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Carbon nanotube reinforced LDPE/SAN blend-based binary thermoplastic nanocomposites: thermo-mechanical and electromagnetic interference shielding properties

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ABSTRACT

Electromagnetic interference (EMI) is electromagnetic wave pollution emitted from natural or artificial sources, causing damage to users, the mother device, and the surrounding electronic instruments. EMI shielding materials of electrically conductive and flexible thermoplastic nanocomposites are increasingly popular due to their low cost and recyclability. An ingenious material solution is two-phased nanocomposites with distinctive structural and electrical characteristics. This work proposes a binary thermoplastic blend nanocomposite system with better EMI shielding than comparable systems. The thermoplastic blend consists of styrene-acrylonitrile (SAN) and a low-density polyethylene (LDPE) masterbatch compatible with multi-walled carbon nanotubes. The injection molding method was used in the production of nanocomposites. Thermo-dynamic and electromagnetic shielding properties of the nanocomposites were investigated. As the LDPE/MWCNT ratio added to pure SAN increased, the storage modulus decreased as expected, and the storage modulus for the MB50 sample was determined as 1.24 GPa with a 50% decrease. The percolation threshold for the two-phase thermoplastic composite was obtained for the MB50 sample containing 10 wt.% carbon nanotubes. In addition, the MB75 sample containing 15 wt.% carbon nanotubes reached an EMSE value of 37 dB. The outcomes have the potential to be applied to produced materials as structural plate elements or shielding casings in commercial applications.

I. INTRODUCTION

The use of electronic devices with advancing technology, developments in the defense industry, and communication technologies have increased the need for electromagnetic shielding. Electromagnetic interference (EMI) is electromagnetic wave pollution emitted from natural or artificial sources, causing damage to living organisms' health in wide frequency ranges, reducing the operating performance of electronic devices adjacent to the electromagnetic wave source, or destroying stored electronic information. For example, people exposed to electromagnetic radiation for an extended period may suffer from several health problems, such as fatigue, memory loss, acceleration, or slowing of the heartbeat [1, 2]. Protection from electromagnetic waves is becoming more critical due to their adverse effects, and the studies on developing protective materials are increasing [3]. Electrical conductivity is necessary for the material to shield against electromagnetic radiation [4]. Therefore, due to their conductivity properties, metals are widely used to protect against electromagnetic waves. However, metals are heavy, have low environmental stability, tend to corrosion, need to be more convenient in the production phase regarding applicability and processability, and bring essential problems such as sustainability [5]. Researchers are studying next-generation materials for the increasing protection requirements, which can replace traditionally used

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*Corresponding Author Tel.: +90-216-218-0018; e-mail: veskizeybek@comu.edu.tr metals due to their limitations. Therefore, the focus is on low-density, light, robust, chemically resistive, and easily processable materials such as polymers or polymer matrix composites as alternative shielding material [6, 7].

Dielectric ceramics, magnetic oxides/ferrites, semiconductors, metal particles/foams, conductive polymers, conductive carbon black/fibers/nanotubes, and other carbon derivatives like graphene, have all been the subject of extensive research during the last decade [4, 8-10]. However, adding nanoparticles at the high rates required to satisfy the prerequisites for an effective magnetic shield often leads to high costs and poor mechanical properties [11]. On the other hand, the main challenge with low loading of conductive fillers is how to form highly effective conductivity pathways [8, 11]. In recent years, the focus has been on developing nanocomposites at low filler content by controlling the phase morphology and microstructure of the two-phase polymer matrix [8, 12]. The phenomenon of double percolation threshold provides advantages over single-phase polymer composites since it is possible to achieve conductivity improvements in electrical conductivity at low concentrations through selective localization of conductive particles in one of the phases or at the interface [8, 9, 11].

