Deeraj *et al. Functional Composite Materials* (2024) 5:13
https://doi.org/10.1186/s42252-024-00060-8

Functional Composite Materials

REVIEW

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A brief review on electrospun polymer derived carbon fibers for EMI shielding applications

B. D. S. Deeraj[1*], Rhiya Paul[1] and Kuruvilla Joseph[1*]

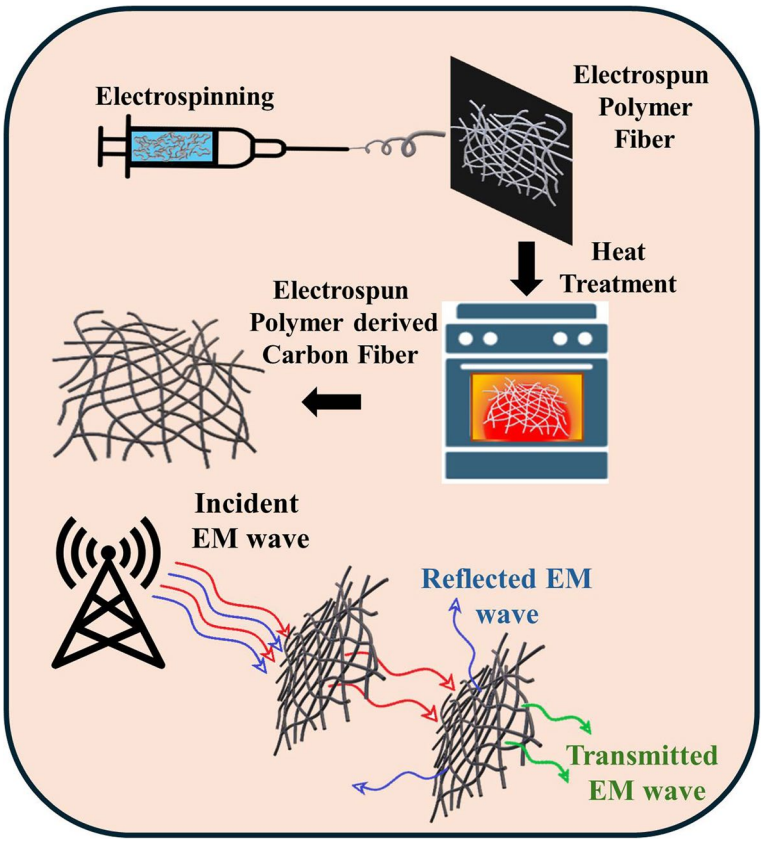
Abstract

The rapid growth of electronic information technology has led to an increase in electromagnetic wave radiation pollution. Therefore, it is imperative to look into shielding materials with superior electromagnetic interference (EMI) shielding capabilities. Because of its exceptional benefits, nanofibers made by electrospinning are capable of the shielding from electromagnetic radiation. This work tries to bring together the details of electrospinning, electrospun carbon and electrospun composite fibers. Furthermore, the preparation, properties and EMI shielding performance of electrospun carbon and composite fibers are presented. This article attempts to provide an overview of the advancements made in the field of carbon fibrous material prepared via electrospinning for shielding applications.

Keywords Electrospinning, Electrospun carbon fibers, Electrospun composite carbon fibers, EMI shielding, Shielding effectiveness

*Correspondence:
B. D. S. Deeraj
deeraj4mech@gmail.com
Kuruvilla Joseph
kuruvilla@iist.ac.in
Full list of author information is available at the end of the article

Graphical Abstract



Introduction

Along with its benefits, technological advancements can sometimes have drawbacks. One such instance is electromagnetic pollution, which is caused by undesired waves that arise from using electronics more frequently. In today's world, our surroundings are full with such stray electromagnetic (EM) wave radiations that would adversely influence the operation of electronic gadgets and are believed to be harmful to living beings. This undesired occurrence is known as electromagnetic interference (EMI), and it is a growing concern these days. The number of electronic gadgets we use or rely on will grow, and the solution to this problem is the development of improved shielding devices capable of blocking this undesired pollution. The concept of protecting against the harmful effects of EMI is known as EMI shielding. The scientific community has stepped up its attempts to create novel shields that enhance shielding effectiveness. [1–10].

The EMI shielding phenomenon is based on the notion of material shields, in which the shielding material blocks the passage of high frequency electromagnetic radiation by reflection or absorption. In basic terms, we use barrier materials as shields that either absorb or reflect the incoming electromagnetic radiation. The more intricate the magnetic and electrical characteristic of the shielding material, the more efficient is the shielding mechanism. The method of shielding depends on numerous parameters, and various shielding materials are being researched, as mention in Sect. 2. Since there are several uses for frequencies in the microwave and radio wave regions, the development of shields in these regions is the primary focus of literature.

This review focuses on electrospun carbon fibers and electrospun composite carbon fibers that are effective for shielding purpose. The Sect. 2 and 3, briefly introduces the concepts of EMI shielding and electrospinning respectively. A brief overview of the enormous potential and possibilities of electrospun fibers is provided. Section 4 and 5 focuses on presenting information regarding the preparation and properties of these

materials and their comparative benefits over other materials. And Sect. 6 presents a brief conclusion for this topic.

EMI Shielding

The scientific community is working to create various materials and their combinations for shielding applications all around the world. We have two mechanisms in which shielding takes place, but researchers are mostly interested in absorption dominated shields than reflection dominated ones. The main reason being that the reflected waves can one again cause 'secondary EMI' scenario, which is not observed in absorption prominent shields.

High magnetization, complex microwave characteristics, and high electrical conductivity are the material section requirements for shielding. Materials for electromagnetic interference shielding are often developed in response to market demands and requirements. The reflection arises from the mismatch in impedance between the air and shield surface [11–14]. Reflection suppresses electromagnetic waves, but for the reasons explained above, it is undesirable. Therefore, it is necessary for EM waves to pass through a shield whose impedance matches that of the air [15]. Thus, the permeability and permittivity of the material are important factors in EM wave penetration [16–18]. Materials with intrinsic dielectric and magnetic losses that can block electromagnetic waves are of interest. The term shielding effectiveness (SE) refers to the capacity of a shielding material to reduce electromagnetic waves (EM). Decibels (dB) are used to express this shielding efficacy. The higher the SE, the greater is the shielding efficiency. The standard criterion for a shield in commercial applications is a SE of at least 20 dB. The total shielding effectiveness of a material (SE_T) is the sum of its shielding effectiveness via absorption (SE_A), reflection (SE_R), and multiple reflections (SE_{MR}).

$$SE_T = SE_A + SE_R + SE_{MR} \quad (1)$$

Four methods are usually used to assess the shield's effectiveness. These comprises of the co-axial transmission line technique, open field method, shielded room method, and shielded box method. A scalar network analyzer can solely measure the signal amplitudes; however, a vector network analyzer (VNA) can determine the signal phases as well as the amplitude. Complex qualities like permittivity and permeability may be measured with VNA. Many reports presents details regarding the shielding theory, testing methods and material requirements for shielding applications [3, 19–28]. In Fig. 1, the representation scheme for shielding is presented.

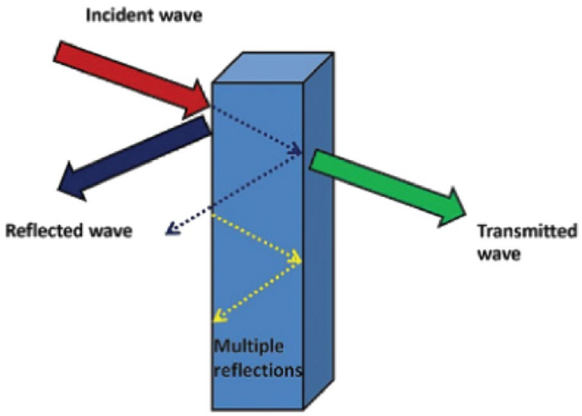


Fig. 1 Mechanism for shielding [8]. Reproduced with permission from Elsevier

Electrospinning

Electrospinning is a process of producing submicron or nano-metered fibers from polymeric solution/polymer melt/ceramic solutions with the aid of electric current [29–38]. Here, a syringe pump is used to feed the polymeric solution into a spinneret at a predetermined pace. The droplets formed at the tip of the spinneret get electrically charged under the applied high voltage. Thus, an electrical repulsion between the charged solution molecules develops inside the droplets [39]. When the repulsive forces exceed the surface tension, spherical droplet deforms into a Taylor cone. Eventually, the cone gets stretched, and a fine-charged jet comes out. Initially, the jet follows a straight line path, then takes a spiral path, and ultimately, gets deposited as fibers, on a ground collector [40].

In the initial phase, the jet has enough acceleration to overcome the Rayleigh instability (due to fluid surface tension), which would otherwise break the jet into droplets [36]. However, as the jet moves forward, its acceleration gets diminished due to the surface tension and the viscoelastic properties of the fluid. Additionally, as the solvent evaporates, the circumference of the jet reduces, leading to the accumulation of charges on the surface, and hence, the electrostatic repulsive forces increase again. Therefore, at this stage, any small perturbations can tamper the stability of the jet, potentially affecting the trajectory of the jet. Two types of instabilities come into play during this point, namely, (i) axisymmetric instability (Rayleigh instability and electric field induced axisymmetric instability) and (ii) non-axisymmetric instability (Bending instability/whipping instability) [41], as depicted in Fig. 2.

The Rayleigh instability causes unwanted bead formation, resulting in defects and discontinuity in fibers, yet

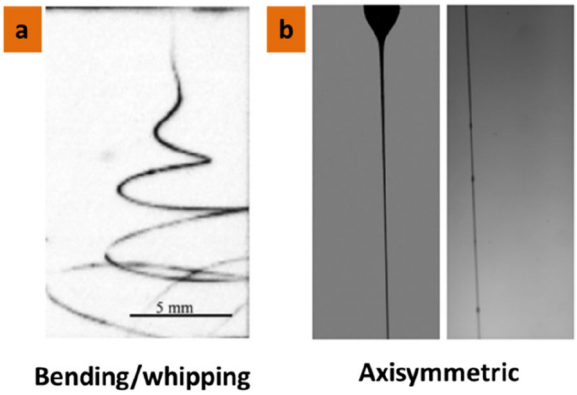


Fig. 2 Types of instabilities in the ejected jet. **a** Reproduced with permission from AIP publishing [42]. **b** Reproduced with permission from Elsevier [43]

this can be overcome using high voltage. This instability is negligible in polymeric solutions of low surface tension. Whipping instability is the most desirable one, as this is critical in the synthesis of nano-fibers [44]. The interplay between aerodynamic forces and lateral electric force cause this instability, i.e., the lateral forces compel the jet to bend and take a spiral trajectory [29]. During this chaotic motion of the fiber jet, the solvent vaporizes, and the jet elongates and stretches tremendously, forming thin, ultra-fine fibers. Thus, the solidified fibers produced during this interval get deposited on the collector. In addition to the instabilities mentioned above, capillary instability and branching stabilities exist during the solidification journey of the jet.

