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MICROWAVE ABSORPTION ANALYSIS ON HEATED EDIBLE SPIRULINA WITH VARIOUS TEMPERATURES

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ABSTRACT

This paper discusses the microwave absorption analysis of edible Spirulina by using WR62 and WR90 rectangular waveguides in conjunction with Agilent P-series Vector Network Analyzer (PNA). Heat might lead to the degradation of spirulina. This phenomenon involves the chemical and physical reaction that is associated with the variation of dielectric properties. These properties determine the propagation mechanism of microwaves within the sample or material. Hence, an assessment method to detect a nutrient change in spirulina due to heat is necessary. In this context, a microwave absorption measurement system was developed to study the reflection coefficient, transmission coefficient, and absorption coefficient of Spirulina tablets over temperature. The transmission/Reflection line method is well-known because it is non-destructive and rapid in analyzing chemical and physical properties. In this work, Spirulina tablet is used since it is a popular food supplement that is believed to be able to treat diseases is and good for health. The reflection, transmission, and absorption measurements were conducted on Spirulina from 12.4GHz to 18GHz.

Keywords: microwave absorption measurement system, P-series vector network analyser, reflection coefficient.

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1. INTRODUCTION

Spirulina has been consumed as a food supplement by the people of Mexico and natives of the Lake Chad area since [1]. It is also known as blue-green algae or cyanobacterium. Nowadays, Spirulina is consumed as a daily supplement because of its nutritional value. Spirulina as an antioxidant is discovered due to the presence of Phycocyanobilin (PCB). PCB is a chromophore bound to a chief protein which is known as phycocyanin. It acts as an enzyme and potent inhibitor of nicotinamide adenine dinucleotide phosphate (NADPH) oxidase. Phycocyanin is the source of pathological oxidant stress in many health disorders [2]. Additionally, Spirulina also scavenges free radicals which trigger several diseases in the human body. There are two types of cyanobacteria, i.e., Arthrospira platensis and Arthrospira maxima. This blue-green alga is a good bacterium for the human body. It is made up of 60% of protein. Protein is most sensitive to high temperatures. It is also found that stressed conditions could change its functional properties.

Since 1970, Spirulina has been analyzed chemically. It has been considered an excellent source of proteins, vitamins, and minerals. Spirulina is a natural “algae” (cyanobacteria) powder that is incredibly high in protein and nutrients., Research has found the health benefits of Spirulina, such that it acts as a protectant against free radicals in the human body [1], acts as an antioxidant [3], inhibitory activity against a wide range of viruses such as HIV-1, an anti-cancer effects, anti-allergic effect and helps in protect against nutritional deficiencies and cellular aging.

Proteins are made up of amino acids attached to form a long chain (20 different types of amino acids). A different sequence of amino acids determines its specific function of it and also its unique 3-Dimensional structure. Its function is as an antibody, enzymes, messenger, structural component, and as a transport or storage. In general, it serves a variety of functions within cells [4]. Protein can be disrupted by several factors, i.e. temperature [5], pH [6], high salt concentration [7], etc. Some researchers reported that overheating destroys certain amino acids. The addition of amino acids to the protein improves the biological value; others have claimed heat damage led to the decrement of digestibility of the heated proteins. Past researchers also have suggested that heat stress inhibits electron transport at the acceptor side of Photosystem II in a cyanobacterium Spirulina platensis [8]. Moreover, past researchers also found that in higher temperature stress, its oxygen and CO_2 assimilation capacities are significantly inhibited if exposed to leaves or algae [9].

The quality of protein needs to be controlled for maximum nutrient intake for the human body. A study has revealed that protein turnover can be used for quality control [3]. Protein turnover is the balance between the synthesis and degradation of proteins. Heat-shock proteins are introduced in controlling protein quality. Spirulina is exposed to stress and certain temperature during manufacturing.

