

## Some Effects of Mineral Fillers on the Electrical Characteristics of Pultruded Composites

by

James G. Vaughan, University of Mississippi  
Ellen Lackey, University of Mississippi  
Elliott Hutchcraft, University of Mississippi  
Lorenzo Bennett, University of Mississippi  
Richard K. Gordon, University of Mississippi

### Abstract

Several studies in the research literature discuss the effects of mineral fillers on the mechanical properties of pultruded composites; however, there are fewer studies that discuss the effects on dielectric/electrical properties. This paper will discuss some effects of mineral fillers and other constituent materials on the dielectric/electrical properties of pultruded composites, especially at gigahertz frequencies. Mineral fillers including various kaolin clays, aluminum trihydrates (ATH), and calcium carbonate were pultruded using standard resin system formulations and methods typical of the pultrusion industry. Experiments were arranged so as to determine the effects of the different fillers on the electrical properties using different resins, different glass reinforcements, and different amounts of filler since each of these has been mentioned in the literature as a possible influence on the electrical properties of composites. Results show that there are differences in the electrical response in the pultruded composites examined and that both electrical and mechanical property data should be considered when selecting a pultruded system.

### Introduction

Due to the inherent electrically insulating and non-magnetic properties of the polymer matrix and E-glass commonly used in FRP composites, most people are familiar with the use of FRP composites for electromagnetic (EM) transparent applications. As a dielectric material, FRP composites are generally known to exhibit very low loss over a range of frequencies. Various commercial composite products are available that take advantage of the EM transparent properties of composites [1-5]. Commercial applications for EM transparent composites range from radomes to cell tower enclosures

to RF transparent buildings for electronics testing. New applications for EM transparent materials are emerging in industries that utilize radio frequency identification (RFID) systems. The use of equipment and containers made from materials such as FRP composites that provide necessary strength and are transparent to the radio frequency signals used in these identification systems allow RFID tags to be read more accurately and allow the RFID antennas to be located within the equipment without interference [5]. Some of these composites are reported to offer losses of less than 1 dB over a specified range of frequencies [2]. However, most available data related to the measurement of the EM properties of composites is proprietary and is not a standard material property that is reported for all composites.

Although FRP composites with non-conducting reinforcement are inherently electrically insulating, the EM properties of all FRP composites are not equivalent [6]. Even for E-glass reinforced composites, the EM properties are affected by the selection of the constituent materials and manufacturing process used for the composite. However, little published data related to EM properties of composites fabricated using a range of commonly used constituent material is available in the open literature. This data is also not commonly provided by composite manufacturers.

### Literature Review

A survey of the literature over the past ten years shows a lot of interest in the effects of mineral fillers on the properties and processing of composite materials. Much of this work has been involved with effects on the mechanical performance of these composites materials while other research has dealt with the electrical and physical property changes expected from these fillers.

At one time, mineral fillers were considered as just inexpensive space-filling materials added to resins to reduce overall costs. However, numerous research articles [7 - 13] have shown the importance of these fillers in increasing the hardness, the elastic modulus, the tensile and shear strength while many times decreasing the flexural strength, and having a very large effect on the flexural fatigue properties. Fillers have also been shown to improve thermal conductivity; improve fire, smoke, wear and chemical resistance; decrease thermal expansion; and improve electrical insulation [13]. The effect of mineral fillers on  $T_g$  was normally minimal while the density of the composite changed according to the specific density of the filler added; the higher the density of the filler, the higher the overall density of the composite. The color of the composite was also shown to be directly related to the whiteness of the mineral filler added [7].

Much of the literature associated with electrical properties of filled composites is related to the effects of conductive fillers to produce electrically conductive composites. Most of this research is based on conductive metal fillers, carbon black or carbon nanotubes, not mineral fillers, and is centered on the concentration of filler

required for an appropriate conductivity based on percolation concepts. For example, nanosize (80 – 200Å) copper sulfide (CuS) particles added to a PVA matrix in the concentration range from 16 to 22% were sufficient to reach the percolation threshold resulting in a  $10^7$  conductivity change [14]. Irregular filler shapes, especially elongated shapes, have been shown to reduce the percolation threshold [15]. The electrical conductivity has also been shown to be strongly dependent on the homogeneity of the fillers within the composite [16].

FRP composites are often selected for applications due to their dielectric or electrical insulating properties. It was shown as early as 1987 that extreme care was needed in selecting E-glass reinforcements for epoxy pultrusions in high voltage electrical insulation applications. The sizing applied to the glass filaments to assist in handling and impregnation was shown to act as an ionic pathway for conduction. If the sizing and the resin do not work together to form a good adhesive bond, then small voids along the interface caused by the poor wet-out were shown to aggravate the electrical problems due to easy diffusion of moisture. The moisture could be due to the curing reaction of the epoxy, due to water wicking along the glass filaments, or water entering at cracks in the glass/resin matrix [17]. One problem with cracking in E-glass pultruded composites has been shown to be due to stress corrosion cracking of the composite structure associated with sodium ion exchange in the E-glass [18]. Under high voltage conditions where electrical discharge in the air generates nitric oxides and nitric acid, the nitric acid assists in stress corrosion cracking of the E-glass surface leading to premature brittle failure. This fact, along with other problems, has led the industry to boron-free glass as a replacement to E-glass [18-19]. Other research has shown that the dielectric breakdown dependence of epoxy composites is associated with the degree of conversion of the epoxy and the type of the applied field. Inorganic fillers tend to improve the breakdown strength of the composite in divergent fields through a mechanism similar in nature to the so-called barrier effect, whereas the same fillers were responsible for a local intensification of the electric field and thus failure of the composite in a quasi-homogeneous field [20]. Alumina fillers have been shown to have better degradation properties to ac electric fields than silica fillers [21-22]. The better performance of alumina fillers was attributed to the higher thermal conductivity as compared to silica.