Additionally, several modifications are available in electrospinning that aids to obtain fibers of different types. Hollow spinnerets are used for the synthesis of hollow fibers and multiple spinnerets are used to increase the efficiency of the electrospinning [45]. Coaxial spinnerets (two concentrically placed hollow needles) are utilized for the fabrication of two fibers in a core–shell manner [46]. The immiscible polymeric solutions are preferred for this coaxial electrospinning as they avoid mixing and inversion of the fluids. Another technique, melt electrospinning, involves the direct usage of polymer melt for electrospinning [47]. This method is particularly advantageous for thermoplastics like PP and PE, which are not soluble in typical organic solvents.

Parameters in electrospinning

There are several factors in electrospinning that play an important role in the production of defect-free fiber. They are briefly presented in this section.

Polymeric solution

This parameter highly affects the properties of the electrospun fiber. This includes the nature of polymer and the solvent, their solution concentration, and so on. Furthermore, the molecular weight of the polymers directly influences the viscosity of the solution. Polymers with low molecular weight (Mw) would result in a less concentrated and low viscous solution, which might result in the development of beads/droplets instead of the fibers. This was substantiated by the work of Anandjiwala and co-workers, where they used PEO of different molecular weights, and at a low Mw (3×10^5 g/mol), fibers with beads were formed and at higher Mw (9×10^5 g/mol), more fine fibers with spindles were achieved[48], as shown in Fig. 3. Polymer solubility is also a significant factor in electrospinning. Plenty of studies have already been conducted to investigate the impact of solubility on the morphology and thickness of fibers. It is concluded that if the polymer is highly soluble in a solvent, it results in electrospraying. In contrast, if the polymer is partially soluble in a solvent, it would result in stable electrospun fibers [40].

For instance, Georgiadou and their team studied the effect of different solvents and the solution concentration on the morphology of the electrospun polylactic acid fibers (PLA) produced[49]. They utilized a wide range of single- and binary solvents to obtain PLA fibers. They optimized the binary acetone and dimethylformamide (DMF) solution to produce nano-metered fibers with minimum defects. They also found that the change in PLA concentration also affects the fiber properties. When the PLA concentration is below 10%, the obtained fibers are in beads on the string fashion; however, as the concentration was increased above 10%, defect-free fibers were obtained. As already mentioned, as concentration is increased, chain entanglements are amplified, leading to enhanced viscosity. Thus, the viscoelastic properties of the fibers increase, which can counteract the stretching force, resulting in defect-free fibers. Such concentration effects were also reported with PEO polymer solutions, where fibers with irregularities were obtained at lower concentrations. However, as the concentration was increased, fine nanofibers with regular morphology were acquired. Thus, there is always a minimum concentration of polymer required for forming fibers, known as entanglement concentration (C_e). Above this concentration, beaded fibers start to form and to produce fine fibers, the concentration should be much higher than the C_e.

Another report shows the influence of salts on the electrospun fibers of polyacrylonitrile (PAN) polymer[50]. Here, they have introduced several salts, such as LiCl, NaCl, $CaCl_2$, and $NaNO_3$, into the PAN/DMF solutions prepared at varying concentrations (4, 6, 8, and 10%). The

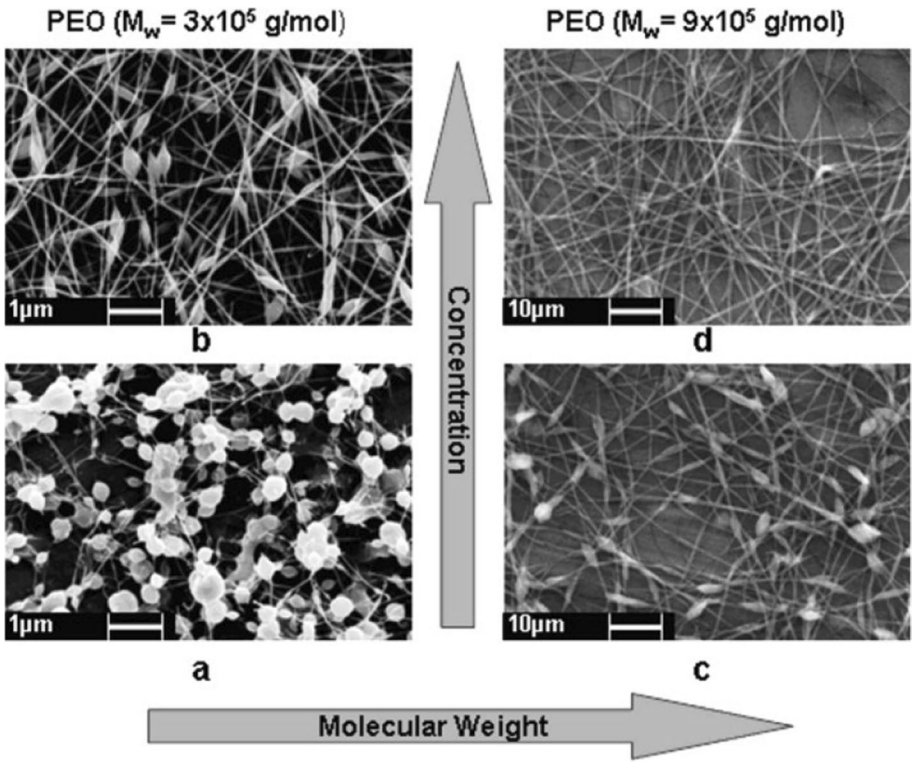


Fig. 3 SEM images of electrospun PEO polymer at different concentrations and molecular weight[48]. Reproduced with permission from John Wiley & Sons

introduction of salts increased the conductance of the polymer solutions, where LiCl-added solutions exhibited the highest conductance. This was reflected in the electrospun fibers, as the inclusion of salts increased the fiber diameter, and fibers containing LiCl had the highest mean diameter of 473 nm. The high conductance, increases the surface charge density, which makes the spinning easier, thus resulting in thicker fibers. This study also suggests that completely insulating polymers can be made conductive by adding some salts and thus easily electrospun as per requirements. Moreover, the viscosity of the polymer solution was not evidently altered by the addition of salts.

Voltage

Every solution has a threshold applied voltage, above which the jet gets ejected from the Taylor cone. However, the effect of voltage/electric field on the diameter and morphology of the fibers is highly debatable. Plenty of research shows that the applied voltage could increase and decrease the thickness of the electrospun fibers. Can-Herrera et al. investigated the effect of applied voltage on the electrospun polycaprolactone (PCL) scaffolds. In this work, they have varied the voltage from 10 to 25 kV and studied the differences in the morphology of the PCL fibers [51]. They found that there was an increase in the thickness of the PCL fiber as the applied voltage increased, as showcased in Fig. 4.

Similarly, Mirzadeh et al. studied the effect of applied voltage (10 kV, 15 kV, 20 kV) on the morphology and

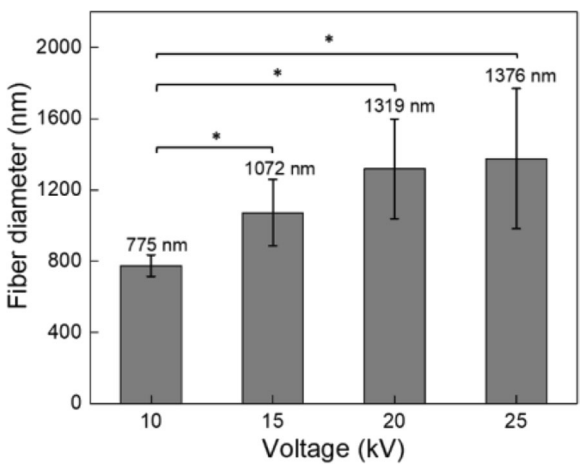


Fig. 4 Graphical representation of change in average diameter of electrospun PCL fiber with respect to the applied voltage[51]. (Open access)

thickness of polyvinylidene fluoride (PVDF) nanofibers [52]. It was concluded that the fibers were of narrow diameter with beads at a lower voltage, and at higher voltage, thick unstable fibers were formed. In another work, the PVDF fibers decreased in diameter as the voltage increased from 9 to 15 kV[53]. However, the trend was reversed as the voltage was further increased to 21 kV. The increasing applied electric field increases the electrostatic force developed on the jet, leading to more stretching, resulting in thin fibers. But at higher voltage, the Taylor cone becomes unstable and disappears, resulting in multiple jet ejections. Thus, the electrostatic force experienced by each cone decreases, resulting in non-uniform coarse fibers with higher diameters [54]. At higher voltages, it is also claimed that the droplet formed at the needle end recedes into the capillary of the tubes, apparently leading to the formation of fibers with a wide range of diameter (Fig. 5).

Solution feed rate

The solution's flow through the needle plays a crucial role in the morphology of the nanofibers formed. A critical solution feed rate for each material solution above causes the tendency for bead formation to be very high[55]. Generally, at a lower feed rate, the amount of solution at the tip of the needle is less and gets sufficiently polarized. But at a higher flow rate, the quantity of solution is high, leading to less polarization and less stretching time, resulting in thick beaded fibers [56]. Shamim and his team worked on the effect of flow rate on the electrospun nylon 6 fibers [57]. Their investigations also suggested that at the lower flow rate (< 0.5 mL/hr), the fibers are fine; however, above 0.5 mL/hr, a considerable amount of solution is getting electrospray, and the rest forms fibers with defects such as beads, weaved structures, and blobs.

Tip-to-collector distance

The distance between the needle tip and the collector is also equally important as the other factors affecting the fiber morphology. This parameter decides the time the jet receives to stretch and solidify into fibers. Here, as the distance is lowered, the probability of attaining thicker beaded fibers increases, and as the distance increases, more defined fine thin fibers are achieved. Nevertheless, increasing the distance beyond a specific limit will not help improve the morphology of the fibers.

Ambient atmosphere

The atmospheric conditions like temperature and humidity also impacts the morphology of the fibers. The rate at which the solvent evaporates and the viscosity of the solution are directly impacted by temperature. As temperature rises, the viscosity of the solution decreases [58]. Likewise, low humidity will lead to immediate drying out of the solution, leading to increased viscosity of the solution. Moreover, as the humidity increases, the solution will take up more water, leading to incomplete drying and solidification of the fibers, forming defective fibers. Also, electric discharge to atmospheric water molecules is high at high relative humidity, resulting in a lower surface charge density on the jet[59].