Liebscher et al. studied the dispersion of two types of graphite nanoplates, namely graphene nanoplates (GnPs) and expanded graphite (EG), in polycarbonate (PC)/SAN = 60/40 wt.% polymer blends. The blends were prepared with a two-stage melt-mixing approach, and the melt's rheological and electrical properties were investigated. They reported that, in contrast to expectations, the electrical conductivity values were low for both new mixes when better dispersion was achieved. On the other hand, a better dispersion increased the reinforcing effect, according to the melt rheological studies [13]. In the studies that carbon nanotube (CNT) used as conductive filler, Bizhani et al. prepared the PC/SAN binary blends (60/40) included with multi-walled carbon nanotubes (MWCNTs) using a melt-compounding process [11]. They revealed that the maximum DC electrical conductivity was 0.0834 S/cm, and the maximum shielding efficiency was 25–29 dB at X-band (8.2–12.4 GHz frequencies) for the 1 wt.% MWCNTs reinforcement ratios. In another study, Liebscher et al. studied the electrical and melt rheological properties of MWCNTs reinforced PC and co-continuous PC/SAN blends [14]. They used the melt-mixing process for mixing the polymers and obtained an increase in electrical resistivity near the percolation threshold of PC–CNT composites and (PC + CNT)/SAN blends at increasing CNT dispersion, proving that higher mixing energies required for better dispersion also result in a more severe reduction of the CNT aspect ratio. Darshan et al. studied the immiscible PBS/HDPE polymers by adding CNT as a nanofiller mixed by a melt-mixing process [15]. The rheological tests suggest that the size of dispersed high-density polyethylene (HDPE) droplets decreased by using malleated PE as a compatibilizer, leading to a double-percolated structure. At the same time, CNTs were preferentially distributed in the HDPE phase. Praveen et al. investigated the compositional parameters $CoFe_2O_4$ and MWCNT/Graphene nanoplatelets reinforced low-density polyethylene (LDPE) polymer given EMI shielding [10]. They declared that 50:5:40:5 ratios were optimal for achieving efficient dielectric, ohmic, and magnetic losses in the X-band for DPE:MWCNT:GNP:$CoFe_2O_4$ composites. Also, they obtained 49 dB as the maximum total shielding effectiveness at 10.3 GHz.

As mentioned in the literature, developing polymer composites with electrical conductivity is possible by applying conductive nanofillers to two-phase blends [10, 14-16]. However, obtaining stable and homogeneous continuous structures is challenging [6]. Additionally, fillers, such as CNTs, are difficult to disperse in the polymer matrix and are potentially harmful and toxic when used in powder form [16, 17]. To minimize the dispersion and toxicity problems in industrial applications, reactive polymer components known as masterbatches containing high concentrations of fillers have been developed instead of powder reinforcement materials [17]. This method is called the melt dilution technique. At the same time, masterbatches facilitate nanofiller dispersion and increase homogeneity in a matrix, decreasing health risks due to dust and providing easy shipping and manufacturing processes on injection and extrusion machines [16, 17]. Perie et al. manufactured fine and better homogeneity polymer nanocomposite by mixing masterbatches of low molecular-weight amino-terminated polyamide-6 (PA6) containing ~10-17 wt.% of MWCNT with maleic anhydride functionalized PE at temperatures above melting of PA6 [17]. They also declared the resistivity of the nanocomposites effectively decreases by adding higher ratios of masterbatches. Clemente et al. studied the conductivity, rheology, and morphology properties of MWCNT/PA66/PA6 nanocomposites produced by the melt dilution technique [16]. According to their results, the nanocomposites processed from the PA6 masterbatch give higher stiffness values. The nanocomposites with 50/50 PA66/PA6 ratios provide the highest electrical conductivity results compared to the other ratios. They stated that this result was due to the nanotubes forming well-dispersed large agglomerates in a continuous mixture morphology, and it is emphasized that the lower viscosity of nanocomposites produced from PA6 masterbatch is another essential factor.

Considering the need for innovative and multi-purpose materials, using conductive nanoparticle-reinforced masterbatches can be an efficient and easy production strategy for obtaining electrically conductive polymers that offer a wide range of usage potential, such as EMI shielding. LDPE is a well-known engineering plastic due to its simple, cheap, abundant synthesis process and excellent environmental stability [10]. However, while it is widely used in many areas, using LDPE at conductivity-required applications such as EMI shielding is impossible because it is electrically insulated. Fortunately, as mentioned, conductive particle reinforcement, such as MWCNT, offers a solution to this issue. SAN has higher elastic modules, perfect tensile, rigidity, hardness properties, satisfactory thermal shock, and chemical resistance than LDPE [18]. SAN/LDPE/MWCNT blends have recently been the topic of inquiry, although various recent investigations have demonstrated that subjected the properties of two-phase nanocomposites. In addition, no study has been reported using melt-mixing and melt-dilution manufacturing methodology on SAN and LDPE polymers. This study aims to design, develop, and produce nanocomposites with a thermoplastic matrix with sensible EMI shielding properties by scalable engineering production methods. For this purpose, dual-phase nanocomposites were fabricated using MWCNT-reinforced low-density polyethylene (LDPE) and styrene acrylonitrile (SAN) with melt-mixing and melt-dilution techniques. A hierarchical and compositional parameters nanocomposite structure was produced to gain electromagnetic shielding capability.

