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Chapter 4

Synthesis of Organic–Inorganic Nanohybrids-Based Polymeric Nanocomposites



Ahmad Shakeel, Komal Rizwan, Ujala Farooq, and Saima Yasin

1 Introduction

Polymeric nanocomposites are of great importance in different fields [74]. Synthesis of hybrid nanocomposites based on organic (polymer) and inorganic components has gained serious attention of researchers due to their extensive range of applications in biomedical, environment, and energy-related areas. Progress in polymer science has created an opportunity to produce an extensive range of materials having superior mechanical, electroactive, and thermal properties [55]. In addition to this direction of developing new nanocomposites, researchers are continuously exploring novel techniques to prepare hybrid nano-materials by combining desirable features of polymers and nanoparticles. In literature, various challenges have been mentioned in preparing polymeric nanocomposites with desired features [68, 87]. The major limitation for producing nanocomposites at a commercial scale is the absence of

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economical methods for nanoparticle dispersion into the polymer matrix. The aggregation of nanomaterials hinders its benefits associated to the dimension (nanoscale), and hence, well-dispersed and isolated nanoparticles within the polymer matrix are needed. Hence, there is a need to develop synthesis methods that are effective on nanoscale yet appropriate for macroscopic processing. Researchers have developed a range of synthesis methods for preparing polymer nanocomposites which include direct processing, in situ polymerization, sol–gel, etc. [44]. Table 1 provides an overview of benefits and limitations of different synthetic routes for polymer based nanocomposites.

Table 1 Benefits and limitations of different conventional techniques for preparing polymeric nanocomposites

Synthesis method	Benefits	Limitations
Melt blending	<ul style="list-style-type: none"> • Well-adapted for mass-scale industrial applications • Economical • Wide spectrum of materials can be employed • Environmental-friendly 	<ul style="list-style-type: none"> • Poor dispersion of nanomaterials, particularly at higher concentrations • High temperature and/or shearing is required
Solution blending	<ul style="list-style-type: none"> • Appropriate for membrane/film formation • Better dispersion of nanofiller • Recommended for thermally sensitive polymer 	<ul style="list-style-type: none"> • Higher capital cost • Compatibility between polymer and solvent is critical • Aggregation of polymer chains after solvent evaporation • Environmental restrictions
In situ polymerization	<ul style="list-style-type: none"> • Enhanced dispersion of nanofiller • Both thermoplastics and thermosets can be used • Permits the grafting or exfoliation of polymers on filler surface 	<ul style="list-style-type: none"> • Complex processing steps • Expensive reactants • Not appropriate for all types of polymers and elastomers
Sol–gel	<ul style="list-style-type: none"> • Higher-quality product • High potential for developing hybrid materials 	<ul style="list-style-type: none"> • Unsatisfactory bonding • High permeability • Expensive raw material
Electrochemical	<ul style="list-style-type: none"> • Short reaction time • Simple and easy operation • Eco-friendly • High-purity product 	<ul style="list-style-type: none"> • Limited surface area of electrode which hinders large-scale production

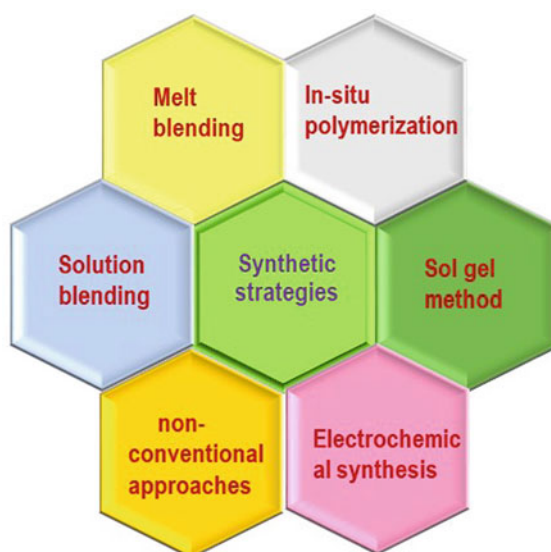
2 Synthesis Routes

Synthesis of polymer nanocomposites via suitable processing technique is vital in order to get high-performance nanocomposites. Several approaches have been developed to prepare polymeric nanocomposites including in situ preparation and direct processing [87]. The direct mixing process involves the dispersion of desired nanomaterial into polymer matrix either by melt, mechanical, or solution blending. However, uniform dispersion of nanomaterial is a major challenge of this approach [79]. In contrast, in situ polymerization technique solves the above-mentioned problem by creating a polymer microenvironment to synthesize the desired nanomaterial from its precursors through series of reactions [54]. This method is gaining interest because of the ease of controlling the morphology and particle size within the composite. The most important processing methods (Fig. 1) for polymer nanocomposites have been explained below.

