

MXENE-REINFORCED POLYMER NANOCOMPOSITES FOR FLEXIBLE WEARABLE SENSORS AND ELECTROMAGNETIC INTERFERENCE SHIELDING APPLICATIONS

Muhammad Danyal Zarin^{*1}, Muhammad Nabeel Sharif², Muhammad Aamir Riaz³, Li Huajing⁴, Gulshan Jabeen⁵, Ayesha Akram⁶, Hassan Imran Afridi⁷, Abdul Rehman⁸

^{*1}School of Intelligent Materials and Future Energy, Anhui Normal University, China

²Department of Physics, University of Poonch Rawalakot, Rawalakot, 12350, Pakistan

³Institute of Physics, Bahauddin Zakariya University, Multan, Pakistan

⁴School of Chemistry and Molecular Sciences, Anhui Normal University China

⁵Department of Chemistry, Bowling Green State University, United States

⁶Department of Chemistry, Thal University Bhakkar, Pakistan

⁷National Centre of Excellence in Analytical Chemistry, University of Sindh, Jamshoro, Pakistan postal address is 76080

⁸Department of Chemistry, University of Agriculture Faisalabad, Punjab, Pakistan

^{*1}danyalzarin1@gmail.com, ²mnabeelsharif377@gmail.com, ³aamirriaz933@gmail.com,

⁴2026209@ahnu.edu.cn, ⁵jabeeng@bgsu.edu, ⁶ayasha.akram.chem@gmail.com,

⁷hassan.afridi@usindh.edu.pk, ⁸470abdulrehman@gmail.com

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Corresponding Author: *

Muhammad Danyal Zarin

Abstract

Multifunctional nanocomposites made of Ti₃C₂T_x MXene and thermoplastic polyurethane (TPU) are presented in this research for applications as flexible strain sensors and electromagnetic interference (EMI) shielding material. The synthesis process of Ti₃C₂T_x MXene involves selective etching of Ti₃AlC₂ MAX phase through LiF/HCl, and subsequent delamination. Nanocomposite films containing 0, 2, 5, 10, and 15 wt% MXene content were prepared and analyzed. XRD results confirmed the formation of MXene along with increased interlayer space, while FTIR results showed hydrogen bonding between surface groups of MXene and the TPU backbone. The morphology analysis using SEM and TEM showed accordion-type layered structures and ultrathin nanosheets. Thermal stability was enhanced with an increase in degradation temperature from 287 °C to 315 °C. Electric conductivity varied from 1.8×10^{-10} S/m in the case of pure TPU to 11.6 S/m in the case of 15 wt% MXene-TPU, with percolation from 5 to 10 wt%. The optimal ratio of MXene-10 yielded a gauge factor of 12.5, response/recovery times of 72/86 ms, and 94.3% response retention for 5000 cycles. Besides, the sensor could monitor human activities. The values of EMI shielding effectiveness varied within 29.6–33.8 dB in the X band and increased from 18.9 to 37.4 dB in the same range of frequencies when thickness changed from 50 to 200 μ m. At the same time, 92.1% of shielding efficiency was retained after 1000 bending cycles. The shielding effect was

based on conductive loss, interfacial polarization, and multiple internal reflections.

1 Introduction

Due to the fast development of flexible and wearable electronics, there is an immediate necessity for the design of new multifunctional materials which would be able to deliver flexibility, strain sensing, and EMI shielding functions at once[1]. In light of the increasing integration of the electronic devices into our daily routines in various applications such as health care monitoring, human-computer interaction, soft robots, and portable communications, it became evident that there was a necessity for the development of materials which could withstand the deformations and retain good electrical properties at the same time[2]. Traditional rigid electronic materials, namely, metals and inorganic semiconductors, due to their mechanical rigidity, fatigue, and poor capability to conform to curved surfaces, were unable to meet these requirements[3].

In this regard, among various two-dimensional (2D) nanomaterials studied for these types of applications, MXenes specifically titanium carbide ($\text{Ti}_3\text{C}_2\text{T}_x$) has been found to be very promising due to their interesting characteristics including metallic conductivity, hydrophilic nature, high aspect ratio, and mechanical stability[4]. These materials were first reported in the year 2011 and since then, they have gained wide popularity for application areas as diverse as energy storage, catalysis, sensing, electromagnetic interference shielding, etc[5]. In general, the structure of MXenes can be represented by the chemical formula of $\text{M}_{n+1}\text{X}_n\text{T}_x$, where M represents an early transition metal, X represents carbon/nitrogen, and T_x represents different termination groups like $-\text{OH}$, $-\text{O}$, and $-\text{F}$, with $\text{Ti}_3\text{C}_2\text{T}_x$ being the most investigated material within this family. Synthesis of $\text{Ti}_3\text{C}_2\text{T}_x$ is usually done through the etching of aluminum layer from Ti_3AlC_2 MAX phase precursor using acidic solution containing fluorides, resulting in formation of ultrathin nanosheets. These nanosheets have high electrical conductivity (up to $\sim 10,000 \text{ S cm}^{-1}$), good

electromagnetic wave absorption capability, and functionalized surfaces[6].

TPU is commonly used as the flexible polymer matrix for nanocomposites due to its high elasticity, tensile strength, abrasion resistance, and biocompatibility[7]. The segmental nature of TPU containing alternating hard and soft segments allows for great stretchability of the polymer chain with maintaining the strength, which makes it an appropriate material for wearable electronics. Combining TPU with MXenes may lead to synergetic effects where MXene network would bring electrical conductivity and electromagnetic shielding capabilities, while TPU will provide flexibility and mechanical strength to the material[8]. However, obtaining such desired effects involves proper dispersion of MXene sheets in TPU matrix and development of the conductive network. In spite of the increasing amount of work dedicated to the study of MXene/polymer nanocomposites, several crucial knowledge areas are yet to be explored, which prevent further incorporation of these materials into functional wearable electronics[9]. Firstly, although some publications describe either the sensing ability or the EMI shielding capability of the composite, systematic analysis connecting the loading ratio of MXenes to the sensitivity of the material as well as its ability to shield EM waves has yet to be performed. There is an insufficient amount of knowledge on how the properties of the conductive network influence the ability of such systems to perform strain sensing and EMI shielding[10]. Secondly, the influence of the thickness of the material on the effectiveness of EMI shielding has not been studied enough yet. In addition, there is little information on the interaction of the thickness of the material and the ratio of MXenes with each other and how they affect the shielding capacity. It is a very important issue since there are usually certain restrictions on the thickness of the shielding layer in practical applications[11]. Finally, the resistance of the sensors and shielding materials to mechanical

wear has not been studied thoroughly. Fourth, the interfacial interactions between the MXene surface functionalization and the TPU functional groups, along with the effects on stress transfer, thermal stability, and electromechanical properties, need further exploration in order to provide guidelines for the development of high-performing nanocomposites. Lastly, combining both strain sensing and EMI shielding properties within a single flexible material platform and performing both functions simultaneously is still an area that needs to be explored further because of the growing demand of such multifunctional materials in next-generation wearables[12].

This study fills these important gaps by designing and characterizing MXene/TPU nanocomposites with systematically controlled $\text{Ti}_3\text{C}_2\text{T}_x$ MXene contents (0, 2, 5, 10, and 15 wt%) for strain sensing and EMI shielding applications. The aims of the current research include: (1) Synthesis of high-purity $\text{Ti}_3\text{C}_2\text{T}_x$ MXene by using optimized selective etching and delamination methods, and fabrication of MXene/TPU nanocomposites with homogeneous nanofiller dispersion; (2) Study of the structural, morphological, thermal and mechanical characteristics of the nanocomposites with respect to their MXene content, especially the interfacial interactions of the terminations on the surface of MXene with TPU; (3) Study of the electrical conductivity and percolation behavior of the nanocomposites to find the correlation between their MXene content and the formation of conductive network; (4) Study of the strain sensing ability of the nanocomposites in terms of sensitivity, gauge factor, response/recovery time, linearity, and cyclic stability (5) to evaluate the EMI shielding capability of the nanocomposites within the X-band frequency spectrum (8.2-12.4 GHz), focusing on the influence of MXene content, nanocomposite film thickness, and mechanical stretching on EMI shielding ability; and (6) to understand the basic structure-property relationship that governs both the strain sensing capability and EMI shielding property of the nanocomposites. With this thorough study, we hope to confirm that MXene/TPU nanocomposites have the potential to serve as

suitable candidates for the next generation of flexible wearables.

2 Materials and Methods

2.1 Materials

The powder of Ti_3AlC_2 MAX phase (particles smaller than 40 μm , purity $\geq 98\%$) served as a precursor for $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. LiF ($\geq 99\%$), HCl (37%), ethanol, and deionized water were employed in the synthesis and purification of MXenes. TPU pellets were selected as a polymer matrix material due to its excellent flexibility, mechanical strength, elasticity, and applicability for wearable electronics. DMF ($\geq 99.5\%$) was chosen as a polymer solvent. Silver paste and copper tape were used to provide electrical contact. All the materials were purchased commercially and used as supplied. Five nanocomposites with varying MXene content were synthesized: 0, 2, 5, 10, and 15 wt% of MXenes were added into the TPU matrix and named TPU, MXene-2, MXene-5, MXene-10, and MXene-15, correspondingly. MXene content was defined by the total dry mass of the MXene-polymer composite.

2.2 Synthesis of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene

$\text{Ti}_3\text{C}_2\text{T}_x$ MXene was prepared from Ti_3AlC_2 via a selective etching method. In brief, 1.0 g of Ti_3AlC_2 was gradually added into an etching mixture consisting of 1.0 g LiF dissolved in 20 mL of 9 M HCl in a magnetic stirrer under 35 °C for 24 h. During this process, selective etching of Al layers from the MAX phase took place[13]. After etching, the suspension was purified by repeated washing with deionized water and followed by centrifugation at 3500 rpm for 5 min cycles until the pH of the supernatant reached around 6. The obtained precipitates were then dispersed in deionized water with the concentration of about 5 mg mL^{-1} . Next, the suspension was sonicated in an ice bath with nitrogen gas flow for 30 min in order to facilitate exfoliation without increasing oxidation excessively. The suspension was centrifuged again at 3500 rpm for 30 min and the dark supernatant with the delaminated $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets was collected. The concentration of

this suspension was diluted to about 2 mg mL^{-1} for further fabrication.