II. EXPERIMENTAL METHOD

2.1 Materials

In this study, acrylonitrile SAN 300 (Kumho, South Korea) and LDPE (Plasticyl LPDE2001, Nanocyl, Belgium) masterbatch containing 20wt% multiwalled carbon nanotubes were used as the thermoplastic polymer matrix. The carbon nanotubes that reinforce the LPDE matrix have >90% purity, approximately 9.5 nm inner diameter and 1.5 µm length, and 250-300 m^2/g surface area (Nanocyl NC7000, Belgium).

2.2 Production of LDPE/MWCNT/SAN Nanocomposites

LDPE/MWCNT/SAN nanocomposites were obtained by mixing different ratios of LDPE/MWCNT masterbatch into the SAN matrix (Table 1). The MWCNTs ratio in the two-phase thermoplastic matrix was a maximum of 20 wt% and a minimum of 0 wt% by the plastic injection method. Engel Spex Victory 80 injection molding machine with 25 cm long co-directional twin screw and 800 kN injection molding force was used for plastic injection. In the production of the samples, a temperature profile of 200 °C was uniformly followed in all heating zones. The specimens were injected into stainless steel molds.

Table 1. Compositions and sample codes of the prepared composite samples

Sample	**LDPE** *(%)*	**SAN** *(%)*	**MWCNT** *(%)*
MB0	0	100	0
MB25	20	75	5
MB50	40	50	10
MB75	60	25	15
MB100	80	0	20

2.3 Characterization

Viscoelastic properties of nanocomposites were evaluated using dynamic mechanical analysis (DMA) under dynamic fatigue loading at varying frequencies and temperatures. DMA in three-point bending mode was performed on rectangular specimens ($80\times13\times3 \pm 0.3$ mm^3) using a Mettler Toledo DMA/SDTA861e analyzer according to ASTM D7028-07. The DMA analyses were performed in temperature scan mode ranging from 30 to 130 °C at a constant oscillation frequency of 1.0 Hz with a heating rate of 5.0 °C/min and three repetitions for each specimen.

Using a Perkin-Elmer 8000 TGA instrument, thermogravimetric analysis (TGA) was used to examine the thermal degradation behavior of the nanocomposites with a heating rate of 10 °C/min in a nitrogen environment.

The two-probe method was utilized to measure the electrical conductivity using an OGSM 830B model multimeter according to ASTM D4496-13. Resistance, resistivity, and conductivity values were calculated using Eq. (1), Eq. (2), and Eq. (3), respectively [5].

$$\mathrm{R} = \frac{\mathrm{V}}{\mathrm{I}} \tag{1}$$

$$\rho = \frac{(\mathrm{R}\times\mathrm{A})}{\mathrm{l}} \tag{2}$$

$$\sigma = \frac{1}{\rho} \tag{3}$$

Here, ρ is resistivity, R is resistance (ohms), V is electric potential difference (voltage), A is cross-sectional area (cm^2), l is contact length (cm), and σ is electrical conductivity (S/cm) [5].

A vector network analyzer (ANRITSU VNA MS2028C) and two WR-90 waveguides were used for EMSE measurement at X-band (8.2-12.4 GHz) (Figure 1). Using a sample holder, samples of $10\times19\times3\ mm^3$ prepared according to IEEE declaration and ASTM-D 4935 were centralized between the waveguides. Scattering parameters, reflection coefficient (S11), and transmission coefficient (S21) were measured [5, 6, 19]. The total EMSE (SETotal) can be found from the sum of SER (SE reflectance), SEA (SE absorbance), and SEM (SE multiple internal reflections) following Eq. (4) [19, 20]:

