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RESEARCH ARTICLE - CHEMISTRY

Electrospun Polyvinyl Alcohol Nanofibers Containing Titanium Dioxide for Gas Sensor Applications

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Abstract

Electrospun nanofibers for gas sensor application were effectively prepared from polyvinyl alcohol and pluronic solution with different percentages of titanium dioxide (TiO_2) nanoparticles. Nanofibers membrane was subject to detailed analysis by scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FT-IR), and thermal gravimetric analysis (TGA). Adding TiO_2 nanoparticles to the blended solution increased nanofibers diameters from 280 ± 20 to 310 ± 30 nm. The gas sensor response of TiO_2 nanofibers (as a function of temperature) was estimated toward liquid petroleum gas (LPG), CO_2, and O_2 and compared with pure nanofibers. The maximum response value (100%) was obtained for LPG at 160 °C with TiO_2 nanofibers (0.01%). These results show promising gas sensing characteristics (such as lower operating temperatures and sufficient gas responses) for those nanofibers materials.

Keywords Nanofibers · Gas sensors · Polyvinyl alcohol · Titanium dioxide · Liquid petroleum gas

1 Introduction

Over the last decades, there has been growing require for accurate, portable, inexpensive, and reliable gas sensors that can differentiate among very low gas concentrations. Typically, gases of interest include NO_2, CO, NH_4, NO, CO_2, SO_2, and other hydrocarbons. These gases can be deleterious to human health if present over a definite concentration [1]. Gas sensors are beginning to appear as a very valuable in several applications such as industrial manufacturing units, environment monitoring, human health, defense, safety, and agriculture [2,3]. The most advantages, features of the gas sensor would be small size, a low-cost device, having low power consumption and high sensitivity [4]. By comparison with metal oxides, conducting polymers present several features such as small response time, room temperature operation, high sensitivity, the possibility of tuning together physical and chemical properties [5]. Pure forms for conducting polymers have a slightly low conductivity as a common feature which easily can be improved by a doping procedure. In overall, removing of some electrons (by electrochemical or chemical oxidation) leaves the polymer backbone charged and the cation radical performances as a charge carrier [6]. The sensing mechanisms have usually depended on one of two ways, the first one is the impact of nanoparticles on the electrical properties of the nanocomposites and the second is the optical features change of the contained surface plasmon resonance of the nanoparticles onto interface with the objective analyte (detected gas) [7].

Electrospinning is an effective way to yield ultra-thin fibers fluctuating in diameters from less than 10 nm to various micrometers via electrostatic stretching [8,9]. This method

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has fascinated increasing attention lately, and approximately over one hundred diverse polymers have been effectively spun into ultra-fine fibers using this technique [10].

Electrospun fibers with a diversity of materials, fine structures, controllable membrane thickness, and large specific surface are likely to be the best candidate as the structure for sensing materials. So far, many experiments are carried out to prepare ultrasensitive gas sensors to detect vapors like NH_3, H_2S, and CO [11–13]. Electrospun fibers with conducting polymer composites, semiconductors, and polyelectrolyte components are successfully applied as a gas sensing interfaces with different fiber arrangements at elevated operating or room temperature [11,14].

Polyvinyl alcohol (PVA) is one of the most common synthetic polymer hydrogels with dopant-dependent electrical, high dielectric strength, optical properties, and good charge storage capacity [15]. Due to its good biocompatibility, it has been utilized in several advanced biomedical applications, e.g., drug delivery system [16,17], contact lenses [18], and artificial organs [19]. PVA is a bad electrical conductor; it turns into conductive by doping with definite dopants. The conductivity of PVA arises as a result of a great physical interaction rate among polymer chains, through hydrogen bonding among the dopant and the hydroxyl groups, and is determined mostly by amorphous area properties [20].

