

## Review Article

# Advances in Nanostructured Polyamide-Based Chemical Sensors

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Nanofibers have shown great potential to contribute to the growing demand for point of care sensors and other kinds of sensors where they are utilized as supporting materials or used to interact with other materials through their functional groups. The choice of a polymer material to be used depends on whether it has the potential to give the properties of the desired sensor. Polyamide nanofibers have properties such as good chemical resistance, very good strength and hardness, high wear resistance, and high surface area to volume ratio, and they are porous, just to name a few. Furthermore, the amide groups in polyamide enable it to interact with other molecules via hydrogen bonding. These characteristics make polyamide nanofibers good candidates for sensor development. Polyamide nanofibers are used nowadays to make various types of sensors. This paper provides a review of some of the reports on the development of nanostructured polyamide-based chemical sensors in the last two decades (2000-2020).

## 1. Introduction

There is an increasing demand for quick, simple, affordable, and reliable sensing materials for application in various fields including environmental monitoring, medical diagnosis, food quality, and agricultural industries. Studies reporting on different types of materials as chemical sensors or biosensors have been conducted. Amongst these materials, polymer nanofibers are emerging as successful and are preferred as chemical sensing materials.

Polymer nanofibers can be synthesized using various methods including electrospinning, self-assembly, phase separation, and templating [1]. The electrospinning technique is the most commonly used owing to its simplicity and cost-effective instrumental setup. Electrospinning is a simple yet low-cost technique for fabricating ultrafine fibers with diameters ranging from several nm to several hundred nm using a high voltage power supply [2]. There are two approaches for electrospinning, viz., needle and needleless based. A basic needle electrospinning setup consists of a high voltage power supply, collector, and a needle nozzle

(Figure 1). During the electrospinning process, the high voltage power supply provides an electrostatic field to charge the surface of a polymer solution droplet and thus induce the ejection of the liquid jet through a spinneret.

The power is applied between a needle and a collector; both are electrically conducting (with opposite charges) separated at an optimum distance between the two. When the charged species in the polymer solution interact with the external electric field, the pendant droplet deforms into a conical structure called a Taylor cone thereby reaching a critical voltage. Once the repulsive electrostatic force overcomes the surface tension of the droplet, a fine charged jet of the polymer solution is ejected from the tip of the Taylor cone. The fast evaporating solvent will cause the polymer jet to solidify to ultrafine fibers while travelling to the collector on which they are collected [2, 4, 5].

Although simple and versatile, needle electrospinning is not widely applied on an industrial scale the main reason being its low productivity (typically less than 0.3 g/h) [6, 7]. The straightforward way to improve productivity is to employ a setup known as “multi-needle electrospinning” by increasing

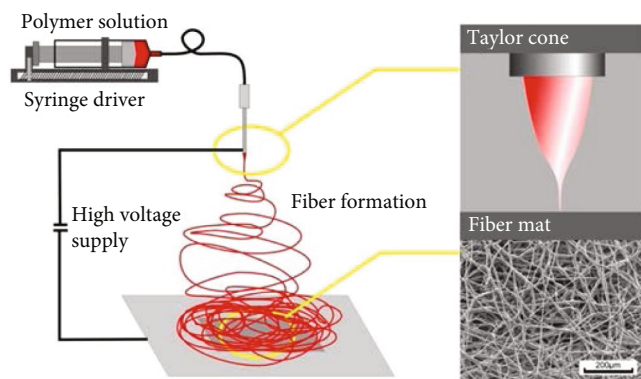


FIGURE 1: A schematic representation of a typical electrospinning setup [3].

the number of needles [8, 9]. However, the limitation of the multineedle electrospinning is that it requires a large working space to avoid interferences between adjacent solution jets, and cleaning systems are needed for an individual needle nozzle to ensure that clogging of the solution does not happen during the nanofiber forming process [7].

Nowadays, needleless electrospinning setups have been developed for large-scale nanofiber production from a compact space. In needleless electrospinning, nanofibers are electrospun directly from an open liquid surface resulting in the simultaneous formation of numerous jets from the needless fiber generator [7, 10]. Rotating spinnerets including balls, coils, cylinders, and cones are normally utilized in this approach [7, 11, 12]. Figure 2 depicts an example of needleless electrospinning. The main disadvantage of the needleless electrospinning strategy is the complexity of the electrospinning setup.

The morphology, structure, and property of the fibrous mats can be controlled by adjusting the properties of the polymer solution and process parameters such as the flow rate, solvents, solution viscosities, applied voltage, humidity, and temperature [14, 15]. The factors that affect the electrospinning process have been explored and reported extensively [16–21]. Fibers and the nonwoven webs generated through the electrospinning process have outstanding properties, which include high porosity, large surface area to volume ratio, and excellent pore interconnectivity [10, 22]. Due to these remarkable characteristics, electrospun fibers have been applied in various areas such as sensors [16, 23], filtration [24, 25], dye-sensitized solar cells [26], and tissue engineering [27]. Different sizes and morphologies of nanofibers are obtained by manipulating factors such as the viscosity of the polymer solution and the electrospinning conditions. This allows for application in numerous fields.

