepared as pellets after mixing with boric acid or fused as discs for analysis. Typical results for the Iron Ore in % are SiO₂ (0.97), Al₂O₃ (0.57), Fe₂O₃ (77.5), MnO (0.35), MgO (0.35), CaO (0.16), Na₂O (< 0.01), K₂O (0.04), TiO₂ (19.1), P₂O₅ (< 0.01), SO₃ (0.14) and that of the Amethyst SiO₂ (97.8), Al₂O₃ (0.11), Fe₂O₃ (1.37), MnO (< 0.01), MgO (< 0.01), CaO (< 0.01), Na₂O (< 0.01), K₂O (< 0.01), SO₃ (0.33). The results show that Tanzania is a very rich country in minerals at that time not explored (Koleleni 2010).

The determination of radon gas and respirable dust concentration in the underground was conducted at Merelani, Tanzanite mines (Kahuluda 2014). Assessment of the concentration of radon gas, radioactive gas and respirable dust in Merelani underground Tanzanite mines was conducted. The estimated effective dose (D) of 1.6 mSv was higher than the external expressive annual effective dose of 1 mSv for public. Overall concentration of respirable dust arithmetic mean was 18.2 g/m³ and geometric mean of 2.1 g/m³ which is higher than the underground mine of 2 g/m³ (Kahuluda 2014). EDXRF was used to determine concentrations of naturally occurring radioactive elements in the rocks of the underground working place. The

minimum levels was 42.2 Bq/m^3 and maximum was 4.2 kBq/m^3 with geometric mean 118.4 Bq/m^3 hot spots have mean radon concentrations of 4 times the ICRP workplace action level of 1000 Bq/m^3 . Radioactive elements was radon gas ($13.37 \text{ } \mu\text{g/g}$), thorium $10.40 \text{ } \mu\text{g/g}$ and potassium ($14478.60 \text{ } \mu\text{g/g}$) ICRP international commission for radiation protection (Kahuluda 2014).

Studies by Mossbauer spectroscopy on Synthetic Minerals (idaite and bornite) and ancient Black –topped pottery from Cyprus, Nile Valley and Africa were made (Makundi 1988). Mossbauer spectroscopy Ray has been used to establish the valence and magnetic structure of synthetic $\text{Cu}_{5.5}\text{FeS}$ (idaite) and $\text{Cu}_5 \text{FeS}_4$ (bornite). Mossbauer measurement was made at 295, 77 and 10K. Findings revealed fast ions, mainly silicate mineral Fe^{3+} ions which provide the red colour (Makundi 1988).

In Dar es Salaam a study of the aerosol contents was conducted and particulate matter on the filters was collected using an Andersen TMPM10 impactor to determine the environmental air pollution. The contents were determined by x-ray fluorescence analysis (Koleleni 2002). In environmental studies, sources of air degradation and their concentrations were found. The combustion processes, which has range of 10 to 800 ng/m^3 for Br, from 30 to 790 ng/m^3 for Pb. The industrial processes which has range of 37 to 883 ng/m^3 for Fe, 14 to 310 ng/m^3 for Cu, 6 to 820 ng/m^3 for Zn. Top soil activities with range of K from 20 to 540 ng/m^3 , Ca from 24 to 3805 ng/m^3 , Ti from 2 to 59 ng/m^3 , Mn from 10 to 386

ng/m^3 . Marine processes contribute a range of Cl from 20 to 310 ng/m^3 and S from 72 to 134 ng/m^3 (Koleleni 2002).

The EDXRFA was used by Munubi 1998 to study elemental composition in city site and rural areas. Composition in the city were dominated by S, Zn, Br and Pb while the rural had correlation with Fe, Ca, K and Ti which domination in coarse particle fractions are correlated to rural area measurement deposit in the atmosphere.

Analysis of serum for the determinations of Fe, Cu, Zn and Br in children in Tanzania was conducted using X-ray Fluorescence Analysis (XRF). The XRF spectrum analysis of human serum was done for 50 malnourished child while control group of 21 health children (Mwiruki 1991). The mean results were 0.15, 0.04, 0.05 and 0.08 mg/l Fe, Cu, Zn and Br, respectively.

EDXRFA of water and plants from Lake Victoria at Mwanza was conducted by Hamza (1996) and Mohammed (1999). Heavy metal pollution ranged from $0.62 - 0.88 \text{ } \mu\text{g/l}$. In Mwanza North results were $164 \text{ } \mu\text{g/l}$ of Fe, $860 \text{ } \mu\text{g/l}$ of Mn, $42.8 \text{ } \mu\text{g/l}$ of Cr. They are mainly of industrial origin while $15.1 \text{ } \mu\text{g/l}$ of Pb, $742.5 \text{ } \mu\text{g/l}$ of Zn and $23.3 \text{ } \mu\text{g/l}$ of Cu are from Soil dust. When compared with International values Fe and Mn have higher values otherwise the rest are within tolerable levels (Hamza 1996). The water plants samples from Lake Victoria influenced by industrial and domestic sewage around Mwanza was measured. Higher concentrations of heavy metals such as K, Ca, Sc, Ti, Mn, Fe, Co, Ni, Zn and Pb were obtained from areas of disturbance

compared to non-disturbed ones (Mohammed 1999).

