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Characterizations of Polymers Doped with Oxides Metals Nanoparticles and Their Applications: A Review

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Abstract

Polymer materials are of significant interest due to their diverse applications (low density, strong mechanics, cost-effective, and intricate shaping). Their potential is further enhanced by doping, which allows control over optical and electrical properties by manipulating the dopant type, concentration, and interaction with polymer chains. Similarly, incorporating oxides and metal nanoparticles modifies physical characteristics of polymers. The surface characteristics and properties of the filler significantly influence the final thermal, mechanical, optical, magnetic, or conductive properties of the nanocomposites. This is particularly relevant for energy and environmental applications where high thermomechanical properties, rheology, and heat stability are crucial. The creation of novel composites with synergistic or complementary behaviors between polymers and inorganic materials is anticipated due to the distinct electrical and optical properties of nanoscale inorganic fillers compared to their bulk counterparts. Research on metal oxide nanoparticles (ZnO, TiO_2, SnO_2) and their application in PANI-based nanocomposites for gas detection exemplifies this approach. Combining organic and inorganic materials unlocks exciting possibilities, paving the way for new applications like biosensors, gas sensors, conductive paints, drug delivery, and rechargeable batteries. Blending polymers with various inorganic nanoparticles further expands the possibilities for creating diverse nanostructures.

Introduction

Even at low nanoparticle concentrations, nanocomposites made by combining nanodispersions with polymers exhibit notable increases in their properties. These materials have a wide range of uses as nanocoatings, are distinguished by their nanoscale dimensions, which range from 1 to 100 nanometers, and aid in the development of conventional processing methods [1]. A great deal of research has been done on the integration of different nanoparticles with polymers to create nanocomposites. The nanoparticles have been categorized into fibrils (1D), sheets (2D), and particles (3D) according to their nanoscale size [2]. These nanoparticles are different from traditional microparticles utilized in composite materials because of their large surface area [3]. The characteristics of polymer materials, which are well-known for their wide range of uses and unique qualities, including low density, shape-forming ability, adjustable electrical behavior, mechanical strength, and affordability, are very advantageous to scientific study [4]. Doping polymers to increase their strength is a significant field of study that involves further manipulating the qualities of the polymers, including their physical characteristics.

It demonstrates that the kind, concentration, and interaction of cognate materials with the polymer chains regulate the electrical and optical characteristics of the selected polymer [4]. The study of understanding conductive polymers has seen a major change with the development of polymer catalysis techniques that have converted polymers from insulators to conductors [5]. Through their impact on the energy levels between the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO), dopants are used to alter the oxidation state of polymers in order to promote conductivity. Doping alters the energy gap value by introducing an intermediate energy level between HOMO and LUMO and reducing the energy gap, according to an earlier study. Increased conductivity is the result of this decrease, which makes it easier for the charge carrier to travel throughout the polymer. As the energy difference narrows, the electrons move from HOMO to LUMO, further improving conductivity [6]. One technique to modify the physical characteristics of polymers is to add filler molecules or semiconducting nanoparticles. The resultant nanocomposites' thermal, mechanical, optical, magnetic, and conductive characteristics are greatly influenced by the filler's intrinsic and surface qualities [7]. Superior thermo-mechanical, rheological, and thermally stable polymeric nanocomposites are of particular relevance for energy and environmental applications [8]. This method uses the unique electrical and optical characteristics of inorganic nanoparticles to generate novel composites based on the synergy between polymer and inorganic fillers. Metal oxide nanoparticles, such ZnO, TiO2, and SnO2, have garnered significant attention in recent times due to their potential in biotechnological applications. Research has indicated that the integration of conductive polymers, such as PANI, with inorganic nanoparticles presents a viable approach towards the development of many applications, such as drug delivery, biosensors, gas sensors, conductive paints, and rechargeable batteries [9]. For instance, studies have demonstrated that the creation of multifunctional nanostructures can occur when PANI is combined with inorganic nanoparticles [10]. In the fields of technology and materials science, conducting polymers—which have special electrical characteristics due to their multi-conjugated structures—are very desirable. For a variety of technological applications, research highlights the need for producing conductive polymer compounds and activated polymers [11].

