POLYMER COMPOSITE MATERIALS

Comparative Analysis of Transmission and Attenuation Coefficients of Silane-Treated Sodium Lime Glass/Polycaprolactone Composites Fabricated by the Melt Blend Technique

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Abstract

The growing use of electronic and wireless devices has increased the need for lightweight, low-cost materials that attenuate microwave radiation. Conventional absorbers based on ferrite and carbon are expensive, environmentally hazardous and non-recyclable. This study investigates waste sodium lime glass (NLG) cullet as a microwave-attenuating filler in a biodegradable polycaprolactone (PCL) matrix, with particular attention to the effect of silane surface treatment. The glass was crushed, ball-milled to a micrometre-sized powder, and functionalised with 3-aminopropyltriethoxysilane (APTES) to improve its compatibility with the hydrophobic polymer. The treated particles were incorporated into molten PCL by melt blending at filler loadings of 12.5, 22.5, 37.5, 50.0 and 62.5 wt%, and the blends were cast in a stainless-steel mould. The complex permittivity of the composites was measured over the X-band (8–12 GHz) using an open-ended coaxial probe connected to a vector network analyser. The transmission coefficient (S21) was measured in a rectangular waveguide, and the attenuation was calculated from it. The finite element method in COMSOL Multiphysics was used to determine the transmission and attenuation coefficients numerically. The results show that the transmission coefficient decreases steadily as filler content increases, consistent with impedance mismatch theory and with effective dispersion of the filler in the matrix. Attenuation therefore rises with filler loading, from its lowest value at 12.5 wt% NLG to its highest at 62.5 wt% NLG. The composites achieved shielding effectiveness between 3.4 and 6.5 dB. These findings show that recycled glass and PCL can be combined into a cost-effective, environmentally friendly microwave shielding material. Such materials can be tailored for use in the electronics, telecommunications and medical industries.

Keywords: Electromagnetic shielding; finite element method; microwave attenuation; polycaprolactone; sodium lime glass

1 Introduction

The rapid proliferation of electronic and wireless communication devices has heightened concern over electromagnetic (EM) interference and radiation. Materials able to attenuate microwave signals have therefore attracted considerable research interest, since they reduce the intensity of EM radiation passing through them and are essential to electromagnetic compatibility (EMC) engineering [1]. Protecting electronic equipment from electromagnetic aggression is commonly achieved by enclosing circuits in a shielding package that reduces the impact of incident waves and electromagnetic coupling [2]. Extensive work has been devoted to developing highly effective shielding materials that extend the service life of electronic devices and limit the effects of EM interference.

Recent progress in microwave absorber technology has produced composite materials, in which two or more constituents are combined in a single or hybrid matrix, that can be tailored for narrowband or broadband absorption at low or high frequencies [3]. Such composites reduce the reflection of EM signals effectively, offer good physical performance and are cheaper to produce [4]. They combine useful properties of their components and can be optimised to give a particular balance of properties for a given application range [5]. Metal oxides are important constituents of advanced composites because their diverse structural geometries and electronic structures make them candidates for biological sensing, optoelectronics and electronics [6]. However, raw-material cost, weight and metal corrosion are recognised drawbacks [7,8]. Researchers have therefore combined such compounds with other materials, including carbon compounds and polymers.

Glass is a non-crystalline, inert, non-porous and fragile material that acts as a thermal insulator. Because it is also impermeable to oxygen and carbon dioxide, glass debris used as a filler in polyurethane resin can reduce collection costs, limit environmental pollution and lower the consumption of natural resources [9]. Glasses are used across a wide range of technologies, from chemically resistant containers and piping to fibre composites, pharmaceutical and sealing glasses, and nuclear-waste immobilisation [10]. Sodium lime glass (NLG), the most widely manufactured glass, is composed mainly of silica (SiO2), sodium oxide (Na2O) and calcium oxide (CaO); its dielectric properties, chemical stability and mechanical strength make it a candidate filler for microwave composites. The hydrophilic glass surface, however, adheres poorly to hydrophobic polymer matrices such as polycaprolactone (PCL) [11]. Silane coupling agents functionalise glass surfaces and create a covalent bridge between the inorganic filler and the organic polymer, improving interfacial compatibility and composite performance.