The nanoporous structures of electrospun nanofibers make it possible to encapsulate other nanomaterials that could be used as sensor probes. Blending electrospun nanofibers with other nanomaterials has shown tremendous advantages such as significantly decreasing the size of the nanofiber material. For example, Pant et al. managed to reduce the size of nylon 6 from 192 nm diameter to 14 nm by blending it with graphene oxide [28]; see Figure 3.

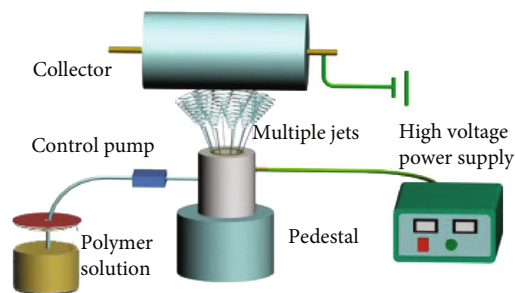


FIGURE 2: Schematic representation of needleless electrospinning using annular spinneret [13].

Sensors based on electrospun nanofibers were observed to have improved sensitivity because of the large surface area to volume ratio of electrospun nanofibers [29, 30]. As sensor materials, electrospun nanofibers have been combined with materials that have optical properties to be employed as colorimetric sensors, or materials that have electric conductivity in this case, they will be utilized as electrochemical sensors. The choice of a polymer depends on the type and characteristics of the sensor as well as the application of the end product. Various polymers including polyaniline [31, 32], polystyrene [33, 34], and polyamide (nylon 6/66) have been used. The latter is one of the popular synthetic polymers and has found several applications [35–37].

Nylon 6 has good mechanical strength, and is chemically stable. Moreover, the mechanical properties can be easily tuned to suit the desired application [38]. The porous nature of nylon 6/66 makes it a suitable host for other substances. Nylon 6/66 also form hydrogen bonding with other substances owing to the presence of the amine groups in its backbone [39, 40]. Taking advantage of the outstanding properties of this polymer, Nasybulin et al. [41] synthesized a material that is electroactive and has mechanical properties by combining polyaniline (an electrically stable polymer) and nylon 6. Merging nylon 6/66 with other substances allows for the fabrication of hybrid materials that have improved physical properties and widen the scope of their application. For example, a blend of nylon 6/66 and nanoclay has a higher Young's modulus and hardness compared to clean nylon 6/66 [42].

It is undoubtable that combining nanomaterials results in a composite material that has superior properties compared to the individual counterparts [43, 44]. Since it is not always possible to get one material that has all properties of the desired application, it is common for researchers to merge materials in the synthesis process to improve existing products or develop a new product that performs better than existing ones. These composite materials find applications in various fields such as in water treatment [45], drug delivery [46], and sensor development [47, 48].

These characteristics of nylon 6/66 have drawn the attention of many researchers to look into sensors that involve it either as a solid support or functionalized with other compounds and utilize the material as a sensor. This study will provide a review of polyamide-based sensors reported in the last two decades (2000 to 2020). Polyamide

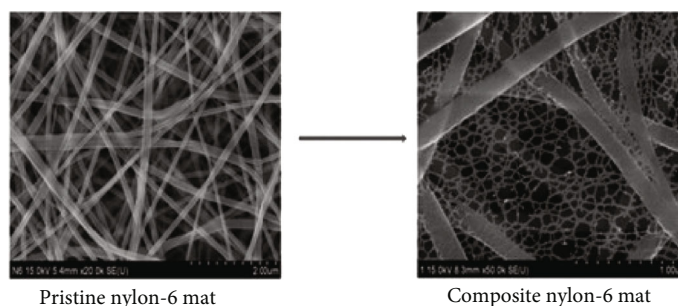


FIGURE 3: An image of nylon 6 mats (a) and an image of composite graphene oxide and nylon 6 mat [28].

6 and polyamide 66 are also known as nylon 6 and nylon 66, respectively. These names will be used interchangeably in this article. The study will focus on the synthesis of the sensor material and the application thereafter. The sections of this paper are divided in terms of the kind of sensor material with the focus on chemiresistor sensors, optical sensors, and electrochemical sensors.

## 2. Nylon 6/66 Sensors

The type of sensor produced from nylon 6/66 is determined by the material combined with the polymer as well as the mode of sensing of the composite material. This section discusses nylon 6/66-based optical, chemiresistors, and electrochemical sensors.