2.1 Direct Processing

This technique is based on the dispersion of nanofiller into the polymer matrix using heating, solvent, or mechanical action, which makes it a top-down approach. This method is extensively used for preparing polymeric composites due to the sustainable bulk production and lower cost. This approach results in nanocomposites with one- or two-dimensional structures having particles within the range of submicron to nanoscale. In order to attain homogenous dispersion of filler into the polymer matrix,

Fig. 1 Various routes for synthesis of organic–inorganic nanohybrids-based polymer nanocomposites



several approaches have been developed such as adding stabilizers or dispersants, adjusting process parameters (mixing speed, temperature, time, etc.), and chemical modification of polymers [87]. There are two main approaches for direct mixing of fillers and polymer.

2.1.1 Melt Blending

Melt blending is a typical procedure to disperse nanofiller into the continuous matrix for producing thermoplastic or elastomer-based nanocomposites. It involves the usage of a suitable processing machine (i.e., extruder or ultrasonicator) for applying required shear forces at high temperatures (usually above the glass transition temperature of polymer) in order to melt the polymer, followed by the addition and mixing of the filler for achieving uniform filler distribution [79]. Typically, melt mixing is performed in the presence of an inert gas like nitrogen, argon, etc. [33]. Melt mixing possesses several inherent advantages such as environmental-friendly due to the absence of any solvent, economical, and convenient because of the compatibility with large-scale industrial processes (i.e., extrusion and injection molding) and applicability for both polar and apolar polymers [54].

Saleh and Jawad prepared the graphene nanoplatelet (Gr NP) and polystyrene-based nanocomposites by dispersing Gr NP into the polystyrene matrix using a small batch mixer at 190 °C and a mixing speed of 100 rpm for 10 min. The prepared nanocomposite was then pressed in a compression molding machine at 25 MPa and 220 °C for 10 min, in order to prepare the samples for characterization [3]. Alig and colleagues explained the correlation between the morphologies obtained for carbon nanotube-based nanocomposite and the processing conditions [6]. Furthermore, they described the dispersion process of filler into the polymer matrix in four steps: (i) wetting of filler aggregates with polymer, (ii) infiltration of polymeric chains into the filler aggregates, (iii) dispersion of filler aggregates by erosion and rupture, and (iv) distribution of individual carbon nanotubes into the polymer. Similarly, different polymeric nanocomposites such as graphene-polypropylene, exfoliated graphite-poly(methyl methacrylate), graphene-polycarbonate, graphene oxide-poly(ethylene-2,6-naphthalate) have been prepared via melt blending for different applications [76].

However, the distribution of filler into the polymer matrix is less efficient in case of melt blending as compared to the solution blending due to the higher viscosity of melt, which can be further enhanced by modifying the surface of fillers, tuning the interactions between the filler and polymer matrix, using solvent together with melt processing, etc. For instance, the distribution of graphene nanoplatelet into the polypropylene matrix was enhanced by using solvent (mixture of *p*-xylene and *N,N*-dimethylformamide)-assisted melt blending process, which eventually resulted in better mechanical properties of nanocomposites [47].

In another study, the dispersion of filler (carbon black, carbon nanotube, and graphene nanoplatelet) into the polymer matrix was enhanced by installing an ultrasonic device at the extruder [96]. The vibration caused by the ultrasonic device helped

in breaking the filler aggregates (or removing the air trapped inside the polymer) during mixing and hence, enhancing the filler distribution. Likewise, the dispersion of graphene sheets in polypropylene (PP) was improved by modifying PP by using triethylaluminum and rare-Ethylenebis(1-indenyl) zirconium dichloride (EBIZrCl₂), which resulted in terminally hydroxylated PP [21]. The modified PP and graphene sheets were first heated in tetradecane at 200 °C followed by melt mixing with PP, in order to prepare the nanocomposite.

Moreover, the application of high temperature for melt blending is known to damage the surface modification of fillers, leading to inhomogeneous distribution of filler. For instance, alkyl ammonium modified organoclays was observed to degrade at temperatures greater than 140 °C, whereas the melt blending temperature was within the range of 190–220 °C [2]. This issue can be solved by using the thermally stable modification of filler or by performing the blending process at lower temperatures [58]. Hence, in order to attain good dispersion of filler and better mechanical properties of nanocomposites, several parameters are important to consider including surface modification of fillers, processing conditions, and compatibility between filler and matrix.

