Microwave dielectric spectroscopy of renal calculi: A large scale study on dielectric properties from 500 MHz to 18 GHz

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ABSTRACT

Inherent dielectric property discrepancy between biological anomalies and healthy tissue enables the microwave diagnostic and therapeutic technologies. To reveal this discrepancy, microwave dielectric properties of many different biological tissues are tabulated. Although the dielectric properties of biological tissues are well documented in the literature, the knowledge on microwave dielectric property behavior of the renal calculi is limited. This work presents ultra wideband dielectric properties of three renal calculi types between 500 MHz to 18 GHz to pave the way for possible application of microwave technologies for diagnosis, treatment, and prevention of urolithiasis. Microwave dielectric spectroscopy is performed on a total of 66 natural stone samples with open-ended coaxial probe technique. The samples belong to three commonly diagnosed renal calculi categories, namely calcium oxalate, cystine, struvite. Analysis of variance (ANOVA) test is performed on fitted Cole-Cole parameters and it was concluded that there is a statistically significant difference between the dielectric properties of the renal calculi types. A patient-to-patient statistical test is also performed and it was concluded that there is no statistical difference between the samples belonging to the same renal calculi category. To this end, based on the relative permittivity discrepancy between the renal calculi types, the category of renal calculi can be identified by measuring the dielectric properties of renal calculi with open-ended coaxial probe technique.

Index Terms — dielectric properties, open-ended coaxial probes, kidney stones, microwave dielectric spectroscopy

1 INTRODUCTION

WITH the advancements in microwave diagnostic and therapeutic technologies, dielectric properties of many different biological tissues as well as biological anomalies have been a research interest [1, 2]. Such anomalies include but not limited to hepatic malignancies, breast tumors, and blood glucose levels [3–5]. One such anomaly affecting up to 14% of the population globally is urolithiasis, known as the formation of stones in the urinary tract, commonly referred as kidney stone (it will be addressed as renal calculi throughout this paper) [6]. The exact pathogenesis of renal calculi is unknown; therefore, the prevention and treatment methodologies is still a research interest. Currently

the renal calculi are either left to be passed naturally or the stones are broken into pieces with lithotripsy treatment. In severe cases, the renal calculi are surgically removed. If the patient is previously diagnosed with urolithiasis, recurrence of the disease can be as high as 75% [7]. Therefore, the patient is advised to follow dietary restrictions after the treatment of urolithiasis. In addition to such recommendations, recurrence of the disease can be further prevented with medications and in the case of recurrence it can be treated through adoption of stone-type-specific measures.

Renal calculi type can fall under one of four major categories: calcium oxalate, uric acid, struvite, and cystine [8]. The type of the renal calculi is currently assessed via X-ray imaging which requires extensive sample preparation as well as an expert to decide the type of the renal calculi based on the crystalline structure of the discarded sample [9]. Determining the stone category is paramount in order to optimize the treatment and prevent the recurrence of the disease [10]. Thus, research on X-ray imaging of renal calculi is ongoing [11]. Although other techniques including chemical and infrared methods have been investigated to determine the renal calculi type, the potential use of microwave dielectric spectroscopy have not been investigated in the literature. To exploit the potential application of microwave technologies for diagnosis, treatment, prevention of the urolithiasis, there is a need to tabulate the dielectric properties of the renal calculi at microwave frequencies.

The dielectric properties of renal calculi at various frequencies have been reported in the literature to explore potential treatment methods and pathogenesis of the disease. Piezoelectric property of the renal calculi at 1 kHz is investigated in [12]. Similarly, in [13], dielectric properties between 10 kHz to 1.5 MHz are investigated to enable the non-invasive disintegration of the renal calculi via the Extracorporeal Shock Wave Lithotripsy (ESWL). The dielectric properties in both studies are given at very low frequencies. Also, during the measurements the samples were either heavily machined or they were obtained from the hospital in powder form and a tablet was formed by adding polyvinyl acetate. Additionally, the number of samples and dielectric property variation is not given for different renal calculi categories. In a more recent study [14], the dielectric properties of struvite grown with single diffusion gel method are reported for designating better lithotripsy parameters. The reported measurements were performed between 1 kHz to 1 MHz frequency range at different temperatures ranging from 30 C° to 80 C°. The reported studies do not cover the microwave frequency range.

