

Utilization of Pineapple Leaf in Fiber-Based Dielectric Composite Material and Its Elemental Composition Analyses

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Abstract—The growing demand for natural fibers in dielectric composite production has accelerated research into plant-based materials, particularly those derived from agricultural waste. Hence, this study attempts to evaluate the effect of processing factors and their elemental composition on the permittivity value of pineapple fiber-based dielectric composites. The dielectric composite was prepared following the randomized experimental conditions of two-level factorial analysis, and the permittivity value was measured using a G-band rectangular waveguide. The most significant factors affecting the permittivity value of the dielectric composites and the best conditions were determined. The elemental composition of the dielectric composite was analyzed through an energy dispersive X-ray (EDX) analysis. The best conditions were obtained at a 1 : 10 ratio of pineapple leaves to distilled water, 50 minutes pulping times with a heating effect, and 5 g of pineapple leaf powder. The highest permittivity value of the composite was recorded at 3.31, with the heating effect being the most significant factor. The elemental analysis of the composite with the highest permittivity value presents that carbon was the dominant element in the composite at 78.05%. The obtained permittivity value exhibited by the composites shows that the pineapple leaf fiber-based dielectric composite could be a potential alternative as an antenna substrate.

1. INTRODUCTION

Tons of agricultural waste are being discarded and wasted by farmers without further exploitation globally. Malaysia is no exception. It is reported that 1.2 million tons of agricultural waste are disposed of in landfills annually, representing 39% of the total waste generated in our country [1]. The amount is forecast to rise in the coming years due to increased demand for food from a growing global population. The relentless generation of agricultural wastes could significantly contribute to environmental pollution, not to mention global warming, due to the release of harmful gases into the atmosphere during the burning of these wastes. Apart from agricultural waste, environmental pollution is also caused by abundant electronic waste, which is non-recyclable and non-biodegradable [2]. Electronic waste could pose a major hazard to human health since most materials are carcinogens. The conversion of agricultural waste into useful raw materials for the materials engineering industries seemed to be a potential avenue for turning waste into valuable materials. Furthermore, agricultural waste has an attractive potential for large-scale utilization due to its being readily available, practically free, and renewable.

Numerous efforts and reports have emerged more recently on waste utilization, particularly agricultural wastes to be used in electrical devices [3–5]. Fabricating a dielectric composite from

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agricultural wastes can reduce the pressure on the environment since the demand for synthetic materials could also be reduced. Agricultural waste-based dielectric materials can be potentially used in electrical applications, such as printed circuit boards (PCBs) made from rice husk [6] and antennas made from banana leaves [5]. The most common materials used to fabricate dielectric materials using agricultural waste are neem oil [3], pineapple leaves [4, 7], sugarcane bagasse [5], rice husk [5], rice straw [5], corn husk [8], coconut oil [9], and banana leaves [9]. Pineapple waste, for example, contains many reusable, highly valuable substances that require the application of innovative scientific and technological extraction methods [10]. The continuous rise in pineapple production has generated a large amount of waste upon harvesting due to the elimination of unwanted parts such as pineapple peel, core, crown, and leaf, which account for 75% of waste [6]. Pineapple leaf fibers have traditionally been extensively used in the textile and paper industries. Due to their high cellulose composition, pineapple leaf fibers have been used as reinforcing agents in composite materials [6, 11–13]. Pineapple leaf can be used as a raw material to produce dielectric material.

Dielectric property is an important property in microwave communications and applications that determines the ability of the material to absorb signals [8, 14, 15]. It controls how well the high-frequency alternating electric field can be absorbed and how quickly the materials can be heated [16, 17]. The electromagnetic signal is absorbed by a good electromagnetic absorber and converted into heat. This is due to the elemental composition present in a material's ability to absorb signals. Therefore, the study of elemental compositions is crucial to understanding how a material behaves since each element is responsible for the dielectric properties of such material. The carbon element, for example, plays a significant role in absorbing or storing the electromagnetic signal, and materials with a higher carbon composition tend to absorb more electromagnetic signal, thereby increasing the permittivity value of the material [14, 15, 18–20]. The elemental composition of the pineapple leaf fiber has been studied by Gadzama et al. [21] to investigate the effect of phase changes before and after chemical extraction. The dispersive energy X-ray (EDX) analysis of the fiber shows that the fibers consist of carbon, oxygen, calcium, sodium, aluminum, chlorine, and nitrogen, with carbon and oxygen being the two most abundant constituents. The EDX analysis of the fiber also shows no significant changes in the extracted nanocellulose [21].

