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Review

Smart Polymers in Micro and Nano Sensory Devices

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Abstract: The present review presents the most recent developments concerning the application of sensory polymers in the detection and quantification of different target species. We will firstly describe the main polymers that are being employed as sensory polymers, including, for example, conducting or acrylate-based polymers. In the second part of the review, we will briefly describe the different mechanisms of detection and the target species, such as metal cations and anions, explosives, and biological and biomedical substances. To conclude, we will describe the advancements in recent years concerning the fabrication of micro and nano sensory devices based on smart polymers, with a bibliographic revision of the research work published between 2005 and today, with special emphasis on research work presented since 2010. A final section exposing the perspectives and challenges of this interesting research line will end the present review article.

Keywords: sensors; smart polymers; detection; sensory devices

1. Introduction

Sensory or smart polymers present the ability to respond, reversibly or irreversibly, to different stimuli. Different sources can cause these responses: Temperature [1], electromagnetic pulses [2], biological molecules [3] or Ph media [4]. The smart polymer, when affected by these stimuli, changes various physicochemical properties, such as solubility, color, fluorescence, or even shape. The research devoted to the synthesis and analysis of the properties of these polymers has increased greatly in recent years, and they are starting to being used in a great variety of sectors, including biosensors [5], biomedical applications [6] and drug delivery [7].

The easy processability of the polymers makes them suitable for preparation in different shapes, such as coatings, films, fibers or wires, and with tuned hydrophilicity. The interaction between the sensor species or receptor motif and the guest in the smart polymer has led to the generation of different materials that are able to be used as sensory devices [8]. The analyte, or target species, produces a quantitative response in the sensory molecule, in terms of a noticeable physical property, which must be properly detected and quantified through recognition and transduction processes, thus producing an easily measurable macroscopic characteristic [9]. Our review work will be focused on chemical sensors based on smart polymers and will not consider physical sensors. Chemical sensors are devices that respond specifically to a particular analyte in an specific way through a chemical reaction, which is used to for the quantitative and/or qualitative determination of the analyte. On the other hand, physical sensors are concerned with measuring physical quantities such as length, weight, temperature, etc. for their own sake.

Although there are different characteristics that can be used to define the sensory behavior, selectivity and sensitivity are of primary significance. Sensitivity defines the detection limit or the minimal quantity of the analyte that can be detected, whereas selectivity is related to the capacity to detect a specific analyte in the presence of other target molecules (in a so-called interference analysis).

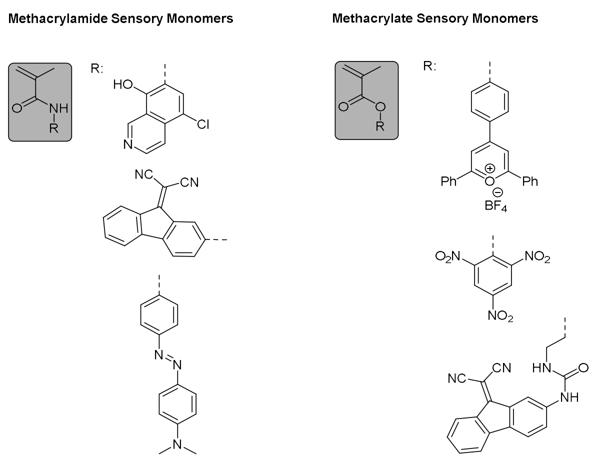
We have organized this review into three different sections. In the first part of the work, the descriptions of the different smart polymers that are used in sensory applications are detailed. In a second, we will present the mechanisms of detection and the main target species that are currently being immobilized and quantified using sensory polymeric materials, including the quickly evolving field of biological applications. Finally, we will describe the main sensory devices that employ sensory polymers that have been developed in the last 10–15 years, especially those focused on the micro- and nanoscale. The review will finalize with a brief summary and outlooks.