Several studies have highlighted the degrading effects of moisture on the dielectric properties as well as breakdown behavior. Most of this work has been conducted at frequencies below 1MHz. It has been stated that the surface OH groups on silica ( $\text{SiO}_2$ ) fillers have a permanent dipole and can act to increase the matrix dielectric constant and moisture absorption characteristics [23]. These effects have been shown to be deleterious to the performance of electronic devices encapsulated by epoxies with silica fillers. However, while studying the

effects of  $\text{SiO}_2$  additions to cyanate ester resins for use as encapsulates for electronic devices, it was shown that untreated silica resulted in only a minimal increase in dielectric constant [23]. Other studies using polyesters filled with kaolin clays showed higher permittivities ( $\epsilon'$ ) and slightly higher loss ( $\epsilon''$ ) than calcium carbonate or quartz [13]. Higher loss implies that the composites absorb more of the signal and are less transparent at the specific frequencies considered. One study on the flame resistance of EPDM composites determined that 180 phr of ATH caused an approximate 50% increase in the dielectric constant (permittivity) with an associated two order of magnitude increase in the volume electrical conductivity [24]. However, other studies using ATH in silicone based polymeric composites have shown that additions of up to 100 phr ATH actually decreased the permittivity of the composite as compared to the unfilled system; further additions of ATH above 100 phr resulted in a gain in the permittivity back to the unfilled state while an increase in ATH to 250 phr increased the permittivity well above the unfilled condition [25-26]. A similar decrease, then increase, in permittivity with moderate additions of silica was noted by Mansour [27], although this study did not show quite the same effect for ATH.

In summary, published studies on mineral fillers have shown that the following effects should be considered when selecting a filler for mechanical or electrical applications: type of filler, the method of processing the filler from the base mineral and any special surface treatments given to the filler, the particle size and particle size distribution, the extent of agglomeration of the filler particles within a given resin system, the particle geometry and aspect ratio, and the amount of moisture absorbed onto the filler or in the composite. The source of the particular mineral filler can also be significant, as it has been shown that impurities and porosity of mineral fillers affect properties. The resin system and the type of glass reinforcement have also been shown to be a concern. While the research literature suggests that these factors may influence the electrical properties of composites, no systematic study has been conducted to determine the separate effects of mineral filler type and loading level, resin system, and glass type on pultruded composite systems meant to be transparent in high frequency ranges commonly used today for many electromagnetic applications. The intent of the present research was to examine these separate effects.

## Experimental Method

**Pultrusion Processing:** Pultruded composites were fabricated using each of the 12 experiments listed in Table 1. For each experiment, a 1" x 1/8" rectangular cross-section part was fabricated. All of the pultrusion processing experiments were conducted at the University of Mississippi using commercial pultrusion equipment and process conditions typical of the industry.

The first 12 experiments listed in Table 1 represent four different combinations of resin systems and E-glasses. To allow determination of the effects of the different types of filler and the loading levels of the filler, the first six experiments used the same resin system and the same glass (System A); thus, the only difference was the type and amount of the mineral filler. Experiments 7 and 8 were conducted to determine the effect of the resin system; these experiments used the same filler and glass with two different types of resins (Systems B and C). Experiments eight and nine allow for a direct comparison of the effects of glass type in that the same resin system and filler type/loading were used with two different types of E-glass (Systems C and D). Experiments 10 and 11 were conducted using ATH to examine the effects of this type of common mineral filler which is known to behave differently than the kaolin clays examined. Experiment number 12 contained no filler in the polyester resin; this experiment served as a baseline for comparison with all other filler containing experiments. After pultruding the product for this study, samples from two experiments (Exp 1 and 4) were taken and subjected to an ASTM D570 30 minute water boil. This was done to determine the extent of the dielectric property changes due to the presence of moisture in these samples. The moisture laden samples from experiments 1 and 4 are identified in Table 1 as experiments 13 and 14, respectively. All samples other than experiments 13 and 14 were measured under room temperature and humidity and received no specific environmental conditioning.

**Permittivity Measurement:** Permittivity measurements were made from 200 MHz – 20 GHz using a dielectric probe that allows for quick measurement of permittivity of the composites. For the measurement setup, vector network analyzers (VNAs) available in the Electrical Engineering (EE) Department at UM were utilized. A more complete description of this measurement setup is available in reference [28]; however, it should be pointed out that the probe only measures a very small area of the specimen and measures through the thickness of the sample (perpendicular to the fiber orientation). For these experiments, the probe was positioned in the center of the width of the rectangular sample and, thus, did not measure the edges of the sample where the most moisture might be expected to occur. The dielectric probe and software allow both the real and imaginary parts of the dielectric constant to be obtained. The real part of the dielectric constant is known as the permittivity, while the conductivity is related to the imaginary part of the dielectric constant; both are known to be a function of frequency. Measurements were made on three separate samples from each pultrusion run. The data were checked for consistency and the median value taken for comparison between experiments.

**Transmission Measurement:** The transmission properties of the pultruded composites were measured using a VNA and waveguide system from 8 - 13 GHz; this frequency range includes the X-band since this is the

primary frequency band for many EM applications. A more complete description of the test setup is given in reference [28].

The S-parameter,  $S_{21}$ , which is directly related to transmission, is typically reported using the decibel (dB) scale. Since perfect transmission would result in  $|S_{21}|=1$ , the highest value on the dB log scale should be 0 dB. This also means that there is no reflected signal at the composite/air interface. If the entire wave is either reflected at the interface and/or absorbed in the composite material, the magnitude of  $S_{21}$  should be  $-\infty$  dB. This measurement is a good representation of real world behavior in that the field that is transmitted to the other side of a composite structure may be initially reflected at the composite interface or absorbed by the composite structure itself; so the absorption by the composite material itself is not the only consideration when utilizing composites for these applications. As depicted in Figure 1, the incident signal can be reflected, absorbed, or transmitted through the material in the waveguide. The  $|S_{21}|$  parameter reported in this research is based on the electric field received at the end of the waveguide. While this technique can be used to compare the transmitted electric field through the waveguide containing each material being examined, this data alone does not quantify the magnitude of signal actually absorbed by the material until the measured parameters are analyzed to extract the information to separate out the absorbed signal in the composite. Based on the data obtained in this study, further research is being conducted to quantify the magnitude of the loss attributable to each material and to obtain its permittivity and loss tangent. The transmission properties are measured along the length of the test sample while the composite is enclosed in the waveguide. For these tests, the samples were 6.137 inches  $\pm$  0.003 inches long. It should be noted that the 6.137" length of the composite sample within the waveguide is a much larger thickness than would be used to fabricate a real-world structure, but the use of this large sample size was selected to both minimize measurement uncertainties and to reduce the effects of variation in the composites for these measurements.

