Chapter 1

Introduction and Literature Review

Chapter 1

1.1. Introduction to conducting polymer nanocomposites

Over the past two decades conducting polymer nanocomposites have been studied with much attention for their various synthetic approaches as well as applications.[1-4] Nanocomposites are multiphase materials which contain more than one component coexist in a nano structural confinement with other components. Conducting polymer nanocomposites are those in which one of the components is a conjugated polymer or its copolymer.[5] Modified properties of components are achieved after combining the conjugated polymer with any other organic or inorganic substrate in composite form.[6,7] Conducting polymers are a class of organic polymers having a continuous conjugated backbone inherently. The advantageous properties of polymeric materials on incorporation with a conducting framework could render advanced applications in appropriate fields. Various conjugated polymers are polyacetylene, polyaniline, polypyrrole, polythiophene, polyphenylenevinylene, etc.[8,9] Conjugated polymers or their derivatives or its copolymers possess conducting or semiconducting nature, along with polymeric features like easy processability, flexibility, lightweight nature, cost effectiveness, and the other unique characteristics of each conducting polymer.[8-11]

1.2. Polythiophene and its derivatives

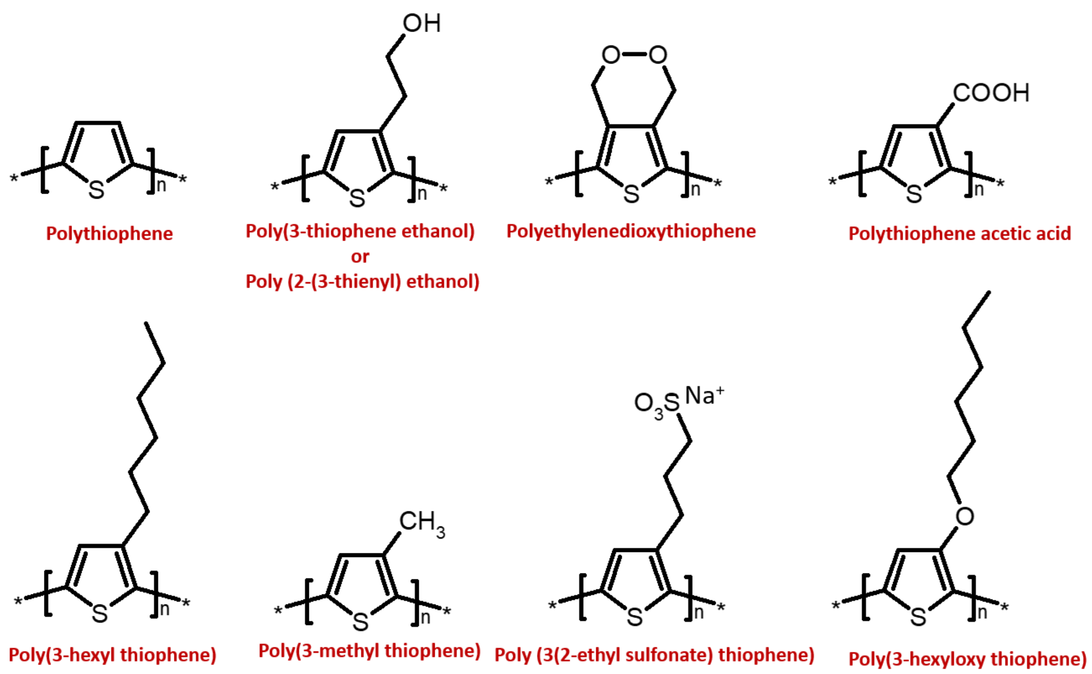

Figure 1.1. *Structure of polythiophene and some of the substituted polythiophenes*