2. Polymers with Sensory Properties

A great variety of polymers can be used as sensory materials. In this review, we have selected the most important polymers that are currently being employed in sensory devices, as well as the polymers that are now undergoing extensive research and development as sensory materials. Thus, the sensory polymers analyzed include molecularly imprinted polymers, polymeric nanocomposites and hybrid polymers, acrylic polymers, polymers with chiral motifs, conjugated or conductive polymers and sensor arrays based on a set of polymers.

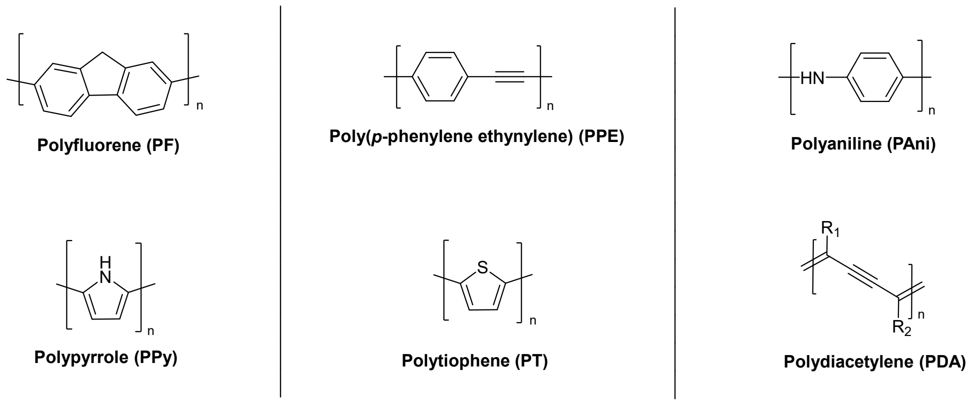
The preparation of molecularly imprinted polymers (MIPs) is carried out by the molecular imprinted technology, in which a polymer is combined in the polymerization process with the target species, which is used as template. Removing the target species creates precise hollow spaces that act as receptor sites for the target. The use of MIPs in different applications has been described previously by different authors [10]. Concerning polymeric nanocomposites and hybrid polymers, these two-phase structures are both formed by organic/inorganic phases, but in a different way. In the case of polymeric nanocomposites, the mixture starts from a polymeric moiety charged with inorganic particles with sizes in the nanometer range (1–100 nm). However, hybrid polymers are also formed of two different natures, but linked covalently, resulting in a single-phase structure [11].

Acrylic polymers belong to the "classical" polymeric materials, and have been analyzed for decades. These kinds of polymers can be used as sensory materials due to their versatility, and also a great variety of sensory units can be anchored chemically to their structure, thus conferring on them numerous advantages. The main acrylic polymers found in sensory applications are polymers prepared with esters of acrylic or methacrylic acid, acrylamide derivatives, and copolymers thereof, as shown in Scheme 1, in which different sensory units that can be precisely anchored to acrylic motifs to later prepare sensory polymers are presented. More examples can be found in the bibliography presented afterwards.



Scheme 1. Some examples of acrylic-based sensory monomers used for preparing sensory polymers for the detection of charged species.

Conductive polymers are formed by main polymer chains that alternate simple and multiple bonds. In the normal state, these polymers are electrically insulating or semiconductors, but they are easily converted to conductor materials by doping. They also present luminescence, and this physical property, along with the electrical signal derived from the (semi)conductivity, can be used as the output signal in sensory devices. Some of the main conducting polymers include fluorene derivatives (PFs), polyaniline (PAni) or polypyrrole (PPy), as shown in Scheme 2 [12].



Scheme 2. Some typical conducting polymers used as sensors.

In recent years, different ligands have been introduced into the main polymer chain to achieve chiral recognition of important chiral analytes. A description of the different ligands that are currently being employed to obtain sensory polymers, mainly in luminescence and colorimetric sensors, can be found in several works in the literature [13,14].

