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Factorial Analysis on the Preparation of Barium Titanate-Epoxy Resin Composite for Antenna Substrate

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ABSTRACT: This study investigates the preparation and characterization of barium titanate-epoxy resin composites, focusing on main factors influencing the dielectric properties of that composite materials. Using a 2^k factorial design, the effects of heating temperature, stirring speed, stirring time, and hardening process on the permittivity were thoroughly investigated. Sixteen samples were prepared and analyzed using Design-Expert software, with permittivity measurements conducted via the waveguide method and a Vector Network Analyzer (VNA) in the 4–6 GHz range. Results show significant impacts from stirring time and speed, with optimal conditions identified as 50°C heating, 500 rpm stirring speed, three minutes stirring time, and room temperature hardening from two-level factorial analysis (TLFA). These findings provide valuable insights into the best fabrication conditions for barium titanite-epoxy resin composites, contributing to the development of antenna substrate with a permittivity value of 7.0208 and a loss tangent of 0.0238 that is suitable for high-frequency communication applications.

1. INTRODUCTION

For optimizing antenna performance, especially on materials for antenna substrates, achieving high permittivity with minimal loss is important. Other than that, the use of composite material in electronic communication systems is on the rise. It is reported that the usage of the composite materials can contribute to high performance and maintain low-cost, miniaturization factors [1]. Multiple methods for fabricating antenna substrates from various materials have been explored, including flexible [2–4], textile [5], agricultural waste [6–9], and composite materials [10–13]. This study aims to identify the key factors that influence the dielectric properties of barium titanate-epoxy resin composites, based on the preparation and characterization processes. Barium titanate ($BaTiO_3$) is renowned for its excellent dielectric properties, making it an ideal ceramic filler in composite materials [13, 14]. By combining with epoxy resin, these composites can offer enhanced performance in high-frequency applications such as antennas [13, 14].

The composite material preparation involves precise mixing of barium titanate powder with epoxy resin and a hardener. The interaction between these components, particularly during the stirring and curing phases, significantly impacts the resulting material's permittivity. It is reported that the epoxy resin alone has a low permittivity value around two to five, making it inadequate to be applied to electronic application [15]. The combination of epoxy resin with high-permittivity ceramic nanopowders, such as barium titanate, to enhance the material's permittivity has been observed in several studies [1, 12, 13, 15–18]. Few studies have highlighted the effect of the temperature, concentration level, and processing method in general on the dielectric properties of composites [6, 7, 19]. However, a comprehensive factorial analysis on heating temperature, stirring speed, stirring time, and hardening process effects on permittivity, particularly at 5 GHz remains underexplored.

This study employs two-level factorial analysis (TLFA) to systematically investigate the impact of four critical factors: heating temperature, stirring speed, stirring time, and hardening process on permittivity of barium titanite-epoxy resin composites. Sixteen experimental runs are presented and analyzed using Design-Expert software to generate experimental conditions and assess their effects on the permittivity of the composites. The goal is to determine the optimal combination of these factors that yields the highest permittivity, making the composite material suitable for antenna substrate applications from TLFA.

Permittivity measurements were conducted using the waveguide method, operating at a range of 4 GHz to 6 GHz. Since this study aims to identify the key factors that influence the dielectric properties of barium titanate-epoxy resin composite and propose potential improvements for future research in this field, the results of this study are expected to contribute to the development of advanced dielectric materials with good performance for high-frequency communication applications.

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2. METHODOLOGY

2.1. Formulation of Dielectric Composite Material

This study utilized composite materials, specifically barium titanate, and epoxy resin. Barium titanate ($BaTiO_3$) is recognized for its role as a ceramic material that serves as a filler in dielectric composites [20]. Epoxy resin is a thermosetting polymer that holds together the fibers of composite materials. Epoxy resin requires a hardener to start the curing process since epoxy does not harden by itself through the cooling or drying process [21].

2.2. Material

In this process, nanosized barium titanate powder and epoxy resin, which are readily available on the market, were utilized. The epoxy resin and hardener were mixed following the manufacturer's recommended weight ratio of 3 : 1, with 37.5 g of resin and 12.5 g of hardener. Additionally, 60 g of barium titanate powder was used. The densities of the epoxy resin and nanosized barium titanate powder are 1.25 g/cm^3 and 6 g/cm^3, respectively [17].

