

## Research article

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# Effect of Fiber Morphology and Elemental Composition of *Ananas comosus* Leaf on Cellulose Content and Permittivity

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### Abstract

#### Keywords

pineapple leaf fiber;  
cellulose;  
permittivity;  
morphology;  
elemental composition

The fiber morphology and elemental composition of pineapple leaf fibers were analyzed to understand their effect on the cellulose content and permittivity value. Cellulose fiber was extracted from pineapple leaf via the alkaline treatment method, and the content was determined using the Kurschner-Hanack method. The permittivity value of the developed cellulose fiber was measured based on the waveguide technique in the G-band. The surface morphology of the developed fiber was examined with scanning electron microscopy. Meanwhile, energy dispersive X-ray (EDX) spectroscopy was used to identify the elemental composition of the pineapple leaf fibers. The findings were that the cellulose fibers with the least diameter and distance between fibers exhibited the highest permittivity value, which was 1.85. The EDX analysis demonstrated that carbon was the commonest elemental in all fibers, and was 55 wt.% of the total element composition. Furthermore, the results showed that the permittivity value increased as the carbon composition increased, and decreased as the oxygen composition increased. Hence, the morphological and elemental studies of the cellulose fiber are useful in determining the permittivity value of the cellulose fiber for material development. The high permittivity value of the pineapple leaf fibers is believed to have great potential for use in electronic components.

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## 1. Introduction

Natural fibers from a variety of resources are extensively transformed and used in numerous applications in the materials engineering industries. This is due to the public awareness of the importance of preserving natural resources, which has led to the invention of more ecologically friendly materials. In this regard, since conventional materials are highly resistant to biodegradation, natural fibers are considered appealing alternatives, particularly those obtained from agricultural wastes [1]. Natural fibers have piqued the interest of researchers as a viable alternative to synthetic fibers due to their biodegradability and noncarcinogenic properties. The utilization of natural fibers in producing value-added materials can reduce pressure on the environment since natural fibers are mostly obtained from agricultural wastes [2]. The renewable properties of natural fibers provide a better solution for the sustainable supply of materials that are less health hazardous to health [3]. Compared to natural fibers, synthetic fibers are expensive and nonbiodegradable, and their use has led to abundant electronic waste [4]. Moreover, the cultivation of natural fibers requires only solar energy for production rather than fossil fuel energy, which is the case with synthetic fibers. The excessive use of fossil fuels in the production of synthetic fibers can deplete valuable natural energy sources and cause polluting gas emissions into the environment [5]. Petroleum, for example, is believed to be limited in the future, and it may become an unreliable energy source. Therefore, the exploration of alternative raw materials with sustainable features is necessary.

Many available plant-based fibers such as pineapple leaf, coir, kenaf, cornhusk, cotton, bamboo, banana leaf, and rice husk, have the potential to be raw materials for natural fiber production [6-9]. Pineapple leaves, for example, are one of the agricultural wastes that are mostly discarded after post-harvesting and are wasted in most pineapple plantations. Pineapple, or *Ananas comosus*, is most commonly consumed as it is, or it can be used to manufacture canned fruit, cordial, and food spread. However, the remaining parts of the pineapple fruit and plants, such as the leaves, core, and peel, are discarded. Tonnes of pineapple leaves are being produced yearly, as shown by the increased size of a pineapple plantation, though only a minor portion is being used for feedstock and energy production [2]. Therefore, the application of natural fibers from pineapple leaves in composite production for industrial use appears to be a positive way of reducing the waste of these renewable materials [1, 10]. Pineapple leaves can also be used as dielectric materials. The dielectric properties of a material are a crucial characteristic in microwave communications and applications that regulate the ability of the material to absorb signals [11]. Electromagnetic (EM) signals are absorbed by good EM absorbers and transformed into heat. Pineapple leaves can be used as dielectric insulators in coaxial cables, capacitors, switchboards [12], and microwave absorbers [13].

Natural fibers commonly consist of cellulose, hemicellulose, and lignin, with cellulose being the primary constituent [14]. Cellulose consists of fibrils bonded together by hydrogen bonds, providing strength and flexibility. The pineapple leaf comprises 70 to 82 wt.% cellulose [14, 15], with each fresh pineapple leaf yielding about 2 to 3 wt.% fiber [2]. The cellulose content differs according to the variety of pineapple plants as well as ecological factors such as location of cultivation. Pineapple leaf has high specific strength and stiffness and is naturally hydrophilic [4] due to its great cellulose composition [2]. Pineapple leaves have superior mechanical properties due to its high cellulose content and relatively low microfibrillar angle [16] and can be used as composite reinforcement to produce biodegradable plastic composites as well as low-density polyethylene (LDPE) composites [2].