Pluronic copolymers consist of hydrophobic poly (propylene oxide) (PPO) and hydrophilic poly (ethylene oxide) (PEO) arranged in a triblock structure (ABA): PEO–PPO–PEO [21]. Beyond their critical micelle concentration (CMC) in aqueous solutions, these two copolymers can self-assemble into nanomicelles with size varies from 10 to 100 nm [22]. Pluronic has well-known industrial applications as thickening, wetting, coating, emulsifying, solubilizing, stabilizing, foaming agents, dispersing, and lubricating [23].

Hyperbranched polyethyleneimine (PEI) is a polyelectrolyte that is used for many applications, such as in the gene delivery treatment [24], and in the catalyst supports improvement [25]. Nevertheless, its uses in biomedical research are boundless due to its cytotoxicity [26]. So, changes in the PEI polymeric spine that reduce its positive charge might be valuable in decreasing the polymer toxicity [27].

Titanium oxide (TiO_2) is related to the family of transition-metal oxide compounds, and it has good gas sensing properties [28] and biocompatibility [29]. Many studies have been described over the last decades involving the preparation of powder TiO_2 for gas sensor applications by deposition on polymer films. But this process depends on the polymer surface charge, chelating/functional groups, and the size of the TiO_2 nanoparticles [30].

This work aims to prepare a small size, low-cost, and high sensitivity nanofibers to be used as a gas sensor device. So, electrospinning technique was applied to prepare those nanofibers using polyvinyl alcohol and pluronic polymers containing different TiO_2 concentrations.

2 Materials and Methods

2.1 Materials

Polyvinyl alcohol (PVA) (Mw = 72, 000) was supplied by Fluka, Switzerland, Titanium (IV) isopropoxide (TTIP); Polyethyleneimine (PEI) (low Mw, 50% solution in water, Mn 1.800 (GPC); typical Mw 2.000), isopropanol, pluronic® F-127 (Plur- F127) were purchased from Sigma-Aldrich Chemicals Ltd. (Germany). The distilled water was used as the solvent to prepare the samples. All the chemicals were analytical graded and used without any additional purification.

2.2 Electrospinning process

The nanofibers mate was prepared in our previous work [17]. Briefly, PVA powder (7.5% w/v) was dissolved in distilled water and stirred (300 rpm) at 50°C for 4h. Then, different pluronic concentrations (0.5, 1, and 2 %) were introduced to PVA solution and stirred for 60 min until the solution became homogeneous. Next, 200 µl of PEI solution was added to the above solution. Finally, different concentrations of TiO_2 NPs (0.01, 0.03, and 0.05 % w/v) with size nearly ∼ 6 (±10) nm—prepared as described in previous work [17]—were added to the polymer blend and stirring overnight to get the final blend of PVA/pluronic/PEI/TiO_2 NPs (Table 1). The electrospinning method was done at room temperature, and the used voltage was 23 KV. The needle tip-to-collector distance was 17 cm, and the used flow rate was 1.3 ml/h. Dry nanofibers have been collected (from the aluminum foil) and kept at room temperature until use.

2.3 Materials characterization

Fourier transform infrared (FT-IR) spectra of PVAB and PVAT nanofibers were recorded on a Fourier transform infrared spectrophotometer (Shimadzu FT-IR-8400S, Kyoto, Japan).

Table 1 Preparation and compositions of the nanofibers (PVA = 7.5%; pluronic = 0.5%; PEI = 200µl)

Ingredient	Designation			
	PVAB	PVAT1	PVAT3	PVAT5
TiO_2(%)	0	0.01	0.03	0.05

The surface morphology of nanofibers was examined by a Joel 6360LA scanning electron microscope (JEOL Ltd., Tokyo, Japan) operated at 5/10 kv voltage acceleration. Nanofibers specimen was fixed on stainless steel stubs with double face tape; layer of gold (10–15 nm) was sputtered on the samples by JFC-1100E sputter (JOEL Ltd., Tokyo, Japan).

Thermal gravimetric analysis (TGA) is a technique in which the sample weight is observed against temperature or time, whereas the sample temperature, in a specific atmosphere, is programmed [31]. The experiments have been done using a Shimadzu thermal analyzer 50 (Japan). The data were acquired in the range 20–600°C under a nitrogen atmosphere with 20 ml/min flow rate and at 10°C /min heating rate.

