

## Research Article

# The Effects of SLS on Structural and Complex Permittivity of SLS-HDPE Composites

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RS-4050 is a rigid epoxy based magnetic castable microwave absorbing material; it has been used in many areas of waveguide application as a microwave waveguide terminations and dummy loads. In recent years, there is a demand for composites material with lower dielectric constant higher loss factor for microwave application. This research, the effect of soda lime silica (SLS) on structural and complex permittivity of soda lime silica-high density polyethylene (SLS-HDPE) composites was conducted in order to explore the possibility of substituting RS-4050 with SLS-HDPE composites as a microwave waveguide terminations and dummy loads. Elemental weight composition of the SLS glass powder and HDPE was identified through scaling of different percentage of SLS and HDPE. X-ray diffraction (XRD) was used to investigate the crystallinity behavior of SLS-HDPE composites. The proposed SLS-HDPE composites material was studied at frequencies 8 to 12 GHz. The study was conducted using waveguide Agilent N5230A PNA technique. The effect of microwave frequency on complex permittivity properties for SLS-HDPE composites of different percentages of SLS and HDPE (10% SLS-90% HDPE, 20% SLS-80% HDPE, 30% SLS-70% HDPE, 40% SLS-60% HDPE, and 50% SLS-50% HDPE) were investigated. Results showed the diffraction patterns reveal good amorphous quality with a genuinely properties structure. The microwave frequency and composites percentages significantly influenced the complex permittivity (real and imaginary) properties of the composites. Moreover, the complex permittivity increased as the percentage of SLS filler increased in the host matrix HDPE as a result of increased in composite density due to less volume being occupied by the filler as the percentage increased. The complex permittivity of the smallest and largest percentages of SLS (10% and 50%) was (2.67-j0.05) and (3.45-j0.35), respectively. The study revealed that the best sample for waveguide application as microwave terminator is 50% SLS as it has the highest dielectric constant, highest loss factor, and highest loss tangent as compared to 10% SLS to 40% SLS. Also 50% SLS has the highest absorption properties as compare to 10% SLS, 20% SLS, 30% SLS, or 40% SLS. The XRD physical structure of the SLS-HDPE composites revealed the absorption characteristics of different percentages of the materials. The SLS-HDPE composites can be applied in the area of waveguide as a microwave waveguide terminations and dummy loads.

## 1. Introduction

RS-4050 is a stiff epoxy built magnetic castable microwave absorbing device; it has been applied in many areas of waveguide application as a microwave waveguide terminations and dummy loads. This material is archetype for construction of low power loads, ring chokes, attenuators,

and other radio frequencies (RF) absorbing components at microwave frequencies. RS-4050 can be useful in standard stock shapes of plates, custom cast configurations, rods and bars, or pourable kits [1]. Unfortunately, RS-4050 contains epoxy which is costly, and it is magnetic based which is not necessary for microwave termination. One particular risk associated with epoxy resins is sensitization [2].

In recent years, there is a demand for composites material with lower dielectric constant higher loss factor for microwave application. Composite material having low dielectric constant high loss factor has the tendency to reflect electromagnetic waves less and absorb more. These characteristics are much needed to minimize the effects of electromagnetic interference (EMI) interference arising from the increasing number of the telecommunication users through mobile telephones, local area networks, wide area networks, and radars systems. Unfortunately composites of low dielectric constant high loss factor are not found naturally; there is need for fabrication of such composites. The common absorbing materials are the mu-metals, that is, an alloy contains copper, iron, nickel, and chromium. Unpolluted iron or specifically referred to as ferrites are frequently used for the fabrication of microwave absorbing composite. However, similar to various metals, ferrites are destructive and corrosive metals, expensive, and heavy, and they are nonbiodegradable. Ferrites have a higher complex permittivity [3]; this means ferrites reflect more; the reflecting characteristic of ferrites is not good for security, as this reflection can be detected by radar or interfere with electronic gadgets. However, soda lime silica glass (SLS) was chosen to replaced ferrites due to its numerous characteristics; SLS has a low dielectric constant and high loss factor, which means when compounding with polymers (HDPE) [4]. SLS-HDPE composites would have the ability to reflect less and absorb more. SLS is biodegradable, noncorrosive, cost-effective, and environmentally friendly.