PCL is a biodegradable, semi-crystalline aliphatic polyester with a low melting point (~60 °C), which makes it easy to process by melt blending. Its biocompatibility, flexibility and dielectric properties make it a suitable matrix for microwave composites [12].

Mao et al. [13] studied the effect of alkaline-earth oxides (SrO, CaO, MgO) on low-temperature fired BaO–Al2O3–SiO2 (BAS) glass-ceramic/Al2O3 composites. The oxides progressively weakened the softening and crystallisation behaviour of the BAS glass-ceramics, enhancing densification, and affected crystallisation more strongly than softening, which widened the densification temperature range. The CBAS/Al2O3 composite performed best, with a dielectric constant of 5.81, low dielectric loss, high bending strength and a favourable coefficient of thermal expansion.

Assis et al. [14] replaced part of the sand in a clay-based ceramic body with comminuted recycled bottle glass at 3, 5 and 10%. Water absorption, apparent porosity, loss on firing and apparent density all improved, most clearly at 10% glass powder. In a related sustainability context, Matos and Coutinho [15] used ground waste glass as a partial cement replacement (0, 10 and 20%) in mortar and found that it improved resistance to chloride penetration and greatly improved sulfate resistance without compromising strength.

Silane coupling agents are widely used to functionalise glass surfaces for polymer composites. Aziz et al. [16] reported improved mechanical and dielectric properties for surface-modified glass fillers in polymer matrices, attributed to stronger filler–matrix interfacial bonding, and aminosilanes such as APTES have been reported to improve the compatibility of glass with polyesters including PCL.

FEM within a rectangular waveguide has been used to characterise PCL-based composites at X-band frequencies. Jimoh et al. [17] evaluated the power loss, attenuation and electric field intensity of PCL/rice husk composites at thicknesses of 5, 10, 15, 30 and 50 mm. The 50 mm sample showed the

lowest power loss (0.76 at 12 GHz) and an attenuation of −16.57 dB at 12 GHz, with electric field intensity falling as thickness increased, indicating the highest microwave absorption capability. Yakubu et al. [18] simulated rice husk/PCL composites of 10, 15, 20, 30 and 50 mm and found the lowest transmission coefficient (0.148468 at 12 GHz) and the least electric field intensity for the 50 mm sample; all thicknesses were considered suitable as microwave dummies. Together these studies support natural-fibre-filled PCL as a cost-effective, environmentally friendly microwave material and validate FEM as an efficient tool where full experimental measurement is unavailable.

Conventional microwave absorbers and attenuators rely heavily on ferrite-based and carbon-loaded materials, which are expensive, environmentally hazardous and non-recyclable [19]. NLG is the most abundantly produced glass worldwide and a significant post-consumer waste stream, yet its potential as a microwave-attenuating filler, particularly after silane surface treatment, has not been fully explored. In particular, the effect of silane treatment on the microwave transmission and attenuation of NLG/PCL composites made by melt blending has not been reported. This study addresses that gap. The complex permittivity of the composites is measured with an open-ended coaxial probe (OECP), and the finite element method (FEM) in COMSOL Multiphysics is used to determine the transmission and attenuation coefficients numerically.

2 Materials and methods

Sodium lime glass (NLG) cullet (waste glass) was used as filler. The coupling agent was 3-aminopropyltriethoxysilane (APTES). The matrix was polycaprolactone (PCL, Mw ≈ 80,000 g/mol). Ethanol (95%), acetone, acetic acid and distilled water were used for cleaning and silane solution preparation. Equipment comprised a pestle and mortar and ball mill, a balance (0.001 g precision), a heating mantle/hot plate with magnetic stirrer, a stainless-steel mould, an open-ended coaxial probe (OECP) connected to a vector network analyser (VNA), and a computer workstation running COMSOL Multiphysics (FEM).

NLG was crushed and ground in a mortar and pestle and then ball-milled to a uniform micrometre-sized powder. The particles were cleaned with acetone and dried at 100 °C for 2 h to remove moisture. A 2 wt% APTES solution was prepared in a 95:5 ethanol–water mixture, with pH adjusted to 4.5–5.5 using acetic acid. The glass particles were immersed in this solution and stirred for 1 h at room temperature, then filtered, washed with ethanol and cured at 110 °C for 30 min to promote siloxane bond formation.