$$SE_{Total}\ (dB) = SE_R + SE_A + SE_M = 10\log(\frac{P_t}{P_i}) \tag{4}$$

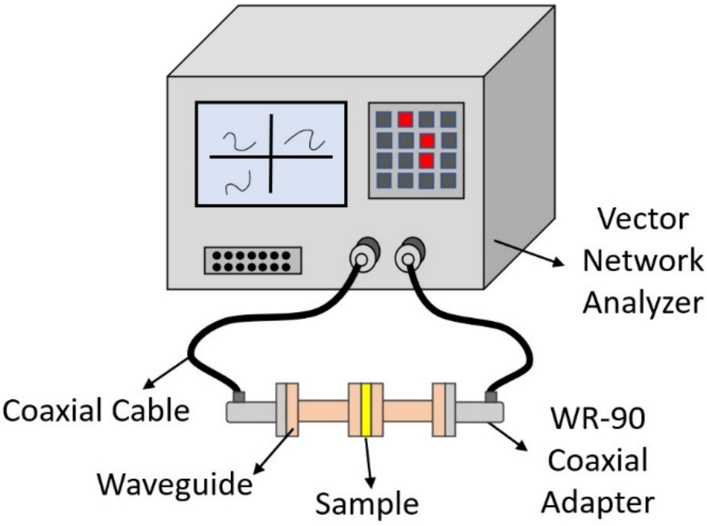

Figure 1. Electromagnetic Shielding (EMSE) Measurement Setup

Here, P_i is the power of the incident, and P_t is the power of the transmitted EM waves. When SE_{Total} is greater than ±10 dB, SE_M can be neglected. In terms of S parameters, the SE_A (dB), SE_T (dB), and SE_R (dB) parameters can be obtained using the following equations Eq. (5), (6), and (7) [20]:

$$SE_A(dB) = 10\log(\frac{1-{S_{11}}^2}{{S_{12}}^2}) = 10\log(\frac{1-{S_{22}}^2}{{S_{21}}^2}) = 10\log(\frac{1-R}{T}) \tag{5}$$

$$SE_T(dB) = 10\log(\frac{1}{{S_{12}}^2}) = 10\log(\frac{1}{{S_{21}}^2}) = 10\log(\frac{1}{T}) \tag{6}$$

$$SE_R(dB) = 10\log(\frac{1}{1-{S_{11}}^2}) = 10\log\left(\frac{1}{1-{S_{22}}^2}\right) = 10\log\left(\frac{1}{1-R}\right) \tag{7}$$

Here, *A* is absorbance, *T* is transmittance, and *R* is reflectance. The sum of reflectance *(R)*, transmittance *(T)*, and absorbance *(A)* is always equal to 1 [21, 22].

III. RESULTS AND DISCUSSIONS

3.1 DMA Analysis

DMA analyses were performed to investigate the viscoelastic behavior of MWCNTs-reinforced SAN/LDPE nanocomposites. The maximum energy stored in a material for one oscillation cycle is the storage modulus (E′). It also provides information about the stiffness-temperature relationship and the load-bearing capacity of the material. The storage modulus–temperature curve of SAN/LDPE/MWCNT composites at different frequency levels is presented in Figure 2. As the temperature rises, the modulus values drop. The reduction in E′ is associated with softening of the thermoplastic matrix at higher temperatures [19]. Pure SAN (MB0) polymer exhibited the highest storage modulus at room temperature with 2.54 GPa. The storage modulus of the MB25 nanocomposite exhibited a 22.4% decrease compared to the pure SAN sample and was determined to be 1.97 GPa. The storage modulus decreased as the LDPE/MWCNT ratio added to the pure SAN increased. The storage modulus of the MB50 sample was measured as 1.24 GPa with a 50% decrease, and the storage modulus of the MB75 sample was measured as 0.67 GPa with a 73.6% decrease. The storage modulus of the MB100 sample is 0.43 GPa. According to the mixing rule, the storage modulus values obtained coincide with the theoretical calculations.

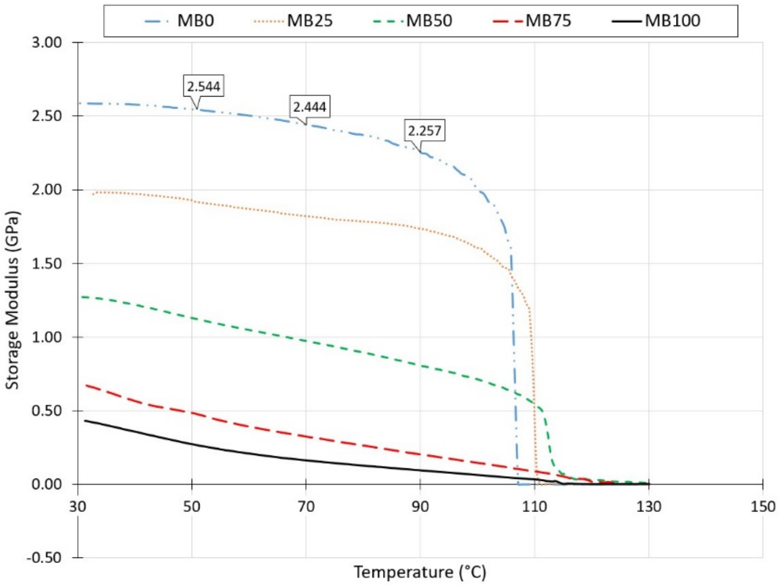

Figure 2. Storage modulus obtained from DMA analysis

DMA analysis results are given comparatively in Table 2. When the glass transition temperatures are compared, the glass transition temperature of the pure SAN sample was determined to be 107 °C. As the amount of LDPE/MWCNT nanocomposite added to the MB0 sample increased, the glass transition temperature increased and reached 130 °C for the MB50 sample. The interaction between the polymer chains and the MWCNTs reinforcements restricted the polymer chains' mobility, which was the main reason for this phenomenon [3].

