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Review

Polymer/carbon nanotube composites: A comprehensive review on fabrication techniques and their consequences

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Abstract: Polymers have attracted significant attention in recent years due to their light weight, low cost, favorable strength-to-weight ratio, and ease of fabrication. Their mechanical and thermal properties have been significantly tailored by reinforcing carbon-based and metal nanofiller materials that possess excellent mechanical and thermal properties. In addition, excellent morphological properties and good aspect ratio of carbon nanotubes (CNTs), graphene, graphene nanoplatelets (GNP), graphene oxide (GO), etc., make them ideal candidates for reinforcement purposes. CNTs/GO have been widely used for the synthesis of polymeric-based composite materials. Moreover, high modulus, better strength, and thermal stability at extremely high temperatures of CNTs/GO motivated various researchers and scientists to make them useful for engineering applications. Their composites with polymer-based composite materials have opened several avenues of application for the automobile, aircraft, textile industries, and biomedical applications. However, during the synthesis of composites, the uniform dispersion into the polymer matrix is a challenge to achieve. Therefore, numerous techniques have been developed by researchers to disperse CNTs/GO throughout the polymer matrix. The selection of the fabrication technique is critically important as the overall performance of the final and developed product is highly dependent on the quality/degree of dispersion, i.e., to what extent carbon particles are dispersed within the matrix. In this review, various fabrication methods for different polymeric composite materials were thoroughly reviewed, and their impact on mechanical and thermal properties were discussed. The review highlights processing methods suitable for the synthesis of polymer composites depending on their applications.

Keywords: CNTs; multi-walled carbon nanotubes (MWCNTs); synthesis techniques; polymers; mechanical properties

1. Introduction

The science of producing composite material is very broad, as it involves all industries that manufacture finished products by using various raw materials [1]. In simple language, a composite material can be defined as a material that is a mixture of two or more materials having properties superior to the parent material [2–4]. Metal matrix composites (MMCs) materials may possess excellent mechanical strength and other properties than those of Polymer matrix composites (PMCs), but their heavy weight, high manufacturing cost, and complex fabrication open the door for alternative light-weight polymer-based composite materials. Figures 1 and 2 show the types of composite materials on the basis of matrix and reinforcement, respectively [5]. Ceramic matrix composites can be defined as a material in which both the base matrix and reinforced material are made of ceramic material, and organic matrix composites, which are also known as PMCs, are composite materials that are made of organic polymers such as resin. These PMCs are further divided into two categories: thermoplastic and thermosetting matrix.

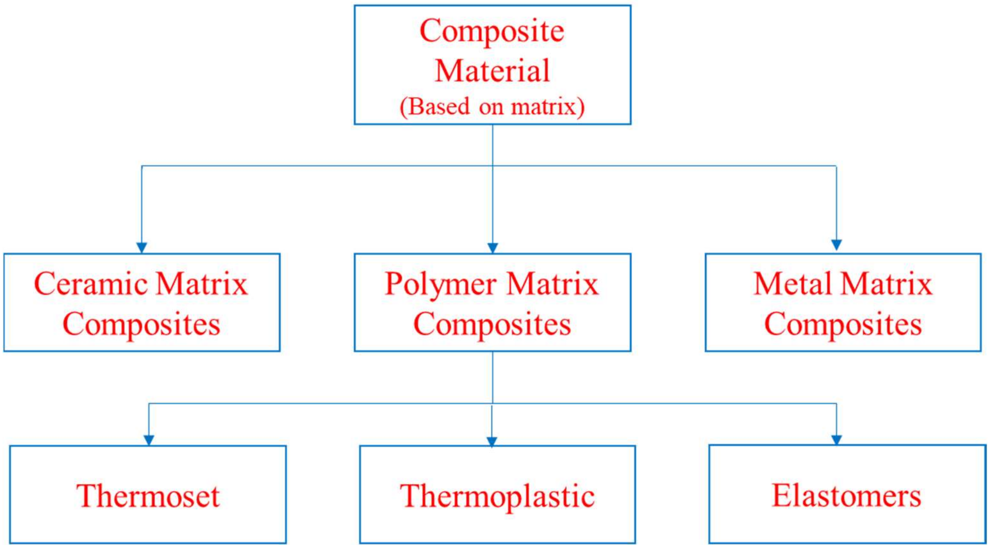


Figure 1. Composite materials based on matrix.

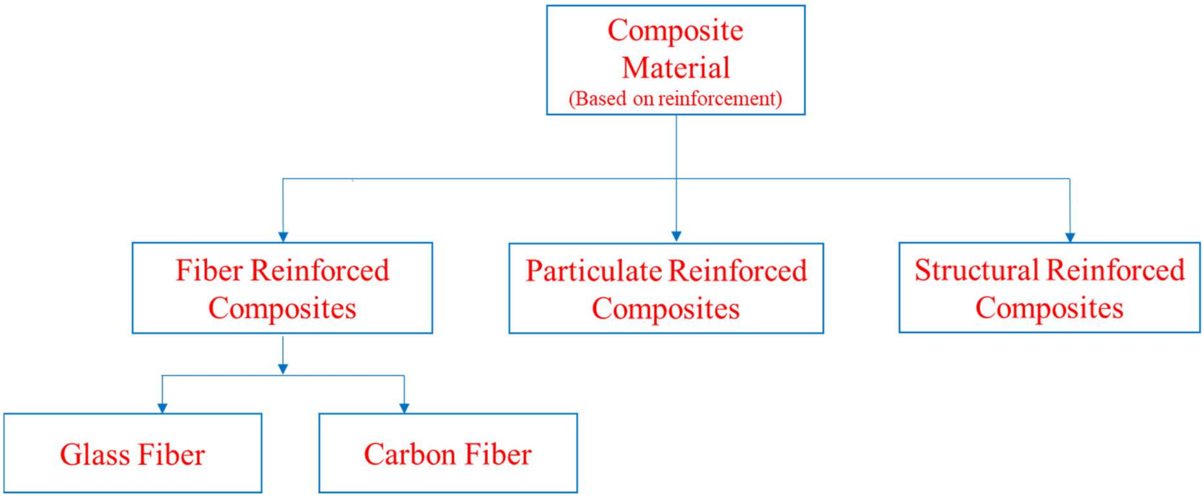


Figure 2. Composite materials based on reinforcement.

Composite materials, based on their reinforcement, can be classified as fiber reinforced, particulate reinforced, and structural reinforced [6]. Rubber composites are also important and belong to a special class of polymer composites in which elastomers act as the primary base matrix. These composites are known for their resilience, flexibility, and ability to withstand higher strain without fracture. Researchers have shown that by reinforcing various kinds of filler additives into the base matrix of rubbers, their mechanical, thermal, and physical properties could be improved, making them an essential material for different industrial applications. Natural rubber, nitrile rubber, and styrene-butadiene rubber are some of the examples of base matrices that are widely used for the synthesis of rubber composites. Some typical applications of rubber composites involve tires, industrial and commercial hoses, gloves, seals, cable insulations, and tubes. Among these materials, fiber-reinforced materials have drawn a lot of attention for the fabrication of plastic-based composite materials for various industrial applications. These fiber-reinforced materials can be glass-reinforced and carbon-reinforced. The carbon fiber materials combine high-strength carbon particles with a virgin polymer matrix [7]. Carbon fiber reinforced materials are considered superior to glass fiber due to the easy processing of small-sized carbon particles than glass fibers [8,9]. The PMCs' materials have been used in almost every field, ranging from sports, protective gears, automobiles, the aviation industry, and other low-load-bearing industrial applications. The PMCs may lack in mechanical performance when exposed to intermediate loading conditions under higher frequencies. Moreover, a polymer material may also fail during its thermal exposure due to its lower melting temperature. Despite these limitations, these polymeric materials have drawn the attention of researchers with great interest because of their light weight, low cost, corrosion-resistant properties, and easy handling during fabrication. Therefore, scientists worldwide have explored these polymeric-based materials for engineering applications by adding some carbon-based filler materials to the polymer matrix.

Since their discovery by Iijima [10], carbon nanotubes (CNTs) have attracted significant attention from researchers due to their excellent mechanical, thermal properties, and better load transfer properties. CNTs are made by converting 2D graphite sheets into a hollow tube-like structure [11]. Some reports also claimed that CNTs are 100 times stronger than steel [12,13] and, possess excellent stiffness and strength [14]. Although CNTs exhibit ballistic charge transport near the Fermi level, in polymer matrices, their contribution to conductivity is limited unless they are well-separated and incorporated into a highly conductive polymer [15]. Still, the small size and good aspect ratio of CNTs make them a suitable candidate to disperse into a polymer matrix. CNTs have a length of a few micrometers and a diameter that can vary up to 100 nm [16]. Moreover, CNTs possess better thermal stability at extremely high temperatures [17,18]. These CNTs are constituted of cylinder-shaped graphene sheets rolled in a cylindrical arrangement [19]. CNTs are further classified into single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) depending on their structure and number of layers [20]. The reinforcing high-strength agent in the base polymer makes it possible to enhance the mechanical and thermal behaviour of the polymer up to a certain limit. The success of fabricating these types of polymer composite materials with improved properties is mainly dependent on the degree of dispersion of filler particles into the base matrix. Techniques using different equipment have been developed in the past to achieve uniform dispersion of these CNTs in a polymer matrix. Researchers have found that different fabricating techniques have different effects on the mechanical and thermal quality of the composite material [21–23]. Various authors fabricated different types of materials by reinforcing CNTs and graphene particles into the polymer matrix and claimed that the synthesis method used was appropriate for improving the overall performance of the composite material [24,25]. For instance, the solvent casting method [26–29], melt-mixing using a twin-screw machine, in-situ polymerization, and additive manufacturing are some of the developed methods traditionally used for the synthesis of polymer-based composite materials. Reports on polymer composites containing CNTs reveal that synthesis techniques play an imperative role for obtaining dispersing these particles in the polymer matrix in order to achieve a final composite material with enhanced properties. Studies have reported that the agglomeration of CNTs results in the poor quality of final composite material in terms of its mechanical and electrical performance; therefore, the uniform dispersion of CNTs particles becomes critically imperative in achieving better mechanical and thermal characteristics of composite material [30,31]. Moreover, higher loading of CNTs lead to agglomeration due to the presence of strong van der walls forces among them [32].