Two-level factor analysis (TLFA) is a factorial analysis method commonly used to determine the effect of certain factors on output. TLFA is a quick analysis in which just two levels of each factor are explored [22]. The factorial analysis is useful for reducing a large number of linked variables to a manageable amount before utilizing them in other analyses, such as multiple regression or multiple analyses of variance [23]. Researchers widely used TLFA to analyze the effects of processing factors during composite preparation. Sallih et al. [24] employed a factorial analysis to investigate the effect of material and processing parameters on the mechanical properties of kenaf fiber-polypropylene composite sheets. Isoda et al. [25] utilized factorial design to prepare the activated carbon from rice husks. Okeke et al. [26] used factorial design to investigate the effect of processing parameters on the mechanical properties of a walnut shell-polyethylene composite. Meanwhile, Gurkan et al. [27] applied a full factorial design approach to obtain maximum adsorption of copper and zinc by groundnut biochar-calcium alginate composites. In this study, TLFA was applied to analyze the most significant factors in dielectric composite preparation to produce a composite from pineapple leaf fibers with good performance. The elemental composition analyses were also performed on the fabricated material in order to investigate the effect of elemental composition on the performance of the developed material. Four factors were evaluated, taking into account the ratio of pineapple leaves to distilled water, pulping time, heating option, and weight of pineapple powder.

2. MATERIALS AND METHODS

2.1. Materials Collection and Preparation

Pineapple leaf (PL) was used in this study and was collected from a pineapple plantation located at Pekan, Pahang, Malaysia. The collected leaf was cleaned to remove dirt and other impurities before undergoing chemical treatment. The PL was chopped into 50 mm long pieces before being treated with sodium hydroxide (NaOH). The leaf was treated with a 5 wt.% NaOH solution at a constant temperature of 100°C. The solution was mixed with distilled water using a mass of pineapple leaves and a mass-to-

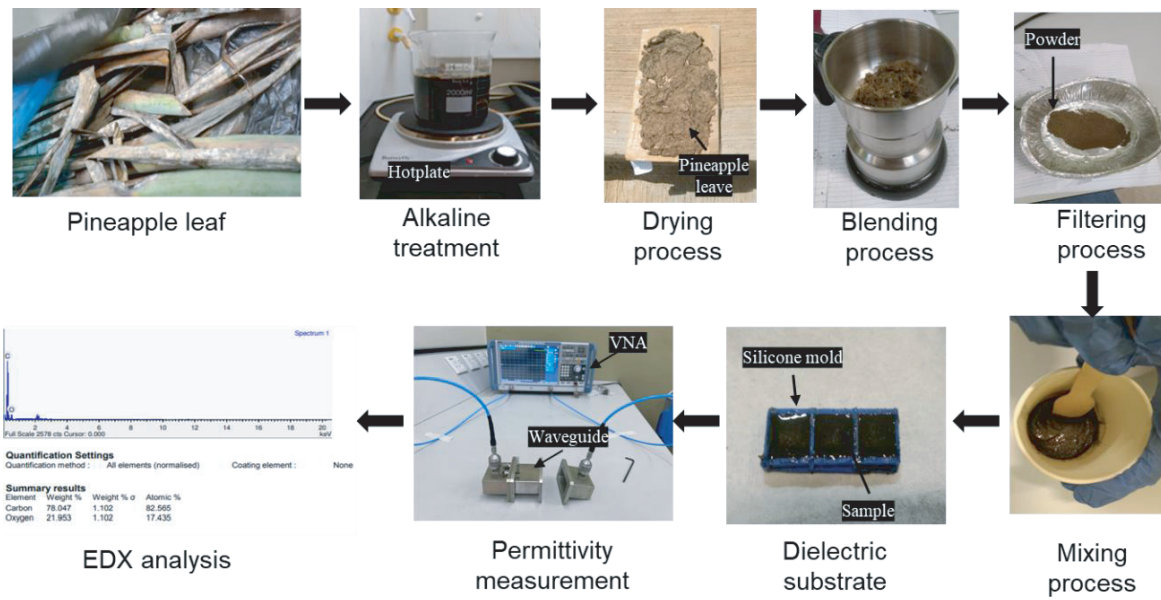


Figure 1. Experimental flow for preparation of dielectric composite.

solution ratio as calculated by Eq. (1). The cool-cooked mixture was filtered and sun-dried for 24 hours after boiling. The sample was then filtered and blended to obtain pineapple leaf powder. The overall experimental flow for this work is portrayed in Fig. 1.

$$5\% = \frac{\text{Mass of NaOH}}{\text{Mass of distilled water}} \times 100\% \quad (1)$$

2.2. Experimental Setup

Two-level factorial analysis (TLFA) was used to determine the significant factors affecting the permittivity value and the best conditions for dielectric material fabrication with a maximum permittivity value. TLFA is an effective statistical method for identifying and evaluating the most important factors [28–30]. Four factors, including the pineapple leaf to distilled water ratio (PL : DW) (1 : 10 and 1 : 15), pulping time (30 and 50 minutes), heating option (heated and non-heated), and weight of PL powder (3 and 5 g), were investigated. The fabrication of the composite was carried out according to the design table as tabulated in Table 1. The table was constructed by the Design-Expert software, in which all factors were completely randomized over 16 experimental runs. The permittivity value obtained was the indicator used to evaluate the performance of the fabricated composites. The outputs were analyzed by the named software through an analysis of variance (ANOVA) with a 95% confidence level. The ANOVA functions to determine the coefficient of the model and verify the significance of the factors chosen. It is also to determine the suitability of the chosen range [31–33].