A curing agent is used to harden the surface of a material by increasing the bonding of the substance's molecular elements. The epoxy hardener undergoes partial curing, meaning that it is partially set but not fully cured, before mixing up with another substance and leaving it totally set.

2.3. Sample Preparation

The production of the sample begins by mixing 60 g of barium titanate with 37.5 g of epoxy solution. This mixture was then stirred using an overhead mixer for two to three minutes. Afterward, 12.5 g of epoxy hardener was added while the solution was heated to either 40°C or 50°C on a hotplate, with stirring speeds adjusted to either 100 rpm or 500 rpm. This process might create air bubbles, which could have decreased the material's impedance, thereby impacting its permittivity and tensile strength [22]. Next, the blend was carefully transferred into a mold, 22.15 mm×22.15 mm, with a depth of 22.10 mm [19], dimensions that match the waveguide opening used for testing the material's permittivity. Finally, the material was left to harden for 30 minutes in an oven set at 60°C, with the moderate heat helping to speed up the curing process. Raising the curing temperature improves the composite's heat deflection temperature and its mechanical properties by enhancing its resistance to deformation under heat. After this initial heat treatment, the composite undergoes a further 24-hour curing period at room temperature. Once cured, the material is extracted from the mold and ready for permittivity testing.

To determine which composite mixture is suitable for the fabrication of antenna, sixteen distinct samples were made. The factors involved during this process, the temperature during stirring, stirring time, stirring speed, and the hardening process, were studied to determine and observe the effect on the permittivity value using 2^k factorial design, where k is the number of factors. The experiment numbers were generated using Design-Expert software. Thus, the process above is repeated with all four factors mentioned above, as shown in Table 1.

TABLE 1. Two-level factorial analysis.

Factor	Description	Value
A	Heating	40°C and 50°C
B	Stirring Speed	100 rpm and 500 rpm
C	Stirring Times	2 mins and 3 mins
D	Hardening Process	Oven Temperature (OT) and Room Temperature (RT)

2.4. Material Characterization-Permittivity Measurement

Permittivity value can be measured with various characterization methods — free space, resonant [23–26], or waveguide techniques [27, 28] — where in this research, the waveguide technique was employed to measure the permittivity value. Additionally, a Vector Network Analyzer (VNA) was used to conduct wide-frequency measurements. The rectangular waveguide, set to operate in the dominant transverse electric (TE_{10}) mode, covered a frequency range of 4 GHz to 6 GHz. The dimensions of the sample were adjusted to match the 22.15 mm height of the waveguide, ensuring it to precisely fill the waveguide's cross-sectional area [29]. Before conducting permittivity measurements on the sample, the VNA underwent a comprehensive two-port Short-Open-Load-Thru calibration to eliminate errors due to device deficiencies. The VNA helped measure the phase and magnitude of S_{21}. To calculate the permittivity together with the loss tangent of the composite material using an inverse method, the phase and magnitude of S_{21} are needed [30].

2.5. Data Analysis Technique

Several methods were employed in analyzing the impact and performance of the four factors on the permittivity of the barium titanate-epoxy resin composite material. Analysis of variance (ANOVA) was used to analyze suitable factors generated by Design-Expert software. It is used to check if there is a significant difference between two or more categorical groups by comparing their means with variance. It is also employed to validate the importance of process factors and understand the level of interaction between these factors [31].

Then, the following analysis was also performed by Design-Expert software, which generated experimental design involving two-level factorial analysis (TLFA). In studying the effect of multiple factors on a response variable effectively by considering all possible combinations of factors at two different levels, a 2^k factorial design is suitable for executing this process. This approach allowed for a comprehensive evaluation of each factor and their interactions. Specifically, a full factorial design was implemented, meaning that all possible combinations of the four factors — heating temperature, stirring speed, stirring time, and hardening process — were tested. Thus, sixteen experimental runs were conducted, corresponding to the full set of combinations for the two levels of each of the four factors. The variable analysis can be generated by Design-Expert and is presented in Table 2.

TABLE 2. Variable analysis with Design-Expert software.