In this work, the impact of different fabricating techniques on the overall performance of the polymer materials has been thoroughly reviewed. The optimal percentage of reinforcement in polymer matrix has been discussed, and its effect on the performance of material fabricated via various methods has been summarized. The major parameters that have been targeted by researchers include mechanical aspects, thermal stability of material at higher temperatures, and electrical properties of the final product. We focused on providing an efficient fabrication technique for enhancing the overall behavior of numerous polymers at a minimum weight percentage of integrating material. A comprehensive review of the developed polymer composite fabricating techniques is imperative to overcome the cost involved in the process. Furthermore, several factors such as cost of final product, efficiency of method, extent of dispersion of filler material, and level of improvement are also essential while selecting a fabrication method for developing composite material.

2. Different fabricating techniques

2.1. Solvent casting method

Solvent casting is a technique that uses a solvent to dissolve polymer material, and the same solvent is used to disperse CNTs separately with different weight percentages (wt%) of CNTs. After that, both solutions are mixed and magnetically stirred for better dispersion of reinforcement in the final solution. After mixing, the final solution containing different loading of CNTs is placed in the oven for removing the solvent by maintaining the temperature at a value depending on the nature of the solvent. After casting, thin films of composite material with different compositions of CNTs are obtained, which are further used to investigate the morphological and other parameters depending on the application of the material. Scientists have extensively explored these solvent casting techniques to synthesize CNTs reinforced polymer composite materials with significantly enhanced mechanical behavior than that of pristine polymers [33–35]. They mainly emphasized dispersing CNTs uniformly throughout the polymer matrix. Zadeh et al. [36] developed polyurethane (PU) composites with different wt% of CNTs (0.05, 0.1, 0.5, and 1 wt%) using the solvent casting method. Authors reported that uniform dispersion of CNTs was achieved throughout the PU matrix, and noticeable improvement in electrical conductivity, Young's modulus, and toughness of PU/CNTs composites had been attained in equivalent to the pure PU. The electrical conductivity for pristine PU was 0.0073 S/cm, but with low loading of CNTs (0.05 wt%), the same was increased to 0.0080, which further peaked to 0.0556 S/cm for 1 wt% CNTs by showing a drastic increment of 6fold over that of pure PU. Researchers further revealed that the Young's modulus and toughness of PU/CNTs composites were also increased with increasing concentration of CNTs up to 0.05 wt.%, and after adding more CNTs, the mechanical performance of the composite material was decreased due to the agglomeration of CNTs particles. Jindal et al. [37] used the solvent casting method to disperse MWCNTs in polycarbonate (PC) by varying the composition of MWCNTs up to 10 wt% and performed scanning electron microscopy (SEM) and observed uniform dispersion (as shown in Figure 3) with this higher wt% of MWCNTs. By adding 10 wt% of MWCNTs, scientists reported a significant enhancement of 136% and 375% in elastic modulus and hardness, respectively, in comparison to the pure PC.

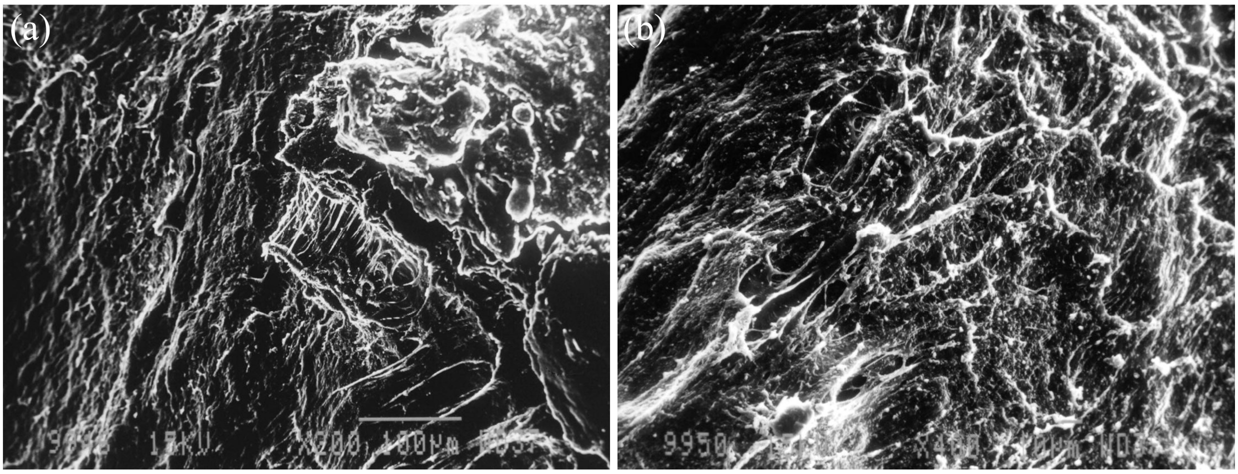


Figure 3. SEM images of PC/MWCNT composites. (a) 5 wt% MWCNTs and (b) 10 wt% MWCNTs (Reproduced from Ref. [37] with permission).

The integration of MWCNTs improved the load transfer [38] capability in PC composite, which further led to an improvement in the mechanical performance of the material. The increment in hardness was possibly due to the stronger short-range linkages between MWCNTs and PC. Researchers also reported that the penetration depth of the composite had been significantly reduced with the increasing composition of MWCNTs, which justified the statement of improved hardness of the composite materials. Gupta et al. [39] also used the solvent casting method to synthesize PU-based reduced graphene oxide (GO) composites at different compositions (0–5 wt%) of GO reinforcement and characterized these composites under Nanoindentation loading conditions at 1 mN load. The hardness for PU was recorded as 58.5 MPa, which ramped to a value of 140 MPa for 5 wt% loading of reduced GO by showing an improvement of 139% when compared to pure PU, as shown in Figure 4.

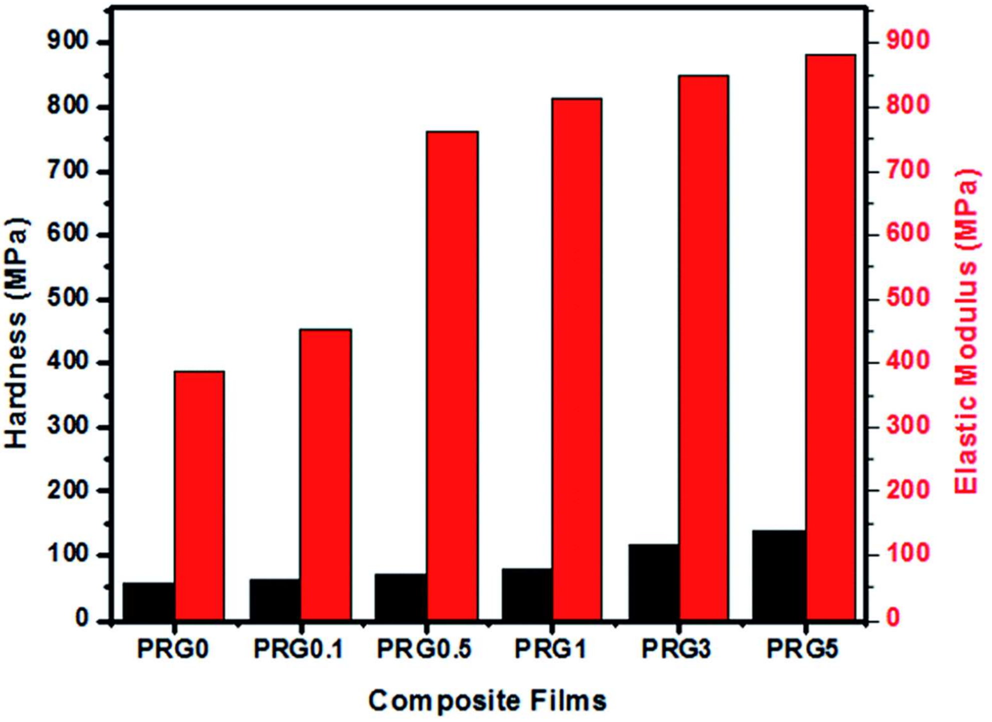
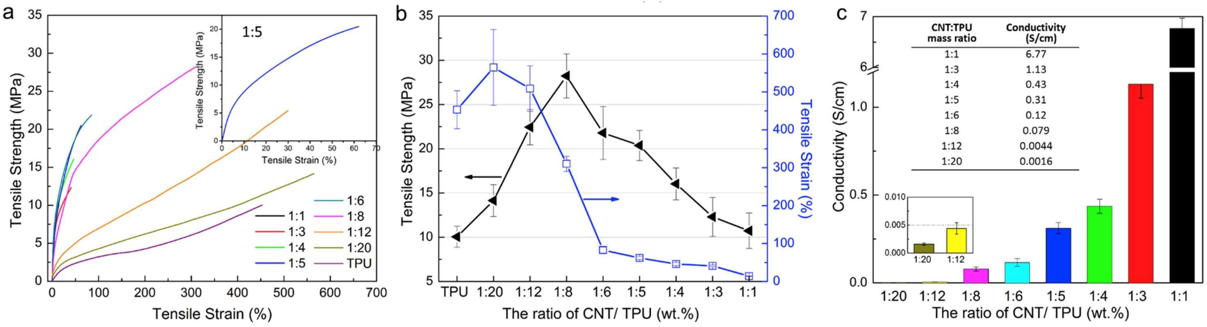


Figure 4. Variation in the hardness and elastic modulus of composite material at 1 mN load (Reproduced from Ref. [39] with permission).

Furthermore, the value of elastic modulus increased from 385.7 MPa for pure PU to 881.7 MPa for 5 wt% compositions, which showed an enhancement of 129% in comparison to pure PU. The significant enhancement in mechanical properties was greatly attributed to the presence of strong interlinkage between PU and acid-treated MWCNTs, which was also confirmed by the SEM images of the fractured surface. He et al. [40] also explored the solvent casting technique to fabricate MWCNTs reinforced thermoplastic polyurethane (TPU) composite fibers. Researchers investigated the effect of MWCNTs content and their alignment on the electrical and mechanical properties of the developed composite fiber. They reported that 1:5 MWCNTs/TPU composites showed better alignment along the direction of the fiber, while for higher concentrations (1:1 and 1:3), MWCNTs formed a dense network in the material, and many pores were visible on the surface. When the concentration of MWCNTs was higher, strong van der Waals forces resulted in the formation of clusters of these particles, which halted the alignment of MWCNTs along the direction of the fiber. However, when the concentration was optimum, the MWCNTs particles got aligned along the fiber direction after coagulation. To investigate the effect of MWCNTs loading on the mechanical behavior of the composite material, researchers performed a filament tensile test and observed that elongation and tensile strength of the material increased when the concentration of MWCNTs was higher. As shown in Figure 5, neat TPU showed a tensile strength of approximately 10 MPa at a maximum strain of 450%, while 1:8 MWCNTs/TPU had a tensile strength of 28 MPa, which is almost 3 times than that of pure TPU.



