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Shielding Effectiveness, Mechanical and Dielectric Properties of NiO/PCL Nano Composites at Microwave Frequency for Electronic Devices

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

There is a continuous generation of electromagnetic fields through operations of electronic devices for this reason it is paramount that these fields be shielded so as to prevent interferences. The, conventional method for shielding these fields are by the use of thin metal foils or sheets. The metals foils are heavy, difficult to fabricate, costly and in many cases not suitable for use in many applications. For these reasons, this work is focused on using materials that is flexible, cost effective and durable, with considerable shielding effectiveness (SE). Hence, polycaprolactone (PCL) and nickel oxide (NiO) nanocomposite were synthesized using the conventional melt blending (CMB) technique. The synthesis method used is fast, easy and produces large mass of controlled composites within a short period. Rectangular waveguide, vector network analyzer, coaxial cable and open ended coaxial probe were used in the measurement of microwave properties. Measured scattering parameter was used to calculate the shielding effectiveness of the NiO/PCL composites. Findings indicates that the dielectric constant increased with increasing filler

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content, where the highest dielectric property was 5.09 for the 62.5 % filler and it was able to shield electromagnetic fields by up to -9.35 dB at the frequency range measured. The average particle size of the NiO nano particles was 40.5 nm using TEM analysis. The best hardness and tensile strength was recorded for the highest loading percentage. It is then concluded that the substrate produced can be tailored for electronic, telecommunication, medical applications and military shield applications.

Keywords: Shielding effectiveness; nano particles; tensile strength; TEM; dielectric and constant; hardness.

1. INTRODUCTION

Polymer nanocomposite is a major factor in the design of novel advanced materials suitable for a variety of different applications, such as electrical engineering and high frequency devices like microwave especially to shielding applications. In the production of systems, polymers reinforcement using different types of organic or inorganic fillers is common practiced to improve mechanical, dielectric properties [1, 2]. Properties of the polymer nanocomposites are affected by factors, like, intrinsic characteristics of each component, contact, shape and dimension of the fillers, and the nature of their interfaces [3]. Knowledge of materials behaviour placed in an electromagnetic field is of immense importance especially when it relates to military hardware, electronics, communication, and industrial applications and shielding [4]. Most applications in engineering do deal with attenuation and shielding effectiveness. It is then paramount to understand characterization of materials for attenuation and shielding in the designing and fabrication of components for microwave operations. Polymers are packed with advantages that run from simplicity of fabrication, light weight, low cost, and excellent insulating property. These advantages has made polymer one of the suitable material for electrical and electronics applications [5].

Researches and literature on polymer reinforced with fibers, metals and ferrites has been on the increase but to the best of our knowledge there is a dearth on research involving polymer and metals and their dielectric, mechanical and shielding properties. In [6], the effect of the various ratios of Fe_2O_3 on the composites of OPEFB/PLA were studied. Their results showed that the dielectric property increased by increasing the Fe_2O_3 ratio in the composites while absorption loss increased with increasing ratio of Fe_2O_3 filler in the composite.

It is reported in [7], that the dielectric properties, scattering parameter and power loss was investigated for epoxy resin (ER) reinforced with different percentages oil palm empty fruit bunch (OPEFB) at 8–12 GHz. Results from investigation showed that the dielectric properties increased with increasing OPEFB percentage in the composites. In [8], it was reported that ferroelectric polymers and copolymers blends were investigated to study its effect on complex permittivity and potential EM wave's absorber. Findings from the investigation showed that there was significant variation in the complex permittivity and attenuation with different fillers concentration. In production systems, polymer reinforcement using different types of organic or inorganic fillers is practiced so as improve its dielectric, mechanical, and tensile strength properties [9]. Researches have been reported that the properties of polymer composites are affected by inherent characteristics of each component, contact area, dimension and shape of the fillers, and the nature of their interfaces [10]. In [11], it was reported that conductive mineral powders such as carbon, barite, and iron (III) oxide were added to waste paper fibres in two different ratios in order to provide electromagnetic shielding. They found that electromagnetic shielding effectiveness of waste paper fibres with mineral powders was of acceptable values. Based on the current research trends on polymer composites, it is suggested that polymer composites is important in the fabrication of good novel materials in the areas of electrical engineering, high frequency, shielding applications, microwave absorption, electromagnetic interference (EMI) and sensors [12]. For shielding effectiveness determination, scattering parameter measurement must be carried out and fully understood. For this study, concentration is based on the effect of the filler content on the matrix and how it affects the properties of the composites prepared. In this work, NiO-PCL composite structures were synthesized, and investigated on dielectric property, mechanical property, morphology and