Doping polymers with fillers and nanoparticles presents a promising avenue for enhancing their performance in future technologies. These improved materials are pivotal for advancements requiring specific qualities such as environmental stability, suitable mechanical properties, cost-effectiveness, and low weight. The research in nanomaterials, particularly the development of smaller materials with unique properties, holds significant future potential [12]. Nanocomposite materials, with applications ranging from gas-separation membranes to contact lenses, catalysts, and bioactive implants, offer diverse prospects [13].

To disperse inorganic Nano fillers in polymer matrices, direct melt-compounding is a common technique [14, 15]. However, a significant challenge lies in the tendency of Nano fillers to aggregate due to their high surface activity, forming micron-sized clusters [14]. Various strategies, including sol-gel/polymerization approaches and surface modification of Nano fillers, have been explored to address this issue [16]. While effective, these methods often involve complex chemical processes, limiting their scalability for large-scale production with diverse Nano filler loadings and combinations [16]. The authors propose a novel method for dispersing inorganic nanoparticles in various polymers through direct melt-compounding, eliminating the need for complex reactions or nanofiller surface modification and paving the way for industrial-scale production of high-performance particle/polymer nanocomposites [17].

Doping methods

Similar to Nano oxides, doping polymers involve introducing foreign substances or modifying their structure to enhance their properties for specific applications. Here are some common doping methods for polymers:

Chemical Doping:

Oxidative Doping: This technique involves exposing the polymer to an oxidizing agent, like iodine or bromine, leading to the removal of electrons from the polymer chain and the creation of positively charged holes. This enhances polymer conductivity, facilitating the movement of charge carriers through the material [18]. Thin-layer films are formed using solutions or suspensions applied through common processes like lacquering or spraying, influencing the rheological characteristics of the coating formulation [19]. Drying procedures, including ambient drying, conventional hot air drying, infrared drying, and microwave energy drying, impact the film or coating qualities [20]. Despite significantly lower coating weights compared to extruded coatings, the intended barrier qualities are maintained [21].

The addition of nanoparticles to coating dispersions, particularly in nanocoatings, offers advantages in terms of material efficiency over multilayer films, aligning with ecological and financial considerations [22]. Moreover, surface coatings can be utilized to modify repellent qualities, such as water-repellent features in paper-based packaging or designs for easy emptying [23]. Popular nanomaterials for paper coatings include starch, ceramics, inorganic pigments, and nanoclays [21].

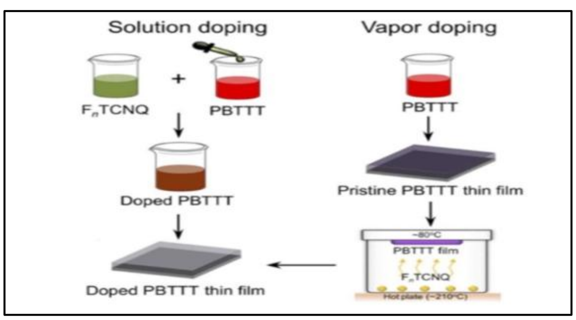


Figure 1: Method of Oxidative Doping

Reductive Doping: This method involves exposing the polymer to a reducing agent, such as an alkali metal, inducing the addition of electrons to the polymer chain and the creation of negatively charged ions. This process can enhance the polymer's conductivity and alter its optical properties [24]. Extrusion, a significant method in polymer processing, involves rapidly melting a polymer with high energy input. The material becomes formable and is forced through the extruder die due to the heat and energy generated by friction between the screws [25]. Throughout the process, the mass undergoes compression, mixing, plasticization, homogenization, chemical transformation, degasification, or gasification [26]. Introducing nanoparticles to polymeric materials can result in various nanocomposites. The dispersion quality of sought-after exfoliated nanocomposites is influenced by the screw and extruder arrangement, with high shear rates favoring exfoliation and longer residence times promoting greater dispersion [27]. The placement of nanoclays also plays a significant role, and successful dispersion or exfoliation depends on the thermodynamic affinity between the nanoclay/nanoparticle and the polymer matrix [28]. Intercalation occurs with weak attractive connections, while exfoliation occurs with substantial interactions [29]. Extrusion/melt processing is an effective method for achieving exfoliation, as depicted in Figure 3 [28].

