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Metamaterial Sensors in Liquid Detection: An Illustrious Review

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Liquid detection has been one of the buzzwords that refer to sensing of various liquids, which include water, chemicals, oils, *etc*. This detection gains more significance with the advent of metamaterials. Metamaterials are proved well suited in antenna designing, cloaking devices, solar cells, and sensors. Many papers reported metamaterial sensors, which can detect liquid chemicals, oils, *etc*. through shift in resonance frequency. Very few papers reported the applications of metamaterial sensors in water treatment. Water purification is one of the prime essentials for the existence of healthy humankind. It can be used to remove unwanted chemicals, contaminants and gases from water. The process of water purification can be done with various methods such as thermal, adsorption, distillation, desalination, reverse osmosis, *etc*. The main aim is to produce water required for specific purpose. In this context, role of metamaterials in water purification is of prime significance. Keeping this in view, an attempt is made to review the importance of metamaterial sensors in liquid detection along with water purification and its importance in comparison with earlier techniques already available.

Виявлення рідини було одним із модних слів, які стосуються зондування різних рідин, до яких належать вода, хемікати, олії тощо. Це виявлення набуває більшого значення з появою метаматеріялів. Доведено, що метаматеріяли добре підходять для розробки антен, маскувальних пристроїв, сонячних батарей і давачів. У багатьох роботах повідомлялося про давачі з метаматеріялів, які можуть виявляти рідкі хемікати, масла тощо через зсув резонансної частоти. Дуже мало статей повідомляли про застосування давачів з метаматеріялів у обробленні води. Очищення води є однією із найважливіших умов існування здорового людства. Його можна використовувати для видалення з води небажаних хемічних речовин, забруднень і газів. Процес очищення води може здійснюватися різними методами, такими як термічні, адсорбційні, дистиляційні, опріснювальні, зворотня осмоза тощо. Основною метою є одержання води, необхідної для певних цілей. У цьому контексті роль метаматеріялів у очищенні води має першорядне значення. Зважаючи на це, зроблено спробу переглянути важливість давачів з метаматеріялів у виявленні рідин разом із очищенням води та важливість їх у порівнянні з попередніми, вже доступними методами.

Key words: metamaterial (MTM) sensors, liquid detection, water purification, sludge volume index, finite integration technique (FIT).

Ключові слова: датчики з метаматеріялів, виявлення рідини, очищення води, індекс об'єму мулу, метод скінченної інтеґрації.

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1. INTRODUCTION

It is not possible to think of any application related to metamaterial without Russian physicist Veselago (1968) who demonstrated negative refraction with tailored materials [1]. He indicated that negative permittivity and permeability values lead to negative refraction in a material. Such material was developed by Pendry after thirty years [2]. Materials tailored for electromagnetic properties beyond their inherent nature are called metamaterials (MTM).

MTM's find various applications in design of antennas, cloaking devices, sensors *etc.* to name the few. Antennas designed metamaterials exhibit lightweight, high gain, high bandwidth and maximum channel efficiency [3]. Cloaking makes objects to be invisible. This technique became prominent with the advent of metamaterials. Cloaking techniques involve acoustic or EM waves with current focus on extension of bandwidth for invisibility of specific objects. Cloaking is achieved by using metamaterials by deflecting certain frequencies [4–19].

Solar cells designed with metamaterials are of prime importance. They can reduce reflected light and increase incident light. At the same time, solar cells designed with MTM's have limited absorption for majority of the solar spectrum. Keeping this in view, various absorbers that utilize solar energy in effective ways were innovated with metamaterials. Semi-circular solar cells with 77% absorption of solar and 84% of visible regions were reported with Ni and SiN materials [20]. Solar cells designed with silicon and metamaterial reflectors are reported to have high efficiency [21]. Apart from these applications MTM's play vital role, when used as sensing devices.

2. METAMATERIAL SENSORS

Sensors have become part of our daily life. They can be designed with different materials; however, sensors developed from metamaterials find various advantages as compared to ordinary materials. In spite of some limitations on usage of metamaterials as sensors [22, 23], they are preferred. Broadband materials can be replaced with sensors as they can operate at single frequency. In addition, MTM sensors possess high sensitivity and resolution [24] making metamaterials significant in enhancing the sensors performance. Even though MTM sensors are widely developed for various applications in measuring humidity, temperature, *etc.*, designing and fabrication of MTM sensors in liquid detection plays a vital role. Liquid detection can be related to chemicals, acids as well as water, oil, *etc*.