## Results

Results of the measurements of all experiments shown in Table 1 indicate that these samples could all be generally categorized as low loss materials over the frequency ranges examined. Figure 2 shows the contrast of the loss of samples from experiments 1 and 12 compared to a composite that incorporates carbon black and is designed to absorb EM radiation. However, within the general category of these samples being low loss materials, some differences were seen as a result of the variation of mineral fillers and moisture levels.

**Effect of Type and Amount of Filler:** To determine the effect of different kaolin clay fillers on the high frequency dielectric properties of the composites, data from experiments 1, 2, and 3 were compared to the no-

filler case, experiment 12. Results of the permittivity measurements and the transmission measurements of the samples are shown in Figures 3 and 4, respectively. As can be seen in Figure 3, all three kaolin clay filled samples show a slight increase in permittivity compared to the no-filler composite, with the two smaller particle size fillers, ASP 600 and ASP 900P being slightly higher than ASP 400P sample. In transmission, the no-filler sample is the most transmissive followed by samples with the small particle size kaolin clays, with the ASP 400P sample being the least transparent. However, these differences are very slight (less than 1 dB). Both of these results compare well with the previous literature results discussed earlier.

Figures 5 and 6 compare the permittivity and transmission measurements of samples containing ASP 400P to  $\text{CaCO}_3$ , both at 20 phr in the same resin and glass composite. As can be seen, both of these filler samples have a slightly higher permittivity than the no-filler sample, but there is no significant difference in permittivity between the two sets of samples. Likewise, the transmission measurements for the ASP 400P and  $\text{CaCO}_3$  samples show no measurable difference between these two samples.

Figures 7 and 8 show the difference that various amounts of filler in the composites exhibit. In Figure 7, a comparison of the permittivity of samples with ASP 400P at 10, 20, and 50 phr is shown compared to the no-filler sample. As expected, the 50 phr ASP 400P sample shows the highest permittivity, although the transmission loss shown in Figure 8 does not show a major difference between the three filler loadings.

Figures 9 and 10 show an interesting effect associated with the two ATH's examined in this study. As discussed in the literature review section [25-27], both ATH and silica have been shown to decrease the permittivity and increase the transmission of low frequency signals when the proper amount of the filler was included in the composite. The present measurements show this same trend for the high frequencies measured in this study. The permittivity decrease seen for the ATH filler samples, compared to the no-filler sample, shown in Figure 9 is the only decrease measured for any experimental condition. However, the decrease in permittivity and the increase in transmission is better for the samples with the low 5 phr amount of ATH as compared to the more typical 150 phr amount. Somewhere between 5 phr and 150 phr, the composite is expected to have a maximum decrease in permittivity and the best transmission value.

**Effect of Resin:** To examine the influence of the resin system, dielectric measurements were made on pultruded product from experiments 7 and 8. These experiments used the same filler and amount and the same type of E-glass; only the resin was different. As seen in Figures 11 and 12, there is no measurable difference in either the permittivity data or the transmission data. At least for these two specific resin systems, one an epoxy

and the other a polyester, there seems to be no influence of the resin on the high frequency dielectric properties.

**Effect of Glass:** To examine the influence of the type of E-glass (along with their associated sizing) on the dielectric properties, two different E-glasses were used with the same filler and resin system. Figures 13 and 14 show that for the E-glasses examined, there was no significant measurable difference in the dielectric properties. However, this result would not necessarily hold true for other formulations of glass.

**Effect of Moisture:** As stated in the literature review, moisture in the composite has been shown to have significant influence on both the dielectric breakdown properties as well as the permittivity and transmission properties. To examine the effect of moisture in this study, pultruded product from experiments 1 and 4 (ASP 400P and  $\text{CaCO}_3$  fillers, respectively) were subjected to a 30 minute water boil procedure per ASTM D570. During the water boil, the samples gained approximately 0.08% weight. As expected, the measured high frequency dielectric properties of these moisture laden samples showed a significant effect. Figures 15 and 16 show that some of the largest changes measured in this study are related to the effects of moisture. Especially affected was the transmission behavior as shown in Figure 16. The loss in transmission due to the presence of a small, ~ 0.08 wt%, amount of water is larger than any effect seen due to filler type or amount, or resin or glass. Therefore, environmental exposure of composites used for EM transparent applications should be considered.

## Conclusions

While all of the composites examined in this study can be considered to be low loss materials, this study has shown that the type and amount of mineral filler has an effect on the high frequency dielectric properties of an epoxy and a polyester glass reinforced composite. The changes in both the permittivity and the transmission of the high frequency signal are not major compared with changes expected with more conducting fillers, but they are measurable. The particular alumina trihydrate (ATH) examined in this study shows an unusual effect in that it actually lowers the permittivity and the loss in transmission, a fact not expected from a typical rule of mixtures approach.

Absorbed moisture appears to be more significant in changing the dielectric properties than the changes in the fillers, resins, or E-glasses examined in this study. The moisture present in the samples due to a 30 minute water boil resulted in a more lossy transmission than any of the other conditions examined. The dipole moment due to the polar nature of water present in these samples is having a significant effect.