STD	Factor A (°C)	Factor B (rpm)	Factor C (min)	Factor D
1	50	100	2	OT
2	40	100	2	RT
3	50	500	2	OT
4	40	500	3	RT
5	50	500	3	RT
6	40	500	2	OT
7	50	500	2	RT
8	50	100	3	OT
9	40	100	3	RT
10	40	100	3	OT
11	50	100	2	RT
12	50	100	3	RT
13	40	500	3	OT
14	40	500	2	RT
15	40	100	2	OT
16	50	500	3	OT

Additionally, the Pareto chart is a graphical tool used to manage model selection for two-level factorial designs and visualize and prioritize the most significant factors influencing permittivity, facilitating the identification of critical variables for optimization. Two different t-limits are depicted on the graph. The higher limit corresponds to the Bonferroni or family-wise corrected t-critical value, while the lower limit represents the standard t-critical value for individual effects tests. Effects that exceed the Bonferroni limit are highly likely to be significant and should be retained in the model. Those that exceed the standard t-value limit might be significant and should be considered for inclusion if they are relevant to the experimenter's context.

3. RESULT AND DISCUSSION

3.1. Two-Level Factorial Analysis (TLFA) of the Composite Material

3.1.1. Analysis of Variance (ANOVA)

Table 3 shows the ANOVA analysis for the factors involved in the process together with the significance model. The F-value and P-value of the significance model are 638.83 and 0.0310, respectively. The model is deemed significant as the F-value indicates good agreement, and the P-value is below 0.05 at a 95% confidence level. The significance of the factors, stirring speed (B) and stirring time (C), on the permittivity value is confirmed by their P-values, both of which are less than 0.05.

TABLE 3. Analysis of Variance (ANOVA) table.

Source	Sum of Squares	Mean Square	F-Value	P-value Prob>F
Model	6.36	0.45	638.83	0.0310
Factor A	0.01	0.01	15.23	0.1597
Factor B	0.30	0.30	420.03	0.0310
Factor C	2.29	2.29	3223.58	0.0112
Factor D	0.04	0.04	53.05	0.0869

The permittivity and loss tangent responses at 5 GHz for sixteen experimental runs are presented in Table 4. The permittivity values range from 4.6917 to 7.0208, and the loss tangent values range from 0.0038 to 0.1313. The highest permittivity occurred under the conditions of Std 5, which involved a heating temperature of 50°C, a stirring speed of 500 rpm, a stirring time of three minutes, and a hardening process at room temperature. The smallest permittivity value was observed under condition Std 7, where the factors contributing to this result were 50°C of heating, 500 rpm of stirring speed, two minutes of stirring duration, and room temperature for the hardening process. In this study, permittivity is the only subject to be further discussed in TLFA since waveguide measurements are acknowledged to have limitations in precisely measuring the low-loss material.

Other than that, Figure 1 illustrates how different factors interact to influence the permittivity at 5 GHz. Orange bars represent positive influences, whereas blue bars denote negative ones. A factor is considered to have a positive effect when there is a proportional increase between the factor and the response value, and a negative effect when the increase is disproportional. Factors that significantly impact the permittivity value are identified by surpassing the t-value limit, which are stirring time (C) and stirring speed (B) being the most influential in this study. Specifically, increased stirring times (C) lead to a rise in permittivity, ranking as the most beneficial single effect due to the more uniform distribution of components throughout the mixture, thereby enhancing material properties. Conversely, a higher stirring speed (B) diminishes permittivity, likely because increased shear forces disrupt mixture uniformity, adversely affecting dielectric properties. Selections for

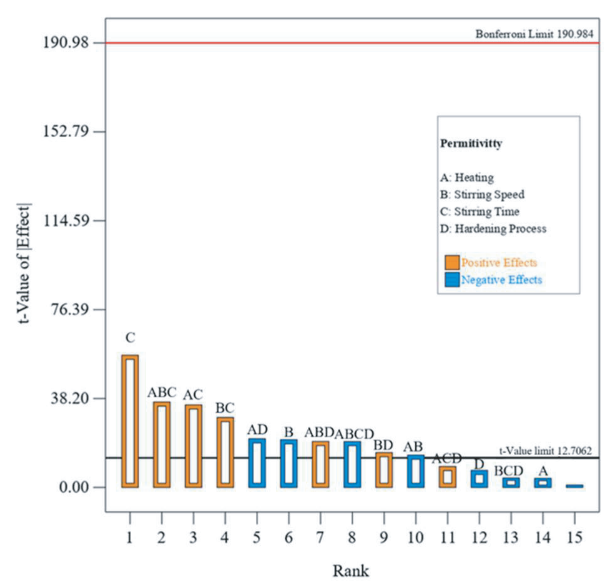

FIGURE 1. Pareto chart for permittivity.