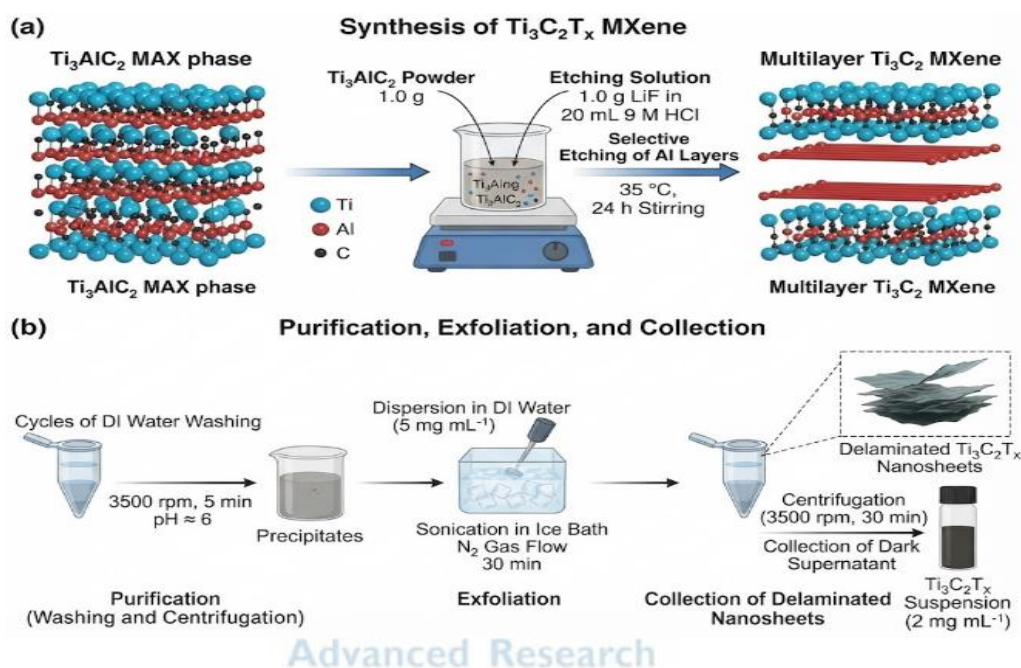


Figure 1: Schematic representation of the synthesis process of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene nanosheets, which includes (a) selective etching of Al in Ti_3AlC_2 by using LiF/9 M HCl at 35°C for 24 hours and (b) washing, centrifugation, N_2 -assisted ultrasonication, exfoliation, and eventually purification of $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets dispersed at 2 mg mL^{-1} concentration.

2.3 Fabrication of MXene/TPU Nanocomposites

In the current study, MXene/TPU nanocomposite films were prepared through a solution-based blending and casting technique. First, the TPU pellets were dissolved in DMF at 10 wt% concentration and stirring at 60°C for 4 hours to get a homogeneous solution of polymers. Specified amount of MXene dispersion was added to the TPU solution to prepare MXene/TPU nanocomposites with 2, 5, 10, and 15 wt% content of MXenes[14].

These mixtures were vigorously stirred at 800 rpm speed for 2 hours and bath sonicated for 30 minutes to promote MXene dispersion in the solution. These solutions were homogenized via high-speed homogenizer at 5000 rpm speed for 10 minutes. The homogenized suspensions were cast

into glass Petri dishes and dried in oven at 60°C for 12 hours to remove most of the solvent. Finally, the films were vacuum-dried at 80°C for 12 hours to remove any remaining DMF. The films prepared had a desired thickness of about 100–150 μm . The pristine TPU film was prepared under the same processing conditions without MXenes and used as the control sample.

2.4 Structural and Chemical Characterization

2.4.1 X-ray Diffraction

X-ray diffraction (XRD) analysis was done using a Cu $\text{K}\alpha$ radiation source ($\lambda = 1.5406 \text{ \AA}$) within an angular range of 5 to 80° at 0.02° intervals. XRD spectra were obtained for Ti_3AlC_2 , synthesized $\text{Ti}_3\text{C}_2\text{T}_x$, pure TPU and MXene/TPU nanocomposites. Change in the peak position and

broadening of MXene (002) plane was used to examine the alteration in layer spacing and structural arrangement after etching and delamination.

2.4.2 Fourier-Transform Infrared Spectroscopy

The FTIR spectra of samples were obtained in the range from 400 to 4000 cm^{-1} at the resolution of 4 cm^{-1} and in 32 scans for each sample. The obtained spectra were used for the determination of typical surface groups in $\text{Ti}_3\text{C}_2\text{T}_x$: -OH, -O, and -F, along with TPU characteristic functional groups. The analysis of characteristic bands after adding MXene was performed.

2.4.3 Raman Spectroscopy

Raman spectroscopic data was recorded from a 532 nm laser source between the wavenumbers of 100-2000 cm^{-1} . Structural modifications in addition to vibrational characteristics of $\text{Ti}_3\text{C}_2\text{T}_x$, along with the effect of adding polymers into the nanocomposite system, were studied through Raman Spectroscopy.

2.5 Morphological Characterization

2.5.1 SEM

The surface morphology and microstructure of the synthesized MXene were analyzed through scanning electron microscopy (SEM). A small amount of the MXene powder was dispersed evenly on the conductive carbon tape fixed on the SEM stub. The powder sample was carefully pressed so that it adheres well and, if needed, coated with a conducting layer before analysis. Micrographs were captured through SEM at the proper accelerating voltage and magnification for analyzing the layered structure and degree of exfoliation of the synthesized MXene and TEM image displaying the ultrathin exfoliated few-layer MXene nanosheets.

2.6 Thermal Characterization

The thermogravimetric analysis (TGA) was done under nitrogen atmosphere at a temperature range

$$\frac{\Delta R}{R_0} = \frac{R - R_0}{R_0}$$

of 30-800 $^{\circ}\text{C}$ with a heating rate of 10 $^{\circ}\text{C min}^{-1}$. The differential scanning calorimetry (DSC) was carried out under nitrogen atmosphere at a temperature range of -50-250 $^{\circ}\text{C}$ at a heating/cooling rate of 10 $^{\circ}\text{C min}^{-1}$. Thermal studies were performed on both pristine TPU and MXene/TPU nanocomposites.

2.7 Mechanical Properties

The mechanical behavior was determined using a universal testing machine, which was provided with a 100 N load cell. Rectangular test samples of about 50 × 10 mm were tested under a crosshead speed of 50 mm min^{-1} . Five or more independent test samples ($n \geq 5$) were subjected to tests for each sample composition[15]. Tensile strength, Young's modulus, % elongation, and toughness were calculated from the stress-strain curves. Additionally, the flexibility of the films was investigated through repeated bending under bending radii of about 5, 10, and 20 mm.

2.8 Electrical Conductivity

The electrical resistance of the nanocomposite films was measured using a four-point probe system.

Electrical conductivity was calculated using:

$$\sigma = \frac{L}{RA}$$

where σ is the electrical conductivity (S m^{-1}), L is the electrode spacing, R is the measured resistance, and A is the cross-sectional area. The electrical conductivity was measured at various MXene concentrations to establish the point where conductive percolation occurs.

2.9 Fabrication of Flexible Strain Sensors

The MXene/TPU films were then prepared in the form of rectangular strips having dimensions of about 30 × 5 mm. These strips had copper tape fixed at both ends of the film using silver conductive paste, forming the electrodes for resistance measurement.

Relative resistance variation was determined by:

where R_0 represents the initial resistance and R represents the resistance under applied strain.

$$GF = \frac{\Delta R/R_0}{\varepsilon}$$

where ε represents the applied strain.

The sensitivity, linearity, working range, response time, recovery time, and detection limit were obtained from the resistance-strain relationship.

2.11 Cyclic and Durability Testing

The optimized sensor was cycled 5000 times with a 20% tensile deformation while monitoring the resistance signal to measure the stability of the signal, recovery of the signal baseline, hysteresis, durability of the mechanical part, and resistance drift. The retention of the sensing performance was measured based on the comparison between the sensing before and after the cycle test.

2.12 Wearable Motion Monitoring

The practical application of this optimized sensor is demonstrated by its mounting on some body parts via adhesive tapes, and simultaneous recording of electrical signals upon flexion of fingers, flexion of wrist, elbow movement, flexion of knee joint, hand clenching, and walking at a sampling rate of 100 Hz. The correlation between the amplitude of resistance change and the level of deformation was evaluated.

2.13 Electromagnetic Interference Shielding Measurements

EMI shielding effectiveness testing was done with a calibrated vector network analyzer (VNA) within the X-Band frequency range from 8.2 to 12.4 GHz. The nanocomposite films were prepared in sizes suitable for the waveguide measurements setup, and the thickness of films was determined with a digital micrometer. The scattering parameters S_{11} and S_{21} were measured for each specimen, which represent reflected and transmitted EM radiation, correspondingly. The experimentally obtained

2.10 Strain-Sensing Measurements

The sensor was strained through tensile strain tests of 1, 2, 5, 10, 20, 30, and 50%, and resistance variations were noted at the same time.

The value for gauge factor was calculated from:

scattering parameters were transformed into reflection (R) and transmission (T) coefficients. The total EMI shielding effectiveness was calculated using:

$$SE_T = -10\log_{10}(T)$$

The reflection contribution was determined from:

$$SE_R = -10\log_{10}(1 - R)$$

The absorption contribution was calculated using:

$$SE_A = -10\log_{10}\left(\frac{T}{1 - R}\right)$$

The multiple-reflection contribution was evaluated where relevant.

2.14 Effect of MXene Loading on EMI Shielding

EMI shielding effectiveness of pristine TPU and MXene/TPU composites with varying amounts of MXene, including 2, 5, 10, and 15 wt%, was assessed in the X-band. Influence of MXene content on shielding efficacy was studied in connection with electrical conductivity, conductive network formation, interfacial polarization, dielectric losses, reflection, and absorption. On the basis of a combination of mechanical flexibility, sensing capability, and EMI shielding effectiveness, the optimum composition of the composite material was determined[16].