High density polyethylene (HDPE) and polycaprolactone (PCL) are widely used in biomedicine. The main advantages of these materials are their biocompatibility and slow degradation accompanied by the release of water and carbon dioxide which makes these polymers good ecologically pure packing material. Therefore, the study of the dielectric and electrical properties of these materials, in specific, the absorption of electromagnetic waves of various frequency bands, is of great scientific and practical interest [5]. The microwave absorbers are processed using different polymeric matrices and polymers have been traditionally considered as right carrier matrices for particles. In some cases, polymers also act as an absorbing component of the polymer-based composites. This is because, instead of the polymer serving as a matrix such as epoxy resin, polyurethane, and rubber, it also helps to improve the electromagnetic (EM) wave absorption properties, as in the case of polyaniline. Polymer-based composites combine both the high EM wave loss of particles and easy processability and multifunctionality of polymers. These materials are hoped to act as ideal EM wave absorber with low density, thin thickness, broad absorption band, high EM wave loss, and even other functionalities [6].

Fillers with unique chemical, physical, and mechanical properties that are intermixed with polymers comprise composite materials with great advantage for technological breakthrough. Conventional metal oxides such as barium titanate ( $\text{BaTiO}_3$ ), titania ( $\text{TiO}_2$ ), alumina ( $\text{Al}_2\text{O}_3$ ), and silica ( $\text{SiO}_2$ ) are widely known as effective reinforcement materials that improve the dielectric and mechanical properties of

polymer-silicate composite. In the last decade, semiconducting oxides ( $\text{SiO}$ ,  $\text{CaO}$ , and  $\text{Na}_2\text{O}$ ) have attracted so much interest due to their potential for different electronic and photonic device applications [7]. Recent researches have shown that the polymer-silicate composites exhibit excellent luminescent, optical, dielectric, and biosensitivity properties. Researches have been conducted in the area of electrical properties of polymer-oxides compositions synthesized by in situ polymerization and melt blending [8].

The complex permittivity properties of polymer composite are mainly modified by the conductive fillers [9]. However, the nature or type of fillers determines the permittivity characterization of polymer composites. Typical conductivity of such composites is due to the formation of a continuous network of filler particles throughout the polymer matrix. This electrically conductive composite material is widely used in the areas of electrostatic discharge dissipation, electromagnetic interference shielding, and various other electronic applications [10]. Polymer matrix composites containing conductive fillers have been extensively studied due to their growing demand for advanced technology and electronic systems [11]. The conductive material could be the right choice for use in shields, especially at low frequencies [12]. They are used in the field of electronics as flexibility conductors and absorption material especially with regard to electromagnetic radiation [13]. In conventional conductive composites, carbon black particles at micrometer sizes are used to achieve desired electrical characteristics. Researches have shown that large filler contents lead to a poor composites [14]; the use of glass powder reinforced polymers has led to the production of composites with unique dielectric and mechanical properties. Filler material comes in different forms; these forms could be in metals, semiconducting oxides, dielectric ceramics, and carbon materials [14]. The study of the complex permittivity and electrical properties of the  $\text{NiFe}_2\text{O}_4$ ,  $\text{ZnFe}_2\text{O}_4$ , and  $\text{Zn}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$  nanoparticles was conducted through chemical (pyrophoric reaction) technique [15]. They found that the interfacial polarization in both dielectric and complex impedance studies of those samples can be attributed to predominant interfacial effect due to their nonmetric grain sizes. The dielectric constant decreases with increasing filler size.

## 2. Experimental

The soda lime silica-high density polyethylene (SLS-HDPE) composites were prepared using melt blend techniques. The Brabender blending machine was used to blend and compound the mixture of SLS and HDPE together. The pellets size and shape of the composites were obtained using oven (Shel Lab. 1330 GX Sheldon manufacturing Inc.) and hydraulic press machine (Fred S. Carver part No.:973110A) at 4 tonnes. For the preparation of the SLS-HDPE composites, a total of 25.0 g was prepared for each composition of the composites. The compositions for easy identification are labeled 50% HDPE, 60% HDPE, 70% HDPE, 80% HDPE, 90% HDPE, 100% HDPE, and 100% SLS. The research was limited to ratio of 50% SLS and 50% HDPE because further addition of SLS filler will result in the flexibility and increase the

TABLE I: Composition of raw materials used in composite preparation.