Electrospun fibers for EMI shielding

Electrospun fibers possess characteristics such as high porosity, a large surface area, and strong interface interactions, which facilitate the absorption, reflection, or scattering of electromagnetic waves [60, 61]. These micro- and nano-scale fibers serve as a platform for both absorbing and reflecting EM waves, making them highly suitable for EMI shielding materials. The method also enables the uniform dispersion of conductive

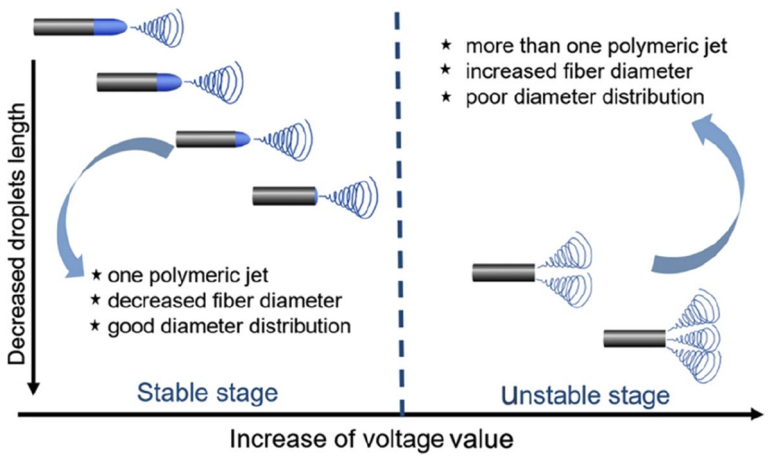


Fig. 5 The schematic representation of the relationship between decrease in droplet size and increase in applied voltage[54]. (Open access)

fillers within polymeric fibers, thereby enhancing the EMI shielding effectiveness of the material.

By incorporating these fibers into various polymeric matrices (including thermosetting and thermoplastic), lightweight and flexible EMI shielding materials with excellent mechanical and thermal resistance can be produced. These composites offer advantages over conventional metal or ceramic-based EMI shielding materials, as they tend to be heavy and more susceptible to corrosion. [62]. Therefore, electrospinning is considered a versatile technique for producing lightweight materials with superior EMI shielding capabilities.

Moreover, electrospinning is an economical and efficient strategy that has been commercialized for synthesizing carbon nanofibers, which find applications in EMI shielding materials, energy storage, and sensors. Other methods for fabricating carbon fibers include chemical deposition and template methods [63, 64]. Despite their existence, the ease of using the electrospinning method has allowed it to surpass these alternatives in the fabrication of carbon fibers. The process of preparing carbon fibers via electrospinning will be elaborated in the subsequent sections of this review article.

Applications of electrospinning

Tissue engineering

Electrospun fibers are effectively utilized in tissue engineering as they are similar to native tissues in texture. The electrospun nanofibrous scaffolds are advantageous over the fibers prepared via other techniques, as they are uniformly aligned and have a high surface-to-volume ratio[65]. Moreover, it can mimic the extracellular matrix and offer high mechanical strength. There are several applications for electrospun nanofibers in tissue engineering, such as bone, cartilage, nerve, vascular, skin, and cardiac. Bio-degradable polymers such as polyhydroxy acids (lactic acid, glycolic acid) and their co-polymers, such as polycaprolactone (PCL), are generally electrospun into nanofibers for biomedical applications. For instance, Semnani et al. synthesized porous nanofiber scaffolds of PCL and Chitosan blends for liver tissue engineering applications[66]. Here, they optimized the collector speed to 90 rpm and the angle of collector wires to 40° to obtain highly oriented fibers with desirable porosity. Cell compatibility of the synthesized fibers was substantiated via the high cell viability of 89.9%. Here, the fibers had a porosity of 79% and a pore size of 12 ± 5 µm, which is appropriate for infiltrating epithetical liver cells in mouse. In another work, conductive nanofibers from polypyrrole /chitosan /collagen /poly ethylene oxide were synthesized to accelerate the tissue repair via the transmittance of tiny electric signals[67]. Adding polypyrrole (PP) could enhance the conductivity of the fibers to 164.274×10^{-3} s/m, placing it within the range of semiconductive polymers. From the MMT results, it was concluded that the fibers with 10% PP showed optimum cell adhesion, growth, and proliferation abilities. Additionally, the mechanical properties of the fibers illustrated its applicability in several types of tissue engineering, including skin, nerve, and heart.

Drug delivery

Electrospun nanofibers are potential carriers in drug delivery as they have high drug loading capacity and high encapsulation efficiency, because of their large surface to volume ratio and tunable porosity. Drugs can be loaded in electrospun nanofibers as crystals or as non-crystals. Several drugs, including antibiotics, RNA, and DNA, have been used in electrospun nanofibers. In a research work, PLA/PCL blended membranes were synthesized using electrospinning, which was found to be effective in the release of bovine serum albumin (BSA) protein[68]. In another report, Abidian and co-workers prepared highly aligned poly(lactide-co-glycolide) (PLGA) fibers (spatial orientation of > 99%) via electrospinning. They studied the effect of fiber alignment on releasing an anti-inflammatory drug, dexamethasone (DEX)[69]. The SEM images of orderly arranged and randomly aligned fibers are displayed in Fig. 6 (a-d). Compared to randomly organized fibers, ordered aligned fibers appear to have a lower burst release and a longer sustained release in vivo, according to the findings, as depicted in Fig. 6(e). The degradation pattern revealed that the random fibers behave as an amorphous system where the hydrolysis is more favored than the aligned fibers, leading to anisotropic degradation in aligned fibers and isotropic degradation in random fibers, as illustrated in Fig. 6 (f, g). This led to a higher sustained release rate of DEX in aligned fibers.

Energy storage

Nanostructured materials contribute significantly to energy storage and conversion devices. Similarly, nanofibers are also utilized in lithium-based batteries (LiBs), supercapacitors, and fuel cells. Commercially, powder materials are used to fabricate electrodes for Li-based batteries. However, migration of Li^+ ions through the powder substances is highly time-consuming, leading to significant volume expansion and causing a poor performance rate. Nanofibers are a potential alternative to address this problem owing to their excellent electrochemical activity, surface-to-volume ratio, and porosity. Here, the electrospun fibers are mainly used as electrodes for LiBs. Hence, several Li-based metal oxides such as $LiCoO_2$, $LiMn_2O_4$, $LiMnO_2$, and $LiFePO_4$ are electrospun to obtain nanofibers. Among this, $LiCoO_2$

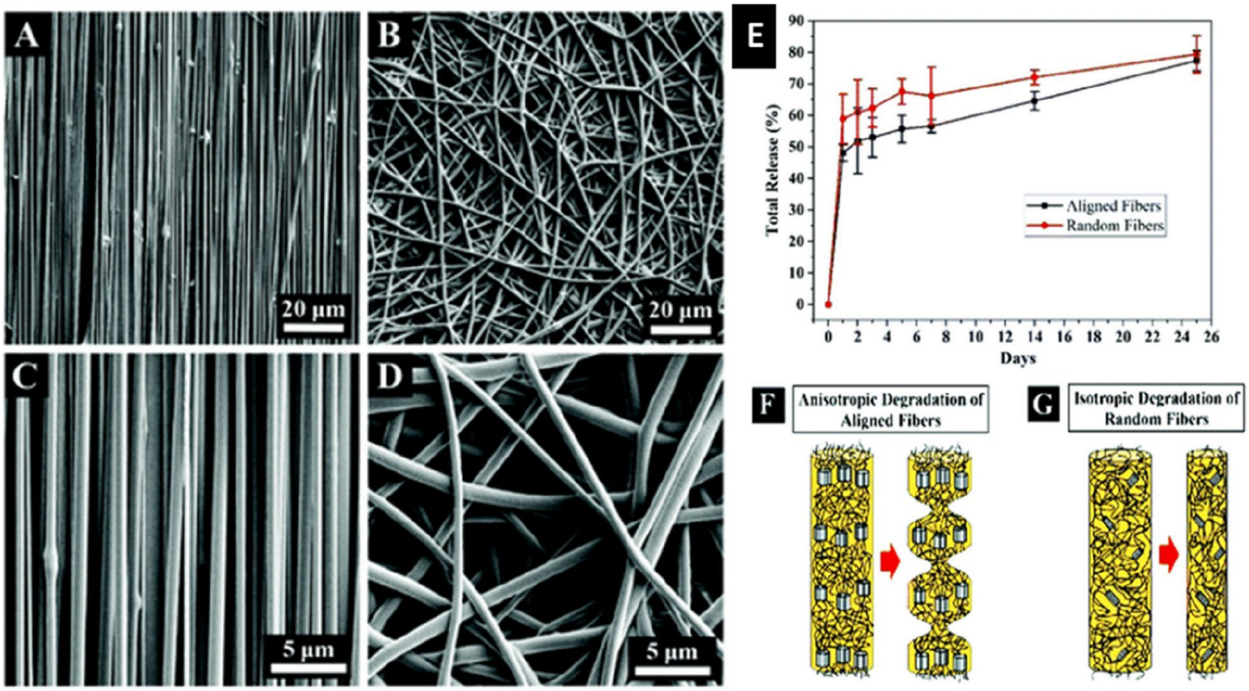


Fig. 6 The SEM images of (**a**) & (**c**) aligned and (**b**) & (**d**) random fibers at different magnification, (**e**) Release profile of aligned and random fibers, (**f**) & (**g**) Schematic representation of degradation modes in aligned and random fibers[69] Reproduced with permission from Royal Society of Chemistry

is a commercially available cathode for LiBs, which was electrospun by Jiao and team [70] to obtain $LiCoO_2$ nanofibers that exhibited a first cycle discharge capacity (FCDC) of 182 mAh/g (higher than that of powder electrodes). However, the cyclability of nanofibers could have been better. Thus, same team fabricated core–shell $LiCoO_2$-MgO via coaxial electrospinning, which delivered 90% of FCDC even after the 40th cycle[71].

Along with these applications, electrospun fibers find ample applications as sensors, protective coatings, catalysis, reinforcements in composites and more[72–80]. In the following section (Sect. 4) details regarding the fabrication of these fibers are mentioned.

Electrospun neat and composite carbon fibers

Electrospinning is a technique used for making a variety of fibers, such as pure polymer fibers, blend polymer fibers, fibers with different morphological architectures and composite fibers with diverse fillers. Composite electrospun fibers are widely employed in material science, particularly for electromagnetic interference shielding. Carbon fibers derived from polymeric source are one of variant that offers very good option for shielding. This section deals with preparation of neat and composite carbon fibers and briefly presents their properties.

Preparation and properties

When it comes to EMI shielding, carbon materials are the obvious choice for this application because of their effectiveness. But, it is relatively expensive as well as challenging to produce multifunctional carbonaceous components that are currently commercially available, like carbon nanotubes, graphene and more. Sophisticated technologies require carbonaceous materials that are flexible, mass-producible, and affordable. One of the most promising solution for this problem is provided by electrospun carbon fibers, which are turbostratic, fibrous carbon materials with high aspect ratio [28, 81, 82]. The ability to alter the morphology and microstructure of electrospun carbon fibers is their most significant benefit compared to other carbon materials, especially carbon nanotubes. By carefully adjusting the electrospinning parameters and setups, we can regulate and manipulate the diameter and profiles of fibers that are electrospun. Furthermore for these electrospun carbon fibers, the microstructure may be adjusted and the fiber interiors can be modified [60, 83–88].