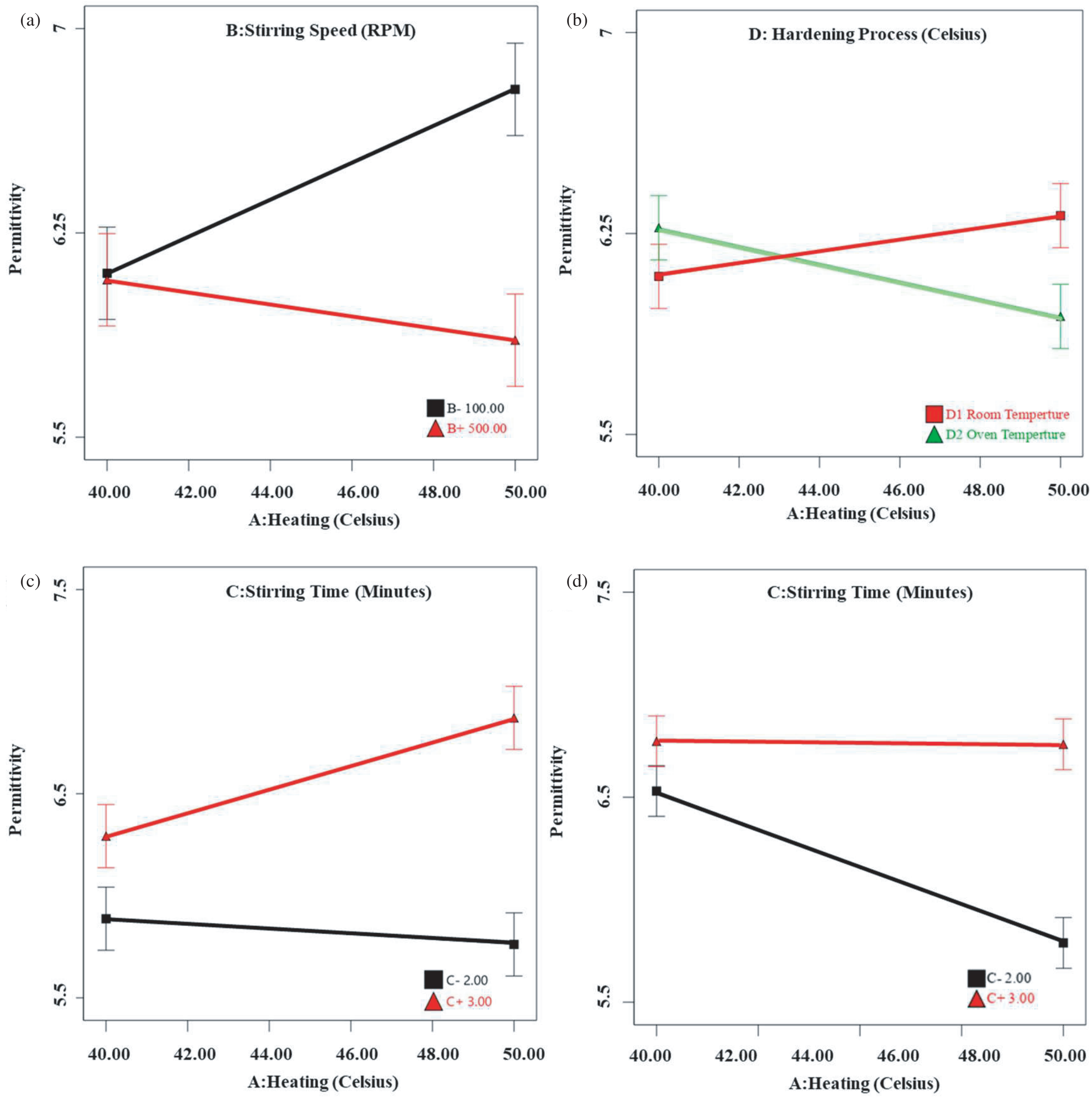

FIGURE 2. Interaction response between variable parameters. (a) The impact of heating temperature and stirring speed on the permittivity. (b) The impact of heating temperature and the hardening process on the permittivity. (c) The impact of heating temperature and stirring duration on the permittivity. (d) The impact of stirring speed and stirring duration on the permittivity.

optimal response are based on achieving the highest values of dielectric permittivity.