Soda lime silica (SLS)		Polymer (HDPE)		Total
Percentage (%)	Mass (g)	Percentage (%)	Mass (g)	Mass (g)
10.0	2.50	90.0	22.50	25
20.0	5.00	80.0	20.00	25
30.0	7.50	70.0	17.50	25
40.0	10.0	60.0	15.00	25
50.0	12.5	50.0	12.50	25
100.0	25.0	00.0	00.0	25
00.0	00.0	100.0	25.0	25

absorption which can generate more heat in the composites and eventually the composites will breakup. The summary of the masses and percentages for each element is presented in a tabular form in Table 1.

The SLS-HDPE composites were prepared via the melt blend technique using the Brabender poly-drive three-phase motor with a drive of 1.5 kW, 3x230 V, 40 A, and speed range of 0-120 rpm. In this method, the machine was set to 170°C for heating; the rotation of the rotors was set to 50 rpm. After the machine has reached the required temperature (170°C), the HDPE was poured into the vial of the Brabender heating block. After 5 minutes the SLS powder 63µm grain size was introduced into the vial. The mixture is left for another 5 minutes before taken out and fabricated into desired dimension. The composites are then moulded according to desired shape and dimensions. In this research, rectangular shape of 0.22 cm x 0.11 cm and 6mm thick was fabricated using an oven (Shel Lab. 1330 GX Sheldon Manufacturing Inc.) and hydraulic press machine (Fred S. Carver part No.:973110A) at 4 tonnes. The oven was set at temperature of (170°C); the composites were then set into the oven and allowed to melt for 30 minutes and after melting the composites were taken to be pressed in a desired shape using hydraulic press at 4 tonnes.

For characterization (electrical and morphological) of samples used in this research 50% HDPE and 50% SLS, 60% HDPE and 40% SLS, 70% HDPE and 30% SLS, 80% HDPE and 20% SLS, 90% HDPE and 10% SLS, 100% HDPE, and 100% SLS; X-ray diffraction (XRD); and vector network analyser (VNA) were used as the need arises.

X-ray diffraction analysis was used to study crystallinity, structure and phase analysis of the samples. The X-ray data were collected using a fully automated Philips X-ray system (PHILIPS PW3040/60 MPD) with Cu-Kα radiation. The X-ray wavelength was 1.5405 Å, and the diffraction patterns were recorded in the 2 theta range (0-90°) with scanning speed of 2°/min. The X-ray crystallinity ( $X_{x-ray}$ ) was computed by Segal's formula utilizing intensity measurement at 2θ of 22.5° and 16.8° (amorphous background) [17, 18]:

$$X_{x-ray} = \left( \frac{I_{002} - I_{am}}{I_{002}} \right) \times 100\% \quad (1)$$

where  $I_{002}$  denotes the maximum of 002 peaks at about 2θ=22.5° and  $I_{am}$  the minimum intensity corresponding to 2θ=16.8°.

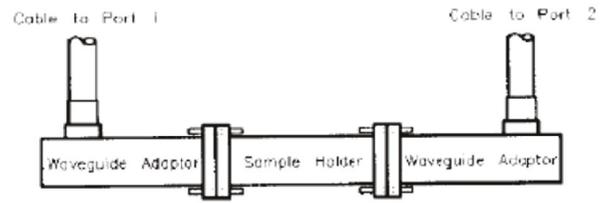


FIGURE 1: Completely filled dielectric waveguide [16].

The waveguide technique was used measured permittivity of SLS-HDPE composites. In this method the total electric propagating through the sample is the same for all the surface area of the sample; hence results obtained can be relied on. The results of dielectric constant, loss factor, and loss tangent obtained using rectangular waveguide are presented in Figures 4, 5, and 6. In guiding electromagnetic waves from point to point, the waveguide was used and the electromagnetic wave was propagated along the waveguide [19]. The completely filled waveguide technique was used to measure complex permittivity of the composites, the calibration method having both the reflection and transmission operation. Figure 1 is a diagrammatic expression showing full two-port set-up with sample inside the wave guide.