**2.1. Nylon 6/66-Based Chemiresistor Sensors.** A good vapour sensor should satisfy the following characteristics: high sensitivity and selectivity, low operating temperatures and temperature independence, fast response and recovery times, and stability in performance [49]. Most vapour or gas sensors are chemiresistors. A chemiresistor sensor changes its electrical resistance when there are changes in its surrounding environment. For the change to take place, there must be direct contact between the targeted analyte and the sensor material. Materials such as metal oxides [50], nanoparticles [51], conductive polymers [52], carbon nanotubes [53, 54], and graphene [55] have been employed to make chemiresistor sensors. These materials can be combined with nylon 6 or nylon 66 to give sensors with outstanding properties. Interaction of the sensing material can be via a covalent bond and hydrogen bonding through the functional groups of nylon, or the materials can be simply encapsulated into the pores of the polymer. An illustration of the chemiresistor sensor principle is shown in Figure 4.

Applying the electrospinning method, Kang et al. prepared a hybrid 1-butyl-3-methylimidazolium hexafluorophosphate (BMIMPF<sub>6</sub>)/nylon 6,6 nanofiber sensor. In this case, the authors noted that there were no covalent bonds formed between nylon 66 functional groups and BMIMPF<sub>6</sub>. The BMIM<sup>+</sup> and PF<sub>6</sub><sup>-</sup> were freely mobilized in the composite material and acted as charge carriers. This resulted in a decrease of resistance with increasing concentration of BMIMPF<sub>6</sub>. When the sensor was tested in four organic vapours, namely, ethanol, methanol, tetrahydrofuran, and

acetone at room temperature, good sensing properties were observed. Exposing the sensor to organic vapours resulted in an immediate decrease of resistance, which increased rapidly on the removal of the vapours. The electrical conductivity of the nanofiber composite was found to be  $1.23 \times 10^8 \Omega\text{sq}^{-1}$  [57].

In another study, nylon 6 was incorporated with graphene oxide to fabricate a sensor for trimethylamine vapour. The composite material was prepared by first synthesizing graphene oxide; the graphene oxide was then reduced onto nylon 6 membranes using a vacuum-assisted self-assembly method. Characterization of the graphene/nylon 6 nanocomposite showed that a noncovalent bond formed between the graphene oxide sheet and the nylon 6 sheet. The limit of detection for the trimethylamine sensor was calculated to be 0.39 mg/L. Weight by weight percentage ratio of 1.92% graphene/nylon 6 composite was found to have a resistance of  $3.839 \times 10^3 \Omega\text{sq}^{-1}$  [58].

The presence of spider-net nanofibers in electrospun nylon 6 which normally have diameters less than 10 nm can improve sensing because of increased free surface area. This phenomenon was observed in a study done by Hosseini-mardi and coworkers [59] where a humidity sensor based on nylon 6/titanium dioxide nanocomposite was fabricated. Electrospun nanofiber mats that had spiderweb-like nanofibers showed better sensing properties compared to those that were without. As water diffused into the nylon 6 nanofiber, hydrogen bonds formed between the water molecules and the C–H, C=O, N–H, and N: groups found in nylon 6. Moreover, an increase in relative humidity escalated the diffusion of water molecules to the nylon 6 network. The water molecules were ionized to H<sup>+</sup> and OH<sup>-</sup>, thereby becoming the source of proton and increased the conductivity of the nanofiber mat. There are more reports on humidity sensors [60].

Biological materials have also been incorporated with polyamide to produce sensors. For instance, Duarte et al. synthesized sensors using polyamide, (Z) octadec-9-enedioic acid, and  $\beta$ -cyclodextrin [60]. They used various molecules for sensitivity and selectivity. The sensors were used to detect volatile organic compounds, and the diffusion/desorption process was found to be reversible for all sensors.

The above sensors showed selectivity towards the analytes of interest. However, they all require a piece of

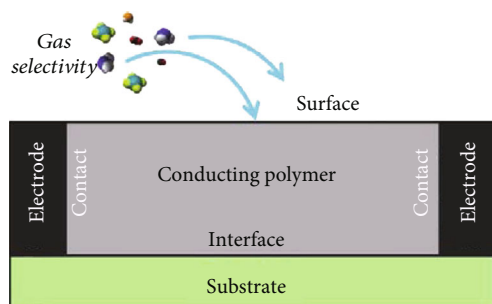


FIGURE 4: Schematic representation of the chemiresistor sensor principle based on chemically sensitive conducting polymer for selective detection of chemical sensing materials [56].

equipment to measure resistance. Certain conditions may hinder their practical applications. In such cases, optical sensors would be an ideal solution. The next section explains and gives more examples of nylon 6 or nylon 66-based optical sensors. The characteristics of some of the polyamide 66-based chemiresistor sensors are summarized in Table 1.