2.15 Thickness-Dependent EMI Shielding

In order to examine the effect of material thickness, optimized films of MXene/TPU with thicknesses around 50, 100, 150, and 200 μm were fabricated. The total SE was evaluated using identical experimental conditions. This thickness dependence was related to the probability of EM

interaction with the conductive network of MXene and the additional length of the path for the absorption process.

2.16 EMI Shielding under Mechanical Deformation

The developed nanocomposite underwent bending with a bending radius of 10 mm up to 1000 cycles. Before and after bending, the EMI shielding efficiency was evaluated using the following formula for the calculation of retention of the shielding effect:

$$SE_{\text{retention}} = \frac{SE_{\text{after}}}{SE_{\text{initial}}} \times 100$$

This experiment was used to determine whether repeated deformation caused significant disruption of the conductive network.

2.17 Mechanistic Analysis

The connection between the nanocomposite microstructure and their performance was established by linking their structural, morphological, electrical, mechanical, sensing, and EMI shielding properties. The sensing mechanism was explained in relation to the structural change in the conductive network of MXene nanosheets due to mechanical deformation. The changes in the tunneling distance and conductive paths between adjacent sheets lead to changes in resistance values under mechanical loading. The EMI shielding mechanism was described in relation to the reflection effect of the conductive MXene network, absorption by the conductive and dielectric losses, interface polarization effects at the MXene/TPU interfaces, and internal reflections.

2.18 Statistical Analysis

Quantitative experiments were carried out on a minimum of three samples for all cases, while mechanical tests were carried out on $n = 5$ samples for each composition. Data obtained were presented in terms of mean values $\pm$ SD. Differences between different samples were statistically analyzed by ANOVA test followed by

Tukey's test. The significance level for statistical difference was $p < 0.05$. Correlation analysis was carried out to explore possible correlations between MXene amount, electrical conductivity, mechanical behavior, and EMI shielding effectivity.

3 Results and discussion

3.1 Structural Characteristics of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and MXene/TPU Nanocomposites

The observed XRD profiles present sufficient data regarding the formation of Ti_3AlC_2 MAX-phase and its further conversion to $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and incorporation into TPU matrix. The initial Ti_3AlC_2 material shows a specific (002) diffraction peak at $2\theta \approx 9.6^\circ$ and additional diffraction peaks at high diffraction angles (for example, around 30° and 40°) typical for the MAX-phase crystal structure[17]. After etching of Al layers, the MAX-phase diffraction peaks decrease significantly, whereas a specific low-angle (002) peak appears at $2\theta \approx 6.8^\circ$. The decrease in the diffraction angle from about 9.6° to 6.8° is a result of the increase in the interlayer distance from about 9.2 Å in Ti_3AlC_2 to 13.0 Å in $\text{Ti}_3\text{C}_2\text{T}_x$, that indicates the removal of the Al layers and an increase in the MXene interlayer galleries. The increase in the interlayer distance can be attributed to the presence of water and surface groups incorporated into the structure of the resulting MXene layers. Following the incorporation of $\text{Ti}_3\text{C}_2\text{T}_x$ in the TPU matrix, the broad diffraction halo of TPU remained prominent at $2\theta \approx 20^\circ$, signifying that the semicrystalline/amorphous structure of the polymer was preserved. In contrast, the MXene-specific (002) reflection appeared in all nanocomposites and showed a gradual move to lower angles in correspondence with increasing amounts of MXene in the nanocomposites. More specifically, the (002) reflection peak shifted from $\sim 6.7^\circ$ for the nanocomposite containing 2 wt% MXene to 6.6° , 6.5° , and 6.4° for the nanocomposites containing 5, 10, and 15 wt% MXene, respectively. The calculated interplanar distance thus increased from ~ 13.2 Å to 13.4, 13.6, and 13.8 Å, respectively. This gradual increase in the MXene gallery is indicative of

structural disorder within the layers and may be attributed to the interaction between the MXene layers and the TPU chains. The intensity of the peaks due to the diffraction associated with MXene became increasingly prominent with an increase in the amount of MXene, as was expected due to the higher amount of crystalline MXene phase in the polymer matrix. There were no visible

reflections associated with Ti_3AlC_2 present in the nanocomposites, which again proved the efficiency of the process of removal of the Al-containing MAX phase during etching. The peak shifts, interlayer spacing, and preservation of the TPU diffraction halo clearly confirmed the successful integration of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene into the TPU matrix.

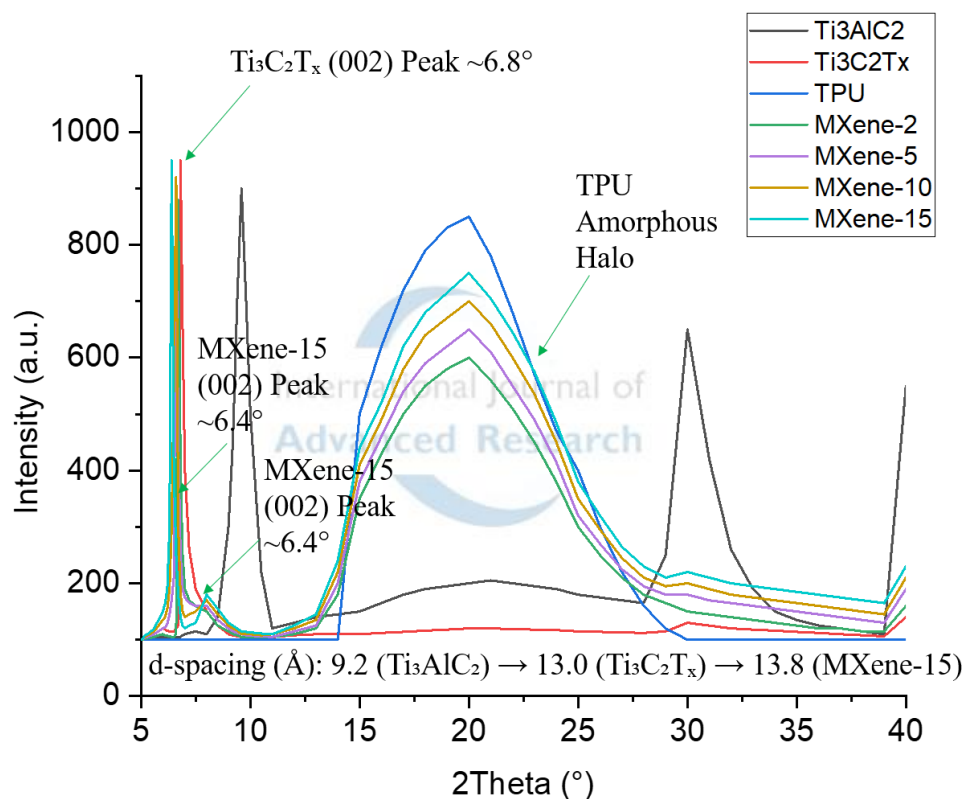


Figure 2: XRD spectra of Ti_3AlC_2 , $\text{Ti}_3\text{C}_2\text{T}_x$ MXene, TPU, and $\text{Ti}_3\text{C}_2\text{T}_x$ /TPU nanocomposites with 2 to 15 wt% of MXene, displaying inhibition of MAX phase reflections, (002) peak shift to lower angles, and increase in the distance between MXene layers in the polymer matrix.

3.2 FTIR Analysis and MXene-TPU Interactions

FTIR spectra confirm the presence of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene in the thermoplastic polyurethane (TPU) polymer and show non-covalent interfacial interactions between the two. The pristine TPU showed characteristic peaks related to the functional groups present in it, such as N-H stretch, aliphatic C-H stretch, carbonyl C=O stretch, and C-O/C-N vibrations. After

incorporating MXene into the polymer, the general spectra of TPU remained the same, which indicates that the basic structure of the polymer did not change.

A progressive increase in the broad absorption band near $\sim 3300\text{ cm}^{-1}$ region was noted with an increase in MXene content. The band is mainly attributed to surface hydroxyl (-OH) groups and reflects the hydrophilic surface terminations of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. Moreover, shifts of the

characteristic TPU absorption bands were noted after the inclusion of MXene. Specifically, the absorption peak of carbonyl stretching shifted from $\sim 1728 \text{ cm}^{-1}$ for pristine TPU to 1722 cm^{-1} for MXene-10. At the same time, the shift of the N-H stretching band occurred from ~ 3320 to 3312 cm^{-1} . The obtained shifts indicate changes in the local chemical environment of the polar functional groups of TPU and hydrogen-bonding interactions between oxygen and hydroxyl surface terminations of MXene and N-H, C=O groups of the TPU chains. Notably, there were no newly

formed strong absorption bands due to the formation of a specific covalent phase as a result of MXene addition. As a result, the observed changes in the spectrum could be related more to interfacial hydrogen bonding, dipole-dipole interaction, and other physical interactions than to the formation of covalent interactions. The latter should enhance the adhesion and stress transfer between the $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets and the TPU matrix, providing the improved properties of the obtained composites.