SE at 8-12 GHz were carried out. The microwave properties of samples were investigated at filler percentages of 12.5 %, 22.5%, 37.5%, 50 %, and 62.5%. The total volume fraction of the composites was set at 40 g. There is dire need to protect electronic components and wireless gadget against electromagnetic interference particularly at microwave frequency. For this singular reason, results from this research would certainly aid in the understanding of material fabrication for EM wave's absorption.

1.1 Theory

The parameters S_{11} and S_{22} are called the reflection coefficients, whereas S_{12} and S_{21} are called the transmission coefficients. These are the parameters directly measured at microwave frequencies. Generally, the S parameters are written in complex form as [13];

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

The reflection coefficient;

$$\Gamma_g = S_{gg} = \frac{b_g}{a_g} \qquad (2)$$

The transmission coefficient;

$$T_{g\to p} = S_{PP} = \frac{b_p}{a_p} \qquad (3)$$

To obtain the total SE, the result obtained via the Vector Network Analyzer (VNA) for the scattering parameter is used using Eq. 4.

$$SE_T(dB) = SE_R + SE_A + SE_M = 10log_{10}\left(\frac{P_t}{P_i}\right)$$
$$= 20log_{10} = 20log_{10}\left(\frac{H_t}{H_i}\right) \qquad (4)$$

where Pi (Ei or Hi) and Pt (Et or Ht) are the power (electric or magnetic field intensity) of incident and transmitted EM waves, respectively.

3. METHODOLOGY

3.1 Preparation of NiO Nanoparticles and NiO/PCL Nano Composites

Pure NiO powder with purity of 99.7 % bought from Sigma Aldrich was sintered for 8 hours at 700 ^{0}C, the sintered granules was then crushed before it was introduced into a high energy milling machine (SPEX 8000D) shaker for 12 hours. The ball to powder mass ratio used in the mechanical alloying was 10:1. After 12 hours in the shaker, the powder was then removed and kept for further investigations.

The NiO-PCL nanocomposites was prepared via the melt blend technique using the Thermo Haake extruder Polydrive three-phase motor with a drive of 1.5 kW, 230 V, 40 A and speed range of 0 to 120 min^{-1}. In this method, the machine is set to desire heating temperature and allowed to heat up. All other components of the machine were set following recommended settings. For our method, the machine is set to 90 ^{0}C for heating, the rotation of the rotors was set to 50 min^{-1}. After the machine has reached the required temperature (90 ^{0}C), the polycaprolactone (PCL) is poured into the vial of the heating block. After 5 min, the NiO nanoparticles is introduced into the vial. The mixture is left for another 20 min before taken out and fabricated into desired dimension using hot and cold press. A 3 mm thick pellet was fabricated from each of the samples prepared while measurement of dielectric constant was carried out using vector network analyzer. The NiO nanoparticles and NiO/PCL nanocomposites were analyzed with transmission electron microscope (TEM), and scanning electron microscope (SEM). Table 1 illustrates the ratio of blending used in this work. Tensile strength and hardness of materials were carried out on an Instron 5585H using the Instron Bluehill 2, version 3.4 software. Tests were performed in accordance with ASTM standard D638 using a crosshead speed of 5 mm/min.

Table 1. Composition of samples prepared

NiO (Nano) %	PCL %	Total Mass (g)
12.5 (5g)	87.5 (35g)	40.0
22.5 (10g)	77.5 (30g)	40.0
37.5 (15g)	62.5 (25g)	40.0
50.0 (20g)	50.0 (20g)	40.0
62.5 (25g)	37.5 (15g)	40.0

3.2 Dielectric Measurement

The network analyzer model used for measurement was the Agilent PNA-L N5230A. Set up is as shown in Fig. 1.