PCL pellets were melted in a beaker on a hot plate at 65 °C. Silane-treated particles were added gradually to the molten PCL in the proportions given in Table 1 and blended thoroughly with a glass rod. The blend was cast into a stainless-steel mould and left to cool and solidify at room temperature. Five samples (A–E) were prepared with different NLG/PCL ratios.

Table 1. Composition of the NLG/PCL composite samples.

NLG filler (g)	NLG filler (%)	PCL (%)
5	12.5	87.5
10	22.5	77.5
15	37.5	62.5
20	50.0	50.0

NLG filler (g)	NLG filler (%)	PCL (%)
25	62.5	37.5

The complex permittivity, $\varepsilon^* = \varepsilon' - j\varepsilon''$, of each sample was measured with an OECP connected to a VNA over the X-band (8–12 GHz). The OECP method is a broadband, non-destructive technique widely used for solid and semi-solid materials. The reflection coefficient (S11) was recorded and converted to permittivity using the standard Agilent OECP model. The dielectric loss tangent was calculated as $\tan\delta = \varepsilon''/\varepsilon'$.

The measured complex permittivity values were used as material inputs in COMSOL Multiphysics. A rectangular waveguide with X-band dimensions was modelled, with each composite placed in the waveguide as a slab of thickness d. The transmission coefficient (S21) was computed for each composition, and the attenuation coefficient was derived from it as in Equation (1) [20]:

$$\alpha = \frac{-20\log_{10}|S_{21}|}{d}\,(\mathrm{dB/m}) \tag{1}$$

where d is the composite thickness in metres.

3 Theory

The test material is positioned within a section of the transmission line, and its characteristics are determined by analyzing both the reflected and transmitted signals. The fringe fields generated by the reflection are used to calculate the transmission and reflection coefficients. Transmission lines are chosen for these measurements because they provide high accuracy, with microwave guides offering strong field confinement. In this experiment, the sample was placed between two waveguide sections and exposed to electromagnetic radiation.

For transmission coefficient measurements, a 10 mm specimen was carefully inserted into the rectangular waveguide (RWG), which was connected via coaxial cable to a network analyzer (Agilent 8570B). To ensure the instrument's reliability, the waveguide was first used to measure air and PTFE, both of which serve as standard reference materials. The sample was machined to fit snugly inside the waveguide, eliminating any air gaps between the specimen and the guide walls.

Figure 1 illustrates the RWG measurement setup. As expected, the transmission coefficient shows a higher magnitude than the reflection coefficient; theoretically, the transmission coefficient should approach unity [21].

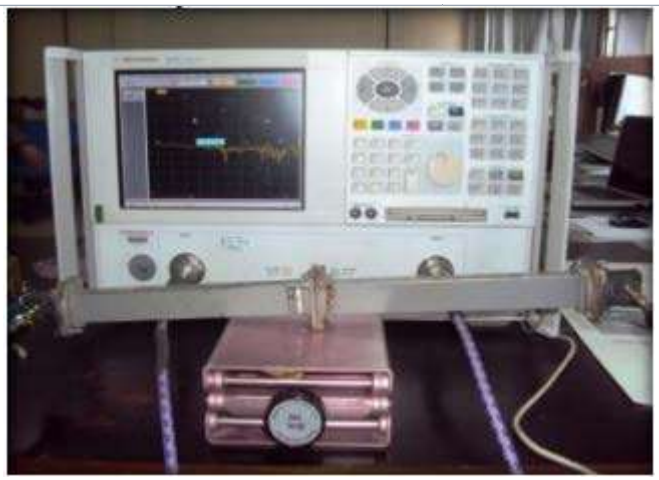


Figure 1. Measurement set-up for S-parameters.

The scattering (S) parameters of a two-port network are defined in terms of wave variables. The nomenclature used for the scattering parameters is as follows: S11 and S22 are called the reflection coefficients, whereas S12 and S21 are called the transmission coefficients. These are the parameters directly measurable at microwave frequencies. In general, the S parameters are written in complex form as in Equation (2) [22]:

$$S_{gg} = \frac{b_g}{a_g}\,(g = 1,\ 2),\ S_{pg} = \frac{b_p}{a_g}\,(p \neq g;\ p,\ g = 1,\ 2) \tag{2}$$