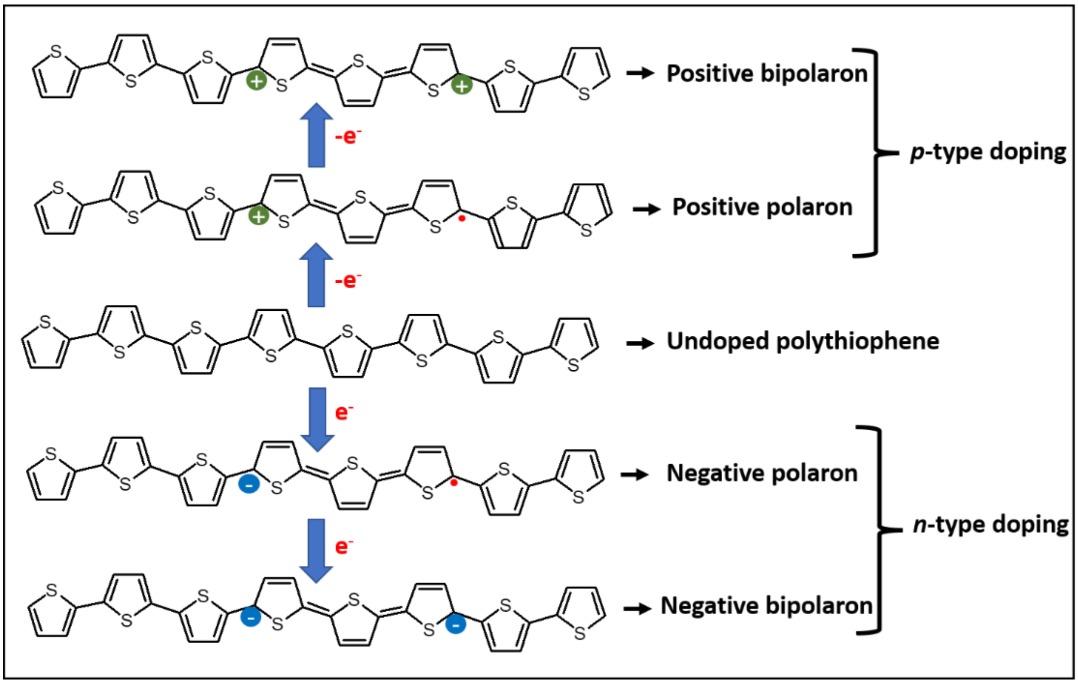

Figure 1.2. *Structures of positive and negative polarons and bipolarons formed in polythiophene via p-type and n-type doping respectively.*

Polythiophene and its derivatives are prominent and promising group of conducting polymer materials, holding unique characteristics such as ease of processability, good optoelectronic properties and environmental stability. Solubility or processability improvements are more prominent in polythiophene derivatives and their copolymers than in unsubstituted states. Structures of some of the substituted polythiophenes taken from literature are given in **Figure 1.1.**[12] polythiophene is distinguishable from other conducting polymers with its polymeric heterocyclic aromatic thiophene ring system existing as extended π-conjugated chains.[13,14] Doping of conducting polymers usually improves their electrical conductivity and shifts the electrochemical potential. Many researchers have reported undoped and doped states of polythiophenes and the importance of doping to result changes in the conductivity performance of polythiophenes. Band gap tuning is usually carried out to obtain desirable electrical properties for conducting polymers. The band gap of polythiophene and its derivatives can be tuned between 1 to 3 eV. Band gap tuning could be done by inserting suitable dopants or with side chain functionalization on polymer aromatic backbone. Polythiophene usually shows conductivity through *p*-type doping. Chemical, electrochemical, or electric methods can achieve the different levels of doping.[14-16] Insertion of a single charge on the polythiophene chain by doping creates polaron and further doping leads to the form bipolaron. Structural illustrations of *p*-type and *n*-type polarons and bipolarons are shown in **Figure 1.2.** The *n*-type doping was also reported in polythiophenes by altering the properties of prepared polythiophene. Jayasundara and co-workers presented a theoretical study of *p* and *n*-type doping on polythiophene and polypyrrole conducting polymers.[16] Polythiophenes might render their individual properties also with suitable fillers in the composite form.[17]

1.3. Organic/inorganic fillers in conducting polymer nanocomposites

The composites of conducting polymers in which polymers serve as matrices and the additional component(s) act as filler(s). Various inorganic or organic fillers can be chosen for nanocomposite preparation. Different fillers such as metals, metal compounds, carbon-based materials, inorganic substrates, and ceramic materials are used. The main factor determining effective nanocomposite formation and better properties is the interaction between different components in the nanocomposites.[18-26] Liu and co-workers reported the effective formation of end functional polythiophene/CdSe nanocomposite having the morphology of interpenetrated network nanorods obtained with the help of successful interaction between the components.[18] Pascariu et al. reported electrochemical preparation of polythiophene-nickel nanoparticle nanocomposites with good conductivity performance.[24] One of the reports of carbon based nanocomposites with polythiophene by Taj et al. was the PEDOT-graphene nanocomposite formation with a narrow band gap and high dielectric properties.[25] Another nanocomposite of polythiophene with clay substrate was reported by Aradillla and co-workers, who suggested the material for ultracapacitor applications.[3,26-28]