3. METAMATERIAL SENSORS IN LIQUID DETECTION

Detection of chemicals having similar dielectric properties is a major task as the principle of detection depends on the change in dielectric parameters of the samples under investigation. In this context, an MTM-based sensor capable of detecting liquid chemicals between 8 GHz to 12 GHz was designed and fabricated. This was achieved by testing different designs using CST microwave studio in which a genetic algorithm was embedded and optimizing the required resonator dimensions.

Any MTM sensor generally consists of a resonator of designated shape and dimensions. Depending on the type of application, the structure of resonator is changed. The main principle behind the design of resonator depends on the frequency shift between simulated and experimental values or a comparison between similar samples in a particular band. Many resonators of different shape and dimensions were reported depending on the sensing application.

Figure 1 shows the MTM based sensor that operates in X-band with proposed dimensions (22.86×10.16 mm^2) that can detect liquid chemicals. As shown in this figure, it consists of three layers. The top layer is a *G*-shaped copper resonator deposited on either side of middle layer, which is FR-4 (Flame Retardant) substrate. The bottom layer is of 10 mm thickness, which can be used to keep the test sample. Figure 1, *a* represent a unit cell compatible with X-band waveguide, which correlates with experimental study.

Figure 2 shows the design of MTM sensor with two ports added on either side with necessary boundary conditions as per the simulation output. During simulation, various boundary conditions are applied in order to measure the *S*-parameters as per the measure-

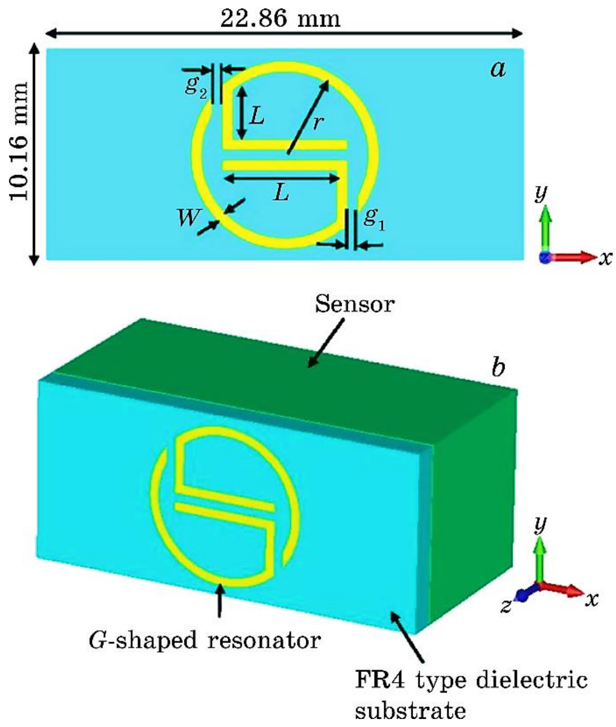

Fig. 1. MTM based sensor for liquid detection (reproduced with permission from [25]).

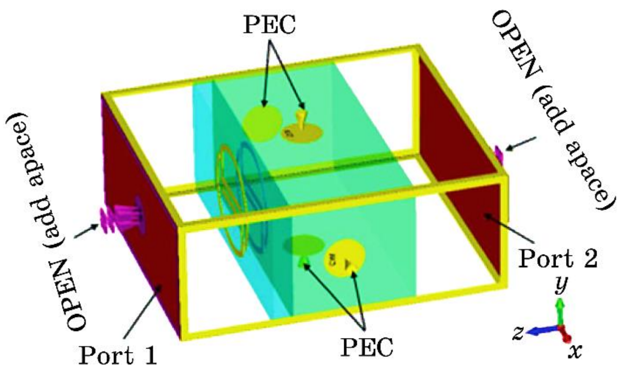

Fig. 2. MTM sensor with simulated boundary conditions (reproduced with permission from [25]).

ments of wave-guide WR-90 as well to assign x–y directions to boundary conditions of perfect electrical conductor (PEC) with z-axis for the direction of propagation.

Figure 3 shows the complete experimental setup with developed MTM-based sensor, waveguide configuration and sample holder with sample under study.