The reflection coefficient is given by Equation (3):

$$\Gamma_g = S_{gg} = \frac{b_g}{a_g} \tag{3}$$

The transmission coefficient is given by Equation (4):

$$T_{g\to p} = S_{pg} = \frac{b_p}{a_g} \tag{4}$$

The transmission coefficients (S21) obtained from the vector network analyzer (VNA) are then used to compute the attenuation of the material under study using Equation (5):

$$\text{Attenuation (dB)} = -20\log_{10}|S_{21}| \tag{5}$$

4 Experimental results

After completing the measurements, the raw data were used to plot the transmission parameter S21 against frequency (Figure 2). Careful inspection of the plot reveals that the transmission coefficient decreases progressively as the amount of NLG filler increases. This trend aligns with the theoretical expectation that denser materials reduce transmission. The highest transmission coefficient was observed at the lowest filler loading (5 g), while the lowest value corresponded to the highest filler loading (25 g). These findings are consistent with impedance mismatch theory, which predicts that materials with higher permittivity exhibit reduced transmission coefficients [23].

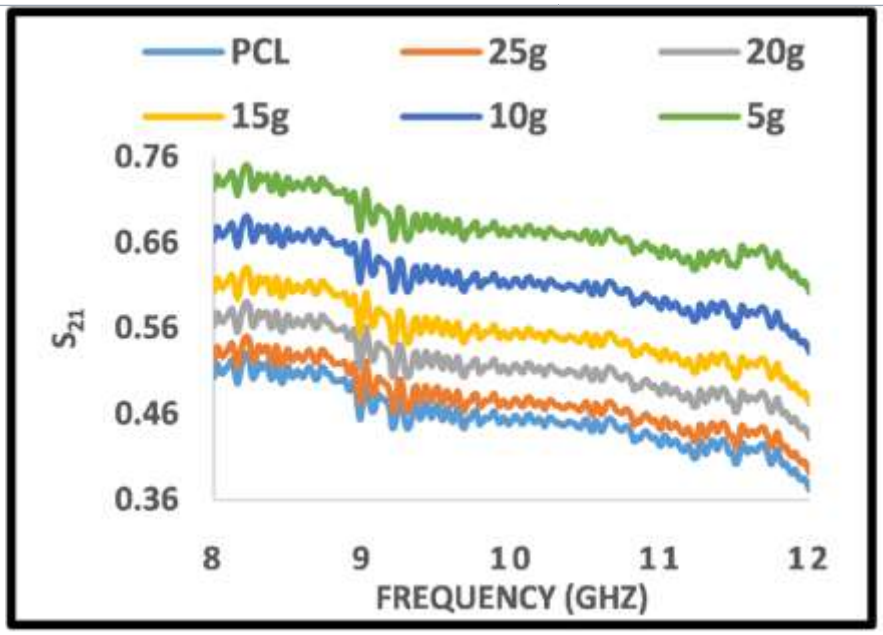


Figure 2. Measured S21 for all samples.

Figure 2 demonstrates that the transmission coefficient decreases with increasing frequency across all samples. This behavior can be attributed to effective dispersion of the filler within the matrix, confirming uniform particle distribution [24]. Such dispersion influences the transmission of electromagnetic radiation, either enhancing or reducing it depending on the distribution of NLG dust. Overall, the findings indicate that higher filler content consistently reduces the transmission coefficient magnitude for all compositions throughout the entire frequency range, as illustrated in Figure 3.