2.3. Composites Fabrication

A mixing procedure was used to create random-oriented pineapple leaf molds from epoxy composites, as pictured in Fig. 1. The composite was prepared by mixing pineapple leaf powder with epoxy. The mixing procedure required special attention since an over-stirred mixture could lead to the release of heat. On the other hand, less stirring leads to an uneven distribution of the pineapple powder. The pineapple leaf powder was heated for three (3) minutes before the mixing procedure. The weight of pineapple powder was prepared according to Table 1. Meanwhile, the weight of epoxy was maintained at 25 wt.%, with 19.5 wt.% epoxy resin and 5.5 wt.% hardeners. The composite was then poured into a mold measuring 22.15 mm wide and 22.15 mm long.

Table 1. Experimental design table for composite preparation and its corresponding permittivity value.

STD.	PL : DW (g/ml)	Pulping Times (min)	Heating Option	Weight of Powder (g)	Permittivity
1	1 : 10	30	Heated	3	2.86
2	1 : 15	30	Heated	3	3.03
3	1 : 10	50	Heated	3	3.00
4	1 : 15	50	Heated	3	3.03
5	1 : 10	30	Non-Heated	3	2.82
6	1 : 15	30	Non-Heated	3	2.74
7	1 : 10	50	Non-Heated	3	2.94
8	1 : 15	50	Non-Heated	3	2.87
9	1 : 10	30	Heated	5	2.88
10	1 : 15	30	Heated	5	3.00
11	1 : 10	50	Heated	5	3.31
12	1 : 15	50	Heated	5	3.12
13	1 : 10	30	Non-Heated	5	2.71
14	1 : 15	30	Non-Heated	5	3.00
15	1 : 10	50	Non-Heated	5	2.80
16	1 : 15	50	Non-Heated	5	2.82

2.4. Composites Characterization

2.4.1. Permittivity Test

Permittivity measurement could be done using either free-space, waveguide [34,35], or resonant techniques [36–39]. In this work, wide-band measurement, i.e., waveguide technique, is preferred in order to provide dielectric value for wide applications. The permittivity was measured using a vector network analyzer (VNA) and a rectangular waveguide [34,35]. The TE₁₀ mode of dominant transverse electric was used in the rectangular waveguide within the frequency range of 4 to 6 GHz in G-band waveguides. The composite was prepared according to the G-band waveguide height of 22.15 mm. The sample must be solid and properly cut with a parallel surface to fill the waveguide's cross-sectional area. The phase and magnitude of the transmission coefficient, S_{21} , were measured using VNA before the value of permittivity and lost tangent of the composite material were estimated using the inverse technique [34,35].

2.4.2. Elemental Composition Analysis

In this section, five dielectric composites with the lowest, average and highest permittivity values were used to analyze the elemental composition. The developed composite was first coated with gold using Quorum Technologies Q300TD sputter coating to improve the image of the sample and prevent charging of the surface. The morphological structure of the composite was observed through the TM3030 Plus Benchtop Scanning Electron Microscope (SEM), and the elements in the fabricated composites were analyzed using energy dispersive X-ray (EDX) analysis.

3. RESULTS AND DISCUSSIONS

3.1. Permittivity Value

Table 1 presents the permittivity values for 16 experimental runs at 5 GHz. The obtained permittivity values ranged from 2.71 to 3.31, depending on the experimental setup. The composite had the highest

permittivity value at a fabrication condition of 1 : 10 PL : DW, 50 minutes pulping times, a heated condition, and 5 g of pineapple leaf powder. Meanwhile, the smallest permittivity value was obtained at an experimental setup of 1 : 10 PL : DW, 30 minutes of pulping times, non-heated conditions, and 5 g of pineapple leaf powder.

3.2. Analysis of Variance (ANOVA)

ANOVA was used to analyze the permittivity value of the composite material based on a 95% confidence level. The statistical analysis is summarized in Table 2. A p-value of less than 0.05 indicates that the model is significant. It was observed that the p-value of the model was 0.0018. The p-values of the main factors of PL : DW, pulping times, heating option, and powder weight were lower than 0.05, which clearly implies that the factors are significant toward the permittivity value of the dielectric composite material. The R^2 value of 0.9997 is close to 1, which shows the high statistical significance of the model, and the model was accepted and could represent the process [40].