The above-mentioned experimental setup was used to measure transmission coefficient ($S21$) with liquid sample. Initially the sample was injected into holder and ($S21$) was measured between 8–12 GHz. This was repeated every sample. In addition, ($S21$) was calculated numerically by taking the dielectric properties of each sample. Both simulated and experimental results confirmed the ability of the designed sensor in detecting various liquids through resonance frequency shift of 250 MHz (transformer oil and diesel oil), 200 MHz (corn, cotton, olive oil, 150 MHz (ethyl alcohol substituted with aniline) and 50 MHz (carbon tetrachloride with benzene. This was achieved by developing a resonator in X-band. It was reported that this sensor could differentiate clean and waste transformer oils, branded and unbranded diesel oils by a resonant shift of 250 MHz even though their dielectric constants are very close of about (2.84 and 2.73) and (2.71 and 2.48). Similar measurements have confirmed 200 MHz shift for cotton, corn and olive oils with 3.2, 3.08 and 2.55 dielectric values. The experimental and simulated results are highly correlated confirming the ability of designed sensor in chemical detection. At the same time, it is of low cost, durable

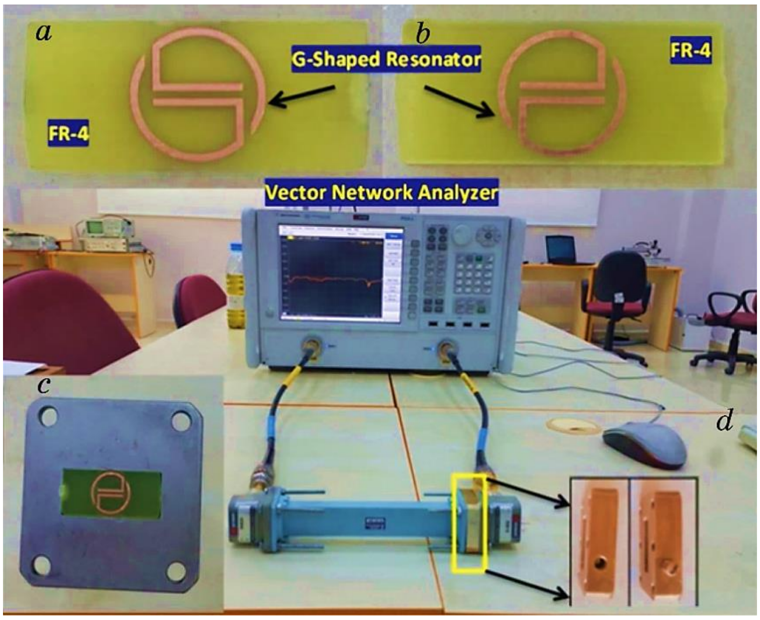

Fig. 3. Experimental setup of MTM sensor (reproduced with permission from [25]).

and highly accurate. This MTM sensor is of high importance in liquid chemical detection [25].

An MTM-based optical sensor for sucrose detection has been reported. The sensor was optimized by developing a metamaterial surface of broad wavelength using E-beam lithography. The detection accuracy of the sensor was very high as compared to an ordinary thin film sensor. The central wavelength of MTM sensor (967 nm) was much higher than the gold film sensor (933 nm). A big shift in wavelength (148.7 nm) between gold film and MTM sensors was reported. This large shift in wavelength is assigned to high resonance intensity experienced by metamaterial surface attributed to low particle size. These outputs confirmed better sensitivity with metamaterials [26]. In continuation, various MTM sensors are designed that include temperature sensors based on semiconductor metamaterials [27], sensors for sensing permittivity of alcohol, *etc.* [28] were reported.

In a similar way, a MTM absorber based on resonator of swastika shape resonator was designed. The sensing ability of this sensor at X-band frequency was demonstrated both theoretically and experimentally. This structure consists of a resonator of swastika shape on top of dielectric layer having gap to fill liquids under test.

Figure 4 shows an MTM-based sensor that can determine liquid chemicals and the ratios in water. It has a swastika shaped copper resonator of width 1mm placed on FR4 substrate with 1.6 mm thickness and 4.2 dielectric constant and 0.02 tangent losses. A reservoir of 3.5 mm thickness between copper plate and resonator was set up to place the chemical liquids for detection.

As every liquid chemical has its own electrical and permittivity values, shift in resonance frequency takes place by bringing a change in absorption value of MTM absorber. This is correlated with systems impedance matching. The swastika resonator is designed as per the numerical results, which have unique properties as compared to other resonators. It is also reported that compatibility between unit cell dimensions and X-band waveguide has been established.

