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Sensors from Electrospun Nanostructures

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Abstract

Nanotechnology exerts a significant influence on materials science, providing new insights into the design of functional materials. One of the most studied areas of nanotechnologies is that of nanofibres, characterised by high specific volume, chemical activity and volume-dependent physical processes. The most promising method of producing nanofibres with various morphologies and functionalities from different materials is electrospinning, where high voltage is applied between the spinneret and the collector to the charged polymer solution (or melt) to draw polymer filaments. This chapter reviews the main electrospinning techniques for producing nanofibres from polymers, provides an overview on the influence of the spinning solution characteristics, the process parameters and the working environment on the process and highlights the many applications of electrospun nanofibres in the field of sensors. Latest advances in this field and the prospects for obtaining new electrospun nanofibre sensors are discussed.

Keywords: nanostructures, electrospinning, electrospun polymers, electrospinning techniques, sensors

1. Introduction

According to the definition adopted by the National Nanotechnology Initiative, nanotechnology is the manipulation of matter with at least one dimension in the range of 1–100 nanometres [1]. At the moment nanotechnology, an interdisciplinary field having confluence of physics, chemistry and engineering [2] is one of the most attractive areas of science [3], with applications in various domains, which radically effected progress in the field of material science [4]. Nanofibre technology, which comprises the synthesis, processing, fabrication and application of nanoscale fibres [5], is one of the main progressions in nanotechnology [6].

2. Electrospinning: principles and main techniques

Among the technologies that can be used to obtain nanofibres (phase separation, template synthesis, self-assembly, melt blowing) [7], electrospinning is one of the most promising technologies, being commonly used for producing nanosize filaments from both organic polymers and inorganic materials [8], as it is uncomplicated, versatile and capable of producing controlled size fibres with high surface to volume ratio, hence more capable to interrelate with the surrounding environment. In addition, the electrospun nanofibre structures are characterised by complex three-dimensional open porous assembly, facilitating even more the interactions with the adjacent environment. The fact that nanofibres have only one dimension at nanoscale, since the others are at macroscopic one, makes it possible to mingle the advantages of nanostructures (high chemical and biological reactivity and electroactivity) with those of conventional solid membranes, such as comfortable manipulation and easy applicability.

A polymer solution or melt can be electrospun if it is able to carry an electric charge and has enough viscosity to be strained without breaking up into droplets. Up to now, more than 200 types of materials have been electrospun into nanofibres, among which natural and synthetic polymers and many hybrid blends [6].

The principle of electrospinning is quite simple: by applying high voltages electrostatic field (e.g. 10–50 kV) to a polymer solution or a polymer melt, the surface of the fluid elongates and first forms a conical shape, known as the Taylor cone. Then, when the electric voltage reaches a threshold value, the jet of charged liquid overcomes the surface tension, leaves the Taylor cone and is drawn to the collector of different potential, forming a nanofibre net [9].

In addition to the classic electrospinning process, many variants have been developed, capable of producing nanofibres with special features: core/sheath nanofibres [10], nanofibres with hollow structures, nanofibres with porous structures [11] and necklace-like and ribbon nanofibres [12].

The elementary electrospinning setup has four parts: a reservoir of polymer in melt or solution form, a spinneret (in the simplest cases, a syringe), the high voltage power supply and the collector, which acts as a counter electrode (**Figure 1**).

Developments in electrospinning technology have made it possible to electrospin, besides polymers, polymers loaded with nanoparticles and functional molecules, ceramic materials and metal oxides. In addition, there have been developed fibres with new special structures [13].

2.1. Factors that influence the properties of 3D nanostructures obtained by electrospinning

In addition to this basic setup showed in **Figure 1**, there are relatively many other approaches to the process, but in all cases the electrospinning process is influenced by the following three categories of factors: the particularities of the spinning polymer solution/melt, the parameters of the electrospinning process and the environment factors.

