



A comparison of techniques for analyzing and studying kidney stones and their accuracy in determining the most likely phases of common stones among patients: a review

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Abstract

Kidney stones are among the chronic diseases that remain prevalent throughout the world, with cases increasing annually. Crystals indicate the formation of these urinary stones, therefore they are one of the most significant factors in the creation of the final form of kidney stones. Despite the considerable number of studies that shed light on this disease, now that many concepts related to this illness remain unclear and incomprehensible, such as the relationship of phases to the sort of stone, or the correlation of the quality of trace elements to the type or form of the final stone. The analysis of kidney stones is the most important step that can be done after extracting the stones from the kidneys or the body. Since the chemical methods used in analyzing these components have been considered unhelpful, due to their inaccuracy, high error rate, the inability to determine the crystalline contents within these stones, so It has become crucial to use and highlight more reliable, more accurate techniques when analyzing these stones in order to fully understand the connection between the formation and phases of stones, as well the sort of the initial phase and the role of elements in the nucleation of these stones. The aim of this review is to compare the common methods used in previous studies and to report which techniques are highly reliable.

Keywords: Kidney stones, spectroscopy, phase, trace elements.

1. Introduction

Kidney stones are solid components consisting of organic and mineral growths inside the kidneys. Crystal components constitute about 95% of the proportion of stones in general, and the other percentages are other organic components (Gernot Schubert, 2006). Kidney stone disease is a serious common health problem that affects the health of patients, and it has been diagnosed as a common disease with an increasing risk in recent years (Knoll, 2007). Urinary tract stone disease has a statistical risk of infection of 1-20% for the lifespan population (López & Hoppe, 2010). While his reappearance and occurrence increases with each day (Chou et al.,

2007). The mechanism of formation of these stones is not fully understood (Miller et al., 2007), therefore the analysis of these stones and the comprehension of the initial phases is very substantial for a proper medical diagnosis first, and then in order to eliminate them. Therefore, the goal of this review is to summarize the techniques that have been used recently in analyzing all aspects related to kidney stones, as well as comparing these techniques with each other, in terms of the features on which they agree and those on which they disagree or present an error rate that differs from each other.

2. Classification of kidney stones based on their chemical composition

2.1. Calcium oxalate group (CaOx)

Calcium Oxalate Monohydrate COM (Whewellite) ($\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$), Calcium Oxalate Dehydrate COD (weddellite) ($\text{CaC}_2\text{O}_4 \cdot 2 \text{H}_2\text{O}$), and calcium oxalate

trihydrate ($\text{CaC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}$) are represent the polymorphism of the hydrates of calcium oxalate (Deganello, 1981).

2.2. Calcium phosphate group (CaP)

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There are three forms of CaP, apatite (basic calcium phosphate) $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH}, \text{CO}_3)$ is the most common either alone or mixed with crystals of CaOx

2.3.Uric acid group (U.A)

Supreme uric acid stones ($\text{C}_5\text{H}_4\text{N}_4\text{O}_3$), are compact, appearing like a central core of loosely aggregated anhydrous U.A crystals surrounding by radiating columnar anhydrous U.A crystals ordered in concentric laminations (Grases et al., 2000). Some

2.4.Struvite

Struvite stones, magnesium ammonium phosphate hexahydrate ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$), they are huge masses of orthorhombic 'coffin-lid'-formed struvite

2.5.Cystine

Cystine stones ($\text{CH}_12\text{N}_2\text{O}_4\text{S}_2$), are dense, amber-colored, somewhat opaque, and with homogenous centers. Upper magnification of the stone and urinary

stones, brushite (calcium hydrogen phosphate dihydrate) ($\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$), and whitlockite (tricalcium phosphate) (OCP) (Khan & Hackett, 1986).

stones display a compact outer layer surrounding a porous friable interior containing of anhydrous U.A, uric acid dihydrate and COM crystals mixed with organic material.

crystals surrounded by spherulitic carbonate apatite crystals and mixed with cellular debris, which frequently contain bacteria (Grases et al., 2000).

deposits exposes a single and unique hexagonal building of the this stone's crystals (Khan & Hackett, 1986).

3. Techniques used for the analysis of kidney stones

3.1.Chemicals' analysis

It is true that most scientific laboratories now do not use this technology and the reason is due to poor analysis and a high error rate. It is a very common method before the discovery of modern techniques in

which the error rate is lower, unlike modern methods, this technique cannot differentiate between chemical compounds (Kasidas et al., 2004).