Table 2. ANOVA for permittivity value of dielectric composite at 95% confidence level.

Source	Sum of Square	df	Mean square	F value	p-value Prob > F
Model	0.36	13	0.027	536.06	0.0018
A: PL : DW	6.291E-003	1	6.291E-003	129.46	0.0076
B: Pulping Time	0.0051	1	0.0051	1045.11	0.0010
C: Heating Option	0.14	1	0.14	2876.88	0.0003
D: Powder Weight	8.701E-003	1	8.701E-003	179.06	0.0055
AB	0.033	1	0.033	674.84	0.0015
AD	1.643E-003	1	1.643E-003	33.80	0.0283
BC	0.020	1	0.020	403.42	0.0025
CD	0.012	1	0.012	253.52	0.0039
Residual	9.719E-005	2	4.859E-005		
Cor Total	0.36	15			
R^2	0.9997				
Adjusted R^2	0.9980				

The interaction between the main factors affecting the permittivity value of the dielectric composite can be described by the final equation in a coded term, as displayed by Eq. (2). The equation presents the model that correlates the interaction between input and output variables with A, B, C, and D, denoting PL : DW, pulping times, heating options, and powder weight, respectively. Meanwhile, Eqs. (3), (4), (5), (6) present the final equations in terms of actual factors differentiated by the categorical factors of PL : DW ratio and heating options, with Eq. (3) presenting STD 5, 7, 13, and 15, Eq. (4) presenting STD 6, 8, 14, and 16, Eq. (5) presenting STD 1, 3, 9, and 12, and Eq. (6) presenting STD 2, 4, 10, and 12.

$$\text{Permittivity} = 2.93 + 0.02A + 0.056B + 0.093C + 0.023D - 0.045AB + 0.001AD + 0.035BC + 0.028CD - 0.013ABC - 0.026ABD - 0.047ACD + 0.046BCD + 9.042^{-3}ABCD \quad (2)$$

$$\text{Permittivity}(1 : 10PL : DW; \text{non} - \text{heated}) = 2.289 + 0.019C + 0.073 \quad (3)$$

$$\text{Permittivity}(1 : 15PL : DW; \text{non} - \text{heated}) = 1.790 + 0.022C + 0.285D \quad (4)$$

$$\text{Permittivity}(1 : 10PL : DW; \text{heated}) = 3.440 - 0.019C - 0.252D \quad (5)$$

$$\text{Permittivity}(1 : 15PL : DW; \text{heated}) = 2.941 + 6.792^{-4}C - 0.0128D \quad (6)$$

3.3. Effect of Independent and Interactive Factors on Permittivity Value

Figure 2 shows a Pareto chart that indicates the response of all factors toward the permittivity value of the dielectric composite at 5 GHz. All main factors are significant on the permittivity value since the bars lie above the t-value limit line, with both factors C and B positioned above the Bonferroni limit line. Also, all factors contributed positively to the permittivity value displayed by the orange-colored bar. Based on a single effect, factor C (heating option) was the most influential factor contributing to the permittivity value of the composites, followed by factor B (pulping time), factor D (powder weight), and factor A (PL : DW ratio).

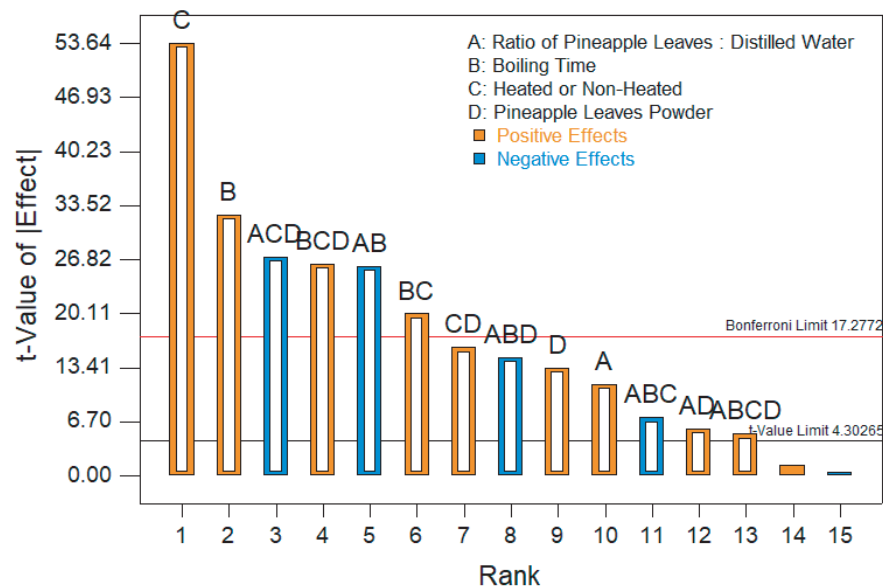


Figure 2. Pareto chart of two-level factorial analysis.

