PREDICTION OF AMMONIA AND IRON CONCENTRATIONS IN WATER BASED ON COMPLEMENTARY DOUBLE SPLIT-RING RESONATOR (DSRR)

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To my beloved parents, thank you.

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ABSTRACT

Ammonia and iron are water contaminants that are of concern in water quality monitoring. According to World Health Organization (WHO), the maximum level of ammonia and iron that can be absorbed by human body from drinking water were about 1.5 mg/L. The excessive ammonia and iron may affect public health, as excessive ammonia and iron will cause health issues, such as skin burn, ingestion problems, and liver damage. Compared with conventional water contaminant testing techniques, microwave sensing is an attractive solution in detecting ammonia and iron, since it is accurate and sensitive without demanding bulky, costly equipment and time-consuming sample preparation. This project aimed to design and develop a rectangular microstrip patch antenna with a complementary double split-ring resonator (DSRR) at 2.38 GHz as preliminary work for the detection of ammonia and iron concentrations. The dimension of the proposed design was calculated based on theoretical equations and simulated using CST MICROWAVE STUDIO$^{®}$. Based on the simulated results, the resonance frequency and *Q*-factor of the proposed design were 2.379 GHz and 396.5, respectively. In order to present the functionality of the sensor, both the simulation and measurement of ammonia and iron are presented in the concentration range from 0 mg/L to 53 mg/L. The resonance frequency of the sensor shifted from 2.337 GHz to 1.952 GHz in iron measurement, meanwhile the resonance frequency of the sensor shifted from 2.3904 GHz to 2.3924 GHz in ammonia measurement. The average and minimum measured sensitivity for iron were 0.007 GHz/mg/L and 0.037 GHz/mg/L, respectively. Based on the results, predictive models were proposed to estimate ammonia and iron concentrations in water. It was found that the first-order of the polynomial model and the second-order of the Fourier model were the best models for the prediction of ammonia and iron concentrations, respectively. The models introduced 0.9965 and 0.9953 of R-squared values, and 0.9698 and 1.36 of Root-Mean-Square-Error (RMSE), for ammonia and iron, respectively.

ABSTRAK

Amonia dan zat besi adalah bahan kimia yang mencemarkan air dan sentiasa diprihatinkan dalam sistem pemantauan kualiti air. Menurut Pertubuhan Kesihatan Sedunia (WHO), tahap maksimum amonia dan zat besi yang boleh diserap oleh tubuh manusia daripada air minuman adalah 1.5 mg/L. Hal ini telah menjejaskan kesihatan awam kerana amonia dan zat besi yang berlebihan akan mengakibatkan pembakaran yang teruk pada kulit manusia, masalah pengingesan, dan kerosakan pinggang dalam tubuh badan manusia. Berbanding dengan teknik-teknik pengujian kualiti air yang konvensional, pengesanan gelombang mikro telah digunakan dalam pengesanan ammonia dan zat besi disebabkan oleh sifatnya yang sensitif, ketepatan yang tinggi, membantu untuk mengurangkan penggunaan peralatan-peralatan yang besar dan juga masa semasa penyediaan sampel. Projek ini bertujuan untuk mereka bentuk antena patch mikrostrip yang bersegi empat dengan DSRR pelengkap pada 2.38 GHz untuk pengesahan amonia dan zat besi. Kerja ini masih dalam tahap awal. Dimensi reka bentuk telah dikira berdasarkan formula teori dan disimulasikan dengan menggunakan CST MICROWAVE STUDIO®. Keputusan simulasi frekuensi resonans dan *Q*-factor adalah 2.379 GHz dan 396.5. Fungsi sensor ditunjukkan dari segi simulasi dan pengukuran, manakala kepekatan amonia dan besi ditetapkan antara 0 mg/L hingga 53 mg/L. Keputusan menunjukkan frekuensi resonans sensor beralih daripada 2.337 GHz ke 1.952 GHz bagi besi, manakala frekuensi resonans sensor beralih daripada 2.3904 GHz ke 2.3924 GHz bagi amonia. Kepekaan purata dan minimum untuk besi adalah 0.007 GHz/mg/L dan 0.037 GHz/mg/L. Pertama model polinomial dan kedua model Fourier adalah model terbaik untuk ramalan kepekatan amonia dan zat besi masing-masing. Ralat punca min kuasa dua dan R-kuadrat untuk model amonia dan zat besi adalah 0.9965 dan 0.9953, dan 0.9698 dan 1.36, masing-masing.