2.4 Nanofibers electrical properties as a gas sensor

Platinum (Pt) contact electrodes were deposited on the surface of nanofibers using the sputtering machine (Deposition System Model Hummer 8.1 USA, a Turbo Sputtering RF & DC Power Supplies (gas/water 5 psi, 220 V, 30 A, 60 Hz)) (t = 10 min, P = 100 WRF). After that, the nanofibers were introduced inside the homemade gas chamber to obtain the electrical characteristics. Several gases (CO_2, O_2, and LPG) were passed individually inside the gas chamber. The nanofibers resistivity was measured, and the resultant response was plotted against gas temperature. The change ratio in the nanofibers resistance (response, S) under a definite gas level (R_a) relative to the resistance in the gas absence (R_g) was then calculated by the following Eq. (1) [32].

$$S = [R_a/R_g] \times 100 \quad (1)$$

3 Results and Discussion

3.1 FT-IR Analysis

Figure 1 shows FT-IR spectra of PVAB and PVAT nanofibers. Attribution of lines in the IR-spectrum of PVAB nanofiber, a broad peak at 3300 cm^{-1} was referring to OH and NH stretching vibration of hydroxyl and amine groups of PVA and PEI, respectively [33]. The peak at 2940 cm^{-1} was associated with CH_2 and CH stretching vibration in PVA and pluronic, respectively [34]. The peak at 1735 cm^{-1} referring to the H–O–H bonding indicates the existence of water molecules in the blending [35]. The peak at 1450 cm^{-1} has been ascribed to frequencies of NH (bending) of PEI. The peak at around 1135 cm^{-1} referring to C–O–C stretching corresponding to the ether of PEO and PPO blocks which confirms the uniform distribution of pluronic additive in the PVA nanofibers [31]. On the other hand, the IR-spectrum of

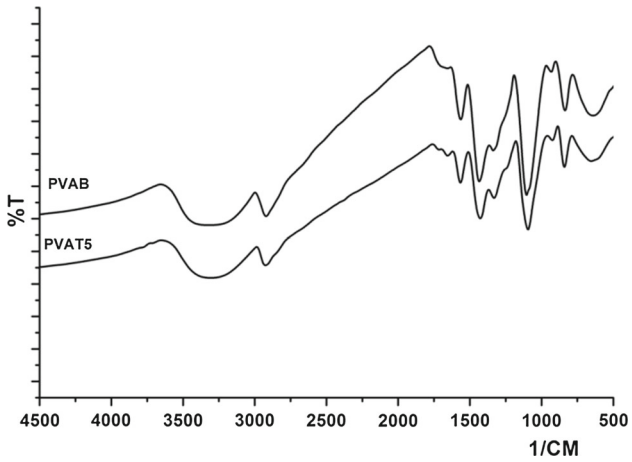

Fig. 1 FT-IR spectra of PVAB and PVAT (PVAB, TiO_2 = 0 %; PVAT5, TiO_2 = 0.05%)

Table 2 TGA phases (°C) for PVAB and PVAT nanofibers

Nanofiber	TGA phases (°C)			
	1	2	3	4
PVAB	25.9–78.1	78.1–199.3	199.3–340.5	340.5–598.8
PVAT1	26.9–70.6	70.6–215.5	215.5–317.8	317.8–599.6
PVAT3	26.4–76.1	76.1–202.3	202.3–320.7	320.7–599.6
PVAT5	26.4–76.1	70.7–201.9	201.9–330.5	330.5–598.9

(PVAB, TiO_2 = 0%; PVAT1, TiO_2 = 0.01%; PVAT3, TiO_2 = 0.03%; PVAT5, TiO_2 = 0.05%)

PVAT shows no difference than IR-spectrum of PVAB, which means the addition of TiO_2 has no effect on the IR-spectrum of PVAB and these results agree with our previously reported results [17].