2.1.2 Solution Blending

Solution blending is another common processing method for producing polymeric nanocomposites. The overall procedure can be divided into three steps: (i) dispersion of nanofiller in appropriate solvent using agitation, (ii) mixing of polymer and nanofiller solutions, and (iii) removal of solvent by evaporation or solvent coagulation [29]. Polymers are typically dissolvable in a variety of solvents including water, cyclohexane, chloroform, acetone, dimethylformamide (DMF), toluene, tetrahydrofuran, etc. [54]. The selection of suitable solvent is mainly governed by the dispersibility of nanofiller and/or solubility of polymer [7]. The same solvent can be used for both nanofiller and polymer matrix. This method facilitates the dispersion of nanofiller into the polymer matrix with the help of solvent. However, this synthetic approach is not suitable for the insoluble polymers. Moreover, the removal of solvent imposes environmental restrictions, which limits its applicability from small scale to industrial scale [79].

For example, Marroquin and colleagues prepared chitosan-based nanocomposite by dispersing Fe₃O₄ and multi-walled carbon nanotubes (MWCNTs) using solvent blending [52]. Firstly, the nanofillers (Fe₃O₄ and MWCNTs) were distributed in distilled water by ultrasonication for 1 h, followed by the addition of chitosan and acetic acid. Magnetic stirring of the mixture was performed for 2 h and then ultrasonicated for 30 min. The prepared mixture was then degassed and vacuum dried to produce the nanocomposite. Likewise, Taha and Alzara reported the development of polyvinyl alcohol (PVA) and SrTiO₃-based nanocomposites [78]. PVA powder was firstly dissolved in distilled water at 80 °C and stirred for 60 min. SrTiO₃ powder was then incorporated into the PVA solution and agitated for 1 h. The resulting mixture

was poured into a glass dish and dried at 80 °C. In another study, the poly(ether-ether-ketone) (PEEK) and MWCNTs-based nanocomposites have been synthesized via solution blending [39]. First of all, *N*-methyl-2-pyrrolidone (NMP) solvent was used to disperse MWCNTs by ultrasonication for 15 min. PEEK powder was dissolved in dichloroacetic acid (DCA) at 185 °C and cooled down to room temperature. The PEEK solution was then added into the MWCNTs solution and manually shaken to mix the solution. The resulting mixture was filtered and washed with methanol and deionized water to remove the residual solvents. The obtained precipitates were dried at 80 °C to obtain nanocomposite powder, which were finally injection molded to prepare the required samples.

In addition to the single polymer matrix, binary/ternary polymer mixture has also been reported in literature to prepare nanocomposites. For example, Lu and colleagues produced nanocomposites, based on boron nitride (BN) as a filler and mixture of polystyrene (PS) and polypropylene (PP) as a polymer matrix, through solution blending method [48]. Polystyrene particles were firstly dissolved in DMF at 80 °C via magnetic stirring followed by the addition of BN platelets into the PS solution and vigorously stirred. The PP microspheres were then added into the PS/BN mixture and coagulated in deionized water. The resultant granules were filtered, washed by DI water, and dried at 80 °C. The characterization samples were prepared by pressing the granules at 170 °C under 10 MPa for 15 min. In order to further enhance the filler distribution and to avoid the filler aggregation and stack up problems, grafting to/from and chemical functionalization approaches are typically used to prepare polymeric nanocomposite. For instance, phase transfer techniques [22], lyophilization methods [20], and surfactants [46] were utilized to facilitate the solution blending of graphene into the polymer matrix.

2.2 *In Situ Polymerization*

In situ polymerization method is a bottom-up approach in which polymers and nanomaterials are formed within the final composite system with the help of series of chemical reactions catalyzed by suitable agents (radiation, heat, initiator, catalyst, etc.) [7]. Typically, there are three approaches for preparing polymeric nanocomposites using in situ method: (i) Nanomaterial precursor is preloaded onto the polymer matrix, which forms required nanomaterial after chemical reaction, (ii) monomers (instead of polymer matrix) along with the nanomaterials forms the starting mixture, and (iii) both monomers and nanomaterial precursors are mixed through an appropriate solvent, followed by the in situ polymerization and chemical reactions to form nanomaterials [87]. The second option (combination of monomer and nanomaterial) avoids the aggregation of nanofiller within the polymer matrix, which eventually results in excellent dispersion of nanofiller and better interfacial interaction with polymers. Moreover, this second approach also alters the physical characteristics of the resultant polymer nanocomposite due to the intercalation or grafting of polymer structures within/on the layers of nanomaterials. This intercalation and homogeneous

distribution of nanomaterials within the polymer matrix allows the development of partially exfoliated structures [29]. However, this method also has several limitations such as requirement of expensive reactants, complex processing steps, and not suitable for all types of polymers and elastomers [79].