Agrochemicals used in farming activities have high potentials in immobilizing heavy metals to agricultural soils. The EDXRFA studied sixty soil samples and concentration of V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As and Pb were determined (Makame2014). Using Factor analysis (FA) and the principal component analysis (PCA), the existence of the elements were derived from the fertilizers and pesticides used in the rice farm at Cheju Zanzibar (Makame2014). A measurement of heavy metal concentrations in Mchicha and Cabbage consumed in Zanzibar by EDXRF analysis was conducted. Resolution of 160 eVMn line Si(Li) detector at 50 W, 50 kV, 10 mA, for 13 elements: Al, Ca, Cr, Mn, Fe, Ni, Zn, Cu, Ag, I, Br, Cd and Pb were detected in the vegetables samples (Fatima 2012). In mchicha samples results were, MDL 1-1.7 µg/g, 1.662-2.55 µg/g 0.6 -1.2 kg/g for Cd, Ni, Cr, and Pb respectively. Cabbage MLD 1.2-2.5, 0.58-2.06 and 0.7-1.3 µg/g for Cd, Ni, Cr, and Pb, respectively. Concentration were higher than respective maximum to tolerable limit set by national and international organisation (TBS 2003, WHO 2001) of 0.2 µg/g, 2.3 µg/g, 0.3 µg/g and 0.2 µg/g for Cd, Cr, Pb and Ni, respectively (Fatima 2012).

In summary the objective of this work was to highlight the peaceful applications of nuclear techniques, which here reference is made to nuclear analysts in the use of nuclear radiations and detectors to obtain information regarding various materials on environment, industrial, geological, archaeological and medical materials. There

are in existence many nuclear related facilities in the market like Energy Dispersive X-Ray fluorescence (EDXRF), Particle Induced X-Ray Emission (PIXE), Electron probe Micro-Mass Analysis (EPMA), Neutron Activation Analysis (NAA) and others are in the developmental stages all the time to improve their accuracy and precision. The x-ray machines, the γ -rays from radioisotope sources and other development have been in place for different countries all over the world and Tanzania should not be left behind. The minimum that can be done is to provide the nuclear/atomic radiation education to our people.

THE ACHIEVEMENT MADE IN TANZANIA

Energy Dispersive X-ray Fluorescence (EDXRF)

Energy Dispersive X-ray Fluorescence (EDXRF) is among the types of X-ray Fluorescence techniques used for elemental analysis applications in Tanzania. In EDXRF spectrometers, all of the elements in the sample are excited simultaneously, and an energy dispersive detector like the Si (Li) or Ge (Li) in combination with a multi-channel analyser is used to simultaneously collect the fluorescence radiation emitted from the sample and then separate the different energies of the characteristic radiation from each of the different sample elements. Resolution of EDXRF systems is dependent upon the detector, and typically ranges from 150 eV – 600 eV. The principal advantages of EDXRF systems are their simplicity, fast operation, multi-element, lack of moving parts, and high source efficiency.

X-ray optics (Fig.1) adds more to EDXRF instrumentation. In a conventional XRF instrumentation, typical focal spot sizes at the sample surface range in diameter from several hundred micrometres up to several millimetres. Poly-capillary focusing optics (Fig.1), collect X-rays from the divergent X-ray source and direct them to a small focused beam at the sample surface with

diameters as small as tens of micrometres (Van Gieken and Markowicz 2002). The resulting increased intensity delivered to the sample in a small focal spot allows for enhanced spatial resolution for small feature analysis and enhanced performance for measurement of trace elements for Micro EDXRF applications.

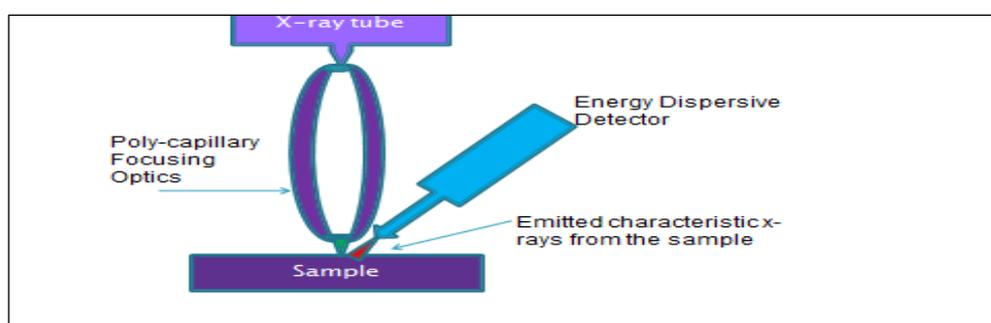


Figure 1: Doubly curved crystal optics directs an intense micron-sized monochromatic X-ray beam to the sample surface for enhanced elemental analysis.