To conclude this brief introduction to the main sensory polymers that are employed in sensory devices, it is necessary to introduce the sensory arrays, which are based on a set of polymers. This disposition is used when the response of only one type of polymer is not selective or sensitive enough for the application, thus the combination of different polymers in an array produces a more effective sensor, increasing the selectivity and selectivity of the sensing procedure. Sensory arrays are employed in a great variety of applications, from gas detection to biological applications [15,16].

3. Mechanisms of Detection and Target Species

The sensing process is based on the change in various physical and chemical properties of the polymers, which vary in the presence of the target species. In this review, the micro and nano sensory devices described are based on the analysis and transduction of this change in physical properties during the sensing process. Following this line, there are different mechanisms of detection that are employed based on different physicochemical characteristics.

First, changes of colorimetric, luminescence (fluorescence or phosphorescence) properties of smart polymers when exposed to target species produces easily quantifiable sensory responses. Color variation depends on selective chemical reactions or specific interactions based on feeble interactions, such as hydrogen bonds. The color change is identifiable by naked-eye or by simple colorimetric techniques (RGB determination) [17,18]. On the other hand, in fluorescence sensors, the sensory behavior includes a receptor that recognizes the target, and a fluorophore responsible for the fluorescence signal. In contrast to colorimetric sensors, the sensing must be quantified using fluorescence spectrometers, in terms of the turn-off or quenching of the luminescence (the deactivation of the luminescence [19]), or the opposite effect, the activation (turn-on) of the luminescence of the sensory species [20,21].

Another typical response is based on changes in electrical resistance due to the surface reaction with different chemicals, thus offering the possibility to measure changes in the resistivity of the polymer in the presence of different analytes (resistive sensors). In this sense, special organically resistive sensors using conducting polymers (CPs) offer great potential to produce flexible, low-cost and lightweight devices with good mechanical properties and tunable electrical conductivity. A similar detection method is used when employing modified electrodes coated with sensory polymers, thus detecting the sensory response throughout electrochemical measurements, mainly cyclic voltammetry [22].

Finally, an interesting method for detecting different species is the use of piezo-resistive quartz crystal micro balances (QCMs). In these devices, the sensory effect is produced due to the mass change of the polymer when the species is absorbed. The mass change produces a variation in the resonance frequency of the piezoelectric crystal, which can easily be measured and quantified. It is important to use appropriate coating materials on the QCM sensor surfaces, to specifically determine their final properties and applications [23].

A wide variety of target species or analytes can be detected using sensory polymeric materials. In this review, we have selected four main groups of species that are currently under extensive research. Heavy metal cations and anions, gases and organic volatile compounds (VOCs), explosives and chemical warfare agents and finally the recent research concerning the biological and biomedical applications, mainly focused in the immobilization and detection of proteins, glucose and different disease markers.

The optical response (colorimetric and luminescence) in sensory polymers is mainly employed in the detection (in aqueous solution) of heavy metal cations and anions. The detection of these ions is carried out using conducting, hybrid and acrylic polymers. In this sense, copper cations are detected employing different polymeric main structures such as polyacetylene, polypentiptycene ethynylene or polyfluorene derivatives using quenching fluorescence techniques, with detection limits in the range of ppm and ppb [24], whereas the detection of Zn^{2+} cations is analyzed using polymers containing (*R*,*R*)-salen-based moieties, which show fluorescence responses when exposed to different zinc cations [25]. Also, sensors based on conducting polyaniline are employed to detect Hg^{2+} cations, by means of resistivity variation, with very low detection limits (ppb) [26]. The importance of the Hg^{2+} detection is also reflected in several recent works [27–30]. Finally, other cations such as Pd^{2+} and Pt^{4+} are detected through the fluorescence variation of different sensory polymers [31,32].

Resistive sensors are mainly focused on the detection of harmful target gases (such as NH_3, H_2S or CO) and VOCs (such as alcohols, CO_2 or environmental humidity). The use of conducting polymers in these resistive devices is especially interesting, producing flexible and low-cost devices with tuned electrical conductivity [33–35]. Some examples include the detection of gases such as hexane or methanol using polymer films with spectroscopic properties [36], the quantification of CO_2 in ethanol solutions by means of branched PEI using the turn-on effect of the fluorescence [37], and the detection of humidity using polymeric films based on 4-vinylpyridimium with fast response times [38].