3.1.2. Interaction Response Between Parameters (Factor)

The integration of the parameter's effects can be more clearly seen in Figure 2, where the heating, stirring speed, stirring times, and hardening process results are depicted.

The interaction between the heating temperature and stirring speed, from Figure 2(a), shows an upward trend, which indicates that when the stirring speed is at 100 rpm as the heating temperature increases, the permittivity value also increases. Figure 2(a) depicts a decreasing trend in the permittivity value as the heating temperature increases when the stirring speed is at 500 rpm. This trend indicates that the interaction between heating temperature and stirring speed significantly affects permittivity. According to a study by Hasan et al. [17], maintaining a stirring speed of 500 rpm throughout the mixing process of composite materials is crucial. In this study [17], the stirring speed is maintained at 500 rpm during the entire mixing process

TABLE 4. Result of the permittivity and loss tangent of the fabricated dielectric material.

Std	Factor A (°C)	Factor B (rpm)	Factor C (min)	Factor D	Permittivity	Loss Tangent
1	50	100	2	OT	5.5574	0.0138
2	40	100	2	RT	5.7441	0.1313
3	50	500	2	OT	4.9885	0.1082
4	40	500	3	RT	6.1376	0.0238
5	50	500	3	RT	7.0208	0.0238
6	40	500	2	OT	6.0592	0.1294
7	50	500	2	RT	4.6917	0.0099
8	50	100	3	OT	6.3202	0.0337
9	40	100	3	RT	6.4598	0.0262
10	40	100	3	OT	6.3629	0.0236
11	50	100	2	RT	6.8185	0.0329
12	50	100	3	RT	6.7355	0.0472
13	40	500	3	OT	6.3317	0.0038
14	40	500	2	RT	6.0182	0.0482
15	40	100	2	OT	6.3324	0.0137
16	50	500	3	OT	6.8972	0.0349

of composite materials. This consistent speed helps prevent the clumping of filler particles, ensuring a thorough mix. Clumping can lead to the formation of larger particles within the composite, which would reduce the effectiveness of the nanoparticles [10].

Figure 2(b) shows the interaction effect between heating and the hardening process on substrate material. It is shown that at 40°C, the permittivity for the oven temperature of the hardening process is slightly higher than the room temperature hardening process. As the temperature increases from 40°C to 50°C, the permittivity slightly increases in room temperature. In contrast, the permittivity for the oven temperature hardening process slightly decreases. This might be due to the higher humidity level in the room temperature than in the oven temperature, which causes the permittivity level to increase as the heating temperature increases. According to Zafar and Gupta [32] works, both the real and imaginary components of permittivity of the barium titanate-epoxy nanocomposites increase with increasing humidity at all temperatures tested (25–70°C) which shows that the permittivity increases with increasing temperature, indicating that the material becomes more polar and exhibits higher dielectric properties at higher temperatures and higher humidity levels which lead to increased permittivity, indicating that the material becomes more polar and exhibits higher dielectric properties in humid environments.

The interaction of the heating and the stirring time towards the permittivity can be observed from Figure 2(c). For the stirring time of two minutes, it can be seen that the permittivity of the composite material remains almost constant or slightly decreases as the heating temperature increases from 40°C to 50°C. However, the stirring time of three minutes shows a significant increase in permittivity value as the heating temperature increases. The graph shows that stirring time significantly impacts the relationship between heating and permittivity. At higher stirring times, the increase in heating leads to an increase in permittivity, whereas at lower stirring times, the increase in heating leads to a slight decrease in permittivity.

The interaction of stirring speed on permittivity for different stirring times can be further observed in Figure 2(d). It shows that the permittivity decreases significantly when the stirring time is shorter (two minutes), and the stirring speed increases from 100 rpm to 500 rpm. On the other hand, the longer the stirring time (three minutes) is, the more the permittivity remains almost constant as the stirring speed increases, which implies that when the stirring time is extended, the permittivity is less sensitive to changes in stirring speed.

3.1.3. Best Solution of Process Factors for the Preparation of Antenna Substrate

The best conditions for preparing an antenna substrate were obtained as the maximum permittivity is more desirable at 50°C heating process, 500 rpm stirring speed, three minutes stirring times, and hardening process at room temperature, as summarized in Table 5. The permittivity value obtained was 7.0208, and this result can be considered for fabricating the antenna substrate.