CNT:TPU mass ratio	Conductivity (S/cm)
1:1	6.77
1:3	1.13
1:4	0.43
1:5	0.31
1:6	0.12
1:8	0.079
1:12	0.0044
1:20	0.0016

Figure 5. (a) Tensile stress-strain curves of TPU/MWCNTs composites, (b) tensile strength and maximum strain at break, and (c) electrical conductivity of TPU at different weight % of MWCNTs (Reproduced from Ref. [40] with permission).

The significant improvement in tensile strength was attributed to the good load sharing and transferring of MWCNTs in the polymer matrix [41]. However, a further increase in MWCNTs content resulted in a decrease in mechanical properties [40], which was due to the inefficient contact and agglomeration of MWCNTs by the strong van der Waals forces among these particles. As shown in Figure 5, the electrical conductivity of the material also increased with the increasing content of MWCNTs. When the concentration was lower (1:20), the value of electrical conductivity was recorded as 0.0016 S/cm, which increased to 6.77 S/cm for higher composition of MWCNTs in TPU (1:1). This improvement in electrical conductivity of the TPU based MWCNTs composite fiber was due to the conductive path formed by the MWCNTs linkages, which is also attributed to highly conductive percolation network [42]. Bansal and his colleagues [43] synthesized hybrid composites of polystyrene with different compositions of CNTs and reduced graphene oxides (rGO); even dispersion of filler materials was obtained after performing SEM analysis. The specimens were also characterized under differential scanning calorimetry (DSC) techniques (in the range of 25 to 170 °C) and nanoindentation tests (at a load of 1000 μN). DSC results showed that for 5 wt% of each GO and CNTs, the mean glass transition increased to 98.40 °C when compared with that of pure polystyrene (92.66 °C). Nanoindentation tests also displayed considerable improvement in the elastic modulus and hardness of the developed material, as shown in Figure 6.

For 10 wt% composite materials, the modulus was recorded as 2.776 GPa, which was 13 % higher than that of pure polystyrene, while the hardness of the 5 wt% filled material was observed as 0.2208 GPa, which showed an increment of 20%. The improved modulus of the composites was ascribed to the excellent properties of filler particles, their even distribution in polystyrene, and the strong interaction between polymer and filler. The finer size of filler particles could also be the reason for the improvement in the mechanical performance of the composite. Reported material with superior mechanical and thermal properties could be applicable for higher-temperature applications.

The TPU composites with varying composition of MWCNTs (0.5, 1, 2, 4 wt%) fabricated via solvent mixing techniques were used by Su et al. [44] to estimate the increase in mechanical properties for exploring the material's applicability in athletic protective gear. The suspension of dispersed

MWCNTs in DMF was used for SEM analysis at an accelerating voltage of 5kV, image showed uniform dispersion of MWCNTs.

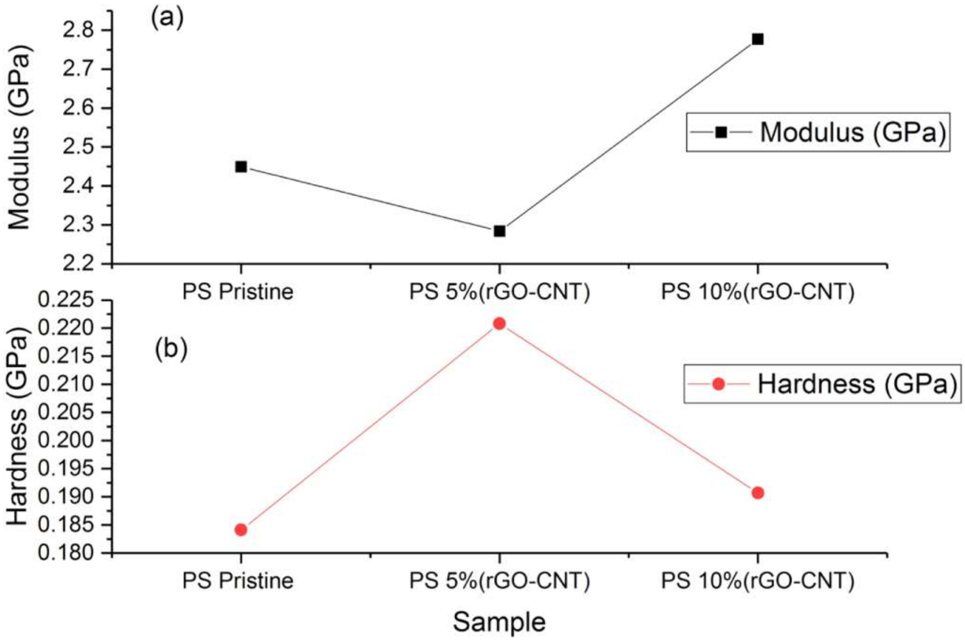


Figure 6. Mechanical properties of polystyrene with different wt% of rGo and CNTs (Reproduced from Ref. [43] with permission).

Thin films of composite material were successfully produced and then moulded into the desired shape by using a compression moulding machine. These specimens were used for tensile testing as per ASTM D-638-14 by using Instron 5567 with a load cell capacity of 30 kN. The dogbone-shaped specimens were cut from the moulded sheets with dimensions of 115 × 19 × 2 mm. For each composition, 12 samples were tested, and their average value was taken. The morphological image, tensile strength, Young’s modulus, elongation at break, and toughness were studied, which were evaluated during testing, as shown in Table 1, Figures 7 and 8.

Table 1. Tensile properties of TPU/MWCNTs composites [44].

MWCNTs (wt%)	Tensile strength (MPa)	Young’s modulus (MPa)	Elongation at break (%)	Toughness (MJ/m^3)
0.0	52.33	412.25	567	47.21
0.5	55.21	430.33	560	48.33
1.0	58.17	450.17	550	49.37
2.0	60.16	480.34	540	50.59
4.0	62.51	507.22	530	51.91

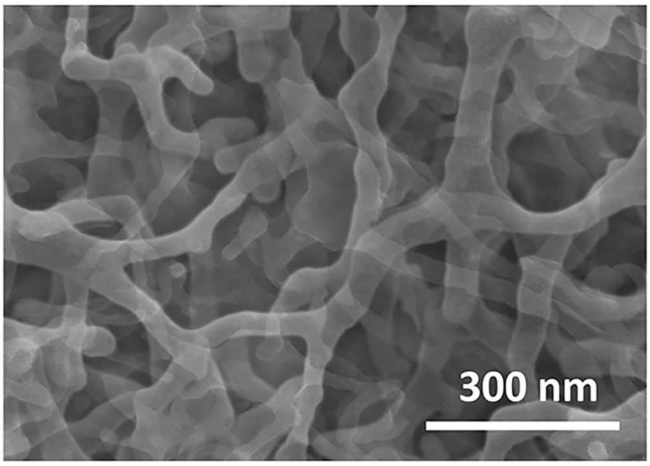


Figure 7. SEM analysis of MWCNTs/DMF dispersion (Reproduced from Ref. [44] with permission).

The values of tensile strength, Young's modulus, and toughness for neat TPU were obtained as 52 MPa, 412 MPa, and 47 MJ/m^3, which were increased to 62 MPa, 507 MPa, and 51.91 MJ/m^3, respectively, for a higher composition of MWCNTs (4 wt%). The improved mechanical performance of the material was ascribed to the high modulus of MWCNTs and better load transfer over the polymer matrix.

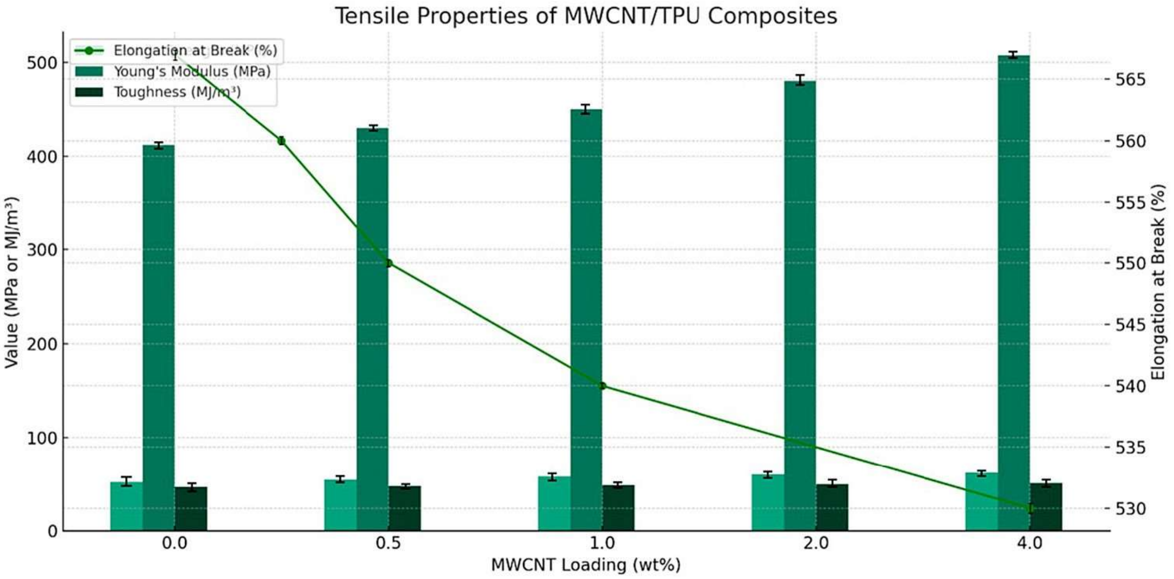


Figure 8. Mechanical properties of TPU/MWCNT composites (Reproduced from Ref. [44] with permission).

Researchers have also performed compressive studies on the same composites by applying compressive load at a rate of 1 mm/min. Results of compressive tests revealed an increment of 23% and 15% in compressive modulus and compressive yield strength in comparison to neat TPU. Researchers concluded that 1–2 wt% is the optimal value of MWCNTs for enhancing the overall mechanical performance of the material under both tensile and compressive loading conditions. The authors suggested that the high surface area and aspect ratio of provided an efficient load transfer between stiff MWCNTs and the polymer matrix, which could be the reason for the significant improvement in the properties of the material. This solvent casting technique was also used by A Katheria and his colleagues [45] for the fabrication of a composite material of poly(ethylene-co-methyl acrylate) or EMA (a copolymer of ethylene and methyl acrylate) reinforced by two filler materials, i.e., Fe_3O_4@g-C_3N_4. The final composite material exhibited an improvement in the value of electrical and thermal conductivity of the material. Reinforcement of Fe_3O_4@g-C_3N_4 (7 wt%) and MWCNTs (3 wt%) into EMA polymer showed a thermal conductivity of 0.92 W/mk, while the electrical conductivity at the same composition was quantified as 10^{-2} S/cm. The exceptional combination of magnetic and thermal conductive properties of Fe_3O_4@g-C_3N_4 and electrical conductivity of MWCNTs was the suggested reason for the improved performance of the material.

