

## A REVIEW ON CONDUCTING POLYMER NANOMATERIAL'S AS SENSORS

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### ABSTRACT

Conducting polymers represent an important class of functional organic materials for next-generation electronic and optical devices. Advances in nanotechnology allow for the fabrication of various conducting polymer nanomaterials through synthesis methods such as solid-phase template synthesis, molecular template synthesis, and template-free synthesis. Nanostructured conducting polymers featuring high surface area, small dimensions, and unique physical properties have been widely used to build various sensor devices. Many remarkable examples have been reported over the past decade.

**KEYWORDS:** fabrication, molecular template, template-free synthesis.

### INTRODUCTION

Conducting polymers have found a wide range of applications in the various fields of electronics, optics, energy devices, medicine, actuators, and composites as a viable alternative to metallic or inorganic semiconductor counterparts.<sup>[1,2,3]</sup> In particular, there has recently been huge demand for developing flexible or wearable electronics, displays, and other devices, in which conducting polymers can ultimately be used as true flexible organic conductors or semiconductors. The most notable property of conducting polymers is their inherent electrical conductivity, which is closely connected to the charge transfer rate and electrochemical redox efficiency. Most conducting polymers act as semiconductors in terms of conductivity although several studies on metallic conducting polymers have been reported. Unlike their inorganic counterparts, a weak intermolecular overlap of electronic orbitals combined with a greater degree of disorder in conducting polymers result in narrow electronic bands and a low mobility of charge carriers. Consequently, conducting polymers have had serious limitations

in specific applications such as transistors and memories. For example, the performance of field-effect transistors (FETs) based on conducting polymers cannot rival that of FETs based on single-crystalline inorganic semiconductors, such as Si and Ge, which have charge carrier mobilities that are about three orders of magnitude higher.<sup>[4,5,6]</sup> The mobilities of FETs based on solution-processed conducting polymers [e.g., poly(3-hexylthiophene)] are generally found to be in the range of  $0.1 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , similar to those of amorphous silicon FETs. A mobility of  $10.5 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  has been the best reported thus far.<sup>[7]</sup> Conducting polymer FETs are not practically suitable for use in applications requiring high switching speeds.

Another example of conducting polymer applications involves electrochromic devices for smart windows and flexible displays. Conducting polymers are one of the most attractive electrochromic materials because of advantages such as high coloration efficiency, rapid switching ability, and diverse colors.<sup>[8,9]</sup> The switching time is one of the important parameters in display technology. The electrochromism of conducting polymers is based on reversible redox reactions accompanying ion exchange, and is completely different from the operating mechanism of FETs.

### **Polymers as sensors**

Conducting polymers have also been used for sensor applications as a signal transducer. There are several important parameters in sensor technology, such as sensitivity, selectivity, and response time.<sup>[10,11]</sup> In most cases, a response time on the order of seconds is enough for human recognition. Thus, it is easier to meet the requirements for response time than for other parameters. The sensing mechanisms of conducting polymers can involve redox reactions, ion adsorption and desorption, volume and weight changes, chain conformational changes, or charge transfer and screening. Compared to the inorganic counterparts, conducting polymers have an advantage in achieving high sensitivity and selectivity by virtue of their chemical and structural diversity. Conducting polymers also share the strengths of polymers over other materials, including low-temperature synthesis and processing, large-area manufacture, flexibility, and cost effectiveness. As a result, conducting polymers can be competitive in sensor applications and sensors are therefore considered to be one of the most practical applications of conducting polymers.

There are several conducting polymers that have been extensively investigated for practical applications. All of them possess high conductivity and good environmental stability, and their polymerization reactions are not only straightforward but also proceed with high yield.