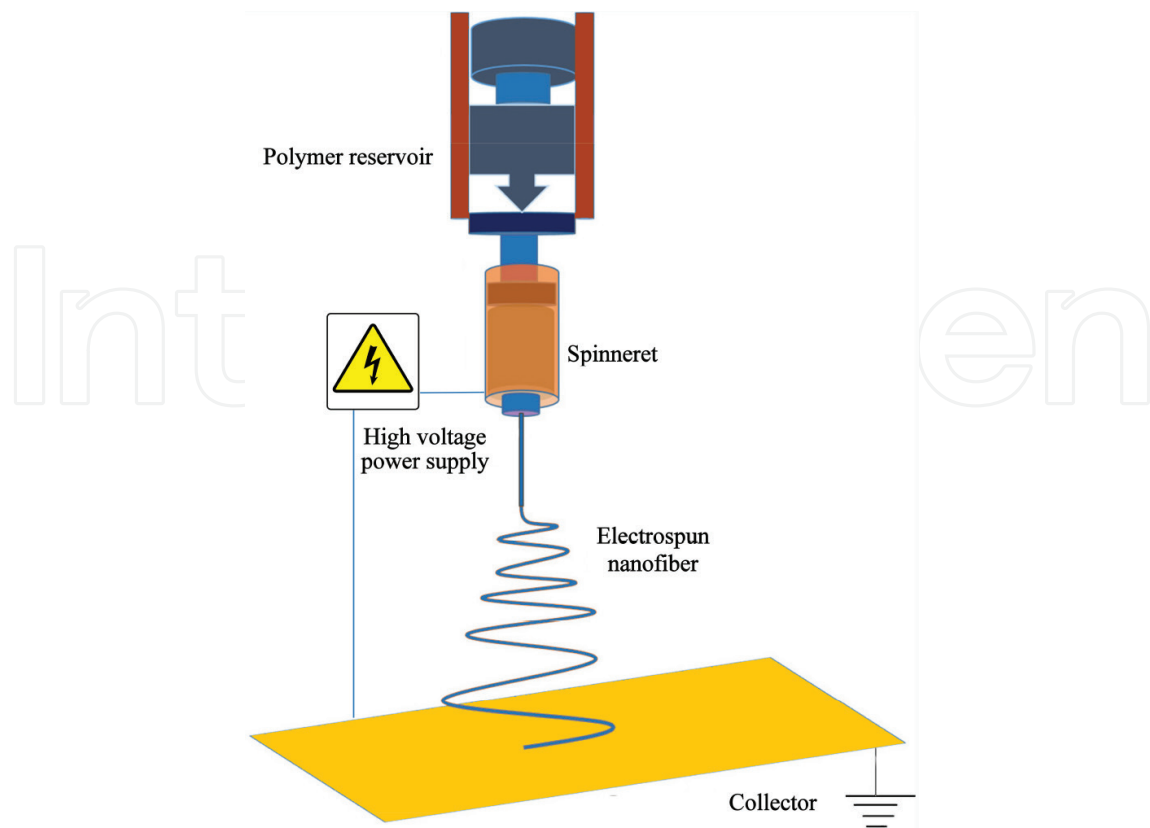


Figure 1. Typical solution electrospinning setup.

2.1.1. Polymer solution characteristics that influence the solution electrospinning process

The main characteristics of the polymer solution with a significant role in the result obtained after electrospinning are concentration, viscosity, conductivity, superficial tension and the volatility characteristics of the solvent.

The solution viscosity is considered to be the dominant variable that determines the fibre diameter. To make electrospinning possible, the viscosity of the electrospun polymer solution must be within a precisely defined range. A too low viscosity leads to interrupted polymer filaments and the appearance of polymer droplets [14], while too high viscosity makes polymer extrusion impossible. The optimum values of viscosity differ according to the molecular weight of the polymer and the solvent used. Of course, there is a close correlation between the concentration of the polymer solution and its viscosity.

An innovative approach to reducing the viscosity of the polymer solution is to apply low-frequency vibrations during the process [15, 16], when vibrations disrupt some of the van der Waals interchain interactions, disordering the polymer chains, which leads to a decrease in the solution viscosity.