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LIST OF SYMBOLS AND ABBREVIATIONS

NH_3 – Ammonia
Fe – Iron
MOH – Ministry of Health
NO_4^+ – Ammonium
DSRR – Double split-ring resonator
CSRR – Complementary split-ring resonator
R^2 – R-square
RMSE – Root Mean Squared Error
WTP – Water treatment plants
ISE – Ion-selective electrodes
NH_4Cl – Ammonium chloride
Fe_2O_3 – Iron (Ш) oxide
H – Hydrogen
N_2 – Nitrogen
OH^- – Hydroxide ions
H_2O – Water
WHO – World Health Organization
EPA – Environmental Protection Agency
NH_2Cl – Chloramine
HOCl – Chlorine
CE – Capillary electrophoresis
IPB – Indophenol blue
OPA – *O*- phthaldialdehyde
UV/VIS – Ultraviolet-visible spectrophotometer
EDTA – Ethylenediaminetetraacetic acid
ISA – Ionic strength adjustor

Fe^{2+} – Iron (II) ions
$Fe(OH)_3$ – Iron (III) hydroxides
O_2 – Oxygen
H^+ – Hydron
phen – 1,10-phenanthroline
FFF – Field-flow fractionation (FFF)
AAS – Atomic absorption spectrometer
ICP-MS – Inductively coupled plasma mass spectrometry
ICP-OES – Inductively coupled plasma optical emission spectroscopy
GPR – Ground-penetrating radar
ORP – Oxidation-reduction potential
MEMS – Microelectrochemical systems
EM – Electromagnetic radiation
SRR – Split-ring resonator
μ_{eff} – Permeability frequency
F – Filling fraction of SRR
ς – Damping factor of metal loss
r – Radius of inner ring
μ_0 – Permeability of free space
f_r – Resonance frequency
a – Length of ring
w – Width of ring
d – Space between the inner and outer rings
c – Speed of light
GPS – Global positioning system
RFID – Radio frequency identification
W – Patch width
ε_r – Dielectric constant
ε_{reff} – Effective dielectric constant
h – Thickness of substrate