When the Spirulina is heated, the molecular structure change. The relationship between temperature and the quality of the Spirulina can be studied via microwave sensing technique. This technique determines the reflection coefficient and transmission coefficient of a medium as a function of frequency, polarization, density, moisture, etc. This technique has been used in many types of research in the food industry for food quality control [10], composition measurements, and the pharmaceutical industry [11]. This technique is considered a relatively inexpensive and rapid technique. Moreover, past research found that the presence of free amino acids or the pH variations and the electrical charges of protein are the factors that can affect the dielectric spectrum at certain frequencies [12]. In general, this technique is also used to measure the microwave absorption of Spirulina. The advantages of this technique are rapid, non-destructive, and accurate.

S-parameters (S_{11}, S_{21}, S_{12}, and S_{22}) are also known as scattering parameters. It is used to determine the complex permittivity and permeability of the linear materials in function of frequency. It describes the electrical behavior of linear electrical networks [13]. S_{11} and S_{22} indicate the reflection coefficient from port 1 and port 2, respectively. Meanwhile, S_{21} and S12 refer to the transmission coefficient that describes the signal flow between port 1 and port 2. In this work, reflection and transmission coefficients were determined from measured S_{11} and S_{21}, respectively. These S-parameters are measured by using the Transmission/Reflection line method. Meanwhile, the absorption coefficient is expressed as

$$\text{Absorption coefficient} = 1 - |S_{11}|^2 - |S_{21}|^2 \quad (1)$$

Microwave absorption elucidates the phenomenon of energy lost when the sample is subjected to electromagnetic waves. The vibration or oscillation of molecules and charge particle lead to collision and friction. Collision and friction result in energy loss. This energy loss is interpreted as absorption. When the Spirulina is heated at various temperatures. There might be an occurrence of a chemical reaction that causes molecular change, i.e., degradation of protein that is associated with break bonding, the release of electrons, etc. These changes vary the absorption activity in Spirulina. Hence, the effect of heating temperature on Spirulina can be investigated through microwave absorption.

In this work, microwave absorption spectroscopy was developed to investigate the effect of heat on the Spirulina S-parameters (reflection coefficient and transmission coefficient). Reflection/transmission methods using open-ended coaxial probes and rectangular waveguides were studied from 12.4 GHz to 18 GHz. In hypothesis, the deterioration of acid amino due to heat could vary dielectric properties significantly [14]. The variation of dielectric properties results in a variation in absorption.

2. METHODOLOGY

2.1 Sample Preparation

In sample preparation, the Spirulina tablet was ground into powder. Then, the 8g of Spirulina in powder is measured using an analytical balance. The Spirulina powder was then compressed into a pellet by using hydraulic pressure. The compression of the pellet is necessary to rule out air void in the pellet to avoid air void before measurement.

2.2 Measurement setup and Calibration

The transmission-reflection measurement system was established using a rectangular waveguide in conjunction with an Agilent E8362B P-series vector network analyzer (PNA). The dimension of prepared samples needs to refer to the requirement of WR62 and WR90. The prepared sample that meets the requirement is placed in a sample holder. The sample in the holder is connected with WR62 and WR90 waveguides. Then, it connected to port 1 and port 2 of the vector network analyzer for reflection and transmission measurement. Before the measurement, calibration needs to be conducted using a standard calibration kit.

All the samples were heated at temperatures of 28ºC, 40ºC; 50ºC, 60ºC, 70ºC, 80ºC, and 90ºC using the heating oven. The heated sample at each temperature was placed in the WR62 and WR90 sample holders. These sample holders with sample were placed in between a pair of waveguides. Meanwhile, both waveguides were connected to port 1 and port 2 of PNA for the Transmission/Reflection line method.