To evaluate the stress effect of shock wave lithotripsy on renal calculi, five categories of renal calculi phantoms are characterized and dielectric properties of the phantoms are reported between 1 Hz to 1 MHz [15]. Phantom materials were preferred due to the laborious procurement process of natural samples. The dielectric properties of the phantoms are reported at very low frequencies and characterized by using the capacitance method.

Another group of studies reporting the dielectric property behavior of the renal calculi samples are performed in an attempt to reveal the pathogenesis of the disease in order to be able to offer treatment and prevention mechanisms. In [16], the DC conductivity of renal calculi at different temperatures is reported. Similar to the other studies, samples were obtained from patients, powdered, and enclosed in a container. In another study, the dielectric properties of the calcium oxalate type renal calculi grown in silica gel are reported between 100 Hz to 1 MHz for temperatures varying from 40 to 110 C° [17]. Dielectric properties between 1 Hz to 1 MHz for temperatures ranging from 40 C° to 100 C° of renal calculi in powder form obtained from the hospitals are reported in [18]. Both studies measured the capacitance of the system to calculate the relative permittivity of the renal calculi samples. These studies were all performed at low frequencies and does now represent the dielectric property behavior at microwave frequencies.

In [19], dielectric properties of artificially grown and naturally obtained three types of renal calculi between 2.2 to 2.9 GHz is given. Although this study presents an insight to the dielectric property behavior of renal calculi at microwave frequencies, the frequency range is limited and the number of samples and their sizes are not detailed in the reported study. Also, the resonant cavity technique is employed during measurements which requires machining of the stones. Therefore, the sample preparation is cumbersome and the measurements can only be performed at a narrow frequency range with the resonant cavity technique.

Microwaves can potentially be utilized for treatment, diagnostics, and prevention of urolithiasis. One potential end application of microwaves is the possibility of in-vivo dismantling large renal calculi with microwave heating via minimally invasive microwave ablation. Another medical application example is utilizing microwaves to determine the category of renal calculi which is currently performed via X-ray diffraction and requires laborious procurement procedures. To explore potential applications of these technique there is a need to tabulate the wideband dielectric properties of renal calculi. Especially the dielectric property discrepancy at microwave frequency range can enable fast and low cost assessment of the renal calculi type. However, the reported studies on dielectric properties of renal calculi at microwave frequencies gives limited information [20]. To this end, in this work the main goal is to tabulate the dielectric property discrepancy between different renal calculi samples to enable the development of microwave devices that can identify the type of the renal calculi. Towards this goal, the dielectric properties of three renal calculi categories, namely calcium oxalate, cystine, and struvite are tabulated between 500 MHz and 18 GHz. Dielectric properties are collected with the open-ended coaxial probe technique. The technique is widely used to collect broadband dielectric property data of biological specimens in laboratory environment.

The remainder of this paper is organized as follows. Section 2.1 describes the samples, methodology used for dielectric property measurements and fitted model is given in Section 2.3 and 2.4, respectively. Dielectric property measurement results are given in Section 3.1, uncertainty analysis is given in Section 3.2, Cole-Cole parameters fitted to the measured dielectric properties are given in Section 3.3, statistical analysis is given in Section 3.4, and a discussion is given in Section 3.5. Finally, the conclusions are drawn in Section 4.

2 MATERIALS AND METHODS

2.1 SAMPLES

Three different types of naturally occurred renal calculi was obtained from the Department of Urology, Cerrahpasa Medical School. The samples were collected from 20 patients, both male and female, by utilizing various treatment methods including Percutaneous Nephrolithotomy (PCNL). A total of 66 stones; that is, 14 calcium oxalate stones, 28 cystine, and 24 struvite were used during the study. Note that the collected number of samples were initially higher; however, part of the samples was excluded from the measurements due to having very small dimensions. A picture of stone samples is shown in Figure 1.