The epoxy-based bis-phenol-A GO nanocomposites were also fabricated using the solvent casting method, and scientists [46] reported even dispersion of GO in the epoxy matrix. Nanoindentation tests were performed on the composite material, and fracture resistance was represented by the ratio of elastic modulus to hardness, which was ameliorated by 24% for a minor composition of GO (0.5 wt%). This rise in fracture resistance of the material indicated that material shifted toward a ductile nature after reinforcing GO. The significant improvement in the fracture resistance was ascribed to the addition of a large-sized GO, which hindered the propagation of the crack through the composite material.

Nanoindentation tests were performed for PU/MWCNT reinforced composite materials fabricated via the solvent casting method [27], and the viscoelastic characteristics of the composite material were measured at varying frequencies (5–250 Hz). Researchers reported that at a higher frequency (250 Hz), for 10 wt% PU/MWCNTs composites, the storage modulus was improved by 148% to that of pure PU. The researchers also noticed an interesting fact that for the same content of MWCNTs, the value of storage modulus at 250 Hz frequency was 13% higher when compared to the value at 75 Hz, indicating that the effect of MWCNTs was more pronounced at higher frequencies. In addition, at lower frequencies, a lower content of MWCNTs (3 wt%) was sufficient to reduce the value of the damping factor (also known as tan δ [47,48]) from 0.20 to 0.15, indicating that after reinforcing MWCNTs, the material becomes more elastic than viscous. This change in the mechanical behavior of the material was attributed to the even distribution of MWCNTs and their linkage with the PU matrix. The list of properties achieved in PMCs by a composite fabricated by the solvent mixing method is tabulated in Table 2.

Table 2. Results of properties of samples fabricated via the solvent casting method.

Polymer	Filler	Tensile strength (MPa)	Hardness (MPa)	Modulus (MPa)	Toughness (MPa)	Electrical conductivity (S/cm)
PU [36]	MWCNTs	-	-	6.8	9.1	0.0449
PC [37]	MWCNTs	-	382.7	6990	-	-
PU [39]	GO	-	140	881.7	-	-
ABS [49]	MWCNTs	-	229	4362	-	-
PU [40]	MWCNTs	28	-	-	-	6.77
PS [43]	CNT/GO	-	2208	2776	-	-
PU [44]	MWCNTs	62.51	-	507.22	51.91	-
Epoxy [46]	GO	-	264.9	4079	-	-
ABS [50]	MWCNTs	-	220	4478	-	-

2.2. In-situ polymerization

This is a chemical process that involves the addition of filler material during the polymerization process. The reinforcement of filler particles into the base matrix of polymer is mainly done during the initial stage, i.e., during the integration of monomers. Kim et al. [51] fabricated Nylon 610 nanocomposites with well-dispersed MWCNTs using an in-situ polymerization technique. SEM images (Figure 9) of the fractured surface of MWCNTs/Nylon 610 composite showed that MWCNTs particles were well adhered and embedded in the Nylon 610 matrix. Researchers also improved the compatibility of MWCNTs with Nylon 610 by enhancing the interfacial interaction between both materials, which further enable a uniform dispersion of MWCNTs into the Nylon 610 matrix. In addition, scientists reported that mechanical and electrical properties were significantly increased with the addition of a small amount (0.1 wt%) of MWCNTs.

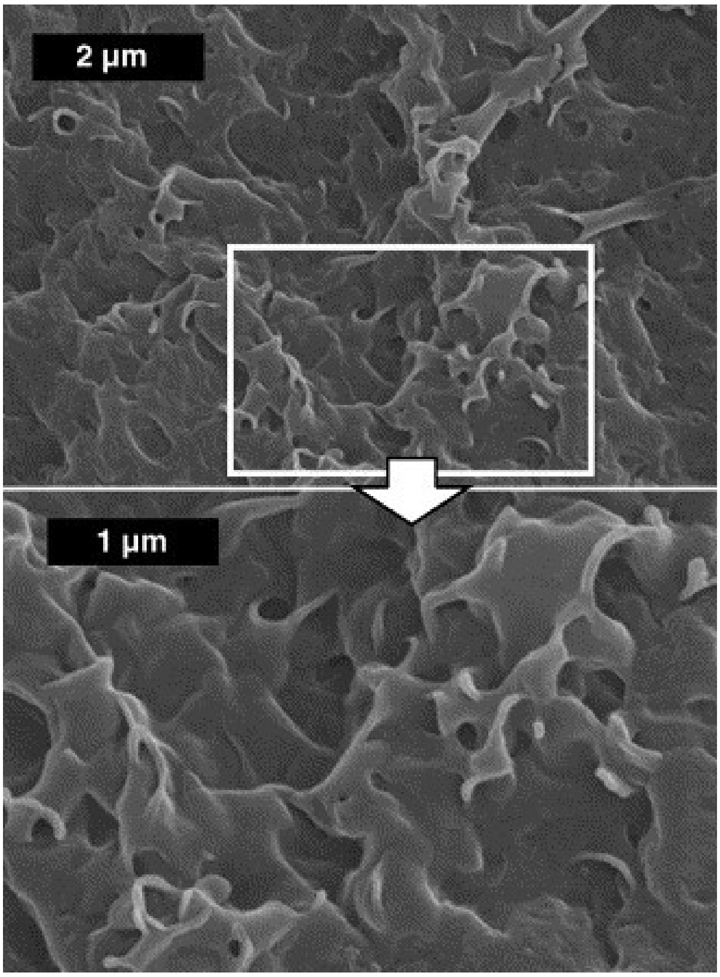


Figure 9. SEM images of the fractured surface of MWCNTs/Nylon 610 composite (Reproduced from Ref. [51] with permission).

The effect of MWCNTs on the properties of Nylon 610 was observed by evaluating the mechanical and electrical behavior of the synthesized composite material. As summarized in Table 3, tensile strength and Young's modulus were observed as 33.5 and 886 MPa, respectively. These values increased significantly by showing an increment of 31% and 52.5% after the incorporation of a small amount of MWCNTs (0.1 wt%). This enhancement in the mechanical properties was an indication of improved toughness and resistance to deformation of the composite material. The researchers reported that during interfacial polymerization, the clusters of MWCNTs were disentangled, which further led to even dispersion of MWCNTs, resulting in improved properties of the material. Conversely, the elongation of the material was reduced considerably, which indicated that the flexibility of the composite was decreased. As MWCNTs are electrically conductive, even a small amount (0.1 wt%) of these particles was sufficient to increase the electrical conductivity by a margin of 5 times. The authors discussed that a certain value of electrical conductivity was needed in the context of electromagnetic interference shielding and electrostatic discharge. A minimal composition of MWCNTs was enough to raise the electrical conductivity from 2.1×10^{-17} S/cm for pure Nylon 610 to 6.1×10^{-12} S/cm for 0.1 wt% MWCNTs/Nylon 610 composite. The researchers further revealed that even dispersion of MWCNTs was the most important parameter to achieve overall improved properties of the material. Another report revealed the successful preparation of polymethyl methacrylate (PMMA) at varying loading of copper oxide (CuO) using in-situ polymerization. Mechanical and morphological studies were performed on these composite materials, and it was reported that the inclusion of CuO into PMMA improved the glass transition temperature and thermal stability of the material. Furthermore, modulus, impact strength, and mechanical strength of the material were also enhanced. Moreover, increasing filler content, temperature, and frequency resulted in a rise in electrical conductivity of PMMA [52]. A group of authors [53] produced CNTs (both MWCNTs and SWCNTs) reinforced PU composites by using in-situ polymerization and reported homogenous dispersion of CNTs in PU. Fourier transform infrared spectra (FTIR) revealed that the introduction of CNTs increased the degree of separation of PU. It was observed that with increasing SWCNTs content, the storage modulus (E') was also increased owing to the restriction imposed on molecular motion because of the well-dispersed SWCNT particles. On the contrary, the microphase separation ascribed to the presence of SWCNTs (as revealed by FTIR) resulted in the decrement of T_g as fewer hard segments were present to obstruct the motion of soft molecules. The results of tensile characterization showed that MWCNTs and SWCNs had different effects on the mechanical performance of the PU. For 2 wt% MWCNTs, the tensile modulus at 50% strain was enhanced by nearly 38% but elongation remained unchanged. However, for 2 wt% SWCNTs, the modulus increased slightly, but a significant rise in tensile strength (60%) and elongation (42%) was observed. It was noted that the introduction of MWCNTs was more beneficial for improving the modulus, while the effect of SWCNTs was more pronounced in improving the elongation of the material. This difference in effect on mechanical performance was attributed to the different rheological behavior of both filler materials. Moreover, minor compositions of MWCNTs and SWCNTs (1 wt%) were sufficient to enhance the thermal conductivity of PU by a margin of 21% and 42%, respectively, in equivalence to neat PU. This noteworthy rise in thermal conductivity was attributed to their excellent experimental value of 6600 $W/m^{-1}/k^{-1}$ [54] for SWCNTs and 3000 $W/m^{-1}/k^{-1}$ [55] for MWCNTs.

Table 3. Mechanical properties of MWCNTs reinforced Nylon 610 composites [51].

Material	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)	Conductivity (S/cm)
Nylon 610	33.5 ± 1.2	886 ± 12.4	11.9 ± 1.4	2.1×10^{-17}
Nylon 610/MWCNTs (0.1 wt%)	43.9 ± 1.1	1352 ± 11.8	4.5 ± 0.8	6.1×10^{-12}

Wang et al. [56] synthesized CNT/polyaniline (PANI) thermoelectric composites with the combination of in-situ polymerization and electrospinning techniques. The process of polymerization aniline and ammonium peroxydisulfate (APS) was executed in the presence of MWCNTs having a diameter of 20–30 nm and a length of approximately 30 μm. For comparison, CNT mixed PANI powder was also prepared by the direct mixing of CNT and denoted by DM-CNT/PANI. The powder of PANI used for direct mixing was fabricated by the same in-situ technique but without adding CNT (10, 20, 30, and 40 wt%) during polymerization progression. The electrospinning method was used for the fabrication of three types of fibers, i.e., pure PANI, CNT/PANI, and DM-CNT/PANI. Before proceeding to electrical characterization, these materials were characterized for morphological studies by using SEM and transmission electron microscopy (TEM) (Figure 10).