A morphological study provides important information for the development of fibers with desired properties. The morphological characteristics of fibers, such as crystallinity, polarity, fiber diameters, composition, and arrangement, are important since they define the electrical, physical, chemical, and mechanical properties of the fibers [17]. The morphological features of a fiber can significantly affect the properties and performance of a material since every feature explains its

effect on that material. Therefore, the morphology of natural fiber is indeed crucial in shaping its properties. For example, fibers with a higher cellulose composition are relatively high in tensile strength due to the cellulose's high crystallinity [15]. This high crystallinity is due to intermolecular bonding being more significant in the crystalline phase [18]. In addition to physical and mechanical properties, morphological structures can also be affected by the chemical or elemental composition of the fibers. For instance, the carbon composition of fibers is believed to be responsible for the high permittivity value of a material. This is because carbon can act as a storage and absorbent material for electromagnetic signals [19, 20].

The application of pineapple leaf fibers in materials engineering as reinforcing agents has been well documented by many authors. However, as far as we know, studies on the morphology and elemental composition of pineapple leaf fiber are scarce. In addition, although pineapple leaf fiber composites possess enhanced mechanical features, their use remains restricted in industrial applications, particularly in electronics components [21]. This restriction is due to insufficient information on their morphological features, which can affect their dielectric properties in terms of permittivity value. Understanding the dielectric behavior of pineapple leaf fibers promotes their use in the materials engineering industry. Therefore, this study was initiated to identify the effects of the fiber morphology and elemental composition on the cellulose content and permittivity values of the pineapple leaf fiber.

## **2. Materials and Methods**

### **2.1 Sample collection**

The leaves of the pineapple (*Ananas comosus*) were utilized as the study material and were collected from a pineapple plantation at Pekan Pina, Pahang, Malaysia. The collected leaves were cleansed to eliminate impurities and were dried in an oven at 100°C. The dried leaves were cut into 2-cm-long pieces.

### **2.2 Cellulose fiber extraction by alkaline treatment**

An alkaline treatment method was applied to extract cellulose from the pineapple leaves. Sodium hydroxide (NaOH) was used in the treatment. The experiment was performed according to the 16 experimental conditions constructed by the Design-Expert software using two-level factorial analysis, with randomized factors of pineapple leaf to NaOH ratio (1:5 and 1:10), NaOH concentration (5 and 10 wt.%), cooking temperatures (60 and 100°C), and pulping times (45 and 75 min). Two-level factorial analysis is a statistical method that was used to identify and evaluate the most significant process factors during fiber extraction [22-24]. The experimental data were analyzed through the analysis of variance (ANOVA) with a 95% confidence level. ANOVA was used to determine the coefficients of the model and validate the significance of the chosen factors [25-27].

One hundred grams of pineapple leaves were initially boiled in a mixture of NaOH and distilled water, in which different NaOH amounts were prepared for the extraction process. The boiled leaf mixture was filtered using a mesh cloth filter and thoroughly washed before being squeezed until a clear effluent was obtained. The squeezed pineapple leaf pulp was dried at 60°C. The dried pulp was then ground to obtain pineapple pulp powder. The cellulose content was then determined by the Kurschner-Hanack method [28], in which nitric acid and acetic acid were used as reagents. Concentrated nitric acid (1.5 mL) and 80% acetic acid (15 mL) were poured into a test tube containing 1 g of pineapple pulp powder. The test tubes were placed in a beaker containing

distilled water and were heated for 20 min at 100°C. The mixture was filtered and dried in an oven at 105°C for 24 h. The weight of the powder was then determined in order to obtain the cellulose content (wt.%).

### 2.3 Permittivity test of cellulose fibers

The permittivity measurement can be performed using the free-space, waveguide [29-31], or resonant [32-35] techniques. In this study, the permittivity was measured using the waveguide technique in the microwave region [29, 30], with the measurement taking place in the G-band, which covers the frequency ranges of 3.95 to 5.85 GHz. The waveguide technique is an established and proven technique and was preferable method in this study because it enables permittivity value measurement over a wide range of frequencies. The resonant technique may be more accurate; however, it is limited to a narrow range of frequencies compared to the waveguide technique.