**2.2. Nylon 6/66-Based Optical Sensors.** Nylon 6/66 on its own does not have optical properties. To synthesize an optical sensor using nylon 6, one would need to combine it with optically active materials such as metal nanoparticles (for example, gold (Au) and silver (Ag) nanoparticles), dyes (e.g., Prussian blue), and conjugated polymers such as polydiacetylene. Optical sensors composed of these optical active materials have been investigated for application in fields including clinical diagnosis and environmental samples. Some applications require that the optically active material be encapsulated into the polymer, while others require that they decorate the surface of nylon nanofiber (Figure 5).

For example, Li et al. fabricated a selective, facile, and sensitive optical sensor strip for  $Pb^{2+}$  with a detection limit of  $0.2 \mu M$  [62]. The sensor was based on an electrospun (nylon 6)/nitrocellulose nanofiber/nanonet membranes implanted with bovine serum decorated AuNPs. The bovine decorated AuNPs were stable, owing to the extremely large and specific surface areas and high porosity of the membranes. When the sensor strip was introduced to  $Pb^{2+}$  liquid, visual color change of the strips from deep pink to white was observed.

With my colleagues, we have developed a colorimetric probe for dopamine detection using unfunctionalized AuNP hosted in nylon 6 fibers [63]. Due to the strong mechanical strength of nylon 6, the nanofibers could be cut to small strips and introduced to aqueous samples of dopamine (the targeted analyte) and interfering species including norepinephrine, epinephrine, ascorbic acid, and uric acid. The purple nanofiber turns navy blue/black in the presence of dopamine and was not responsive to the interfering species.

The effectiveness of gold nanoparticles in detecting pharmaceutical analytes is notable. These nanoparticles are used in the formulation of sensors for various analytes. For example, Abedalwafa et al. [64] produced a colorimetric sensor for the determination of metronidazole utilizing gold nanoparticles capped with melamine. The sensor was sensitive

towards the targeted analyte resulting in a low limit of detection of 2 nM.

Researchers have also explored the possibility of using metal alloys for optical sensing. Silver-copper (Ag-Cu) alloy nanoparticles stabilized with glutathione and hosted in electrospun nylon 6 nanofibers were used to detect nickel (II) [65]. The sensor selectively detected  $Ni^{2+}$  in the presence of selected transition metals, and its sensitivity effect towards other alkali and alkali earth metal ions was found to be negligible.

In another study, a colorimetric sensor based on copper-gold alloy nanoparticles supported in electrospun nylon 6 nanofiber for detection of ascorbic acid (AA) was described [66]. The white nanofibers change color from white to blue due to the aggregation of the alloy nanoparticles showing selectivity for AA in the presence of interfering species at pH 2–7. In another study, polyaniline was blended with polyamide 66 to produce nanofibers with spiderweb-like structures [67]. The authors noted a color change from brilliant brown to green when the sensor material was introduced to L-ascorbic acid.

Other researchers investigated and reported on a study about impregnating metal salts like gold salt into nylon 6 nanofibers for sensing purposes [68]. On the interaction of the sensor material with the reducing sugars in alkaline media, the gold salts formed AuNPs thereby resulting in a color change from white to purple. Nylon 6 has also been utilized as a supporting material for biological thiols, and the material was used to detect chloropicrin in the air [69]. A colorimetric response was noted on the addition of the Ellman's reagent.

A nylon electrospun nylon 66/cobalt chloride nanofiber membrane has also been studied for optical detection of relative humidity [70]. In this case, cobalt chloride was applied as the color indicator. Nylon 66, acting as a solid support, also played an important role in providing an increased number of binding sites. The results obtained indicated that the fabricated nanofiber membrane has the potential to be applied in a visual hygrometer. The effectiveness of the sensor in detecting relative humidity is shown in Figure 6.

Giachet et al. fabricated two types of sensor materials for optical detection of pH [71]. Cotton and polyamide 66 fabrics were dyed with curcuma powder and used as optical sensor materials. Both dyed materials displayed a yellow color which changed to red on contact with basic solutions. The polyamide-based sensor was observed to change color in a wider pH range compared to the cotton-based sensor. The color change was not affected by the ionic strength of the solutions. Utilizing a UV-visible spectrometer, the authors were able to assess the reversibility of the color change of the fabrics. Other textile optical sensors have been reported [72].

The role of dyes in colorimetric methods is notable. A dye can be combined with a polymer material such as polyamide 6/66 and used as a sensor material. Two or more dyes can also be blended to achieve the same. For instance, methyl yellow was combined with nylon 6 to produce a sensor for formaldehyde gas (Figure 7). The sensor could detect formaldehyde with insignificant interference from other

TABLE 1: Features of polyamide 66-based electrochemical sensors.