1.4. Carbon nanotubes as fillers in conducting polymer nanocomposites

Carbon nanotubes are unique one-dimensional conducting nanotubular materials exhibiting unique physical, electrical, and mechanical properties. They are generally classified as single-walled and multiwalled carbon nanotubes. Single walled nanotubes can be considered as folded graphene sheets, whereas later is multi-folded. The difficulty of using carbon nanotubes is poor processability due to the inherent bundling nature.[29,30] Various functionalization strategies on the walls of carbon nanotubes can be adopted to decrease this self-aggregation tendency. Carbon nanotubes could act as suitable fillers in conducting polymer nanocomposites due to (i) the opportunity of polymers to wrap on the surface of carbon nanotubes utilizing weak non-covalent CH-π or π-π interaction or (ii) covalent grafting of polymers on the defective sites of carbon nanotubes walls.[31-33] Carbon nanotubes also possess a template effect for attaching polymers around it; thereby maintaining the nanotubular morphology.[23,30] Single and multiwalled carbon nanotubes have their advantages in nanocomposites; multiwalled carbon nanotubes have the overall electronic structure less destructible on chemical treatments due to the presence of multi folds of tubular graphene layers. Multiwalled carbon nanotubes are economically more viable and could exhibit more excellent mechanical properties than single-folded carbon nanotubes.[34-36]

1.5. Synthetic strategies and development of conducting polythiophene-carbon nanotube nanocomposites

General strategies used to prepare polythiophene carbon nanotubes nanocomposites are

1. Carbon nanotubes or functionalised carbon nanotubes are ultrasonicated in the presence of polymer matrix.
2. In-situ polymerisation of polymer in the presence of dispersion of carbon nanotubes
3. Grafting of polymer chains from the surface of carbon nanotubes.[32]

The processability of nanocomposites is a crucial factor to be considered in the preparation of nanocomposites. The processing of nanocomposites also needs significant attention to tune the composites for specific applications. Polythiophene-carbon nanotube nanocomposites could usually be made up of aerogels, thin films, pellets, conductive inks, etc. The morphological features, distribution of different components in the composites and solvent dispersibility helps to fabricate them into suitable form.[37,38]

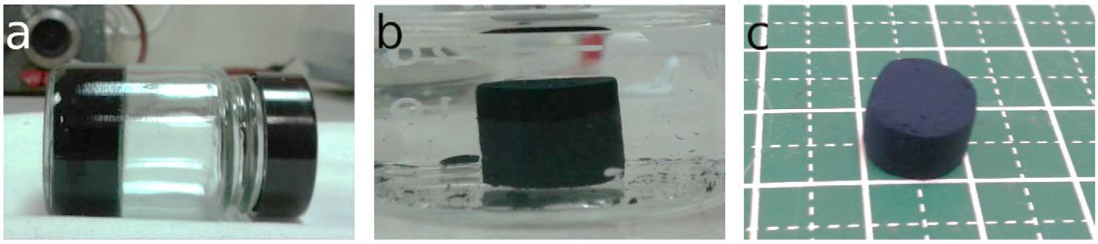

Figure 1.3. *PEDOT:PSS/CNT nanocomposites hydrogel (a), alcogel (b) and aerogel (c) (adapted from Cheng et al. 2017).*