The material under test (MUT) height was adjusted to 22.15 mm in order to fit the height of the waveguide. Meanwhile, the width was adjusted to not exceed the waveguide's width of 47.55 mm. The MUT was positioned at the center of the waveguide to obtain high-sensitivity detection of transmission signal changes since the electric field is strong at the center. A vector network analyzer (E5071C) was connected to the waveguide to measure the magnitude and phase of transmission parameters.

An inverse technique was used to predict the permittivity, or dielectric constant, of the MUT, based on the measured transmission parameter. The transmission parameter was determined by the electromagnetic theory formula by estimating the initial value of the dielectric constant [36]. The estimation was repeated until the calculated and measured transmission parameter values were in reasonable agreement. The final estimation value was then adopted as the dielectric constant value of the MUT.

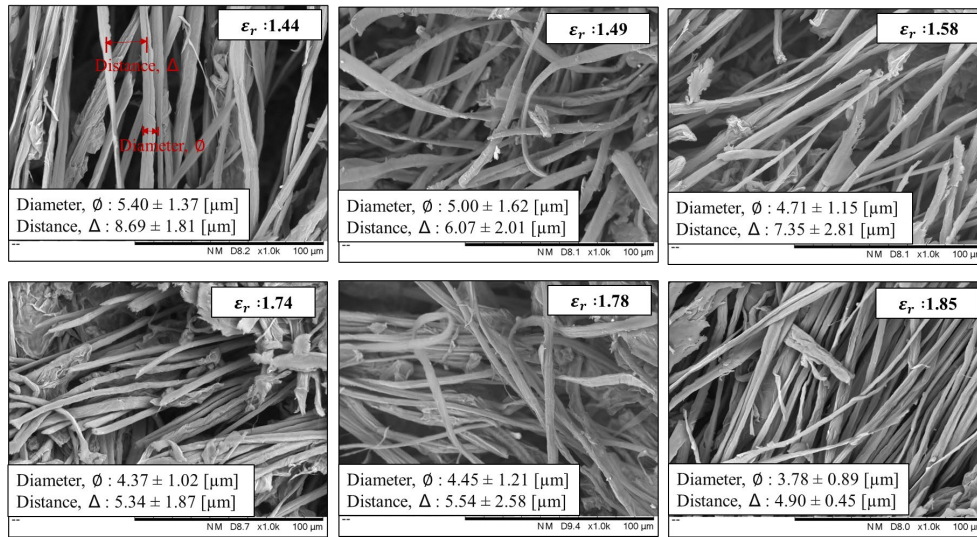
### 2.4 Analysis of the morphological structure and elemental composition of cellulose fibers

The surface morphology of cellulose fibers from pineapple leaves was examined from the top view with scanning electron microscopy (SEM; Hitachi/TM3030 PLUS, Japan). The observation was conducted on the six selected MUT samples according to their highest, average, and lowest permittivity values. The MUTs were layered with 3 nm gold by a vacuum sputter coater (Quorum Technologies Q300TD) to provide a conductive surface on the MUT. The conductive surface can prevent charring and charging, and can enhance the secondary electron signal, producing an excellent SEM image of the fibers. The accelerating voltage was adjusted to 15 kV. The elemental composition present in the fibers was identified through energy-dispersive X-ray (EDX) spectroscopy with a TM3030 PLUS (Hitachi). The elemental composition of the fiber samples was revealed theoretically using backscattered secondary electrons (BSE).

## 3. Results and Discussion

### 3.1 Effect of morphological changes in cellulose fiber and permittivity value

The effect of morphological changes in the cellulose fibers was observed and analyzed in terms of their fiber diameter and distance between fibers on the permittivity value obtained. Figure 1 displays the SEM images for the six selected MUT pineapple leaf fiber samples.