Figure 3 presents the effect of the main factors on the permittivity value of the dielectric composite. The heating of the pineapple leaf powder (Fig. 3(c)) before the mixing procedures significantly affects the permittivity value of the developed dielectric composite. This is due to the existence of more carbon elements in the heated composite. The increased carbon elements resulted in an increased permittivity value [41]. Pulping time is also one of the main factors contributing to the high permittivity value of the composite. As seen in Fig. 3(b), the increase in pulping times significantly increased the permittivity value. This is due to more cellulose being extracted with longer pulping times since more pineapple leaves are delignified.

The two interacting factors that contribute the most to the permittivity value are depicted in Fig. 4. The interaction between pulping times (B) and the heating option (C), as displayed in Fig. 4(a), shows that the heating of the pineapple powder plays a significant role in the permittivity value increment. Both heated and non-heated samples require longer pulping times to maximize permittivity values. Likewise, the interaction between the heating option (C) and powder weight (D), as presented in Fig. 4(b), also demonstrates that the heating option is important in producing composite materials with high permittivity. The nonparallel line connecting both factors demonstrates that there is an interaction between them, and that the interaction has an effect on the permittivity value. The maximum permittivity value is obtained at 5 g of powder weight in a heating condition.

Therefore, the best condition to produce dielectric material was selected based on the highest permittivity value. Based on the values, the best conditions for maximizing permittivity were a 1 : 10 PL : DW ratio, 50 minutes of pulping time, a heated condition, and 5 g of pineapple leaf powder with a 3.31 permittivity value. Jayamani et al. [4] investigated the permittivity value of pineapple leaf fiber-reinforced epoxy composites as a function of fiber loading. It was discovered that as the fiber concentration in the composites increased, so did the permittivity value. The highest permittivity value