3.2.Scanning electron microscopy

This technique produces high-resolution images of the surfaces of the examined material, where surface morphology and crystal structures can be identified. Its working principle is to use high-resolution power for point scanning by employing electron beams focused on raster lines (Vivek K Singh & Rai, 2014). SEM furnish details about the kind of the crystalline composition, crystals form, inner structure, components position, crystalline conversions, crystallite dimensions distribution, and specific data

on the near joining between crystals and the organic components as well on the association between various crystalline kinds (Charafi et al., 2010).

Daudon and colleagues studied the mesoscopic range of renal stone structures, specifically whewellite, by this technique (Daudon et al., 2009), and so it was used by Daudon as a diagnostic tool for primary hyperoxaluria.

3.3.Energy dispersive x-ray analysis (EDX) coupled with SEM

Or elemental distribution analysis (EDAX), X-rays are used in this technique, the percentages of the constituent elements of the samples examined through this device are obtained, and here lies the advantage that makes this device coupled with the SEM device, as the normal SEM device cannot obtain the percentages of ionic concentrations that make up the sample and thus knowing the crystals, their nature, and components. As a result of this coupling, a single system is produced through which a high imaging capacity can be obtained for the

samples to be studied and analyzed (Marickar et al., 2009). The spectrum of EDX microanalysis contains both semi-qualitative and semi-quantitative information (Scimeca et al., 2018).

Marickar and his colleagues studied the percentages of the concentrations of the elements by examining the stones with this technique, and thus knowing the morphological features of the crystals that make up all the stones (Marickar et al., 2009). While in another work, the stones were examined by EDXRF,

and the percentage of trace elements was determined in order to understand the role of these elements and their relationship to the patients' diet (Srivastava et al., 2014). Whereas to examining the shape and spatial distribution of the phases that were identified

in kidney stones, Ali and others used this technique to obtain results identical with other techniques that were applied, and the results were promising (Uvarov et al., 2011).

3.4. Particle-induced emission (PIXE)

The beam of heavy energetic particles such as protons with an energy greater than 1 MeV (sometimes up to 4 MeV) is used to induce the emission of X-rays of the element. What distinguishes this technique is its detection limits (usually at a concentration level of $0.1\text{--}10\text{ mg kg}^{-1}$), especially for materials with a low atomic number. One of the most important reasons that enables this technique in laboratory use is the possibility of combining the feature of providing spatial maps of the elements with high accuracy at the micrometer scale, in addition to the high sensitivity (Verma, 2007).

Pineda and his colleagues, used micro-PIXE technology to understand and clarify the concentrations of trace elements inside the stones, and thus study the statistical analysis of these elements for two groups of stones, one from Sudan, and the other from South Africa, respectively (Pineda-Vargas et al., 2009). While it was used in another study to obtain preliminary maps of the distribution of calcium oxalate within the kidney stones, and thus trace the patterns of trace elements inside the stones. Micro-PIXE technology gives important information by which to reveal the mechanisms through which the growth and stimulation of the renal calculi is carried out (Pineda et al., 1995).

3.5. Polarization microscopy (PM)

The principle of its work is the interaction of the crystals inside the kidney stones with its polarized light, and therefore the amount of refraction, or reflection, or double reflection of light, are all functions and parameters that determine the stone

components include minerals (Gernot Schubert, 2006). It requires grinding the stones, and it must be mixed with a liquid with an appropriate refractive index in order for the analysis condition to be achieved correctly.

3.6. Synchrotron radiation X-ray with computed microtomography μ CT

In the synchrotron accelerator, an electron is released within a circular path with a diameter ranging from 10 meters to 100 meters (and some are more than that), the magnetic field controls the path of the electron, and thus produces an electromagnetic spectrum with a wide field that includes X-rays, infrared and ultraviolet rays, etc. The reason for using this type of radiation is that the resulting radiation is more intense than that produced by conventional X-ray devices, thus obtaining a monochromatic beam with high concentration and very low spacing, in addition to the synthesis and high brightness, the characteristics of this technique make it a very important tool in the study of

distribution maps of trace elements within renal calculi (Manzoor et al., 2019).

Stevens and colleagues used μ CT to study the mechanism of stone formation within the Randall plaque and the role of apatite in the growth process within the Randall plaque (Stevens et al., 2015). While Kaiser et al. used μ CT to investigate the micro-structure of minerals localized in nephrolithiasis (Kaiser et al., 2011). Manzoor et al. worked on the study of the texture and pattern of trace metal deposition in addition to the work of 3D analysis of the porosity and spatial variance of kidney stones by SR- μ CT (Manzoor et al., 2017).