Sensor material	Analyte	Sensor performance	Ref.
1-Butyl-3-methylimidazolium hexafluoro-phosphate (BMIMPF <sub>6</sub> )/nylon 6,6 nanofiber	Ethanol, methanol, tetrahydrofuran, and acetone	EC = $1.23 \times 10^8 \Omega\text{sq}^{-1}$	[57]
Nylon 6/graphene oxide	Trimethylamine	LOD = 0.39 mg/L Resistance = $3.839 \times 10^3 \Omega\text{sq}^{-1}$	[58]
Nylon 6/titanium dioxide	Humidity	Sensing improved by presence of spiderweb-like nanofibers	[59]
Polyamide, (Z) octadec-9-enedioic acid, and $\beta$ -cyclodextrin	Volatile organic compounds	Diffusion/desorption was reversible	[60]

\*EC: electrical conductivity; \*\*LOD: limit of detection.

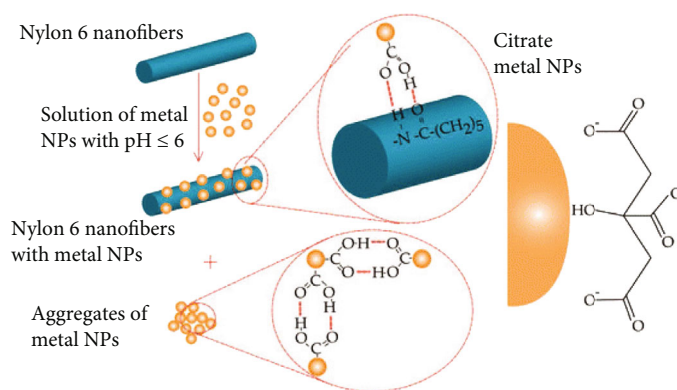


FIGURE 5: Postulated mechanism of pH-induced assembly of metal nanoparticles on the surface of nylon 6 nanofibers [61]. Reprinted with permission from ref. [61]. Copyright (2008) American Chemical Society.

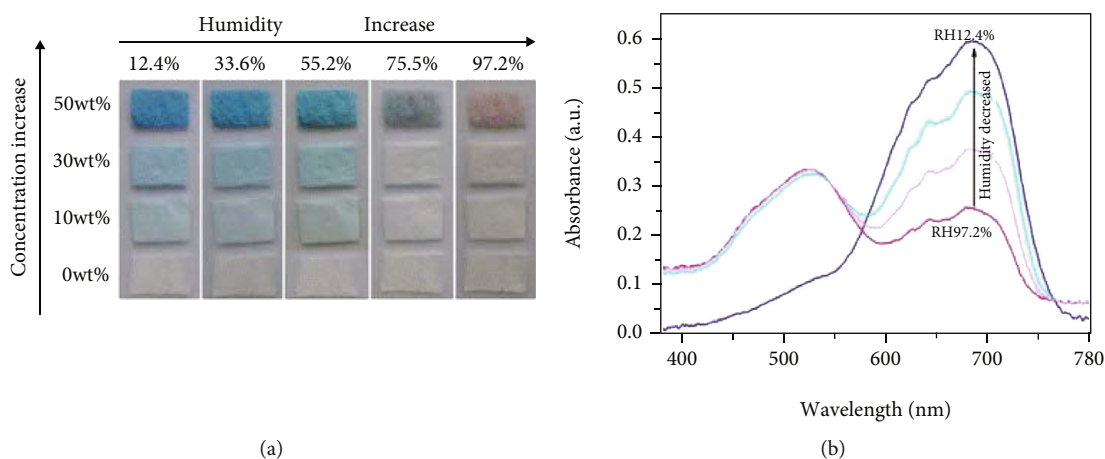


FIGURE 6: (a) The photographs of PA66/CoCl<sub>2</sub>·6H<sub>2</sub>O NFMs containing different concentrations of CoCl<sub>2</sub>·6H<sub>2</sub>O: 0, 10, 30, and 50 wt% in different RH. (b) Humidity dependence of visible absorption spectra (380–780 nm) of PA66/CoCl<sub>2</sub>·6H<sub>2</sub>O (50 wt%) [70]. Reprinted from [70].

volatile compounds [73]. A blend of meta-aramid and dye 3 was impregnated onto electrospun nylon 6 nanofibers and used as a sensor for ammonia gas. The sensor responded very fast to ammonia (10 s) at room temperature. Moreover, the authors reported that after washing for 30 min, the sensing material retained its character and reversibility [74]. Another sensor for formaldehyde was reported by Jung

et al. [75]. The sensor material comprises of bromophenol blue encapsulated into nylon sheets. On exposure to formaldehyde, the sensor changes color from bluish green to yellow; this change was not observed on commonly interfering species.

The properties of some of the polyamide 6/66-based optical sensors are summarized in Table 2.