3. RESULTS AND DISCUSSIONS

3.1 Measurements during the Heating Process (Real-Time)

Figure-1 shows the variation of the reflection coefficient and transmission coefficient over the frequency with different temperatures of Spirulina. As shown in Figure-1(a), S11 is decreased as frequency lower than 15GHz for temperatures higher than 60℃. This can be related to the increase of permittivity of the Spirulina at a temperature of 60℃ above. It is suggested at a frequency between the range 12.4GHz to 15GHz for temperatures lower than 50℃ is the best to detect the effect of high temperature on the Spirulina. As for the Transmission coefficient, energy transmitted shows an increment at frequency 15GHz for the temperature lower than 50℃ and shows decrement for temperature above 60℃. This is due to more energy reflected at this frequency, so electrical cannot be transmitted. At frequency 18GHz, absorption he high for temperatures less than 60℃.

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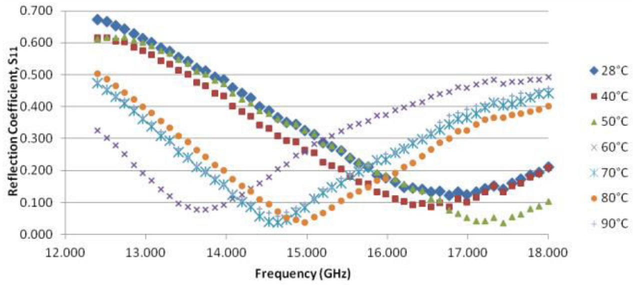

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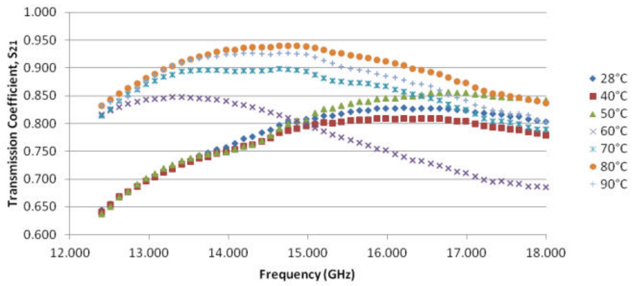

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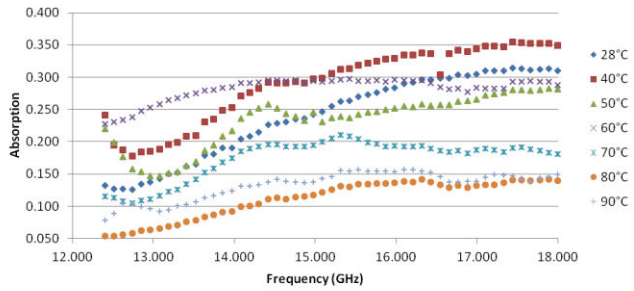

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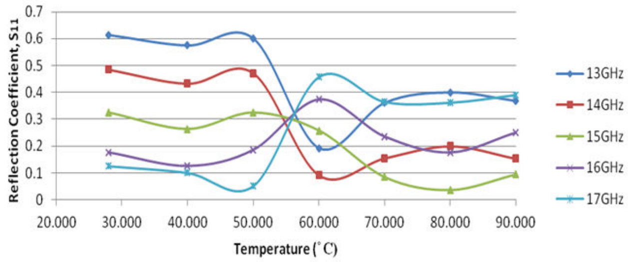
Figure-1. The variations of (a) reflection coefficient, S11, and (b) transmission coefficient, S21, and (c) absorption over frequency for different temperatures.

Five frequencies were selected as shown in Figure-2 to discuss the variations of reflection coefficient and transmission coefficient over temperature. Frequency 17GHz is the highest at a temperature of 60℃ and followed by frequency less than that. The highest frequency shows reflect more with an increment of temperature. This is because of the polarization of the Spirulina. However, the transmission coefficient shows the opposite of reflection. This is due to S11 being high; therefore the energy cannot be transmitted more than the reflected energy. However, absorption drastically decreases for five frequencies starting at a temperature of 60℃. This is because, at this temperature, the ability to absorb energy decrease of the loss drastic change of protein structure.