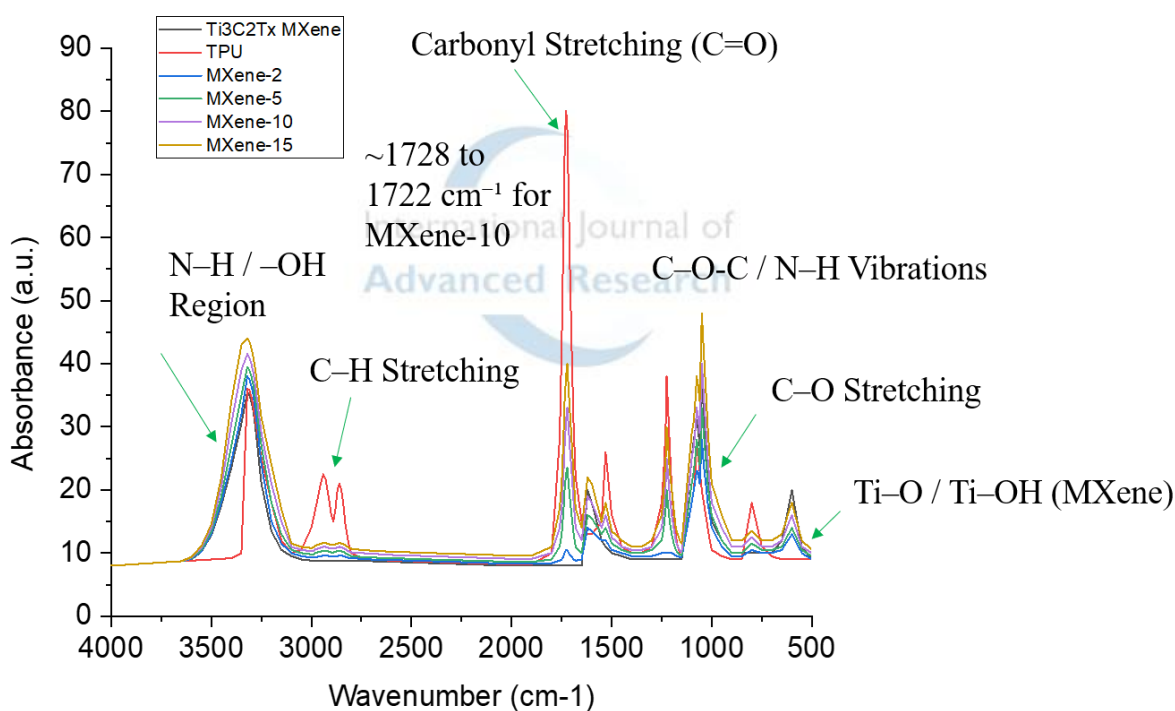


Figure 3: FTIR spectra of pure $\text{Ti}_3\text{C}_2\text{T}_x$ MXene, TPU, and $\text{Ti}_3\text{C}_2\text{T}_x$ /TPU nanocomposites containing 2-15 wt% MXene, which contain distinct functional groups and shifts of spectra caused by hydrogen bonding and other interactions at interfaces.

3.3 Scanning electron Microscopy (SEM)

Figure 4 shows some morphological characteristics of the MXene material that has been utilized in high-performance polymer nanocomposites. The multilayered structure of the MXene is evident from the scanning electron microscopy (SEM) in Figure 4a. It has an accordion type structure with an open microstructure between layers. This

unique structure is formed by chemically etching the MAX phase to create a multilayered structure of MXene. Also, the TEM image in Figure 4(b) shows that the bulk material was successfully exfoliated to ultra-thin and transparent 2D nanosheets of MXene. When incorporated in flexible polymer matrices, such high aspect ratio nanosheets are capable of providing a highly

conductive and densely inter-connected network. Such structure is vital for achieving a high sensitivity of piezoresistive effect in flexible wearable devices because any micromechanical deformation will change the conductive path between two flakes. At the same time, metallic

conductivity and high density of charge carriers in the MXene 2D nanosheets lead to multiple reflections and absorption of electromagnetic radiation. Thus, these materials are extremely suitable for EMI shielding.

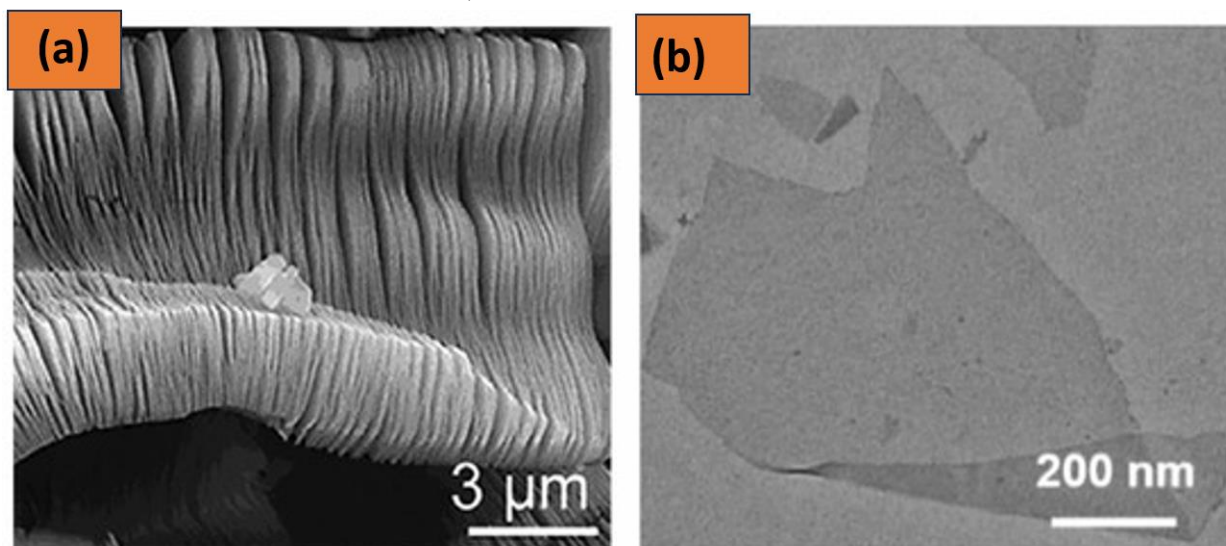


Figure 4: Structural description of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene: (a) SEM image displaying the typical multilayered folded structure of the etched MXene material, and (b) TEM image displaying the ultrathin exfoliated few-layer MXene nanosheets.

3.4 Thermal Stability of MXene/TPU Nanocomposites

Thermal stability of thermoplastic polyurethane (TPU) without MXene addition and TPU nanocomposites containing MXenes were studied through thermogravimetric analysis (TGA) as illustrated in Figure 5. It is obvious that the degradation curve of TGA illustrated in Figure 5a indicates single step primary degradation for all compositions. Importantly, the degradation profile gradually moves to higher temperature range as the amount of MXene increases from 0 to 15 wt%. This improved thermal stability is shown graphically in Figure 5b, whereby the onset degradation temperature gradually rises from about 287 °C for the pure TPU sample to 293, 300, 309, and 315 °C for the 2, 5, 10, and 15 wt% MXene/TPU nanocomposites, respectively. With the approximate 28 °C rise in the degradation

temperature of the nanocomposites with the maximum MXene content, there is an appreciable enhancement in the thermal stability of the material system. This is mainly due to the inherent thermal stability of MXene as well as its barrier effect in the polymer matrix. The high aspect ratio of the MXene nanosheets leads to longer diffusion paths of the degradation products and reduced mobility of the polymer chains, thus delaying the thermal decomposition. Moreover, the increasing residual mass at higher temperatures also signifies the influence of the thermally stable inorganic MXene phase. Therefore, the results clearly indicate the effective improvement in thermal stability by MXene incorporation into the TPU system, which is highly beneficial for its use in flexible wearable sensors and EMI shielding applications.

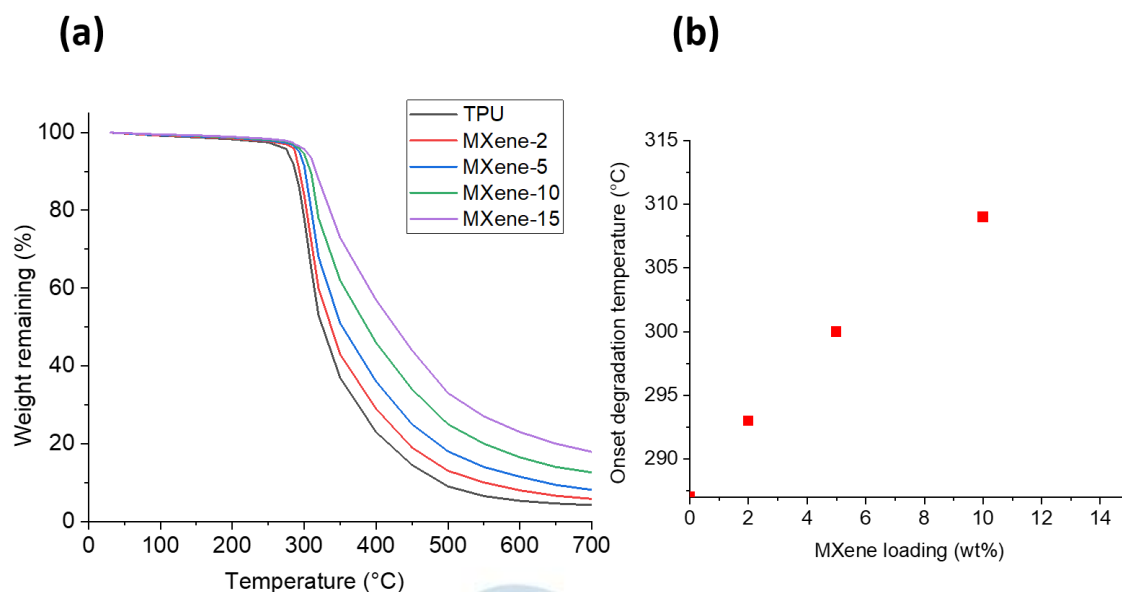


Figure 5: Thermogravimetric analysis (TGA) of pristine TPU and MXene-TPU nanocomposites: (a) TGA curves for mass loss as a function of temperature for various amounts of MXene (0-15 wt%), and (b) effect of MXene concentration on the onset temperature of thermal degradation.

