Mineralogical and Morphological Studies on Human Urinary Stones

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ABSTRACT: The formation of human urinary stones is a worldwide problem for affecting the large number of people. The urinary stones are formed in the kidney due to the deposition of few minerals and crystalline materials. Human urinary stone samples were analyzed by powder X-ray diffraction (XRD) and Scanning electron microscopy-Energy dispersive X-ray spectroscopy (SEM-EDX). The present study has been carried out to identify the minerals and elements; those are present in the urinary stones and attempt to establish its morphological structure and elemental compositions.

Keywords: Human urinary stones; XRD; SEM-EDX; Minerals.

1. INTRODUCTION

Urolithiasis is the third most common urological disease affecting a large number of people in different age groups irrespective of gender (Ghosh et al., 2009; Knoll, 2007). In most of the industrialized countries, around 20% of population is suffering from urolithiasis (Marickar et al., 2009a; Taylor et al., 2004). In India, urolithiasis affects about 2 million people every year, and the incidence is comparatively low in southern India (Girija et al., 2007). Formation of urinary stones is the most painful and prevalent urological disorders of the urinary system (Davidson et al., 2005). Qualitative and quantitative estimations of human urinary stones containing calcium oxalate monohydrate, calcium phosphate, magnesium ammonium phosphate, calcium carbonate and uric acid can also be identified by FT-IR and FT-Raman techniques (Selvaraju et al., 2012a; 2012b). Different analytical methods such as XRD, XRF, PIXE, SEM with EDX and FT-IR can provide vital information regarding chemical and structural formations of urinary calculi (Uvarov et al., 2011; Carpentier et al., 2011; Galassini et al., 1989).

In the present study the minerals and morphological changes of different samples of human urinary stones is evaluated using XRD and SEM methods. The urinary stone samples were analyzed for the concentration of elements like Ca, Cu, Fe, K, Mg, Na, P, S and Zn by EDX method.

2. MATERIALS AND METHODS

Human urinary stone samples were collected from Department of Urology, Rajah Muthiah Medical College Hospital (RMMC&H), Annamalai University, Annamalainagar, Tamil Nadu, South India. The samples were numbered sequentially S_1- S_4. The stones were washed with deionised water to remove debris such as blood, mucous and casts. Then the urinary stones were kept for further analysis. The oven dried at 60 °C for one hour to remove moisture content and then the dried stone samples are ground into a fine powder by using an agate mortar and pestle. The XRD patterns of the well powdered samples are characterized by powder X-ray diffraction analysis using an X-ray diffract meter model Rigaku D/max-2500 with monochromatic Cu-Kα source (λ=1.54056 Å) using tube voltage and current of 40 kV and 100mA, respectively. The samples are scanned over the range (2θ) 10°-80°. The X-ray diffraction of patterns of the urinary stone samples are obtained at Rigaku D/max-2500 XRD Diffractometer available at University of Madras, Guindy campus, Chennai Tamil Nadu, South India.

The morphology studies and elemental composition of the human urinary stone samples (S_1-S_4) are studied in JEOL-JEM-5610 LV Scanning Electron Microscope, equipped with an energy dispersive X-ray analyzer (OXFORD EDS) available at
Centralized Instrumentation and Service Laboratory (CISL), Department of Physics, Annamalai University, Tamil Nadu, South India.

Fig. 1. Human Urinary Stones

3. RESULTS AND DISCUSSIONS
3.1. Powder X-ray diffraction method

Human urinary stone samples were analysed for in this study using powder X-ray diffraction technique. In group I (S1-S4), of four urinary stone samples were found to be a mixture of calcium oxalate monohydrate (COM) and calcium phosphate (Apatite) with major constituents of calcium oxalate monohydrate and calcium phosphate in minor constituents. The observed results from XRD patterns are indicate high level of calcium oxalate and calcium phosphate (JCPDS-20-0231; 09-0432) (Wilson et al., 2010; JCPDS, 1977) and are known to crystalline in monoclinic and hexagonal systems. These results are good agreement with FT-IR results (Selvaraju et al., 2012a). The overlap of powder X-ray diffraction pattern of urinary stone in group I samples are shown in Fig.2.

The XRD patterns are observed results indicate this component mostly contains calcium oxalate monohydrate (COM) and calcium phosphate (apatite) is known to crystalline in monoclinic and hexagonal system respectively. The minerals are identified to calcium oxalate monohydrate (PDF 20-0231) and calcium oxalate dehydrate (PDF 17-0541). Similar results already are reported (Wilson et al., 2010; ICCD, 2005; Orlando et al., 2008). From the XRD results, it may be concluded that the presence of whewellite and weddellite are observed in the human urinary stones.

Fig. 2. Powder X-ray diffraction pattern of human urinary stones (S1-S4).

3.2. SEM-EDX analysis

Human urinary stone samples (S1-S4) are analyzed in this study using scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX). Fig. 3 (S1-S4) shows the morphological of human urinary stone samples and the results indicate that calcium oxalate monohydrate and calcium phosphate crystals are appeared in the spherical and irregular granular crystalline shape and its confirm the presence of calcium oxalate monohydrate and calcium phosphate (Wilson et al., 2010; Marickar et al., 2009b).

Fig. 3. Scanning electron micrographs and EDX spectra of human urinary stone.

4. STATISTICAL ANALYSIS

Statistical analysis is performed using SPSS software program, version 11.5. The results are expressed as mean and standard deviations. A probability level (p-value) of less than 0.05 is considered statistically
significant. This analysis is carried out with 99% confidence level.

4.1. One Way Anova

Comparisons of elemental concentration of human urinary stones are performed. While comparing five groups of urinary stones, we can observe that the probability value is less than 0.05 level of significant. Table 1 and Fig.4 show the mean and standard deviation and the relative distribution of trace elements in human urinary stones. It reveals that the presence of calcium and phosphorus are in predominant elements and moderate concentration of magnesium, copper, sodium and whereas concentrations of iron, sulphur and potassium are in smaller amounts.

Table 1: The mean and standard deviation of trace elements in human urinary stones

<table>
<thead>
<tr>
<th>Element</th>
<th>Mean</th>
<th>Standard Deviation</th>
<th>p-value</th>
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<tr>
<td>Calcium</td>
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S- Significant; p<0.05 at level of significance.

5. CONCLUSIONS

The present study indicates that the human urinary stones are having different morphological structures and minerals of calcium oxalate monohydrate, calcium oxalate dihydrate, calcium phosphate, calcium phosphate hydroxide, struvite (magnesium ammonium phosphate) and uric acid, respectively, were identified by XRD and SEM-EDX.

The correlation between calcium and certain elements are relevant to the process of stone formation has been established. Finally, established both XRD and SEM-EDX can be used as analytical tools in obtaining valuable information for the nature of crystalline structure and morphological variability of the elemental composition of urinary stones.

REFERENCES

7. JCPDS (1977), Joint Committee on Powder Diffraction Standards.