3.3 Morphological Characterization

The microstructure of all the samples were studied using a Hitachi S-3400N SEM at

Fig. 1. Measurement Set-up for (a) T/R Coefficients (b) Sample fitting (c) Complex permittivity (d) Shorting Block and Probe Sensor

20kV.The samples were coated with gold and palodium with a sputter coater (Emitech K 550X) so as to increase their conductivity and eliminate electromagnetic charges before placing the samples in the SEM and TEM vacuum chamber. The duration for coating of the samples is 3 minutes and the thickness recommended for coating is 4 nm.

4. RESULTS AND DISCUSSION

4.1 Surface Morphology

Observation on Fig. 2(a) showed areas where particles overlaps one another which could be attributed to the cohesive bonding force of NiO nano particles [14, 15]. The morphology and size distribution were almost uniform based on an Image Tool Software (Image J). The diameter of the particles sizes obtained for the NiO nano particles was in the range of 40.9 – 50.1 nm. Fig. 2(b) is the SEM image of the pure PCL after melting, the surface morphology shows that the PCL was indeed melted to required morphology at the melting temperature.

Micrograph shown in Fig. 3 is the fractured surface of the NiO-PCL nanocomposites. It shows the NiO nanoparticles as white particles dispersed throughout the matrix of nanocomposite. The increase in agglomeration can be attributed to an increase in specific surface area, associated with reduced particle size, resulting in higher adhesion forces [16].

4.2 Mechanical Properties

Fig. 4 shows that the tensile strength increased with NiO nano particles percentage from 4.30 MPa to 5.83 MPa at 10 % filler composition. This increase is attributed to the addition of stiff NiO nano particles into the polymer matrix, in addition, the uniform dispersion of NiO nano particles resulted in good load transfer causing an improved mechanical properties at a high loading percentage.

Fig. 5 shows that the hardness of NiO/PCL nano composites increases with increase in filler percentage that was incorporated into matrix. The highest value of hardness recorded at 62.5 % filler loading might be attributed to large surface area due to close boundary spacing in the domain walls [17].