Polypyrrole (PPy) features low oxidation potential and good biocompatibility, and the advantages of polyaniline (PANI) include that the monomer is very inexpensive. Polythiophene (PTh) has many useful derivatives, one of which is PEDOT developed by Bayer AG (Leverkusen, Germany). PEDOT features optical transparency and can be soluble in water with polystyrene sulfonate (PSS). Owing to these desirable characteristics, PPy, PANI, PTh and their derivatives have become leading materials in various applications fields. This review addresses research efforts to fabricate and manipulate nanostructures mainly consisting of the representative conducting polymers for sensor applications, and highlights remarkable recent examples with a focus on materials functionalization, transduction mechanisms, and device characteristics in sensor applications.<sup>[12,13]</sup>

### **Fabrication of Conducting Polymer Nanomaterials**

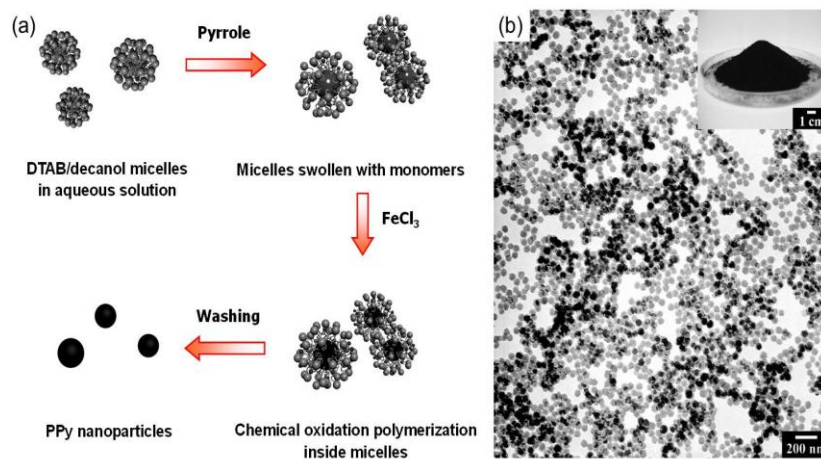
Precise control over the size and morphology of conducting polymers at the nanoscale is essential to improving the performance of related sensors. As mentioned, polymers are highly unstable at the nanometer scale, which is one of the greatest obstacles in building polymer nanoarchitectures. Nevertheless, numerous efforts have been made to fabricate polymer nanomaterials with well-defined size and morphology, and various types of conducting polymer nanostructures have been fabricated in a controlled fashion.<sup>[14]</sup>

Conducting polymers have traditionally been synthesized via chemical or electrochemical oxidation polymerization. The overall process includes the oxidation of monomers, followed by the coupling of the charged monomers to produce the polymer chains. Chemical polymerization is advantageous for large-scale production at low cost, while electrochemical polymerization offers the possibility of in-situ formation, such as on an electrode for a sensor device. Conducting polymers can be obtained in the presence of various oxidizing agents. Oxidation polymerizations with acid or peroxide initiators result in insulating materials that require a post-doping process. Metal salts that can act as both oxidizing and doping agents are used to conduct the oxidation polymerization, which directly yields polymers in a conductive state. For example, ferric salts including FeCl<sub>3</sub> and Fe(ClO<sub>4</sub>)<sub>3</sub> are widely employed. Owing to their electrical conductivity, conducting polymers can grow electrochemically without oxidizing agents on an electrode. It is possible to tailor the polymer thickness by controlling the applied potential, polymerization time, and electrolyte.

Conducting polymer nanostructures have been fabricated with the aid of templates during the polymerization process. The synthetic routes are traditionally classified into three classes

depending on the kind of template: hard template synthesis, soft template synthesis, and template-free synthesis.<sup>[15]</sup> However, it is sometimes ambiguous to distinguish the hard and soft templates. For example, track-etched polymer membranes and polymer nanofibers are both soft, but they are categorized as a hard template and a soft template, respectively. Therefore, the templates are classified here as solid-phase and molecular templates as a new standard. Molecular template synthesis has strengths compared to solid-phase template synthesis. The most widely used molecular templates include surfactants, liquid crystals, and polyelectrolytes. Because molecular template synthesis is comparatively straightforward and cost-effective, it is suitable for large-scale production. However, the molecular templates are not robust, static entities, leading to considerable difficulty in obtaining the desired nanostructures. Surfactant templating is a typical example of molecular template synthesis. Surfactants have the ability to self-assemble into ordered molecular structures called micelles. The micelles exist in various forms, and their dimensions generally range from a few to a few tens of nanometers, making them very suitable as templates. Microemulsions consisting of thermodynamically stable micelles with size less than 50 nm have been used in polymerization techniques to prepare polymer nanoparticles. However, micelles are highly sensitive to the surrounding environment, which makes it very difficult to achieve stable microemulsion systems for polymerization, preventing their widespread utilization for industrial products. One of the strategies for making stable microemulsion systems is to use co-surfactants like long-chain alcohols. A notable example is described in Figure 2a.<sup>[16]</sup> A cationic surfactant, dodecyltrimethylammonium bromide (DTAB) was used to obtain spherical micelles reinforced with decanol in aqueous solution. The decanol can play a role in retarding the diffusion of monomers through the aqueous phase. Consequently, monodisperse PPy nanoparticles (60 nm in diameter) were obtained on a gram scale, which is a very large quantity in laboratory-scale synthesis. Figure 2b shows a TEM image of the PPy nanoparticles and the inset shows a photo of a Petri dish containing 12 g of the nanoparticles.