3.5 Mechanical Properties

As seen in Figure 6, the mechanical properties of the MXene-reinforced TPU nanocomposites displayed a clear relationship with the MXene content. The Young's modulus value rose linearly from 9.8 MPa of pristine TPU up to 29.8 MPa of 15 wt% MXene/TPU nanocomposites (Figure 6a), indicating an approximately three-fold increment of the stiffness of nanocomposites. Such an improvement could be explained by the stiffness of MXene nanosheets and effective restriction of polymer chains movement inside the TPU matrix. At the same time, the rise of the modulus values indicates effective mechanical interaction between MXene and the TPU matrix. In the case of tensile strength (Figure 6b), a similar behavior was observed. Tensile strength increased from 18.6 MPa of pristine TPU to 21.4, 25.8, and 31.7 MPa with MXene content of 2, 5, and 10 wt%, respectively. The highest value of tensile strength of 31.7 MPa at 10 wt% MXene is equal to 70% improvement compared to pristine TPU. This improvement is mostly due to the effective stress

transfer from the TPU matrix to highly dispersed high-aspect ratio MXene nanosheets due to good interfacial interactions. With the addition of 15 wt% MXene, the tensile strength was reduced slightly to 29.4 MPa, indicating the onset of poor stress transfer efficiency with the increased amount of the filler. On the contrary, elongation at break was decreased significantly with the increase in the MXene concentration (Figure 6c), starting from 486% for neat TPU and reaching values of 452%, 414%, 362%, and 286% for 2, 5, 10, and 15 wt% MXene, respectively. Such decrease in ductility is associated with the increased rigidity of the composite and limited chain mobility of the TPU matrix. At higher concentrations of the filler, formation of aggregates of nanosheets can cause local stress concentration points and reduce the ability of polymer chains to move when undergoing tensile strain. Altogether, the strength-ductility trade-off becomes obvious, where 10 wt% MXene gives an optimum balance between tensile strength and mechanical reinforcement.

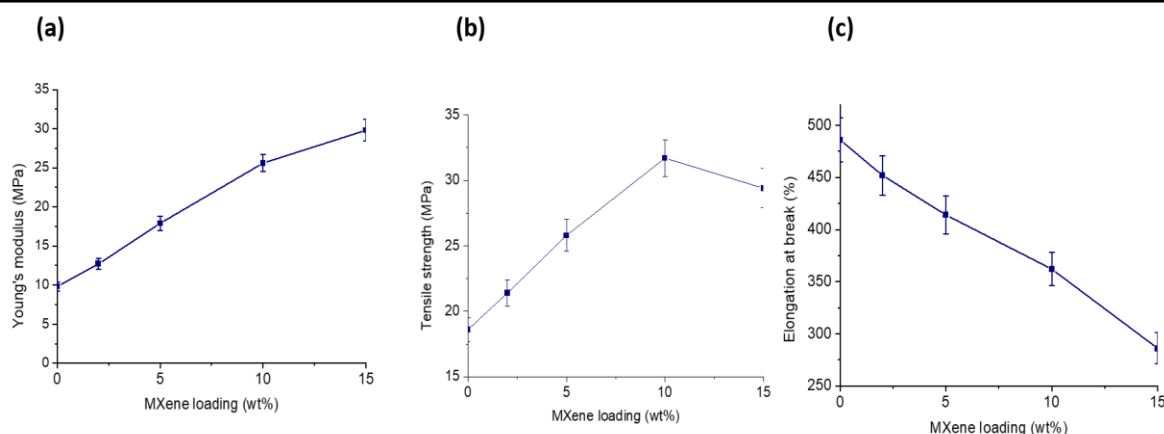


Figure 6: Properties of TPU and MXene/TPU nanocomposites as a function of MXene content: (a) Young's modulus, (b) tensile strength, and (c) elongation at break. The error bars represent the standard deviations for the replicates.

3.6 Electrical Conductivity and Percolation Behavior

Electrical conductivity of the TPU nanocomposites doped with the MXene revealed a clear dependency on the content of MXenes, which is typical for the development of conductive network in the polymer matrix. Initial pristine TPU possessed very low electrical conductivity of $1.8 \times 10^{-10} \text{ S m}^{-1}$, which confirms its mainly insulating character. Addition of 2 wt% MXenes led to a noticeable increase in conductivity to the level of $3.6 \times 10^{-5} \text{ S m}^{-1}$, which confirms that the first role of highly conductive MXene nanosheets was played in the process of charge transfer. The conductivity value increased to $8.9 \times 10^{-3} \text{ S m}^{-1}$ when the amount of MXene reached 5 wt%. The dramatic increase can be explained by the smaller distance between the nanosheets and a higher possibility of their electrical contact and tunneling. A special feature in the behavior of electrical conductivity was noted between 5 and 10 wt% of MXene: there was a significant increase of the conductivity value from 8.9×10^{-3} to 2.8 S m^{-1} . It suggests that the formation of a more continuous network of MXenes in TPU takes place, which means that the electrical percolation threshold is situated in this region. An even higher MXene amount of 15 wt% increased the electrical

conductivity value up to 11.6 S m^{-1} . It is obvious that further conductivity improvement at higher MXene concentrations occurs due to the creation of a denser conductive network structure, which creates more effective routes for the electron transfer, thus decreasing the electrical resistance of the composite.

Generally, the conductivity of the material increased by about 11 orders of magnitude from pure TPU to 15 wt% MXene nanocomposites. The change in conductivity shows the possibilities of such composites in terms of multifunctional applications. The nanocomposites with concentration ranges near the threshold of conductive network formation have excellent perspectives for use as piezoresistive sensors since their mechanical deformations may affect the inter-sheet distances and conductive contact points, leading to variations in electrical resistance. On the other hand, increased contents of MXene, especially 10% and 15%, offer better inherent conductivities and hence can be used for applications where there is need for better charge transport, for example, EMI shielding. This shows that the loading of MXene offers a good way to tailor the electrical properties of TPU while retaining its multi-functional nature.

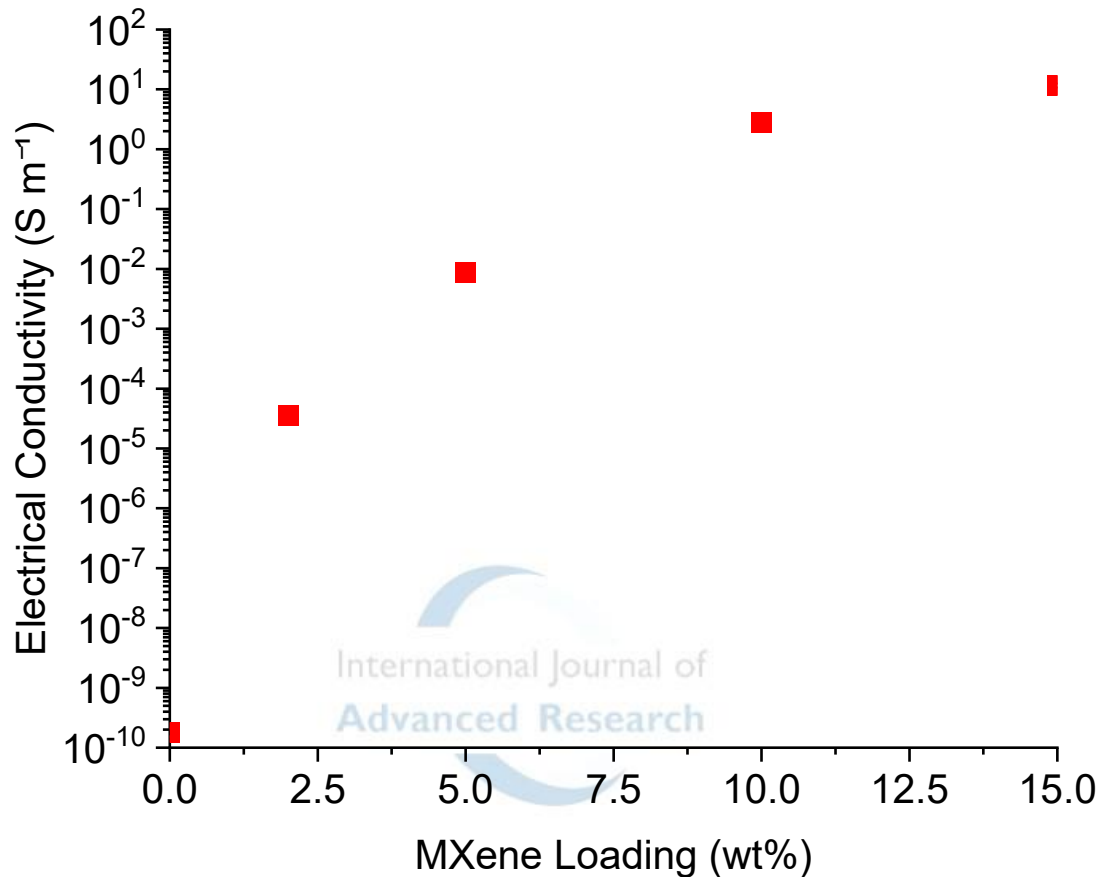


Figure 7: Conductivity of polymer nanocomposites reinforced by MXenes as a function of MXene content (weight %), demonstrating percolation point and transition from insulator to conductor for the fabrication of wearable electronic devices and electromagnetic shielding applications.

3.7 Strain-Sensing Performance

Electromechanical strain sensing performance of the TPU/MXene nanocomposites was studied using tensile deformation under a strain range of 1-50%. Pure TPU showed very little changes in its electrical resistance during deformation, which is because of its intrinsic insulating properties. Contrarily, addition of MXene greatly improved the electromechanical strain sensing performance of the polymer matrix by inducing strain-dependent changes in its electrical resistance[18]. In all tested compositions of TPU/MXenes nanocomposites, MXene-10 nanocomposite displayed a continuous increase in $\Delta R/R_0$ values with increasing strain values. The $\Delta R/R_0$ values were found to be equal to 0.08, 0.42, 0.96, 2.18,

3.71, and 6.25 at 1%, 5%, 10%, 20%, 30%, and 50% strain, respectively. Such an increase in the resistance of MXene-10 nanocomposites is due to increasing deformation and disruption of the conductive network of MXene within TPU matrix. It is worth noting that in low strains, the conductive network was still mostly intact, hence a relatively lower resistance value. As the degree of tensile deformation increases, the separation and rearrangement of neighboring MXene nanosheets cause an increase in electrical resistance as the effective number of conductive paths decreases. There was also reasonable repeatability in the response of the MXene-10 nanocomposite when loaded and unloaded, suggesting that the conductive network is capable of reversible

deformation in the elastomeric TPU matrix. The significant change in resistance and reversibility of

the electromechanical response show promising applications in flexible/wearable strain sensing.