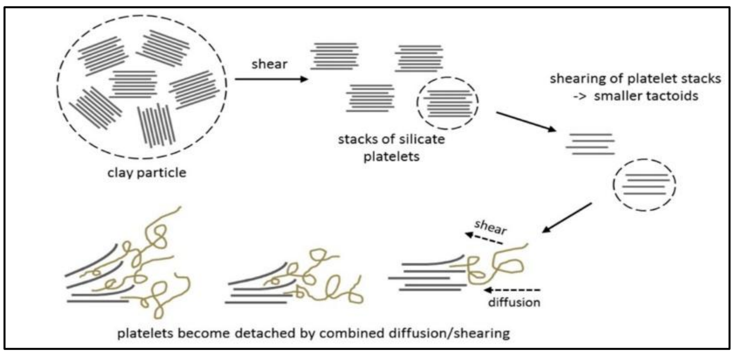


Figure 2: Mechanism of exfoliation and dispersion of clay during melt Adapted from [28].

Physical Doping:

Nanodeposits: This method involves physically blending the polymer with another substance, such as a conductive filler or a dye [30]. The inclusion of fillers enhances the polymer's conductivity, while dyes can modify its color or other optical properties [31]. Nanocomposites and nanostructured materials are generated through conventional or innovative techniques, including vapor deposition, chemical reduction, pulsed laser deposition, mechanical milling, magnetron sputtering, layer-by-layer coating, dip coating, sol-gel coating, TUFT (tubes by fiber templating) process, electrochemical deposition, sol-gel techniques, or electro-hydrodynamic processing (comprising electrospraying and electrospinning) [32].

Nanocoatings, formed at the nanoscale, are instrumental in surface functionalization, imparting specific qualities such as antibacterial, self-healing, flame retardant, gas barrier, etc. [33]. Extensive research in various fields, including biomedicine, packaging, sensors and electronics, textiles, solar panels, lithium-ion batteries, building materials, and the automobile industry, has explored the applications of nanocoatings [34].

Radiation Doping: This method involves subjecting the polymer to high-energy radiation, such as gamma rays or ultraviolet light, representing a promising approach for semiconductor doping. This process induces flaws in the polymer chain, resulting in modifications to its mechanical or electrical characteristics [35]. Nuclear transmutation doping (NTD), particularly through mechanisms involving thermal neutrons ("neutron" doping) and γ-rays (photonuclear doping), has gained popularity for uniformly doping silicon ingots across a sample's volume [36]. While prior attention primarily focused on these mechanisms, there is a growing interest in using charged particle nuclear processes for NTD due to the availability of high-intensity charged particle beams. This heightened interest is attributed to charged particles' effectiveness in influencing semiconductor crystal characteristics in localized areas [37].

Electrochemical Doping:

Electro spraying: Electrospinning and electrospraying, integral components of electro-hydrodynamic processing techniques, involve applying a high-voltage electric field to a polymer solution or melt. Electrospinning produces continuous polymer fibers in the submicrometer range, influenced by factors like viscosity, surface tension, and conductivity of the polymer solution, as well as process parameters. Electrospraying, a cost-effective method, breaks liquids into small particles through electro-hydrodynamic force, creating droplets under electric repulsion. Electro-hydrodynamic processing (EHDP) offers advantages over traditional deposition methods, with electrospray deposition (ESD) demonstrating high efficiency (up to 80%) and reduced process steps [38]. In recent years, both electrospinning and electrospraying have been successfully scaled up to an industrial level using multinozzle injectors by companies like Bioinicia S.L., despite previous attempts to industrialize the electrospinning process being limited. An experiment involving the casting method and co-doping of different concentrations of titania nanoparticles with a laser dye blend (DCM) in polystyrene demonstrated notable effects on the optical properties. The addition decreased the magnitudes of direct electronic transitions, impacting energy gaps. This modification resulted in decreased values for refractive index, dispersion energy, and single-oscillator energy while increasing absorption and extinction coefficients in all produced films. The presence of titania nanoparticles also led to an increase in crosslinking, as evidenced by Urbach's tail, showcasing the success of the technique in terms of cost, results, and manufacturing speed [39].