Aerogel substances are distinguishable with their properties of high specific surface area, low density, and 3-dimensional porous structure. The unique nanotubular morphology of carbon nanotubes surrounded by polythiophene polymer delivers the possibility to form advantageous aerogels. Various applications of aerogels are contamination treatment, Cerenkov detector, thermal insulators, and energy storage. Besides that, biocompatible aerogels are interesting for bio implant and drug delivery. Good conductivity of the aerogels might lead to binder-free supercapacitor electrode preparation.[38-41] Cheng and co-workers reported free-standing aerogel fabrication from PEDOT: PSS/CNT nanocomposites having high specific surface area and good electrochemical performance. In this report, a hydrogel was first converted to alcogel, followed by treatment with supercritical CO_2, which resulted in the formation of free-standing aerogel (photographs of hydrogel, alcogel and aerogel adapted from the literature are given in **Figure 1.3.**).[39] Idumath et al. conducted a detailed review of emerging trends in polymer aerogels in which they remarked carbon nanotubes-polymer nanostructures could act as promising candidates for aerogel preparation.[40] Polythiophene carbon nanotubes thin/thick films are the most demanded preparation with nanocomposites due to their distinctive properties to obtain flexibility, lightweight nature, easy fabrication, etc.[14,29,42,43] Transparent thin films are also prepared using carbon nanotube nanocomposites.[43] Methods such as spin coating, solution sorting, spread casting, vacuum filtration, etc. are commonly used for nanocomposites thin film preparation. All the techniques seek the formation of stable dispersion of nanocomposites in suitable solvents.[14,29,42,43] Wang et al. reported polythiophene-carbon nanotube nanocomposites films for chemiresistor application. They found that thicker films were needed to produce a sufficient signal-to-noise ratio.[42] Another work done by Liyanage and co-workers addressed the fabrication of thin film transistors of poly(3-dodecylthiophene)-CNT nanocomposites, and its preparation was reported by dispersion based-sorting technique.[43] Pellet formation with nanocomposites is another form of fabrication of carbon nanotube-conducting polymer nanocomposites. Binder-free pellets have more significance. Carbon nanotubes alone do not give strong binder-free pellets, but composite preparation with environmentally stable polymers can render the effective formation of binder-free, firm, and non-deliquescent pellets.[44-48] It is easy to study the electrical conductivity of nanocomposites samples in pellet form. Gas sensing applications are mainly reported with pelletized form of polythiophene-carbon nanotube nanocomposites. [44-45] A recent report of ammonia sensing application of polythiophene carbon nanotube nanocomposites was conducted by Husain and co-workers and they conducted the study in the pelletized form of the composites.[44] Another possibility is the preparation of conducting ink with polythiophene carbon nanotube nanocomposites. The self-bundling nature of carbon nanotubes obstructs effective dispersion and thereby hinders conducting carbon ink preparation.[34,48] Polythiophene/CNT conductive inks were not much progressed in literature today. But the possibility of using conducting polymer-wrapped carbon inks can simplify the ink printing method and thereby reduce the overall cost of application. Chen et al. fabricated conductive ink made up of MnO_2/PEDOT/MWCNT nanocomposites with poly-tetrafluoroethylene in ethanol and demonstrated their application in flexible micro-supercapacitor (see **Figure 1.4.**).[48]

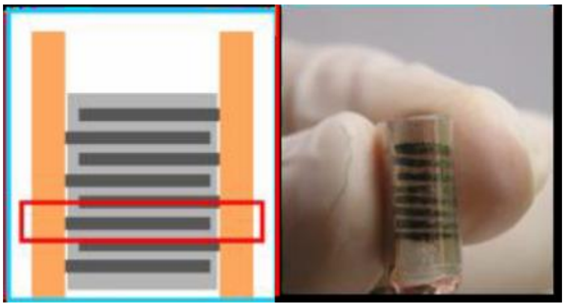

Figure 1.4. *Flexible micro-supercapacitor fabricated from MnO_2/PEDOT/MWCNT nanocomposites conductive ink with poly-tetrafluoroethylene (adapted from Chen et al. 2014).*

1.6. Applications of conducting polythiophene-carbon nanotube nanocomposites

Polythiophene-carbon nanotube nanocomposites are desirable materials in different applications such as sensors, suitable nanocomposite precursors for synthesising higher order structural architectures, supercapacitor electrodes, electromagnetic interference (EMI) shielding, photovoltaic cells and photodiodes, transistors, thermoelectric films, conducting adhesives, battery electrodes, aerospace applications, etc. The properties developed are characteristics of both polythiophene and carbon nanotube fillers which depend on their ratio of mixing and on the interaction between the components to determine the appropriate field(s) of application of the nanocomposites prepared. Significant applications of polythiophene-carbon nanotubes nanocomposites are discussed briefly below.