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## Biographies

James Vaughan: Dr. Vaughan is a Barnard Professor of Mechanical Engineering at The University of Mississippi (UM) and is the director of the Composite Materials Research Group at the University.

Ellen Lackey: Dr. Lackey, CCT-I, is an associate professor of mechanical engineering at UM. She works in the area of composites processing and characterization.

Elliott Hutchcraft: Dr. Hutchcraft is an assistant professor of electrical engineering at UM. His research area is EM measurements and modeling.

Lorenzo Bennett: Mr. Bennett is an M. S. student in electrical engineering at UM. His research area is EM measurements and modeling.

Richard K. Gordon: Dr. Gordon is an associate professor of electrical engineering at UM. His research area is in numerical methods in electromagnetics.

**Table 1. Fillers, resins, and glasses examined**

<b>Exp #</b>	<b>Filler</b>	<b>phr</b>	<b>Resin System</b>	<b>E-Glass</b>	<b>System</b>
1	ASP 400P	20	AOC P920 Polyester	OC 366	A
2	ASP 600	20	AOC P920 Polyester	OC 366	A
3	ASP 900P	20	AOC P920 Polyester	OC 366	A
4	CaCO <sub>3</sub>	20	AOC P920 Polyester	OC 366	A
5	ASP 400P	50	AOC P920 Polyester	OC 366	A
6	ASP 400P	10	AOC P920 Polyester	OC 366	A
7	ASP 400P	20	EPON 9500 Epoxy	PPG 2001	B
8	ASP 400P	20	AOC 706 Polyester	PPG 2001	C
9	ASP 400P	20	AOC 706 Polyester	OC 366	D
10	ATH	5	EPON 9500 Epoxy	PPG 2001	B
11	ATH – SB432	150	AOC 706 Polyester	OC 366	D
12	No filler	0	AOC 706 Polyester	OC 366	D
13	ASP 400P w/ H <sub>2</sub> O	20	AOC P920 Polyester	OC 366	A
14	CaCO <sub>3</sub> w/ H <sub>2</sub> O	20	AOC P920 Polyester	OC 366	A

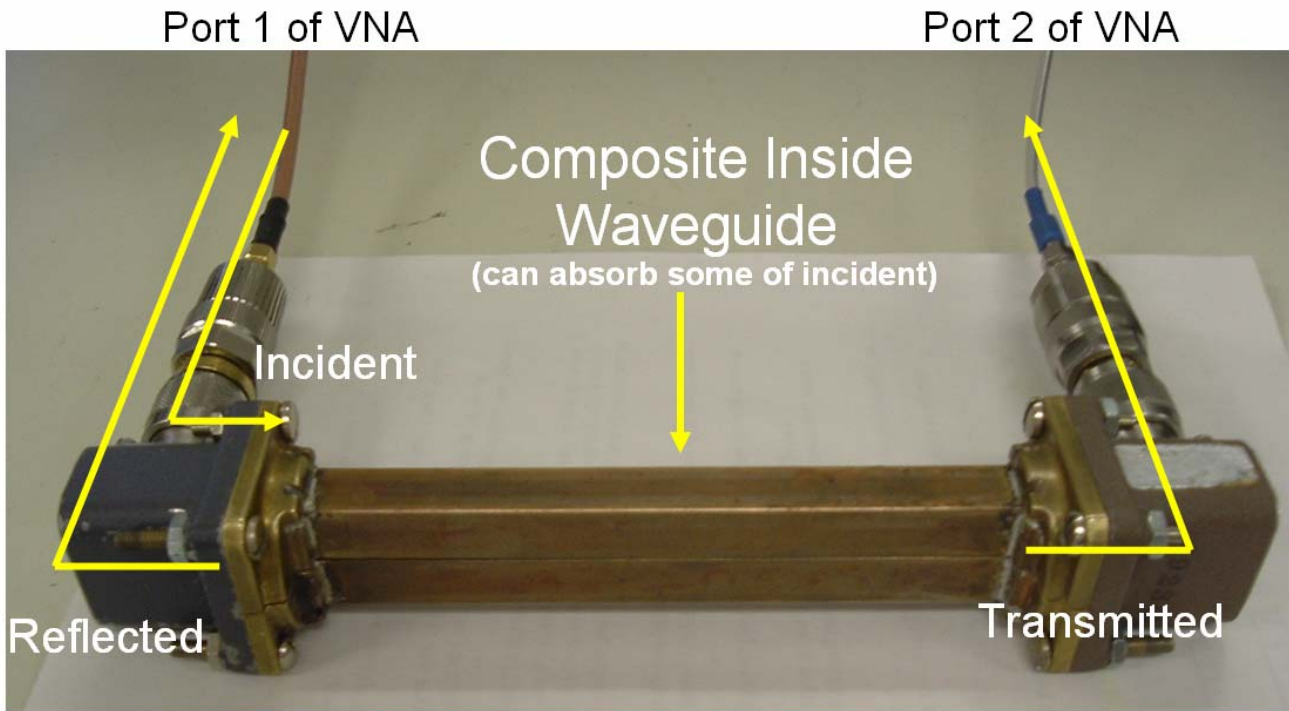


Figure 1. Photograph of the waveguide used to measure the transmission behavior of the composite samples.