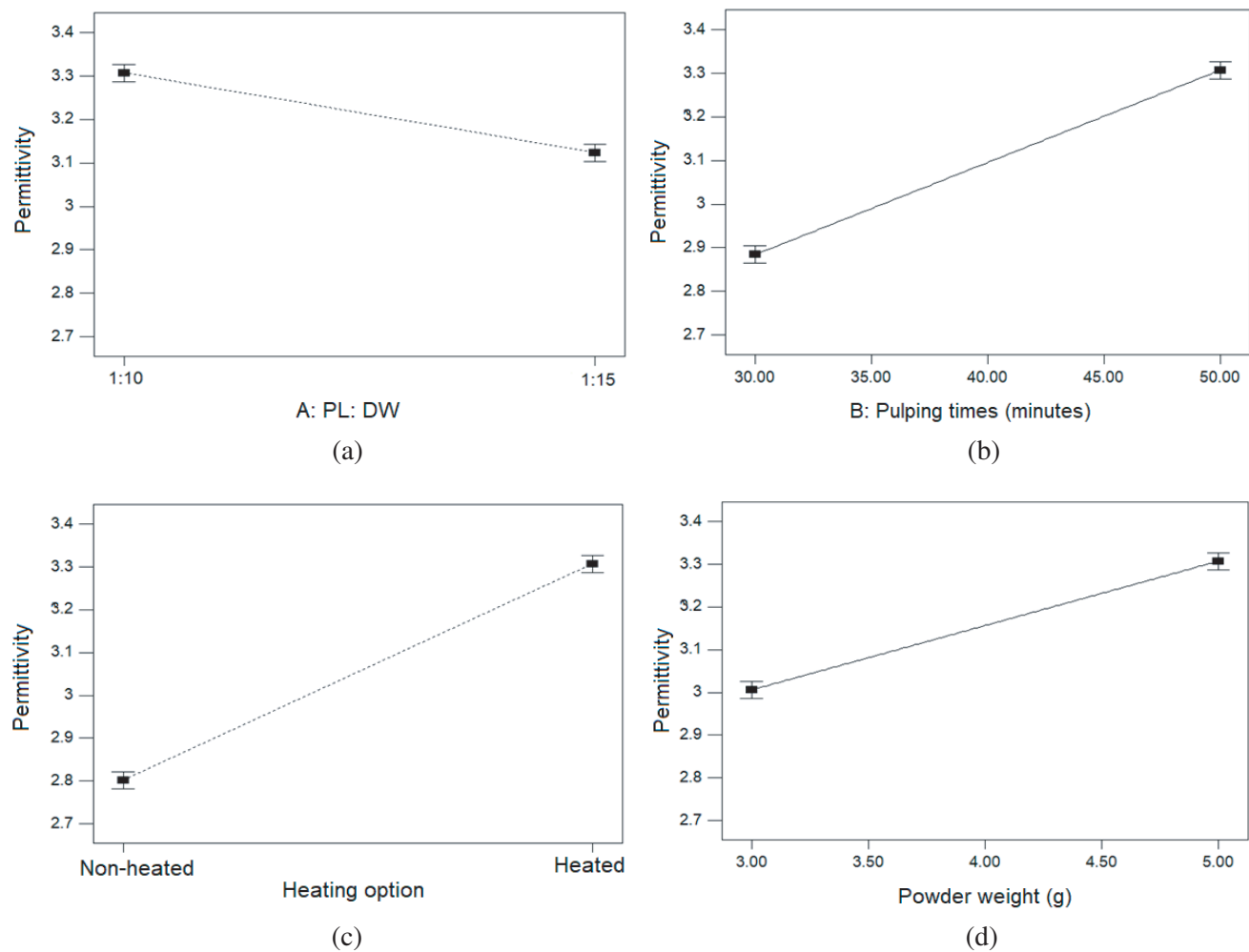


Figure 3. Effect of main factors on permittivity value of dielectric composite at best conditions.

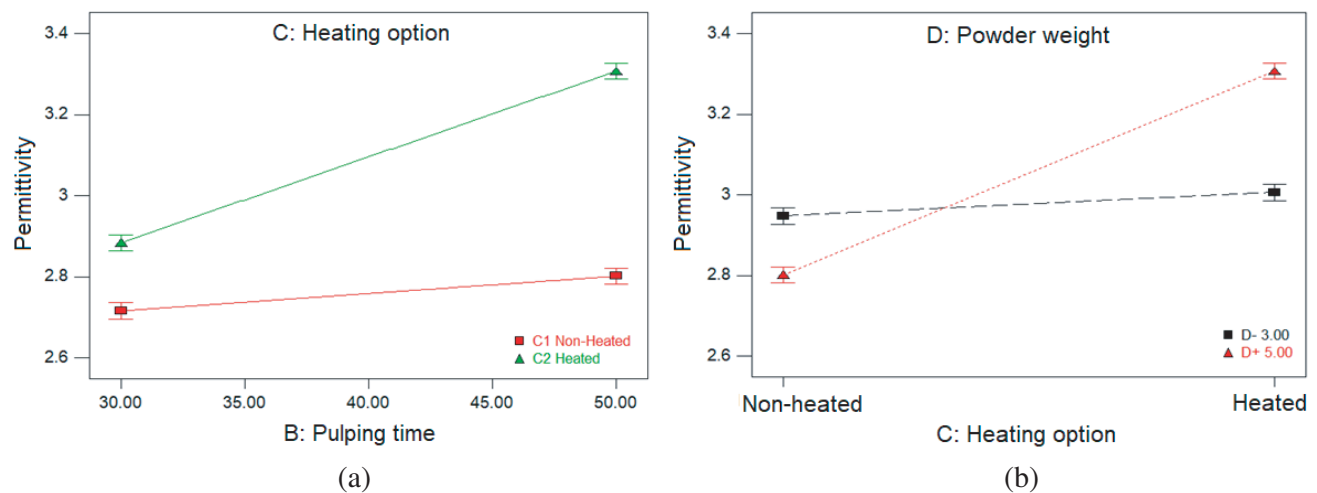


Figure 4. Interaction effect between main factors of (a) BC and (b) CD on permittivity value of dielectric composite.

of 4.2 was obtained at 20% fiber concentration at 3 MHz. The high permittivity value obtained may be attributed to the increase in fiber loading since the presence of more pineapple leaf fibers in the composites resulted in increased interfacial polarization due to heterogeneity. Zulkifli et al. [5] studied the permittivity values of sugarcane bagasse, banana leaf, rice husk, and rice straw for microwave communication applications. The permittivity values obtained vary depending on the epoxy loading and materials used. Banana leaf composites exhibited the highest permittivity value with 4.22 at 50% epoxy loading, followed by rice husk and rice straw with 3.39 and 3.23, respectively. Meanwhile, the sugarcane bagasse composite shows the lowest permittivity value of 2.18 at the same epoxy loading.

Patra and Bisoyi's [42] evaluation of the permittivity value of sisal fiber-reinforced polyester composites revealed that the composites had a permittivity value of up to 6.6. The high permittivity value obtained is due to cobalt naphthalene being used during composite preparation, which acts as an accelerator. Table 3 summarizes the findings of the current study with other research. The value obtained from the current study is acceptable and comparable with other research. Therefore, the permittivity value of the pineapple leaf fiber composite obtained in this study could be regarded as high, and further exploitation as a dielectric composite is possible.

Table 3. Comparison of permittivity value between this and previous study.