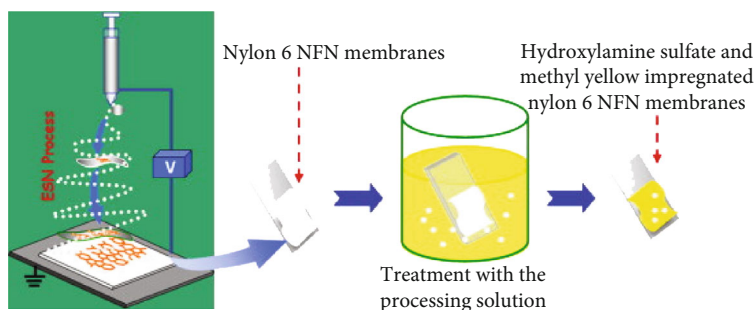


FIGURE 7: Schematic representation of the production process of a formaldehyde gas sensor [73].

TABLE 2: Features of polyamide 6/66-based optical sensors.

Sensitive material	Method	Analyte	Sensor performance	Ref.
Bovine serum albumin decorated AuNPs	Colorimetric	Pb <sup>2+</sup>	LOD = 0.2 $\mu$ M	[62]
AuNPs	Colorimetric	Dopamine	LOD = $5 \times 10^{-7}$ M Linear range = $5 \times 10^{-7}$ – $5 \times 10^4$ M	[63]
Melamine-functionalized gold nanoparticles assembled on polyamide nanofiber membranes	Colorimetric spectrophotometric	Metronidazole	LOD = 2 nM Linear range = 2 to 100 nM	[64]
Ag-Cu alloy nanoparticles	Colorimetric	Ni <sup>2+</sup>	LOD = 5.8 $\mu$ g/mL	[65]
Cu-Au alloy nanoparticles	Colorimetric	Ascorbic acid	LOD = $1.76 \times 10^{-2}$ mg/L Linear range = $1.76 \times 10^{-2}$ – $1.76 \times 10^5$ mg/L	[66]
Polyaniline	Colorimetric	l-Ascorbic acid	LOD = 50 ppm	[67]
Au salt	Colorimetric	Reducing sugars	—	[68]
Cobalt chloride	Colorimetric	Humidity	RH = 12.4 – 97.2%	[70]
Curcuma powder	Colorimetric UV-vis spectrophotometer	Textile	—	[71]
Methyl yellow	Colorimetric	Formaldehyde	Good reproducibility under cyclic sensing	[73]
Meta-aramid/dye 3	Colorimetric	Ammonia gas	Linear range = 1 – 10 ppm	[74]
Bromophenol blue (BPB) and nylon sheet	Colorimetric	Formaldehyde gas	LOD = 50 ppm	[75]
Biological thiols and Elleman's reagent	Colorimetric	Chloropicrin vapour	Linear range = 0.00 – 1.50 mM	[69]
4-(p-Nitrobenzyl)pyridine	Colorimetric	1,3-Dichloropropene, methyl iodide, and methyl bromide	LOD = 0.2, 0.1, and 0.1 ppm, respectively	[76]

**2.3. Nylon 6/66-Based Electrochemical Sensors.** Examples of composite materials involving polyamide 6/66 for electrochemical sensing reported in the last two decades are discussed in this section.

In 2015, Andre et al. [77] modified fluorine doped tin oxide electrodes with zinc oxide decorated polyamide/polyaniline nanofiber that was then utilized as a sensor for hydrazine. According to the authors, overall improvement in terms of the electroactive area and electron transfer kinetics was noted. The sensor was effective in detecting hydrazine resulting in a limit of detection of 0.35  $\mu$ mol.

Hong and coworkers reported on an ammonia gas sensor based on a composite polyaniline-nylon 6 fabric [78]. Unlike other sensors that use electrospun nanofibers of the

nylon 6, these authors used nylon 6 fabrics. The composite material showed excellent sensitivity and fast response towards ammonia gas when it was doped with monocarboxylic acids.

Tsai et al. prepared an electrochemical sensor for the detection of ascorbic acid utilizing gold/aniline-pentamer-based electroactive polyamide composite [79]. The improved electrochemical response of the composite sensor material was demonstrated using cyclic voltammetry. The limit of detection was found to be 2.75  $\mu$ M. In another study, aniline-pentamer-based electroactive polyamide was modified on the surface of the carbon paste electrode and employed for detecting ascorbic acid [80]. The detection range for the ascorbic acid sensor was 0.005–0.7 mM, and the detection limit was 0.005 mM.

TABLE 3: Features of polyamide 66-based electrochemical sensors.