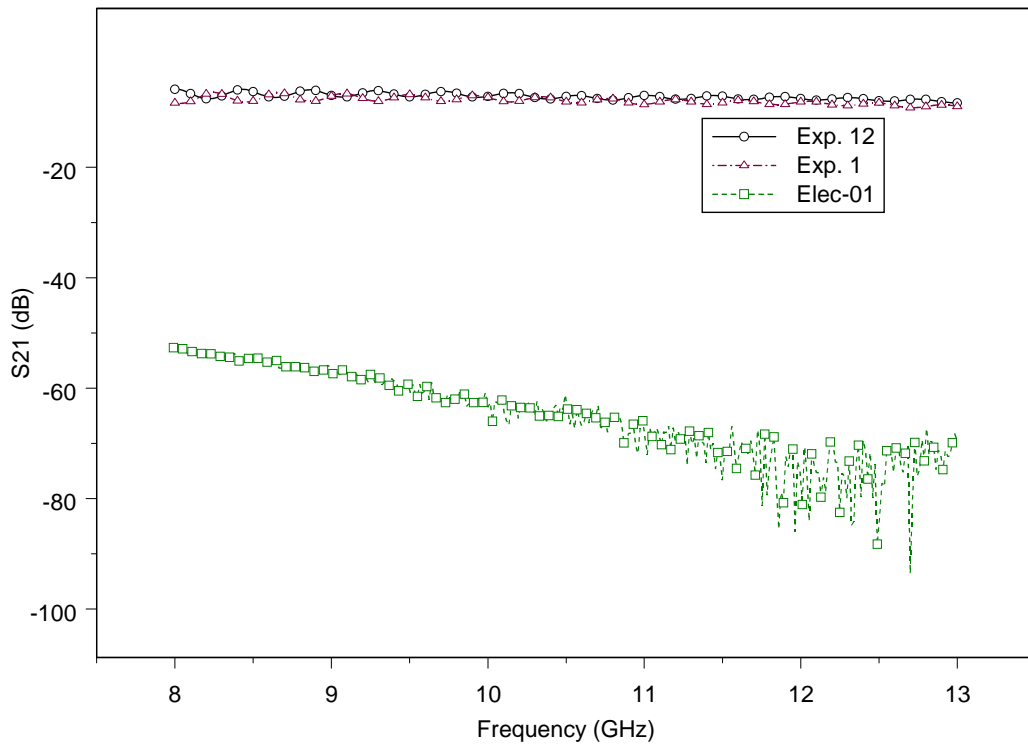


Figure 2. Plot of the transmission of composites in this study as compared to a highly absorbing conductive composite incorporating carbon black (Elec-01).

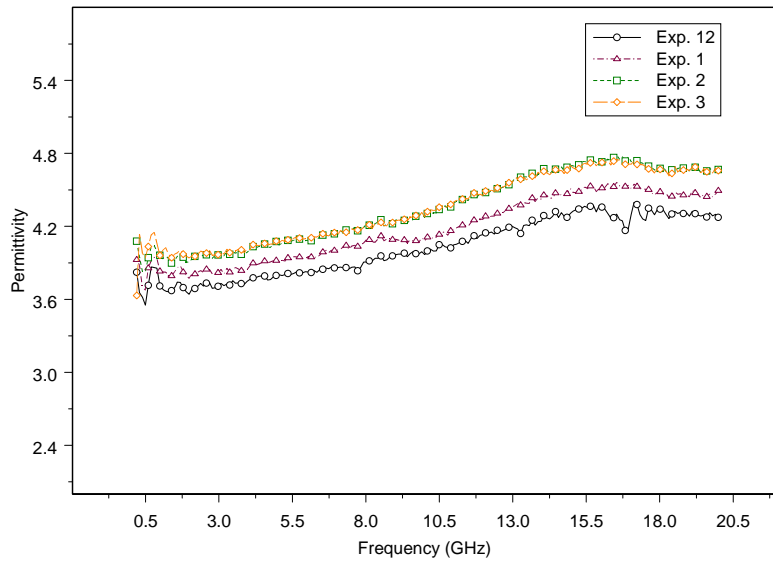


Figure 3. Plot of permittivity versus frequency for three different kaolin clay fillers.

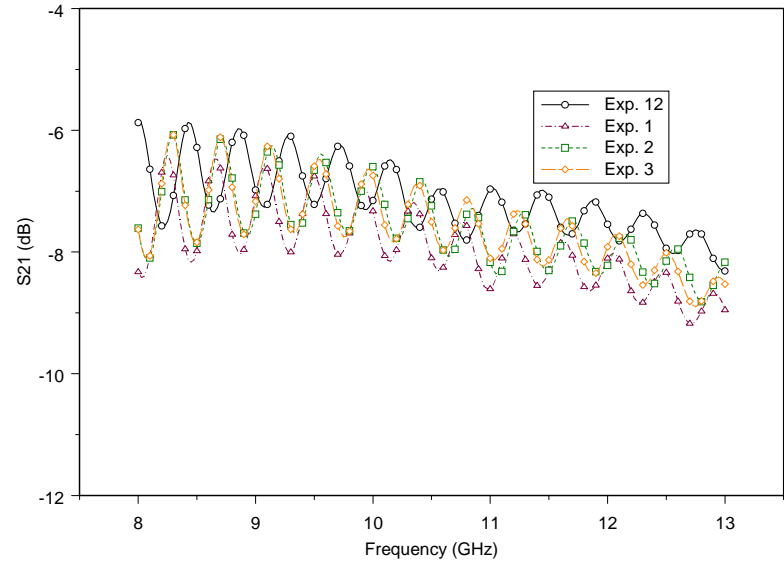


Figure 4. Plot of transmission measurements versus frequency for three different kaolin clay fillers.

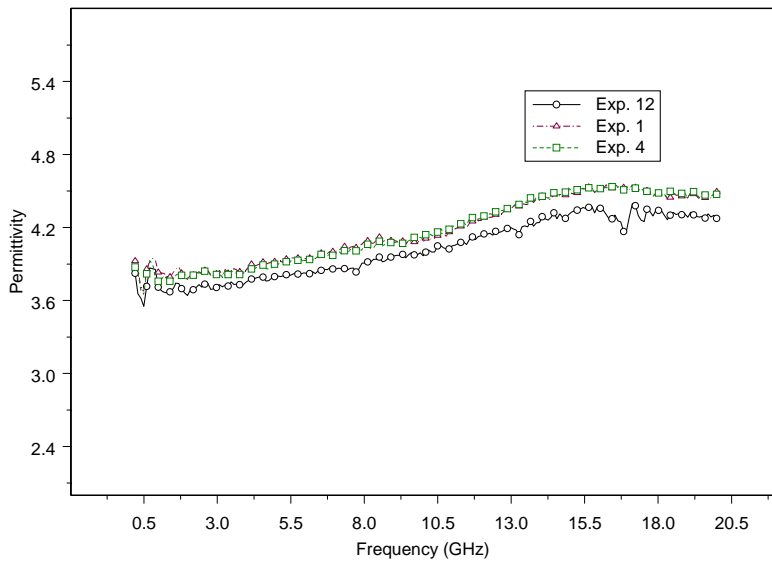


Figure 5. Comparison of permittivity of ASP 400P to  $\text{CaCO}_3$ .