Due to the increasing terrorist menace and the reinforcement of security measures, the detection of explosives and harmful substances, mainly in gas phase, is one of the most important envisaged applications of sensing polymers. Taking into account all the experience acquired in gas sensing, new and different developments have been carried out in this field. For example, films based on pentiptycene and tetraphenylehylene moieties can detect picric acid aqueous media, from the fluorescence quenching effect, with very low detection limits (ppb) [39]. Hydrazine vapors are also detected using a film based in poly(phenyl ethers) [40]. A related research line is focused on the use of films from acrylic polymers to detect TNT vapors and also in aqueous media [41–43].

Other dangerous anions, such as fluorides and cyanides, can also be detected using sensory polymeric materials. For example, fluoride anion F^- is detected trough the formation of coumarins in fluorinated poly(phthalazinone ether), activating the fluorescence signal [44]. On the other hand, the detection of CN^- using hybrid polymers containing Cu^{2+} have been carried out using different polymers containing several backbone families, such as functionalized hybrid polyacetylene [45], or dicyano-vinyl containing a conjugated polymer [46], with very low detection limits (in the range of ppb).

Undoubtedly, the study of biological and biomedical applications of sensory polymers has increased exponentially in the last 10–15 years. Different biomolecules, proteins or, especially, glucose can be detected accurately using different sensory devices based on smart polymers. In this review, we note the most recent works, but it is important to note that this field is evolving quickly. We then include some interesting examples: Folic acid can be detected using a sensor based on MIPs formed by electropolymerization of the thermosensitive amine-terminated poly(*N*-isopropylacrylamide), and then measuring, using cyclic voltammetry techniques, changes in the electrochemical properties in presence of the analyte [47]. Proteins such as myoglobin are detected using nanofilms based on poly(hydroxyethyl methacrylate-*N*-methacryloyl-(*L*)-tryptophan methyl ester), which is coated on a gold surface. Then, surface plasmon resonance measurements are preformed, detecting the presence of the myoglobin with a sensitivity of 26.3 ng/mL, and also with great selectivity against other species, such as lysozyme, cytochrome and bovine serum albumin. This sensory system has been applied to real samples from myocardial infarction patients [48]. Protein detection is also carried out capturing and quantifying amino acids and neurotransmitters of X-linked inhibitors of apoptosis protein, using polymeric sensors with anchored juglone backbones [49]. As stated before, glucose is detected in many different ways, due to the medical importance for the control of diabetes. For example, nano and micro porous shaped polypyrrole films can be used to detect glucose through voltammetry measurements [50]. Hormones such as testosterone are also possible to detect by their ability to bind to an acrylic acid film [51], whereas melamine can be quantified directly in real milk samples using a fluorescent MIP chemosensor based on a rhodamine derivative, with low detection limits (1.55×10^{-4} M) [52].

To list the recent works focused in the synthesis and properties of sensory polymers, we present in Table 1 a bibliographic review concerning the use of smart polymers as sensors for the detection of the target species described previously: different cations and anions, gases and volatile organic compounds, explosives and harmful substances and to conclude, the detection of biomolecules.

Table 1. Detection of analytes using sensory polymers. (For additional information, please see reference [53]).