1.6.1. Sensors

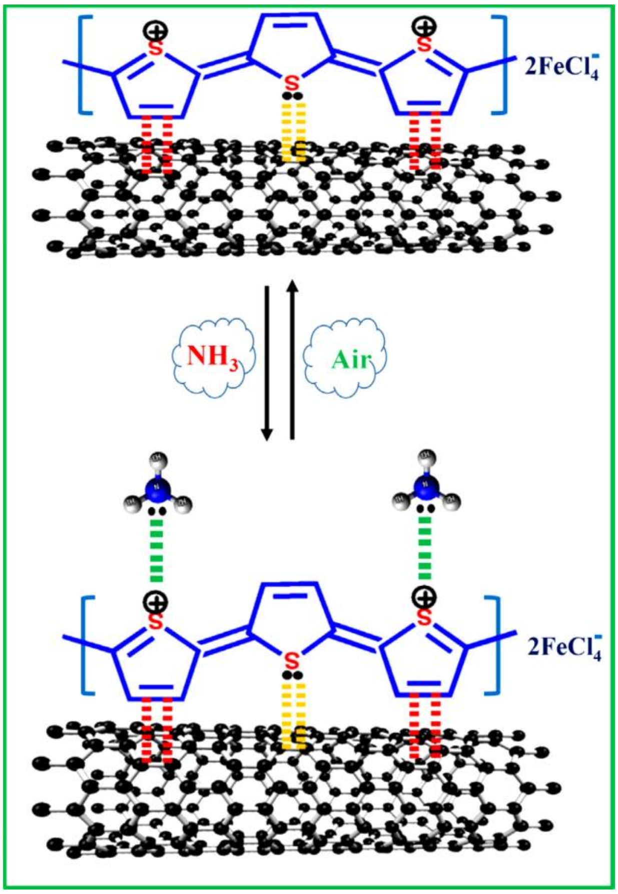

Figure 1.5. *Reversible ammonia sensing using polythiophene-carbon nanotube nanocomposites (adapted from Husain et al. 2020)*

Polythiophene-carbon nanotube nanocomposites are emerging as sensor materials which include electrochemical sensing, chemiresistor sensing, chemical sensing, mechanical sensing, and voltammetric sensing methods.[42,44,45,49,50] The development of low-cost portable sensors using conducting polymer nanocomposite systems is a new field of research applying its relevant properties. Carbon nanotubes are important materials distinguishable by their property of sensitivity to environmental changes. Surface functionalization with polymers and their conducting nature can further correlate with the sensing properties of carbon nanotubes. Zhang et al. reported developing a chemiresistive sensor using polythiophene functionalized carbon nanotubes to detect n-methylphenethylamine (NMPEA) and various volatile organic compounds. This chemiresistive sensor combined on circuit boards could develop wireless communication with a cell phone accessory or computer. Combining polymer components having various recognition groups to conducting carbon nanotubes enable them to adopt suitable sensing mechanism based on the property related to the recognition of the group on the polymer.[49] Hussain et al. established a reversible chemical sensor based on electrical conductivity difference for nanocomposites interacting with the analyte ammonia (see **Figure 1.5.**).[44]

1.6.2. Preparation of higher-order nanocomposites

Higher-order nanocomposites are emerging as an exciting way of preparing more efficient nanocomposites from carbon nanotubes- polythiophene binary nanocomposites.[51-55] The availability of an excellent structural platform helps to accommodate other materials on it. Therefore, the formation of well-oriented carbon nanotubes- conducting polymer nanocomposites is the prime factor which helps to build them up to higher-order structural architectures. Structural modification with additional fillers will lead to integrated properties of individual components. [51-53] Interaction of binary polythiophene-CNT nanocomposites with the other component would act as another important factor to form stable higher-order composites. This factor also acts as a reason for enhancement in the properties of nanocomposites.[51,52] A report on higher-order nanocomposites by Wan and co-workers revealed incorporation of Pt nanoparticles to polythiophene-carbon nanotubes nanocomposites could effectively enrich them as an electrochemical sensor for Bisphenol A detection.[52]