L_{eff} – Effective length

ΔL – Fringing length/ extension of length

L – Patch length

L_g – Ground length

W_g – Ground width

W_f – Width of inset

Z_o – Characteristic impedance of the line

fi – Length of inset

L_f – Extension length of inset

L – Inductance

C – Capacitance

Z_1 – Impedance 1

Z_2 – Impedance 2

L_{gap} – Total equivalent inductance

L_1 – Inductance 1

L_2 – Inductance 2

C_1 – Capacitance 1

C_2 – Capacitance 2

α_{avg} – Average ring length

α_{ext} – Half-side length of square SSR

C_{pul} – Capacitance per unit length of the metal strip

γ – Wire loop of square geometry

W – Patch width

G_{pf} – Gap between patch and feed line

L_o – Length of outer ring

L_i – Length of inner ring

g – Width of ring

ls – Gap between rings

g_s – Gap of rings

t – Height of patch

ΔLp_1 – Series inductance

ΔCp_2 – Series capacitance

Cc1 – Coupling capacitor

S – Sensitivity

Δfr – Resonance frequency shifting

Δεr – Step size of the dielectric constant

M_1 – Initial concentration of the stock solution

V_1 – Volume of the stock solution

M_2 – desired concentration

V_2 – Volume of added water

PCB – Printed Circuit Board

FKEE – Faculty Electric and Electronic Engineering

UTHM – Universiti Tun Hussein Onn Malaysia

SSE – Sum of squares due to error

n – Total number of observations

i – Iteration of test set

$y(i)$ – Predicted value

$f\big(x(i)\big)$ – Actual value

SSR – Sum of square roots

k – Number of independent variables

ADS – Advance Design Software

VNA – Vector Network Analyzer

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Ammonia (NH_3) and iron (Fe) are common water contaminants that are present in the water supply. Ammonia is a colourless compound that can be found naturally with low concentrations in the human body and the environment but with high concentrations in industrial products, such as household cleaning solutions. The overuse of fertilizers in agricultural activities, which became industrial wastes and landfill leachate, have exposed humans to excessive ammonia, as it might spread on the ground and flow into the water supply. Excessive ammonia may result in the eutrophication of water surface, depletion of oxygen, and toxicity for aquatic organisms [1]. For humans, ammonia can lead to some health issues, such as skin burn, ingestion, and inhalation problems [1]. Iron is an essential element in the human body; however, excessive iron in the human body can lead to hemochromatosis, which can bring damage to the liver, pancreas, and heart [2]. The high concentration of iron in water is normally caused by the clogging of iron precipitates in home water softeners or filtration systems, and the internal corrosion of the distributing pipeline [3]. According to the Ministry of Health (MOH) Malaysia, the permitted limit of ammonia and iron in raw water should be lower than 1.5 mg/L and 1.0 mg/L, respectively [4].

Generally, water contaminant detection is carried out in laboratories, which involves the use of international-standard laboratory instruments and portable testing kits. These techniques are manually handled by a group of trained and skilful technicians or laboratory assistants [5]. The techniques provide highly reliable results, but the costs of sample preparation and testing increase when different types of tests and a large number of samples are involved. Moreover, the process of sample collection is time-consuming especially when the sampling point is in urban areas. In the pursuit of a low-cost yet highly reliable water contaminant detection technique, investigations on microstrip structures have been done extensively.

Microstrip resonators have been used extensively in water quality monitoring [6], [7], material characterisation [8], [9], and single-cell feasibility detection [10]. Microstrip resonators work via characterising the spectral behaviour of a sample below 100 GHz, which contributes to a low-cost embedded system compared with laboratory-on-board techniques [11]. The microstrip line is often coupled with metamaterial structures to provide better sensitivity and accuracy in a limited range of frequency bandwidth [12]–[14]. This technique has been used for thin-film and microfluidic sensing [15], [16], nitric acid detection [17], and water contaminants sensing, such as the detection of nitrate, phosphate, and ammonium (NO_4^+) [18], [19] and the measurement of other parameters, such as pH level, conductivity, and dissolved oxygen (DO) [18]. The measurement of water quality is observed via changes in microwave response, such as changes in S_{11} and S_{21} parameters and resonance frequency. The performance of the sensor is promising, as it can detect concentrations as low as 0.5 mg/L for nitric acid detection in water [17] and 0.1 mg/L separately for nitrate, phosphate, and ammonium detection in water [18].

In this project, a rectangular microstrip patch antenna with a complementary double split-ring resonator (DSRR) was proposed for ammonia and iron detection. The DSRR was considered, as it allows better electric field concentration at a lower frequency [20], [21]. A DSSR with the square unit cell performs better in terms of return loss and higher quality factor (*Q*-factor) compared with those of the conventional complementary split-ring resonator (CSRR) and circular structure [22]–[25]. In order to establish reliable predictive models for ammonia and iron concentrations, the experiment work was conducted extensively on water samples with concentrations ranging from 0 mg/L to 53 mg/L. The performances of the proposed models were evaluated based on the parameters of R-squared and root mean squared error (RMSE).

1.2 Problem Statement

Water is a vital element for life sustainment [26]. In Malaysia, ammonia and iron are common contaminants found in the water for filtration in water treatment plants (WTPs) [27]. A high concentration of ammonia in water may interfere with the filtration process of manganese and iron, as most of the oxygen is consumed by nitrification and thus causes the water to become earthy-tasting and mouldy [27]. Conventionally, the detection of ammonia and iron is conducted either in a laboratory or on-site, which involves expensive and bulky laboratory instruments, such as spectrophotometry equipment, portable testing kits, and ion-selective electrodes (ISE). These techniques are less accurate when the concentration is too low (spectrophotometry) [28] or high (ISE) [29]. In addition, the techniques create environmental issues from the extra wastes or rubbish, such as testing strips (from the API test kit) and hazardous chemical reagents, such as sulfuric acid and hydrochloric acid [30]. Also, the techniques' measurement results are strongly dependent on the sample's condition, such as bubbles (for spectrophotometry). Furthermore, these techniques involved complicated sampling processes, such as sampling, storage, handling, and analysis.