### 3. Results and Discussion

The XRD patterns of 100% SLS, 100% HDPE, and SLS-HDPE various percentages are presented in Figures 2 and 3. The pattern of XRD of 100% SLS is shown in Figure 2 and broad peak was observed at 2θ = 16°–38° which indicates that it is amorphous [20], while Figure 3 shows that 100% HDPE has diffraction peaks at 2θ = 21.0° and 24.1°; this is in agreement with result obtained by Patwary and Mittal [21], which can be assigned to pure or 100% HDPE. The presence of two sharp diffraction peaks in pure or 100% HDPE in Figure 3 illustrates the semicrystalline nature of pure HDPE. Increase in SLS filler percentage gradually shows decrease in the intensity of the peaks in a diffraction signals as more SLS fillers are added. The declined in the crystallinity is associated with the increased in the rigidity of the particles structure, which then result in increase in permittivity of filler [22]. This increase is anticipated to increase the electrical properties of composites [23]. Both the physical and chemical properties

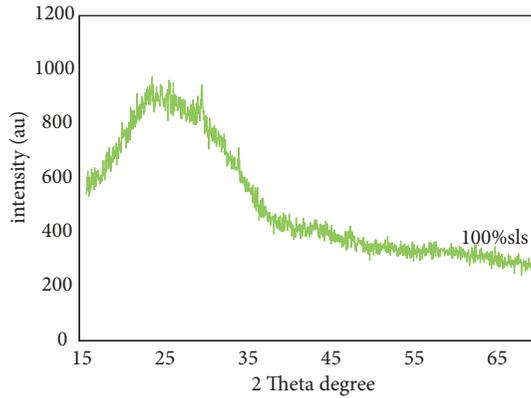


FIGURE 2: XRD spectrum of 100% SLS composites.

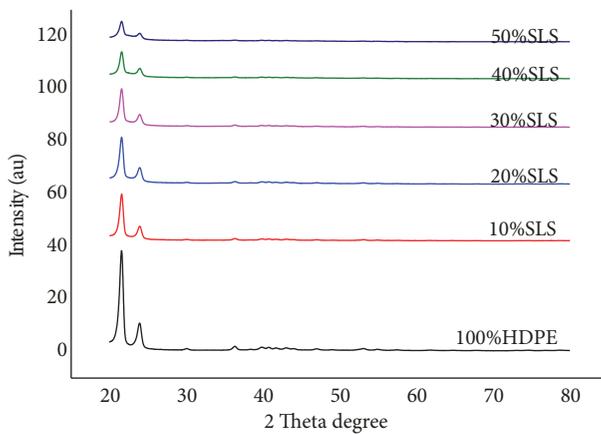


FIGURE 3: XRD spectrum of 100%HDPE and SLS-HDPE composites.

of particle are largely reliant on the void, particle size, ratio of the amorphous, and crystalline phases as cited by Murillo et al. [24] and Richard et al. [25].

The patterns of XRD of all SLS-HDPE composites do not display any secondary peaks signifying that the material samples were pure samples. In the XRD profile, the pattern of diffraction of 100% SLS is compatible with the research conducted by [26]. The crystallinity and sharp peaks of the HDPE were found to be at 10% SLS. Careful observation shows that the diffraction peaks for sample of 20% SLS at  $21.0^\circ$  and  $24.1^\circ$  suggests decrease in peaks intensity. The higher the % of HDPE, the higher the relative peak intensity of diffraction. However, after compounding, the intensity of sharp peaks of the HDPE appears to be reducing as the % of SLS filler increases; broad halo peak also increases. The diffraction patterns reveal good amorphous quality with a genuinely properties and structure [26]. From the results presented, undoubtedly the crystal structural intensity of the materials has declined after an increase in the percentage of SLS fillers.

The permittivity of SLS-HDPE composites was measured using rectangular waveguide technique. The results of dielectric constant, loss factor, and loss tangent obtained using

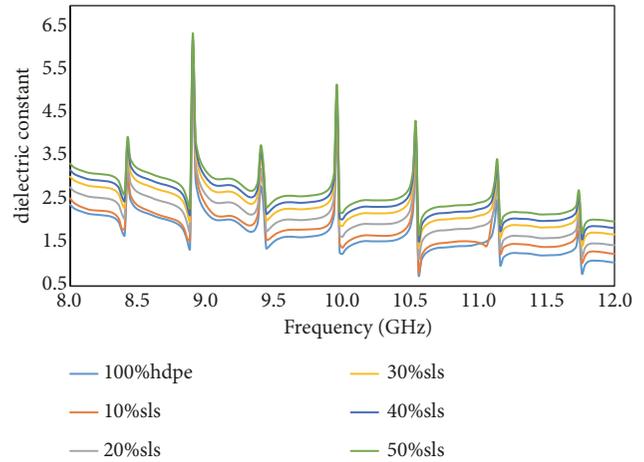


FIGURE 4: Variation in dielectric constant of SLS-HDPE composites.