3.7. X-ray absorption spectroscopy (XAS), and X-ray absorption near-edge spectroscopy (XANES)

Synchrotron radiation sources have effectively helped to understand materials of very little order, including trace elements, due to the high brightness of these rays (Chassot et al., 2001). Forming a perception about the nature of the relationship between the structure of the mineral elements that make up the stones and their locations inside the stones or even within the mineral proteins, and

therefore this technique allows us to analyze the elemental without the interference of absorption (Poswal et al., 2012).

As for (XANES), it is an easy method that employs synchrotron light to determine the valence of the excited atoms, as well as the ability to determine the electrical capabilities of the atoms surrounding the

excited atom. Thus, it is a good way to know the chemical bond by using the XANES spectra X-ray absorption spectroscopy is particularly valuable for describing biological calcium phosphates for several valued explanations. In the beginning, calcified deposits are frequently low crystalline and therefore hard to characterize through conventional techniques. In addition, probe's size is very adequate to create a mapping of a biological sample without any

3.8.Thermogravimetric (TGA)

The principle of work of this method is to record the changes of each: the temperatures, as well the weight loss of the samples continuously until it reaches 1000 degrees. And because each material is characterized by its own thermal absorption, by knowing the initial and final temperature and the amount of weight change in comparison, the proportion of the constituent elements of the samples is known (Kasidas et al., 2004).

This method was used by many researchers and was reported in their literature, Rose and others

particular preparation. In consequence, unique force point of X-ray absorption spectroscopy (XAS) is that the chemical state of the pathological calcification is conserved (Daudon & Bazin, 2012). Russell et al. studied stones with X-ray absorption near-edge spectroscopy (XANES) and obtained unknown compounds around the K edges of sulfur, phosphorous, and calcium.

recommended this method for the analysis of kidney stones because of its speed (ROSE & Woodfine, 1976), while D'Ascenzo and others in their work, they reported the results of differential thermal analysis (DTA) by TGA, thus coming up with the DTA-TG technique (D'Ascenzo et al., 1983), Which has great accuracy in addition to the speed of the examination, while Materazzi et al., reported that coupling with FTIR gives the results of identifying and recording the fine decomposed material at each specified temperature (Materazzi et al., 1995).

3.9.Laser-induced breakdown spectroscopy (LIBS)

A microplasma with a nano-life (very short) is generated, while the samples to be examined are bombarded with a high-energy laser. During this procedure, the molecules are separated in the atomic and ionic form to generate the emission spectrum. This emitted spectrum from the plasma is a distinct case for the excited state of atoms, and thus obtaining the properties of the constituent elements of the sample by means of emission (Parigger et al., 2003).

This optical technique is an effective method for sensitive and rapid analysis of kidney stones (Vivek Kumar Singh & Rai, 2011). While the samples were examined in another work in order to obtain the spatial distribution of the trace elements within the renal stones, so that this technique is described as a very suitable technique in order to obtain integrated information about these elements(Vivek K Singh et al., 2009).

3.10.Infrared spectroscopy IR- FTIR

In short, in this spectroscopy, the samples to be examined are irradiated with infrared laser pulses, and the reason is to create vibrations within the atoms of the samples, and depending on the absorption spectrum of the examined samples, these spectra are recorded and finally analyzed to find out the initial components of the samples (Kasidas et al., 2004). Sample preparation for this method is effortless, as it does not demand mixing the specimen with an infrared inactive material, for instance, potassium bromide, prior to investigation. Even in problematic cases in which a single water molecule divides the two compounds (weddelite and whewellite), calcium oxalate identification is extraordinarily easy by infrared spectroscopy (Daudon & Bazin, 2012) .

Channa and his colleagues used this technique to come to the conclusion that calcium oxalate and uric acid stones were the most prevalent stones in patients in Hyderabad and neighboring areas (Channa et al., 2007). Sandra Tamosaityte, emphasizes the importance of this technique by studying a number of kidney stones and examining their chemical composition using this technique, as it enables the effectiveness of this technique in terms of being sensitive to organic and inorganic components as well. In addition to their conclusion that the analysis by FTIR spectroscopy showed that the similarity between the stones, or even the difference between some of their properties, does not mean the possibility of saying that they are of the same types, and vice versa (Tamosaityte et al., 2013).