Electrospun carbon fibers are derived from a precursor polymer source. To prepare carbon fibers, the pre-required condition is a carbon-rich polymer precursor that can yield carbon fibers after undergoing heat treatment. The most popular method for preparing

and modifying the morphology of carbon nanofibers is electrospinning (to achieve fibrous structure), followed by thermal treatment. A high-voltage power source, solution-feeding equipment, and a collecting device are commonly employed in the electrospinning process (Sect. 3). A scheme of electrospinning is presented in Fig. 7. To produce carbon fibers, the precursor nanofibers are then pre-carbonized, carbonized, or graphitized as presented in Fig. 8. For the thermal treatments, researchers use ovens, high temperature furnaces and different carbonization atmospheres. Electrospun polymer derived carbon have wide array of application potential in materials field, spanning from sensors energy devices (battery, supercapacitor, fuel cell, solar cells and hydrogen storage), environmental devices, catalysts, biotherapeutics applications and EMI shielding. The overall performance, structure and properties of the prepared carbon fibers depends on the electrospinning step (it determines the shape, size and profile of fibers), pre-carbonization step (an important step based on the type of polymer) and carbonization/graphitization step (carbonizing parameters like heating rate, time and temperature). Lee et al. [81] presented a detailed review on the microstructural tuning, surface tuning and cross sectional tuning of carbon fibers. Guo et al. [82] and Sharma et al. [28], presented their reviews on electrospinning based fibrous materials in EMI shielding.

In 1999, Chun et al. presented the first report on thermally treated electrospun carbon nanofibers [91]. In 2003, Kim and Yang worked on these carbon webs for super capacitor application [92]. After that much research was not pursued until 2016, later which there is an exponential increase in the research groups and research outputs in this field in various applications, as of 2020, over 1000 research articles are reported in this field [81]. As mentioned earlier, the choice of polymer precursor is very important for the resultant carbon fibers. Many possible polymers are tried for this purpose and a few of them are widely used, as they are carbon-based and are utilized because they can easily produce microstructures that are turbostratic or graphitic.

Many polymers are explored as precursor materials for preparing carbon fibers. They include, polyacrylonitrile (PAN)[93–98], pitch [99], polyimide (PI) [100, 101], polyvinyl alcohol (PVA) [102], cellulose [103], polyvinylpyrrolidone (PVP) [104], lignin [105], polyvinylidene fluoride (PVDF)[106] and more. Interest in case of bio-based polymer derived carbon fibers is increasing rapidly and much work is reported with those polymers. In one hand, when thermally treated at low temperatures, polymer such as PAN form aromatic rings in linear polymeric structures. Conversely, at elevated temperatures, the aromatic rings in pitch and phenolic resin undergo intermolecular crosslinking to form graphitic or turbostratic structures.

Owing to the high carbon output and good thermal stability, PAN is the frequently used commercial carbon fiber precursor. It is also the precursor that is being explored the most for electrospun carbon fibers [91, 107]. Carbonized fibers often possess a turbostratic microstructure that is less-ordered and falls between perfect graphitic and amorphous forms. Despite having a 78%

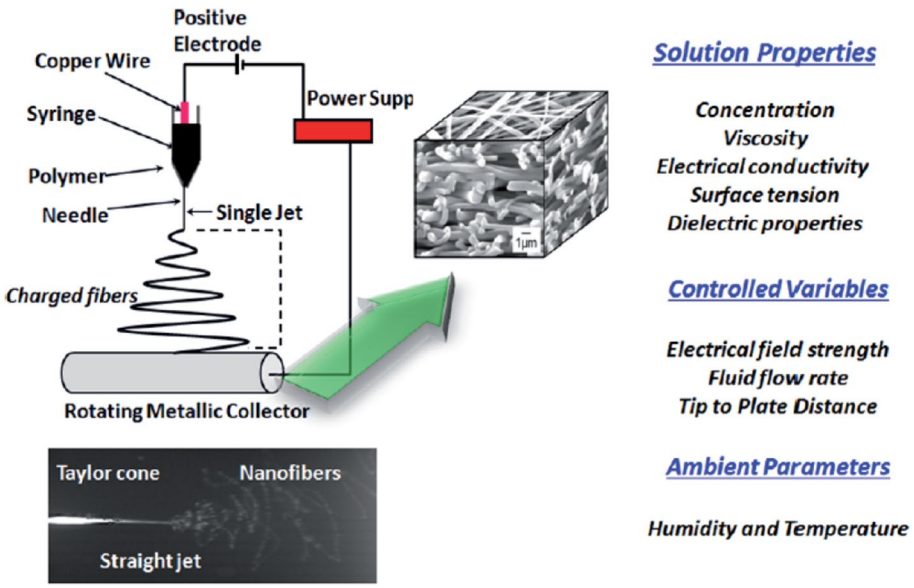


Fig. 7 Electrospinning setup scheme and parameters [89]. (open access)

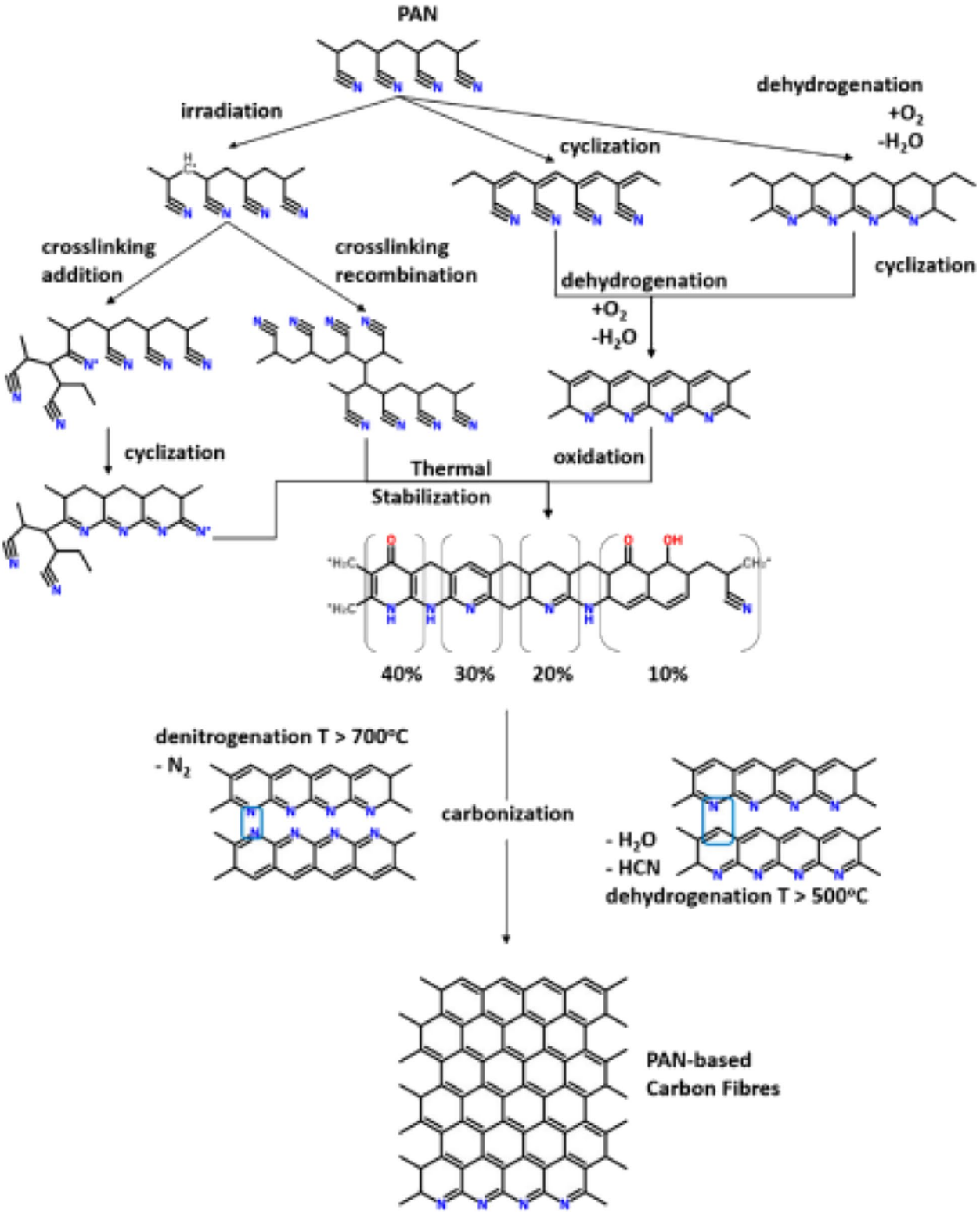


Fig. 8 Chemical evolution during PAN based carbon fiber preparation process [90]. (open access)

sp^2 hybrid bond composition, electrospun carbon fibers have less developed crystallites and amorphous phases than carbon fibers due to the less developed precursor nanofibers [81]. This is due to the fact that the tensile tension that created during the electrospinning process of the precursor nanofibers is not as consistent as the stress generated during the process of post-stretching that is usually employed with carbon fiber precursors.

Carbon nanofibers may be effectively produced by electrospinning PAN and then going through two steps: stabilization and carbonization. This procedure is similar to the manufacturing of traditional carbon fibers. Numerous stabilization and carbonization protocols for electrospun PAN nanofibers have been studied by research groups. The stabilization of these fibers was done in hot air oven at 200–300 ˚C, and the carbonization was done in an inert environment at high temperatures that can go upto 2800 ˚C [108–115]. Progressive and multi-stage heating processes covering stabilization and carbonization (Fig. 9 (a, b & c)) were devised to minimize mass loss and dimension shrinkage [116]. In Fig. 9(d-j) and Fig. 10, microscopic images of carbon fibers with different variants are presented. Because of narrow fiber diameters and the drawing action of the electrospinning, carbon nanofibers from electrospun PAN, in contrast to their traditional counter parts, display low strength [117]. In case of electrospun PAN fibers, the polymer chains may relax to certain degree due to the presence of solvent traces. Because of this the same imperfect chains are estimated in the carbon fibers, as they are retained [118]. This orientation loss may be reason for the comparatively inferior mechanical features of the prepared carbon fibers. And when these nonwoven mats are subjected to stress, the stress is only shared with relatively small area because of the randomness and fiber stacking. This results in weak mechanical property of individual fibers or nonwoven mat[113]. Hence the uses of electrospun carbon fibers are limited to functional applications