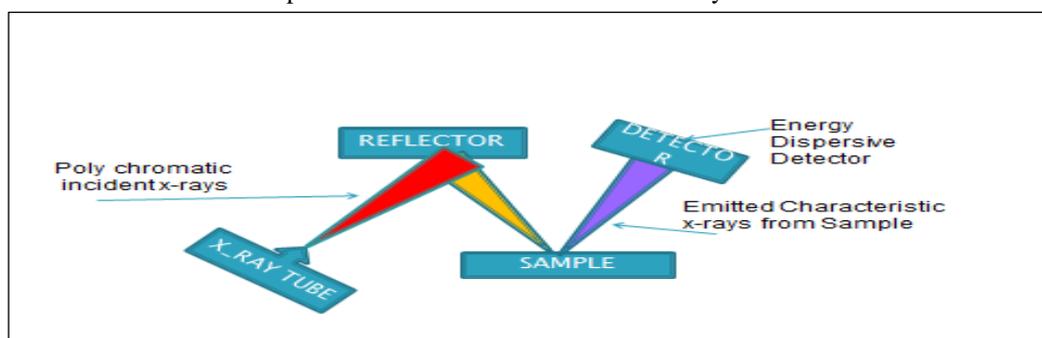


Figure 2: Total Reflection X-Ray Fluorescence (TXRF) experiment setup.

The TXRF(Fig.2) facility includes most of the main equipment used in the EDXRF set-up with a TXRF module and a sample holder attached to the x-ray tube. The reflected beam, in a total reflection condition, is incident on a sample carrier, which is a reflecting surface at critical condition. The

characteristic x-rays from the sample are detected by the Si (Li) detector and associated electronics. Calibration was conducted using liquid standards deposited as thin films on quartz plates as explained elsewhere (Koleleni 2001). Total reflection X-ray fluorescence (TXRF) is a surface

elemental analysis technique often used for the ultra-trace analysis of particles, residues, and impurities on smooth surfaces. It is currently an important tool for wafer surface contamination control in semiconductor chip manufacturing. TXRF is an EDXRF technique in total internal reflection geometry (Van Gieken and Markowicz 2002). An incident beam hit the sample at angles below the critical angle thus reflection of almost 100% of the exciting photons. In this configuration, the advantage of TXRF over conventional XRF is a reduced background contribution which was caused by Compton scattering and thus improve sensitivity.

In a conventional XRF geometry (Fig.1), the angle of incidence is greater than the critical angle and the primary radiation penetrates into the sample and the Compton scattered

appear in the sample. In a conventional TXRF geometry (Fig.3), the angle of incident X-rays is less than the critical angle and the primary radiation is reflected off of the sample surface and does not enter into the detector. This reduces the background radiation significantly and eliminates the Compton scattered peak.

Doubly curved crystal optics can be used to focus X-rays to small spots for localized TXRF elemental analysis. A slit is used to restrict beam divergence in the scattering plane to less than the critical angle in order to meet the total reflection requirement. The flux density on the reflection surface is several orders of magnitude higher than that of conventional TXRF systems yielding high elemental sensitivity.