Electrospinning fundamentally requires the transfer of electrical charge from the electrode to the polymer droplet at the end point of the syringe needle, so a minimum electrical conductivity in solution is therefore essential for the process; solutions lacking conductivity cannot be electrospun.

A correction of insufficient polymer solution conductivity can be obtained by adding an electrolyte, when the increased number of charges results in an increase in the solution elongation capacity, favouring the formation of smooth, small diameter fibres [17].

Surface tension, strongly influenced by the nature of solvent, is a very important factor in electrospinning; basically, if all the other conditions are established, surface tension controls the upper and lower limits of the range in which the electrospinning can be achieved [18, 19]. Surface tension can be adjusted by varying the polymer/solvent ratio in the spinning solution or by adding surfactant to the solution, which ensures the formation of more uniform fibres [20].

2.1.2. Processing parameters that influence the solution electrospinning process

The applied voltage is one of the most important parameters of the electrospinning process, as it influences directly both the dynamics of fluid flow and the morphological characteristics of the electrospun fibres.

Only when the applied voltage exceeds a certain threshold value, load charged polymer jets are ejected from the Taylor cone. The size of this threshold voltage depends on the nature of the polymer-solvent system [21].

It is generally accepted that an increase in the applied voltage leads to an increase in the deposition rate, thus there is a greater probability of defect formation [22, 23]. The length and diameter of the electrospun fibres decrease with the increase in applied voltage without any change in pore size [20]. For nanofibres produced with low voltage, a uniform morphology with fewer defects and drops is obtained [24].

The distance between the electrode and the collector is important because, if correctly set, can control the morphology and diameters of the nanospun fibres. If the distance is too small, the fibres will not have enough time to solidify before reaching the collector, while if the distance is too long, it is possible to obtain fibre with beads at the surface [25]. A slight change in this distance significantly influences the characteristics of the fibres, the diameter of which becoming smaller as the distance to the collector increases.

The flow rate of the polymer solution within the syringe is an important process parameter; generally, a lower flow rate is recommended to give the polymer solution sufficient time for polarisation. If the flow rate is too high, fibres with many beads are formed due to the insufficient time for the polymer filament to solidify before reaching the collector [26].

2.1.3. Environmental parameters that influence the solution electrospinning process

The influence of the humidity of the environment in which electrospinning takes place is manifested in terms of fibre morphology, deposition orientation and solvent evaporation rate [27]. At very low humidity, a volatile solvent can be dried very quickly, while high humidity helps to discharge static electricity to electrospun fibres. In addition, at high humidity, condensation may occur at the surface of the fibre due to the cooling of the surface of the jet determined by the rapid evaporation of the solvent, and the air flow can interrupt the formation of fibres, causing ruptures [20].

The temperature of the electrospinning environment has a significant influence on the process, as the evaporation rate of the solvent decreases exponentially with the decrease in temperature; when the evaporation process of the solvent becomes slower, the jet takes a longer time to solidify, which can lead to defects in fibre formation [28]. Consequently, temperature control is essential to adjust the evaporation rate of the solvent and the viscosity of the solution [29].

At low atmospheric pressure, the polymer solution in the syringe tends to flow, causing an unstable jet initiation, and at very low pressures, electrospinning cannot be achieved due to direct discharge of electrical charges.

2.1.4. Particularities of melt polymer electrospinning

Melt electrospinning has a number of advantages over solution electrospinning, mainly linked to the absence of solvent, which reduces costs and makes the process more environmentally friendly [30]. In addition, by melt electrospinning, it is possible to transform into nanofibre polymers for which there are no suitable solvents, such as polyolefin or polyethylene terephthalate, or mixtures of polymers for which it is difficult to find a unique solvent for all the components [31, 32]. Unlike the solution electrospinning, where frequently a nonwoven fibrous mat is obtained, the melt electrospinning can produce filaments which can be used in knitting or weaving processes [33]. In this case, the melt electrospinning process (known as melt electrospinning writing) uses a moving collector, which exerts a translational movement sufficiently fast for the rectilinear deposition of the polymer jet [34].

In addition to these advantages, there are difficulties arising from the particular equipment required (**Figure 2**) and the high viscosity and low electrical conductivity of melt polymers.