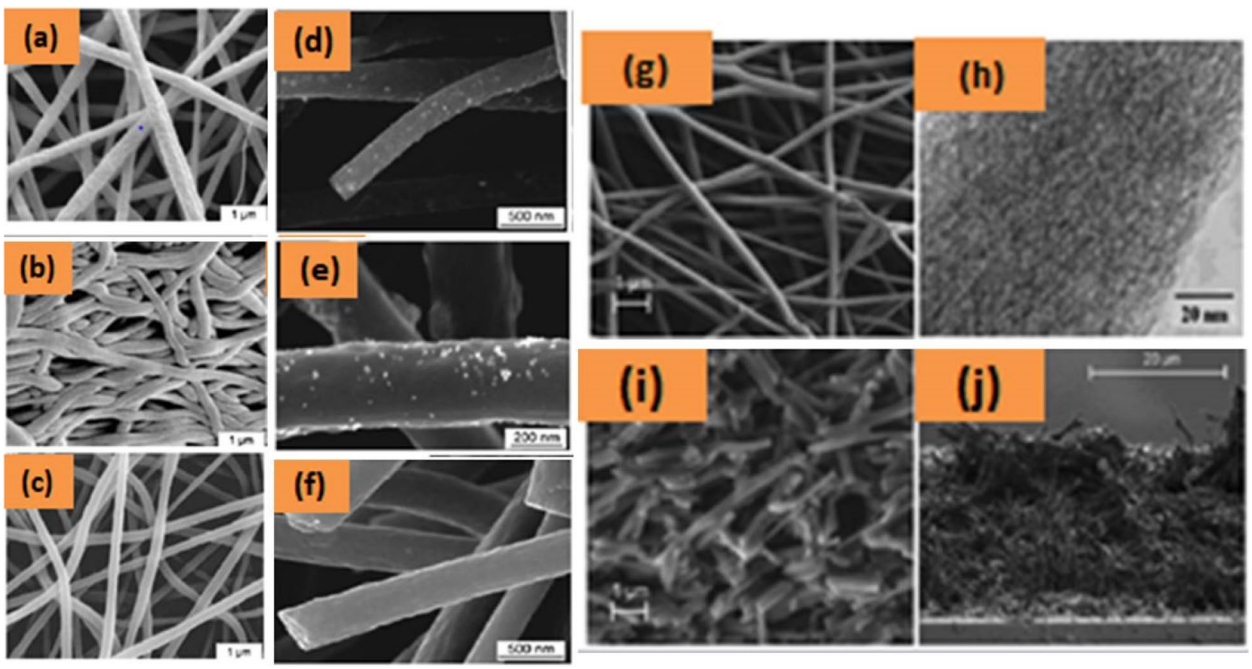


Fig. 9 Scanning microscope image of (**a**) electrospun PAN fibers; (**b**) carbon nanofibers from two-step heating; (**c**) carbon fibers from multi -step progressive heating [116]. Reproduced with permission from Elsevier. **d**, **e** SEM images of carbon/SnO_2 fibers(**f**) derived carbon fiber [122]. Reproduced with permission from American Chemical Society. Carbon nanofibers (**g**) SEM image; (**h**) TEM image; (**i**) top-view of fibers and (**j**) cross section view of fibers [123]. Reproduced with permission from American Chemical Society

(See figure on next page.)

Fig. 10 SEM images (**a**) neat carbon fibers [124]. Reproduced with permission from Elsevier. **b** carbon black loaded fibers [125]. Reproduced with permission from Elsevier. **c** carbon nanotubes loaded fibers[126]. Reproduced with permission from Elsevier. **d** graphene loaded fibers [127]. Reproduced with permission from Elsevier. **e** Porous fibers [87]. Reproduced with permission from John Wiley & Sons. **f** hollow fibers [128]. Reproduced with permission from Elsevier (**g**) fibers with 2 channels (**h**) fibers with 4 channels [129]. Reproduced with permission from Royal Society of Chemistry

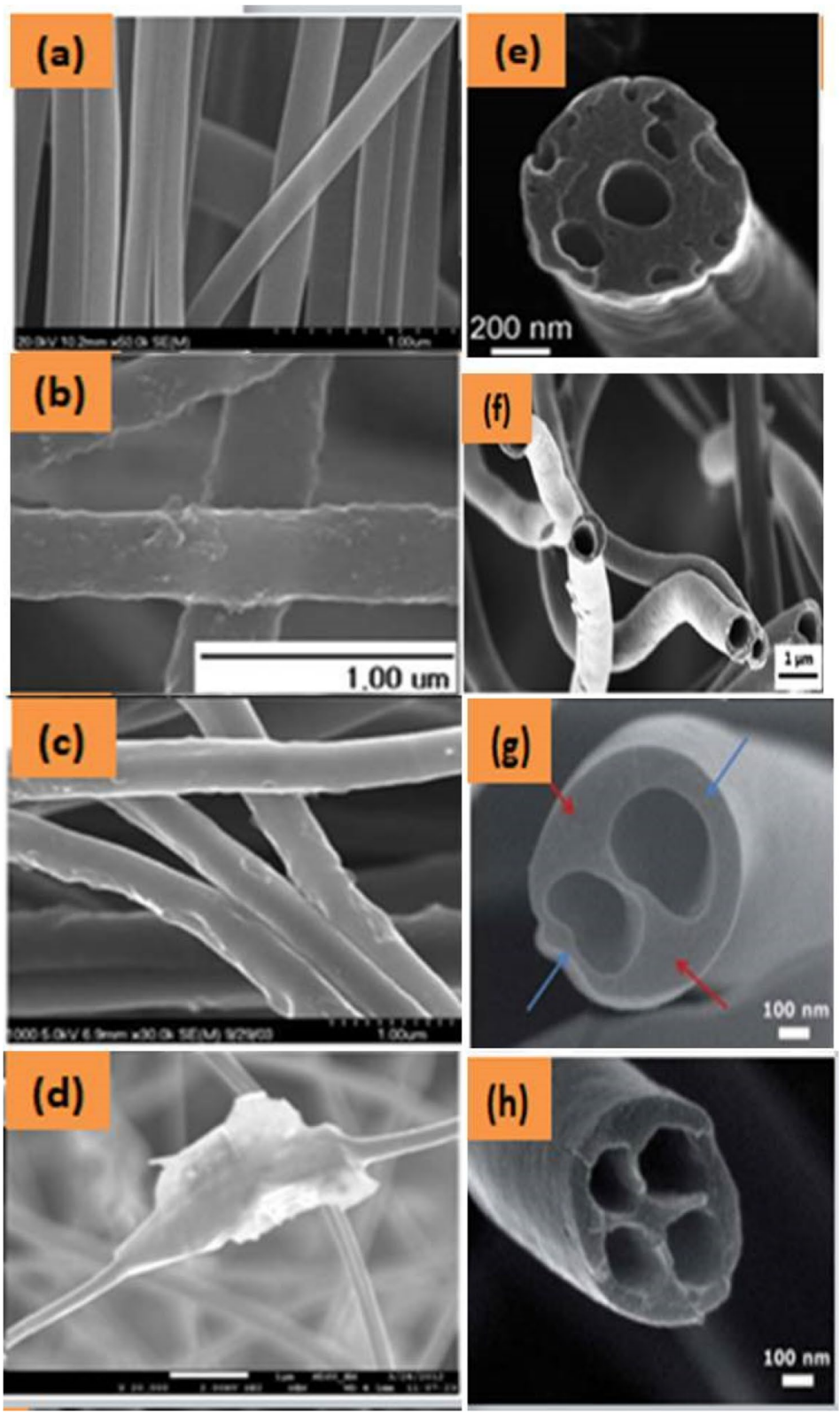


Fig. 10 (See legend on previous page.)

rather than high mechanical strength applications. Based on previous studies, it was observed that, the more the molecular orientation and the degree of pre-oxidation, the higher is the PAN derived carbon fiber's mechanical properties and researchers have worked with different strategies to improve them [97, 119–121].

By using carbonization and electrospinning processes, Bayat and colleagues showed the EMI shielding capabilities of pure carbon nanofibers in their study [130]. Here, carbonization of PAN was carried out at 700 °C and 900 °C, leading to the formation of carbon fibers, with an enhanced electrical conductivity of 0.05 ± 0.004 Scm^{-1} and 2.6 ± 0.9 Scm^{-1} at 700°C and 900°C, respectively. In another work, the carbonization temperature was varied between 800°C to 1700°C to investigate its effect on the mechanical properties of the synthesized carbon fibers. The results disclosed that, as the temperature was increased to 1400 °C, the strength of the carbon fiber raised to 3.52 ± 0.64 GPa from 1.86 ± 0.55 GPa. The Young's modulus also peaked to 191 ± 58 GPa for the fibers which were fabricated at 1700 °C. However, due to microstructural mismatches between the amorphous and crystalline phases, the strength fell to 2.05 ± 0.70 GPa at 1700 °C [124]. PAN-based carbon nanofibers have an electrical conductivity ranging from 1.95 to 7.69 S cm^{-1}, and reports have indicated thermal conductivity of 0.012 W m^{-1} K [81, 131].

Electrospun fibrous materials offers excellent capability to be employed in shielding [132, 133].Even though, carbon fibers and electrospun pure ceramic fibers exhibit potential in EMI shielding, several problems needs to be addressed. Such fibrous materials fall within a narrow segment and their mechanical brittleness severely restricts their practical uses. To address these issues, adding fillers to the electrospun fiber would be an excellent choice. As the composite fiber significantly increase the variety of raw materials available on one hand, and on other hand the filler reinforced fibers would see a notable improvement in their mechanical properties, particularly with regard to their flexibility. Typically, electrospun composites are filled with various ceramic materials to modify the dielectric characteristics, magnetic fillers to boost the magnetic properties, and electric conductive fillers to increase the electric conductivity. Composite carbon fibers contain other additives in addition to fibrous carbon, such as metal nanoparticles, metal oxides, ceramic fillers, magnetic fillers, and carbon compounds. Researchers are now developing methods to incorporate metal organic frameworks and MXenes into carbon fibers (Table 1).

Electrospun neat and composite fibers for EMI shielding

Usually electrospun polymer fibers, their composites, and post treated fibers are used for EMI shielding. The whole agenda is to enhance the dielectric or magnetic properties of these fibers and use them form electromagnetic wave shielding. Information on additives and post-treatments for electrospun polymer fibers are not included in this section as the interest is of carbon fibers. This section will specifically address composite carbon fibers and electrospun carbon fibers that are polymer derived and are employed for EMI shielding.