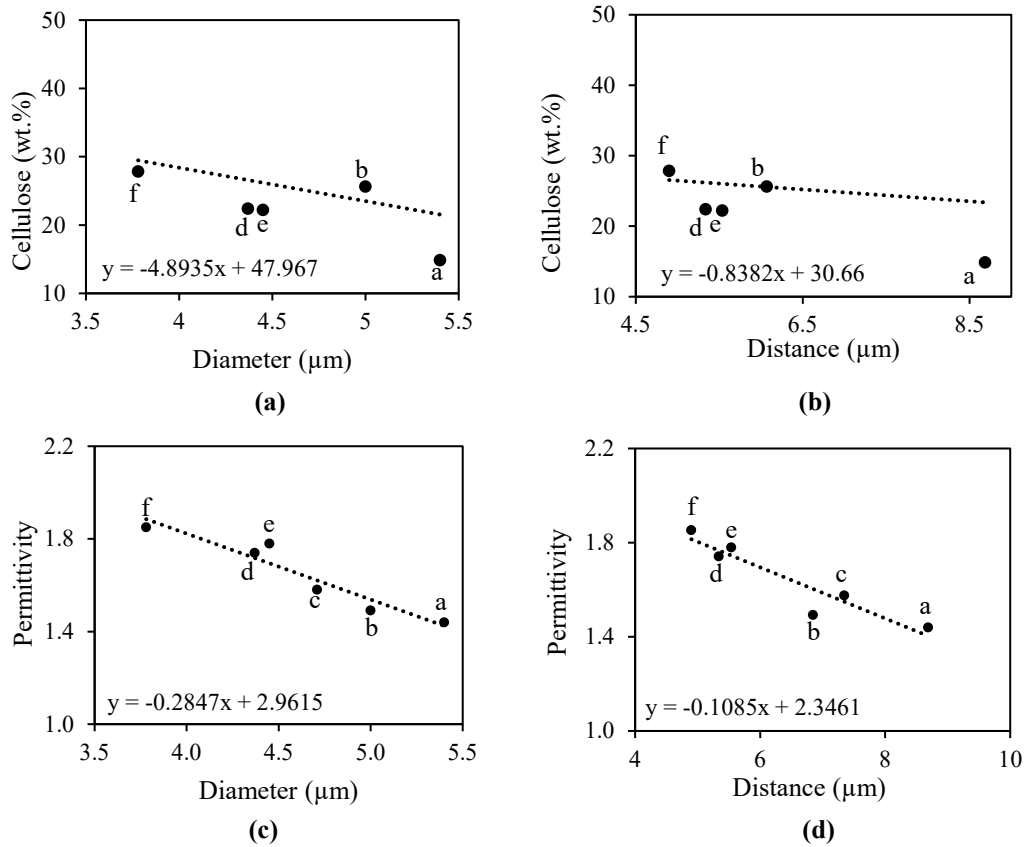


**Figure 1.** SEM image for each pineapple leaf fiber sample

The images show that the permittivity value increases as the cellulose fiber diameter decreases and vice versa. The highest permittivity value of 1.85 was obtained with the smallest cellulose fiber diameter of  $3.78 \pm 0.89 \mu\text{m}$ . In the meantime, the lowest permittivity value of 1.43 was achieved in a sample with a cellulose fiber diameter of  $5.40 \pm 1.37 \mu\text{m}$ . From the results, we found that the resultant small diameter of the cellulose fibers benefited not only their permittivity but also mechanical properties. Both mechanical and dielectric properties (permittivity) depend on the porosity, density, and fiber morphology properties such as fiber diameter [37]. Fibers with smaller diameters possess better mechanical properties [38]. The smaller the fiber diameter, the higher the strength and modulus since finer fibers exhibit superior features due to the higher level of ordering of molecular chains and increased crystallinity [39].

A similar trend was also observed for the distance between cellulose fibers (Figure 2), where the permittivity value decreases as the distance increases. This is due to the increase in porosity with distance. This means that the dielectric properties are highly dependent on the porosity of the fiber [40]. The presence of large air voids between the cellulose fibers resulted in the low permittivity values due to the fiber's high porosity. The loose arrangement between fibers due to the large air voids caused a reduction in the fiber content in the pineapple leaf, thereby reducing the permittivity value. As investigated by Li *et al.* [40], the fiber content played a significant role in determining the permittivity value of kenaf fiber-filled polyurethane foam. The permittivity of the composites increased significantly with increased fiber content. Similar findings were also observed by Jayamani *et al.* [12], in which the dielectric properties of the jute and bamboo fibers significantly increased with increasing fiber content. The increase in permittivity as fiber content increases is due to the increased number of polar hydroxyl groups in the pineapple leaf fiber [41, 42].

Table 1 summarizes the cellulose content (wt.%) and the permittivity values for each sample. In a similar way to the permittivity values, cellulose fiber diameter and the distance between cellulose fibers showed parallel behavior with cellulose content. As shown in Table 1, the diameter was proportionally related to the distance between fibers. The distance between fibers increases as the diameter increases. The increase in fiber diameter and distance between fibers results in a reduced value for cellulose content (Figure 2). This may be the result of the reduced fiber content



**Figure 2.** The value of cellulose content across the (a) diameter, (b) distance between fibers, and the permittivity values of pineapple leaf fiber across (c) diameter, and (d) distance between fibers. Note that in both Figure 2 (a) and (b), some data points were not included in the Figure since the values were exceptionally large.