The main feature of the polymer melt that influences electrospinning is viscosity. As the viscosity of the melt is at least one order of magnitude greater than that of the polymer solutions, it is essential to reduce it. This is usually done by raising the temperature (without degrading the polymer) or by adding additives such as cationic surfactants [35, 36].

Another parameter related to the polymer melt is the molecular weight of the polymer, which influences the diameter of the obtained nanofibres. When the molecular weight of the electrospun polymer is higher, the Melt flow index is lower and the diameter of the fibres is bigger. Because in the melt electrospinning, the viscosity cannot be controlled by adjusting the polymer/solvent ratio, like in the solution electrospinning, it is essential to choose a suitable molecular weight of the polymer to achieve a stable and reproducible electrospinning process [33]. It is believed that the optimal range of molecular weights for melt electrospinning is substantially lower than that characteristic of solution electrospinning. The process is also influenced by the stereoregularity of the polymer, the isotactic polymers producing finer fibres than the atactic ones [32].

The conductivity of the polymer melt decisively influences the stability of the extruded polymer jet. A too high conductivity determines the jet's instability, while a too small one will cause reduced electrostatic traction forces. It is considered that average conductivity values in the range of 10^{-6} – 10^{-8} S/m prerequisites for a stable electrospinning process [33].

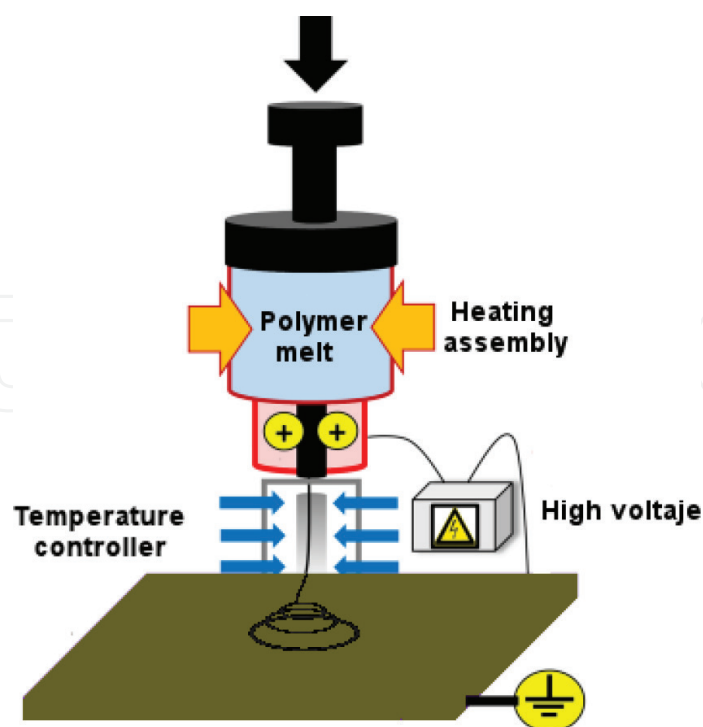


Figure 2. Melt electrospinning setup.

In what regards the processing parameters that influence the melt electrospinning process, it can be seen that, unlike the solution electrospinning, there is a direct proportionality relationship between the fibre diameter and the melt polymer flow rate.

Concerning the effect of the applied voltage on the diameter of the fibres, information is contradictory, but the overall conclusion is that for each polymer there is an optimum voltage range [37–40].

The distance between the spinneret and the collector is relatively lower in melt electrospinning than in the solution process (usually 3–5 cm). This distance influences the cooling process of the polymer melt jet and the shape of the fibres. Flat fibres may appear at smaller spinneret to collector distances, while larger distances affect the accuracy of depositing filaments on the collector in the melt electrospinning writing process [34].

3. Electrospun sensors

Sensors, which convert energy to detect concentration changes of a specific substance and communicate the information in the form of an electrical or optical signal [41], have gained special importance in recent decades, due to their numerous applications in areas such as monitoring environmental factors, health and wellbeing or detection of dangerous vapours [42].