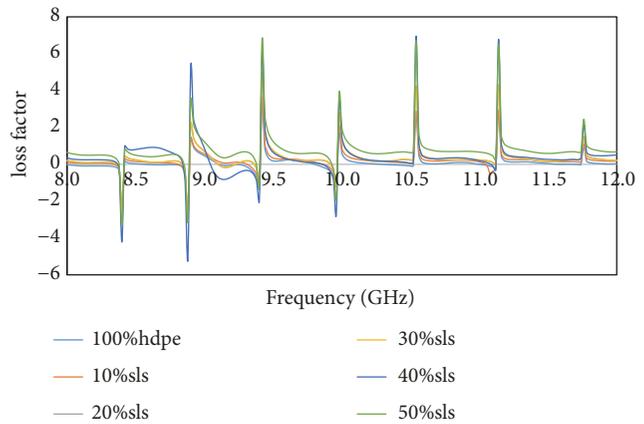


FIGURE 5: Variation in loss factor of SLS-HDPE composites.

TABLE 2: Mean complex permittivity for all composites.

Sample		$\epsilon_r$
%SLS	% HDPE	
00	100	2.48-j0.03
10	90	2.67-j0.05
20	80	2.89-j0.08
30	70	2.97-j0.10
40	60	3.33-j0.15
50	50	3.45-j0.35

rectangular waveguide are presented in Figures 4–6. In this method the total electric propagating through the sample is the same for all the surface area of the sample. Figures 4, 5, and 6 show the variation in real  $\epsilon'$  (dielectric constant), imaginary  $\epsilon''$  parts (loss factor) of the permittivity, and the loss tangent  $\tan \delta$ , respectively, using the X111644A X-Band waveguide (TRL) method of the Agilent 85071 software kit Material Measurement. Table 2 shows the mean complex permittivity for all samples. Observation on Figure 4 shows that the dielectric constant is calculated for the 50% SLS-HDPE composites with a dielectric constant of 3.2928 at

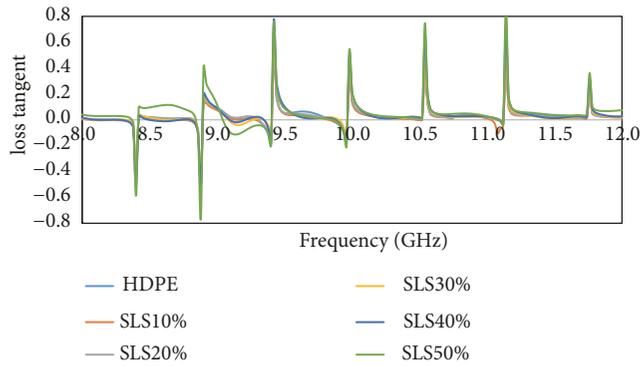


FIGURE 6: Variation in loss tangent of SLS-HDPE composites.

8 GHz which decreased to 3.1178 as frequency reaches 10 GHz. This decrease in dielectric constant is attributed to the dipole relaxation as the composites lags behind the fast change of applied field [27, 28]. That is, a dielectric (or dielectric composite) is an electrical insulator that can be polarized as a result of an applied electric field. When a dielectric is placed in an electric field, there is no flow of electric charges through the material as they do in an electrical conductor but slightly shift from their regular equilibrium points causing dielectric polarization [29]. As a result of dielectric polarization, positive charges are displaced in the direction of the field and negative charges shift in the opposite direction. This creates an internal electric field that reduces the overall field within the dielectric itself. Further observations showed a systematic gradual decrease in the dielectric constant as frequency increases for all the SLS-HDPE composites. The decrease in dielectric constant with frequency can be explain due to the effect of polarization taking place in the composites due to the continuous varying of electric field. It is this component or field of the microwave that is responsible for the interaction of the material with the electromagnetic waves [30]. Figures 4–6 showed some form of oscillation in the dielectric constant, loss factor, and loss tangent of the composites. This is as a result of the composites thickness relatively thin [31]. As it is difficult to fix in thick sample of the composite into the waveguide tube without leaving an air gap between the sample and the waveguide walls, the thickness of the composite reduces to barely 6 mm to be able to be fixed into the waveguide tube without leaving an air gap between the sample and the walls of the waveguide tube. The spikes or half wavelength  $\lambda/2$  effect in the graph is repeated due to multiple reflection effect in the sample, and this multiple reflection occurred as a result of the thickness of the sample which is 6 mm; the sample back and front surface reflect due to thinner size of sample which could not be absorbed [31, 32].