**a)** Schematic illustration of the preparation of PPy nanoparticles in a cationic surfactant (DTAB)/co-surfactant (decanol) emulsion system; **(b)** TEM image of monodisperse PPy nanoparticles prepared through micelle templating (inset: photograph showing a Petri dish containing 12 g of PPy nanoparticles obtained in a single polymerization reaction). With permission from.<sup>[13]</sup>; Copyright 2005, Wiley-VCH Verlag GmbH & Co. KGaA.



This example indicates potential for use of the microemulsion technique for the efficient mass production of nanoparticles. However, the technique requires high surfactant concentration, which is problematic in terms of cost and environmental pollution. Another study was reported for producing.

PPy nanoparticles using a kind of dispersion polymerization, where water-soluble polymers provide steric stability. It is known that dispersion polymerization produces polymer microbeads. Jang *et al.* were able to produce PPy nanoparticles just by using a polymeric stabilizer, polyvinyl alcohol (PVA), in aqueous solution.<sup>[17]</sup> Figure 3a,b represent the synthesis process in which ferric ions were anchored on PVA chains via coordination bonding. The polymerization of pyrrole monomers proceeds only with ferric ions on PVA chains, which allows enough steric stability to yield nanometer-sized particles, exhibits the SEM images of the resulting PPy nanoparticles, in which the size of the nanoparticles was surprisingly as small as *ca.* 25 nm in diameter. Considering that previous studies were limited to the synthesis of micrometer-sized particles with steric stabilizers, this approach is very innovative.

### Sensor Applications

A variety of sensors have been formulated using conducting polymers in different transduction modes. The transduction modes can be divided into five main classes based upon the operating principle into *conductometric*, *potentiometric*, *amperometric*, *colorimetric*, and *gravimetric* modes.<sup>[18,19]</sup> The conductometric mode uses changes in electrical conductivity in response to an analyte interaction. The conductivity of a conducting polymer material bridging the gap between two adjacent electrodes is commonly measured as

a function of analyte concentration, and it can be also monitored with a fixed potential in solution.

Potentiometric sensing mode is based on analyte-induced changes in the chemical potential of a system when no current is flowing. The change in the open-circuit potential of the system is monitored, which is mostly proportional to the logarithm of the concentration of analyte. The chemical and diffusion processes have to be at equilibrium conditions in the potentiometric mode for a thermodynamically accurate signal to yield. Amperometric mode refers to either single-potential amperometry or variable-potential amperometry. The principle of amperometric sensing is to measure the current generated by the redox reaction of an analyte at a sensing (working) electrode, where the current is subject to Faraday's law and a dynamic reaction achieving steady-state conditions in the system. Voltammetric mode is also a variant of amperometric mode. It monitors the change in current while varying the applied potential. Colorimetric sensors quantitate changes in optical absorption characteristics, which depend on the local electronic structure. The sensitivity of the bandgap of conducting polymers to analyte-induced changes provides a useful means to create this kind of sensor.

Lastly, gravimetric mode takes advantage of a weight change in a conducting polymer as a result of analyte-polymer interaction. Minute weight changes in the polymer can be normally monitored using a quartz crystal microbalance. Numerous conducting polymer sensors based on these transduction mechanisms have been devised in order to detect various chemical and biological species.