A high specific surface area and a very porous structure are vital for high sensitivity and quick response of a sensor [31]. This is why electrospun nanomaterials, having large specific surface area and high and tunable porosity [43], have found frequent use in the field of sensors of many types, such as chemiresistive, optic (fluorescent and colorimetric), acoustic

wave (piezoelectric) or photoelectric [13]. Electrospun nanostructured sensors display faster adsorption and minimised bulk effects when compared to the conventional sensors [44].

3.1. Chemiresistive electrospun sensors

Chemiresistive sensors, which work by measuring resistance variation when in contact with the substance to be detected, have a high miniaturisation potential, making them applicable to portable devices. Semiconductive polymers, metal oxides, metals and conjugated compounds are used to obtain such sensors [42, 45].

Polymers that exhibit conductivity, such as polyaniline (PANI), polypyrrole (PPY) and poly(3, 4-ethylenedioxythiophene) (PEDOT), are used as gas sensors [46]. For example, PEDOT, insoluble in pure state, becomes dispersible in water after doping with poly(styrenesulfonate) (PSS) and can be electrospun, the obtained nanofibres being used for the detection of inorganic compounds such as nitrogen oxide and ammonia and organic substances (ethanol, methanol and acetone) by measuring resistance variation [47, 48].

Polyaniline (PAni) doped with (+)-camphor-10-sulfonic acid (HCSA) has been electrospun and proved to be excellent ammonia sensors, while the electrospun fibres obtained from undoped PAni exhibited very good nitrogen oxide detection capabilities [49]. Polyaniline electrospun nanofibres also proved to be effective as moisture sensor, when the response to changes was given by impedance variation. It was found that the sensor exhibited a rapid, reversible and very sensitive change in impedance as a function of adsorbed water molecules [50]. A sensor from electrospun nanofibres from polyaniline/polyethylene oxide (PAni-PEO) doped with 10-camphosphulfonic acid (HCSA) and deposited on a pair of gold electrodes modifies resistance, having a 0.5 ppm detection threshold for ammonia [51].

Chemiresistive sensors from PAN/PAni electrospun fibres showed good sensitivity to NH_3 gas at room temperature and this sensitivity increased with increasing gas concentration [52].

Ammonia sensors from quartz crystal microbalance coated with electrospun polyacrylic acid (PAA) membranes showed high sensitivity at low ppb level, but this sensitivity depended on the morphology of the nanofibres, the load of the quartz crystal microbalance with the electrospun membrane and the relative humidity [53].

Another ammonia sensor was obtained by depositing a P-type conductive PAni onto the surface of n-type semiconductive TiO_2 electrospun fibres. The sensitivity of this sensor substantially increases with the increase of NH_3 concentration [54].

Electrospun nanofibres from PANI and PANI/ZnO were used to detect HCl and NH_3 vapours at room temperature, recording a decrease in the resistance of the sensors when exposed to HCl vapour, respectively an increase in resistance when exposure to NH_3 vapours. The PAni/ZnO sensor has a high sensitivity response at room temperature with better repeatability compared to that of the pure PANI sensor [55].

An ammonia sensor based on a single PAni nanofibre demonstrated a very low detection value (under 1 ppm) and a response time under 10 s [56].

Electrospun nanofibres of poly(o-anisidine)-polystyrene doped with (+)-camphor-10-sulfonic acid proved to be effective in water and ethanol determination, showing a high sensitivity and rapid response and recovery [57].

Electrospun conductive polypyrrole nanofibres coating a copper interdigital electrode has been used to produce an aliphatic amines gas sensor with low detection limit (0.42 ppm for *n*-butylamine), quick response and good repeatability at temperatures between 90 and 200°C [58]. PANi/poly (vinyl pyrrolidone) nanofibres containing urease have been used as NO₂ sensors, showing a significant conductivity increase in the presence of levels of NO₂ in the range of 1–7 ppm [59].

Electrospun nanofibres of Al-SnO₂/PANi were used as hydrogen sensors, having high sensitivity at low temperature (48°C) [60], while sensors made of PANi/TiO₂:SnO₂ nanofibres placed onto an epoxy glass substrate detected hydrogen at even lower temperature (27°C) showing good sensitivity [61].