Doping polymers applications by metal oxide nanoparticles

Conductive polymers (CPs) and metal oxide polymeric composites (MOPCs) generate nanostructures that are essential for the implementation of nanodevices in the areas of energy, electronics, healthcare, and the environment in order to build intelligent nanopatterns. CP and MOPC nanostructures are widely used in biosensing, chemical sensing, controlled drug delivery, wound healing, filtration membranes, antireflective surfaces, electrodes, diodes, and field-effect transistors due to their diverse structural, electrical, optical, plasmonic, and mechanical properties. These materials are essential to nanotechnology because they can be extruded into fibers, foams, gels, and thin films. Conductive polymers and metal oxide composites are used in the electrical, environmental, energy, and healthcare sectors because to their exceptional mechanical flexibility, chemical inertness, optical behavior, and biocompatibility. Studies reveal that their attributes and performance are significantly influenced by structural sizes, flexible morphologies, and doping tunability [40].

Furthermore, materials and structures designed at the nanoscale exhibit elevated volume ratios, surface areas, and enhanced performance metrics [41]. These nanostructures, with atomic sizes ranging from 1 to 100 nm, exhibit intricate forms, spatial organization, high surface area, and density. Enhancements in these fields are being driven by novel structures and devices that use particular kinds of conducting polymers and metal oxides [42].

Electronics and Energy:

Conductive polymers: Due to their notable strengths, stability, moderate oxidation, ease of electrochemical polymerization, and high heat resistance, polyphenylenes (PP) are advantageous, particularly in applications requiring electroluminescence [43]. Poly(3,4-ethylenedioxythiophene) (PEDOT), a widely used commercial conductive polymer, offers stability, low redox potential, transparency, and high electrical conductivity. PEDOT finds applications in electrochromics, antistatic coatings, photovoltaics, biosensors, batteries, and nanoelectronics [44]. To address PEDOT's insolubility issue, researchers have successfully doped it with either tetramethacrylate or poly(styrenesulfonate) (PSS), resulting in PEDOT:TMA or PEDOT:PSS, respectively [45]. PSS dramatically enhances the conductivity of PEDOT, resulting in PEDOT:When exposed to or post-treated with solvents such as dimethyl sulfoxide or ionic liquids, PSS is effective for transparent electrodes, flexible electronics, electrochromic materials, polymeric capacitors, thermoelectrics, and UV-stabilized photovoltaics [43]. Conversely, TMA transforms PEDOT into a p-type conducting material, offering conductivity ranging from 0.1 to 5 S cm1. TMA is noncorrosive, readily cross-linked, and soluble in organic solvents, making it advantageous for dye-sensitized solar cells (DSSCs), carbon nanotubes, glucose sensors, patterned organic light-emitting diodes (OLEDs), and $TiO2$ nanocomposites [46].

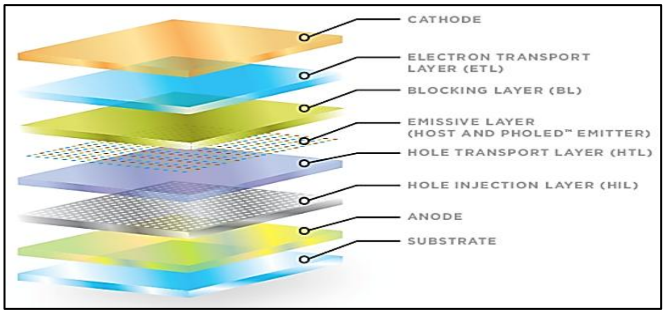


Figure 3: Organic lightemitting diodes (OLEDs)