In few cases, electrospinning based carbon fibers are incorporated into a polymer (thermoplastic or thermosetting) matrix during the fabrication process in order to develop electrospun carbon fiber/ polymer composites. The mechanical performance of electrospun carbon fiber

Table 1 List of few literatures with regard to electrospun fibers in shielding applications

Material	Remarks	Reference
Fe_3O_4/carbon nanofiber composite	Increased conductivity	[130]
ZrO_2-embedded electrospun carbon fibers	Increased permittivity	[115]
Carbon black super P/carbon fiber	Increased effectiveness	[134]
PEDOT:PSS-polyvinylpyrrolidone coated carbon nanofiber	Increased effectiveness	[135]
Barium titanate with carbon	Aligned fibers, Increased effectiveness	[136]
Nb_2O_5 nanoparticles with N-doped carbon nanofibers	Increased effectiveness	[137]
N-doped carbon nanofibers containing La0.85Sr0.15CoO3 − δ	Increased effectiveness	[138]
Alkali-treated PAN nanofibers with graphene oxide composite films	nanofibers intercalated to graphene and microgasbag structure	[139]
TaC/Fe_3C-Fe composite fibers	Increased effectiveness	[140]
Tantalum carbide/carbon fibers	Increased effectiveness	[141]
$CoFe_2O_4$/C carbon fibers	Increased effectiveness	[142]
PAN based carbon fibers (Fe_2O_3, $BaTiO_3$ & MWCNTs)	Increased effectiveness	[143]
carbon fibers and silicone with an alternating multilayer structure	Increased effectiveness with number of layers	[144]

reinforced composites will depend on a number of factors, including porosity, the level of infiltration, the alignment and bonding interactions between carbon fibers and matrix, and the interaction between surfaces. Several techniques, including layer-by-layer design, dip-coating, and in-situ polymerization, have been reported as being used along with electrospinning to prepare electrospun carbon fiber reinforced composites [60]. Few reports like directly mixing the carbon fibers and polymers are also available[145]. Thus prepared composites are used for many value-added applications including EMI shielding. In Fig. 11, details regarding preparation of electrospun neat fibers, electrospun composite carbon fibers and electrospun fiber/polymer composites are illustrated.

Bayat et al. created carbon fibers and demonstrated how the graphitization of carbon at high temperatures increases conductivity. They also found a rise in the EMI SE of the fibers from 0 to 15 dB [130]. Another study found that neat carbon fibers that were heat-treated to 2100 ˚C had an EMI shielding effectiveness of 20 dB[115]. In one study, carbon nanofibers that were electrospun combined with carbon black super P (CBSP@CNFs) and their polydimethylsiloxane (PDMS) composites were produced [134]. The schematic for preparation is presented in Fig. 12. CBSP@CNFs has outstanding EMI shielding capability due to their high electrical conductivity of 2.5 S cm^{-1} and displayed higher shielding effectiveness over 8.2–26.5 GHz range. Within the range, the maximum and mean values of shielding effectiveness were 55.8 and 50.7 dB, respectively. Figure 13 shows the shielding effectiveness of the neat and carbon black loaded carbon fibers over the range. There appears to be a substantial correlation between EMI SE and electrical conductivity, as seen by the large improvement in EMI SE observed with an increase in CBSP loading. The EMI SE of neat carbon fiber and carbon black loaded carbon fibers with PDMS was presented in Fig. 13 (d, e and f). Each of the three PDMS composites; neat carbon fibers and carbon black loaded carbon fibers, has an average EMI SE value of 17.8 dB, 33.1 dB, and 45.2 dB, respectively. In Fig. 13 (g), the photographs of prepared composites are presented. A conductive network is created by carbon black, which increases polarization loss. Because of graphitization and the formation of a conductive network during carbonization, it was shown that carbon fibers and carbon black loaded carbon fibers show a notable increase in conductivity. Because CNFs have a porous structure, incoming electromagnetic waves are able to travel through the layers and be reflected by all of the layers. The absorption process predominates because

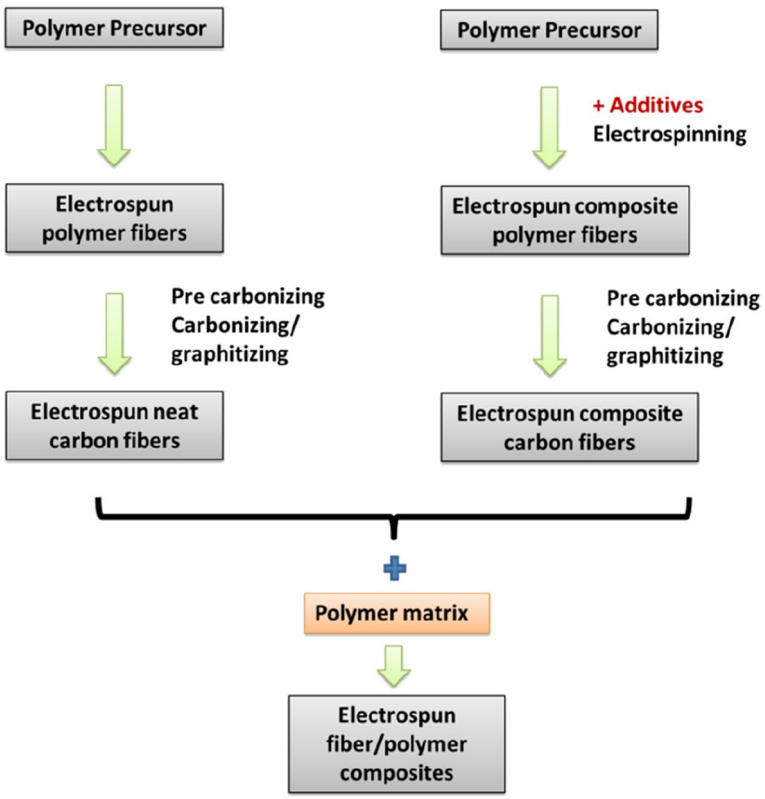


Fig. 11 Scheme of preparation of electrospun neat fibers, electrospun composite carbon fibers and electrospun fiber/polymer composites

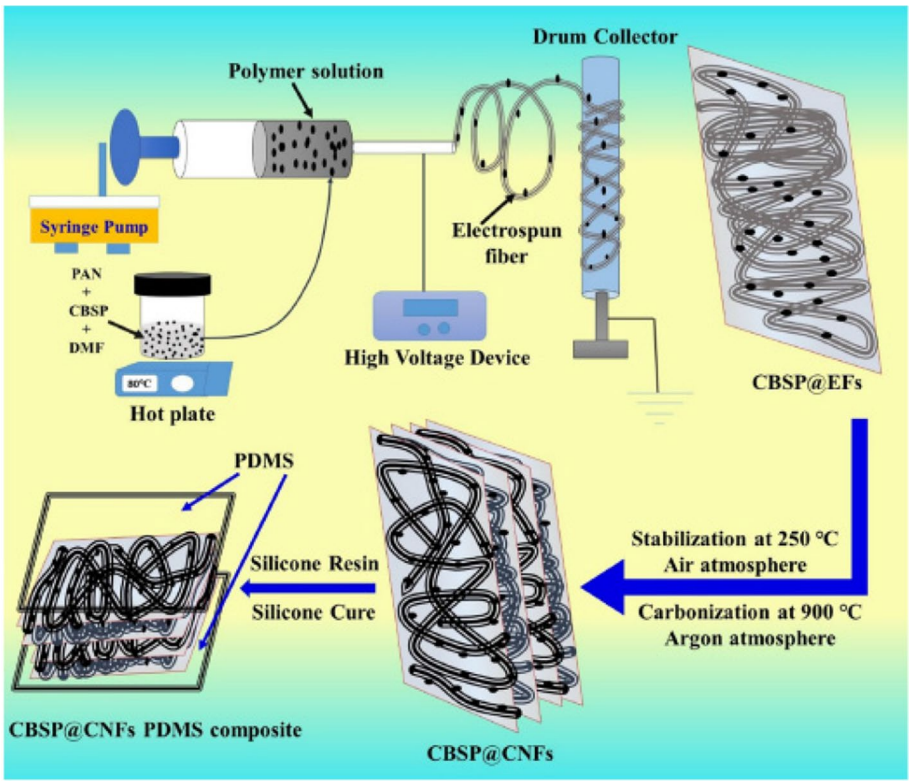


Fig. 12 Fabrication process of carbon fibers and their PDMS composite [134]. (open access)

of the rise in interfacial polarization and multiple reflections caused by the layered structure of CNFs. As seen in Fig. 13 (h), the collective effect of all these elements helps to enhance the final material's overall EMI shielding performance. The authors conclude that outstanding EMI shielding capability and electrical conductivity are the results of the efficient interaction between CBSP and CNFs.

Sharma et al. [135] reported fabrication of PEDOT:PSS and PVP coated, electrospun carbon fibers and their PDMS composites. The preparation method was presented in Fig. 14(a) and Because of the synergistic effect, the resulting material demonstrated high EMI shielding performance of 44 dB in the 8–26.5 GHz frequency range. These materials demonstrated, at a minimal PEDOT: PSS-PVP loading a high absolute shielding effectiveness value of 5678 dB cm^2 $g^{-1.}$ The proposed shielding mechanism displayed in Fig. 14 (b), suggests an absorption dominant EMI shielding approach over reflection. According to the authors, these results suggest that these composites have potential as lightweight, flexible, and thin EMI shielding materials in real-world applications. In another work, barium titanate with carbon fibers was prepared by electrospinning and carbonization in both aligned and non-aligned patterns, and the properties were compared [136]. They observed that the aligned fibers with barium titanate displayed a superior EMI shielding effectiveness of almost 81 dB (0.24 mm), while non-aligned fibers displayed a SE of near 59.2 dB. They pointed out that the alignment of optimally doped CNF may create new avenues for the commercial manufacturing of flexible, lightweight, and effective shielding materials. In a work, Nb_2O_5 nanoparticles with N-doped carbon nanofibers were prepared and employed for EMI shielding applications [137]. Another work demonstrates an approach of preparing lightweight N-doped carbon nanofibers containing $La_{0.85}Sr_{0.15}CoO_{3-\delta}$ for shielding applications[138].