Electrospun polyamide nanofibrous membranes have been proved to be effective as glucose biosensors, displaying a sensitivity of 1.11 µA/mM and a detection limit of 2.5×10^{-6} M [62].

TiO₂ fibre mats obtained by electrospinning from a dimethyl formamide solution of poly (vinyl acetate) showed high sensitivity to NO₂ and H₂ with reversible response and a response times of the order of 1 min [63].

Sensors for humidity and KCl were obtained from electrospun poly (lactic acid)/polyaniline fibres deposited onto interdigitated microelectrodes. The sensitivity of the sensor depends on the poly (lactic acid)/polyaniline ratio in the blend [64].

Electrospun nanofibre sensors have found applications in the medical field as well. A sensor composed of a electrospun conductive nanofibres mat with polyacrylic acid grafted on its surface and a sensing element made of conducting polymer with covalently attached oligonucleotide probes is effective in determining the non-Hodgkin's lymphoma gene, with a detection limit of 1 aM (1×10^{-18} mol/L) and high selectivity [65].

Sensing electrodes for catechol detection were obtained by electrospinning a PANi solution with dispersed multiwall carbon nanotubes followed by the reduction of the amino groups and the addition of polyphenol oxidase on the nanofibres [66].

3.2. Optical electrospun sensors (colorimetric and fluorescent)

Optical sensors work by altering the colour or intensity of their fluorescence by the analyte [42]. These sensors, which are usually based on a technology that allows miniaturisation, low cost and in-situ usage, show high sensitivity and selective response towards various analytes [67].

Compared to other methods of obtaining nanofibres, electrospinning provides nanofibres for optical sensors, which are easy to manufacture and operate, relatively low-priced and with customizable properties, such as diameter, morphology and porosity [68].

There are three ways to obtain optical sensors by electrospinning: inserting chromophores into a transparent polymer without optical properties; using as substrate polymers having the ability to absorb/emit light and the functionalization of the electrospun polymer with active optical nanosystems [42].

Electrospun core-shell nanofibre with polymethyl methacrylate shell and a core made of phase change thermochromic material show thermochromic phase change comportment and can be used in detecting foreign materials in a specific environment [69].

A colorimetric sensor made of electrospun porphyrinated polyimide (PPI) nanofibrous membrane proved to be effective in hydrogen chloride detection, showing high sensitivity and fast response time. When exposed cu HCl vapours, the polymer changes its colour from pink to green, as a result of the protonation of the neutral porphyrin [70], and the colour intensity increases as the concentration of HCl is higher.

Colorimetric strip sensors to detect uranyl (UO_{22}^{+}) have been produced using electrospun cellulose acetate nanofibre mats that incorporated 2-(5-Bromo-2-pyridylazo)-5-(diethylamino) phenol. The colour in these sensors changes from yellow to purple in the presence of uranyl at pH 6.0, and the detection limit can extend to 50 ppb, with very low interference ability in the presence of other metal ions [71].

Colorimetric sensors have been produced incorporating gold nanoparticles into electrospun polystyrene nanofibres. These sensors, which do not employ any complex instrumentation, showed very good response towards oestrogenic compounds such as 17β -estradiol, with high sensitivity [72].

Optically active electrospun nanofibres can be used as sensors to detect heavy metals using colorimetry and fluorescence methods [68].

Colorimetric sensors based on nanofibres electrospun from a mixture of 10, 12-pentacosadiynoic acid and poly (ethylene oxide) and polyurethane can be used to detect *Escherichia coli* [73].

Membranes from electrospun polyamide 66/cobalt chloride proved to be effective in determining humidity, due to the colour change property of cobalt salt, which turns from blue to pink as the relative humidity increases over 12.4%. The sensor has high sensitivity, fast response time, good reproducibility and long-time stability [74].

Mats from electrospun cellulose acetate fibres doped with the chromogenic and fluorogenic amine-reactive blue dye Py-1 act as colorimetric sensors detecting biogenic amines [75].