Hence, a cost-effective, high-sensitivity, compact-sized, and potentially safe sensor is needed to monitor the concentrations of ammonia and iron. In this work, a rectangular patch antenna with a complementary DSRR was proposed as a microwave sensor to detect ammonia and iron. The changes in the sensor's resonance frequency would be observed when the sensor was exposed to water samples with different concentrations and the measurement data would be used for predictive model development

1.3 Hypothesis

Microstrip structures have been applied as an attractive solution for microfluidic sensing of medical analytes [12], chemical substances [31], and blood glucose [32]–[34] and the detection of salt and sugar contents [35]–[37], zinc, copper, and lead [38], [39]. Since each material has unique dielectric properties depending on concentration and composition, the response of the sensor is reflected from its variation in terms of cross-mode insertion loss [40], return loss [35], resonance frequency shift [32]–[34], [38], [41] or *Q*-factor [42]. It has been hypothesised that the concentration levels of ammonia and iron affect the dielectric properties of water, thus causing the shift in resonance frequency.

1.4 Aim

This project aimed to design a highly sensitive and low-cost sensor for ammonia and iron detection.

1.5 Objectives

This research work was embarked to achieve the following objectives:

a) To design a rectangular microstrip patch antenna with a complementary DSRR with resonance frequency at 2.38 GHz using CST MICROWAVE STUDIO$^{®}$.

b) To measure the resonance frequency of the sensor when exposed to water samples with different ammonia and iron concentrations.

c) To propose predictive models for predicting the concentrations of both ammonia and iron based on the shift of resonance frequency.

1.6 Scope of Study

The scope of the research is as follows:

a) Iron (Ш) oxide was used to prepare samples of different iron concentrations.

b) 15 samples in the concentration range from 0 to 53 mg/L were prepared using the stoichiometry technique. This concentration range was selected, based on the measurement limits in conventional techniques of iron concentrations, such as indophenol reaction and ammonia selective electrode (0.0025 mg/L to 3

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APPENDIX A

LIST OF PUBLICATIONS

1. Josephine Ong Ning Ting, S. K. Yee, "Review on Water Quality Monitoring Technologies." Indonesian Journal of Electrical Engineering and Computer Science, Institute of Advanced Science and Technology, vol 6, pp. 1416, 2020.
2. S. L. Yeoh, S. K. Yee, N. T. J. Ong, S. H. Dahlan, C. K. Sia, “Prediction of Ammonia Concentration in Water Based on Microwave Spectroscopy”, Bulletin of Electrical Engineering and Informatics (BEEI), Universitas Ahmad Dahlan, vol 9, pp. 898, 2019.
3. N.T.J. Ong, S.K. Yee, S.H. Dahlan, A.Y.I Ashyap, C.K. Sia, "Empirical model for ammonia concentration prediction in distilled water", Proceedings of Mechanical Engineering Research Day 2020, pp. 172-173, 2020.
4. Yee SK, Ong NTJ, Lim SCJ, Mohd Zin NS, SH Dahlan, AYI Ashyap, CF Soon, "Microwave sensing of ammonia and iron concentration in water based on complementary double split-ring resonator”, Sensors and Actuator Reports 3 (2021), 100044, 2021.

APPENDIX B

VITA

The author was born in January 20, 1994. She went to SMK Tung Hua, Sibu, Sarawak, Malaysia, for her secondary school. She is a postgraduate student who is studying at Universiti Tun Hussein Onn Malaysia, Parit Raja, Johor, Malaysia. She graduated from UTHM with a bachelor’s degree in Electronic Engineering. She was a research assistant at the Centre for Electromagnetic Compatibility (EMCenter) under the Research Centre for Applied Electromagnetics, UTHM, in a research project to develop a model for water quality detection using the microwave sensing technique. She participated as a presenter in three conferences to present the findings of the research. She has co-authored five papers in area of microwave sensing applications. She is currently a member of the Board of Engineers Malaysia.