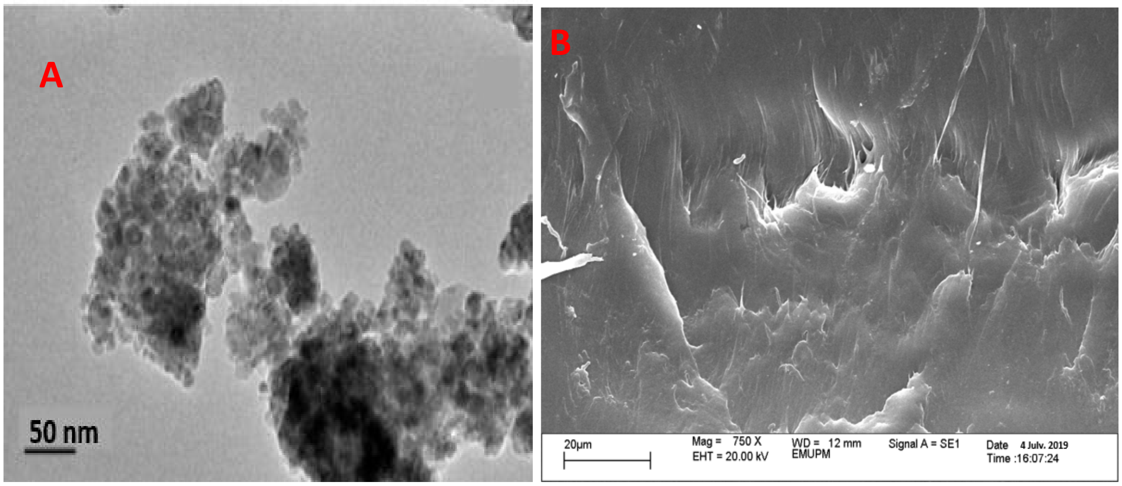

Fig. 2. (a) TEM image of NiO nano-particles (b) SEM image of PCL

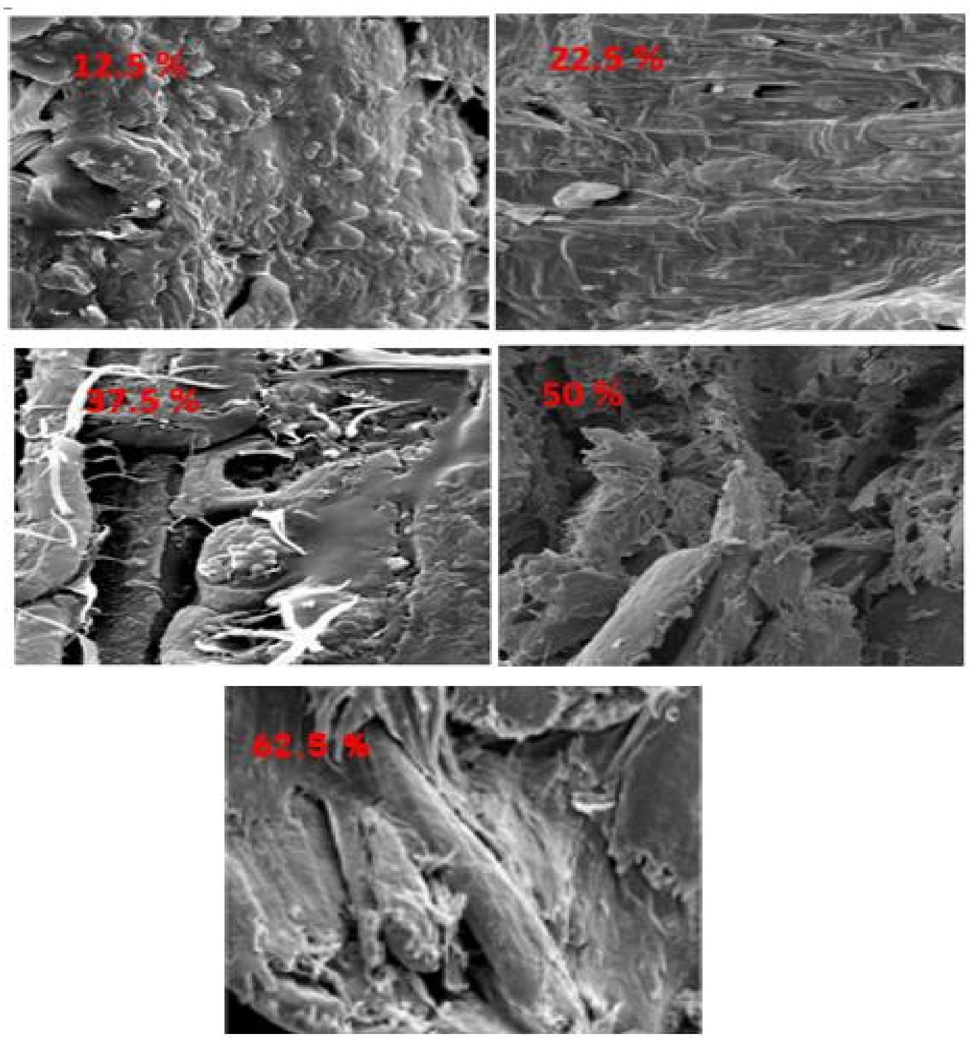

Fig. 3. SEM images for NiO/PCL nano composites

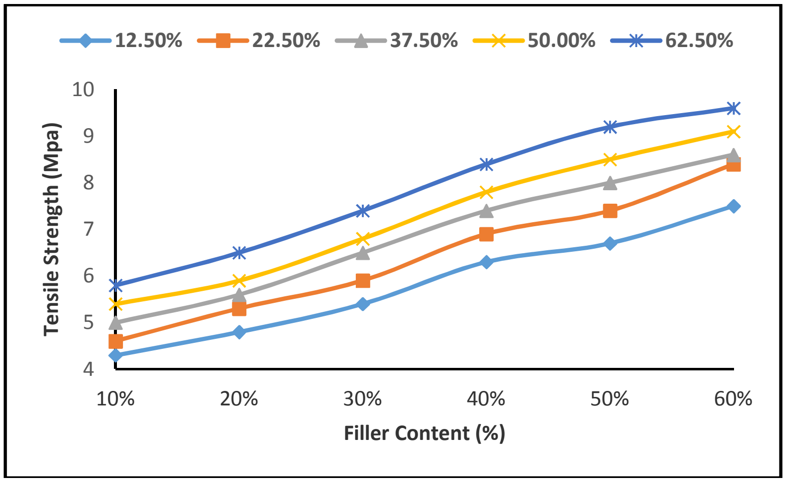

Fig. 4. Tensile Strength for Filler (%) of NiO/PCL Composites

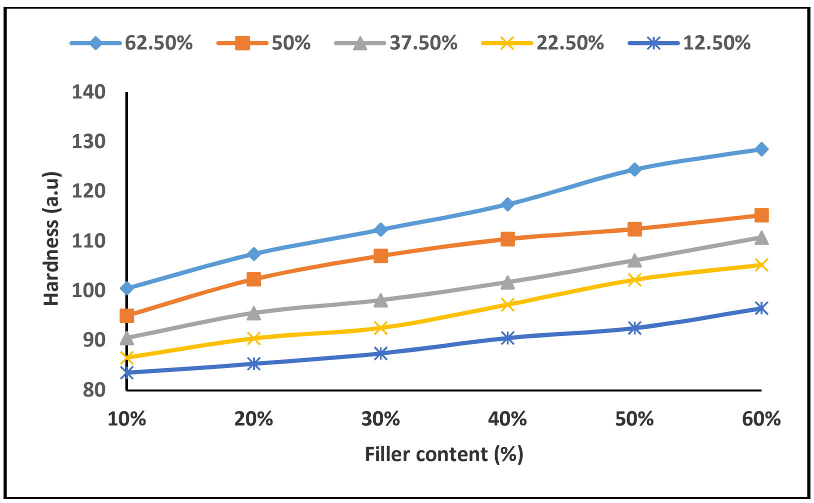

Fig. 5. Hardness for Filler (%) of NiO/PCL Composites

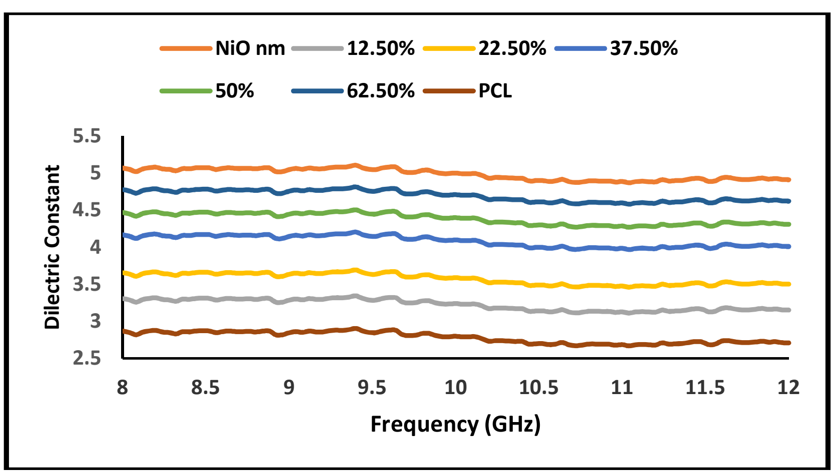

Fig. 6. Dielectric constant of NiO nano, PCL and NiO/PCL nano-composites

The dependence of dielectric constant on frequency can be explained using interfacial polarization [18]. Based on the effective medium theory, complex permittivity of polymer-based composite can be increased by adding fillers with higher permittivity values [19] as observed in Fig. 6.

Fig. 7, suggest that NiO nanofiller can change the property of the composites from a medium loss material to a dispersive high loss material which is attributed to the high loss filler material.

Fig. 8 is in agreement with impedance mismatch theory where materials with highest permittivity depicts lower transmission coefficient values [20, 21].

The significant difference in the increase of reflection coefficients (Fig. 9) at higher filler content might be attributed to the fine distribution and volume of the filler in the matrix.