Raw materials	Permittivity value	Frequency	References
Pineapple leaf	3.31	5 GHz	Current study
Pineapple leaf	4.20	3 MHz	[4]
Sugarcane bagasse	2.79	1–20 GHz	[5]
Banana leaf	4.22	1–20 GHz	[5]
Rice husk	3.39	1–20 GHz	[5]
Rice straw	3.21	1–20 GHz	[5]
Sisal	6.60	10 kHz	[42]

In terms of the durability of the materials produced, bio-based materials from agricultural waste are inevitably prone to degradation due to their minimal resistance to environmental degradation. This could be a merit for bio-based fiber composites since they can be disposed of easily, reducing environmental pollution [44]. Nevertheless, the durability and lasting duration might be elongated when being composited with epoxy. This is due to the nature of the epoxy, which can withstand heavy pressure and is practically impossible to break once dried, owing to its high mechanical strength [45]. Also, the use of natural fibers in dried powder form during fabrication could increase the durability of the material as it was diluted into the epoxy system. However, to date, the long-lasting nature of bio-based composites remains a concern due to the undesired degradation of the material with constant exposure to severe environmental damage.

3.4. Elemental Composition

Five samples were selected based on the lowest, average, and highest permittivity values for SEM analysis, which involves EDX analysis to find the elemental composition of the composite material. The samples were chosen to compare the elemental composition of the developed composite between the maximum and minimum permittivity values to understand how elemental composition can affect their permittivity value. Also, the selection was made to obtain an initial observation between the highest and lowest permittivity values since the obtained permittivity values were barely different among samples; hence, it is insignificant to analyze all samples. The correlation between the permittivity value and element composition of the composite material is discussed in this section.

3.4.1. Carbon Composition

Figure 5(a) displays the interaction between the carbon percentage and permittivity value. It is clearly portrayed that the increase in carbon composition in the composite material significantly increased the

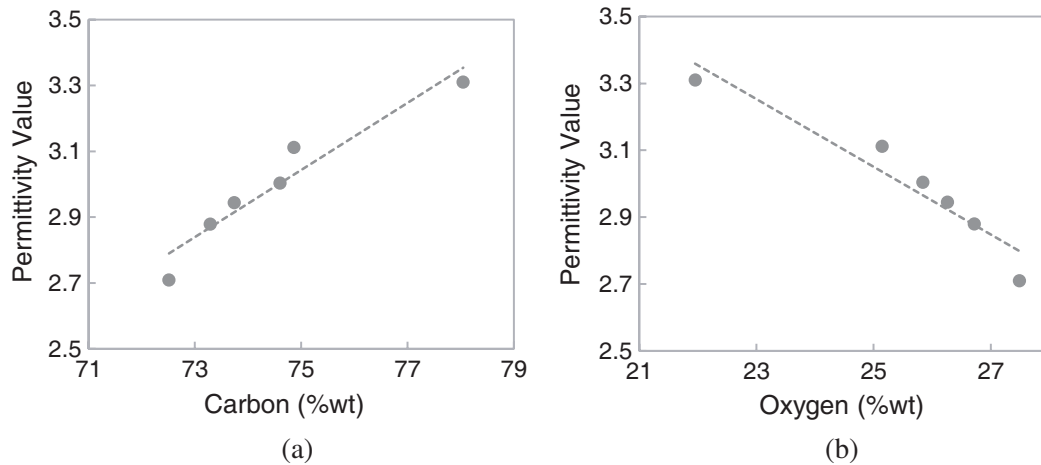


Figure 5. Interaction between (a) carbon and (b) oxygen with permittivity value.

permittivity value. The lowest carbon percentage of 72.51% contributed to the lowest permittivity value of 2.71. Meanwhile, the highest permittivity value of 3.31 was attributed to the high carbon composition of 78.05%.

The carbon percentage for the highest permittivity value is shown in Fig. 6. The EDX analysis shows that carbon composition was dominant in the dielectric composite. The amount of carbon in the formulated dielectric material affects the permittivity value of the material. As mentioned earlier, the high permittivity of a material is the main indication of its high carbon content [41]. Carbon acts as an absorbent and storage material for electromagnetic signals, and materials with a higher carbon composition tend to absorb more electromagnetic signals [5], thus increasing the permittivity value of the material. This is due to the properties of carbon that aid in the absorbance and storage of the electromagnetic signal in a material [43]. Meanwhile, the lower carbon percentage in the dielectric composite will produce a lower permittivity value due to the low carbon composition, which will reduce the ability of the composite to absorb or store the electromagnetic signal.