**Table 1.** Fiber diameter and distance between fibers on cellulose content and permittivity

Sample	Diameter ( $\mu\text{m}$ )	Distance between cellulose fiber ( $\mu\text{m}$ )	Cellulose (wt.%)	Permittivity ( $\epsilon$ )
a	$5.40 \pm 1.37$	$8.69 \pm 1.81$	14.83	1.44
b	$5.00 \pm 1.62$	$6.07 \pm 2.01$	25.62	1.49
c	$4.71 \pm 1.15$	$7.35 \pm 2.81$	39.38	1.58
d	$4.37 \pm 1.02$	$5.34 \pm 1.87$	22.36	1.74
e	$4.45 \pm 1.21$	$5.54 \pm 2.58$	22.20	1.78
f	$3.78 \pm 0.89$	$4.90 \pm 0.45$	27.81	1.85

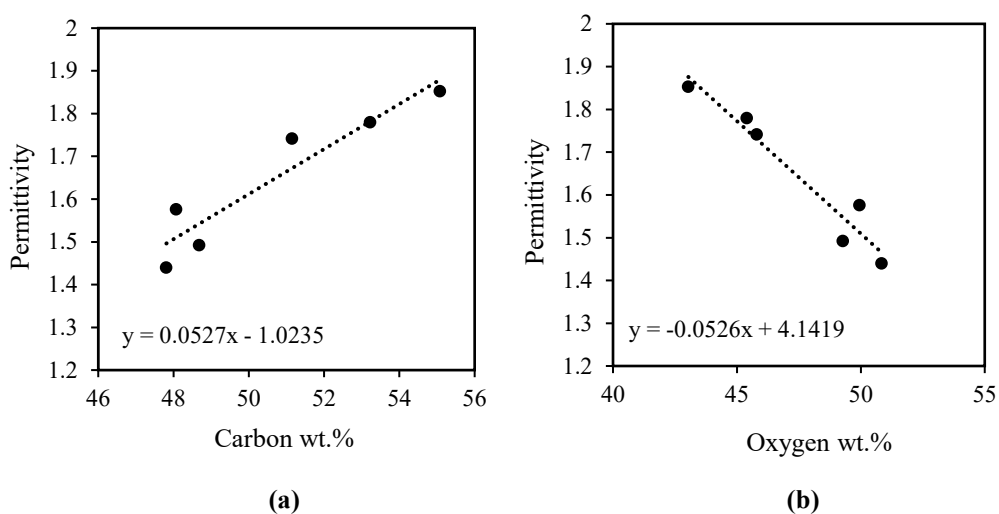
in the leaf as distances between fibers increase in the sample. The fibers were tightly packed together due to their small diameter. As seen from the SEM images (Figure 1) for sample (a), the loose arrangement of fibers with large air voids caused the cellulose content to be the lowest among other samples, at 14.83 wt.%.

### 3.2 Effect of elemental composition on cellulose content and permittivity value

The EDX analysis of the pineapple leaf fiber shows that the pineapple leaf consists of carbon (C), oxygen (O), sodium (Na), magnesium (Mg), and calcium (Ca) elements. An EDX analysis of pineapple leaf fibers treated with 1% NaOH by Gadzama *et al.* [15] revealed that the fibers consist of the same elements as in this current study except for Mg and three more elements of aluminum (Al), chlorine (Cl), and nitrogen (N). Table 2 presents the percentage amount of each chemical element. It was observed that C and O were the two main elemental constituents in the pineapple leaf fiber, which is in agreement with the result obtained by Gadzama *et al.* [15], with 71.04 wt.% C and 24.85 wt.% O. It is also interesting to note that the permittivity value increased as the C percentage increased from 47.81 wt.% to 55.08 wt.%, as portrayed in Figure 3a. Therefore, the high permittivity value exhibited by the pineapple leaf fiber can be associated with its high carbon composition. The decrease in permittivity of the cellulose fiber is due to the loss of the polar functional group. According to Lett and Ruppel [43], the increase in permittivity has been attributed to the presence of polarizable electrons in the fibers.