Type of Analyte	Target Species	References
Cations and anions	Hg^{2+}	[26–30,54–63]
	Zn^{2+}	[64–69]
	Cu^{2+}	[20,24,70–74]
	Fe^{2+},Fe^{3+}	[57,73,75–79]
	Al^{3+}	[58,78,80,81]
	Cr^{3+},Cr^{6+}	[27,58,73,78]
	F^{-}	[44,82,83]
	CN^{-}	[45,46,74,84–88]
	Other cations	[19,31,32,72,79,89,90]
Gases and Volatile Organic Compounds	NH_3	[35,91–94]
	CO_2	[37,95,96]
	H_2O	[38,97–100]
	VOCs	[17,18,22,23,36,101–104]
Explosives and harmful substances	PETN [1]/RDX [2]	[105]
	TATP [3]	[106]
	DCP [4]	[107]
	Hydrazine	[40,108]
	DNB [5], DNT [6]	[21,109,110]
	TNT [7]	[41–43,105,110–112]

Table 1. *Cont.*

Type of Analyte	Target Species	References
Biomolecules	Drugs	[113–117]
	Hormones	[51,118,119]
	Neurotransmitters	[120–122]
	Sugars/Saccharides	[50,122–130]
	Amino acids and proteins	[48,131–135]
	Metabolites	[47,136,137]
	Other biomolecules	[52,62,63,138,139]

[1] PETN—Pentaerythritol tetranitrate; [2] RDX—1,3,5-trinitro-1,3,5-triazinane; [3] TATP—Triacetone triperoxide; [4] DCP—Diethyl chlorophospite; [5] DNB—2,4-dinitrobenzene; [6] DNT—2,4-dinitrotoluene; [7] TNT—2,4,6-trinitrotoluene.

4. New Micro and Nano Sensory Devices Based on Smart Polymers

The development of new sensory polymers has opened new possibilities in the preparation of micro- and nanodevices, which are essentially focused on detecting and quantifying different physical properties that change due to the sensing effect. The quantification of these properties is related directly to the amount (sensitivity) and the type (selectivity) of the target species. In this sense, sensory devices based on smart polymers are evolving quickly, and in this review, we will present the most representative. We will analyze the micro- and nanodevices based on polymeric nanofibers, films and coatings, sensory arrays (which are extensively used in electronic noses), QCMs, microfluidic devices, modified electrodes with smart polymers, and also sensory devices based on polymer field-effect transistors. We will also include a brief description of sensory chips, which are gaining interest for biological and biomedical applications, the use of micro and nanoporous materials, and finally a couple of references related to the sensory devices based on LIPSS (Laser Induced Periodic Surface Structures), as well as sensory devices related to the SERS (Surface Enhanced Raman Spectroscopy) effect, which can be used for the sensor enhancement effect. This part of the review will also include a deep bibliographic outlook starting from the early 2000 s and extending up to the present day, ending with a schematic table covering the most important research works presented in this investigation line.

Polymeric nanofibers present great interest due to their combination of high surface area, large porosity and interconnected porous structure, thus conferring them with high selectivity and sensitivity, reversibility and also fast response time. Different physical properties can be measured using these materials, thus leading to several detection mechanisms. Some of them have already been detailed in at the beginning of Section 3, such as resistive and optical responses. In this case, we also include the surface acoustic wave (SAW) sensors, in which the sensing mechanism is related to the analysis of the change in the velocity of an acoustic wave on a piezoelectric substrate surface, which is caused by the adsorption process of different analytes. There are different applications in which polymeric nanofibers can be used, such as biosensors (especially glucose sensors for the diagnosis and treatment of diabetes, due to the high sensitivity and selectivity to glucose and glucose oxidase). Other analytes detected with these devices are fructose, methanol or hydrazine, and also hormones and amino acids, such as thyroxine and methionine. The review presented by Wang et al. resumes the fabrication process and applications of sensory devices based on nanofibers [140].

Due to their easy processability, polymers can be transformed into films or coatings. In this second case, the sensory monomer, or macromolecules with receptor motifs, is dispersed or anchored, generally in small proportions (up to 5 wt %) acting as coating in a given substrate. The deposition takes place usually using spraying or sputtering techniques of the polymer solution using air brushes. The thickness of the coating varies between a few nanometers to 10 µm. An interesting work has been published by Fitzgerald et al. [36] analyzing the use of polymeric sensory thin films, and also the use of MIPs in sensory applications is investigated in the review paper published by Ye and Mosbach [141]. Concerning use of polymeric films, these materials are produced through a radical polymerization of the monomers together with the sensory monomer, which is subsequently employed in the detection, through the different mechanisms (colorimetry, fluorescence, etc.), explained previously. These materials are mainly employed for the detection of several vapors or substances in aqueous media. Polymeric films are obtained with thicknesses of up to 100 μm, and can be modulated in terms of flexibility, hydrophilicity or transparency.