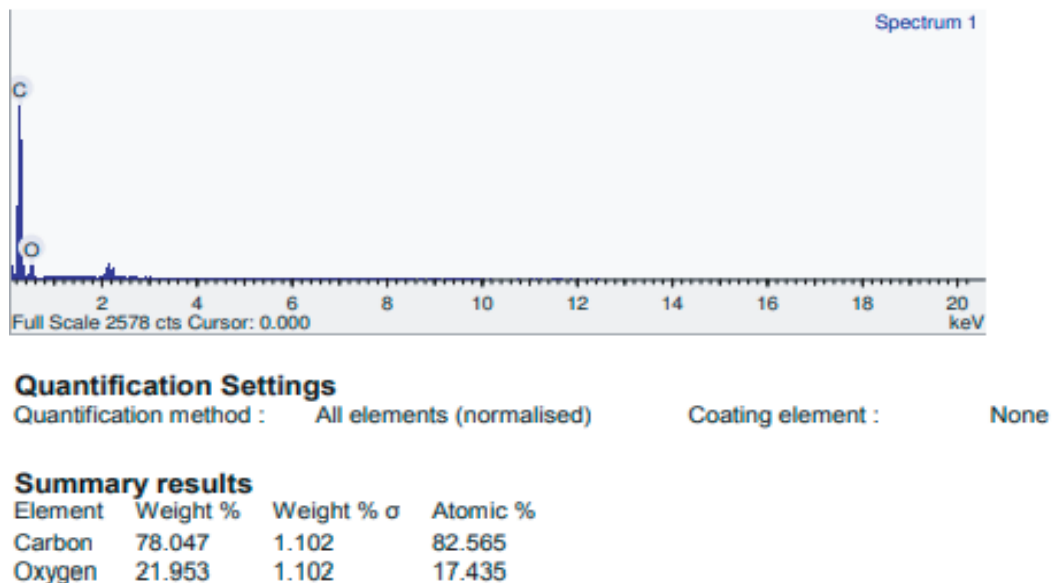


Figure 6. EDX analysis of dielectric composite with the highest permittivity value.

3.4.2. Oxygen Composition

The interaction between the oxygen percentage and the permittivity value is displayed in Fig. 5(b). Unlike carbon, oxygen has a different effect on the permittivity value of the dielectric composite. The permittivity value decreased as the oxygen concentration increased. The high oxygen percentage of 72.49% in the composite material resulted in a low permittivity value of 2.71. Hossain and Roy [43] mentioned that the permittivity value decreased as the oxygen element increased in the dielectric material. This is due to the formation of bubbles with oxygen inside the dielectric material, known as pores. The pores are the void spaces in the circular form that are filled with oxygen. The more oxygen is in the composite material, the less ability it has to absorb or store electromagnetic signals, reducing its permittivity value.

4. CONCLUSION

The permittivity value of the pineapple fiber-based dielectric composite obtained in this study shows that the agricultural waste-based material can be developed into a dielectric composite. The best conditions to produce a dielectric composite with the highest permittivity value of 3.31 were determined at processing conditions of 1 : 10 PL : DW, 50 minutes of pulping times, heated conditions, and 5 g of pineapple leaf powder. The percentage of elemental components in the fibers is crucial in determining their effects on permittivity value. The permittivity value of the developed composite obtained in this study shows that agricultural waste, particularly pineapple leaf, could be a potent substitution for expensive, non-biodegradable synthetic materials used to formulate dielectric material.

Based on the results obtained in this study, among the potential applications that can be proposed for the pineapple leaf fiber-based material are antenna substrates and printed circuit boards (PCB). The limitation of this current study would be the continuous supply of pineapple waste. This is due to uncontrolled external factors such as weather conditions, with floods in particular, which might devastate the pineapple plantation, contributing to the limited supply of the pineapple leaf. In addition, limited storage capacity and proper storage are other limitations of this study. Therefore, to overcome the storage issue, the fabrication procedures were conducted immediately upon sample collection to avoid microbial infections, which could ruin the fiber composition. This precaution is crucial as the materials are prone to natural degradation. For large-scale production, especially for industrial use, it is suggested to produce the powder form of the pineapple leaves in large amounts so that storing the materials could be easier. This is due to the long lifespan of the dried powder form compared to unprocessed raw pineapple leaves when being properly stored. Also, it is critical to consider the long-term durability and stability of the produced dielectric composite material. It is recommended to investigate its performance under different environmental conditions, such as exposure to various chemicals, temperature variations, and humidity levels.

Information on the effects of processing factors on a dielectric composite fabrication is necessary to produce the material with the desired properties. The findings of this current study could contribute to the selection of significant processing factors. With some modifications to the range of the selected factors, the dielectric composite with the desired permittivity value could be obtained. The best condition obtained in this study could be used to perform an optimization experiment, which could further maximize the permittivity value of the developed dielectric composite. Because of their unique and desirable properties in the formulation of dielectric composites, pineapple leaves can be as appealing as conventional materials under the right formulation and processing conditions.

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