The bonding strength in reinforced composites between carbon fibers and matrix is quite low, despite the fact that electrospun carbon fibers have numerous great features. For improved electrospun carbon fiber reinforced composites, it is therefore imperative to alter the surface of carbon fibers. Enhancing the interfacial bond strength of carbon fibers may be effectively achieved by surface treatment and many methods are available for it [60, 146]. There are two categories of CNF modification; chemical modification and physical modification. By making the material's surface rougher and enabling the creation of more contact points, physical modification

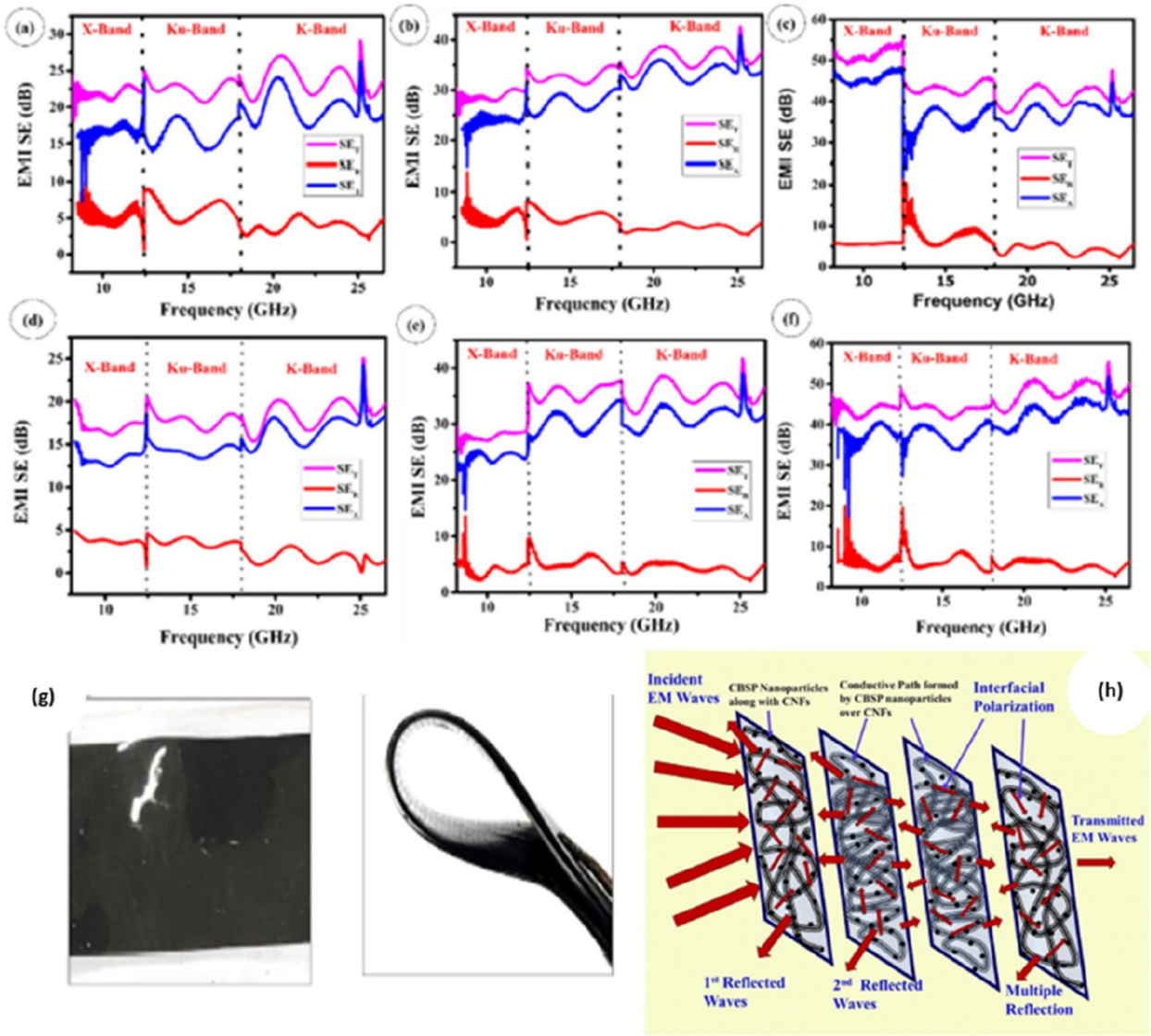


Fig. 13 Shielding effectiveness of (**a**) neat carbon fibers (**b**) carbon black loaded samples (CBSP@CNFs-50) (**c**) carbon black loaded samples (CBSP@CNFs-100) (**d**) neat carbon fiber PDMS composite, (**e**) PDMS composite with CBSP@CNFs-50, and (**f**) PDMS composite with CBSP@CNFs-100. **g** Photographs of CBSP@CNFs PDMS composite. **h** Shielding mechanism of CBSP@CNFs [134]. (open access)

can improve the specific superficial region. To improve interfacial adhesion, electrospun carbon fibers can be functionalized chemically by adding reactive functionalities that can form covalent bonds with matrix phase. In summary, the goal of each of these surface treatments is to enhance the interfacial performance through surface roughness, chemical bonding at the surface, or wettability.

In a work, In order to produce graphene-carbon fiber composite, the research team adopted an efficient method of annealing alkali-treated polyacrylonitrile nanofibers with graphene oxide composite films [139]. The findings demonstrate that during the thermal treatment procedure, carbon nanofibers were intercalated into graphene sheets and microgasbag structure was produced. These materials have superior strength of 10.4 MPa and interestingly, the prepared films exhibited density of 0.678 g/cm^3,superior conductivity of 1.72×10^5 S/m and an significant EMI SE of 55–57 dB. The authors observed that the notable improvement in shielding effectiveness may be associated with the joint action of carbon nanofibers and graphene, the establishment of an efficient conductive arrangement, and the shape of microgasbags. Further investigation using simulation revealed that the primary

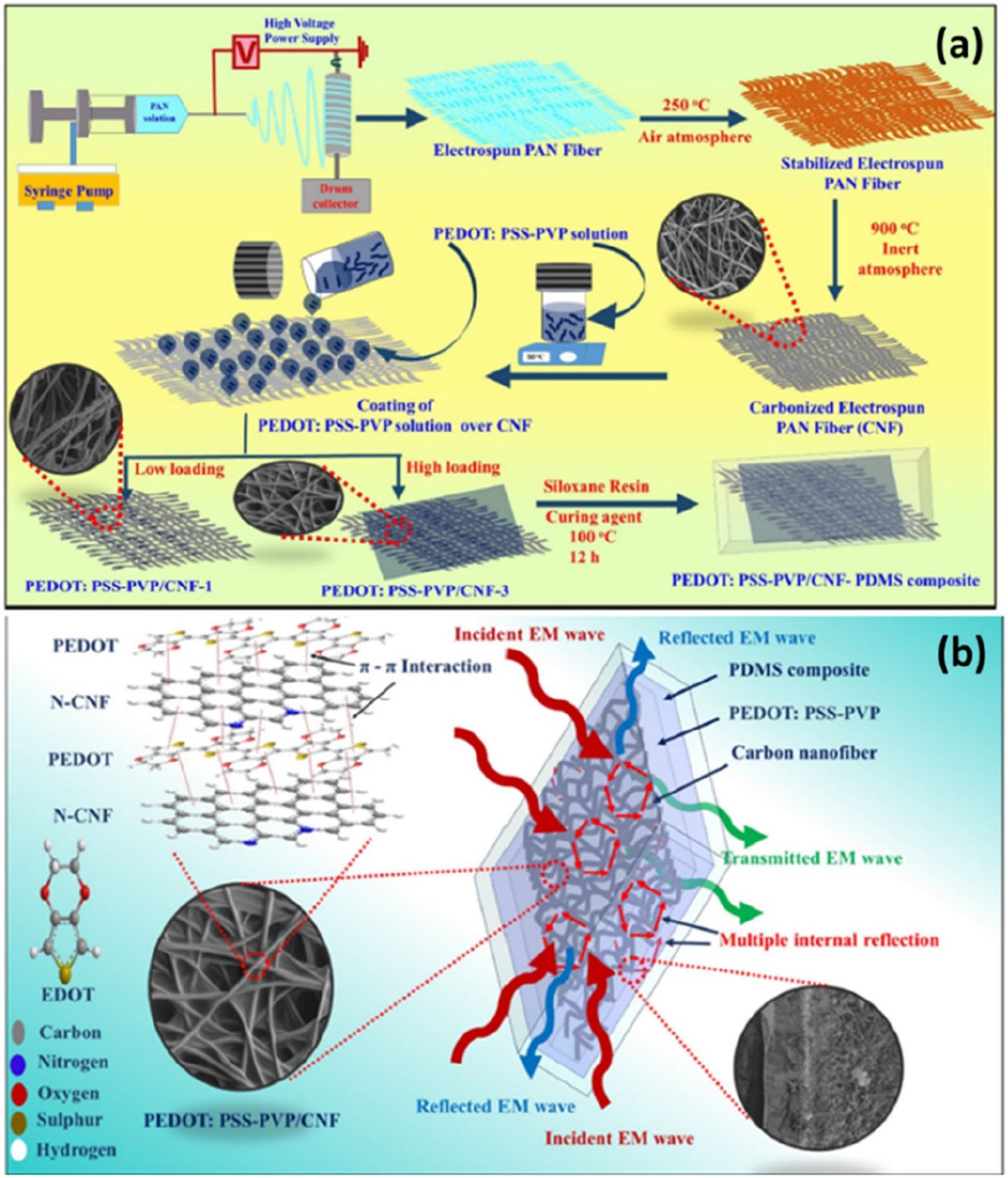


Fig. 14 **a** Scheme of preparation of PEDOT:PSS-PVP/carbon fiber-PDMS systems (**b**) Schematic mechanism of shielding [135].Permission pending from Elsevier

causes of the underlying process are the loss by conduction and numerous reflections brought about by the unique shape of the resulting fibers.

The scientific community is intrigued by research focusing on the incorporation of conductive and magnetic additives in carbon materials. In a work[140], the

authors prepared TaC/Fe_3C-Fe composite fibers via electrospinning process and pyrolysis. They have reported a conductivity of 15.4 Scm^{-1}, a saturation magnetization of 13.3 emu g^{-1}, and a low density of 0.34 $g.cm^{-3}$. They demonstrated that the highest shielding effectiveness of 46.4 dB is attained at 0.18 mm thickness by altering the Ta and Fe mass ratio. Additionally, they pointed that the loss mechanism mostly relied on reflection. Another study describes the development of conductive frameworks using electrospinning and pyrolysis with tantalum carbide (TaC) nanoparticles. [141]. They observed that the composite fabrics have tensile strength of almost 9.5 MPa with good flexibility. These TaC particles in optimum amount interconnect, resulting in conductivity of 10.4 S cm^{-1} and shielding effectiveness of up to 37.7 dB. Because of its pore structure, reflection serves as the primary basis for the shielding process. In another work[142], electrospinning process is used to prepare cobalt ferrite/carbon nanofibers employing isopropanol solvent media. The sintering process was done in different temperatures and electromagnetic shielding performance is analyzed in 8.2–12.4 GHz frequency level. The resultant $CoFe_2O_4$/C carbon fibers displayed noteworthy EMI performance of 30–35 dB.