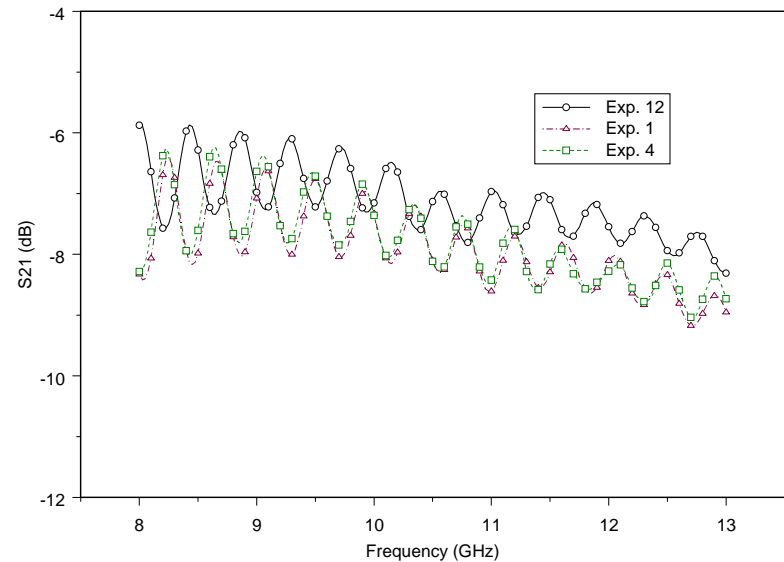


Figure 6. Comparison of transmission measurements of ASP 400P to  $\text{CaCO}_3$ .

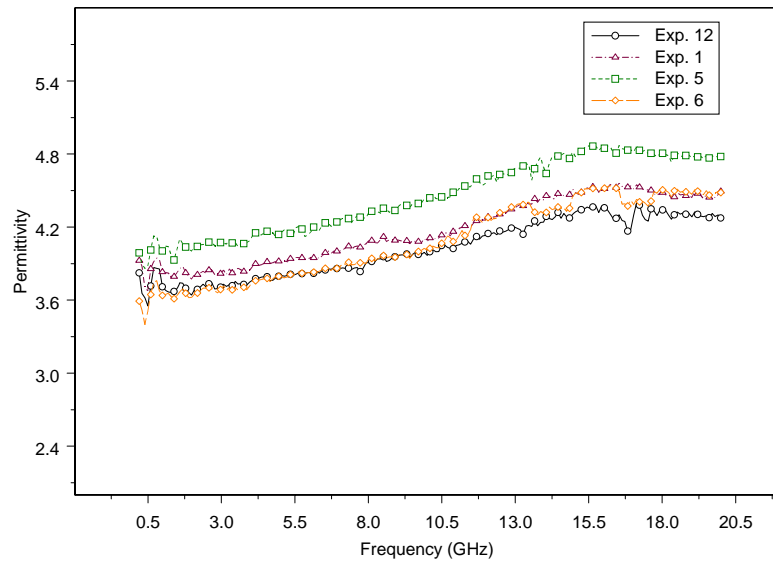


Figure 7. Permittivity comparison for various amounts (phr) of ASP 400P compared to no-filler.

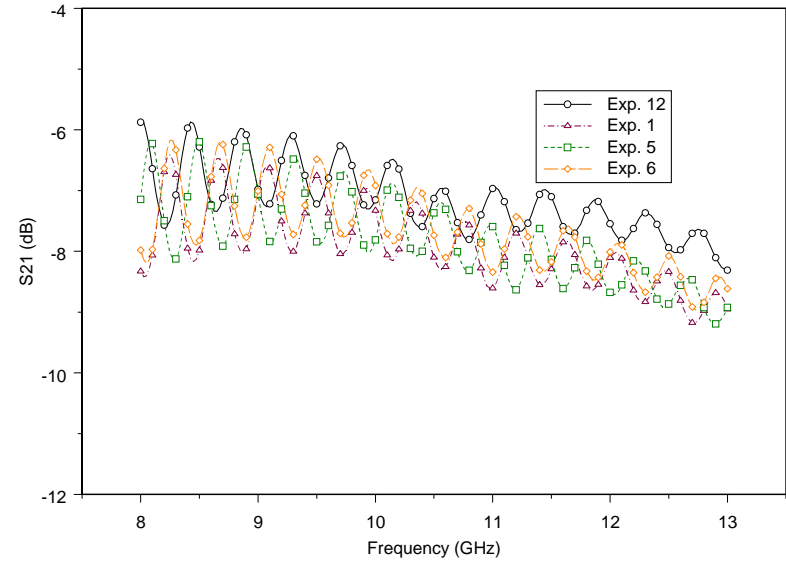


Figure 8. Transmission comparison for various amounts (phr) of ASP 400P compared to no-filler.