1.6.3. Supercapacitor applications

Incorporating polythiophene with carbon nanotube makes them a suitable combination for supercapacitor electrode applications by properly tuning their properties.[56-60] Carbon nanotubes are promising electrode materials due to their structural and electronic properties.[57,59] Electrical double-layer formation on the surface of carbon nanotubes is due to their capacitance performance known as electrical double layer capacitance (EDLC).[56-59] Polythiophene exhibits good redox properties with less percentage EDLC characteristics and thereby exhibits pseudo capacitance.[56,58] Many authors have reported polythiophene carbon nanotube nanocomposite based supercapacitors with high energy efficiency and good power density.[56-60] One study conducted by Zhang et al. reports the supercapacitor application of polythiophene-carbon nanotube nanocomposites obtained with electropolymerization in ionic liquid microemulsion followed by composites formation.[56] Binder and additive-free supercapacitor electrodes are more attractive. For capacitance performance, Lota et al. prepared binder-free electrodes with PEDOT and carbon nanotubes.[58] Literature studies revealed that many reports of polythiophenes nanocomposites with single and multiwalled carbon nanotubes can render good capacitor performance.[57]

1.6.4. EMI shielding

Electromagnetic interference is present in devices that utilize, transfer or distribute electrical energy. Increased use of cellular towers, electronic devices, wireless networks, etc., seeks more efficiency in material related to EMI shielding.[61] As nanotechnology progresses in various materials science fields, establishing efficient EMI (Electromagnetic Interference) shielding materials is considered a major area of research interest. Research related to EMI shielding materials pay attention to both synthesis and application. Continuous conducting fillers in nanocomposites for EMI shielding applications are most efficient rather than discontinuous fillers.[62,63] Conventional EMI shielding materials are metal-based systems with many demerits such as high density, low resistance to corrosion and poor mechanical properties. Using carbon nanotubes as the filler in nanocomposites provides good mechanical strength, improved electrical conductivity and high corrosion resistance. The incorporation of polythiophene with carbon nanotubes to obtain nanocomposites having well-organized conducting networks deliver an efficient platform for high EMI shielding efficiency (EMI SE). The use of polythiophene pursues more attention related to its remarkable characteristics, such as low density, high environmental stability, good conductivity, and potential flexibility. Developments of EMI shielding materials based on polythiophene and polythiophene derivatives in the doped state as composites with carbon nanomaterials were reported.[63-66] One of the recent reports by Preetham Bhardwaj et. al. is that polythiophene graphene grafted three-dimensional carbon fibre nanocomposites with good performance in antistatic and microwave shielding applications.[66]

1.6.5. Photovoltaic cells and photodiodes

Recently conjugated polymer-based photovoltaic devices have been developed vastly for their peculiar properties, including lightweight nature, flexibility, continuous conducting framework, and good energy conversion efficiency.[33,67-73] Along with good environmental stability, and processability, the association of efficient photoconversion groups as substituents in connection with the existing conjugated framework were reported.[69] Highest power conversion efficiency in polymer photovoltaic devices is achieved with bulk heterojunction (BHJ) materials rather than single component and bilayer solar cell materials.[33,73] Polythiophene could act as good electron donors in combination with suitable electron acceptor materials like C_{60} that offer maximum energy efficiency up to 7 %. Carbon nanotubes are considered good electron acceptor materials in BHJ photovoltaic devices with suitable polymeric materials like polythiophene derivatives due to ballistic conduction pathways and higher carrier movements. Planar nano-heterojunctions with conducting nanomaterials such as carbon nanotubes could define excellent energy-efficient substrates for exciton dissociation.[33] The impeding factor for obtaining good energy efficiency in such devices is (i) the aggregation tendency of carbon nanotubes on dispersing with polymeric materials and (ii) intermixed state of metallic and semiconductor carbon nanotubes. The exciton dissociation and photogeneration of photovoltaic nano-heterojunction of polythiophenes and carbon nanotubes/graphene were studied.[33,72,73] Rahman et al. demonstrated single-walled carbon nanotubes with nanostructured ITO electrodes for a photoelectric application. Here functionalized polymers were attached with carbon nanotubes with van der Waals and electrostatic interactions.[70] Later, a work was reported by Yu et al. in which covalently grafted regioregular poly(3-hexyl thiophene) with graphene was fabricated as a heterojunction photovoltaic device using a simple solution processing approach.[67] Another work by Habisreutinger and co-workers demonstrated P3HT-wrapped carbon nanotube nanocomposites to enhance the hole extraction efficiency of perovskite solar cells.[70] Recently, Ahmed et al. reported polythiophene carbon nanotube nanocomposites as counter electrodes in dye sensitized solar cells.[71]