As seen at the end of Section 2, sensory arrays are finding interesting applications in many research fields, due to their versatility and the possibility of detecting, using a single device, different species. A sensory array consists, basically, of a device containing several sensory polymers, thus broadening the number of analytes that can be detected using a single device. In this way, in biomedicine sensory chips arrays are used to detect different proteins and clinically relevant biomolecules to prevent the apparition of diseases [142]. At this point, it is necessary to remark on a specific type of sensory array that has been recently developed, denoted electronic noses, which are specifically focused on the detection of odors and flavors. The scientific development and industrial applications have increased in parallel during the last decade, as shown in the review published by Arshak et al. [143], with special attention to a specific application that has attracted a lot of interest concerning the characterization of olive oil and wine, as described in the review work presented by Rodríguez-Méndez et al. [144].

QCMs are employed as sensory devices taking advantage of the piezoelectric effect. In these devices, the sensory effect is produced due to a mass change of the polymer when the target species is detected, thus originating an electrical signal (change of resonance frequency) that can be easily measured and quantified. In QCM sensors, the smart polymer directly coats the substrate, and detecting an electrical signal shortens the response time, enhances the sensitivity, and also reduces the variability. Sensory devices based on QCMs modified with smart polymers are being employed in a great number of applications. For example, in the detection of organic vapors and harmful gases, as described in the review of Nanto et al. [145], in biological applications such the immobilization of antibodies and DNA, and also in the detection of heavy metal ions (copper, cadmium or gold cations), as described in several published works [146,147].

Microfluidic and nanofluidic technologies emerged in the early 2000 s, and have recently become a very interesting fabrication technique for obtaining micro and nano sensory devices based on smart polymers. Although the detection methods employed in these devices have already been described, electrochemical measurements enhance their selectivity and sensitivity, and for this reason, they are widely used. This detection method is based on the electrical modulation of the analyte species that occurs under redox reactions, with electroactive species being detected through cyclic voltammetry measurements. Two main applications have recently been the focus of research efforts. First, biological and biomedical applications, such as the detection of proteins and glucose [127,128], including a deep review published recently covering the applications of the sensory systems integrated in microfluidic devices [148]. The other interesting applications follows a classical research line described previously, and is focused on the detection of heavy metal ions, which is crucial for environmental and safety reasons [149]. Following this line, in situ measurements, especially, for controlling environmental pollution, have gained interest, as detailed in the review recently published on this topic [150].

Another type of sensory devices uses modified electrodes with smart polymers. In these sensory devices, analytical methods allow the utilization of electrochemical measurements (cyclic voltammetry) to determine the sensing effect by means of electrical signals, thus improving the detection and quantification experiments. The modification of these electrodes can be done in several ways, and using different materials, as described in the review published in 2016 by Naveen et al. [151]. Non-modified electrodes based on metal or graphene are coated with sensory polymers, (conducting polymers or polymer nanocomposites), then the electrochemical properties are measured, and the voltammetry results are correlated directly with the detection and quantification properties of the sensor. The applications in which these sensory devices are employed have been also increased greatly in the last decade. For example, detection of heavy metal ions (such as palladium), is carried out using graphene modified electrodes [152]. The detection of VOCs, such as phenol, ethanol or methanol is also analyzed using modified electrodes, as well as the quantification of quantification of humidity, which is carried out using electrodes modified with conducting polymers [99]. Finally, as seen throughout all of this review, biological applications, mainly protein detection, are currently under investigation using these devices. We can remark on applications concerning the electrochemical biosensing of glucose and lactic acid [153] and different medications and drugs, such as anticancer substances [154] or phenobarbital [155]. A deep bibliographic review was published in 2016, with special attention given to biological and biomedical applications [156]. Additionally, following this line, the use of polymer field-effect transistors has gained a lot of attention in recent years, with different works being published related to the detection of H_2S and ammonia [157,158]. A review focused on gas sensors based on these devices has recently been published by Lv et al. [159].