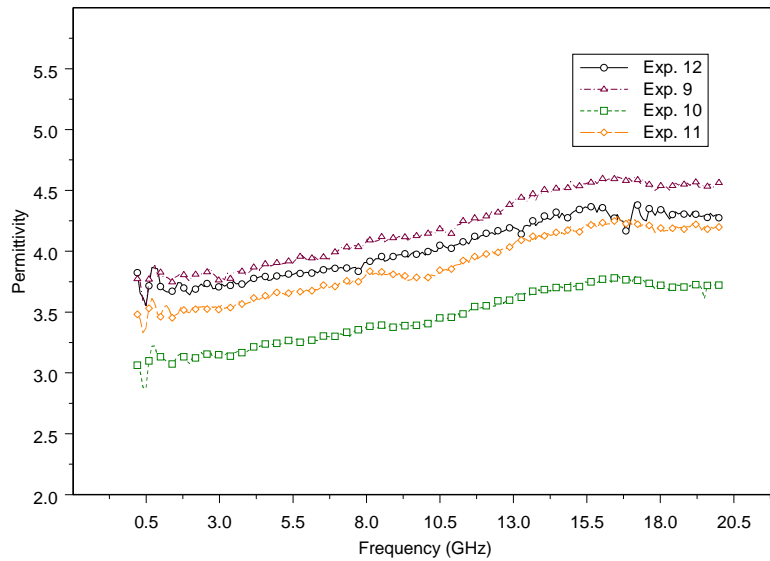


Figure 9. Permittivity of the ATH filled composites compared to no-filler and a ASP 400P filled composite.

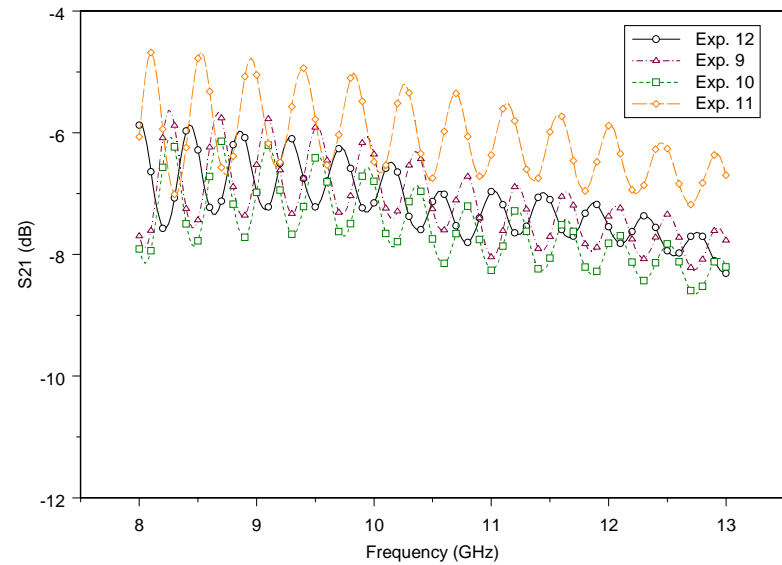


Figure 10. Transmission measurements for ATH filler samples.

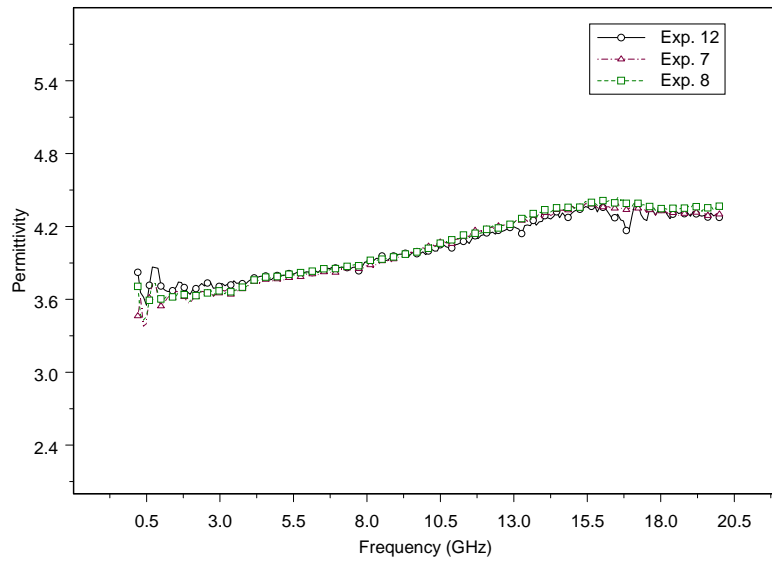


Figure 11. Permittivity comparison of two different resins, an epoxy versus a polyester.

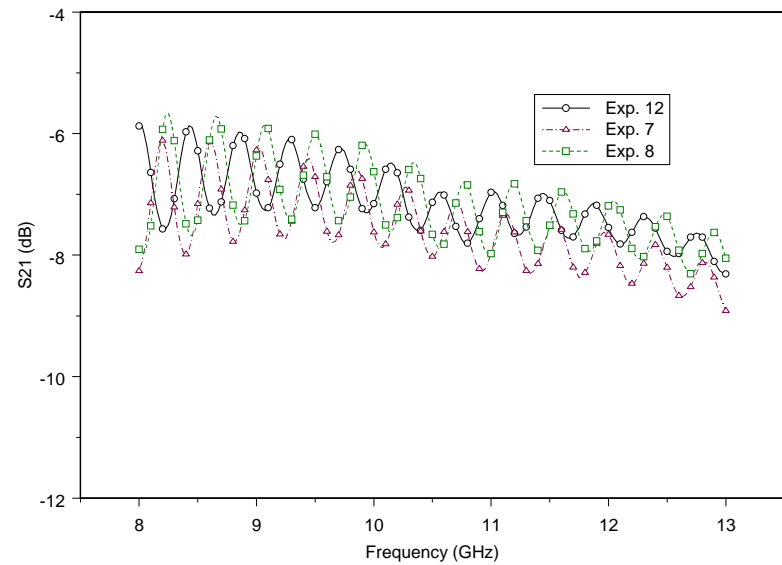


Figure 12. Transmission comparison of two different resins, an epoxy and a polyester.