**Table 2.** Elemental composition of the pineapple leaf fiber

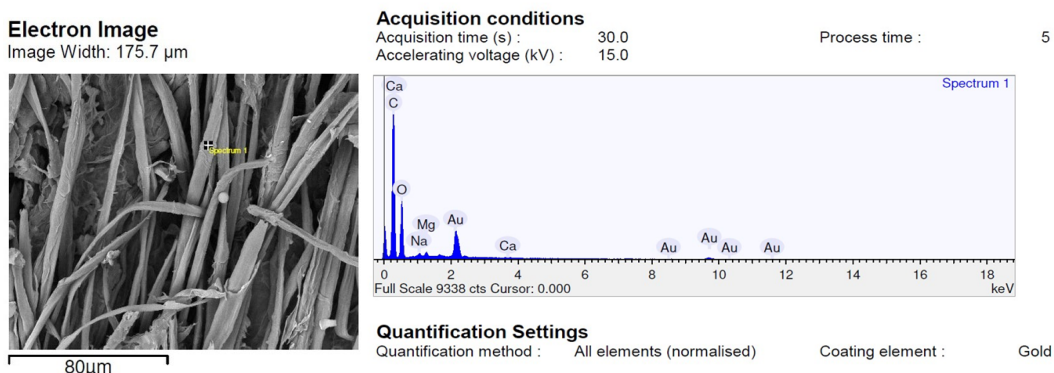
Label	C (wt.%)	O (wt.%)	Na (wt.%)	Mg (wt.%)	Ca (wt.%)	Cellulose (wt.%)	Permittivity ( $\epsilon$ )
a	47.81	50.85	0.17	0.68	0.48	14.83	1.44
b	48.68	49.28	0.9	0.15	0.64	25.62	1.49
c	48.07	49.95	1.01	0.45	0.53	39.38	1.58
d	51.15	45.81	1.23	1.29	0.53	22.36	1.74
e	53.22	45.41	0.61	0.52	0.23	22.20	1.78
f	55.08	43.04	0.46	0.79	0.21	27.81	1.85



**Figure 3.** Effect of (a) carbon and (b) oxygen composition on the permittivity value of the cellulose fibers



The percentage of O behaves differently from C, where the increase in the amount leads to a decreased permittivity value, as seen in Figure 3b. According to Bamzai *et al.* [44], the permittivity value of single titanium dioxide (TiO<sub>2</sub>) crystals is different for those having sufficient oxygen content and those with oxygen deficiencies. The crystal with a reduced oxygen content displays a higher permittivity value. The effect of C and O on cellulose content is generally similar to its effect on the permittivity value. The cellulose content increases as the C composition increases and decreases with an O increment. Figure 4 presents the EDX graph for sample (f), which refers to the highest permittivity result.



**Figure 4.** EDX analysis of the sample with the highest permittivity value (sample f)

The carbon composition was the highest in the sample, at 55.08 wt.%, followed by oxygen at 43.04 wt.%. The composition of Na, Mg, and Ca in all samples ranged from 0.15 to 1.23; hence, the values can be assumed to not contribute much to the cellulose content and permittivity of the fibers. Furthermore, the values of each element in the cellulose fibers vary widely, making it difficult to explain their behavior in relation to cellulose content and permittivity values. Nevertheless, the doping or substitution of Mg<sup>2+</sup> and Ca<sup>2+</sup> ions can reduce the dielectric properties of barium strontium titanate ceramics [45]. The incorporation of Mg<sup>2+</sup> ions in the Ni-Zn ferrite shows improved dielectric properties of the ceramic filler [46]. Depending on the type of material being doped, these ions have different effects on the dielectric properties of one material when substituted with other chemical elements. It is, therefore, important to acknowledge and understand how every single element behaves since their composition can affect the performance of the composites.

## 4. Conclusions

The morphological and elemental composition of the pineapple leaf fibers were analyzed. The effect of the morphological features of the cellulose fibers on their dielectric properties was analyzed, where the finest fiber diameter contributes to a high permittivity value. The loose cellulose fiber arrangement between fibers caused the permittivity value to be reduced. Also, the elemental composition of the fibers significantly affects their permittivity value, with the one having more carbon composition having a higher permittivity value. Therefore, the findings of this study reveal that the morphological features of the cellulose fibers and their corresponding elemental composition are essential to developing materials with the desired properties. The doping of carbon into a material could further improve its permittivity value. The high permittivity value of the



pineapple leaf fibers suggests that the cellulose fiber could be exploited further to be used as electrical and electronic components.

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