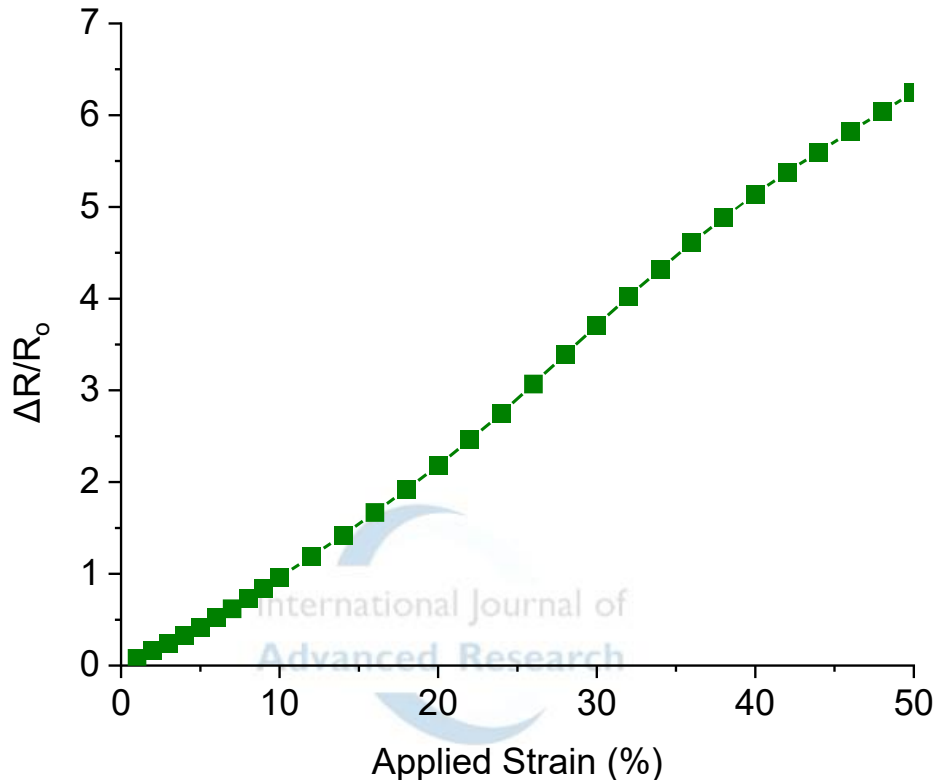


Figure 8: Strain-dependent relative resistance variation ($\Delta R/R_0$) of the MXene-10/TPU nanocomposite subjected to tensile deformation for strains between 1 to 50%.

3.8 Gauge Factor and Sensitivity

Strain Sensing Effectiveness of the MXene-10 Nanocomposite The strain sensing behavior of the MXene-10 nanocomposite material in terms of gauge factor (GF) as a function of tensile strain is illustrated above for a working strain range of up to 50%. Two distinct ranges of behavior for the sensing response can be observed. In the range of low strain values (1-10%), the GF increases slightly from 8.00 to 9.60, showing sensitivity to the initial strain application. However, in the higher strain

range (10-50%), the GF shows a more significant increase and reaches the maximum value of around 12.50 at 50% strain. This increased sensitivity in the range of higher strains is mainly due to the deformations and further separation of neighboring sheets of MXenes within the polymer matrix. Such separation increases the distances between the sheets and disrupts the conductive network, which results in a larger change in the electrical resistance value per unit strain.

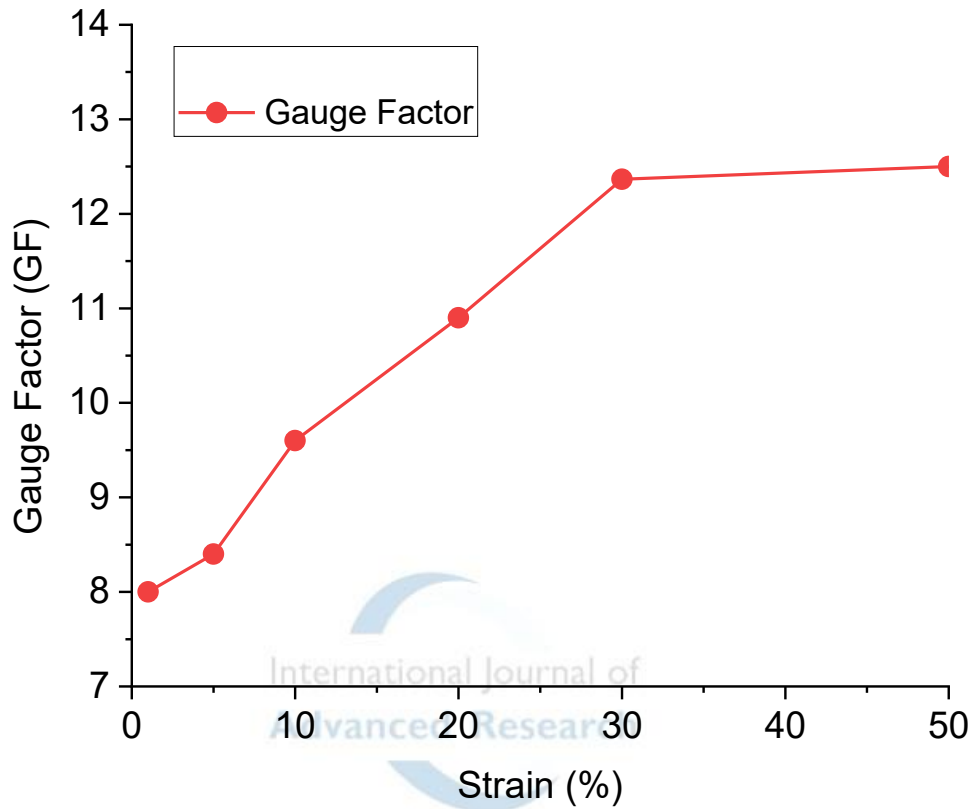


Figure 9: MXene-10 nanocomposite gauge factor (GF) versus the applied tensile strain (%) showing the two-regions strain sensing behavior.

3.9 Response Time and Cyclic Stability

The electromechanical responses and stability of the optimized MXene-10 sensor are shown in Figure 10a and 10b. In 10% tensile deformation, the sensor exhibits fast and reversible sensing behavior, with a response time of approximately 72 ms and a recovery time of approximately 86 ms. The fast response and recovery times suggest a quick change and recovery of the conductive network under mechanical deformation and unload. Moreover, cyclic stability testing in 20% tensile deformation shows great mechanical

stability of the sensor, in which the sensor retains nearly 94.3% of its original response after 5000 repeated loading-unloading cycles. There is only a slight resistance drift found during the entire deformation process, suggesting that the sensor has good stability and reliability of its sensing response. This high cyclic stability of the sensor can be attributed to the intrinsic elastic recovery nature of the TPU matrix, thus contributing to restoring the original composite and interfacial contacts and conductive paths among the MXene nanosheets after releasing the strain.

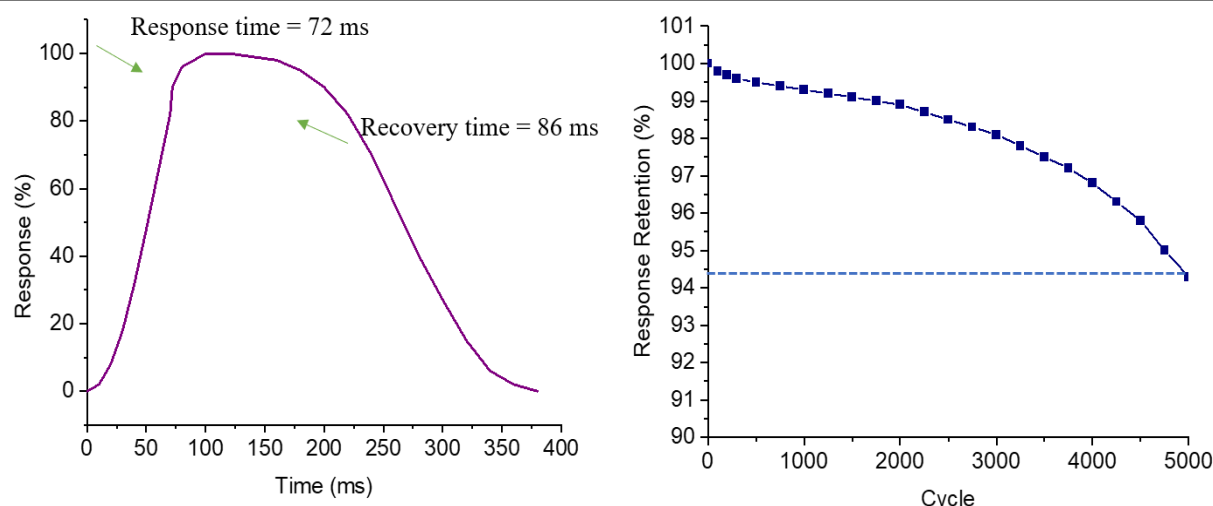


Figure 10: Performance characteristics of MXene-10 electrochemical/electromechanical sensor upon optimization: (a) Time response and recovery for 10% elongation, resulting in response time of ~ 72 ms and recovery time of ~ 86 ms. (b) Relative response retention (%) plotted against number of loading-unloading cycles up to 5000 cycles under 20% strain, indicating durability with 94.3% retention (dashed line).

3.10 Wearable Motion Monitoring

Dynamic electromechanical behavior of the MXene-10 wearable sensor upon repeated finger movement monitoring is illustrated in Figure 11. With regard to repetitive bending and un-bending actions monitored over a 10-second period, the wearable sensor revealed clear and consistent changes in the relative resistance ($\Delta R/R_0$). Following the application of mechanical deformation, there was a quick increase in $\Delta R/R_0$, which resulted in the appearance of very well-defined peak responses before quickly returning

back to the base-line after cessation of the mechanical strain applied. Peak responses were highly consistent over five successive cycles with $\Delta R/R_0$ of 3.5%, 3.6%, 3.5%, 3.5%, and 3.5%, respectively. Such consistency in peak response is an indication of stable and reproducible electromechanical transduction. On the other hand, signal recovery towards base-line level following each bending action indicates proper response not only to mechanical deformation but also to its cessation.