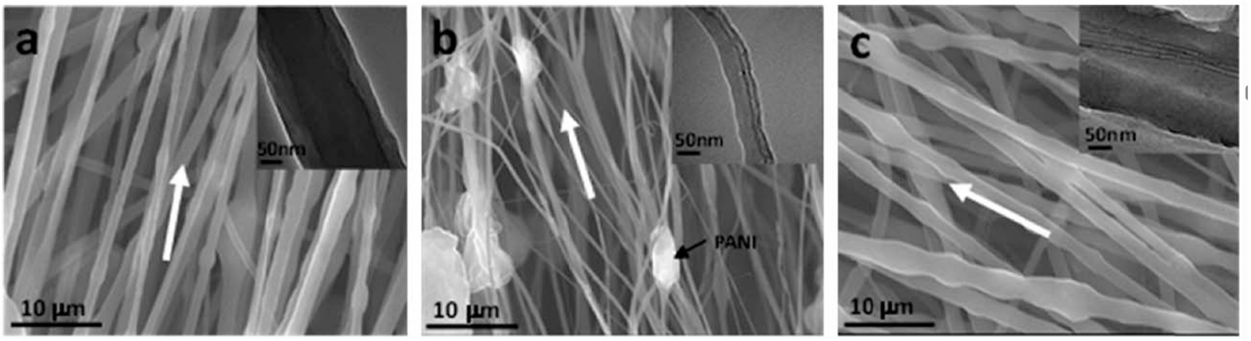


Figure 10. SEM image of fiber of (a) PANI, (b) DM-CNT/PANI (40 wt%), and (c) CNT/PANI (40 wt%). The inserts are the TEM images of a single fiber (Reproduced from Ref. [56] with permission).

It was noted that some segregated particles of PANI were present in the DM-CNT/PANI composite (Figure 10b). Therefore, less homogeneity in the dispersion of CNT was observed for DM-CNT/PANI composite due to the presence of segregated PANI molecules. In contrast, for CNT/PANI fibers, no such segregation of PANI particles was seen, which led to the uniform dispersion of CNT, and also CNT were aligned parallel to fibers' axis. Electrical conductivity (σ) and Seebeck coefficient (S) were the other properties considered for evaluation by using CNT-reinforced PANI composites. For CNT/PANI composites fabricated by in-situ polymerization, results showed that both σ and S increased with the increasing content of CNT. At 40 wt% composition of CNT, σ and S were up to 17.1 S cm^{-1} and 10 µV k^{-1}, respectively, which were 10 and 2 times higher when compared with neat PANI. During the process of polymerization, the molecular arrangement of PANI was improved by π–π interaction between CNT and PANI, which further led to an increment in the carrier mobility and, as a result, conductivity and Seebeck coefficient were increased [57]. In another attempt, scientists used in-situ polymerization for the synthesis of MWCNTs reinforced PU composites after treating MWCNTs with nitric acid and silane acid as a coupling agent [58]. The researchers reported that by varying the composition of MWCNTs from 0.5 to 3 wt%, the overall performance of the material was significantly improved. At 2 wt%, dispersion of MWCNTs was uniform and there was a little agglomeration. By adding MWCNTs, the glass transition temperature of the material was enhanced, and the results of the TGA study showed that the thermal stability of the material was improved. Furthermore, a minor wt% of MWCNTs (0.5 wt%) was found to be sufficient for improving the mechanical properties of the composite material. In comparison to unfilled PU, the impact strength, elongation rate at break and tensile strength of MWCNTs filled PU were increased by 93.8%, 23.5% and 25.1%, respectively. The excellent properties of MWCNTs and their better load transfer properties were suggested reasons for the noticeable improvement in the properties of the material. Additionally, it was reported that by increasing MWCNTs loading up to 1 wt%, the volume resistivity of the material decreased dramatically, but further addition of MWCNTs (beyond 1 wt%) showed that the resistivity reached a stable value and no further change was observed. This indicated that the electrical conductivity of the material was improved after the addition of MWCNTs, and the percolation threshold was about 1 wt%.

The synthesis of a novel composite of PU/epoxy (PU/EP) was prepared with the reinforcement of graphene oxide nanosheets (GONS) using the in-situ polymerization technique [59]. Initially, a polymer of PU was synthesized by varying the composition of GONS. After that, EP and a curing agent were added to prepare PU/GO/EP nanocomposites. The morphological analysis showed that GONS were uniformly dispersed into the PU/EP matrix, and a strong interfacial interaction between GONS and the PU/EP matrix was formed. A minor content of GONS (0.066 wt%) was sufficient to increase the tensile modules of PU/GO/EP from 218 to 257 MPa. Additionally, an improvement of 52% and 103% was reported in the tensile strength and elongation at break, respectively, for PU/GO/EP composites. Furthermore, Thermogravimetric tests showed that the addition of GO into the PU/EP matrix improved the thermal stability of the PU/GO/EP nanocomposite material. This could be due to the involvement of the high aspect ratio of GO particles acted as a barrier and constrained the release of small molecules, leading to better thermal conductivity. Another group of researchers [60] also fabricated polypropylene (PP) composites via the in-situ polymerization approach at varying compositions of MWCNTs (0.1 to 8 wt%). Investigators evaluated the thermal behavior of PP composites in the context of crystallization temperature and reported an increased value of crystallization temperature with an increase in MWCNTs. The presence of MWCNTs as nucleating agents led to crystallization occurring at a higher temperature than that of pure polymers. The improvement achieved in properties by PMCs for the sample fabricated by in-situ ploymerization is presented in Table 4.

Table 4. Properties of materials fabricated by in-situ polymerization.

Polymer	Filler	Tensile strength (MPa)	Hardness (MPa)	Modulus (MPa)	Toughness (MPa)	Electrical conductivity (S/cm)
Nylon [51]	MWCNTs	43.9	-	1352	-	6.1×10^{-12}
PC [53]	MWCNTs	9.11	-	5.13	-	-
PANI [56]	CNT	-	-	-	-	17.1
PU [58]	MWCNTs	15.58	-	-	-	-
PU/epoxy [59]	GO	27.6	-	297	-	-
Polyamide [61]	MWCNT	131.1	-	3400	10.6	1.6×10^{-6}

2.3. Melt mixing

Melt mixing of polymer composite material has also drawn much attention as it can produce mechanically and thermally improved polymeric composite materials even at a low percentage of filler material [62]. This method involves melting and processing of the material using a twin screw extruder. This extruder used for the dispersion of filler material into polymer matrix by maintaining some parameters such as melting temperature, running time, and rpm of the screw. Chen et al. [63] used melt mixing process to reinforce MWCNTs into PU matrix by varying the composition of MWCNTs and they used twin screw extrusion machine for the mixing of both materials. Results of SEM images (Figure 11) indicated homogeneous dispersion of MWCNTs throughout the polymer matrix, and a strong interfacial bonding between MWCNTs and polymer was responsible for the improvement in mechanical properties of composites. Young’s modulus for pure PU was 4.96 MPa, which peaked to 135.1 MPa for the highest loading of MWCNTs, showing a dramatic increment of 27 times. Moreover, tensile strength for composites containing 9.3 wt% MWCNTs was observed as 2.4 times higher than that of pure PU. Researchers further concluded that the significant improvement in the mechanical properties was achieved without sacrificing the elongation at break.

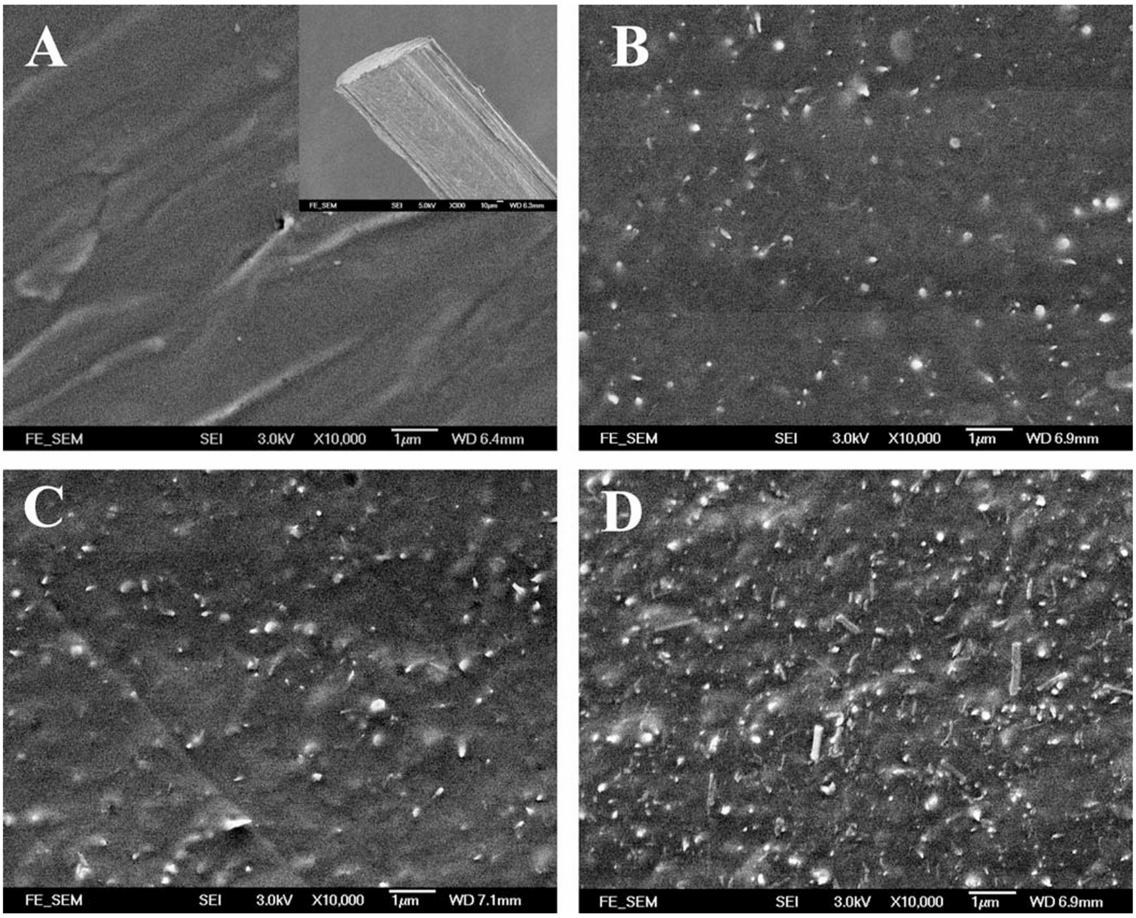


Figure 11. SEM image of (a) pure PU, and PU composite fibers containing (b) 5.6, (c) 9.3, (d) 17.7 wt% MWCNTs (Reproduced from Ref. [63] with permission).