3.11.Powder X-ray diffraction (XRD)

The principle of X-ray radiography devices is to rely on recording the intensity of the rays passing through the examined samples by detectors, and thus the internal structures of the sample become visible due to the different absorption of the inner layers inside the sample (Spieř et al., 2009). The obtained diffraction patterns include additive contributions of various microstructural and macrostructural characteristics of a specimen. By the position of the peak, most of the contributions can be investigated, such as lattice parameters, space group, chemical composition, macro stresses, or qualitative phase

analysis. Rely on the intensity of the peak, detailed knowledge on the atomic positions, temperature factor, or occupancy, as well as texture and quantitative phase analyses, easy to be acquired (Dinnebier & Billinge, 2008). Kidney stones have also been described using X-ray diffraction (XRD) techniques by many researchers (Ancharov et al., 2007) (Daudon et al., 2009) (Orlando et al., 2008) (Uvarov et al., 2011), the goal is to determine the final stages of kidney stones and thus identify the mechanism of formation of these stones and help doctors give or describe the treatment.

3.12.X-ray fluorescence

X-ray fluorescence spectroscopy is mostly utilized to know the qualitative and quantitative chemical analysis, specifically, in electron microscopes. XRF spectrometry is dependent on the wavelength-dispersive standard, which is that single atoms emit a relative abundance of X-ray photons of energy or wavelength characteristics which will be evaluated (Oyedotun, 2018). The beam coming from the source ejects the atom's electron from its internal orbitals, as a result of this process an excited atom is produced, and thus this atom emits a high-energy beam to get rid of the excited state. The emission of these rays is distinct, and therefore the intensity values of these rays can be combined to find out the varying levels of intensity, which in turn will be transformed into

information about the concentrations (Weltje & Tjallingii, 2008).

Singh and others, studied the multiple components of kidney stones by WD-XRF, and qualitative and quantitative determination of all major and trace elements was performed (Vivek K Singh et al., 2017). While Oztoprak and his colleagues, used this technique for comparative analysis, studying the ratio of hydrogen to carbon inside the samples, and thus important news about organic compounds such as uric acid and its relationship to the primary formations of gravel.

Table 1. Comparison of the different methods used recently in the analysis of kidney stones

Technique	Advantages	Limitations
WCA	<ul style="list-style-type: none"> • low cost • Easy to apply 	<ul style="list-style-type: none"> • low accuracy-destructive • No capability to crystals recognition • Only the chemical components can deferment
ESEM	<ul style="list-style-type: none"> • Non-conductive, no coating needing • Pressure controllability for good spatial accuracy • ESEM images permit a good observation at the mesoscopic scale of the whewellite which forms the urolithiasis stone (Daudon et al., 2009) 	<ul style="list-style-type: none"> • Does not provide a clear definition of the sample composition • Expensive, time consuming • Cannot distinguish between crystalline and non-crystalline materials (Amidon et al., 2009)
TGA	<ul style="list-style-type: none"> • Fast and accurate quantitative results (ROSE & Woodfine, 1976). • Determine the age of the stones (ROSE & Woodfine, 1976) • Quantitative determination of each hydrate of the calcium oxalate when present together with uric acid or magnesium ammonium phosphate (Kaloustian et al., 2003) • suitable combination with XRD (Basiri et al., 2012) 	<ul style="list-style-type: none"> • Difficulty in identifying the components of the stone due to the disintegration of compounds at high temperatures (Kasidas et al., 2004) • Destructive the samples
PM	<ul style="list-style-type: none"> • Low cost, very fast analysis • Requires a small amount of sample • Very effective with stones such as weddellite and whewellite 	<ul style="list-style-type: none"> • Require a high subjective experience • The components of uric acid, purine groups and calcium phosphate cannot be identified (Gernot Schubert, 2006)