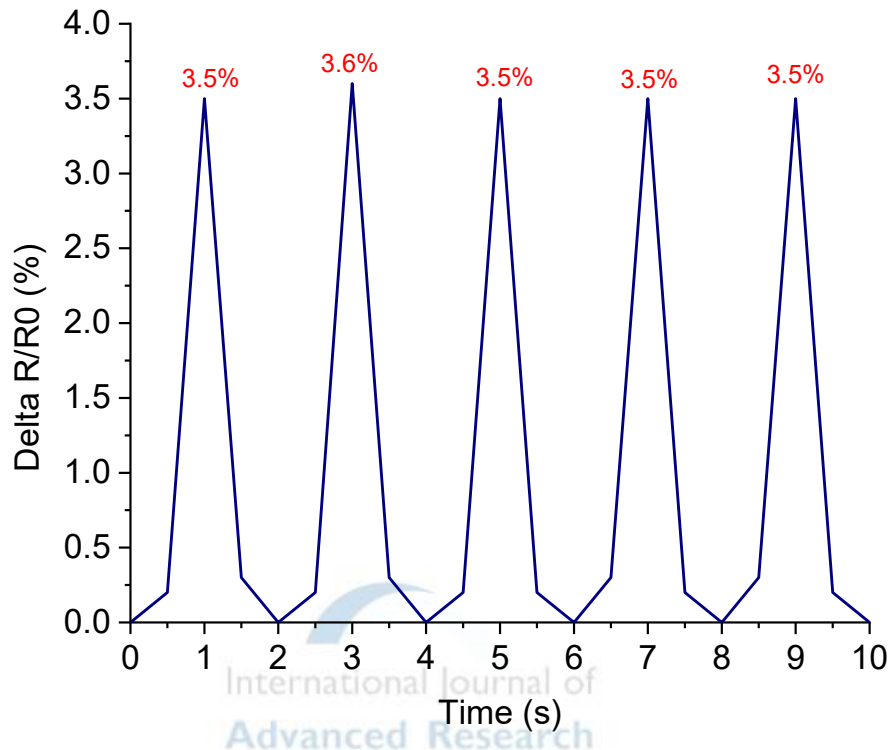


Figure 11: Response of relative resistance ($\Delta R/R_0$) to repeated bending and stretching of the wearable motion sensor for 10 seconds, showing the repeatability of electrical output with peak amplitudes of 3.5-3.6% of its value.

3.12 Frequency-Dependent EMI Shielding Behavior

The shielding efficiency of the nanocomposite MXene-10 at different frequencies in relation to EMI has been evaluated for the X-band frequency range (8.2–12.4 GHz) as indicated in Figure 12. In the frequency range investigated, the total shielding effectiveness (SE_t) was constant, rising from 29.6 dB at 8.2 GHz to 33.8 dB at 12.4 GHz. In this regard, the increase in SE_t is estimated to be approximately 4.2 dB over the frequency range. The gradual rise in SE_t with increasing frequency can be ascribed to the effects of frequency-dependent polarization phenomena, interfacial charge accumulation, and enhanced electromagnetic interactions within the heterogeneous MXene/TPU composite. The formation of conductive channels by the MXene

enables electrical conductivity with attendant dielectric losses, while the interfaces within the composite promote interfacial polarization and internal reflections.

The lack of significant sharp peaks or narrow resonance effects also implies that the shielding behavior does not involve a localized resonance-based mechanism. In contrast, the fairly smooth frequency dependency of the shielding effect shows broadband absorption based on the contributions of conduction, polarization, and interface loss effects in the nanocomposite material. Overall, the consistently high SE_t value of about 29.6–33.8 dB confirms the capability of the MXene-10/TPU composite material to act as a broadband EMI shielding material in the X-band frequency range.

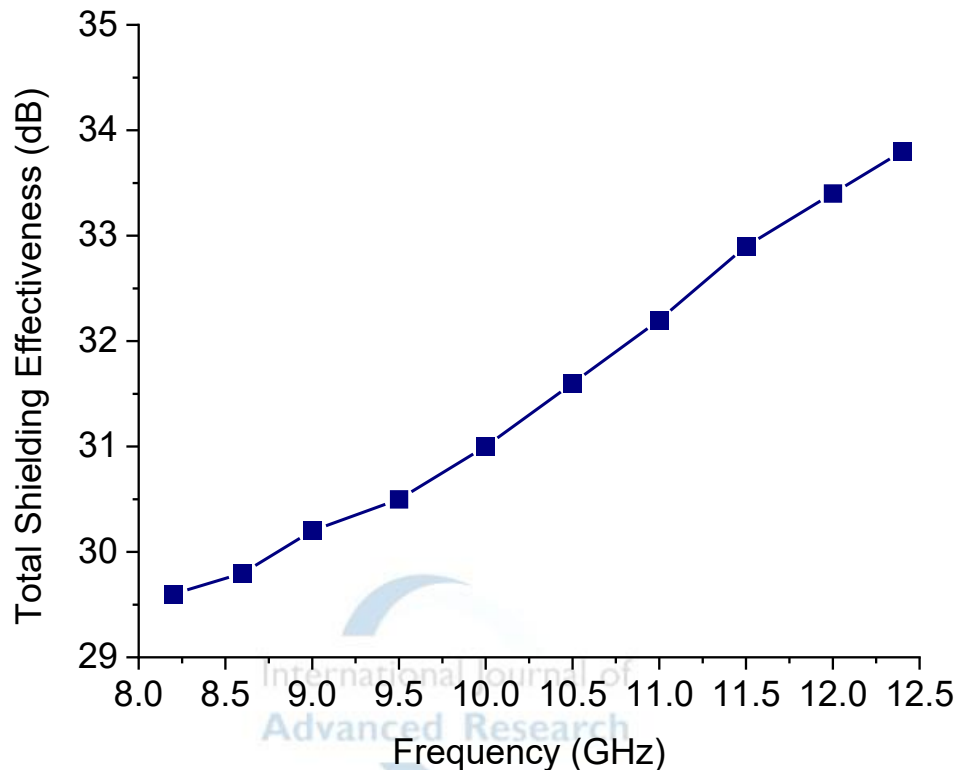


Figure 12: The frequency-dependent total shielding effectiveness (SE_t) of the MXene-10/TPU nanocomposite in the X band (8.2–12.4 GHz) range, showing the presence of a broadband stable shielding behavior with increasing attenuation values as the frequency increases.

3.13 Effect of Nanocomposite Thickness

Figure 13 illustrates the dependence of the EM interference shielding properties of the MXene-10 nanocomposite on film thickness. An obvious improvement of the shielding effectiveness (SE_t) value was noted when the thickness of the film increased from 50 to 200 μm . Thus, the SE_t value increased from 18.9 dB at 50 μm to 26.7, 31.8, and 37.4 dB at 100, 150, and 200 μm , respectively. It clearly shows that the increase in thickness significantly influences the electromagnetic attenuation in the MXene/TPU composite.

It should be emphasized that the improved performance of shielding at larger thickness is associated with higher volume fraction and continuity of conductive paths in the composite material, leading to effective electrical conduction and electromagnetic energy dissipation. Moreover, the longer path length allows for a higher level of interaction of the incident electromagnetic radiation with the composite material, which leads to a conductive loss, dielectric loss, interfacial polarization, and many other phenomena inside the heterogeneous system. As a result, the film of 200 μm showed the highest SE_t value of 37.4 dB.

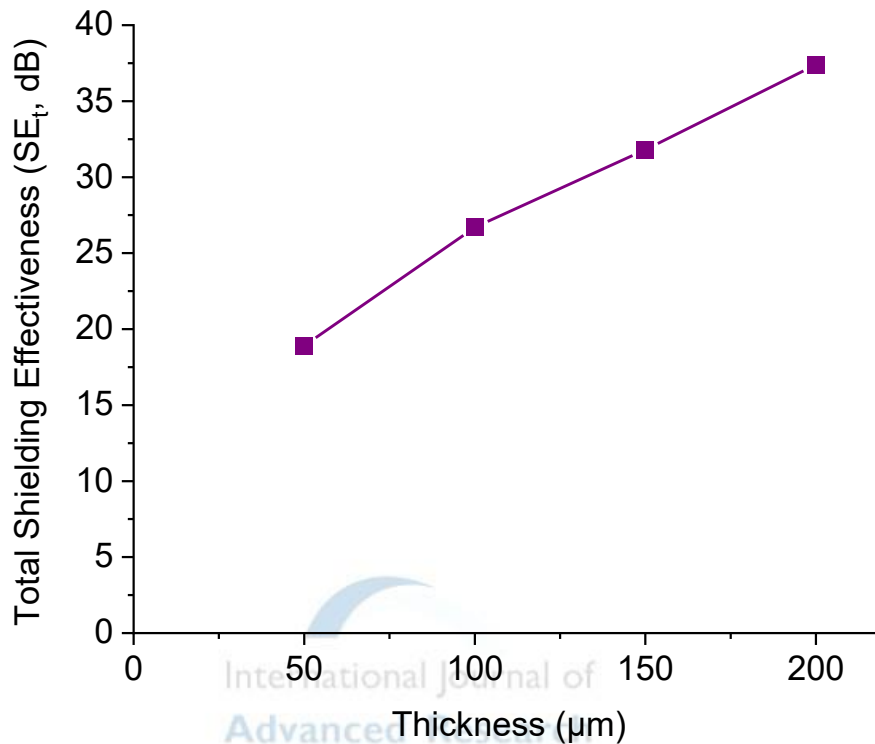


Figure 13: MXene-10/TPU nanocomposite films' total electromagnetic interference shielding effectiveness (SE_t) dependent on thickness, which exhibits better shielding effectiveness with increasing film thickness from 50 to 200 μm.

3.14 EMI Shielding after Repeated Bending

The mechanical reliability of the optimized MXene-10 nanocomposite film was determined by studying the total electromagnetic interference shielding effectiveness (SE_t) of the film under repeated bending tests with a bending radius of 10 mm. It can be seen from Figure 14 that the initial value of SE_t of 31.8 dB showed a steady decline with the increase of the bending cycles, decreasing to 29.3 dB after 1000 cycles. Although there was a slight decrease in the SE_t of the film, its shielding effectiveness remained at around 92.1%, which is only a decline of 2.5 dB.