Interestingly, the glass transition temperatures of the MB75 and MB100 samples were similar to those of the MB50 sample, and it was determined that increasing the ratio of MWCNTs above 10 wt% did not increase the thermal stability.

Table 1. Comparison of storage modulus and glass transition temperature values obtained from DMA analysis

Sample	**Tg** *(°C)*	**Difference** *(%)*	**S. Modulus** *(GPa)*	**Difference** *(%)*
MB0	107	-	2.54	-
MB25	111	3.73	1.97	22.44
MB50	130	17.11	1.24	37.05
MB75	130	0	0.67	45.96
MB100	130	0	0.43	35.82

3.2 TGA Analysis

Figure 3 shows the results of the TGA analysis of MB0 and MB100 samples. The degradation of LDPE in sample MB100 started at 450 °C and ended at 530 °C. The mass remaining after thermal degradation in the MB100 sample, which contains 20% MWCNTs, proves the presence of carbon nanotubes (Figure 3a).

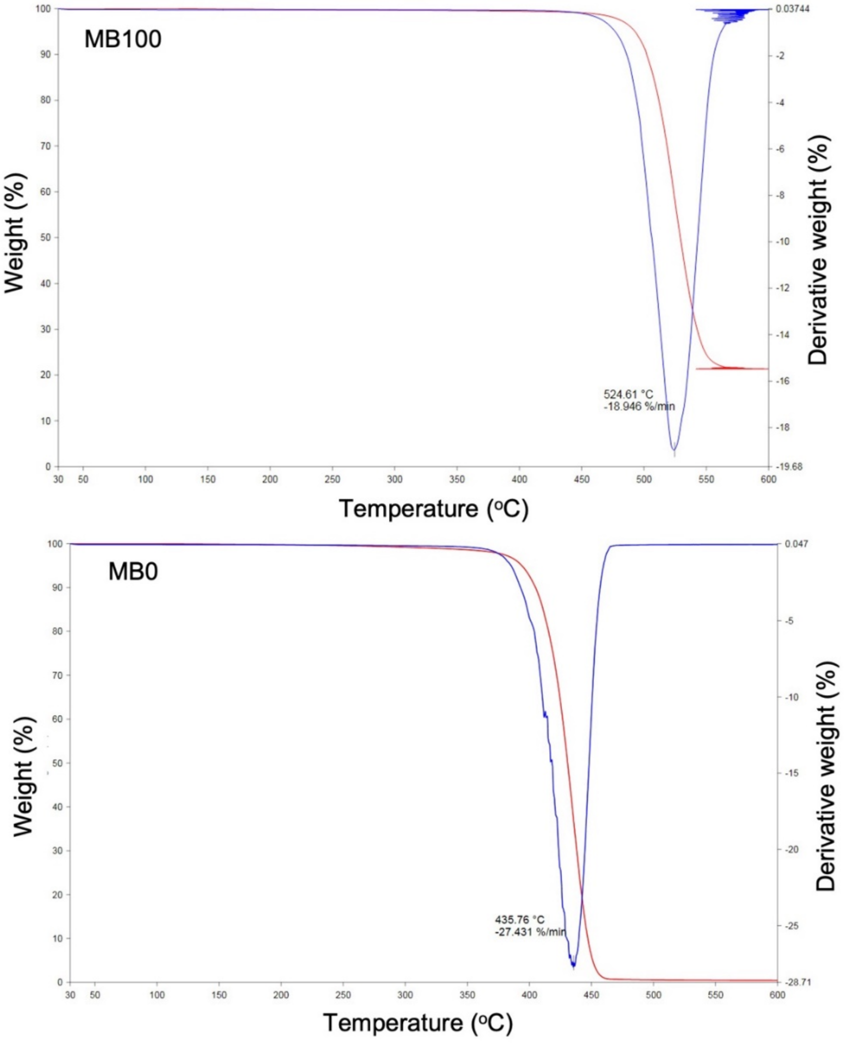

Figure 1. TGA analysis results **(a)** MB100, (b) MB0

The TGA curve of the MB0 (pure SAN) sample is shown in Figure 3b. Degradation started at 370 °C and ended at 530 °C. The conversion of aromatics starts at 370 °C and continues until monomeric acrylonitrile starts to appear at 405 °C. The C-H vibrations of aromatic species are much stronger than the nitrile vibration. Both aliphatic and aromatic -H vibrations are accompanied by weak nitrile absorption. This continues until the degradation is complete [23].