Sensor material	Method	Analyte	Sensor performance	Ref.
Fluorine doped tin oxide electrodes with zinc oxide decorated polyamide/polyaniline nanofiber	Impedance	Hydrazine	LOD = 0.35 $\mu\text{mol}$	[77]
Polyaniline-nylon 6 fabric doped with monocarboxylic acids	Conductometric	Ammonia gas	Reasonable sensitivity and reversibility for $\text{NH}_3$	[78]
Gold/aniline-pentamer-based electroactive polyamide composite	Voltammetric	Ascorbic acid	LOD = 2.75 $\mu\text{M}$ Linear range = 0.05 – 0.75 mM	[79]
Aniline-pentamer-based electroactive polyamide modified on the surface of the carbon paste electrode	Voltammetric	Ascorbic acid	LOD = 0.005 mM Linear range = 0.005 – 0.7 mM	[80]
Polyamide/polypyrrole coated with reduced graphene oxide	Impedance voltammetric	Malathion pesticide	LOD = 0.8 ng/mL	[81]
Indium tin oxide electrode modified with electrospun polyamide 6/poly(allylamine hydrochloride) (PA6/PAH) nanofibers functionalized with multiwalled carbon nanotubes	Differential pulse voltammetry	Dopamine	LOD = 0.15 $\mu\text{mol/L}$ Linear range = 1 – 70 $\mu\text{mol/L}$	[85]
Polyamide amine-Au nanoparticle (PMA-Au) nanocomposites and horseradish oxidase-graphene antibodies (HRP-G-Abs)	Voltammetric	Salbutamol	Linear range = 0.1 ng/mL to 1 $\mu\text{g/mL}$ LOD = 0.03 ng/mL	[86]
Nylon 66 film deposited onto screen printed electrodes	Differential pulse cathodic stripping voltammetry	Chlorothalonil	LOD = $1.53 \times 10^{-8}$ M Linear range = 0.1 – $2.8 \times 10^{-6}$ M	[83]
Nylon 66 graphite HB pencil electrode	Differential pulse cathodic stripping voltammetry	Chlorothalonil	LOD = $0.94 \times 10^{-8}$ M Linear range = 1 – $26 \times 10^{-7}$ M	[84]
Fluorine doped tin oxide electrode modified with polyamide 6/chitosan electrospun nanofibers and gold nanoparticles	Voltammetric	$\text{Cd}^{2+}$	LOD = 0.88 $\mu\text{g/L}$ Linear range = 25 to 75 $\mu\text{g/L}$	

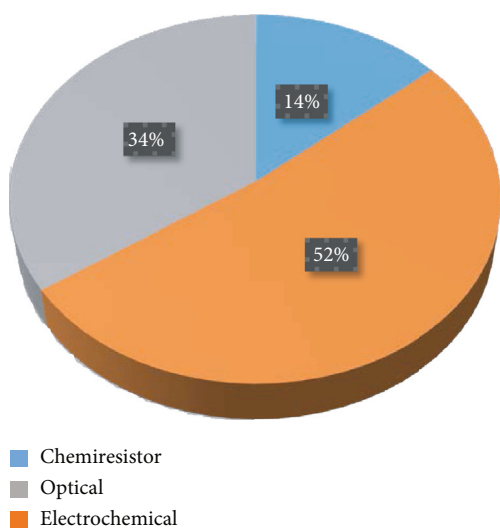


FIGURE 8: Percentage of N 6/66-based sensor respect to the number of papers.

Good conductivity and high selectivity are crucial for an electrochemical sensor. Thus, combining materials that can produce a composite with the two properties is important. Migliorini et al. [81] developed an electrochemical sensor for the organophosphorus pesticide, malathion using a blend of polyamide 6, and polypyrrole nanofiber surface modified

with graphene oxide. Reduction of graphene oxide was achieved using chemical and electrochemical strategies. The former yielded a material that exhibited higher electrical conductivity. When applied to detect malathion, it showed high sensitivity, and a limit of detection of 0.8 ng/mL ( $S/N = 3$ ) was recorded. Utilizing the traditional fluorine doped tin electrodes, Migliorini and coworkers further produced a sensor for cadmium (II) by decorating a composite of nylon 6 and chitosan nanofiber with gold nanoparticles. The nanofiber material was used to modify the electrode and assessed its sensor properties by square voltammetry. The sensor could detect cadmium with negligible interference from mercury (II), lead (II), and copper (II) [82].

Another electrochemical sensor for a pesticide based on polyamide 66 was reported by Thanalenchumi and coworkers [83]. The authors modified a screen-printed electrode with a film of nylon 66. A significant increase in the peak currents of the nylon 66 modified material was observed compared to the unmodified screen-printed electrode. The sensor showed good selectivity toward chlorothalonil in real water samples. The electrochemical studies were carried out using differential pulse cathodic stripping voltammetry.