The overlapping behavior for the 12.5 and 22.5 % filler composites might be as a result of the minimum particle distribution and infinitesimal weight percent difference [22]. It therefore connotes that the reflection coefficients at this filler composition is influenced mainly by the polymer.

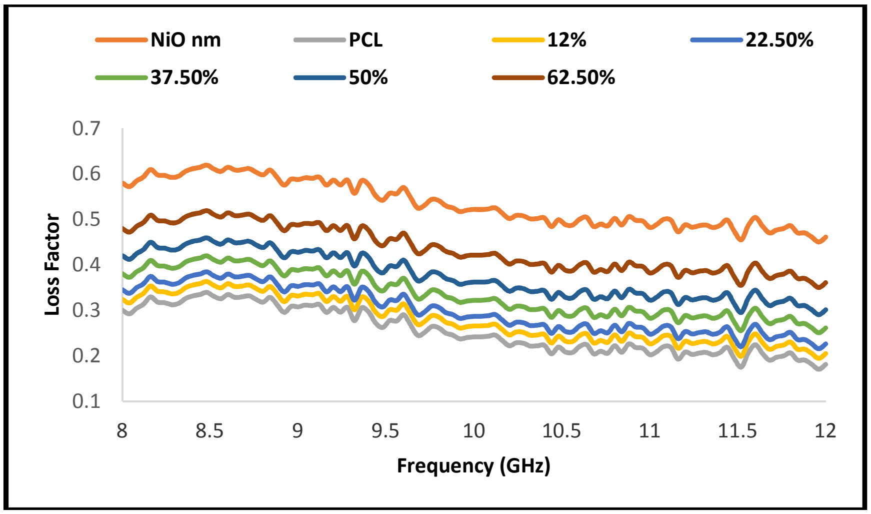

Fig. 7. Loss factor of NiO nano, PCL and NiO/PCL nano-composites

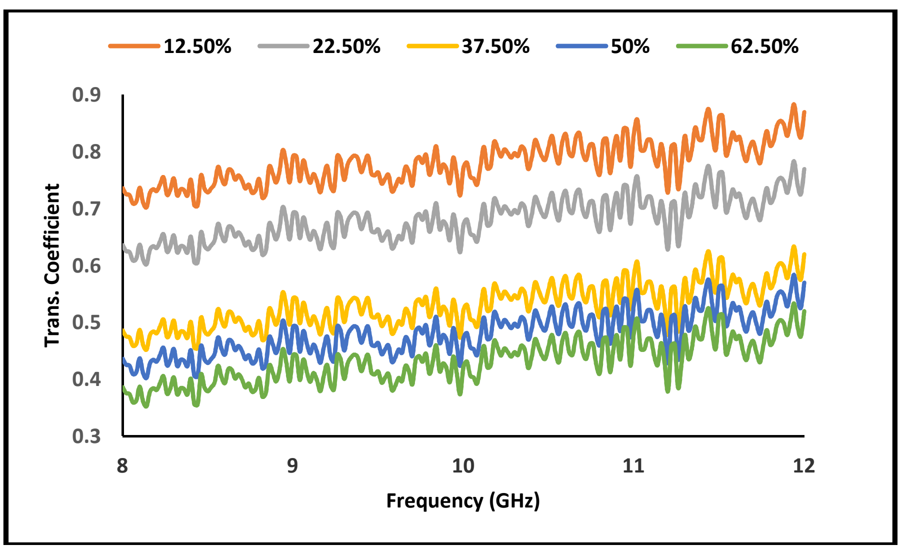

Fig. 8. Transmission Coefficient of NiO/PCL Nanocomposites

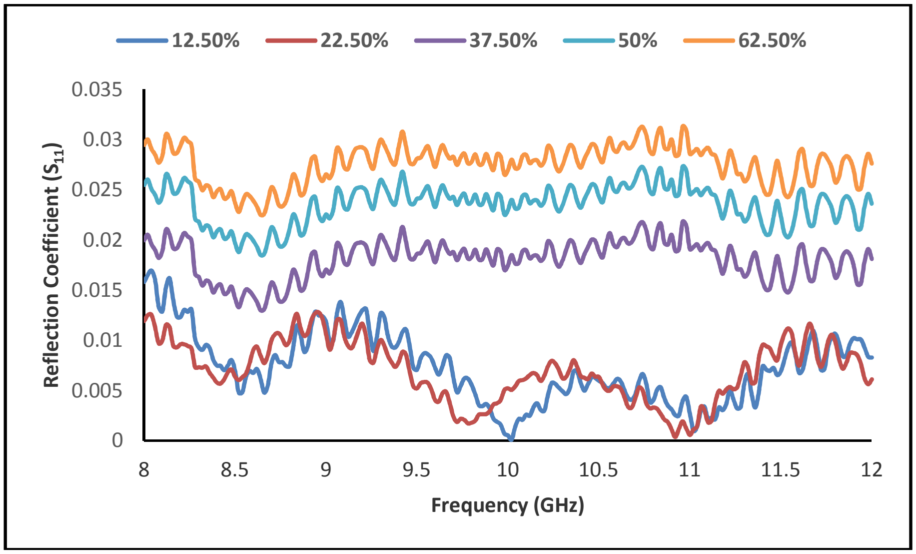

Fig. 9. Reflection Coefficient of NiO/PCL Nanocomposites

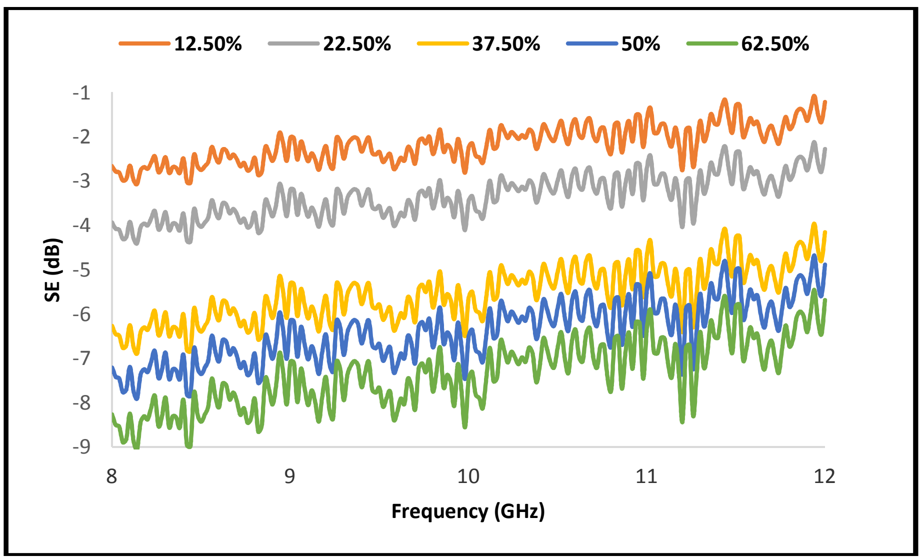

Fig. 10. Shielding Effectiveness of NiO/PCL Nanocomposites

Observation on Fig. 10, confirms that shielding is dependent on the interaction of composites particles fraction and frequency hence higher shielding as particle sizes of the composites decreases [23].

The result suggests that an average of -9.35 dB of SE could be obtained by using a filler of 62.5% in the composites matrix. A comparative study based on SE performance between existing materials and the newly introduced metal/polymer composite-based SE is depicted in Table 2.

Table 2. Comparison of SE for existing materials and present study

Materials	SE (dB)	Reference
NiO/PCL Nano	-9.35	This study
Rice Husk/PCL	-12.40	[2]
MWCNT	40.07	[5]
OPEFB/PCL	-6.35	[6]
Recycled Fe_2O_3	-9.0	[16]
Corn Husk Powder	-7.20	[22]

Based on the values obtained for the dielectric constant and the linear regression coefficient