Jindal et al. [64] fabricated acrylonitrile-butadiene-styrene (ABS) based composites at different compositions of MWCNTs (3, 5, and 10 wt%) by using the melt mixing method. SEM image (Figure 12) for 10 wt% ABS/MWCNTs composite showed the reasonable dispersion of MWCNTs through the ABS matrix.

Under quasi-static loading, it was revealed that for 10 wt% composition, hardness and elastic modulus of ABS/MWCNT composite materials were improved by 49% and 61% when compared to pure ABS. Scientists also performed Dynamic Mechanical Analysis (DMA) and examined the specimens under dynamic loading conditions by varying the frequency of load up to 200 Hz, and the results of DMA indicated a significant improvement in the storage modulus of the composite at a composite of 10 wt% of MWCNTs. The value of storage modulus for 10 wt% composite was greatly improved by nearly 58% to 75% for a frequency range up to 200 Hz, in equivalence to pure ABS. Researchers further suggested that better load transfer properties of carbon particles and their adhesion with the polymer matrix were the reasons for improved mechanical behavior under both quasi-static and dynamic loading conditions.

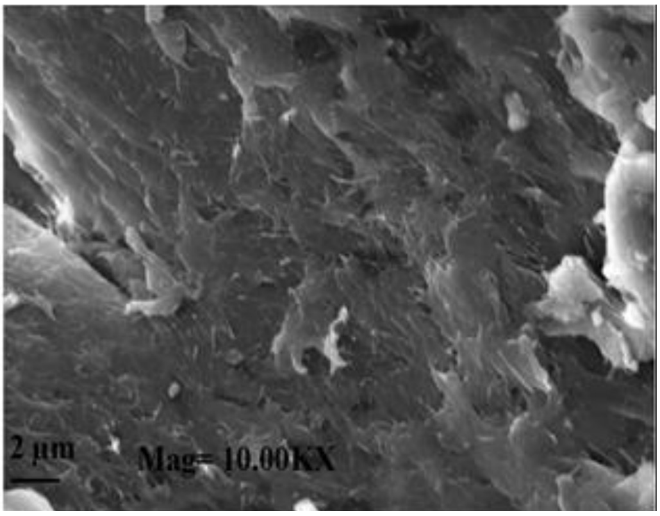


Figure 12. SEM image of 10 wt% ABS/MWCNTS composite at a scale of 2 µm (Reproduced from Ref. [64] with permission).

The melt mixing approach was also explored by Liu et al. [65] for the formation of MWCNT reinforced Nylon 6 (PA6) nanocomposites with different reinforcement of MWCNTs by using a Brabender twin-screw mixer at a speed of 100 rpm for 10 min by maintaining the temperature at 250 °C. Thin films of composites with a thickness of 0.5 mm were fabricated using a compression moulding machine at a pressure of 150 bar and a temperature of 250 °C. TEM revealed fine and even dispersion of MWCNTs throughout the Nylon 6 matrix. SEM images of the fractured surface (Figure 13) of composite materials not only reconfirmed the homogeneous dispersion of MWCNTs in the Nylon 6 matrix, but also evidenced a strong interfacial interaction of MWCNTs with the matrix.

Composite specimens with different compositions of MWCNTs (0, 0.2, 0.5, 1, and 2 wt%) were prepared and their mechanical properties were evaluated using different tests, i.e., tensile testing, nanoindentation, and DMA. The DMA studies were performed by varying the temperature of specimen in the range of −100 to 200 °C. The results of mechanical properties are summarized in Table 5.

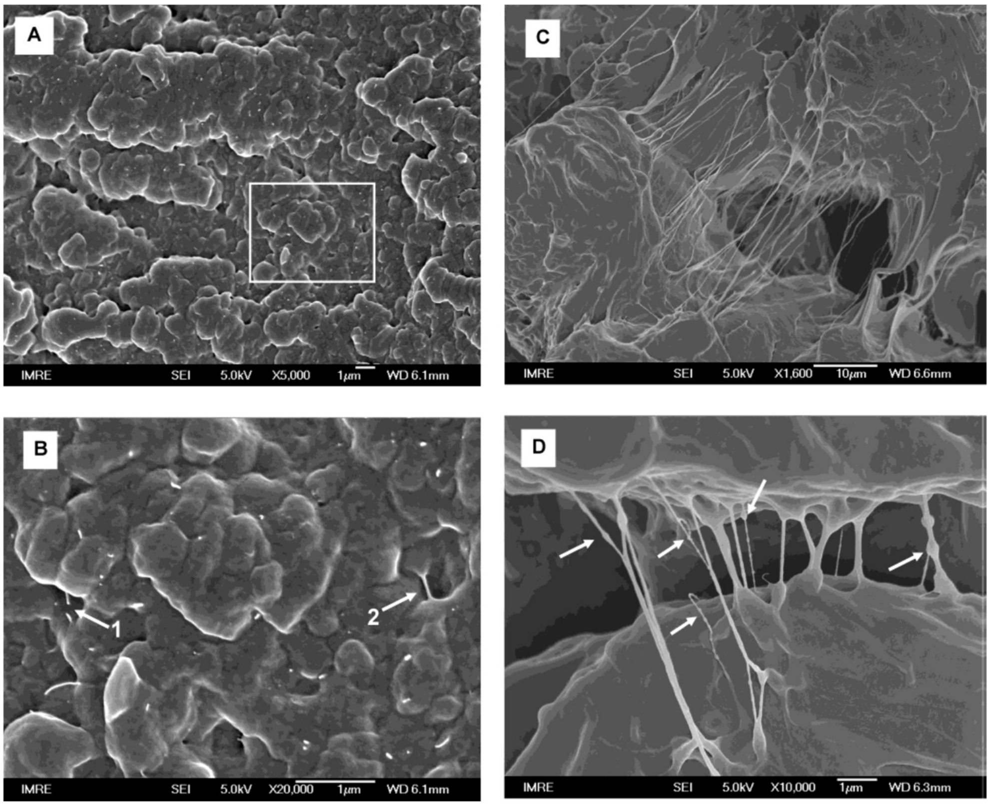


Figure 13. (a) SEM image of failure surface with 0.5 wt % MWCNTs in PA6, (b) enlarged SEM of the selected region, (c) micro cracks linked by stretched nanotubes, and (d) enlarged SEM image of stretched nanotubes (Reproduced from Ref. [65] with permission).

Table 5. Mechanical properties of PA6 at different compositions of MWCNTs [65].

Mechanical properties	Neat PA6	PA6/MWCNTs (99.8/0.2)	PA6/MWCNTs (99.5/0.5)	PA6/MWCNTs (99/1)	PA6/MWCNTs (98/2)
Yield strength (MPa)	18 ± 2.2	33.7 ± 1.3	35.4 ± 0.3	40.3 ± 3.1	47.2 ± 1.2
Tensile modulus (MPa)	396 ± 36.6	682.5 ± 65.0	765.5 ± 45.9	852.4 ± 77.0	1241.8 ± 22.8
Modulus (GPa)	2.08	2.55	2.57	3.14	2.97
Modulus (GPa)	1.18 ± 0.02	1.33 ± 0.03	1.60 ± 0.12	2.02 ± 0.02	-
Hardness (GPa)	0.06 ± 0.001	0.08 ± 0.005	0.10 ± 0.005	0.11 ± 0.003	-

It was reported that the melt mixing method was efficiently used for the production of MWCNT reinforced PA6 composites with improved mechanical properties. With the addition of 2 wt% of MWCNTs, the elastic modulus ramped from 396 to 1241.8 MPa by showing an enormous rise of 241%, while the yield strength rose from 18 to 47.2 MPa, which was about 162% higher when compared to neat PA6. Furthermore, it was also seen that with increasing content of MWCNTs, the storage moduli of the composite material were also increased. At 0 °C, the value of storage modulus for neat PA6 was observed as 2.08 GPA, which ramped to 2.97 GPa for 2 wt% composites, which was 43% higher, and this value further increased by 57% when the temperature rose to 120 °C. The noticeable improvement in the value of storage modulus was attributed to fine dispersion, excellent mechanical strength, and high aspect ratio of the MWCNTs. Another interesting study reported the synthesis of MWCNTs or MWNT reinforced polyethylene (PE) with PC and polyamide (PA6) by using a melt mixing approach [66]. Different high loadings of MWNT (24, 35, and 44) were incorporated into the PE matrix (PE used as a carrier component for MWNT) during in-situ polymerization, and the final composites for PC and PA6 were prepared using a twin screw extruder.

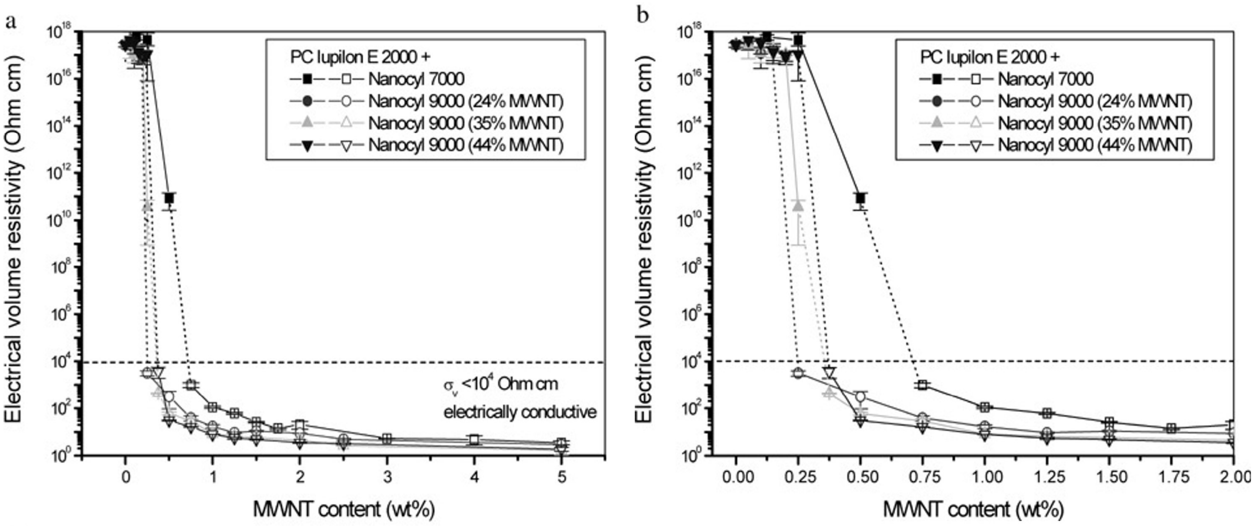


Figure 14. Electrical volume resistivity versus MWNT* (MWNT refer to MWCNTs) content comparing the use of (a) pure MWNT material and (b) MWNT concentrates in PE (Reproduced from Ref. [66] with permission).