To collect the dielectric property data with open-ended coaxial probe technique, the probe tip should be fully in contact with the material under test. Therefore, it is advised to use the open-ended coaxial probe for solid materials with smooth surface. Since the renal calculi samples had rough surfaces, a measurement site from each stone was designated and filed to obtain a smooth surface. The diameters of the samples were approximately ranging from 0.5 to 2.0 cm.



Figure 1. Discarded struvite samples obtained from female and male patients.

2.2 MEASUREMENT SET-UP

Dielectric property measurements are performed with a slimform open-ended coaxial probe with an aperture diameter of 2.2 mm. The probe is connected to Agilent N5242A PNA-X Microwave Network Analyzer with an RF cable. Commercially available Agilent's 85070E software is used during the calibration and measurement procedure. An illustration of the measurement set-up is shown in Figure 2a and open-ended coaxial probe with the material under test is shown in Figure 2b.

2.3 MEASUREMENT PROCEDURE

The probe is calibrated with the standard open, short, and deionized water calibration procedure. The dielectric properties of methanol are measured to verify the calibration at the frequency of interest. Although open-ended coaxial probe measurement technique has several advantages such as broadband measurement capability and minimal sample preparation requirements, the technique suffers from high error rates [21]. Therefore, the measurements should be performed with meticulous care; that is, both the measurements and calibration should be performed multiple times. Calibration should be verified with a known liquid, the measurement from the same point and different parts of the sample should be collected, the microwave cable connection as well as probe tip should be inspected, the contact between the probe tip and sample should be ensured. To this end, after the calibration is verified with a known liquid (methanol is used in this work as a known liquid) the aperture of the probe is washed with distilled water and dried with a cotton cloth and carefully placed against the sample's smooth surface for measurement. A minimum of 5 measurements were collected from each measurement point. Multiple measurement points were taken whenever the sample surface was available since some samples had highly irregular rough surfaces. Total number of dielectric property measurements collected for calcium oxalate, cystine, and struvite are 70, 130, and 112, respectively. Note that all measurements are performed at 20 °C. Additionally, both cable connection and probe tip is inspected to ensure the measurement system is stable during measurements.

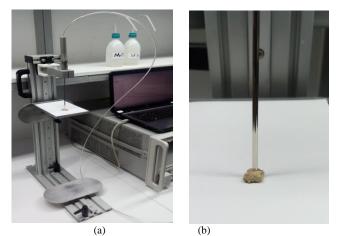


Figure 2. Dielectric property measurement set-up: (a) open-ended coaxial probe, PNA-X, and laptop computer, (b) open-ended coaxial probe with material under test.

2.4 MATHEMATICAL MODEL

Mathematical models such as Cole-Cole and Debye are used to represent the dielectric property behavior over a wide frequency range with a few parameters in the literature [22]. The mathematical models have also been utilized to identify the differences between samples [23]. In this work, Cole-Cole parameters was fitted by using Particle Swarm Optimization (PSO) algorithm. The method is detailed in [24, 25]; therefore, it is will be briefly explained in this paper. Cole-Cole Equation (1), is used to mathematically model the wideband dielectric property behavior of various materials including biological tissues with frequency dispersive characteristics. In this work, a single pole Cole-Cole equation is used to model the dielectric behavior of renal calculi for all three categories.

$$\widehat{\varepsilon_{(\omega)}} = \varepsilon_{\infty} + \frac{\Delta\varepsilon}{1 + (j\omega\tau)^{(1-\alpha)}} + \frac{\sigma_i}{j\omega\varepsilon_0}$$
(1)

where ε_{∞} is the relative permittivity at field frequencies, $\Delta \varepsilon$ is the difference between ε_s , the static permittivity, and ε_{∞} is the relative permittivity at high frequencies ($\Delta \varepsilon = \varepsilon_s - \varepsilon_{\infty}$), τ is the relaxation time for a dispersion region, α represents the broad distribution of relaxation time constant, and σ_i is the ionic conductivity. Euclidean distance, given in Equation (2), is calculated and given to the PSO algorithm as a function to evaluate the goodness of the fit.