In the quest of eliminating toxic electrode materials such as mercury-based electrodes, researchers are looking into the use of nylon as an alternative. An example of such was reported by Thanalechumi et al. [84] where a nylon 66

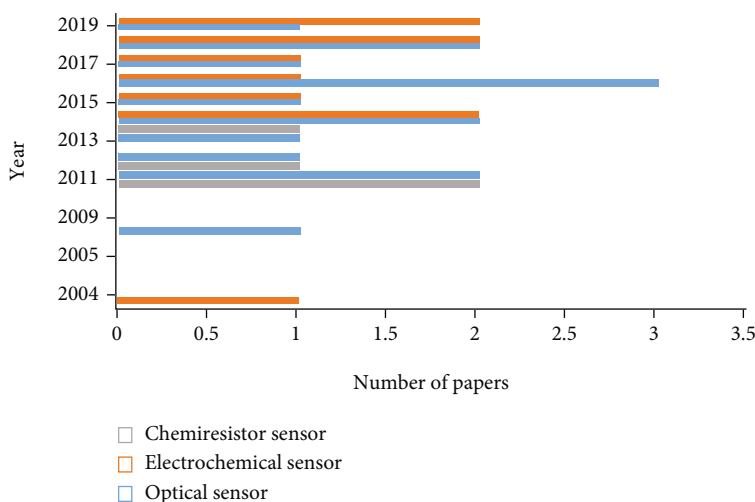


FIGURE 9: The number of N 6/66-based sensors published per year.

modified graphene HB pencil electrode was utilized as an electrochemical sensor to determine chlorothalonil in water. Using the sensor, a low detection limit ( $0.94 \times 10^{-8}$  M) and a linear range from 1 to  $26 \times 10^{-7}$  M were obtained. It is noteworthy that the conductivity properties of the reported sensor were superior compared to the mercury-based electrode. The characteristics of some of the polyamide 6/66-based electrochemical sensors are summarized in Table 3.

Figure 8 shows the percentage of the chemiresistor, optical, and electrochemical sensor papers discussed in this paper. Chemiresistor sensors amount to only 14% of all N 6/66-based sensors reported. This could be due to the challenge with the materials that are normally used in gas sensing such as metal oxides including ZnO-Ca [87], Ga-ZnO [88], Ca-V-ZnO, and  $V_2O_5$ -TiO<sub>2</sub>. These materials showed outstanding performance in detecting gases. However, the operating temperatures are high, ranging from 200 to 450°C. This is a problem since nylon melts at 265°C but can only retain its properties at temperatures that are  $\leq 150^\circ\text{C}$ . More studies on sensors combining N 6/66 with graphene and dyes such as porphyrins can improve the number of reports in this area.

The percentage of optical sensors is the highest (52%). This could be because these types of sensors maximize the outstanding properties of N 6/66 thereby using its porous structure to host sensitive materials such as metal nanoparticles, dyes, and conductive polymers. The ability to form hydrogen bonding makes it ideal in sensing and widens the scope of application in terms of the analytes that can be determined. Hence, the greater number of publications was compared to chemiresistor sensors and electrochemical sensors. The challenge with some of the optical sensors discussed herein is relying on eyeball detection. If the analyst is color-blind, this is a major drawback. The continual use and development of image processing software like ImageJ will turn things around in this area. For both optical and electrochemical sensing, incorporating biological compounds with N 6/66 will produce good materials and improve the number of publications.

Figure 9 depicts the number of articles published on N 6/66-based sensors. Only one paper was published between the years 2000 and 2008. Since 2011, there is at least one publication per year in this area with the highest number of articles reported in 2017. It is undoubtedly that there is still a lot to be done to bridge the knowledge gap that is still big.

As researchers are trying out the feasibility of various materials, it will be interesting to see studies combining polydiacetylene (PDA) and N 6/66. Such a sensor is expected to have dual properties; PDA has conductive properties and has been used as a chromophore in developing colorimetric probes. Thus, it is possible to produce an optical sensor and electrochemical sensors from the combination.

### 3. Conclusions

The use of polyamide 6/66 in the development of chemiresistor sensors, optical sensors, and electrochemical sensors is at its infant stage. The limited number of reports especially between 2000 and 2010 attests to this. It is noteworthy considering that from the past decade at least three papers are published annually in this area owing to the increasing knowledge and advances in technology. The characteristics of polyamide 6/66 make it a good candidate for solid-state-based sensors. It is expected that more solid-state-based polyamide sensors will be developed to address the challenges of liquid state sensors. Moreover, the new image processing techniques will strengthen the real-time detection of analytes. It would be interesting to see the application of nylon 6/66 in gas/humidity sensors moving from just focusing on chemiresistors; colorimetric sensing of gases using nylon is an area that still needs to be explored.

### Consent

No consent was necessary.



## Conflicts of Interest

The author declares no conflict of interest.

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