Electrochromic devices: Smart windows, employing polymers doped with metal oxides like titanium dioxide (TiO_2), undergo color changes with applied voltage, providing energy-efficient control over natural light and heat in buildings [47]. Conducting polymers (CPs) consist of both single- and double-bonded polymeric chains [48]. Due to their p-electron backbones, conjugated polymers enable copolymerization and various levels of chemical or electrochemical doping. These materials' range of properties, from insulating to conducting, light weight, and cost-effectiveness, allow them to find diverse commercial applications in gas, optical, and biosensors etc. [49]. Dopant concentrations significantly impact the electrical conductivity of CPs, modifying their electrochemical and structural characteristics. CPs exhibit cyclic redox, electrical stimulus release, and/or ion-exchange properties, making them useful for drug release and sensing with specific polymerizations and dopants [50]. Concerns in doping CPs include the potential impact of larger molecule dopants on structural characteristics and the leaching or diffusion of tiny molecular dopants affecting other device layer properties [51]. Nanodevice systems often employ conducting polymers, with notable examples being copolymers of polyaniline (PANI), polypyrrole (PPy), polyphenylene (PP), and poly(3,4-ethylenedioxythiophene) (PEDOT). Additional constituents include poly(pphenylene) (PPP), poly-p-phenylene-sulphide (PPS), poly(p-phenylene-vinylene) (PPV), poly(3-hexylthiophene) (P3HT), poly(p-phenylene-erephthalamide) (PPTA), polythiophene (PT), polythiophene-vinylene (PT-V), poly(pphenylene) (PPP), and the ladder-like polymer poly(benzimidazobenzophenanthroline) (BBL). Polyaniline (PANI) stands out due to its high stability, low cost, strong electrical conductivity, light weight, and oxidative polymerizing nature. PANI undergoes a transition from insulator to conductor when exposed to protonic acid, making it promising for various applications such as batteries, supercapacitors, sensors, and protective coatings [52]. PANI nanocomposites with metal oxides like MnO_2 or TiO_2 exhibit enhanced characteristics compared to standalone PANI [53].

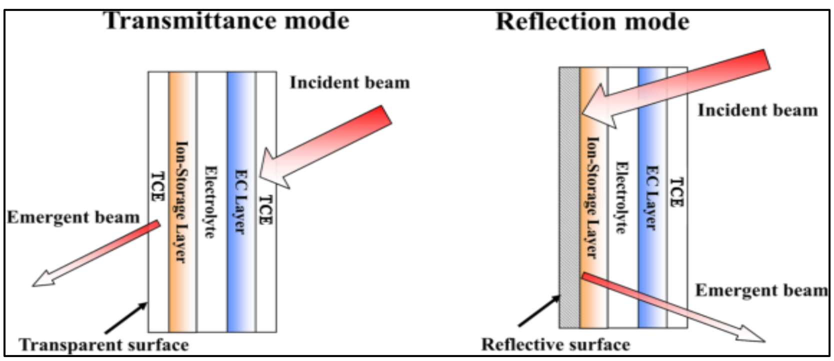


Figure 4: Modes of Electrochromic Device (ECD) Operation

Sensors and Catalysis

Gas sensors:

Rapid industrialization and modernization have led to a surge in environmental contamination, necessitating continuous monitoring and pollution prevention efforts [54]. To safeguard both the environment and human health, the identification of hazardous gases like CO, NO_2, NH_3, SO_2, CO_2, and hydrocarbons is crucial. Various sensor types, including surface acoustic wave sensors, immunosensors, chemiresistive sensors, electrochemical sensors, and biosensors, are employed for different applications. The chemiresistive sensor, valued for its affordability, ease of use, and rapid response, stands out as an effective transduction unit, commonly used in commercially available gas sensors [55]. Chemiresistive sensors translate chemical information through changes in the electrical resistance of a two-point contact, requiring minimal supporting electronics for assessment. Nano-treating conducting polymers, such as polymers doped with metal oxides like zinc oxide (ZnO), can significantly alter their operating mode. These polymers exhibit varying electrical conductivity when exposed to specific gases, enabling the detection of gases with remarkable sensitivity and selectivity [56]. By optimizing the gas sensor's performance through both adsorption and composition-structure-dependent charge transport, the resulting sensors enhance the detection of target analyses [57]. Typically, chemiresistive sensors involve depositing an active layer over an array of electrodes to detect changes in electrical resistance in the presence of target gases [55].