Figure 5, *a* shows the fabricated MTM sensor with dimensions as per simulation output. The sample under study (Fig. 5, *b*) has been placed into the X-band waveguide. Figure 5, *c* shows the reservoir for liquid chemical. X-band waveguide is fed by VNA between 8 and 12 GHz for each sample separately. Simulation was done using finite integrate technique to find MTM structures absorption of liquid chemical depending on its electrical property. With the help of Vector Network Analyser, loss tangents and dielectric constants for acetone, ethanol, water, methanol, *etc.* are measured in specified frequency range. It was reported that absorption ratios significantly

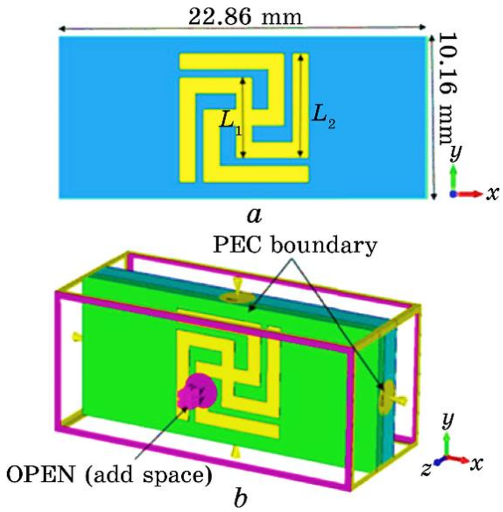

Fig. 4. Swastika-shaped MTM resonator (reproduced with permission from [29]).

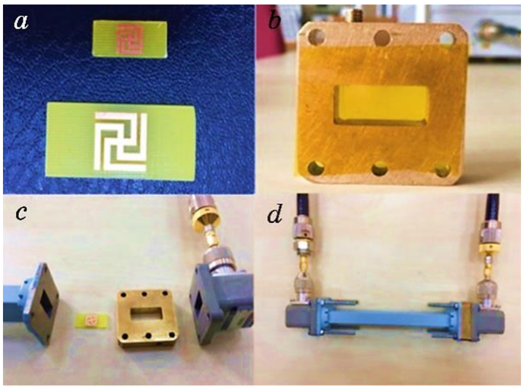

Fig. 5. (*a*)—Fabricated MTM sensor with simulated dimensions; (*b*)—X-band waveguide; (*c*)—reservoir (reproduced with permission from [29]).

differ between the investigating samples. In addition, the shift in resonance frequency estimates their density rate accurately. It is also demonstrated that absorption properties of the MTM sensor varies with resonator dimensions. Both observed and simulated results inferred the linear relation between resonance frequency and permittivity of swastika shaped MTM sensor for the samples under study. Since they are highly correlated, this sensor might be considered for detecting different liquid chemicals [29].

3. METAMATERIAL SENSORS IN WATER TREATMENT

Huge amount of wastewater was being processed throughout the world [30]. In this sewage water is one which was the wastewater released after consumption for household purposes like washing, flushing, *etc*. This water is generally made to accumulate in a sewer facility, which is black (due to bathing, dishwashing) and grey (toilet water) in colour. It is a combination of paper wrappers, sanitary items, soap residue and chemical composition of waste material with foul smell. This results in sewage pollution creating threat to environment and human health apart from affecting agriculture, aquatic and biodiversity. Hence, this water needs to be treated that is known as sewage treatment. Sewage treatment involve various processes [31] that include pre-treatment, primary, secondary, tertiary and sludge treatments. Pre-treatment is the first step, in which large materials such as bottles are scanned and reduced to smaller particles. These particles are removed and the water is ready for primary treatment. During primary treatment, heavy particles settle down the surface while biological content will be removed during secondary treatment. Even though secondary treatment removes most of the virus and bacteria, components like nitrogen, phosphorus, *etc.* may be present in the wastewater. This can be removed through tertiary treatment. This water can be reused as industrial water. Further treatment may help to use this water for cleaning. Beyond all these treatments, wastewater can be treated using sludge treatment. In this process, disintegration of organic matter and hydrolysis of macromolecular components leads to usage of solid deposits of sludge as fertilizers and for production of energy, *etc.* [32].