This indicates the excellent stability of the electromagnetic shielding capability of the MXene-10 nanocomposite film under mechanical deformations. The stability is mainly due to the flexible matrix of TPU, which is capable of undergoing repeated mechanical bending and sustaining the connectivity of the conductive network of MXenes. Although the repeated bending may lead to some disruptions of the connectivity, the network is still well-interconnected enough to ensure the electromagnetic attenuation capability.

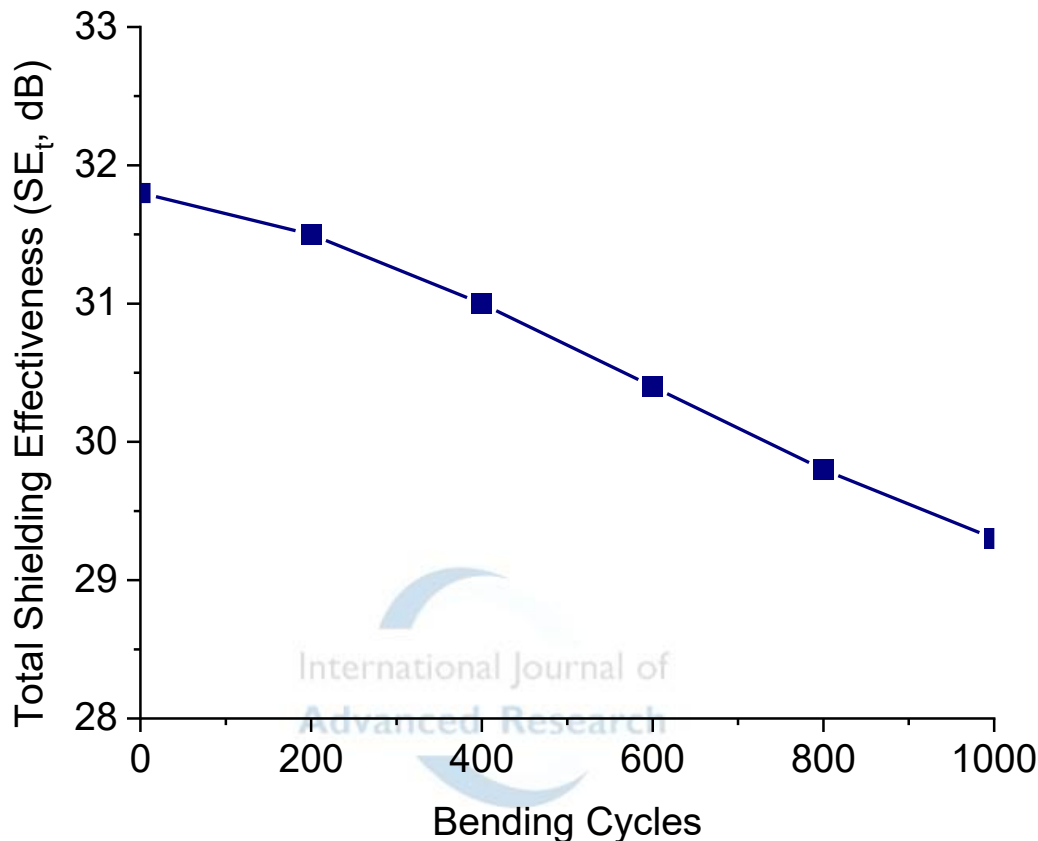


Figure 14: Durability of the MXene-10/TPU nanocomposite film under mechanical deformation through repetitive bending at a radius of 10 mm with respect to the conservation of total electromagnetic interference shielding effectiveness (SE_t).

3.15 Structure-Property Relationship and Functional Mechanism

Figure 15 shows the evolution of the MXene conductive network inside the TPU matrix, as well as the strain sensing and EMI shielding mechanism that takes place at each stage. With only 2 wt% MXene loading, there is very little connectivity between the nanosheets, causing them to be separated from each other in the polymer matrix. As a result, there is little interaction between particles and thus not enough conductive network to provide effective charge transfer. Upon further increase in the amount of MXene to 5 wt%, more compact arrangement of nanosheets and formation of contacts between them take place, marking the beginning of a percolated conductive network. At 10 wt% MXene

content, good connectivity of the conductive network is achieved along with sufficient TPU connectivity, which provides a proper balance of electrical conductivity and flexibility of the composite. The strain sensing is controlled by the effect of deformation on the conductivity of the composite system. In the state of tensile strain, the elongation of the TPU matrix causes an increase in the distance between adjacent nanosheets of MXene, hence, an increase in the tunneling distance and the contact resistance. This results in an increase in the electrical resistance of the composite system ($\Delta R/R_0$). The removal of strain leads to the reconnection of the MXene nanosheets, thus, enabling the conductive network and electrical signal generation.

On the other hand, EMI shielding mechanism includes several processes responsible for electromagnetic energy attenuation. On interacting with the composite surface, some of the incident EM radiation is reflected owing to the conductive MXene network and the consequent impedance mismatch, thus forming the reflection part of the process (SE_r)[19]. The radiation penetrating into the composite further encounter's conductive losses, interfacial polarization, and reflections from many MXene-TPU interface surfaces[20]. This helps in

increasing the path length and converting the EM energy into heat energy inside the composite, thus making a contribution towards the absorption part of the process (SE_a). The schematics show the synergy that exists between the two materials, where the MXenes offer electrical connectivity for conductivity and EM radiation suppression, while TPU ensures the structural continuity and flexibility of the composite. Such a synergy between the two materials at moderate MXenes concentration results in a flexible strain sensor as well as a good EMI shielding material.

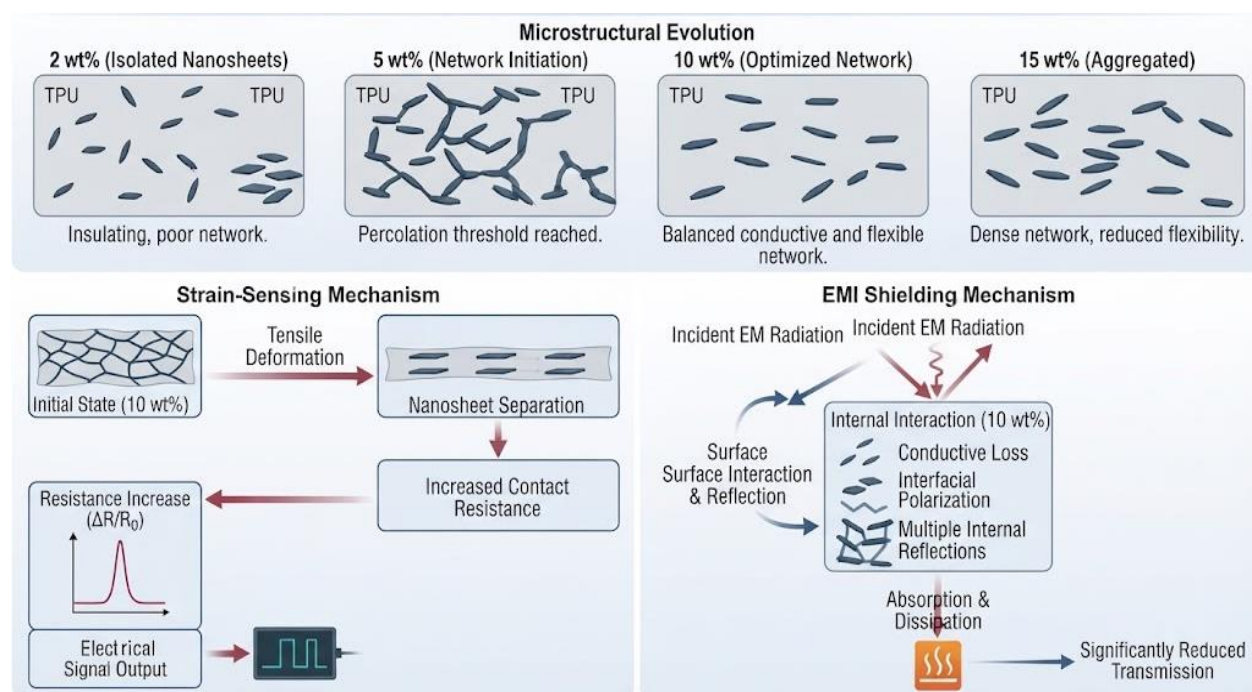


Figure 15: Schematic representation of the structure-property relationship and functional mechanisms of MXene/TPU nanocomposites, illustrating the development of the MXene conductive network with varying concentrations (upper), electromechanical strain sensing effect due to deformation (lower left), and reflection, absorption, polarization, and multiple reflection effects involved in electromagnetic interference shielding (lower right).

4 Conclusion

In this research work, Ti_3C_2Tx MXene/thermoplastic polyurethane (TPU) nanocomposite films with multifunctionality for flexible strain sensing and electromagnetic interference (EMI) shielding applications have been successfully prepared. The MXene nanosheets were prepared by a simple method of

etching the MAX phase Ti_3AlC_2 with a solution of LiF/HCl followed by exfoliation. The MXene nanocomposites with 0–15 wt% MXene were prepared and characterized by several techniques. The successful formation of MXene and increase in layer-to-layer distance were confirmed by XRD analysis, and the hydrogen bonding interactions in between the functional groups of MXene and the

TPU were confirmed by FTIR spectroscopy. SEM images of MXene nanosheets showed accordion-like layered morphology and exfoliated nature of the MXene sheets. The thermal stability of the nanocomposites increased due to increased onset decomposition temperature from 287 to 315 °C. Young's modulus increased while the elongation at break decreased with increasing MXene content in nanocomposites. The electrical conductivity increased from 1.8×10^{-10} to 11.6 S/m with increasing MXene from 0 to 15 wt% and percolation occurred in the range 5 to 10 wt%. For X band, the shielding effectiveness of EMI was between 29.6-33.8 dB, while increasing from 18.9 to 37.4 dB due to the increase in thickness from 50 to 200 microns. Post 1000 bending cycles, 92.1% of shielding effectiveness was achieved. Conductive loss, interfacial polarization, and multiple internal reflections were the reasons behind it. Scalable fabrication, environmental stability, integration with wireless technology, and shielding effectiveness for other frequencies are required in future research.

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