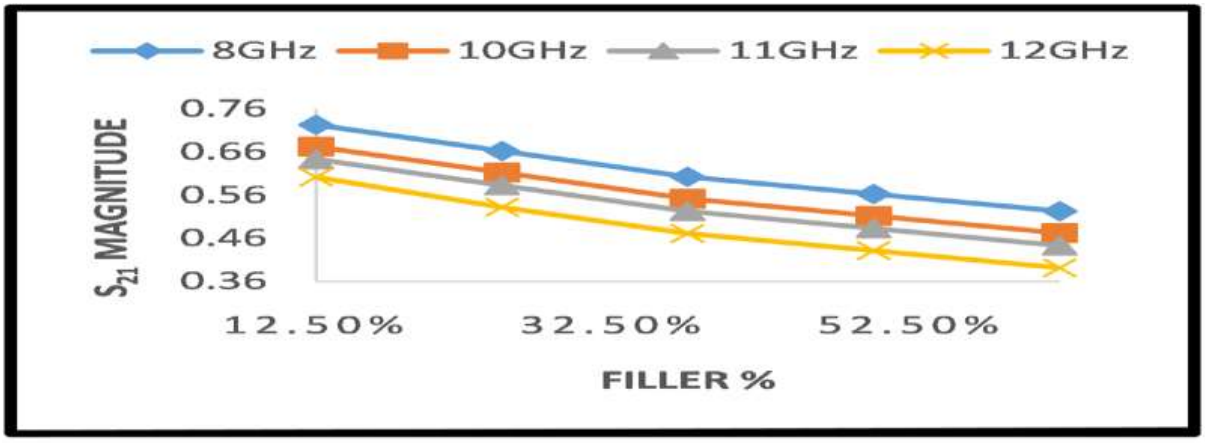


Figure 3. NLG filler content versus transmission coefficient.

The sinusoidal waveform observed across the frequency range is attributed to impedance mismatches among the waveguide's input impedance, the sample's surface impedance, and the coaxial cable's characteristic impedance [25]. To determine the attenuation of the NLG–polymer pellets, the raw transmission coefficient data were applied to Equation (5). The resulting calculations, presented in Figure 4, reveal that the lowest attenuation occurred at 12.5% NLG filler content, while the highest attenuation was recorded at 62.5%. These findings are consistent with the S21 data, as composites with reduced transmission exhibit greater absorption.

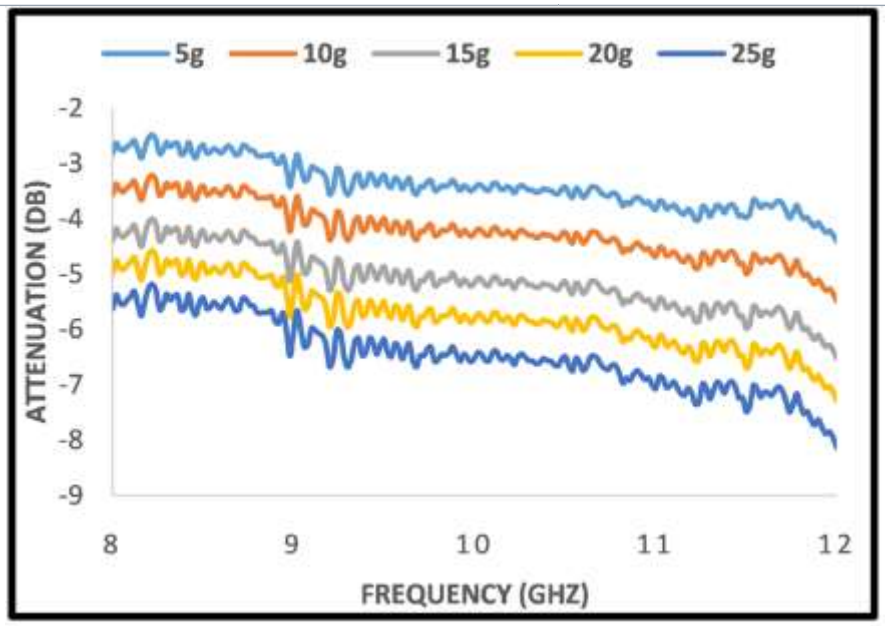


Figure 4. Calculated attenuation of the different compositions of NLG–PCL composites.

The graph illustrates a steady increase in attenuation with rising filler content. This trend supports the theory that denser materials reduce transmission and may be linked to effective dispersion of the filler within the matrix [26]. Uniform particle distribution influences the attenuation of electromagnetic radiation, with the degree of dispersion of NLG particles in the matrix determining whether attenuation increases or decreases.

5 Conclusion

The synthesis of NLG–polymer composites was successfully accomplished in this study. Electromagnetic wave propagation through a rectangular waveguide (RWG) and the attenuation behavior of the samples were examined at microwave frequencies. The investigation focused on transmission coefficients, attenuation, and surface morphology of the prepared composites.

Shielding effectiveness refers to the ability of specialized materials to reduce electromagnetic interference (EMI) by limiting the penetration of fields or waves into a designated enclosure. The findings confirm that the developed composites are suitable for microwave shielding applications, with effectiveness ranging between 3.4 and 6.5 dB. Based on these results, the materials can be tailored for practical use in the electronics, telecommunications, and medical industries.

Conflicts of interest

The authors declare no conflicts of interest regarding the publication of this paper.

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