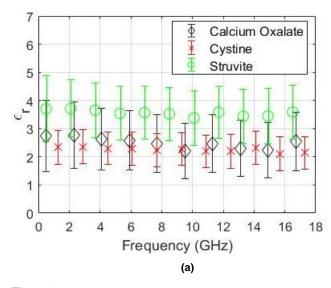
$$e = \frac{1}{N} \sum_{i=1}^{N} \left[\left(\frac{\varepsilon_{\omega_i}^{\prime} - \hat{\varepsilon}_{\omega_i}^{\prime}}{median(\varepsilon_{\omega_i}^{\prime})} \right)^2 + \left(\frac{\varepsilon_{\omega_i}^{\prime\prime} - \hat{\varepsilon}_{\omega_i}^{\prime\prime}}{median(\varepsilon_{\omega_i}^{\prime\prime})} \right)^2 \right]$$
(2)

where ε'_{ω_i} and ε''_{ω_i} are the measured dielectric properties, ε'_{ω_i} and ε''_{ω_i} are the fitted dielectric properties , and N is the number of points used within the frequency range of 0.5 to 18 GHz. If the Euclidean Distance is larger than the error threshold given to the algorithm, a new set of solution is produced by the PSO algorithm. During the Euclidean distance calculations 18 points (N=18) were considered. If the Euclidean distance is larger than the error

threshold given to the algorithm, a new set of solution is produced by the PSO algorithm. Otherwise, the algorithm stops and returns the fitted Cole-Cole parameters.

Euclidean distance was assessed as a fitness function for the algorithm since it is a measure used for determining the goodness of fitting. The goodness of fitting is further evaluated by calculating the root mean squared error (RMSE) and root mean squared relative error. Since both measured and fitted values are complex values to calculate RMSE and RMSRE, absolute value of the complex permittivity is calculated. The formulas for RMSE and RMSRE are:

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (\left| \varepsilon_{w_i} \right| - \left| \widehat{\varepsilon_{w_i}} \right|)^2}$$
(3)



$$RMSRE = \sqrt{\frac{1}{N} \sum_{i=1}^{N} \left(\frac{\left|\varepsilon_{w_i}\right| - \left|\varepsilon_{\widehat{w_i}}\right|}{\left|\varepsilon_{w_i}\right|}\right)^2} \tag{4}$$

where N is the number of frequency points (N=176 in this work), $|\varepsilon_{w_i}|$ is the absolute value of the measured complex permittivity, $|\widehat{\varepsilon_{w_i}}|$ is the absolute value of the complex permittivity calculated with fitted Cole-Cole parameters.

3 RESULTS

3.1 MEASURED DIELECTRIC PROPERTIES

The median of the relative permittivity and conductivity measurements for each renal calculi type with error bars are shown in Figure 3a and Figure 3b, respectively. The median is used instead of mean since mean is known to be sensitive to outliers. Error bars shows the standard deviation of the measurements. Median relative permittivity of calcium oxalate

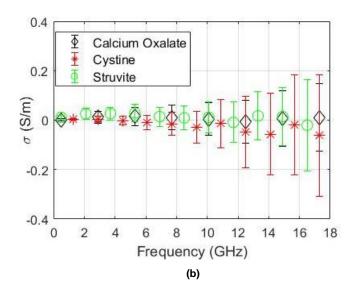


Figure 3. Median dielectric property measurements of calcium oxalate, cystine, and struvite obtained from patients; (a) median relative permittivity comparison (b) median conductivity comparison.