For PC and PA6 composites, the temperature of the extruder was kept at 280 and 240 °C, respectively, and the extruder was rotated for 15 min at a speed of 50 rpm. The extruded material was then moulded into sheets with a diameter of 60 mm and a thickness of 0.35 mm, and these sheets were used for electrical characterization. Morphological characterization results indicated that MWNTs were evenly dispersed into the PE polymer, and this dispersion was transferred to the matrix polymer in order to get a uniformly dispersed composite material. Scientists claimed that the same technique may be used for other polymers that possess polar behavior and are incompatible with PE. Electrical results showed that the improved dispersion of MWNT via this novel method resulted in the reduction of electrical thresholds in comparison to the direct inclusion of the same CNTs under the same experimental settings. As shown in Figure 14, the lowest concentration of MWNT (24 wt%) caused the lowest electrical percolation thresholds, while the concentration with a higher content of MWNT (44 wt%) resulted in a lower value of resistivity. Potschke et al. [67] prepared melt-mixed composites using two different techniques for incorporating CNTs into a polymer matrix. In the first method, master batches of CNTs/PC composites, which were commercially available, were diluted during melt mixing with the use of different kinds of pure PC (powder and granules), whereas in the second case, CNTs were directly incorporated into PC using a small-scale micro compounder. In the first case, for the controlled dilution process, the MWNT should be dispersed homogenously in the polymer. The results of electrical studies showed that for all types of PC material used, the percolation started with the loading of MWNT in the range of 0.875 to 2 wt%. Therefore, the authors concluded that that composites with 1.5 wt% were electrically conductive, as an even distribution of MWNT was achieved at this minimal concentration. In the second case, the SEM images (Figure 15) showed that the direct incorporation of MWNTs resulted in the next to good distribution of MWNTs.

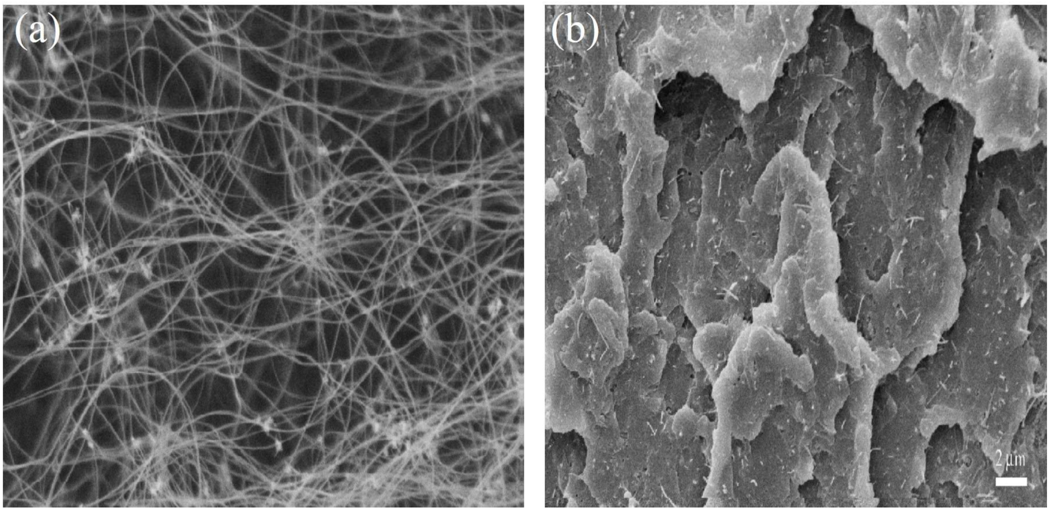


Figure 15. SEM of (a) pure MWNT and (b) MWNT/PC composite at 1.5 wt% (Reproduced from Ref. [67] with permission).

The micrographs of pure MWNT (Figure 15a) showed that these tubes are quite straight, less tangled, and relatively long, and it was assumed that some of these long tubes were broken during melt mixing due to a high viscosity of the polymer. The SEM image (Figure 15b) of the composite material revealed that no agglomeration was observed in the polymer matrix. In this case, electrical percolation was initiated at 3 wt% of MWNT. The mechanical characterization revealed that after reinforcing MWNT directly, the Young's modulus was increased while elongation at break was reduced dramatically. Table 6 shows the property enhancement in PMCs fabricated by the melt mixing method.

Table 6. Properties of materials fabricated by the melt mixing method.

Polymer	Filler	Tensile strength (MPa)	Hardness (MPa)	Modulus (MPa)	Toughness (MPa)	Electrical conductivity (S/cm)
PU [63]	MWNTs	120	-	135.1	-	-
ABS [64]	MWCNTs	-	223	3311	-	-
Nylon 6 [65]	MWNT	47.2	-	1241.8	-	-
PP [68]	MWCNT	-	-	5300	-	4.3×10^{-2}

Bhagat et. al. [69] developed electrically conductive MWCNT/PP/high-density polyethylene (HDPE) nanocomposites via the melt mixing method using two different heating treatment methods. In the first method, the composites of PP (70%), HDPE (30%) were prepared at varying compositions of MWCNTs by mixing all three materials in an internal mixer with two screws rotating in opposite directions. The mixing was carried out at 200 °C and 40 rpm. In the second method, the three materials with a similar amount were initially melt mixed at 140 °C for 10 min, and after that, the temperature was raised to 200 °C. For comparison analysis, composites of PP and HDPE were also prepared with both methods. The researchers observed that at 1 wt% of MWCNTs, PP/HDPE composites prepared by the second method displayed a better conductive nature than those prepared by the first method. This might be due to the formation of an effective MWCNT-MWCNT network in the second case, which led to good conductivity.

2.4. Additive manufacturing

These types of techniques were also explored by researchers for the fabrication of CNT-reinforced polymer composite materials. Additive manufacturing not only produces precise parts but also enables the synthesis of highly customized parts with excellent mechanical, electrical, and thermal properties [70]. In comparison to very high temperatures involved in other fabrication methods, these additive manufacturing techniques, such as selective laser sintering (SLS), deal the approaches to synthesize complex components with desired mechanical, thermal, and electrical properties by using composite powder material. Other 3D printing techniques, such as fused deposition molding (FDM), have also gained popularity for the synthesis of polymer composite material [71]. To combine filler material into a polymer matrix, twin screw wire extrusion process can be used, which fabricates the required composite wire for the FDM machine [72]. Researchers [73] presented a powder-based SLS method to prepare electrically conductive material with complex geometry. The composite powder of two polymers, i.e., polyamide 12 (PA12) and PU, was prepared by varying the concentration of CNTs from 0.1, 0.5, and 1.0 wt%. A comparative study for electrical conductivity was made, and it was revealed that at 0.5 wt % loading of CNTs, sintered composites showed higher electrical conductivity than that fabricated via hot moulding techniques. Furthermore, it was also claimed that the direct current (DC) conductivity for CNT/PU sintered composites was higher than that of CNT/PA12 sintered composites. A small composition of CNTs (0.5 wt%) was sufficient to enhance the electrical conductivity of PU and PA12 to 10^{-4} and 10^{-5} S/cm, respectively. In addition, the researchers also discussed that the composites of both CNT/PU and CNT/PA12 fabricated by sintering possess lower thermal conductivity than that of synthesized via compression moulding. The laser sintered CNT/PU composites due to porous micro-channels had highly porous structure which easily dissipated energy eliminating phonon propagation, which further led to poor thermal conductivity. At 1 wt% loading of CNTs, the thermal conductivity of hot-compressed PU and PA12 composites increased to 4.28 and 16.9 W/km, respectively. The reason for lower thermal conductivity of laser sintered polymer composites was the presence of pores and cavities in the polymer matrix; therefore, it was difficult to synthesize fully condensed specimens, as sintering is a pressure-less technique. These cavities contributed to the reduced thermal conductivity by prohibiting the thermal wave propagation.

In another study, scientists used 3D printing techniques to prepare ABS and MWCNTs composites [74]. Two composites were prepared for the mechanical characterization, and the results were compared with pure ABS filament. In the first case, MWCNTs were dispersed in the ABS matrix by using a melt mixing approach, and pallets were obtained, which were further processed in a filament maker and passed through a screw driven by a motor. In the second case, an airbrush filled with MWCNTs dispersed in solvent was used to spray MWCNTs on the ABS filament. Tensile results showed that pure ABS filament had a tensile strength of 41 MPa, which peaked to 51.5 and 62 MPa for MWCNTs dispersed ABS and MWCNTs coated ABS, respectively. While tensile modulus for MWCNTs dispersed ABS and MWCNTs coated ABS was 2125 and 2350 MPa, respectively, which was higher than that of pure ABS (1955 MPa). Researchers also performed flexural and impact testing on the specimens and revealed that the incorporation of MWCNTs into ABS (dispersion and coating) resulted in a significant enhancement in flexural modulus and impact strength.

A scalable method was developed by Vidakis et al. [75] for the development of multifunctional filaments of composite material. The powder of MWCNTs enriched polylactic acid (PLA) polymer was synthesized using a mechanical mixing method. After that, suitable filaments of these composites having a diameter of 1.75 mm were prepared using a single screw extruder machine. The concentrations of MWCNTs were kept as 0.1, 0.5, 1.0, 2.5, and 5.0 wt%, and the effect of this filler addition on the mechanical, electrical, and thermal behavior of the PLA was evaluated. The tensile characterization of the specimens indicated that with the addition of MWCNTs up to 1 wt%, the tensile strength and tensile modulus increased significantly. However, further addition of MWCNTs beyond 1 wt% revealed that the values of these properties reduced significantly and then obtained an optimal value. At 1 wt%, significant improvements of 34% and 33% were observed in tensile strength and modulus, respectively. The results of flexural studies showed that the flexural strength and modulus for pure PLA were observed as 66.8 MPa and 2.24 GPa, respectively. With the addition of 5 wt% MWCNTs, these values further increased to 99.5 MPa and 3.21 GPa, respectively. The possible reason for this improvement could be that the presence of MWCNTs as nucleating agents possibly increased the crystallinity of PLA. The researchers also claimed that the involvement of MWCNTs in to PLA matrix increased the conductivity of the PLA. The Thermogravimetric analysis of these composites revealed that all PLA/MWCNTs nanocomposites were thermally stable at 270 °C, and no weight loss was observed at this temperature. However, a further temperature rise resulted in the thermal degradation of the composite, and beyond 390 °C, all the composite specimens were fully decomposed.