The production of polypyrrole and reduced graphene oxide (r-GO)-based nanocomposites using chemical oxidative in situ polymerization method has been reported [75]. The nanomaterial was dispersed in CTAB/APS mixture using ultrasonic bath for 10 h. Pyrrole monomer was then added to this stable suspension, and the required nanocomposite was obtained after the polymerization. The prepared nanocomposite was washed, followed by overnight drying in a vacuum oven at 60 °C. Likewise, polymeric nanocomposites based on polyaniline and montmorillonite using in situ polymerization approach have been fabricated [41]. In short, clay particles were distributed in distilled water using ultrasonicator for 10 min. Then, the solution of aniline in HCl was slowly added to the clay suspension, followed by the slow addition of APS (oxidizing agent) under magnetic stirring. For complete polymerization at 0 °C, the suspension was stirred for 4 h and a dark green polymer nanocomposite was obtained by vacuum filtration. The obtained composite was washed with water and acetone followed by overnight drying in vacuum oven at 50 °C.

Hou and colleagues reported the preparation of nanocomposites based on waterborne polyurethane (WPU) as a polymer matrix and combination of graphene oxide (GO) and carbon black (CB) as nanomaterials [36]. First of all, the mixture of monomer (isophorone diisocyanate) and polypropylene glycol was stirred in a container at 85 °C for 3 h. Then, mixture of NMP/DMPA along with the few drops of dibutyltin dilaurate was incorporated into the system under stirring for 2 h, to obtain NCO-terminated prepolymer. Nanomaterials (CB and GO) were then dispersed in the prepared mixture and stirred at high speed for 2 h. Neutralizer (TEA) and viscosity reducer (acetone) were added, and the mixture was stirred for 45 min and cooled down to 45 °C. Mixture of EDA and water was then added dropwise under extensive stirring, and the nanocomposite dispersion was finally obtained. Similarly, the highly conductive polymeric nanocomposites based on polypyrrole and Zeolite nanoparticles using in situ polymerization method have been synthesized [35]. Firstly, nanoparticles were dispersed into the mixture of distilled water and CH₃Cl by stirring for 1 h. Then, monomer was incorporated into the suspension with continuous stirring for 4 h. After 3 h, the obtained composite material was filtered, washed, and dried in an oven. The oxidizing agent (FeCl₃) was then added dropwise to initiate the polymerization process at 0 °C or 25 °C. In another study, Nicosia and colleagues compared the photocatalytic activity of TiO₂-poly(methyl methacrylate) nanocomposites synthesized by solution blending and in situ polymerization for water pollution remediation [60]. The results revealed that the chemical-physical interactions between the nanomaterials and polymer matrix was significantly influenced by in situ polymerization approach, which eventually boosted the photocatalytic degradation of dyes.

2.3 Sol–Gel Method

Synthesis of polymeric nanocomposites via sol–gel method is an old technique; however, recently, it is getting famous for preparing structurally advanced and functionalized hybrid materials. This method is quite similar to the in situ polymerization method, which involves the nanomaterial precursor and polymer matrix along with the suitable reagent (solvent, heating, radiation, etc.). In short, the nanomaterial precursor is dissolved in a suitable solvent and mixed with polymer matrix in precise molar ratios [87]. This mixture then undergoes a series of hydrolytic (or non-hydrolytic in case of organic solvents) and condensation reactions followed by nucleophilic substitution reaction facilitated by polymer matrix, which results in sol formation. Sol can be understood as a colloidal suspension having almost non-interacting particles. This sol then converts into a (wet) gel system due to the crosslinking reactions and the formation of interconnected network of particles facilitated by agitation and/or temperature changes. This wet gel eventually leads to the formation of polymeric nanocomposite after the drying procedure [67]. Typical nanomaterials used for preparing polymer nanocomposites through sol–gel technique include alumina, silica, titania, and vanadia. However, silica-based materials are commonly used because of their lower cost and highly stable Si–O bond [65]. In this method, polymer nanocomposites are synthesized by having either physical or chemical interactions between the inorganic nanomaterials and organic polymers. This method does not involve separate steps or additional energy to distribute the nanomaterials within the polymer matrix. Other benefits of this technique include high purity and uniformity of product, low sintering temperature, easily controllable reaction, better forming ability, etc. On the other hand, this method also inherits some drawbacks such as requirement of expensive and toxic precursors, difficulty in making crystalline nanocomposites, brittle fracture of product due to the volatility of solvents, and longer preparation times (i.e., few days to weeks) [79].