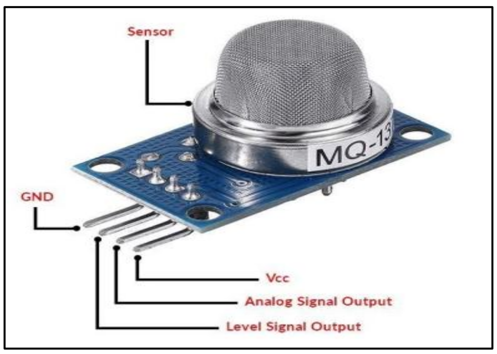


Figure 5: Gas sensors

Free-standing porous membranes play a crucial role in various applications, including air filtration, water treatment, biomedical engineering, and catalysis [58]. These membranes are typically constructed from polymers for mechanical stability and natural flexibility [59]. However, ceramic membranes offer a broader range of options and can operate at high temperatures exceeding 100–200 °C or in extreme conditions [59], making them suitable for catalytic applications exposed to severe environmental conditions [59]. The preparation of ceramic porous membranes involves colloidal processing, where ceramic powder is dispersed in a solvent with a suitable dispersant, and various additives are added to enhance processability and introduce porosity [60]. Integrating metal oxides like TiO_2 into polymer membranes enhances their catalytic efficiency for various chemical reactions, such as water splitting for hydrogen production or pollutant degradation [61]. Catalysts have historically played a vital role in meeting various requirements, such as minimizing chemical use and addressing environmental issues through improved reaction selectivity to avoid waste byproducts [62]. In many industrial operations, including petrochemical and refining processes, energy generation, and specialty chemical synthesis, catalysts, primarily consisting of metal oxides and precious metals in powder form, have proven indispensable [63].

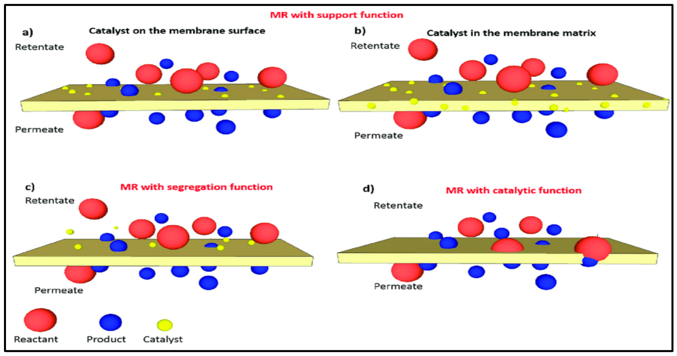


Figure 6: Catalytic membranes[64]

Biomedical and Environmental:

Rising animal ailments prompt intensified efforts to develop new medications, yet their effectiveness hinges on precise administration—correct dosage, targeted delivery, and minimal interaction with unintended targets to avoid severe side effects [64]. The burgeoning field of biomaterials significantly influences patient treatment [65]. Despite decades of progress in medications, antibodies, enzymes, and vaccinations, the need for drug delivery systems (DDSs) persists. Conventional methods face challenges like solubility issues, rapid drug breakdown, potential tissue injury, unfavorable pharmacokinetics, low biodistribution, and insufficient selectivity. DDSs, employing amphiphilic components, enhance solubility, act as drug carriers, and improve protection against degradation, minimizing unintentional extravasation. Enhanced permeability, retention effects, or ligand-mediated targeting in DDSs contribute to reducing side effects, delaying renal clearance, and increasing drug concentrations in targeted tissues [64]. Early drug delivery systems (DDS) employed solid polymers for extended drug release in agriculture before transitioning to biomedicine [66]. The field has since grown, with lipid-based cancer treatments like Ara-C liposomal, Doxil, and DaunoXome available. DDS applications expanded to diverse materials—silicon, iron oxide, gold, and organic compounds [67]. Polymers, with their versatile processing into micelles, dendrimers, membranes, fibers, foams, nanoparticles (NPs), and hydrogels, stand out. This adaptability facilitates treating various medical conditions. For example, Lupron Depot, employing poly(lactic-coglycolic) acid (PLGA) microspheres, treats advanced prostate cancer. While biodegradable polymers like poly(lactic acid) (PLA) and PLGA allow sustained release, stimulus-sensitive or smart biomaterials, using metal oxide-doped polymers, offer controlled drug release under specific conditions like temperature or pH, enhancing drug effectiveness and minimizing side effects [64].