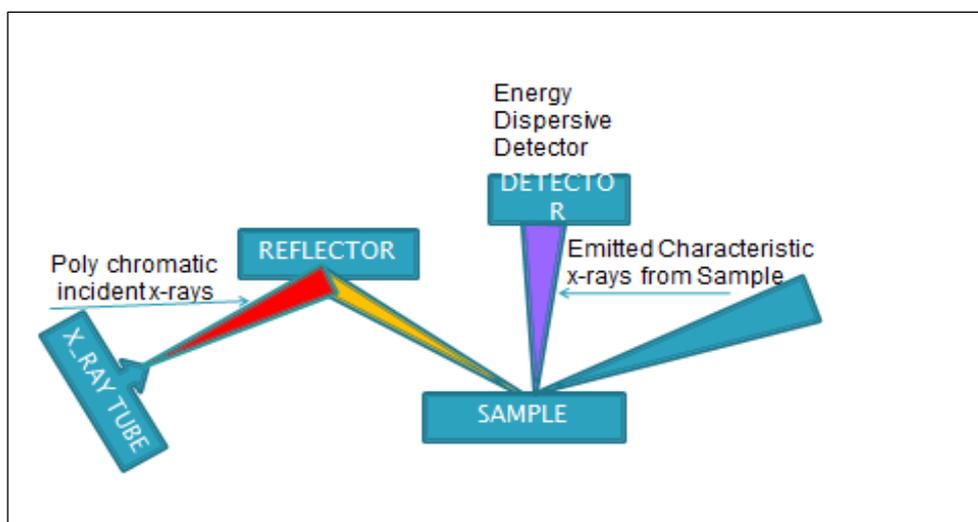


Figure 3: Schematic illustration of TXRF

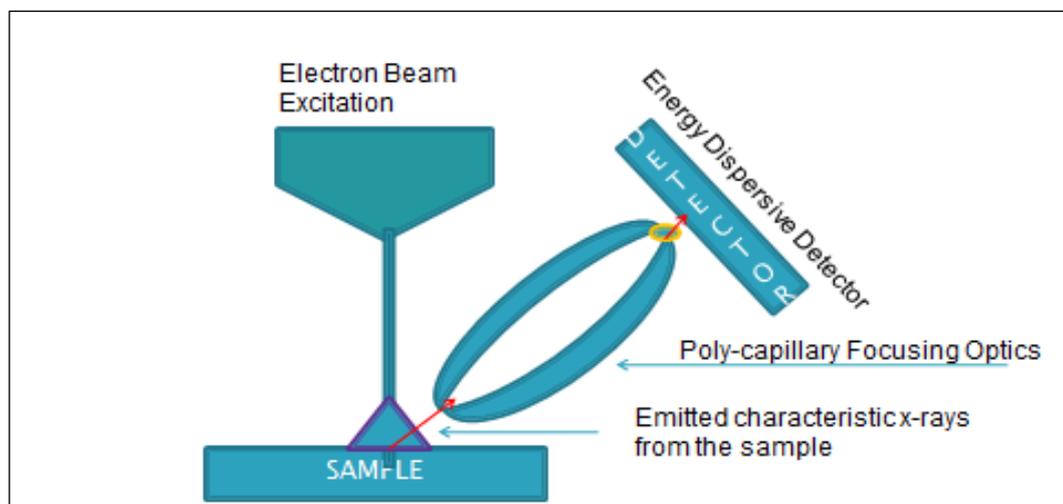


Figure 4: Schematic illustration electron excitation XRF

X-Ray Fluorescence in Scanning Electron Microscopy (SEM)

Electron-probe X-ray fluorescence (EP-XRF) analysis has been widely used in scanning electron microscopy instrumentation for elemental analysis of specimens. With the x-ray fluorescence microscopy in the SEM (Fig.4), the surface of a solid sample is excited with a highly-focused energetic beam of electrons which induces X-ray fluorescence from the elements within the sample.

The major advantage offered by the electron excitation, as opposed to X-ray excitation, is the small probe size, typically < 1 mm, and its high excitation efficiency, especially in the low-energy range. However, EP-XRF has some major limitations. First, the electron source causes Bremsstrahlung background, which limits the sensitivity of EPXRF analysis. Secondly, EPXRF is a

surface-sensitive technique and cannot be used to measure under layer materials in a sample. In contrast, XRF analysis using X-ray excitation offers much higher detection sensitivity, especially for high-energy X-rays. Under layer analysis is possible due to much better depth penetration of X-rays in comparison with the electron probe. Furthermore, XRF analysis allows the sample to be measured as it is, while a certain level of sample preparation is required for EPXRF, as it requires that sample to be conductive.

The capability of excited X-ray fluorescence can be integrated into an existing SEM (Fig.5) system using an X-Beam unit containing a poly-capillary optic (Van Gieken and Markowicz 2002). The approach extends the capability of the SEM at low cost by using existing hardware and software on the SEM.

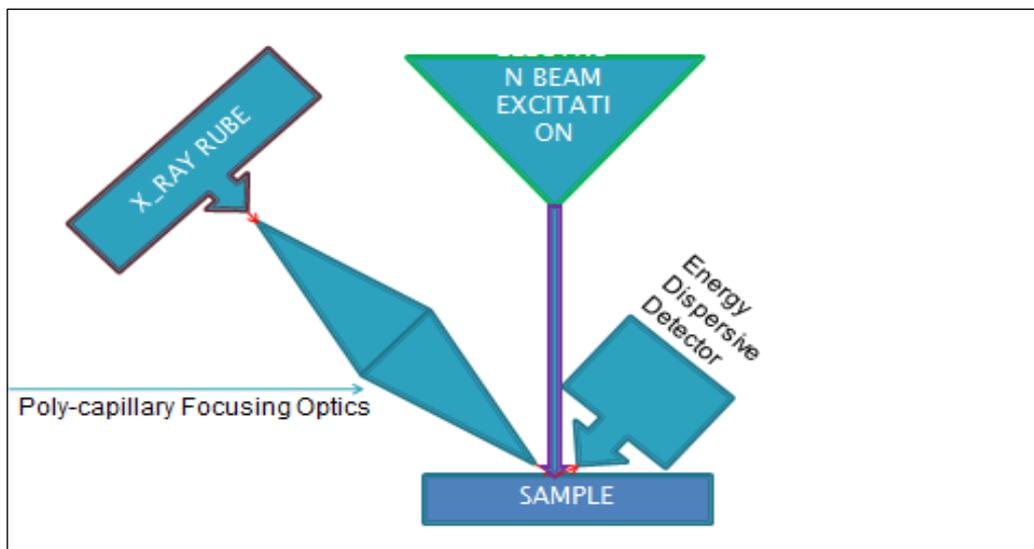


Figure 5: The schematic illustration of SEM

OTHER NUCLEAR TECHNIQUES AS WAY FORWARD

Particle Induced X-ray Emission (PIXE)