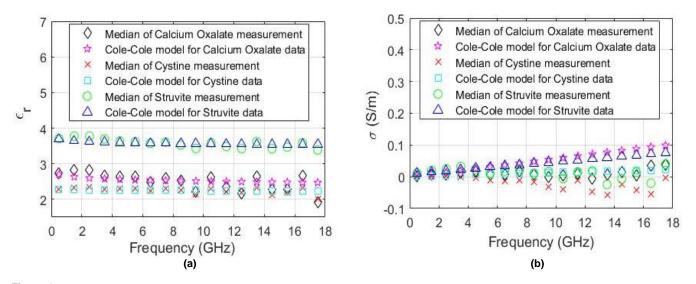


Figure 4. A comparison of fitted Cole-Cole model output and measured median complex permittivity for calcium oxalate, cystine, and struvite; (a) real part of the complex permittivity obtained from the measurement and Cole-Cole model, (b) conductivity values obtained from the measurement and Cole-Cole model.

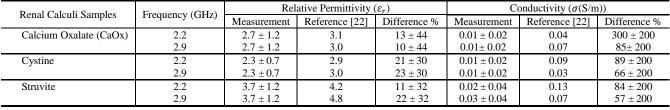


Table 1. Relative permittivity and conductivity comparison of measurements and literature data [22].

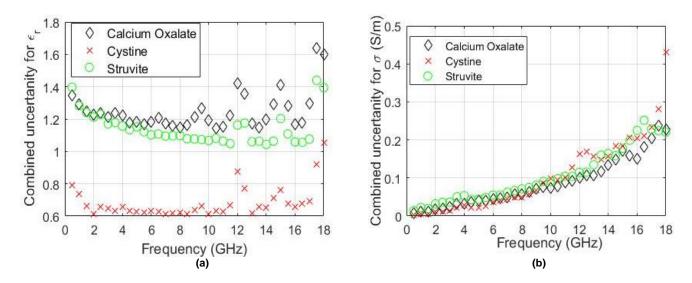


Figure 5. Combined uncertainties with respect to frequency for calcium oxalate, cystine, and struvite; (a) uncertainty in measured real part of the complex permittivity, (b) uncertainty in measured conductivity.

and cystine has a slight difference $(14\% \pm 39\%)$ between 500 MHz to 18 GHz. The relative permittivity of the struvite is higher than calcium oxalate by 27 ± 34% and cystine by 37 ± 7% between 500 MHz and 18 GHz. The median conductivity of all measured renal calculi are very similar; that is, close to 0.01 ± 0.02 at 2.2 GHz and close to conductivity of other low loss dielectrics such as wood.

Comparison of the measured dielectric properties with literature are given in Table 1. Note that only one study is reported on dielectric properties of renal calculi at microwave frequencies; therefore, the measured dielectric properties are compared with [22]. The median relative permittivity measurements are close to literature data (with a maximum difference of $23 \pm 30\%$) at reported frequencies as shown in Table 1. Cystine and calcium oxalate has close relative permittivity where calcium oxalate's relative permittivity is higher than relative permittivity of cystine. The median relative permittivity difference between cystine and calcium oxalate is $14 \pm 39\%$. This relative permittivity behavior also matches with the literature data as shown in Table 1. Reported relative permittivity of struvite is higher than calcium oxalate (1 ± 2.4) and cystine (1.4 ± 1.9) , also agrees well with the literature data. The median difference between the permittivity of struvite and calcium oxalate is $27 \pm 34\%$ and the median difference between struvite and cystine relative permittivity is 37±7%. Reported conductivity values of the stones are very low, similar to the measured conductivity values in this work as shown in Table 1.

3.2 UNCERTANITY ANALYSIS

The uncertainty of the measurements is calculated by evaluating Type A and Type B errors. Type A error is obtained by calculating the standard deviation of the measurements for each renal calculi category and it is divide by the square root of the number of measurements for each frequency. Similarly, Type B error is obtained for each renal calculi type and each measurement frequency by dividing the half distance between the maximum and minimum measurement with square root of three. The combined uncertainty is then calculated with Equation (5),

$$u = \sqrt{u_A^2 + u_B^2} \tag{5}$$

This method has also been used in the literature to calculate the uncertainty of dielectric property measurements [2]. Combined uncertainty for the relative permittivity and conductivity is given in Figure 5a and Figure 5b, respectively. Mean combined uncertainty for relative permittivity of calcium oxalate, cystine, and struvite are $\pm 1.22, \pm 0.67$, and ± 1.12 , respectively. Mean combined uncertainty for conductivity of calcium oxalate, cystine, and struvite are $\pm 0.09, \pm 0.1$, and ± 0.1 , respectively. Finally, one contributing factor to measurement uncertainty is the drift of the vector network analyzer (VNA). To minimize this error, the VNA is turned on three hours before the measurements.