High-speed stirring is essential to distributing filler particles evenly throughout the composite mixture. This process should be performed swiftly to minimize the formation of trapped air bubbles, which can adversely impact the permittivity value. According to [33], increasing the stirring speed tends to produce more bubbles. Thus, ensuring the mixture is evenly distributed but with minimum bubble production is essential. This paper proposes the utilization of 500 rpm but only stirred within three minutes to overcome the production.

TABLE 5. Suggested best solution for antenna substrate fabrication.

Factors	Value
A: Heating (°C)	50
B: Stirring Speed (rpm)	500
C: Stirring Times (min)	3
D: Hardening Process	Room Temperature
Permittivity	7.0208
Loss Tangent	0.0238

3.2. Application and Result Analysis of Antenna Fabrication

As shown in the optimal solution in Table 5, the fabrication of the sample reveals that barium titanate has values of 7.0208 and 0.0238 for permittivity and loss tangent, respectively. The high permittivity of this composite material influences the size of the patch antenna, making it suitable for the design of compact antennas, a beneficial attribute for applications that require smaller devices. Additionally, the low loss tangent indicates that dielectric losses are manageable, which supports satisfactory antenna efficiency. These properties confirm the suitability of the barium titanate-epoxy resin composite for patch antenna production.

3.2.1. Antenna Performance

Figure 3 illustrates the construction of the patch antenna, employing barium titanate and epoxy resin as the substrate materials, with the antenna dimensions set at 26.6 mm by 18.6 mm. Measurements for the antenna were carried out using a VNA and a rectangular waveguide across frequencies ranging from 4 GHz to 6 GHz. Additionally, the optimization of the antenna's length and width was performed using CST software.

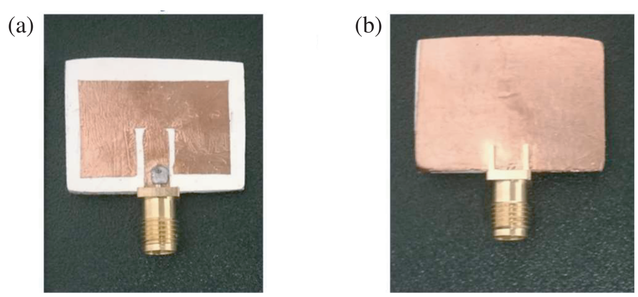

FIGURE 3. (a) Top view and (b) bottom view of fabricated microstrip.

3.2.1.1. Analysis of Width of Patch toward Return Loss

Figure 4 illustrates the patch antenna's varying width affecting the return loss performance. The patch width of 26.6 mm results in the best return loss performance within 5 GHz frequency at −37.96 dB. The performance of other width lengths (28.6, 30.6, and 32.6 mm) shows slightly higher return loss values, implying less efficiency but still giving significant return loss results. Achieving low return loss is important in maximizing power transfer and minimizing signal reflection.

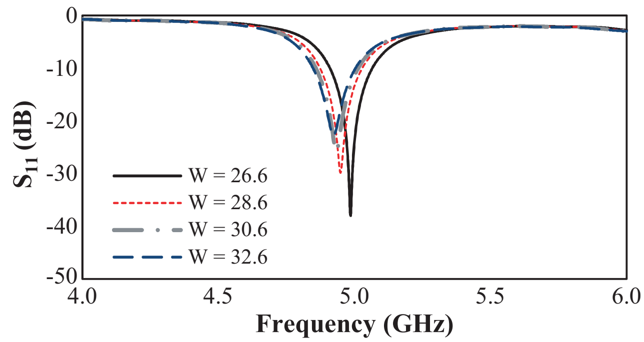

FIGURE 4. Effect of width of patch toward return loss.

3.2.1.2. Analysis of Length of Patch toward Return Loss

Figure 5 shows the effect of the length of the patch antenna towards return loss performance. The patch length of 18.6 mm suggests the best return loss performance, −37.96 dB, within 5 GHz. The performance of another length of patch size (16.5, 22.5, and 22.6 mm) shows higher return loss values, indicating a less efficient but still significant return loss performance than $L = 18.6$ mm. To optimize the design, this result gives important information because it shows the minimal signal reflection and maximum efficiency at the target frequency.