3.3 Electrical Conductivity and EMSE

Electrical conductivity and resistivity test results are given in Figure 5. The produced materials present various conductivity (and conversely resistivity) values according to the doping ratios and mixing parameters. As expected, the MB0 sample shows completely insulating properties with infinitely large resistivity and zero conductivity values. On the other hand, SAN/LDPE/MWCNT nanocomposites show a balanced property with varying amounts of carbon nanotubes. Accordingly, the resistance values of MB25, MB50, MB75, and MB100 samples are 3.48×10^5, 858, 134, and 67 Ωcm, respectively. In addition, the conductivity values of MB25, MB50, MB75 and MB100 samples are 0.3×10^{-6}, 0.11×10^{-3}, 0.74×10^{-3} and 0.15×10^{-2} S/cm, respectively. The graphs show that increasing the MB ratio decreases resistivity and, conversely, increases conductivity due to the growing conductive particle chain [11, 19, 24].

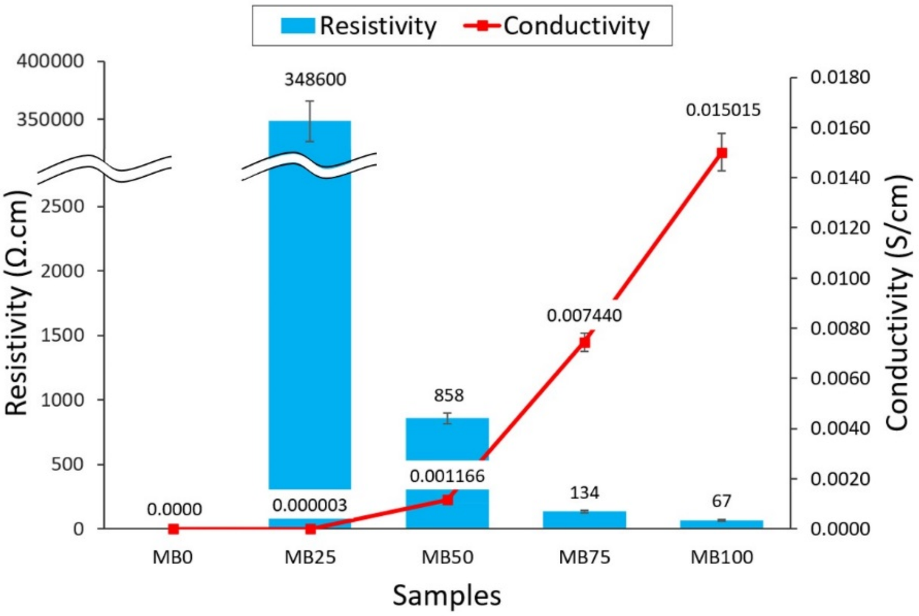

Figure 4. Electrical conductivity and resistivity results

As the MB content increases from 25 to 100 wt.%, the CNT content rises, and the SAN content decreases compared to the total sample percentage. Thus, the trend of reducing resistivity (increasing conductivity) is accelerated. Conductivity increases slightly between 0% and 50% ratio levels, while a significant increase occurs between MB50 and MB100 samples. Therefore, 50% ratio levels can be referred to as the percolation threshold level to change the conductivity characteristic of the composites [5, 25]. The rate of increase in conductivity was calculated as 1000 times from MB25 to MB50 and 10 times from MB50 to MB100. Increasing the amount of CNT per unit volume promotes the improvement of electron charge transport in the material [19, 25, 26]. The obtained gain proves that electron charge transport occurs through the composite's extensive conductive network formed by CNTs. Thus, the required minimum conductivity level, a prerequisite for electromagnetic shielding, is achieved.

