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Studies of some elements in urinary stones by proton induced X-ray emission (PIXE) technique

N K Sharat Singh[1], H N K Sarma[1]*, Sanjeev Kumar[2] and J Arunachalam[2]

[1]Department of Physics, Manipur University, Canchipur, Imphal-795 003, Manipur, India

[2]National Centre for Compositional Characterization of Materials (CCCM), Hyderabad-500 062, Andhra Pradesh, India

E-mail hnksarma@rediffmail.com

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Abstract : The levels of trace elements in urinary stones were investigated by PIXE technique at CCCM, Hyderabad using protons of 2.5 MeV energy. Some of the common elements detected were Ca, Fe, Cu, Zn and Sr and the distribution of the said elements were observed at various region of the stone.

Keywords : PIXE, urolithiasis, trace element

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PIXE has been widely used in various branches of sciences including biological [1–3], geological [4], environmental [5] and archeological [6] purposes since its practical use of analytical purpose started in 1970 [7]. In Biological Science, its important role is to investigate the trace elements in the biological systems such as in tissues for its multi-elemental detection capability, high sensitivity and non-destructive elemental analysis. Moreover, it can analyse most of the elements having atomic number greater than 15 [8].

Human tissues are made up of many elements. Such constituents of living materials are usually divided into three main groups [9] : the major elements, composed of H, C, N, O, Na, Mg, P, S, Cl, K, Ca; the trace elements, composed of F, Si, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Se, Mo, Sn and I; toxic elements, composed of Be, Hg, Pb, U and transuranium elements. Out of these groups, trace element have an important function in biological systems and take a major role in the smooth functioning of the organs for the life process. The deficiencies or excesses of certain trace elements may lead to many diseases and pathological disorders. One of the hazardous, painful disease in human is stone formation which is common in both sexes. The stones are formed in urinary tract, kidney, gall bladder *etc* and calculi in urinary tract have been known to occur in man and animal since antiquity. The incidence of urolithiasis is fairly high in south east including several regions of India [10]. Manipur is a north-east state of India, adjoining Myanmar with a population over 22 lakhs and is facing an acute problem of urolithiasis, hence urgent attention is needed.

Some eleven stones removed from different patients were collected from Regional Institute of Medical Sciences (RIMS), Imphal and washed with distilled water to remove possible blood constituents and dried at room temperature by keeping it in a dessicator. The stones were ground by an agate mortar. The powder samples (0.4 gm by weight) were mixed with pure graphite powder in the ratio 9 : 1. The mixture thoroughly ground, homogenised with binder 200 μl (polyvinyl alcohol + distilled water) and pressed into pellets of thickness 2 mm, diameter 13 mm with a pressure of 30 kN m^{-2}. A proton beam of diameter 3 mm, beam current 2 nA, energy 2.5 MeV from 3 MV Tandetron accelerator at National Centre for Compositional

Corresponding Author

Characterisation of Materials (CCCM), Hyderabad, India was used to irradiate the samples under vacuum condition (5×10^{-5}). The beam was diffused by an aluminium diffuser before irradiating the samples. The samples were fixed on the double sided adhesive tapes in the PIXE chamber at 45° to the beam direction and mounted on a ladder. A planar P-type Ge-detector (Euresis Mesures Type EGX 100-01, Be window thickness 40 μm) cooled at liquid nitrogen temperature (77° K) with full width half maximum (FWHM) of 155 eV at 5.9 keV was used to detect the characteristic X-rays emitted from the target. A polythene sheet of thickness 300 μm was used as an absorber (filter) and kept it in front of the detector to attenuate the bremsstrahlung background and prominent low energy X-ray peaks. The data was acquired on a Pc based MCA in 1024 channels. A typical spectrum of the stone sample is shown in Figure 1. The spectral data were analysed using the GUPIX-99 interactive software and coal fly ash (NIST standard) was used as a standard for the analysis, hence the concentration of the elements detected were found out. Pd internal standard [11] and pure single material as well as bone standard [12] were also reported for the analysis of the kidney stones.