Sludge disintegration technique may be thermal, mechanical, chemical or microwave radiation based. It is reported that releasement of organic matter depends on final temperature [33], which affect dielectric properties of materials. Dielectric properties play important role in determining its EM-field distribution in a given material. The electrical properties of Waste activated sludge (WAS) which prevalent in sewage treatment plant has been reported by [34] with the help of open-ended coaxial probe technique. In this context, MTM sensor that sense variation in complex permittivity with resonance frequency shift is of importance. Metamaterials are tailored to control and manipulate electromagnetic waves [35]. Since the year 2000 when negative permittivity and permeability are demonstrated by metamaterials [36], various applications like antenna designing, cloaking devices have been stimulated [37]. Similarly, metamaterial absorbers are being used in various fields [38, 39]. Energy harvesting applications with microwave metamaterial absorbers have been developed [40, 41]. In continuation, metamaterial sensors that sense temperature, pressure, liquid chemicals, *etc.* are being reported [42–45]. Majority of MTM sensors are designed to study the change in complex permittivity through shift in resonance frequency based on capacitive effects [46]. For a given material, dipoles behave as per the complex dielectric constant that depends on its physical and chemical properties [47–55]. The impact of environmental parameters on complex permittivity using open-ended coaxial probe was reported [56].

Sludge volume index (SVI) is one of the *parameters in process control, which describes the sludge settled in an aeration tank of an activated sludge.* It is measured dividing amount of sludge settled in thirty minutes for one litre sample by concentration of suspended solids. Its value should range between 50–150 mL/g beyond which measures need to be taken to control the sludge levels. Various techniques to measure SVI has been earlier reported [57, 58], but for the first time a MTM sensor was reported for prediction of SVI and validate in microwave range. This technique demonstrated better visibility than the regular method of using a coaxial probe in which complex permittivity values are being measured. Later, an MTM sensor was designed as per the frequency of operation through simulation using FIT. Such MTM sensor was simulated and developed with the help of CNC (computerized numerical control) controlled PCB machine. Its ability was tested with vector network analyser (VNA) whose results demonstrated high sensitivity and effectiveness of designed sensor for water treatment. The designed sensor structure has two square copper resonators front and back along with another resonator with meander lines from left top to the right bottom of substrate made with Isola material of low dielectric loss. A sensor layer of thickness 10 mm was placed behind the backside resonator whose dimensions are compatible for X-band. The front and rear sides of waveguide sample holders are covered with kapton film of 25-μm thickness that was considered in simulation to correlate with experiment similar to lab setup.

Using this sensor, SVI index was measured in three stages of wastewater treatment given by water entry point, aeration and exit stages. Measurements of dielectric constant and MTM sensor are considered. Three samples tested between 8 GHz and 12 GHz for entrance, aeration and exit stages are reported to vary from 67 to 32.54, 39.64 and 56. Secondly, the measured dielectric constants of wastewater samples were simulated by assigning these values to a microwave simulator. The resonant frequencies of entrance, aeration and exit stages of water are found to be 10.208 GHz, 10.024 GHz and 10.232 GHz with about 230 MHz bandwidth indicating the ability of sensor in distinguishing different stages of water samples. These results are compared with experimental values obtained from network analyser, which are highly correlated with small differences in resonance frequency. Hence, this technique suits determination of liquid type [59].

4. CONCLUSIONS

This paper mainly reviewed the role of MTM sensors in liquid detection with prime focus on designed resonators and their successful implementation. It is observed that MTM sensors used in detection of liquids replace the traditional coaxial tube technique in X-band range of frequency. Mainly we observed that replacement of coaxial tube technique along with simulation is being carried out simultaneously. These sensors could differentiate between clean and waste transformer oils by a frequency shift of 250 MHz even their dielectric constants are very close by a difference of 0.11. Similarly, a frequency shift of 200 MHz was observed for olive, corn and cotton oils with dielectric constants 2.55, 3.08 and 3.2. Similar is the case with branded and unbranded diesels. This sensor proved to be successful in terms of speed, economical and accuracy. Apart from liquid detection, MTM sensors are proved very useful in water treatment centres. In this review, we observed an excellent MTM sensor that can detect sludge volume Index (SVI) efficiently. For the first time in the journey of MTM sensors this was reported, which may lead to many applications in detecting water quality in future. To design this sensor, complex permittivity values of water samples are being measured. The simulated and experimental results proved the efficiency of this sensor with high sensitivity and its effectiveness for usage in water treatment. For a given water treatment centre, five experimental values for same sample were reported to have about 0.5% standard deviation at resonance frequency. The simulation values are highly correlated with experimental values for entrance, aeration and exit of the water treatment centre with a shift of 72 MHz, 16 MHz and 88 MHz, respectively. A minimum of 230 MHz frequency shift exist between different samples under study, which can determine water quality. This journey of MTM sensors in water treatment may lead to new inventions in detecting and purifying water quality with low cost and high speed masking the existing conventional techniques.

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