For instance, Factori and colleagues reported the development of polymer nanocomposite based on ZnO and poly(vinyl alcohol) (PVA) via sol–gel technique under microwave heating [26]. PVA was firstly dissolved in hot DI water followed by the incorporation of ethyl alcohol, acetic acid, and $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ into the solution. The prepared solution was then poured into the round bottom flask and placed on a microwave reactor. The mixture was maintained at 100 °C via microwave irradiation of varying power as a heating source, which eventually resulted in polymer nanocomposite. Phase inversion phenomenon was further used to prepare polymer nanocomposite films. Likewise, Besancon et al. used non-hydrolytic sol–gel technique along with reactive extrusion to produce polypropylene (PP) and titanium dioxide-based polymer nanocomposites [16]. PP was firstly added into the co-rotating twin screw extruder at 200 °C followed by the addition of nanomaterial precursor and other reactants. After adding reactants, the temperature was increased up to 240 °C in a certain region of extruder. A vacuum pump was installed to extract the by-products. The prepared nanocomposite was obtained at the exit of die and cooled under air flow. In order to further remove the residual by-products, Soxhlet extraction technique was

used with ethyl acetate as a solvent at 110 °C for 72 h. In another study, Behnam and colleagues fabricated the polyurethane and carbon nanotubes (CNTs)-based polymer nanocomposites with enhanced thermal stability using sol–gel method [14]. In short, CNTs were distributed in tetrahydrofuran (THF) and sonicated for 30 min followed by the addition of polyurethane, tetraethyl orthosilicate (TEOS), and DI water into the mixture. Then, formic acid was incorporated into the mixture under continuous stirring until complete solvent evaporation.

2.4 Electrochemical Synthesis

Electrochemical synthesis is a simple chemical procedure for preparing polymeric nanocomposites, which is typically performed on electrochemical workstation. Commonly used monomers for this processing technique include aniline, thiophene, and pyrrole [65]. Generally, there are three electrodes present during the electrochemical synthesis: (i) working electrode, (ii) counter electrode, and (iii) reference electrode. This methodology is an excellent way to directly prepare the polymeric nanocomposite film on the surface of electrode. The main controlling parameters of electrochemical synthesis process include current density or applied potential and the amount of charges integrated in the system. This approach offers many advantages including operational simplicity, short reaction time, eco-friendly, and easier to control. Moreover, this technique avoids the use of oxidant; hence, greater purity of the final product can be achieved. However, the mass production using this technique is not feasible because of the limited surface area of the working electrode [79].

For example, Fani and colleagues reported the fabrication of polymeric nanocomposite biosensor based on polypyrrole, reduced graphene oxide, and gold nanoparticles via electrochemical method [28]. In short, the working electrode (screen printed carbon electrode) was first cleaned with H_2SO_4 and DI water followed by drying at room temperature. Then, the mixture of reduced graphene oxide, pyrrole, HAuCl_4 , and l-Cys in PBS was sonicated for 20 min and electrodeposited onto the electrode surface using voltammetric sweep. Similarly, Saeb and Zenali synthesized the polymeric nanocomposite (polyaniline- TiO_2 -gold nanoparticles) sensor for hydrazine detection using electrochemical technique [71]. Firstly, the electrode (glassy carbon electrode) surface was polished using 0.3 mm alumina-based abrasive paper followed by rinsing with deionized (DI) water. The electrode was then sonicated for 10 min in a mixture of water and ethanol, in order to eliminate the remaining alumina particles. TiO_2 nanoparticles were dispersed in DI water by using ultrasonicator for 30 min and then dropped onto the electrode surface and allowed to dry at ambient temperature. After drying, the electrode was rinsed with DI water to remove the residual nanoparticles. The prepared electrode was then submerged in a mixture of aniline and H_2SO_4 , and the electro-polymerization of aniline was performed with a potential window of -0.2 to 0.8 V for 5 cycles. Then, the solution of Au in chloroauric acid was electrodeposited on the electrode surface using cyclic voltammetry.