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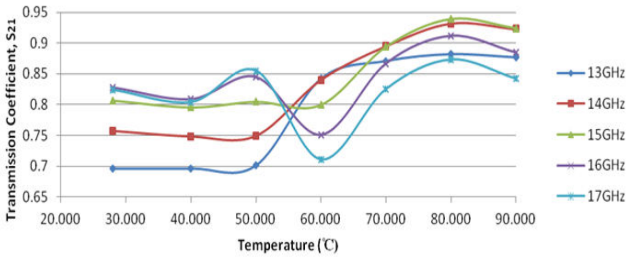

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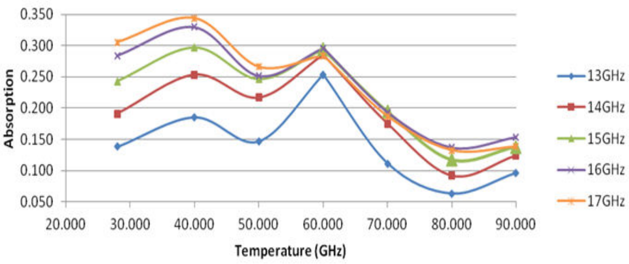

(c)

Figure-2. The variations of (a) reflection coefficient, S11, and (b) transmission coefficient, S21, and (c) absorption over temperature for five selected frequencies.

3.2 Measurement after the Hearing Process (Non-Real-Time)

Figure-3 shows the variation of the reflection coefficient and transmission coefficient over frequency for different temperatures. The electric signal reflected high at frequency 13GHz and frequency 17GHz. The lowest reflected electric signal occurs at around 15GHz until 16GHz. Reflection coefficients supposedly decrease when the temperature is increasing. This is due to the ionic conduction of Spirulina. Thus, it is studied that the reflection coefficient expected, will show an increment with increasing frequency with an increase of temperature. Moreover, because of the ionic conduction, it is expected that the transmission coefficient is high between frequencies 15GHz to 16GHz.

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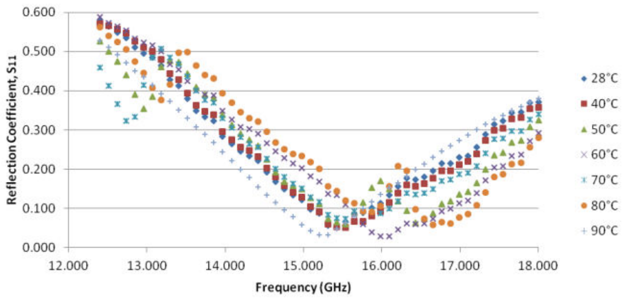

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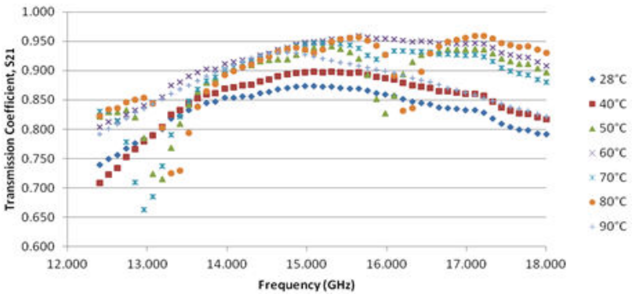

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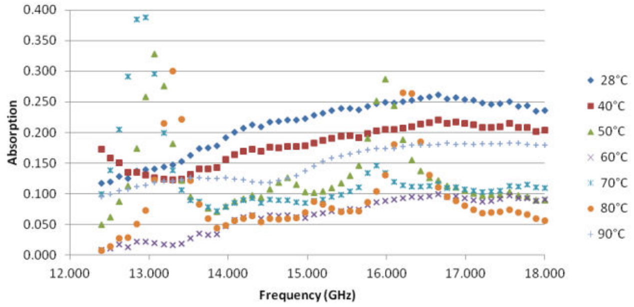