EMSE results as a function of frequency are presented in Figure 5. Sample MB0, i.e., pure SAN, gives an EMSE value of -6.08 dB. This value is normal as the material is intrinsically insulating, and the measurement detected no conductivity. The other samples show similar EMSE characteristics; all samples except sample MB25 reach the commercially required -30 dB EMSE values [19, 27, 28]. The calculated EMSE values of samples MB25, MB50, MB75, and MB100 are -26.44, -35.52, -37.17, and -36.99 dB, respectively. EMSE values increased with the addition of MB due to increased electrical conductivity. The improvement rates for MB25, MB50, MB75 and MB100 were 334%, 484%, 581% and 506% respectively compared to MB0. The material's electrical conductivity is crucial for attenuating EM radiation [19, 29].

Increasing the MB ratio promotes the formation of electrically conductive chains, and high conductivity improves the impedance mismatch between the material surface and air [3, 5, 29]. As mentioned in the conductivity results section, the electrical conductivity increases and the EMSE increases when the MB ratio approaches the threshold level of 50%. As stated in the literature, an electrical conductivity level of about 0.01 S/cm is sufficient as a potential application for EMI shielding [19]. This is supported by the fact that MB50, MB75, and MB100 samples exceed the commercially required EMI shielding level.

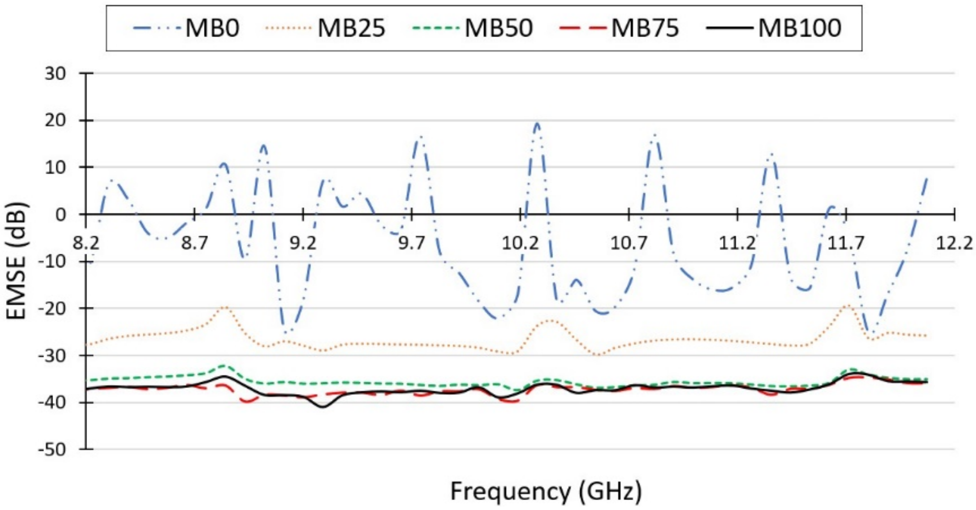

Figure 5. EMSE Results of SAN/LDPE/MWCNT nanocomposites

Figure 6 represents the obtained SEA and SER values of the samples, revealing the primary mechanism of the EM shielding of the produced material. The SEA curves show a similar trend with the obtained EMSE responses. While absorbance curves are clustered around -35 dB, reflectance curves are on the zero line. The SEA and SER curves of the MB0 sample were positioned around zero since the total EMSE value was calculated very low. The SEA and SER values of the MB0 sample were calculated as 0.19 and -6.28 dB, respectively. It is thought that the shiny outer surface of the material causes the reflection.