Particle-induced X-ray emission or proton-induced X-ray emission (PIXE) is a technique used in the determination of the elemental make-up of a material or sample. When a material is exposed to an ion beam, atomic interactions occur that give off EM radiation of wavelengths in the x-ray part of the electromagnetic spectrum specific to an element. It is non-destructive elemental analysis technique now used routinely by geologists, archaeologists, art conservators and others to help answer questions of provenance, dating and authenticity.

The technique was first proposed in 1970 by Sven Johansson of Lund University, Sweden, and developed over several years with his colleagues Roland Akselsson and Thomas B Johansson as shown in figure 6 (Johansson et al. 1970). Recent extensions of

PIXE (Johansson et al. 1995, Carlsson et al. 1981, Maenhaut and Malmqvist 2002) using tightly focused beams (down to 1 μm) give the additional capability of microscopic analysis. This technique, called micro-PIXE, can be used to determine the distribution of trace elements in a wide range of samples. A related technique, particle-induced gamma-ray emission (PIGE) can be used to detect some light elements.

Accelerated ions are used to bombard the sample. The fast moving particles knock and excite the electrons from one energy level to another in the sample. When these missing electrons are replaced within an atom, a characteristic X-rays are emitted. By detecting and analyzing the x-rays and identification of most of the elements in the sample can be done. Then it is possible to obtain the absolute concentrations of the elements.

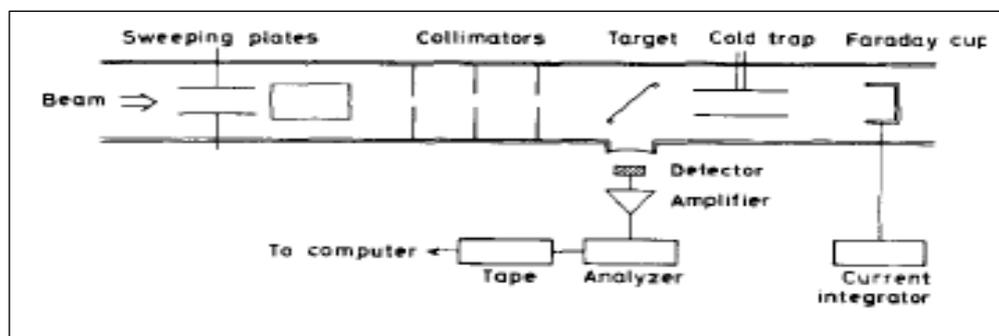


Figure 6: The schematic illustration of PIXE experimental arrangement (Johansson et.al. 1970)

PIXE can be used to measure simultaneously with Rutherford Back Scattering (RBS) on the 45 beam line and on the Micro beam line. Users frequently use PIXE (and RBS) with the scanned micro beam to map the surface location and concentrations of elements (Johansson et al. 1995).

Electron microprobe (EMP)

An electron microprobe (EMP), also known as an electron probe micro analyser (EPMA) or electron micro probe analyser (EMPA), is an analytical tool used to non-destructively determine the chemical composition of small volumes of solid materials (Van Gieken and Markowicz 2002). It works similarly to a scanning electron microscope: the sample is bombarded with an electron beam, emitting x-rays at wavelengths characteristic to the elements being analysed. This enables the abundances of elements present within small sample volumes (typically 10-30 cubic micrometres or less) to be determined. The concentrations of elements from beryllium to plutonium can be measured at levels as low as 100 parts per million (ppm). Recent models of EPMA can accurately measure elemental concentrations of approximately 10 ppm.

Neutron activation analysis (NAA) is a nuclear process used for determining the concentrations of elements in a vast amount of materials. NAA allows discrete sampling of elements as it disregards the chemical form of a sample, and focuses solely on its nucleus. The method is based on neutron activation and therefore requires a source of neutrons. The sample is bombarded with neutrons, causing the elements to form radioactive isotopes. The radioactive emissions and radioactive decay paths for each element are well known. Using this information, it is possible to study spectra of the emissions of the radioactive sample, and determine the concentrations of the elements within it. A particular advantage of this technique is that it does not destroy the sample, and thus has been used for analysis of works of art and historical artifacts. NAA can also be used to determine the activity of a radioactive sample (Pollard and Heron 1996).

If NAA is conducted directly on irradiated samples it is termed **Instrumental Neutron Activation Analysis (INAA)** as shown in Figs. 7 and 8. In some cases irradiated samples are subjected to chemical separation to remove interfering species or to concentrate the radioisotope of interest, this technique is known as **Radiochemical Neutron Activation Analysis (RNAA)**.