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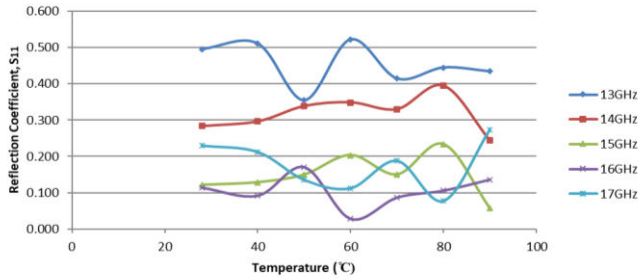
Figure-3. The variation of (a) reflection coefficient, S11, (b) transmission coefficient, S21, and (c) absorption over frequency for different temperatures.

Five frequencies were selected as shown in Figure-4 to discuss the variation of reflection coefficient and transmission coefficient over temperature. Temperature 60℃ is taken as the reference temperature since most proteins will denature at this temperature. The graph shows fluctuating graph because of the effect of changing the electric field when heating. The reflection coefficient is highest at 13GHz it is found that in this frequency, the energy is not high enough to go through the sample of Spirulina. Therefore, in this frequency, an electrical signal is transmitted less. It happens because Spirulina acts as low ionic conduction. As for absorption, it decreases drastically when > 60℃ due to the change of Spirulina molecular structure.



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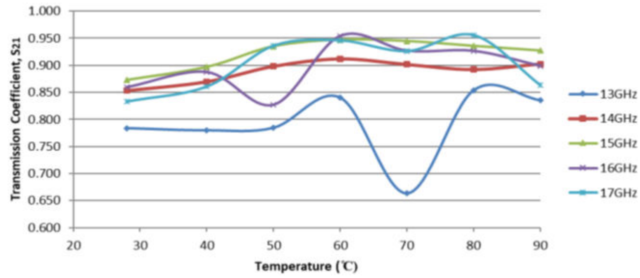

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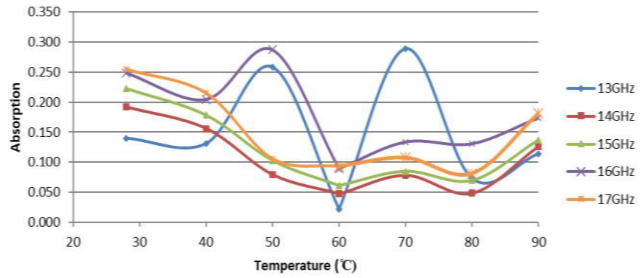

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Figure-4. The variation of (a) reflection coefficient, S11, (b) transmission coefficient, S21, and (c) absorption over temperature at five selected frequencies.

3.3 The comparison between the Coefficient of Determination, R^2 of S_{11}, S_{21} and Absorption within 20 ºC -60 ºC and 60ºC -90ºC for Real-Time Measurements

Table-1 shows the variations of the coefficient of determination, R^2 between two ranges of temperature for reflection coefficient, S_{11}, transmission coefficient, $S_{21,}$ and absorption coefficient during real-time measurement (measurement was conducted during the heating process). R^2 indicates the relationship between parameters involved with temperature. Linearity of S_{11}, $S_{21,}$ and absorption coefficient with temperature was investigated via a linear equation, y=mx+cas shown in Table-1 y represents S11, S21, and absorption coefficient as tabulated in Table-1(a)-(c). 13 GHz, 14 GHz, 15 GHz, 16 GHz, and 17 GHz were selected to establish these relationships. The temperature range for the development of linear equation is from 20 ºC -60 ºC and 60ºC -90ºC. At 60℃, exhibits the highest sensitivity. The reflection coefficient increase after a temperature of 60℃ except at frequencies ranges 16GHz to 17GHz. Therefore, the transmission coefficient shows decrement from 13GHz to 14GHz. Absorption is high at a frequency 16GHz when temperature>60℃. This frequency is appropriate to be used to study the absorption for temperature >60℃.