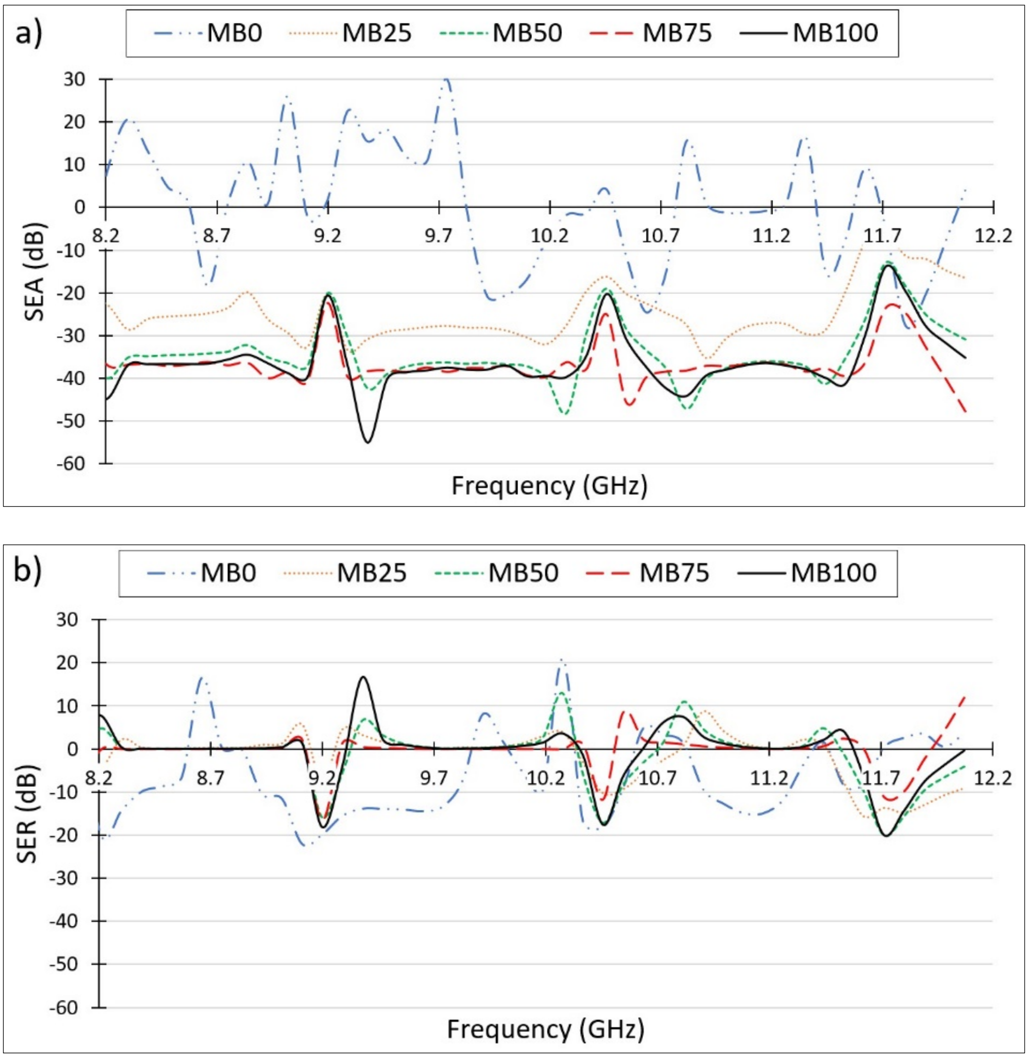

Figure 6.a) SEA, b) SER results of the samples

The SEA values were calculated as -24.48, -34.11, -36.75, and -36.23 dB for samples MB25, MB50, MB75, and MB100 respectively. According to the findings, the percentage of the SEA values compared to the total EMSE was calculated as 92.4, 95.9, 98.8, and 97.9% for samples MB25, MB50, MB75, and MB100, respectively. The SER values were found as -1.96, -1.41, -0.41, and -0.75 dB for samples MB25, MB50, MB75, and MB100, respectively. The calculated values reveal that more than 95% of the shielding performance is due to the absorption mechanism. While the electromagnetic waves are absorbed, they follow the conductive networks inside the material, disappearing into low-value heat [19, 20, 24]. The structure's conductive network size directly affects the absorption capacity. Findings show the manufactured composite can be an excellent alternative to absorbance-dominated EMI materials, primarily required in some sectors such as aviation, defense, etc.

The study aims to add an EMI shielding ability to a permeable structural thermoplastic polymer without complicating its production process. The mixing of MWCNT/LDPE masterbatch with SAN leads to obtaining a structural thermoplastic blend with an excellent EMI shielding performance without drastic changes in its processability, according to the DMA analysis [11, 17, 19].

IV. CONCLUSION

In this study, carbon nanotube-reinforced composites containing two different thermoplastic matrix phases (SAN and LDPE) were fabricated using product engineering processes. The thermo-mechanical and electromagnetic shielding properties of the nanocomposites were investigated. As the LDPE/MWCNT ratio added to pure SAN increased, the storage modulus decreased as expected, and the storage modulus for the MB50 sample was determined as 1.24 GPa with a 50% decrease. The percolation threshold for the two-phase thermoplastic composite was obtained for the MB50 sample containing 10 wt.% carbon nanotubes. In addition, the MB75 sample reaches an EMSE value of 37 dB, offering the potential to be used in commercial applications as shielding cases or structural plate elements. Finally, the primary strategy of the study, combining dilution and mixing production methods, was successfully implemented according to the findings. MWCNT added to LDPE in large amounts could be dispersed in the SAN phase, enabling the material to gain electromagnetic shielding ability, primarily by increasing electrical conductivity.

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