Other researchers focused on the development of CNT and graphene nanoparticles (GNP) reinforced polyether ether ketone (PEEK) composites filament by using fused filament fabrication (FFF) method [76]. To confirm uniform dispersion of the filler material, stir mixing was used before proceeding to melt compounding. Subsequently, twin screw extruder was used to prepare filament with a diameter of 1.75 mm. The DSC results showed that in comparison to unreinforced PEEK, the melting and the crystallization peaks were shifted toward higher temperatures. The melting temperature of GNP (3 wt%) and CNT (1 wt%) reinforced PEEK was increased by 1 and 2 °C, respectively, while the crystallization temperature was improved by 3–4 °C and 5–6 °C, respectively. This increment indicated that the presence of the CNT and GNP in PEEK eased its crystallization. However, further increment in wt% of reinforcement material resulted in negligible rise in degree of crystallinity, which was ascribed to the agglomeration of GPN and CNT. Thermal expansion behavior of the composite material was also evaluated, and it was observed that below the glass transition temperature, the coefficient of thermal expansion (CTE) was reduced by 16% and 26%, respectively, for 3 and 5 wt% GNP/PEEK nanocomposites. On the other hand, the incorporation of CNT also reduced the CTE of PEEK but in a lesser extent. For 1 and 3 wt% CNT-enriched PEEK, the CTE was lessened by 9% and 18%, respectively. The tensile characterization showed that the Young's modulus of the pristine PEEK was found to be 3.15 GPa, which ramped to 3.89 and 3.77 GPa for GNP/PEEK (5 wt%) and CNT/PEEK (3 wt%), respectively. Singh et al. [77] prepared MWCNTs reinforced ABS composites via twin screw extruder method and filament having 1.75 mm diameter was prepared for 3D printing and claimed that additive manufacturing can enhance the mechanical behaviour of the 3D printed entities. It was reported that by adding 1, 2, and 3 wt% of MWCNTs, the mechanical properties of ABS were significantly improved. Another report presented the results of mechanical and electrical properties of 3D printed MWCNTs reinforced ABS composites by using FDM techniques [78]. The dispersion of MWCNTs was achieved using a twin screw micro compounding extruder equipped with a back-flow channel capability. The lumps of composite were further processed in a single screw extruder to produce a filament of 1.75 mm diameter. At 7 wt % loading of MWCNTs, tensile strength of the composite material was improved by 288% when compared with unfilled ABS. However, a further surge in MWCNTs concentration resulted in the transition of the material from ductile to a brittle nature. Furthermore, for higher loading of MWCNTs (10 wt%), the electrical conductivity was found to be 7 times than that of pure ABS. Another interesting observation regarding the measurement of fluidity of the material was made using Melt Flow Index (MFI) techniques. In this method, a constant load was applied to push the molten material through a standard orifice [79]. It was noted that for samples having 10 wt% MWCNTs, the value of MFI was 164 times lower than that of pure PU. A remarkable point was revealed that this reduced MFI of the composite material made the filament extrusion process more problematic because of the breaking of the filament.

2.5. Advantages and disadvantages of composites prepared by different methods

Each synthesis method has its advantages and limitations. For example, the solvent blending method is simple in use and the quality of dispersion of filler particles is excellent, but it may involve the use of toxic solvents [80]. The removal of these solvents is time-consuming, and there is a possibility of some residual, which may affect the overall performance of the material. In-situ polymerization results in outstanding matrix-filler interaction, but it is a time-consuming process and often requires high expertise [81]. The prime benefits of melt mixing are that it is solvent-free and can be scaled up, but it requires a higher temperature, which can degrade the quality of the product. Additive manufacturing, on the other hand, is helpful for generating complex geometries and customized products, but poor interfacial bonding often results in limited mechanical properties.

2.6. Applications of polymer composites

Polymer composite materials having a polymer as their base matrix are applicable in a variety of engineering fields due to their light-weight, better strength-to-weight ratio, corrosion resistance, and lower initial and maintenance costs. The overall properties of these composite materials are found to be superior to their parent materials. These improved properties of polymer-based composites make them ideal materials for the aerospace and aviation industry [41], automobile engineering [82], electrical and electronics, sports industry, and for the synthesis of biomedical implants. Some key benefits of using polymer-based composite materials for engineering applications include improved fatigue and corrosion resistance, which further enhances the service life of the components. In addition, the light weight of these composites helps in reducing the overall weight of the final part. Moreover, the use of polymers for the development of engineering products provides good design flexibility with tailored mechanical and thermal properties [83].

3. Discussion

In this review, we have synthesized a broad range of research on the fabrication of polymer composites, focusing on the incorporation of carbon-based nanomaterials like CNTs and graphene to enhance mechanical and thermal properties. The studies examined highlight the potential of these composite materials across applications, from automotive and aerospace to biomedical and athletic gear.

This review has also specifically established the role of fabrication techniques in the properties of PMCs. The solvent casting, in-situ polymerization, and melt mixing have all demonstrated efficacy in producing polymer composites with improved mechanical strength, stiffness, and thermal stability. Additive manufacturing techniques are also emerging as promising methods for creating complex and customized composite parts.

The reviewed studies highlight that the key role of fabrication techniques in achieving uniform dispersion of the reinforcing filler within the polymer matrix is crucial. It is also observed from various reports that agglomerations of CNTs or graphene can lead to diminished mechanical performance and inconsistent properties [30–32]. Therefore, optimizing fabrication parameters to ensure adequate dispersion is a key challenge in the field.

The concentration of the reinforcing filler is another important factor [59,71]. While increasing the filler content generally enhances mechanical properties up to a certain point, exceeding an optimal concentration can lead to agglomeration and a decline in performance. This optimal loading varies depending on the polymer, filler, and fabrication method.

3.1. Comparison of fabrication techniques

Each fabrication technique presents its own set of advantages and limitations:

- Solvent castings enable good control over filler dispersion but involves the use of solvents, which can have environmental and safety implications [34].
- In-situ polymerization can promote strong interfacial bonding between the filler and the polymer but may be limited to specific polymer systems [52].
- Melt mixing is a scalable technique suitable for industrial production but may face challenges in achieving uniform dispersion, especially at high filler loadings [67,68].
- Additive manufacturing offers design flexibility but can result in porous structures that affect thermal and mechanical properties [71,70].

3.2. Future directions

While significant progress has been made in the development of polymer composites, several areas warrant further investigation:

- Focus is needed to address dispersion challenges. Novel strategies are needed to improve the dispersion of nano-fillers in polymer matrices, particularly at high loadings, to fully capitalize on their reinforcing potential.
- New development is occurring for the modeling of composite materials; though quite significant progress has been made in modeling pure materials. Still, computational modeling can play a greater role in predicting composite behavior and optimizing fabrication processes for PMCs.
- More work is needed in the field of life cycle assessment. Evaluating the environmental impact of different fabrication techniques and composite materials is essential for sustainable development.
- PMCs have taken front stage in many applications. Exploring new applications for polymer composites in emerging fields such as flexible electronics, energy storage, and biomedical devices.

4. Conclusions

The present review highlights on following key findings:

- Uniform dispersion is crucial for the performance of PMCs. Across all fabrication techniques, the most critical factor influencing the mechanical, thermal, and electrical properties of polymer composites is the uniform dispersion of carbon nanofillers (like CNTs and MWCNTs) within the polymer matrix. Agglomeration significantly hinders performance improvements.
- The major onus of property enhancement lies in the fabrication method. Different fabrication techniques (solvent casting, in-situ polymerization, melt mixing, and additive manufacturing) impact the final properties of the composites. Each method has unique advantages and challenges related to filler dispersion and interfacial bonding.
- The review highlights that significant property enhancements are achievable. When carefully fabricated, polymer composites with carbon nanofillers show substantial improvements in various properties, including:

(1) Mechanical properties: increased Young's modulus, tensile strength, hardness, toughness, and storage modulus.

(2) Thermal properties: enhanced thermal stability and sometimes thermal conductivity (though additive manufacturing can sometimes lead to lower thermal conductivity due to porosity).

(3) Electrical properties: significant improvements in electrical conductivity, even at low filler concentrations.

- Key methods to improve the interfacial interaction are in-situ polymerization and melt mixing. In-situ polymerization often leads to excellent interfacial interaction between the polymer and filler, while melt mixing is effective in achieving fine dispersion and strong bonding, both contributing to superior mechanical properties.
- A wider customization can be achieved by Additive Manufacturing, enabling customization. Additive manufacturing techniques (like SLS and FDM) enable the production of complex geometries and multi-functional composites with improved electrical conductivity. However, challenges such as potential porosity and reduced thermal conductivity can arise depending on the process and materials.

This review effectively highlights our understanding of key fabrication techniques for PMCs, particularly those incorporating carbon nano-fillers, i.e., CNTs. The key parameters required to control for achieving better properties are the degree of filler dispersion, which largely depends on the fabrication technique. While each fabrication method, solvent casting, in-situ polymerization, melt mixing, and additive manufacturing, offers distinct advantages and has demonstrated considerable success in enhancing mechanical, thermal, and electrical properties, they also present unique challenges, primarily related to achieving and maintaining homogeneous filler distribution. Here, we summarize the experimental findings, including quantitative improvements across polymer-filler systems and provide valuable insights for researchers and engineers. The detailed analysis emphasizes the importance of process parameters that influence dispersion and, consequently, material properties are particularly useful for optimizing composite synthesis. This article is a resource for the polymer community, guiding the selection of appropriate fabrication methods based on desired end-use applications and highlighting the persistent need for innovative strategies to overcome dispersion challenges for unlocking the full potential of polymer nanocomposites.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

Author contributions

Dinesh Kumar: conceptualization, writing-original draft, methodology; Amika: investigation, formal analysis, validation; Deepak Kumar: methodology, visualization, resources; Parminder Singh: data curation, resources, analysis; Arvind Singh Chauhan: writing-review & editing, investigation, analysis; Sahil Kapoor: analysis, resources, visualization; Suneev Anil Bansal: main supervision, project administration, writing-review & editing.

Conflict of interest

The authors declare no conflict of interest.

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