Table-1. The variations of coefficient of determination, R^2 over 20 ºC -60 ºC and 60ºC -90ºC for (a) reflection coefficient, S_{11} (b) transmission coefficient, $S_{21,}$ and (c) absorption coefficient at 13 GHz, 14 GHz, 15 GHz, 16 GHz, and 17 GHz (real-time).

(a)

Temperature (℃)	Frequency (GHz)	R^2	*m*	Linear equation
20-60	13	0.5954	-0.0114	y=-0.0114x+1.0043
60-90		0.6101	0.0057	y =0.0057x -0.095
20-60	14	0.5953	-0.0105	y =-0.0105x +0.8391
60-90		0.4518	0.0023	y =0.0023x -0.023
20-60	15	0.2520	-0.0014	y =-0.0014x +0.3534
60-90		0.5191	-0.0054	y =-0.0054x +0.5215
20-60	16	0.5600	0.0059	y =0.0059x -0.0482
60-90		0.4495	-0.0043	y =-0.0043x +0.5795
20-60	17	0.4073	0.0086	y =0.0086x -0.2012
60-90		0.3485	-0.002	y =-0.002x +0.547

(b)

Temperature (℃)	Frequency (GHz)	R^2	*m*	Linear equation
20-60	13	0.5958	0.0041	y =0.0041x+0.5527
60-90		0.6994	0.0012	y =0.0012x+0.781
20-60	14	0.4918	0.0023	y =0.0023x+0.6721
60-90		0.7937	0.0029	y =0.0029x+0.683
20-60	15	0.1176	-0.0001	y =-0.0001x+0.8075
60-90		0.745	0.0042	y =0.0042x+0.5185
20-60	16	0.3643	-0.0018	y =-0.0018x+0.8884
60-90		0.6615	0.0045	y =0.0045x+0.5185
20-60	17	0.3425	-0.0026	y =-0.0026x+0.9163
60-90		0.6488	0.0044	y =0.0044x+0.482

(c)

Temperature (℃)	Frequency (GHz)	R^2	*m*	Linear equation
20-60	13	0.5684	0.0029	y =0.0029x+0.0524
60-90		0.6383	-0.0052	y =-0.0052x+0.5215
20-60	14	0.6054	0.0023	y =0.0023x+0.1322
60-90		0.7458	-0.0057	y =-0.0057x+0.5941
20-60	15	0.2213	0.0010	y =0.001x+0.2245
60-90		0.7918	-0.0055	y =-0.0055x+0.5955
20-60	16	0.0221	-0.0004	y =-0.0004x+0.3058
60-90		0.7652	-0.0048	y =0.0048x+0.5583
20-60	17	0.2726	-0.0013	y =-0.0013x+0.3566
60-90		0.8188	-0.0049	y =-0.0049x+0.5542

3.4 The Comparison between the Coefficient of Determination, R^2 of S_{11}, S_{21} and Absorption within 20 ºC -60 ºC and 60ºC -90ºC for Non Real-Time Measurements

Table-2 shows the variations of the coefficient of determination, R^2 between two ranges of temperature for reflection coefficient, S_{11}, transmission coefficient, $S_{21,}$ and absorption coefficient during non-real-time measurement (measurement was conducted after the heating process). Non-real-time measurement (at room temperature), transmission coefficient exhibits the most optimum relationship at 15GHz for temperature 20℃ to 60℃and 20℃ to 60℃ where R^2 is 0.9682 and 0.9571, respectively. Both the reflection coefficient and absorption coefficient are bare to secure R^2> 0.9 for both ranges of temperatures at the same frequency

Overall, it can be noticed that R^2 for nonreal-time measurement (Table-1) is higher than for real-time measurement (Table-2). It might be attributed to the conduction of measurement during the heating process (real-time measurement). In real-time measurement, the vigorous activity of molecular change occurs. It generates heat noise during measurements.