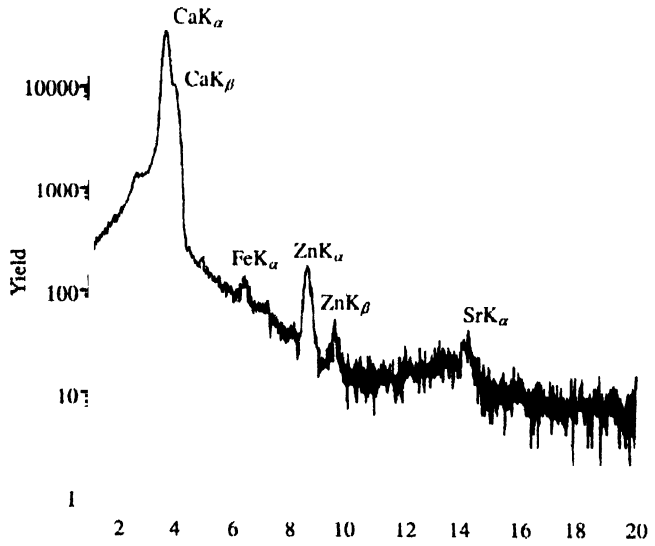

Figure 1. A typical PIXE spectrum of a kidney stone.

The elemental concentrations of the elements so far detected are shown in Table 1. As evident from the table, it is seen that Ca is found to be the major constituent element in all samples and other remaining elements *viz.* Fe, Cu, Zn, Sr in trace levels. This calcium is responsible for the formation of calcium carbonate ($CaCO_3$), calcium oxalate ($CaC_2O_4.H_2O$), hydroxyapatite [$Ca_{10}(PO_4)_6(OH)_2$], brushite ($CaHPO_4.2H_2O$) [13] in kidney stone. Moreover, the elemental concentrations are different for different

Table 1. Concentration of elements in kidney stones analysed at CCCM

Serial number	Stones removed from	Sample ID code	Concentration (Ca%, rest ppm)				
			Ca	Fe	Cu	Zn	Sr
1	kidney	Sl. 2	1.74	5.0	—	11.0	3.0
2	do	Sl. 4	2.6	—	—	8.0	8.0
3	ureter	Sl. 6	2.7	11.0	—	24.4	1.0
4	do	Sl. 9	7.7	25.0	—	10.5	1.2
5	do	Sl. 11	0.7	8.0	—	75	3.5
6	do	Sl. 12	2.8	2.0	—	29.0	2.1
7	kidney	Sl. 17	1.67.	8.0	5.0	56	0.8
8	do	Sl. 20	19	10.0	18.0	22.0	1.7
9	do	Sl. 21	5.8	—	—	4.0	—
10	do	Sl. 24a	0.4	—	—	—	—
		Sl. 24b	1.9	—	—	4.76	—
		Sl. 24c	0.195	1.2	—	—	—
11	do	Sl. 25a	0.0071	2.0	—	—	—
		Sl. 25b	0.0064	14.0	3.7	2.0	—
		Sl. 25c	0.2	86.0	14.5	12.3	2.8

Note . Serial number 10 and 11 are big kidney stones weighing 116 gms and 108 gms respectively. The concentration of these stones are investigated at different sites: 24a, 24b, 24c represent inner (core) region, middle region (region in between core and outermost surface), outermost region respectively and so on.

stones irrespective of both sexes. In particular, samples (Sl. 24 and Sl. 25) were analysed by selecting three different regions *i.e.* inner, middle outer regions. Here, the elemental concentration is also found to be different for each of the regions and no perfect relation among the elements were found. This leads to the decision that the elements were not distributed uniformly in the entire volume of the stone which corroborates the earlier findings of Gallassini *et al* [14] where concentration variation was observed when scanned over different parts of the same stone. The change in zinc status reflects probability of immune response inhibition which might lead to progression of disease concerned [15]. Nevertheless, the formation of stone remains an enigma. There are many factors for its formation and some of them may be due to genetic, nutritional, and environmental factors. The most acceptable idea is that kidney stone formation starts due to the supersaturation of the urine. There are three mechanisms which are of importance in kidney stone formation. These are nucleation, crystal growth and aggregation [16] and nucleation refers to the birth of submicroscopic molecular species of critical size within the supersaturation solution [17].

Urinary stones were analysed by PIXE technique and the elements such as Ca, Fe, Cu, Zn and Sr were

detected out of which Ca was found to be the major constituent followed by Fe, Cu, Zn, Sr in trace levels. Further, the elements were not distributed uniformly in the stone.

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