In another study, Yan and colleagues fabricated the multi-layer polymer nanocomposite (chitosan silver nanoparticles) coating for pH-dependent controlled release of active compounds using a two-step electrochemical synthesis approach [86]. The electrodeposition of chitosan was performed by using a two-electrode workstation: (i) working electrode made of stainless steel wire of 0.4 mm diameter and (ii) counter electrode made of platinum wire. All the electrodes were sonicated in ethanol, acetone, and water for 5 min for removing the impurities. Then, the two electrodes were partially immersed in chitosan solution (chitosan and nitric acid), and a constant current density (2.5 A m^{-2}) was applied for particular time. In the next step, the chitosan-coated electrode was soaked in the solution of AgNO_3 and NaNO_3 for 12 h to allow complete loading of silver ions. The electrode was removed from the solution, then gently washed with DI water followed by the application of a cathodic voltage (5 V) for particular time to allow the electrochemical reduction of silver ions to the nanoparticles. In the end, the prepared polymeric nanocomposite was washed with DI water, removed from the electrode surface using tweezers, and freeze-dried for further analysis.

2.5 Nonconventional Methods

In addition to the above-mentioned techniques, researchers have investigated some other approaches as well such as template-based synthesis, electrospinning to produce polymer nanocomposites with improved properties due to the better dispersion of nanofiller into the polymer matrix.

2.5.1 Template-Based Synthesis

In this approach, an inert material acts as a framework or skeleton for the in situ polymerization of monomer. The template-based synthesis is typically used to prepare polymer nanocomposites with porous structures. For example, Wang et al. reported the sacrificial template synthesis of N-doped carbon, molybdenum disulfide (MoS_2) nanosheets, and polypyrrole (PPy)-based hollow nanocomposites for sodium storage performance [84]. The result showed that the MoS_2 nanosheets were protected by PPy and N-doped carbon via Mo–N bonds, which inhibits the volume change and prevents the aggregation and fracture of electrode. Moreover, the external PPy and inner N-doped carbon effectively accelerated the Na-ion transport. Likewise, Zhang et al. [94] produced PPy nanostructure with platinum and gold nanoparticles using a reactive template based on manganese oxide (MnO_2) nanowires. The reactive template (MnO_2 nanowires) induced one-dimensional polymerization of monomers, and the simultaneous dissolution of template provided the hollow tube-like structure. The nanoparticles content in the prepared nanocomposites can simply be varied by manipulating the amount of precursors. However, this technique is not suitable when complex composite system is required.

2.5.2 Electrospinning Method

This method is typically used to prepare polymer nanocomposites in the form of nanofibers by applying high voltage. This approach is a modification of solvent blending; i.e., polymer and nanomaterial are dispersed/dissolved into a solvent to obtain homogeneous solution. Then, a hydraulic syringe is filled with this solution, and the nanofibers are extruded under high voltage to overcome the surface tension. For example, [19] utilized electrospinning technique to prepare poly(ethylene oxide) or poly(vinyl pyrrolidone) and iron oxide nanoparticles-based nanofibrous network. Metal organic framework based on poly(methyl methacrylate) and zirconium via electrospinning technique has been synthesized [53]. This approach is highly suitable to produce uniform morphology and size of nanofibers. Table 2 provides an overview of different polymer nanocomposites along with their synthesis route.

3 Conclusion

Synthesis of hybrid nanocomposites based on organic (polymer) and inorganic components has gained serious attention of researchers due to their extensive range of applications. Integration of inorganic nanomaterials to polymeric components is excellent tool to confer their unique characteristics to polymeric materials. Polymeric nanocomposites showed great conductivity, large surface area, high porosity, significant catalytic, electric and optical potential. These composites may possess weak interactions like hydrogen bonding and Van der Waals forces, and some nanocomposites possess strong interactions at the interface as well. Different synthetic routes have been developed for the preparation of polymeric nanocomposites as in situ polymerization, melt blending, solution blending, sol–gel, and electrochemical synthesis. The polymeric nanocomposites synthesized through these approaches showed intriguing features which have been explored in applications of electronic devices, different sensors, and in biomedicines. Different synthetic approaches have different limitations such as direct mixing process involves the dispersion of desired nanomaterial into polymer matrix either by melt, mechanical, or solution blending. However, uniform distribution of nanomaterial is a major challenge of this approach. In contrast, in situ polymerization method solves the above-mentioned problem by creating a polymer microenvironment to synthesize the desired nanomaterial from its precursors through series of reactions. This method is gaining interest because of the ease of controlling the morphology and particle size within the composite. Still problem exists to enhance the conductivity control, the morphology, shape size, and composition of polymeric nanocomposites, so future developments must focus on improving the synthetic protocols, and novel assembly approaches should be introduced.