The result reveals that the higher the SLS the higher the  $\epsilon'$ ; also the higher the SLS the higher the  $\epsilon''$ . This is attributed to increase in loss factor as the % HDPE is decreased and the % SLS filler increased. The higher the  $\epsilon''$ , the higher the reflection and lower transmission is observed as the % of SLS filler is increased. Observation on Figure 4

shows that the dielectric constant measured for the 50% SLS-HDPE composites having the dielectric constant of 3.2928 at 8 GHz which decreased to 3.1178 as frequency reaches 10 GHz. This decline in dielectric constant is attributed to the dipole  $\text{Si}^+$  and  $\text{O}^-$  relaxation as the composites lags behind the fast change of applied field in the wave guide [27]. Further observations showed a systematic gradual decline in the dielectric constant as frequency increases for all the SLS-HDPE composites. The decline in dielectric constant as frequency increases can be attributed to the permittivity dispersion which is interfacial polarization, that is, accumulation of charge at the interface between the HDPE and SLS within the composite due to an external field [33].

Declassification of HDPE from a low loss material to a high loss material can be clearly seen from the result of the loss tangent where the loss of the 50% SLS microparticles is 0.02 which is less than 1. Also, there is sequential increase in loss tangent of all the composites as the SLS microparticles content increases. The trend obtained for the loss tangent is in agreement with that of the loss factor for all composites. The result from Table 2 reveals that the higher the %SLS in the host matrix HDPE the higher the  $\epsilon'$ ; also the higher the %SLS in the host matrix HDPE the higher the  $\epsilon''$ . This is attributed to increase in loss factor as the % HDPE is decreased and the % SLS filler increased. Figure 4 shows an increase in the dielectric constant as the percentage of SLS filler increases in HDPE host matrix. The higher the  $\epsilon''$ , the higher the reflection and lower transmission is observed as the % of SLS filler is increased. The increase in the SLS filler percentage brings about increase in thermal conductivity of the composites and that brings about increase in absorption which increases the loss factor of the composite (Figure 5).

The high permittivity observed at the lower frequency range is due the heterogeneous conduction in the multiphase of the composites [28]. For the imaginary part of permittivity (loss factor), the loss factor measured for the 50% SLS-HDPE composites is 0.3589 at 8 GHz which is good for microwave absorption and microwave termination. The gradual increase in loss factor of the composites with increase in frequency is in conformity with measurement carried out by [34]. The increased value of loss factor at higher frequencies suggests that they are lossless materials at microwave frequencies [35]. Higher dielectric constant and loss factor attributed increasing SLS particles in the polymer matrix [35]. The dielectric loss rises due to the localized motion of charge carriers.

#### 4. Conclusion

SLS-HDPE composites were prepared using melt blend technique. Seven different percentages of SLS and HDPE from 10% SLS and 90% HDPE to 50% SLS and 50% HDPE composites, and also pure or 100% SLS and 100 %HDPE, were analyzed based on their dielectric properties. The best sample for waveguide application as microwave waveguide terminations and dummy loads is 50% SLS as it has the highest dielectric constant, highest loss factor, and highest loss tangent as compared to 10% SLS to 40% SLS. Also 50%

SLS has the highest absorption properties as compared to 10% SLS, 20% SLS, 30% SLS, or 40% SLS.

Results from the XRD analysis in Figures 2 and 3 showed that the SLS-HDPE composites are amorphous. The permittivity of the materials was measured using the rectangular waveguide method. The effect of the different percentages of SLS fillers on the dielectric properties of SLS-HDPE composites was analyzed for the whole frequency range between 8 GHz and 12 GHz (X-band). The dielectric constant and loss factor of the SLS-HDPE composites varied from 2.67 to 3.45 and 0.05 to 0.35, respectively, in the X-band frequency. It was found that both dielectric constant and loss factor values of SLS-HDPE composites increased with increasing % of the SLS.

### Data Availability

The XRD and waveguide data used for this research are not yet made public because we are still working on it for further achievement, but it will soon be made public when we finished working on it.

### Conflicts of Interest

The authors declare that they have no conflicts of interest.

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### Supplementary Materials

Graphical abstract. (*Supplementary Materials*)

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