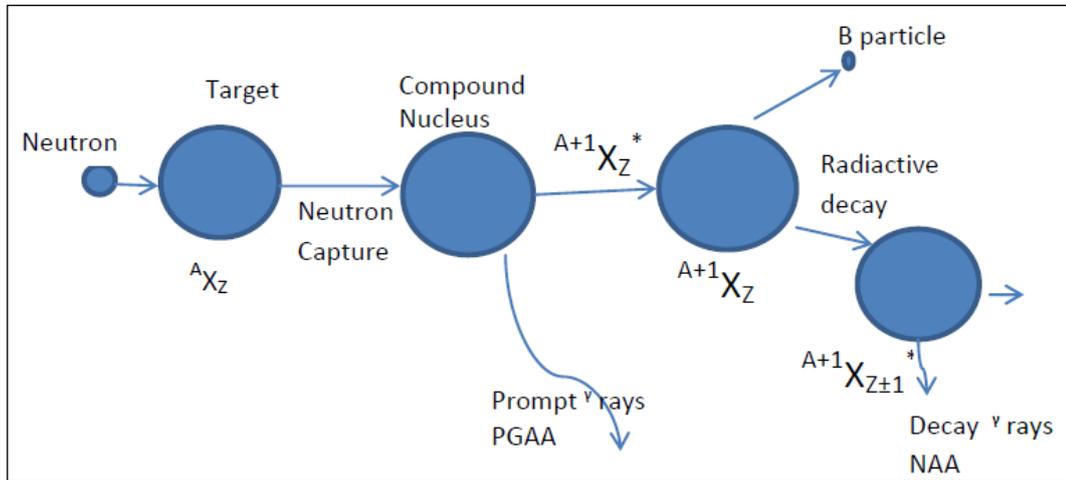


Figure 7: Illustration of NAA experimental arrangement

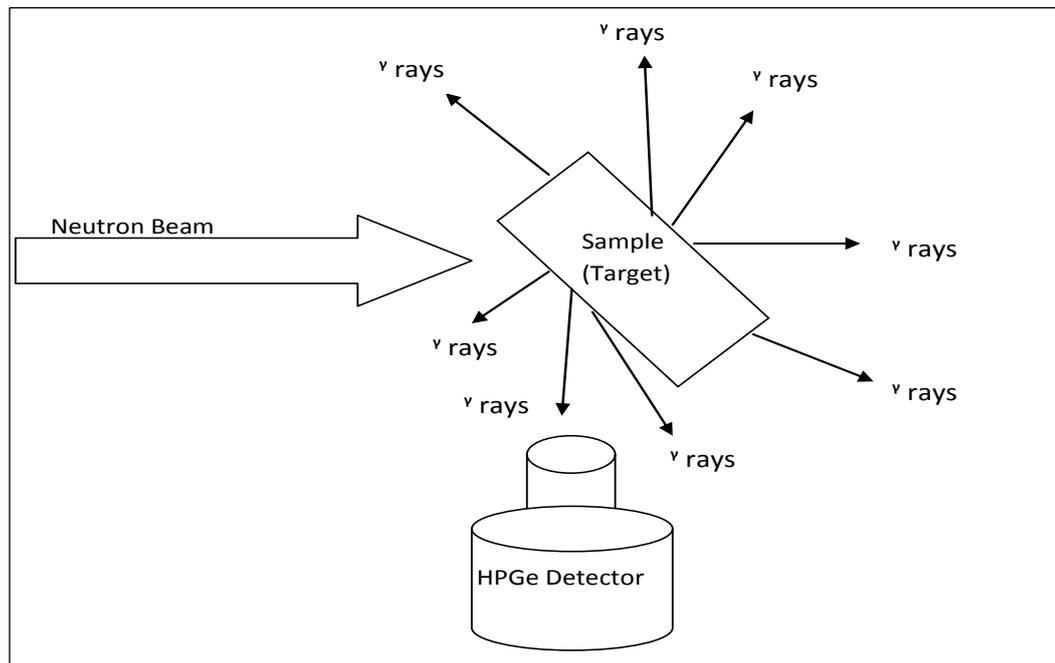


Figure 8: A schematic illustration of NAA excitation and detection arrangement

NAA can perform non-destructive analyses on solids, liquids, suspensions, slurries, and gases with no or minimal preparation. Due

to the penetrating nature of incident neutrons and resultant gamma rays, the technique provides a true bulk analysis. As different

radioisotopes have different half-lives, counting can be delayed to allow interfering species to decay eliminating interference. Until the introduction of ICP-AES and PIXE, NAA was the standard analytical method for performing multi-element analyses with minimum detection limits in the sub-ppm range (Pollard and Heron 1996). Accuracy of NAA is in the region of 5%, and relative precision is often better than 0.1%. There are two noteworthy drawbacks to the use of NAA; even though the technique is essentially non-destructive, the irradiated sample will remain radioactive for many years after the initial analysis, requiring handling and disposal protocols for low-level to medium-level radioactive material; also, the number of suitable activation nuclear reactors is declining; with a lack of irradiation facilities, the technique has declined in popularity and become more expensive (Pollard and Heron 1996).