In this overview, we spotlight the advancements in intrinsically conducting polymers (ICPs), recognized as semiconducting or conductive polymers, among stimuli-triggered drug delivery systems (DDSs). ICPs, organic materials with metallic electrical and optical properties, exhibit exceptional features such as processing flexibility and ease of synthesis. Functioning as conductive and electrochemically active biomaterials in DDSs, ICPs respond to electrical and/or electrochemical stimuli due to their conjugated π-system, allowing electron transit. The distinct electrical structure of ICPs, characterized by alternating single (σ) and double bonds (π), dictates their conductivity. The doping process induces radical cations/anions (polarons) or dications/dianions (bipolarons), with reversible de-doping demonstrated in a biocompatible ICP, poly(3,4-ethylenedioxythiophene). P-doping withdraws electrons, creating positively charged holes, while n-doping injects electrons, generating negative charges. Oppositely charged ions balance the electrical charge. As the doping level increases, ICPs exhibit enhanced electrical conductivity. Approximately twenty-five known ICPs, produced through electrochemical or oxidative chemical polymerization, include commonly used materials like polypyrrole (PPy), PEDOT, and polyaniline (PAni) for their stability, biocompatibility, and excellent electrical and electrochemical characteristics [68].

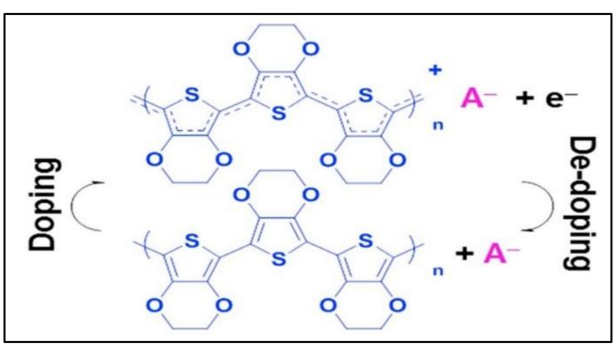
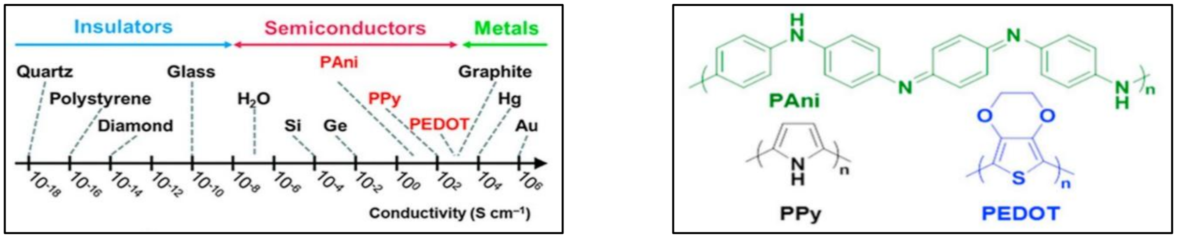


Figure 7: Reversible redox activity of Poly(3,4-ethylenedioxythiophene) (PEDOT) via oxidation and reduction reactions known as doping and de-doping.[64]



(a) (b)

Figure 8 (a) Scale of material's conductivity. Adapted with permission from [64]. (b) Chemical structure of the most employed ICPs (PAni, PPy and PEDOT).