3.3 COLE-COLE PARAMETERS

The Cole-Cole parameters are fitted to the median of each measured dielectric properties of renal calculi category. Fitted

Table 2. Cole-Cole parameters fitted to the median measurement results.

	Renal calculi Type			
Cole-Cole Parameters	Calcium Oxalate (CaOx)	Cystine	Struvite	
\mathcal{E}_{∞}	1.2	1.28	1.32	
$\Delta \epsilon$	3.79	3.22	5.59	
τ	12.87	20	1.34	
α	0.9	0.97	0.96	
σ (S/m)	0	0	0.01	
Euclidean Distance (%)	1.3	0.56	0.67	
RMSE	0.17	0.07	0.09	
RMSRE	0.07	0.03	0.03	

Cole-Cole parameters are given in Table 2. The ε_s parameter is reflecting the relative permittivity of the renal calculi at microwave frequencies where cystine has the lowest and struvite has the highest relative permittivity. This result is consistent with the measured relative permittivities. As it can be seen from Figure 3a, cystine has lowest relative permittivity values specifically at lower frequencies. It can also be seen on Table I the permittivity of cystine is smaller than both calcium oxalate and struvite by 0.4 ± 1.9 and 1.4 ± 2.4 , respectively at 2.2 GHz. The ionic conductivity parameter for all three categories are very low, the ionic conductivity of the struvite is slightly higher than other two categories and also in agreement with the measurements. Euclidean distance used as a fitness function input for the PSO algorithm is also given in Table 2 and it is lower than 1.3% for all three renal calculi categories.

The Cole-Cole model output along with the measured real part and imaginary part of complex permittivity are shown in Figure 4a and Figure 4b, respectively. Cole-Cole plots are in well agreement (see the error parameters in Table 2) with measured median relative permittivities. The conductivity measurement of the struvite slightly differs from Cole-Cole plot at higher frequencies. This could be due to the measurement error. Cole-Cole parameters can be utilized for practical applications to represent the dielectric property behavior with few parameters and to analyze the dielectric property behavior.

3.4 DATA ANALYSIS

To analyze the dielectric property behavior in the whole frequency range, the Cole-Cole parameters are fitted to median of the dielectric property data collected from each renal calculi sample. Note that only three Cole-Cole fittings were performed in the earlier section whereas in this part the Cole-Cole fittings are performed for each sample. The procedure explained in Section 2.4 is followed for fitting of the Cole-Cole model. Since more than one measurement was collected from each sample, the medians of the measurements are taken and the median is used as a reference for each Cole-Cole fitting. A total of 66 fittings were performed. Examples of the Cole-Cole fittings for Struvite samples are shown in Table 3.

The sources of variability in the dielectric properties is analyzed by using the Cole-Cole parameter value $\Delta \varepsilon$ which is the difference between the static permittivity ε_s and relative permittivity ε_{∞} . A nested analysis of variance (ANOVA) was utilized to compare patient-to-patient and sample-to-sample variability. The analysis was performed on all three renal calculi categories. To satisfy the distributional assumptions required for ANOVA analysis, close number of stones is selected from each patient. Note that the availability of these samples depends on the number of renal calculi per patient and renal calculi type of the patient. Therefore, for calcium oxalate, the number of stones for each patient was close enough; that is, ranging from 1 stone per patient to 4 stones per patient; therefore, all the stones were analyzed. However, for cystine and struvite, the amount of stone per patient was not similar. For cystine the number of stones per patient and for struvite there was two patients with 2 and 22 stones per patient, respectively.