Standards and reference material

The quality assurance and quality control (QA/QC) program pertaining to the instrument calibration make use of standards from National Bureau of Standards (NBS), National Institute of Standards (NIST), United States Geological Standards (USGS), National Institute of Environmental Standards (NIES) and International Atomic Energy Agency (IAEA) reference materials whose values are either certified or well established (Rueter et al. 1975).

CHALLENGES

The spectra given in Figures 9 and 10 are from EDXRF in Tanzania for plant and aerosol sample, respectively while that given in figures 11, 12 and 13 are from micro PIXE analysis of quartz and aerosol samples. These show that the two methods are not necessarily competing but complementing each other.

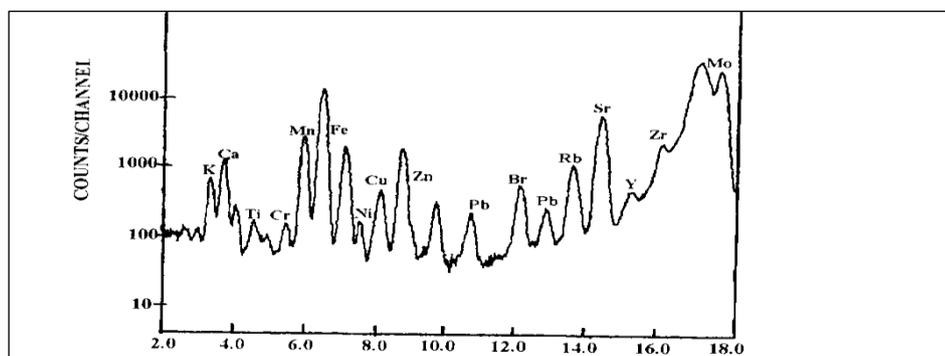


Figure 9: XRF spectrum of plant sample

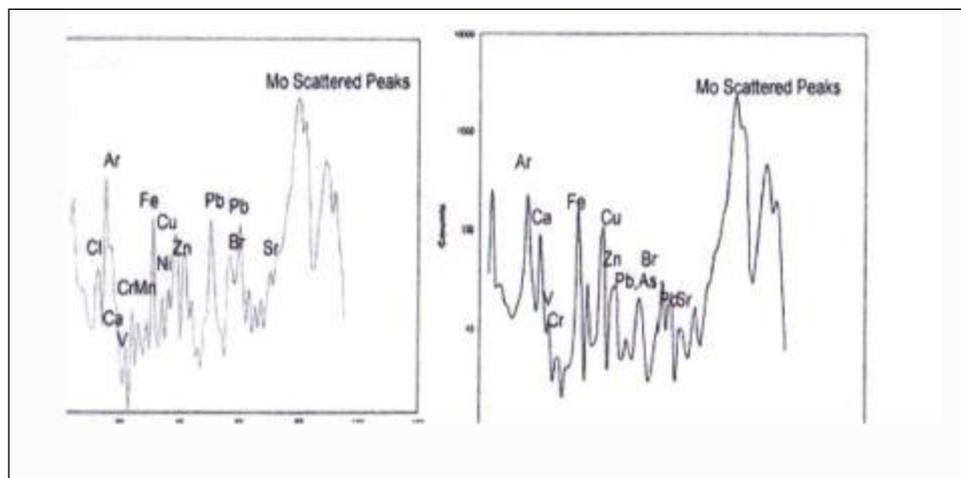


Figure 10: XRF spectrum of aerosol sample

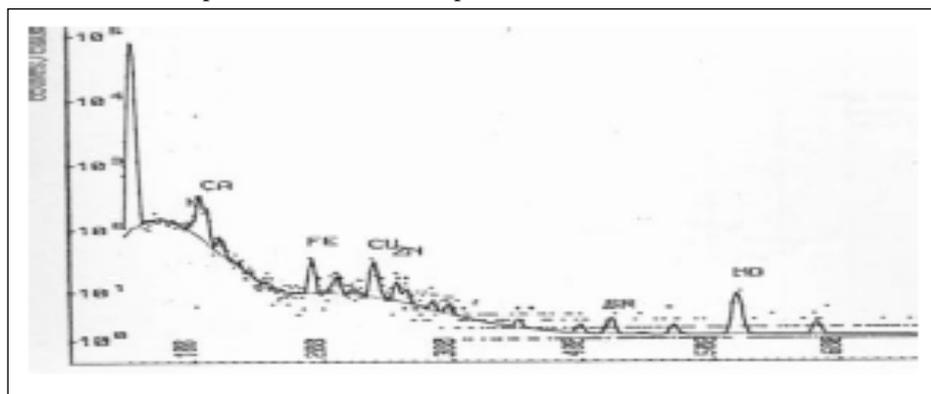


Figure 11: Micro-PIXE spectrum of quartz blank sample (Koleleni 1988)