FT-IR	<ul style="list-style-type: none"> Determine the spatial distribution of mineral components (Daudon & Bazin, 2012) Identification of non-crystalline materials (Lehmann et al., 1988) Require small amount of samples to analyze suitable combination with TGA or SEM (Basiri et al., 2012) 	<ul style="list-style-type: none"> Difficulty in qualitative analysis in stones containing uric acid and calcium phosphate (Gernot Schubert, 2006) Time consuming in the case of potassium bromide (Gernot Schubert, 2006) Difficulty identifying some small components such as urate, uric acid dihydrate, whewellite, and weddellite or reverse, etc (G Schubert, 2006)
LIBS	<ul style="list-style-type: none"> Simple, inexpensive, compact, portable, Liquids, solids, gases, no preparation, Simultaneous all-channel spectral acquisition (Hahn & Omenetto, 2012). Capability to reveal high and low atomic number elements 	<ul style="list-style-type: none"> Destructive (Hahn & Omenetto, 2012) The overall detection efficiency is relatively low (Hahn & Omenetto, 2012) Damage caused by high-energy laser pulses
EDX-SEM	<ul style="list-style-type: none"> High spatial resolution and the ability to analyze small sample areas (Linke et al., 2004) non-destructive (Linke et al., 2004) 	<ul style="list-style-type: none"> Limited in the sample size. The detection limits of SEM/EDX are approximately 0.1 wt% (Linke et al., 2004) Low sensitivity to trace elements and to elements with atomic number less than 11 (Linke et al., 2004) Expensive
μCT	<ul style="list-style-type: none"> Higher resolution details in 3-D (Yin et al., 2013) Three-dimensional quantitative analysis of porosity and spatial variation of intact renal calculi stones (Manzoor et al., 2019) Provides high spatial resolution and chemical specificity as well as with minimal sample manipulation (Zhu et al., 2014) 	<ul style="list-style-type: none"> No nm scale investigation Low accuracy Require a subjective experience Does not support analysis for chemical composition
XANES	<ul style="list-style-type: none"> smaller amount of each sample Provide information on chemical bonds in the coordination shell, specifically the second coordination (Siritapetawee & Pattanasiriwisawa, 2008) Accurate description of the average electronic state (Daudon & Bazin, 2012) Geometric information about first neighbors (Daudon & Bazin, 2012) 	<ul style="list-style-type: none"> Difficulty distinguishing between scattered atoms that have close atomic numbers Difficulty knowing the number of backscatterers
PIXE	<ul style="list-style-type: none"> Non-destructive High sensitivity and quick results (Carmona et al., 2010) generate elemental maps (Pineda & Peisach, 1994) low background radiation (Miranda, 1996) lower secondary excitation in thick targets (Miranda, 1996) 	<ul style="list-style-type: none"> high uncertainties in fundamental constants (Miranda, 1996) effect of energy and lateral straggling (Miranda, 1996)
XRD	<ul style="list-style-type: none"> Non-conductive, reliable Accuracy Easy preparation Differentiation of whole crystalline ingredients 	<ul style="list-style-type: none"> Detection limit Expensive No nanocrystalline detection Struggle to recognize compounds in situation of overlapping
XRF	<ul style="list-style-type: none"> Accurately in the qualitative and quantitative chemical analysis (Dinnebier & Billinge, 2008) Non-destructive 	<ul style="list-style-type: none"> high expensive Most of the commercial devices available are not able to give promising results in cases of materials containing less than 11 elements (Taggart et al.,

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- Simple sample preparation
 - Absorption depends on the atomic number, as in the case of excitation (Oyedotun, 2018)
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- 1770)
 - Nearly all initial differences are measured as numbers rather than concentrations (Taggart et al., 1770)
 - Low sensitivity to isotopes of the elements (Oyedotun, 2018)
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4. Results and discussions

We found that the XRD and IR (FT-IR) spectroscopic measurements for the analysis of chemical monotonies are the most widely used in the researchers' literature because they are relatively fast and accurate in addition to being non-destructive to the samples.

Most researchers consider thermometry (TG) to be the best method for the detection and characterization of calcium oxalate hydrate. Most researchers in the most common phase classification processes among CaOx and CaP stones rely on the scattered XRD technique.

We found that most researchers rely on EDX-SEM technology to document test results from other devices with EDX-SEM results to know and compare the results and know the extent of their validity, which gives a general idea that the EDX-SEM device was approved by most researchers because they used it to detect crystals and the type of phases in Gallstones.

Also, we note from the published literature based on the techniques referred to in the literature of this review, that only seven major components have an appearance equal to or more than 1%. The most common constituents are calcium oxalates, which are whewellite (monohydrate), and weddellite (dihydrate), with a recurrence rate of 68% and 43% respectively. The frequency of occurrence of apatite is 33%, while the percentage of bruchet does not exceed 2%, for stones of infection (struvite) is 6%, for uric acid is 10%, for uric acid dihydrate is 6% (uric acid monohydrate is not appearing0, for cystine is 0.3%, xanthine and dihydroxyadenine, are very occasional.

The comparison established in this review proved that most of the devices used cannot be the dividing line between the reality of the phases of kidney stones and the results that these devices provide us with, and the fact that the completion between the devices to detect the chemical content of phases and surface morphology can only be done by using assemblage devices.

5. Conclusions

There is no specific method or combination of devices that provides all the information required to determine the main components and characteristics of kidney stones . But the agreement between SEM-

EDAX and XRD, makes it the most common way to understand the chemical components and morphology of these stones.

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