### **Chemical Sensors**

There are many chemical species of concern that must be detected, including toxic gases, volatile organic compounds, alcohol, and humidity. Various conducting polymer nanostructures have been used to detect them. Chemically synthesized polymer nanoparticles suspended in solvents can be simply deposited on a prefabricated electrode by drop casting to construct a sensor substrate. Kwon *et al.* investigated the gas sensing properties of PPy nanoparticles with diameters of 20, 60 and 100 nm. They found that decreasing the nanoparticle size produced a noticeable increase in the sensitivity toward ammonia gas. It was anticipated that the increased sensitivity can be attributed to the higher surface-to-volume ratio of the 20-nm nanoparticles compared to the 60 and 100-nm nanoparticles.<sup>[20]</sup> The use of conducting polymer nanoparticles obtained via micelle templating and electrospinning in sensor applications has also been reported. Highly sensitive chemiresistive sensors based on



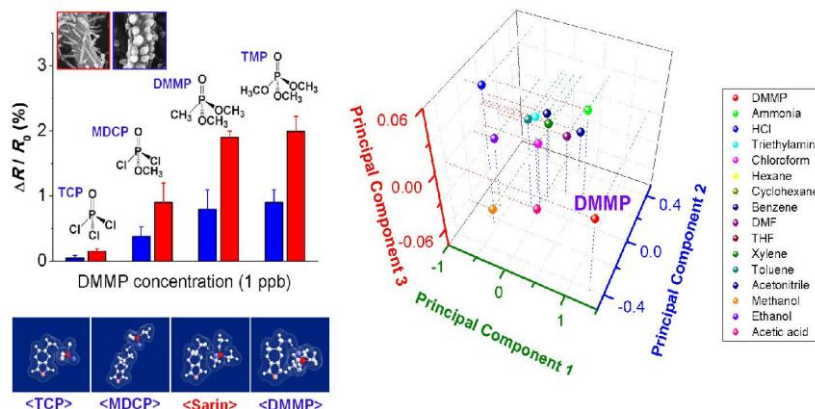
PPy nanotubes were devised to discriminate volatile organic compounds and toxic gases.<sup>[21]</sup> PPy nanotubes were transferred onto a polydimethylsiloxane substrate using a dry-transfer method and then micro-patterned gold electrodes were deposited onto the nanotubes by thermal evaporation. Other conducting polymer nanostructures such as PPy nanoparticles, PEDOT nanorods and PEDOT nanotubes were also integrated into electrode substrates to induce discriminative responses toward individual analytes. The lowest detectable concentration of the sensors was *ca.* 0.01 ppm for ammonia, selectively.

Electrochemical polymerization allows controllable polymer deposition on an electrode. Recently, PPy nanowires grown electrochemically on a microelectrode substrate were used as conductometric transducer to detect hydrogen gas at room temperature.<sup>[22]</sup> The diameters of the nanowires were in the range of 40–90 nm and their lengths were on the order of several tens of micrometers. It was found that the PPy nanowires were stacked at high density and a number of the nanowires bridged the gap between the electrodes, The sensor was exposed to different concentrations of hydrogen gas at room temperature in a closed chamber, during which the resistance of the PPy nanowire electrode was monitored. Upon exposure to hydrogen gas, a drop in resistance was observed. The resistance of the PPy nanowires is expected to decrease when exposed to hydrogen, which is a reducing gas. The sensors had a linear detection range of *ca.* 600–2500 ppm for hydrogen gas, and their sensitivities were found to be dependent of the amount of the deposited nanowires. On the other hand, the sensors showed a loss in sensitivity due to carbon monoxide gas interference, which has remained a critical issue to be solved. The detection of hydrogen at low concentrations is very important, because it is explosive in the presence of oxygen. Interestingly, it has been found that PANI has the ability to interact with hydrogen. Kaner and Weiller demonstrated that hydrogen interacts directly with doped PANI nanofibers to induce a small change in the conductivity of the nanofibers.<sup>[23]</sup> The direct mass uptake of hydrogen by PANI nanofibers was also observed using a quartz crystal microbalance (*ca.* 3% relative to the nanofiber mass). A plausible mechanism of the hydrogen/PANI interaction involves hydrogen interacting with doped PANI at the charged amine nitrogen sites, followed by the dissociation of hydrogen with the formation of new N–H bonds at the amine nitrogen of the PANI chain.<sup>[53,54]</sup> Subsequently, charge transfer between adjacent amine nitrogens returns the PANI back to its original doped, emeraldine-salt form with a release of hydrogen.