Table 2 Overview of different polymer nanocomposites prepared via different techniques

Polymer material	Inorganic material	Composite preparation method	Application	Refs.
Polyamide	Organoclay	In situ polymerization	–	[10]
Polyaniline	Montmorillonite	In situ polymerization	Removal of dye	[41]
Polypyrrole	Molybdenum diselenide	In situ polymerization	Photocatalytic degradation of dye	[57]
Poly(methyl methacrylate)	Titanium dioxide	In situ polymerization	Photocatalytic degradation of xenobiotic	[60]
Polyaniline	Graphene oxide-camphorsulfonic acid	In situ polymerization	Electromagnetic wave absorption and anti-corrosion	[85]
Polyaniline	Graphene oxide	In situ polymerization	Removal of dye	[4]
Polyaniline	Manganese dioxide	In situ polymerization	Energy storage	[38]
Polypyrrole	Zeolite	In situ polymerization	Electrode for super-capacitors	[35]
Polyimide	Paraffin-barium titanate	In situ polymerization	Energy storage	[64]
Polyaniline-chitosan	Montmorillonite	Intercalation and in situ polymerization	Removal of dye	[56]
Poly(<i>o</i> -anisidine)	Carbon nanotubes-graphene	In situ polymerization	Electrode for super-capacitors	[17]
Poly(methyl methacrylate)	Graphene nanoplatelets	In situ polymerization	Gamma-ray absorption	[15]
Polyaniline	Phosphotungstic acid-titania	In situ polymerization	Anode for methanol fuel cells	[45]
Polyaniline	Reduced graphene oxide-zinc oxide	In situ polymerization	Electrode for super-capacitors	[62]

(continued)

Table 2 (continued)

Polymer material	Inorganic material	Composite preparation method	Application	Refs.
Polyaniline	Graphene oxide-titanium dioxide	In situ polymerization	Photocatalytic degradation of dye	[12]
Polyaniline	Cadmium sulfide quantum dots	In situ polymerization	Solar cell and photovoltaic	[66]
Polyaniline	Graphene oxide-manganese sulfide	In situ polymerization	Electrode for super-capacitors	[88]
Polypyrrole	Zinc spinel ferrite-silicon dioxide	Sol-gel and in situ polymerization	Electromagnetic wave absorption	[32]
Polypyrrole	Nickel zinc ferrite	In situ polymerization	Degradation of organic pollutants	[59]
Polypyrrole	Zinc ferrite-graphitic carbon nitride	In situ polymerization	Degradation of organic pollutants	[23]
Poly(methyl methacrylate)	Reduced graphene oxide-hematite	In situ polymerization	Energy storage	[82]
Waterborne polyurethane	Graphene oxide	In situ polymerization	Anti-corrosion	[36]
Polypyrrole	Aluminum fumarate metal-organic framework	In situ polymerization	Removal of heavy metal	[90]
Polyaniline	Nickel zinc ferrite	In situ polymerization	Electromagnetic wave absorption	[43]
Poly(butylene succinate)	Nano-boehmite	In situ polymerization	–	[40]
Poly(methyl methacrylate)	MXene-zinc oxide	In situ polymerization	Dielectric materials	[83]
Polyindole	Copper-alumina	In situ polymerization	Sensing of ammonia gas	[72]
Polypyrrole	Titanium dioxide	Polymerization followed by sonication	Degradation of organic pollutants	[89]

(continued)

Table 2 (continued)

Polymer material	Inorganic material	Composite preparation method	Application	Refs.
Polythiophene	Zinc oxide	Sol-gel and in situ polymerization	Degradation of organic pollutants	[27]
Poly(vinyl alcohol)	Zinc oxide	Microwave-assisted sol-gel	UV shielding and anti-microbial	[26]
Polypropylene	Titanium dioxide	Sol-gel and reactive extrusion	–	[16]
Poly(vinyl alcohol)	Zinc oxide	Sol-gel and self-propagation	Electrode material and electrochemical sensor	[1]
Poly(vinyl chloride)	Cadmium sulfide	Sol-gel and spin coating	Photocatalytic degradation of dye	[70]
Polyamide	Hydroxylated boron nitride	Melt blending	Thermal management	[34]
Poly(methyl methacrylate)	Alumina	Melt blending	–	[9]
Poly(vinyl alcohol)-poly(vinyl pyrrolidone)	Montmorillonite	Melt blending	Dielectric material	[73]
Poly(lactide)-polycaprolactone	Silicon dioxide-multi-walled carbon nanotubes	Melt blending	EMI shielding	[49]
Polypropylene	Multi-walled carbon nanotubes	Melt blending	Wear resistance	[63]
High-density polyethylene	Calcium carbonate	Melt blending	Dielectric material	[5]
Polypropylene-poly(lactide)	Nanoclay	Melt blending	Biodegradable material	[51]
High-density polyethylene	Electrochemically exfoliated graphene	Melt blending	–	[11]
Linear low-density polyethylene-poly(ethylene-co-methyl acrylate)	Graphene-carbon nanotubes	Melt blending	–	[61]