Two other interesting and emerging sensory devices are the so-called sensory chips and the sensory devices based on microporous and nanoporous materials. Concerning sensory chips, these are handleable devices that can be used directly on human skin to carry out in situ biological analysis (detection of proteins or immobilization of disease markers), and also temperature monitoring. It is important to remark that chips can be also developed in the form of sensory arrays, thus detecting different biological molecules using a single chip. For these reasons, they have attracted special attention in the last years, with an incipient development. A detailed list of recent research papers will be detailed next, but we will remark on the review published in 2015 by Chuan-Guang et al., devoted to the analysis and detection of azobenzene derivatives using polymeric sensing chips [160], as well as an interesting research paper presented by Song et al. concerning antigen–antibody recognition using biological chips based on poly(*N*-isopropylacrylamide) [161]. Another interesting research line investigates the use of micro- and nanoporous materials in sensory devices. These materials present several advantages over traditional sensors, especially the increased interfacial area, which leads to higher sensitivity compared to solid materials. The use of microporous and nanoporous materials is specifically focused on developing new porous substrates in which the sensory polymer is deposited. Some examples of applications include the detection of nitroaromatic vapors [111] or the immobilization and quantification of lipids [162].

To conclude this section, we detail in Table 2 the publications covering the new micro and nano sensory devices based on smart polymers, following the scheme presented previously: devices based on polymer nanofibers, polymer films and coatings, sensory polymeric arrays, modified quartz crystal microbalances, sensory devices prepared from microfluidic techniques, the use of modified electrodes coated with sensory polymers, sensory chips and finally sensory devices based on micro- and nanoporous materials.

Table 2. Overview of micro and nano sensory devices using smart polymers. (For additional information, please see references [141,144,145,148,150,151,156,160]).

Group of Sensory Devices	Type of Response	References
Polymer nanofibers	SAW Electrical Optical	[163,164] [34,35,118,165–167] [17–19,168]
Polymer films and coatings	Optical Electrochemical Electrical	[39,48,169–176] [115,177–179] [93]
Sensory polymeric arrays	Optical Electrical	[89,180–182] [183–185]
Quartz Crystal Microbalances	Piezoelectric	[14,114,130,146,186–192]
Microfluidic devices	Optical Electrical	[27,128,150,193–195] [148,190]
Modified electrodes	Electrochemical	[26,99,152–155,196–202]
Sensory chips	Optical	[161,193–195]

Table 2. *Cont.*

Group of Sensory Devices	Type of Response	References
Micro and nanoporous materials	Optical Electrochemical	[111,203–205] [162,206]
LIPSS and SERS effect	Optical	[207,208]

5. Conclusions

The present review has focused on the analysis of recent developments in the research and development of sensory polymers and the preparation of micro and nano sensory devices using these smart materials. It has been remarked that in the last 10–15 years, the interest in this research field has increased noticeably, with a great number of publications and review articles published to date. Apart from classical applications in gas or metal cation detection, the emerging field related to biological and biomedical applications is especially interesting. In parallel, the improvement in the experimental techniques to determine optical, electrical or mechanical properties have enhanced the sensitivity and selectivity of the sensory devices.

It is important not to forget the difficulties and challenges that must be overcome in these devices, such as the improvement of selectivity, the increasing of the number of target species, with special attention on biomedical and biological applications, and also the reduction of the detection or response time. These research advancements should be carried out together with the industrial applications, then improving the production methods in order to obtain and commercialize cheap and portable sensory devices.

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