A nanofibre-based fluorescent sensor for Ni^{2+} determination, even in the presence of other competing metal ions, was obtained by electrospinning pyridylazo-2-naphthol-poly(acrylic acid) polymer functionalized with 1,10-carbonyldiimidazole and 1,8-diazabicyclo[5.4.0]undec-7-ene [76]. The sensor showed high sensitivity and selectivity in its fluorescence towards Ni^{2+} , and the detection was simple, rapid and selective, without any need of additional sample handling phases.

Optical fluorescence sensors based on poly (acrylic acid)-poly (pyrenemethanol) polymers have proven effective for the detection of metal ions (Fe^{3+} and Hg^{2+}) and 2,4-dinitrotoluene (DNT), showing high sensitivity due to the high-volume surface ratio of the nanofibre membrane structures [77].

A fluorescence sensor based on a pentyptcene conjugated polymer was used for Copper (II) cation detection, with high selectivity and sensitivity [78].

A fluorescent film sensor made of electrospun nanofibres from a copolymer of vinyl naphthalimide and methyl methacrylate was used for Cu^{2+} detection, showing high sensitivity (a detection limit of $20 \times 10^{-6} \text{ M Cu}^{2+}$) [79].

A picric acid fluorescent sensor was obtained by electrospinning a solution of 5-(N-carbazole styryl)-1, 3-dimethyl-barbituric acid and polystyrene in dimethyl formaldehyde/tetrahydrofuran upon an amino-functionalized glass. The emission intensity of the fluorescent sensor is not significantly influenced by common interferents and the fluorescent nanofibres can be regenerated [80].

Fluorescence sensors for detecting traces of toluene vapours have been produced by embedding fluorescent CdTe quantum dots in electrospun polyvinyl alcohol nanofibres, showing sensitivity and fast time response [81].

A fluorescence sensor for Cu^{2+} sensing, which displays good sensitivity and selectivity, was obtained from electrospun rhodamine dye-doped polyester nanofibres [82].

A fluorescent sensor for the rapid detection of bacteria was made from electrospun membranes of a boronic acid copolymer, poly (4-vinylphenylboronic acid-co-2-(dimethylamino) ethyl methacrylate-co-n-butyl methacrylate), showing high affinity and towards both Gram-negative and Gram-positive bacteria [83].

Optical sensors based on electrospun polyvinyl alcohol nanofibres with embedded nanocomposite cerium oxide nanoparticles can be used to detect radicals in solutions. These nanocomposite nanoparticles are fluorescent with observable emission under near-UV excitation [84].

3.3. Other electrospun sensors

Besides the chemiresistive and optical sensors, other categories of electrospun nanofibre sensors have been developed.

An electrospun nanofibre piezoelectric sensor made of polyvinylidene fluoride (PVDF) nanofibre nonwoven webs proved to be effective in acoustic sensing for various applications [85].

A humidity sensor was obtained by depositing polyaniline composite nanofibres on surface acoustic wave resonator, demonstrating very high sensitivity, fast response and good sensing linearity [86].

Sensors based on gas adsorption on electrospun PAni fibre mats have been used to determine carbon dioxide. The process is reversible, and the adsorption process is fast enough to make quick detection possible [87].

Surface acoustic wave sensors are piezoresistive sensors [88]. Such a sensor based on creased polypyrrole film with electrospun polyvinyl alcohol nanowires as spacer showed high sensitivity, low detection limit and good stability [89]. Other piezoresistive sensors have been obtained from polyvinylidene fluoride and its blend with polyoctafluoropentyl acrylate [90].

4. Conclusions

Due to their small size and high surface-to-volume ratio, many sensors based on electrospun nanofibres have been used lately for analyte detection, proving great sensitivity and very rapid response time, all in elevated stability conditions. Nanotechnology is constantly developing towards new challenges in many areas including sensors. In future developments, it can be expected that the problems that affect the efficacy of electrospun nanofibre sensors are to be addressed and solved, beginning with a more accurate control of the process in order to achieve better control over the size and morphology of the nanofibres and continuing with increasing pore uniformity for more sensitivity and improving the reversibility of the sensors.

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