(continued)

Table 2 (continued)

Polymer material	Inorganic material	Composite preparation method	Application	Refs.
Poly(butylene adipate-co-terephthalate)	Multi-walled carbon nanotube-zinc oxide	Melt blending	Packaging material	[30]
Poly(vinyl alcohol)	Strontium titanate	Solution blending	Dielectric materials	[78]
Chitosan	Graphene oxide	Solution blending	Electrical and opto-electronic devices at high frequency	[25]
Poly(ether-ether-ketone)	Multi-walled carbon nanotubes	Solution blending	High-temperature applications	[39]
Poly(ethylene-co-vinyl acetate)	α -zirconium phosphate	Solution blending	–	[95]
Poly(vinyl pyrrolidone)-poly(ethylene oxide)	Alumina-silica	Solution blending	Optical and opto-electronic devices	[24]
Polyaniline-sulfonated polyetherimide	Sodium alamate-multi-walled carbon nanotubes-titanium dioxide	Solution blending	Hydrogen storage	[13]
Polystyrene-polypropylene	Boron nitride	Solution blending and hot pressing	Thermal management material	[48]
Polyurethane	Functionalized graphene-functionalized carbon nanotubes	Solution blending	–	[69]
Poly(vinyl alcohol)	Graphene oxide-sodium montmorillonite	Solution blending and evaporation	Removal of dye	[8]
Poly(vinyl alcohol)-poly(vinyl pyrrolidone)	Nanographene	Solution blending	–	[37]
Polypyrrole	Reduced graphene oxide-gold nanoparticles	Electrochemical synthesis	Virus DNA detection	[28]

(continued)

Table 2 (continued)

Polymer material	Inorganic material	Composite preparation method	Application	Refs.
Poly(<i>N</i> -phenyl- <i>o</i> -phenylenediamine)	Titanium carbide-titanium dioxide	Electrochemical synthesis	Uric acid detection	[91]
Chitosan	Silver nanoparticles	Electrochemical synthesis	Biomedical and tissue engineering	[86]
Polypyrrole	Reduced graphene oxide-multi-walled carbon nanotubes	Electrochemical synthesis	Dopamine detection	[42]
Polyaniline	Titanium oxide-gold nanoparticles	Electrochemical synthesis	Hydrazine detection	[71]
Polyaniline	Reduced graphene oxide-gold nanoparticles-manganese dioxide	Electrochemical synthesis	Electrode for super-capacitors	[80]
Polyaniline	Reduced graphene oxide-silver nanoflower	Electrochemical synthesis	DNA detection	[81]
Poly(3,4-ethylenedioxythiophene)-poly(thiomethyl 3,4-ethylenedioxythiophene)	Gold nanoparticles	Electrochemical synthesis	Nitrite detection	[31]
Polyaniline	Graphitic carbon nitride-cadmium oxide	Electrochemical synthesis	Detection of antibiotics	[18]
Chitosan	Manganese oxide-aluminum oxyhydroxide	Electrochemical synthesis	Removal of organic pollutants	[77]
Polyaniline	Graphitic carbon nitride-titanium dioxide	Electrochemical polycondensation and soaking adsorption	Removal of organic pollutants	[97]

(continued)

Table 2 (continued)

Polymer material	Inorganic material	Composite preparation method	Application	Refs.
Poly(vinyl pyrrolidone)	Bismuth oxybromide	Precipitation	Removal of organic pollutants	[92]
Polythiophene	Bismuth oxyiodide	In situ precipitation	Removal of organic pollutants	[50]
Polythiophene	Manganese oxide	Mechanical mixing and sonication	Removal of organic pollutants	[98]
Polyaniline	Indium oxide	Impregnation method	Removal of organic pollutants	[93]

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