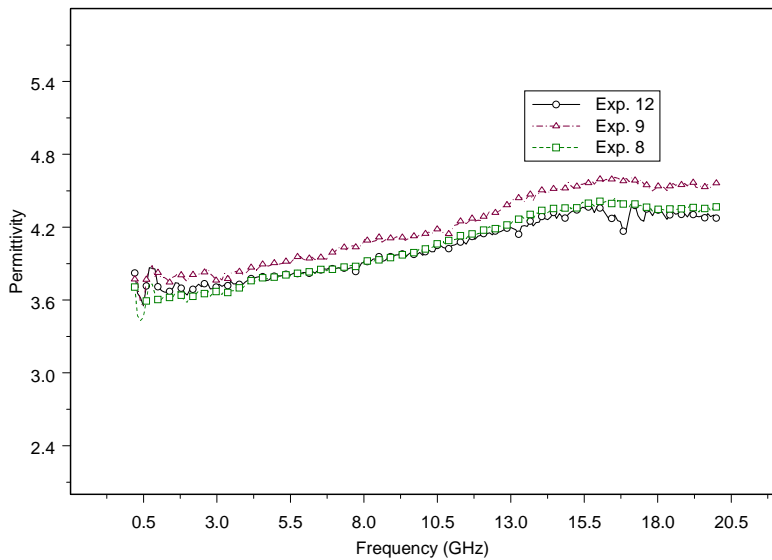


Figure 13. Permittivity comparison between two different glasses.

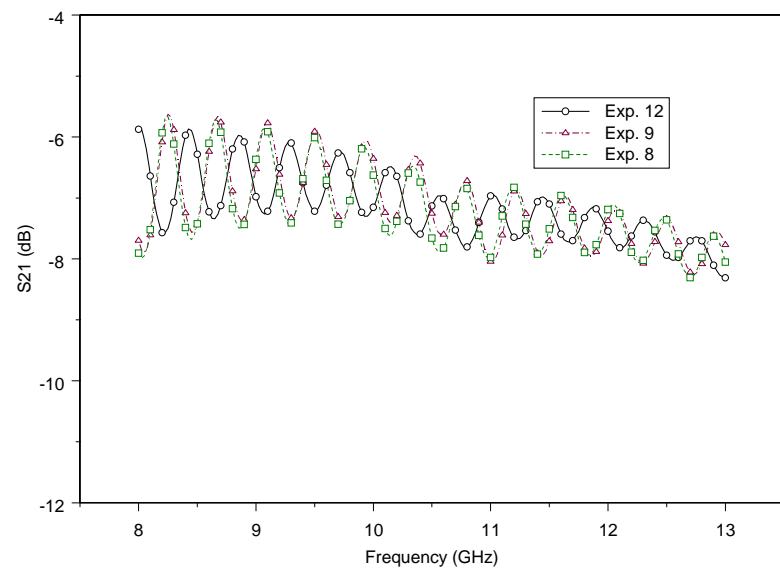


Figure 14. Transmission comparison between two different glasses.

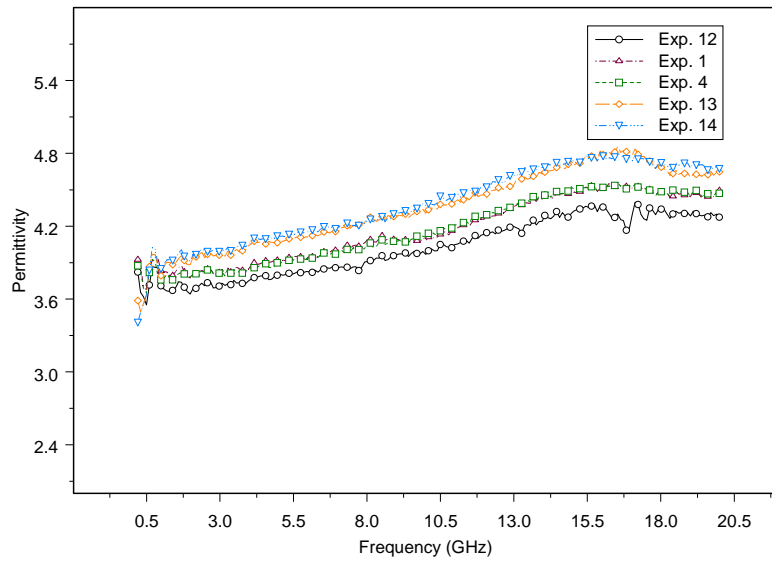


Figure 15. Permittivity versus frequency showing the effect of moisture in the composite.

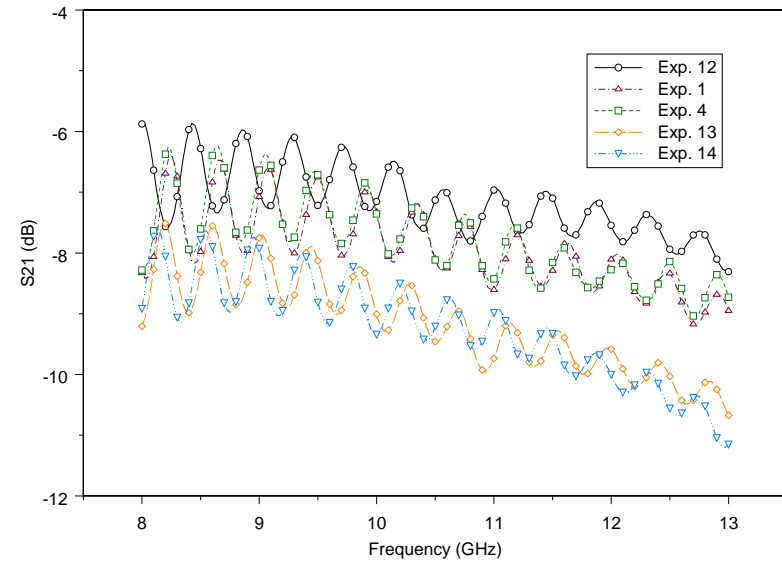


Figure 16. Effect of moisture on the transmission behavior of the composite.