Table-2. The variations of coefficient of determination, R^2 over 20 ºC -60 ºC and 60ºC -90ºC for (a) reflection coefficient, S_{11} (b) transmission coefficient, $S_{21,}$ and (c) absorption coefficient at 13 GHz, 14 GHz, 15 GHz, 16 GHz and 17 GHz (non real-time)

(a)

Temperature (℃)	Frequency (GHz)	R^2	*m*	Equation
20-60	13	0.0181	-0.0008	*y*=-0.0008*x*+0.5051
60-90		0.4072	-0.0023	*y*=-0.0023*x*+0.6296
20-60	14	0.9287	0.0022	*y*=0.0022*x*+0.2175
60-90		0.2616	-0.0025	*y*=-0.0025*x*+0.5162
20-60	15	0.8378	0.0025	*y*=0.0025*x*+0.0407
60-90		0.3514	-0.0036	*y*=-0.0036*x*+0.4277
20-60	16	0.1439	-0.0016	*y*=-0.0016*x*+0.1734
60-90		0.9472	0.0034	*y*=0.0034*x*-0.1679
20-60	17	0.9212	-0.004	*y*=-0.004*x*+0.3501
60-90		0.306	0.0037	*y*=0.0037*x*-0.1156

(b)

Temperature (℃)	Frequency (GHz)	R^2	*m*	Equation
20-60	13	0.5730	0.0016	*y*=0.0016*x*+0.7262
60-90		0.0634	0.0018	*y*=0.0018*x*+0.6659
20-60	14	0.9779	0.0019	*y*=0.0019*x*+0.798
60-90		0.3618	-0.0004	*y*=-0.0004*x*+0.9299
20-60	15	0.9682	0.0024	*y*=0.0024*x*+0.8044
60-90		0.9571	-0.0007	*y*=-0.0007*x*+0.9907
20-60	16	0.2750	0.0021	*y*=0.0021*x*+0.7896
60-90		0.9034	-0.0017	*y*=-0.0017*x*+1.0518
20-60	17	0.9185	0.0039	*y*=0.0039*x*+0.7198
60-90		0.4626	-0.0022	*y*=-0.0022*x*+1.0873

(c)

Temperature (℃)	Frequency (GHz)	R^2	*m*	Equation
20-60	13	0.0825	-0.002	y=-0.002x+0.2282
60-90		0.0047	0.0006	y=0.0006x+0.0781
20-60	14	0.9652	-0.0048	y=-0.0048x+0.3315
60-90		0.5212	0.0021	y=0.0021x-0.0801
20-60	15	0.9812	-0.0053	y=-0.0053x+0.375
60-90		0.6499	0.0021	y =0.0021x-0.071
20-60	16	0.3425	-0.0037	y=-0.0037x+0.3708
60-90		0.8773	0.0025	y =0.0025x-0.0578
20-60	17	0.9148	-0.0056	y=-0.0056x+0.4154
60-90		0.4684	0.0024	y=0.0024x-0.0638

4. CONCLUSIONS

The main intention of this research is to distinguish the fresh and heated Spirulina through investigation of the effect of heat on Spirulina through the Transmission/Reflection line method. The measurement results provide a fundamental knowledge of wave propagation to characterize Spirulina. Subsequently, it justified the feasibility of this method in characterizing Spirulina. In this research, Spirulina can be characterized during the heating process with various temperatures via real-time and non-real-time reflection, transmission, and absorption measurement. The distinguishable results can be noticed due to different heating temperatures. The presence of other substances via degradation of nutrients (e.g., protein) in Sprulina during the heating process led to the variation of electrical properties and characteristics of the Spirulina. This variation could vary the propagation mechanism of electromagnetic waves in Spirulina. Apart from that, it can be concluded that the transmission coefficient, S_{21} can provide the most promising relationship at 15GHz due to its high R^2 at different frequency ranges.

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