To overcome disadvantages such as stiffness, brittleness, insolubility, low biodegradability, and poor processability in intrinsically conducting polymers (ICPs), blending them with more flexible and/or biodegradable polymers to create electroactive blends and copolymers is a common strategy. The biocompatibility of ICPs has been extensively studied using various cell lines, including fibroblasts, endothelium, bone, keratinocytes, myoblasts, neural, glial, and mesenchymal stem cells. The biomedical industry witnessed a surge in the use of ICPs following the confirmation of their biocompatibility with biological systems. However, ensuring the non-toxicity or very low toxicity of ICPs requires the removal of unreacted monomers, excess dopant ions, or residual solvents. Additionally, materials with nanofeatures may alter toxicity values, potentially leading to adverse biological effects due to increased surface area. Continuous evaluation of the toxicity of ICP-based materials is crucial to guarantee the safety of devices utilized for biomedical purposes [69].

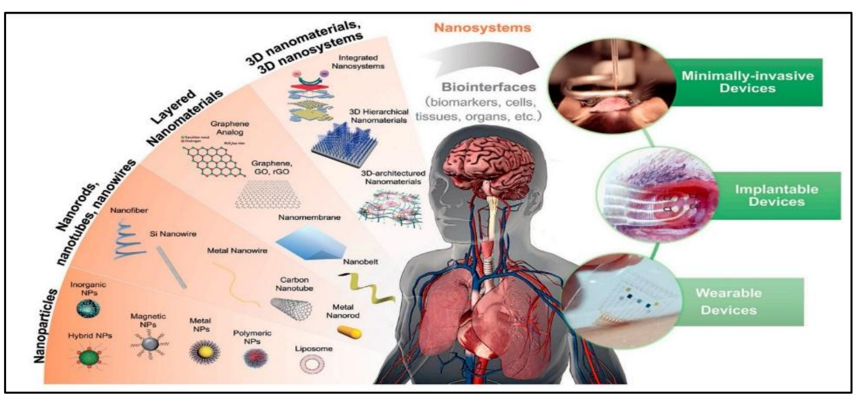


Figure 9: Scheme of different nanostructures. Adapted with permission from [70].

Antibiotics, considered the "golden standard" for treating bacterial illnesses since their discovery, face challenges due to the emergence of antibiotic resistance, leading to a resurgence in infectious disorders. To combat this, the exploration of novel antimicrobial drugs has gained importance. Utilizing metal and metal oxide nanoparticles (NPs) is one approach to address antibiotic resistance in bacteria, with research highlighting the antifungal effects of metal oxide NPs. The ongoing COVID-19 epidemic has intensified efforts to search for affordable and potent antiviral medications [71, 72].

Silver nanoparticles (Ag2O NPs) and their components exhibit potent bactericidal, fungicidal, and antiviral properties, with diverse applications in nanomaterials. Researchers emphasize their use in catalyzers, chemical sensors, optoelectronic devices, and targeted drug administration. Ag2O NPs demonstrate significant antibacterial potential, particularly in biocompatible materials for bone implants. Biomedical applications include wound treatment, cancer therapy, oxidative stress shielding, ulcer therapy, and photocatalytic destruction of pharmaceutical micro-pollutants. These NPs are also effective in coating medical equipment and wound dressings to prevent infections. Surface-coated antimicrobial food packaging sheets, comprising plastic/biopolymer films and synthetic/naturally occurring antimicrobials, are explored, emphasizing distinct coating processes for depositing antimicrobials onto the film substrate [73, 74].

Conclusions

Polymer nanocomposites, comprising a polymeric main phase and nanoscale dispersion phases, exhibit substantial property enhancements even at modest nanoparticle loading. Nanocomposites, defined by at least one exterior dimension between 1 and 100 nm, can undergo standard wet and dry processing. The physical characteristics of polymers can be altered by incorporating filler or semiconductor nanoparticles, impacting thermal, mechanical, optical, magnetic, or conducting properties. Combining organic polymers with inorganic nanofillers creates novel materials with synergistic or complementary behaviors. Conducting polymers, beneficial in electrochemical processes, offer improved conductivity and surface area. Doping polymers with nanoparticles enhances their chemical and physical properties. Future applications include contact lenses, gas-separation membranes, catalysts, and bioactive implant materials. However, challenges exist in achieving uniform dispersion due to nanofillers' high surface activity, leading to cluster formation. Various techniques, such as sol-gel processes and organic modification, aim to address this issue.

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