Im et al.[143]. prepared carbon fibers with two filler combinations in PAN; they used PAN with FB (Fe_2O_3 & $BaTiO_3$) and PAN with FBC (Fe_2O_3, $BaTiO_3$ & MWCNTs). Following the spinning and heat treatment, they fabricated the carbon composite fibers and analyzed their EMI shielding properties. In Fig. 15 (a, b), the SEM images of FB and FBC are depicted, and the SE and share of reflection and absorption are presented in Fig. 15 (c and d). At low frequency of 800 MHz, we can see a SE value of 36 and 40 dB, respectively. But these values lowered to 24 and 29 dB later on with increase in frequency. This is corresponding to the dielectric and magnetic losses at higher frequencies. Due to the presence of MWCNTs, the SE of FBC is more than FB as the samples higher permittivity and permeability. In the case of FB and RBC, the average EMI SE of 32 and 37 dB are reported respectivel. Interestingly, when we consider the case of FB, the main shielding phenomenon is reflection dominated, almost contributing to 64%. But, in case of FBC sample, this changes to absorption dominated

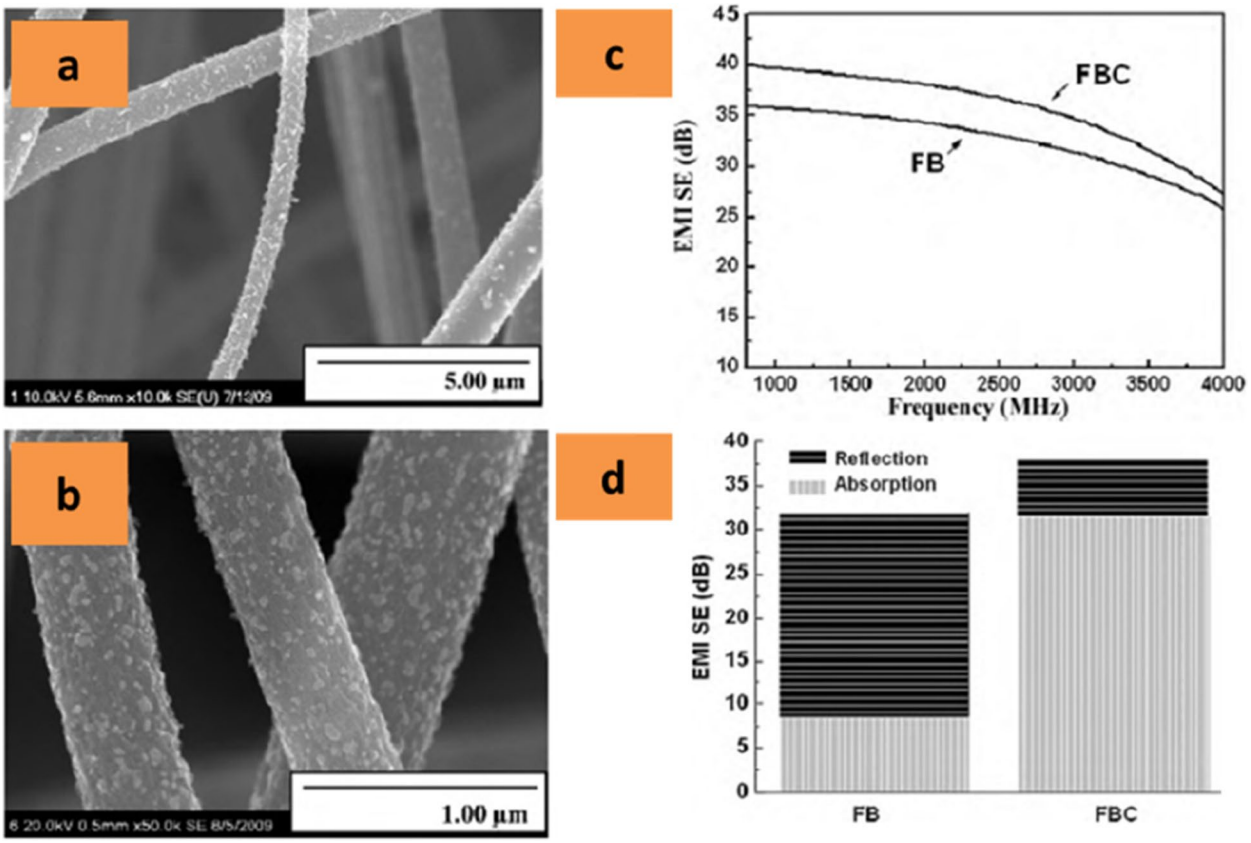


Fig. 15 **a**,**b** SEM images of FB and FBC (**c**) SE of fibers, (**d**) share of SE [143]. Reproduced with permission from Elsevier

shielding behavior, the major contributor being the addition of MWCNT.

Im et al. [125] in a study reported PAN based electrospun carbon nanofibers at different thermal treatments and varying loading of carbon black and fluorinated carbon black. Here the idea is carbon black used in the carbon fibers, has high conductivity and low cost, and after fluorination process enhance the filler's adherence and dispersion in a matrix. They demonstrated that better conductivities and shielding efficiencies are achieved by thermal treatments and the fluorination of carbon black. Carbon composite webs had a high EMI shielding efficacy of 50 dB and an conductivity of 38 S/cm. Kang et al. [147] produced a web of composite carbon fiber to examine the EMI shielding qualities. These $BaTiO_3$ and Fe_3O_4 webs were created using the electrospinning followed by carbonization processes. The samples are termed as CF (0%), 20FCF (Fe_3O_4 20%), 20BCF ($BaTiO_3$ 20%), FB37CF (Fe_3O_4:$BaTiO_3$ 20% (7:3)), FB55CF (Fe_3O_4:$BaTiO_3$ 20% (5:5)) and FB73CF(Fe_3O_4:$BaTiO_3$ 20% (3:7)). It was established that the carbon nanofiber web with EMI shielding and the nanoparticles added had better absorption properties than the carbon nanofiber web without the nanoparticles. In samples with 20 wt.% $BaTiO_3$ the highest EMI SE of 23 dB is obtained, while, carbon fiber with 20 wt.% Fe_3O_4 have EMI SE of 20.5 dB. Regardless of the ratio, all samples comprised of both fillers showed SE of 20 dB.

Li et al. [144] presented flexible and composite films by using electrospun carbon fibers and silicone with an alternating multilayer structure (AMS). The performance was evaluated in different layers and improvement in property is observed with increase in layers. The EMI SE and individual contribution of mechanisms of carbon fibers is shown in Fig. 16 (a, b). The EMI shielding performance of the AMS structure is shown in Fig. 16 (c, d). Remarkably, the shielding property was not declined even after 1000 cycles of bending, as presented in Fig. 16 (e). The photograph of the film is presented in Fig. 16(f). High conductivity, a large surface area, good interface bonding strength, and an alternating multilayer structure are all credited with these exceptional qualities.

In the case of electrospun derived carbon fibers, the collective application in EMI shielding is due to the following reasons. They are (a) excellent conductivity, (b) porous structure that can contribute in absorption and reflection phenomenon, (c) in case of carbon fiber/polymer composites, the interface interactions between the reinforcing carbon fibers and polymer matrices, (d) improved conduction loss and interfacial polarization loss, (e) in case of composite carbon fibers, the additive property also positively imparts the shielding performance, (f) thickness of mat or number of mats used. These types of materials are believed to be excellent wave-absorbing and highly temperature resistant for futuristic applications in EMI shielding.

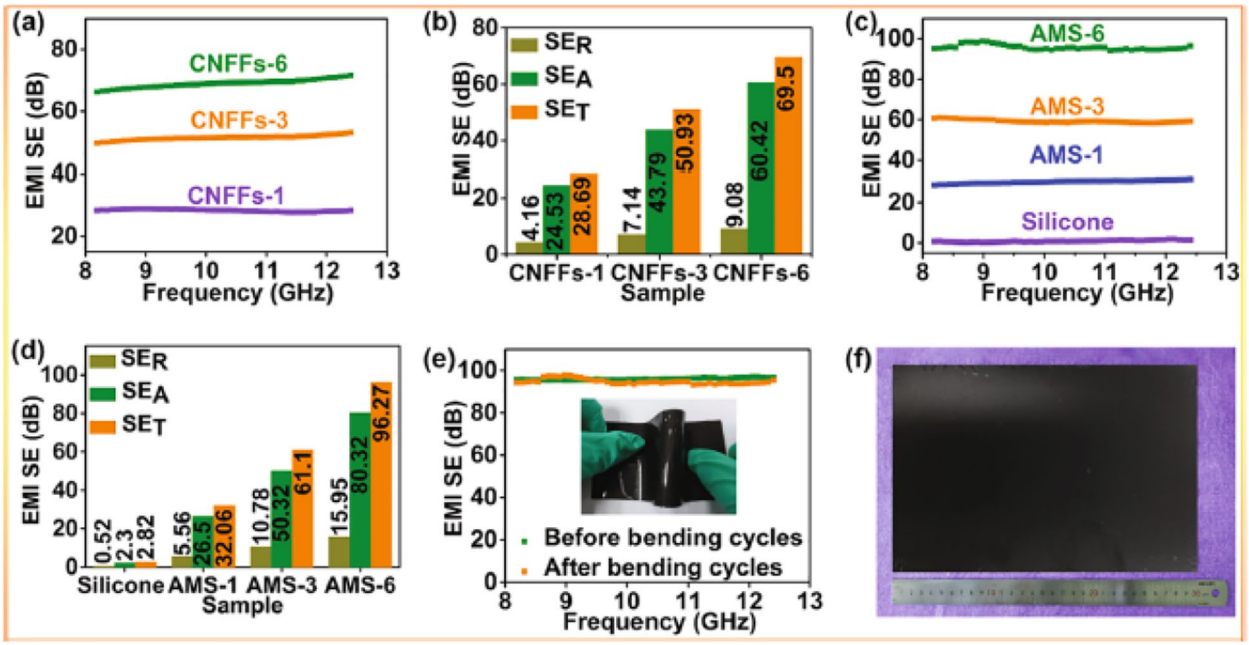


Fig. 16 **a** shielding performance of fibers (**b**) average SE of fibers (**c**) shielding performance of AMS films (**d**) average SE of AMS films (**e**) Shielding effectiveness of samples prior to and following 1000 bending cycles (**f**) photograph of AMS film [144]. Reproduced with permission from Elsevier

Conclusion

This review focuses on the preparation, properties and EMI shielding ability of carbon fibers prepared via electrospinning process. There are few issues to be resolved before considering mass production, like establishment of quality control of morphology and microstructure. This review shows that electrospinning, followed by stabilization and carbonization, is an easy procedure to create carbon nanofibers with hierarchical architectures. Applications for these materials go beyond EMI shielding and include adsorption, sensor development, energy conversion and storage, and more. The relationships between the preparation conditions and nanofibers architecture will be the main focus of futuristic studies. As more and more research is reported, electrospun carbon fibers produced by electrospinning are expected to become even more attractive members of the carbon family. These materials are effective and efficient materials to be employed as shields in wide frequency spectrum. The substantial increase of studies in this field demonstrates the efficacy of these thin, flexible, and light-weight materials in EMI shielding applications.

Acknowledgements
The authors thank IIST for necessary support.

Authors' contributions
B. D. S. D. Conceptualization, Writing – original draft; Writing – review and editing, R. P. Writing – original draft, K. J. Supervision, Writing – review and editing.

Funding
The authors did not receive any external funding.

Availability of data and materials
No datasets were generated or analysed during the current study.

Declarations

Competing interests
The authors declare no competing interests.

Author details
[1]Department of Chemistry, Indian Institute of Space Science and Technology, Thiruvananthapuram, Kerala 695547, India.

Received: 16 February 2024 Accepted: 11 July 2024
Published online: 20 September 2024

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