3.2 Thermal Properties

Table 2 shows four phases for all samples: the first one presumably refers to the evaporation of the residual water trapped in the nanofiber's surface; the second one shows the presence of a chemical degradation process coming from bond cleavage (C–C bonds) in the polymeric backbone of the blended nanofibers [36]; the third one and fourth one show melting point and decomposition of the nanofibers networks. We can conclude that the presence of TO_2 in the nanofibers networks nearly did not show any significant effects on thermal stability. These results agree with previously reported data about ZnO addition effect on poly(methyl methacrylate) thermal degradation [37].

3.3 Scanning Electron Microscope

Figure 2 shows many trails to get smooth and beadless nanofibers by using different pluronic concentrations (0.5, 1,

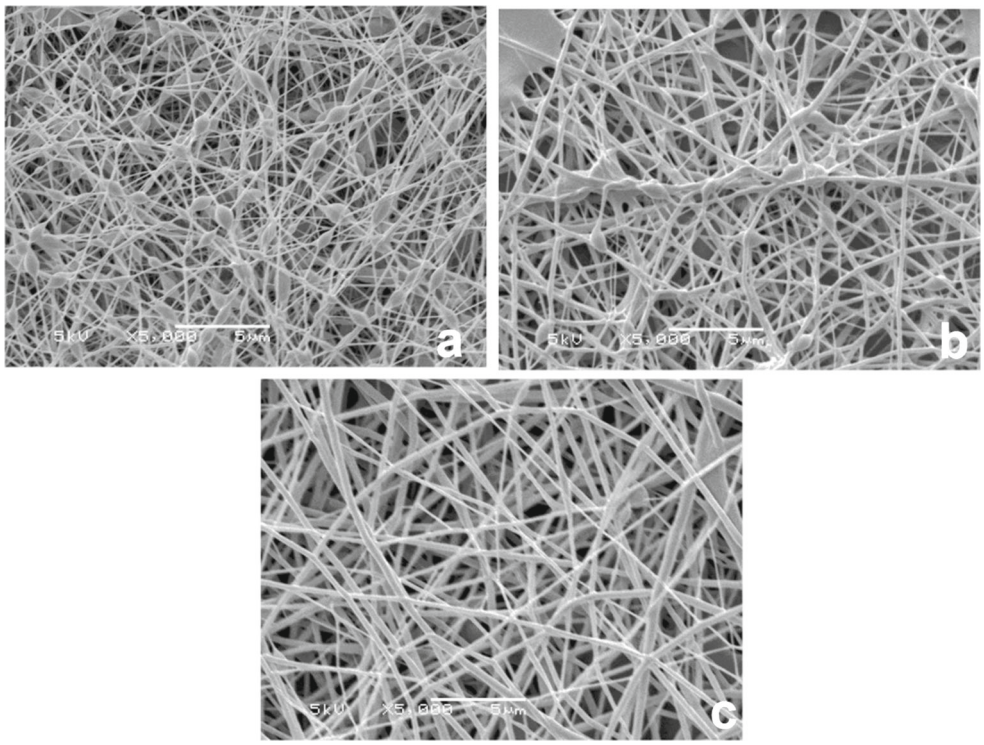

Fig. 2 SEM images of PVAB with different pluronic concentrations (**a** pluronic = 2%; **b** pluronic = 1%; **c** pluronic = 0.5%)

