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# *In situ* analysis of human teeth by external PIXE

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ABSTRACT

The elemental profiles of the enamel, cementum and caries of human teeth were analysed by the external proton induced X-ray emission studies. Ten elements namely P, Ca, V, Mn, Fe, Cu, Zn, As, Sr and Pb were estimated in the present study. P and Ca were found to be the major elements whereas all other elements were found in trace level. It was observed that the respective concentrations of elements namely P, Ca, Fe, Zn and Pb in enamel are more than those in cementum. Concentration of P ranged between 6.37% and 25% whereas Ca ranged between 12.94% and 43.36%.



## 1. Introduction

Elements play an important role in human health. Deficiency and excess of these, resulting from exposure to both the natural and man made environment, can lead to a wide variety of clinical effects [1]. Teeth are reported to be suitable indicators of trace element exposure for a wide range of elements. Since teeth accumulate a variety of trace elements, it is very interesting to study the elemental distribution in human teeth to evaluate biological processes. The knowledge of the spatial distribution of trace elements in tissues is involved in many biological functions of living organisms. In this way, elemental distribution in teeth can provide information about physiology of elements, environmental influence, contamination by metallic amalgams used as restorative materials and dietary habits [2].

Non-destructive analyses of the major and trace elements present in biological samples are of great importance, because these materials are sometimes too precious to be analysed destructively. Ion beam techniques have been widely employed in dentistry and their sensitivity for trace elements in various thick tissue specimens was attentively investigated [3–6]. Teeth are not homogeneous in elemental composition as demonstrated in earlier studies [7–9] and consequently thick targets of tooth enamel, caries, and cementum were used for analysis. It has been reported that the concentration of Pb in teeth can be used as an index of environmental pollution [10–12]. Lead is preferentially incorporated and stored in calcified tissue such as teeth [13].

Among various elemental analysis techniques, atomic absorption spectroscopy (AAS) and inductively coupled plasma mass spectroscopy (ICP-MS) are becoming more routine, but they are destructive chemical methods involving sample dissolution procedure and their inability for analysis of samples in solid form. The electron probe microanalyser (EPMA) technique is useful for the analysis of even a mono-mineral grain, but is limited to detection of elements at concentration more than 200 ppm. In this context, multi-elemental non-destructive accelerator based external proton induced X-ray emission (external PIXE) technique looks more useful for *in situ* analysis of samples. It is fast, simultaneous, reliable, quantitative, multi-elemental and non-destructive with an excellent sensitivity in the ppm level and detection limits across a wide range of atomic numbers [14]. On the other hand, the samples can be irradiated as such in air which is extremely useful in analysing any specific area of the samples. For all of these advantages, external PIXE technique was preferred for the current study for *in situ* analysis of the human teeth samples.

## 2. Materials and methods

### 2.1. Sample collection

Five carious human teeth samples were collected from the City Hospital, Berhampur, India from five individuals in the age group of 31–54. To keep contamination of the samples to a minimum, proper care were taken for the collection of specimens and their handling prior to the analysis. The samples were acid cleaned and

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washed with deionized water and then with alcohol. The specimens were sealed in individual plastic containers in deep freezer till the irradiation.

### 2.2. Sample irradiation

The *in situ* elemental analyses of the teeth samples were carried out in the external PIXE set-up at Institute of Physics, Bhubaneswar which is the unique of its kind in India and was installed by us in 2003 [15]. The proton beam of 3 MeV energy obtained from the 3 MV tandem type horizontal pelletron accelerator (Model: 9SDH-2, make: National Electrostatics Corporation, Madison, USA) was used for irradiation of teeth. The proton beam was collimated by a graphite collimator to a beam size of 1-mm diameter. The beam was extracted into air using a Kapton$^{TM}$ foil (8-μm thick) at the exit point of a vacuum scattering chamber [16]. The beam was first focused and centered at the target location inside the scattering chamber and then let through the thin Kapton foil placed at the exit port. The Kapton foil is used as exit window due to its several special characteristics like low beam-induced background emission, minimal energy loss and resistance to radiation damage. The beam was allowed to travel a few centimeters in air after which it irradiates the samples. Beam charge measurements were carried out by using a rotating vane chopper designed by us [17].