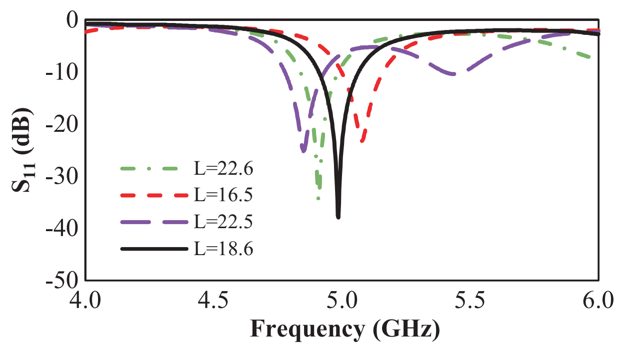

FIGURE 5. Effect of the length of patch toward return loss.

3.2.1.3. Performance of Patch Antenna

Figure 6 shows that the antenna resonates around 5 GHz by observing the depth of the S_{11} parameters at this frequency. The S_{11} parameters indicate how much power is reflected from the antenna, which means the lower the value of S_{11}

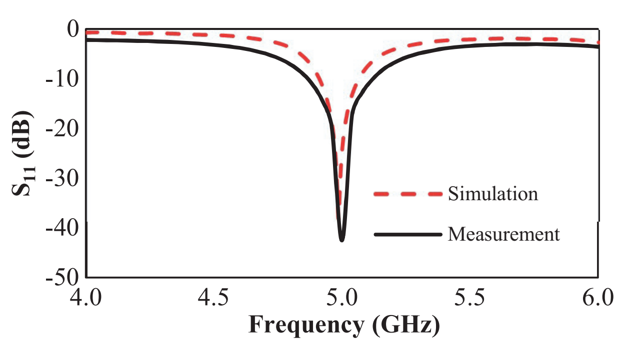

FIGURE 6. Comparison of the S_{11} parameter for simulation and measurement of patch antenna.

is, the less the power is reflected. The measurement shows a minimum -42.52 dB at the resonant frequency, indicating minimum power reflection. At the same time, the simulation result shows a slightly higher minimum value -38.12 dB, indicating a good result, though not as optimal as the measured result. Minor differences between the simulation and measurement results could be due to the fabrication process or measurement setup. Still, both the simulated and measured results show a bandwidth where S_{11} is below -10 dB using barium titanite-epoxy resin composite as the substrate, providing a decent bandwidth for the antenna, making it suitable for application around the 5 GHz frequency range. Using barium titanite-epoxy resin composite as a substrate likely contributes to good dielectric properties, such as high permittivity and low loss, which are advantageous for antenna performance.

3.2.1.4. Voltage Standing Wave Ratio (VSWR)

Figure 7 demonstrates that the patch antenna exhibits a VSWR of 1.137 at 5 GHz, suggesting an almost ideal impedance match with the transmission line. A VSWR below two is typically considered adequate for most antenna applications, while a VSWR of one represents a perfect match, ensuring that all the power from the source is transmitted efficiently with no reflections.

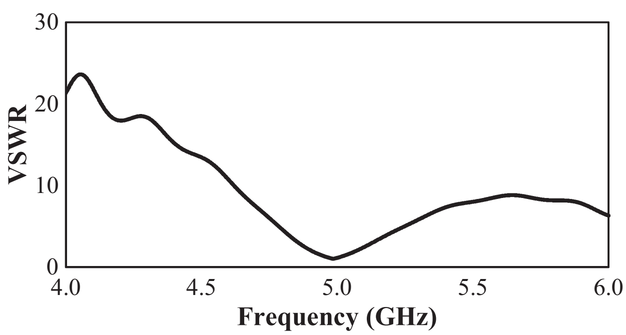

FIGURE 7. Voltage standing wave ratio analysis at 5 GHz.

4. CONCLUSION

A factorial study has been performed to determine the principal factors that enhance permittivity in dielectric materials. The findings suggest that through specific combinations and interactions of elements, it is possible to develop materials with high permittivity, ideal for high-frequency usage. This evaluation was conducted at a frequency of 5 GHz using a specific rectangular waveguide. The results indicated that the optimal TLFA conditions comprised a heating temperature of 50°C, a mixing speed of 500 rpm for three minutes, and subsequent curing at room temperature. Under these specified conditions, the composite material attained a permittivity of 7.0208, qualifying it as a suitable substrate for antennas. Further investigation into the elements that influence dielectric permittivity could involve optimization techniques. Future studies might focus on improving aspects such as the pouring technique to reduce the presence of air bubbles, which negatively affect the material's dielectric performance.

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