1.6.6. Transistors

The area of flexible electronics with organic thin film transistors (OTFTs) and organic semiconductors is an emerging study which could potentially replace conventional inorganic semiconductor-based electronic materials.[74] Conducting polymers or other small organic molecules having semiconductor properties are attractive for their solution processability, low-cost manufacturing, lightweight and flexible nature and low-temperature synthetic routes. However, less charge mobility and the short lifetime of such electronic materials limited its application.[74-76]

Conducting carbon nanotubes are promising materials for integrating with conducting polymers in organic field effect transistor systems because of their one-dimensional nanostructure with electronic charge transfer capability.[76] Application of carbon nanotubes in such electronic materials still faces challenges from their self-bundling nature and practically infusible and insoluble character. A suitable combination of conducting polythiophene with carbon nanotube having electronic interaction between components and good morphology features is attractive to electronic material devices.[74-76] The switching speed of transistors is directly proportional to field effect mobilities (μ) and inversely proportional to channel length (L). Reducing the line width between the printed lines increases the transistor efficiency.[74] Park and co-workers fabricated OTFT electrodes of PEDOT: PSS/CNT having a small 7 μm channel length using an electrohydrodynamic jet printing technique. They demonstrated the composite with good dimensional stability also.[74] Soon after, another work done by Lee et al. developed PEDOT/PSS composite with single-walled carbon nanotubes that was reported as an efficient bilayer thin film transistor.[77] Recently, Kandpal and co-workers prepared poly(3-hexyl thiophene) (P3HT): MoS_2 : multiwalled carbon nanotube nanocomposites and fabricated as an electrochromic diode for rectification application by utilizing redox behavior of P3HT.[78]

1.6.7. Thermoelectric materials

Thermoelectric materials directly convert heat energy into electrical energy. The use of thermoelectric materials in the field of electrical energy production might promise sustainable development by converting waste heat energies released from various sources.[79] Efficiency of thermoelectric materials can be determined with good figure of merit and power factor.[80] Carbon nanotubes are striking as thermoelectric materials because of their excellent mechanical strength, high conductivity, thermal stability, low toxicity, and lightweight nature.[79-84] Incorporation of conducting polymers like polythiophene in composite form would be more better thermoelectric material as it could gain ease of processability, tunable molecular structures and mechanical flexibility.[79,81] Polythiophenes are environmentally stable, and many of the reports based on polythiophene and its derivatives show good thermal stability and low thermal conductivity; thereby, they are suitable for fabricating thermoelectric nanocomposites.[84] Recent reports on polythiophene-carbon nanotube nanocomposites, among which Hu and co-workers reported poly(3,4-ethylenedioxy thiophene)/carbon nanotube nanocomposites, which exhibited good thermoelectric performance with power factor 19.00±1.43 $\mu Wm^{-1}K^{-2}$.[79] He et al. prepared thermoelectric film made up of carbon nanotubes modified with thermally cleavable polythiophenes. The substrate-free thermoelectric film was prepared by solvent evaporation with the figure of merit of $3.1x10^{-2}$ and power factor of 28.8 $\mu Wm^{-1}K^{-2}$ at 25°C.[80]

Many other applications were also reported with polythiophene-carbon nanotube nanocomposites. Ma and co-workers reported headspace solid-phase microextraction (HS-SPME) based on gas chromatography for polycyclic aromatic hydrocarbons having low boiling points. They demonstrated the experiment in real soil samples containing naphthalene, acenaphthene, 1-methyl naphthalene and fluorene.[85] Ostrovsky et al. reported an innovative auditory neuron multielectrode array interfacing using polythiophene carbon nanotube nanocomposites for clinical cochlear implant systems.[86] Kwon and co-workers reported another application of carbon nanotubes web with carboxylated polythiophenes to assist battery electrodes for high performance.[87] Literature reports of applications of polythiophene-carbon nanotube nanocomposites revealed the importance of the combination of conducting polythiophene and the conducting filler carbon nanotubes in commercial as well as industrial applications. There are many possibilities for developing innovative nanocomposites using more efficient synthetic strategies or tuning the properties of components of nanocomposites for suitable applications.

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