The samples along with the NIST (National Institute of Standards and Technology) Bone Ash international standard were irradiated with maximum beam current of 15 nA. An Si (Li) detector (active area 30 mm$^2$) having energy resolution of 170 eV at 5.9 keV placed at 90° with respect to the beam direction was used to detect characteristic X-rays emitted from the target [15]. The detector has an entrance beryllium window of 8 μm thickness. A 25-μm thick aluminium absorber (with 6% hole) was kept in front of the detector to attenuate the bremsstrahlung background and the dominant low energy X-ray peaks [18]. Spectra were recorded by using a PC based multi channel analyser. The PIXE spectral analyses were performed using GUPIX-2004 software. This provides a non-linear least square fitting of the spectrum, together with subsequent conversion of the fitted X-ray peak intensities into elemental concentrations, utilizing the fundamental parameter method (FPM) for quantitative analysis [19]. The uncertainty in the concentration estimation for the major element is 0.1–0.2% for Ca and P whereas for other elements it is found to be between 8% and 13% [20–21].

## 3. Results and discussion

The external PIXE spectrum of the enamel of a tooth are given in Fig. 1. Ten elements namely P, Ca, V, Mn, Fe, Cu, Zn, As, Sr and Pb were estimated in the current study. It can be observed from Table 1 that the respective concentrations of elements namely P and Ca in enamel are more than those in cementum. Concentration of P ranged between 6.37% and 25% whereas Ca ranged between 12.94% and 43.36%. Since human teeth contains hydroxyapatite [$Ca_{10}(PO_4)_6(OH)_2$] as their primary inorganic constituent which contains calcium and phosphorous in it, hence the higher amount of Ca and P is obvious in the teeth samples. Ca and P showed less concentration on caries as compared to enamel and cementum, whereas they showed highest concentration on enamel. The decrease in Ca and P concentration on caries are due to presence of contaminants deposited during food intake and due to depletion of hydroxyapatite. On the other hand, the higher concentration of Ca and P in tooth enamel may be attributed to the higher concentration of hydroxyapatite in them because human dental enamel is the hardest tissue in the body with 92–96% of inorganic matter, 1–2% of organic material and 3–4% of water in weight [22]. The concentrations of Ca and P by weight percentage should be 39.8 and 18.5 respectively for a stoichiometric hydroxyapatite with a Ca/P weight ratio of 2.15. On this basis, the concentration of Ca in enamel in the teeth samples is close to this value and P concentrations show slightly higher values. While the concentration of Ca in cementum varies between 20.1% and 30.2%, P concentrations don't show much variation as compared to the theoretical values. On the other hand, Ca and P elemental concentrations are severely depleted in the carious region. Calcium salts provide rigidity to the

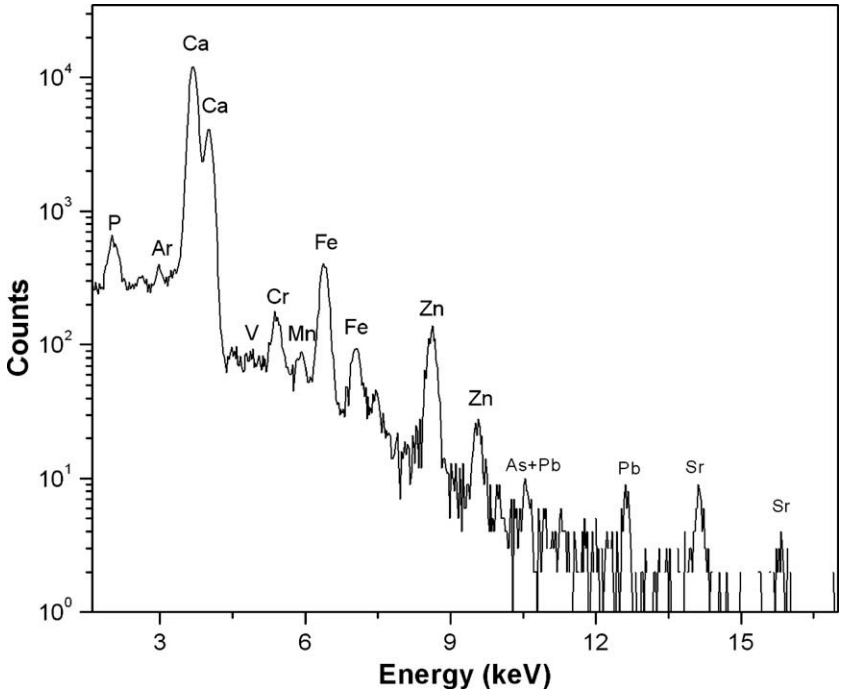


**Fig. 1.** External PIXE spectrum of a tooth enamel.