 Table 3. Cole-Cole parameters fitted to the measurements collected from

struvite samples.					
Cole-Cole	Measurements collected from struvite				
Parameters	Sample 1	Sample 2	Sample3		
\mathcal{E}_{∞}	1.12	1.11	1.9		
Δε	4.48	5.97	4.46		
τ	7.64	7.41	9.92		
α	0.92	0.91	0.84		
σ (S/m)	0	0	0		
Euclidean Distance (%)	0.88	0.49	0.51		

A patient-to-patient ANOVA test is conducted to test the variability between patients of the same category. The test is performed at a 0.05 level of significance (alpha value). The statistical probability values are shown in Table 4 is higher than the level of significance. This shows that there is no significant statistical divergence in variability between samples belonging to same renal calculi categories. Indicating that the dielectric properties only depends on the renal calculi composition and thus it can be used to categorize the renal calculi.

	Renal calculi Type			
ANOVA	Calcium	Cystine	Struvite	
	Oxalate (CaOx)			
Total number	7	9	2	
of patients				
Total number	14	22	10	
of used stones				
p value \geq	0.11	0.21	0.80	

Table 4. Statistical probability values for patient to patient analysis.

Another ANOVA test was conducted to assess the variability between the categories at a 0.05 level of significance. A statistical probability value ($p \ge 0.03$) shows that there are statistically significant differences between the three renal calculi categories.

3.5 DISCUSSION

Development of microwave technologies for health care applications requires a broad knowledge of dielectric property behavior of biological materials at the frequency of interest. This work presents the dielectric properties of three renal calculi types. The median relative permittivity of cystine is slightly lower than calcium oxalate (14 ± 39 %. median difference in the whole frequency band) and the relative permittivity of struvite is higher than both calcium oxalate and

struvite by 0.4 ± 1.9 and 1.4 ± 2.4 , respectively at 2.2 GHz. The conductivity of all renal calculi types is very small at the whole measured frequency range.

Finally, the ANOVA test revealed that the variance of relative permittivity between renal calculi categories is statistically significant while there is no statistically significant difference found in same renal calculi category between patient to patient analysis. From the statistical analysis and the median differences in the whole frequency band between the relative permittivity of renal calculi types it can be concluded that by measuring the relative permittivity of renal calculi with openended coaxial probes and comparing the measurement with the data set, the renal calculi type can be identified.

Renal calculi are classified as calcium oxalate, uric acid, struvite, and cystine depending on their chemical composition. Indeed, the relative permittivity discrepancy between different renal calculi types stem from differences at molecular level. However, determination of exact relations between chemical composition of materials and the dielectric permittivity is subject of electrochemistry. On the other hand, the water content is a strong indicator for higher dielectric permittivity values for biological materials. When the molecular formulation of the struvite is analyzed, the molecular formula includes water molecule, which could be a contributing factor to higher dielectric properties than other renal calculi types.

Lastly, the renal calculi type can be separated by comparing the measured values with the existing data even with the reported uncertainty values. This could be achieved by repeating the measurements while following the best measurement practices or it could be possible to classify the renal calculi type via single measurement through implementation of classification algorithms.

4 CONCLUSIONS

Current medical protocols for management of urolithiasis requires determination of chemical composition of renal calculi, namely the category of the discarded renal calculi, to eliminate recurrence of urolithiasis. This is a necessary step to prescribe the preventive measures such as dietary restrictions and in some cases medications to the patient. The median relative permittivity discrepancy between cystine and calcium oxalate, struvite and calcium oxalate, struvite and calcium oxalate, struvite and cystine are 14 \pm 39%. , 27 \pm 34%, 37 \pm 7%, respectively between 500 MHz and 18 GHz. Based on the inherent dielectric property discrepancy, open-ended coaxial probe technique can emerge as an alternative technology to determine the renal calculi type which opens up the possibility of a faster, possibly low cost, and automized alternative technology to existing one.

ACKNOWLEDGMENT

This project has received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Sklodowska-Curie grant agreement No 750346.

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