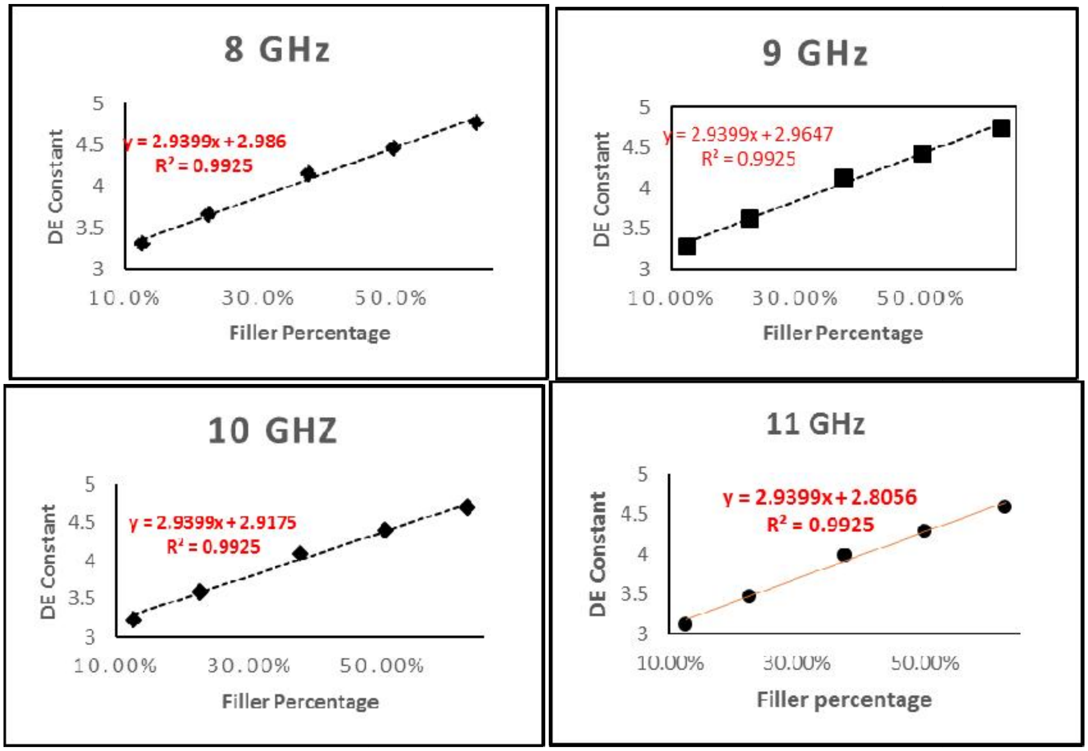

Fig. 11. Regression Analysis for the Dielectric Property of NiO/PCL Nanocomposites

Table 3. Regression coefficients for dielectric constant with respect to frequency

Frequency (GHz)	Equation	Regression coefficient (R^2)
8.0	$\varepsilon' = 2.9399x + 2.9860$	0.9925
9.0	$\varepsilon' = 2.9399x + 2.9647$	0.9925
10.0	$\varepsilon' = 2.9399x + 2.9175$	0.9925
11.0	$\varepsilon' = 2.9399x + 2.8056$	0.9925

correlation, showed that the dielectric constant values were correlated to filler percentage increase since all values were close to unity as shown in Table 3. Fig. 11 showed that the sensitivity of all the composites as filler content increases varies orderly with loss factor and there is a strong sensitivity in the range of 2.99 to 2.81 from 8 to 11 GHz. The regression equations can be used to predict the percentages of NiO nano filler in the PCL host matrix.

5. CONCLUSION

In this work the structural, mechanical, dielectric and SE properties of NiO/PCL nano composites were investigated. Results obtained for the composites showed a fractured inhomogeneous surface while the NiO filler prepared was in nano dimension. The dielectric properties of the samples increased with increasing filler content to a maximum value of 5.09 while the loss factor followed the same trend as the filler content increases.

The transmission coefficients decreases with increases in filler loadings for all samples which is evident in the result for the dielectric property, as high dielectric materials tend to absorb more of electromagnetic radiation. The calculated SE for the composites showed lowest values at 8 GHz and highest at 12 GHz.

Results confirms that the 62.5 % NiO nano filler when introduced into the matrix was able to produced shielding effectiveness of -9.35 dB. Based on the result obtained, it is suggested that NiO nanofiller can change the property of polymeric composites from a medium loss material to a dispersive high loss material due to their high loss property. Hence, the material produced in this work can be tailored for shielding in electronic, telecommunication and medical applications.

DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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