Conducting polymers can be complexed with many species to create composite materials that are sensitive to certain chemical agents. PANI nanofibers treated with  $\text{CuCl}_2$  showed the ability to detect hydrogen sulfide with change in resistance by 4 orders of magnitude.<sup>[24]</sup> The  $\text{CuCl}_2$  in the PANI nanofibers reacts with hydrogen sulfide to yield HCl, finally resulting in the further doping of the nanofibers. This reaction scheme has been extended to the detection of other specific analytes such as phosgene<sup>[25]</sup> and arsine.<sup>[26]</sup> Phosgene, a colorless toxic gas with a permissible exposure level of 0.1 ppm, was used as a chemical weapon during World War I. Arsine is also a highly toxic gas used in the semiconductor industry, with a permissible exposure level of 50 ppb. Water-soluble amines such as ethylenediamine, phenylenediamine, and metanilic acid were found to make PANI nanofibers sensitive to phosgene. In addition, water-soluble metal salts were readily incorporated into PANI nanofibers, such as  $\text{CuCl}_2$ ,  $\text{CuBr}_2$ ,  $\text{CuF}_2$ ,  $\text{Cu}(\text{O}_2\text{CCH}_3)_2$ ,  $\text{Cu}(\text{NO}_3)_2$ ,  $\text{EuCl}_2$ ,  $\text{NiCl}_2$ ,  $\text{FeCl}_3$ , and  $\text{CoCl}_2$ . The salts were screened to find the best candidates for detecting arsine. The  $\text{CuBr}_2$ /PANI nanofiber composite gave the best response with a change in resistance of more than an order of magnitude upon exposure to arsine. One of the notorious toxic gases is sarin, which is an organophosphorous compound. Flexible nerve agent sensors based on hydroxylated PEDOT nanotubes (HPNTs) with unique surface substructures were recently demonstrated.<sup>[27,28]</sup> Axially aligned HPNTs were produced via electrospinning process under a magnetic field, and then integrated into a conductometric substrate. Dimethyl methylphosphonate (DMMP) is commonly used as a simulant for sarin. Upon exposure to DMMP, the HPNTs showed a sharp increase in resistance. The reaction time was as fast as *ca.* 1 s, and the recovery time was as good as 3–25 s. The lowest detectable concentration was as low as 10 ppt. Figure 8 provides information on the selectivity of the HPNTs toward similar organophosphorus compounds such as trimethyl phosphate (TMP), methyl dichlorophosphate (MDCP), and trichlorophosphate (TCP), used as nerve gas simulants. The hydroxyl side chain of PEDOT was found to interact with the nerve agent simulants through hydrogen bonding. The hydrogen-bond strength was calculated to increase in the order  $\text{TCP} < \text{MDCP} < \text{sarin} \approx \text{DMMP} < \text{TMP}$ , which would lead to different response intensities with the same tendency. The response of HPNTs against 15 volatile organic compounds, chosen as potential interferents, was examined and compared to responses from other sensing materials using principal components analysis. The first three principal component scores are plotted in Figure 8c. Unique signatures of the analytes could be observed, denoted by their segregation into separate regions of the plot, allowing identification of individual analytes by their responses. Particularly, DMMP exhibited clearly differentiable components, which validated



the selective recognition ability of HPNTs. The response of the HPNTs was found to be durable even under mechanical deformation, which provided the possibility of fabricating a wearable nerve agent sensor.



## CONCLUSION

A number of studies have demonstrated that conducting polymer nanomaterials are promising candidates for building state-of-the-art sensors, due to their unique advantages over other materials. The most important parameters that determine the sensor performance include response/recovery time, sensitivity, selectivity and stability. A great deal of effort has been directed toward enhancing these parameters over the past decades. The response and recovery times and the sensitivity have experienced impressive improvements with great advances in nanotechnology. However, selectivity is still a challenge. Detecting target species in a complex environment remains a difficult task, and is hindering the widespread application of conducting polymer-based sensors. Most sensors have been demonstrated only under controlled laboratory conditions, while actual samples comprise a range of interferent chemicals and substances. A conducting polymer by itself lacks the specificity or selectivity toward target species, and thus, it is crucial to judiciously functionalize the polymer with appropriate receptors. Another concern is that conducting polymers may degrade over time, even in dry, oxygen-free environments. New efforts should be aimed at improving the stability of the sensor response.

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