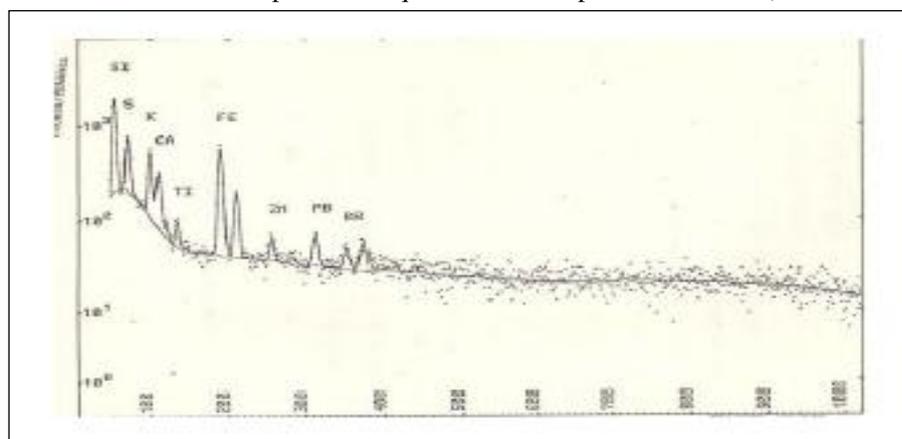


Figure 12: Micro-PIXE spectrum of aerosol sample (Koleleni 1988)

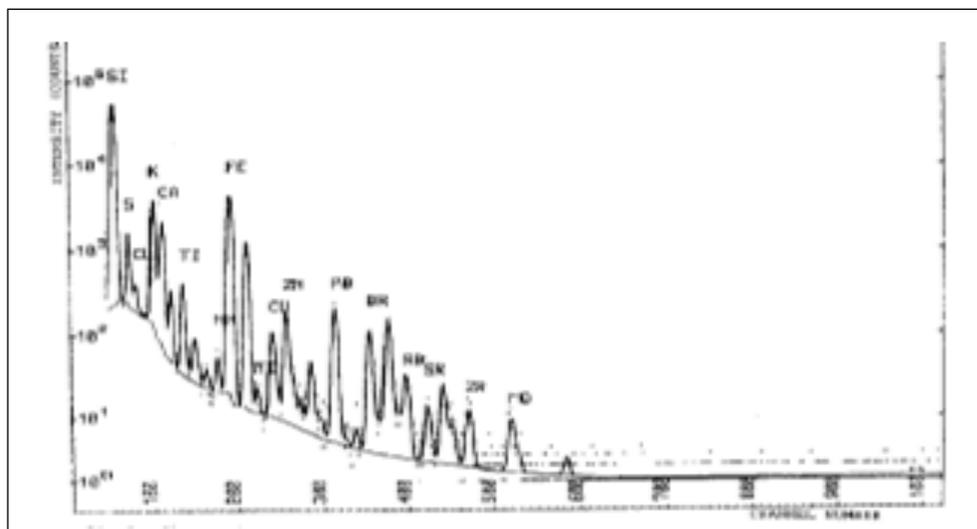


Figure 13: Micro-PIXE spectrum of aerosol(Koleleni 1988)

The report on the performance of the XRF spectrometer including the detection limits attained, which ranged from 0.01 to 10 ng cm⁻² using collimators of 6 and 8 mm diameter under excitation conditions of 50 kV, 35 mA. The accuracy of the measurements was checked using IAEA SOIL-7 and NIST 3087a Certified Reference Materials. The experimental values differed by <5% from the certified values. The total reflection x-ray fluorescence (TXRF) facility added as a module to the existing XRF system provides a better detection limits between 0.1 and 100 pg for most of the elements measured (Koleleni 1987, Koleleni and Kondoro 2005). Several experiments can be done by PIXE, SEM, Electron Probe (EMP) where more information may not be necessarily available in all types of XRF. However the limitations are on the financial side, skills and trained manpower.

CONCLUSIONS

An XRF analysis system based on both EDXRF and TXRF components has been shown to be a very powerful analytical tool. After a series of calibrations the detection limits found were of the order of 10 ng cm⁻² for EDXRF and 10 pg for TXRF. Competing and complimenting techniques such as PIXE, NAA, and EPMA are given. In PIXE, main features include accelerator where protons are used to excite the samples which is more expensive than photons used in EDXRF. The same trend applies to NAA where neutrons from a rather expensive reactor is used to excite the sample and in Electron Probe micro Mass Analysis EPMA where electrons are used to excite the Sample. In this paper the questions of how the Nuclear/Atomic radiations may be used for peaceful applications as nuclear techniques specifically to Tanzanian situations are raised. The capacity for investigations of different types of samples in geology, environmental, medical,

biological and chemical samples were discussed as the way forward by adding more equipment.

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