Compositional and morphological analysis of kidney-stones using PXRD and SEM

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Stone formation in human urinary tract, commonly called kidney stones, is a major medical problem around the world. A combination of powder X-ray diffraction (PXRD) and scanning electron microscopy (SEM) has been used to analyze the compositional and architectural variations of 50 kidney stones retrieved from patients of eastern India. Quantitative phase analysis has been carried out following the Rietveld analysis of PXRD data collected with CuKα radiation using a Bruker D8 Advance Diffractometer operating in the Bragg-Brentano geometry [1]. The urinary calculi samples analyzed can be classified into four groups on the basis of quantitative estimates of constituent phases as (1) oxalate (whewellite, weddelite or mixture of whewellite and weddelite being the major component i.e. > 50 wt%) (2) uric acid (anhydrous/hydrated uric acid and ammonium acid urate or their mixture accounting for > 50 wt%) (3) phosphate (struvite, hydroxyapatite or their mixture constituting > 50 wt%) and (4) mixed (when none of the above groups having composition > 50 wt%). The present study reveals that oxalate group is the most abundant type (82%) of kidney stones in eastern India, followed by uric acid (12%) and phosphate (4%). Among the oxalate group, about 40% of kidney stones (16 out of 41) were monophasic i.e. composed exclusively of whewellite (calcium oxalate monohydrate). Structural characterization of different regions (core, middle and outer layers) of five representative kidney stones clearly demonstrated compositional variation along the radial direction i.e. from the nucleus towards the periphery, with the core part composed of 100 wt% of whewellite, while other regions were mixture of different amounts of whewellite, weddelite, anhydrous uric acid, hydroxyapatite, brushite and ammonium acid urate[2]. The SEM images of calculi samples show different levels of organization, resulting from an agglomeration of crystallites having diverse shapes and sizes. The plate-like and octahedral bipyramidal geometries of whewellite and weddelite crystallites are apparent in the SEM images of kidney stones containing these phases. While the characteristic rhomboidal blocks of uric acid crystals are clearly visible in the SEM micrograph of calculus containing 98% of anhydrous uric acid, the hydroxyapatite crystallites exhibit a spherulitic morphology. The methodology described can provide precise information about the quantitative phase composition as well as possible growth mechanism of urinary calculi. The initial stage of stone formation is mostly related to hyperoxaluria/ hypercalcicuria, which increases the calcium oxalate supersaturation in urine to induce nucleation of whewellite crystals. Subsequently, the nuclei consisting predominantly of whewellite crystallites would grow in size due to deposition of other minerals depending on the prevailing pathogenic conditions during different stages of stone formation.


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