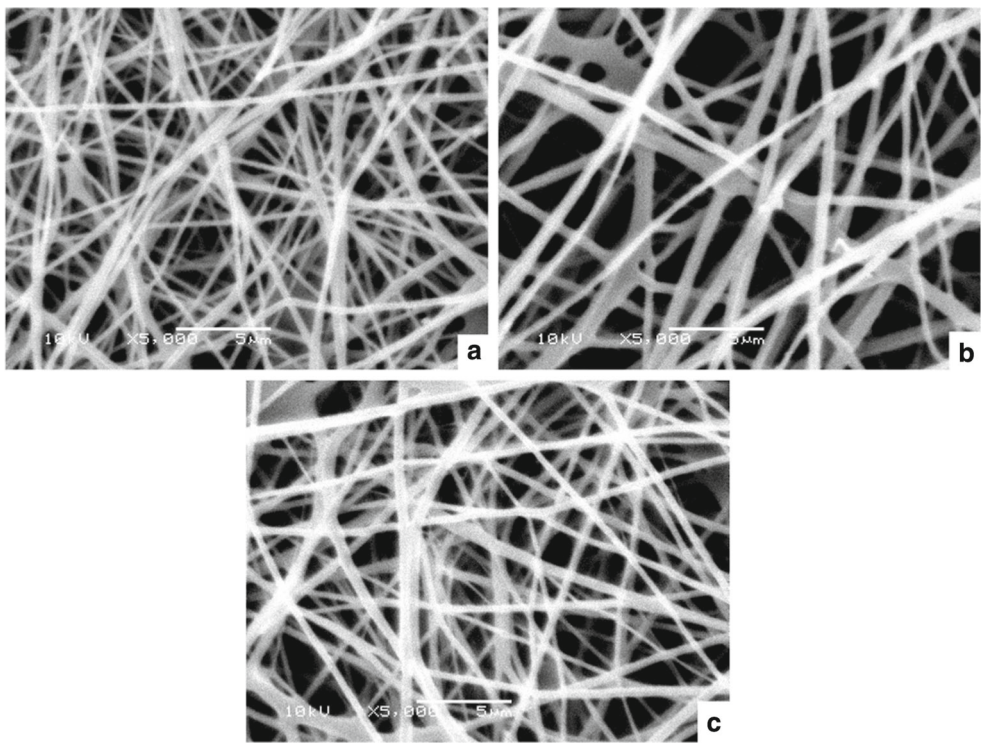

Fig. 3 SEM images of PVAT (**a** PVAT1, $TiO_2 = 0.01\%$; **b** PVAT3, $TiO_2 = 0.03\%$; **c** PVAT5, $TiO_2 = 0.05\%$)

and 2%), and the best concentration was 0.5%. Further, the TiO_2 NPs addition did not show any effect on the nanofiber morphology (Fig. 3a–c). On the other hand, the TiO2 NPs addition to the solution effect on the nanofibers diameter average as the diameter average increased from 280 ± 20 (for PVAB) to 310 ± 30 nm (for PVAT 0.05%). This behavior mainly attributed to increasing TiO_2 NPs in the PVAT blend solutions during the electrospinning process causing physically agglomerations of TiO_2 on the nanofibers' surface and change rheology of the fiber surface [38]. In conclusion, the diameter size was changed depending on TiO_2 NPs concentration.

3.4 Nanofibers Gas Sensing Performance

Figure 4 shows the responses of PVAB and PVAT nanofibers toward different gases. The used temperature was determined depending on TGA analysis (Table 2). The working temperatures (T_w) were specified by measuring the nanofiber response ($S = R_a/R_g$) at several temperatures while keeping the gas concentration constant. Figure 4a shows that the PVAT1 nanofibers give a higher response (65%) for CO_2 gas at a temperature of 160 °C. On the other hand, the response value of PVAT1 nanofibers decreased to 60% for O_2 gas at T_w of 180 °C (Fig. 4b). Finally, maximum response (100%) was observed for LPG gas (Fig. 4c) at T_w of 160–170 °C. The response of the PVAT1 nanofibers sensor is higher than that of PVAB, PVAT3, and PVAT5 nanofibers in the concentration range examined here. These results indicate that TiO_2 has significant effects on T_w lowering and response enhancing.

This improved gas sensing behavior can be demonstrated by the interchange of electrons between TiO_2 nanoparticles and the molecules of nanofibers. Therefore, the variation in the resistance is greater than that of the pure nanofibers (PVAB). The general performance levels of gas sensors depending on semiconducting oxides are affected by various parameters like nanostructures of the oxides, catalytic additives, and the geometry [39]. Our results show that, TiO_2 addition can improve the gas sensing performance to a great degree [40].

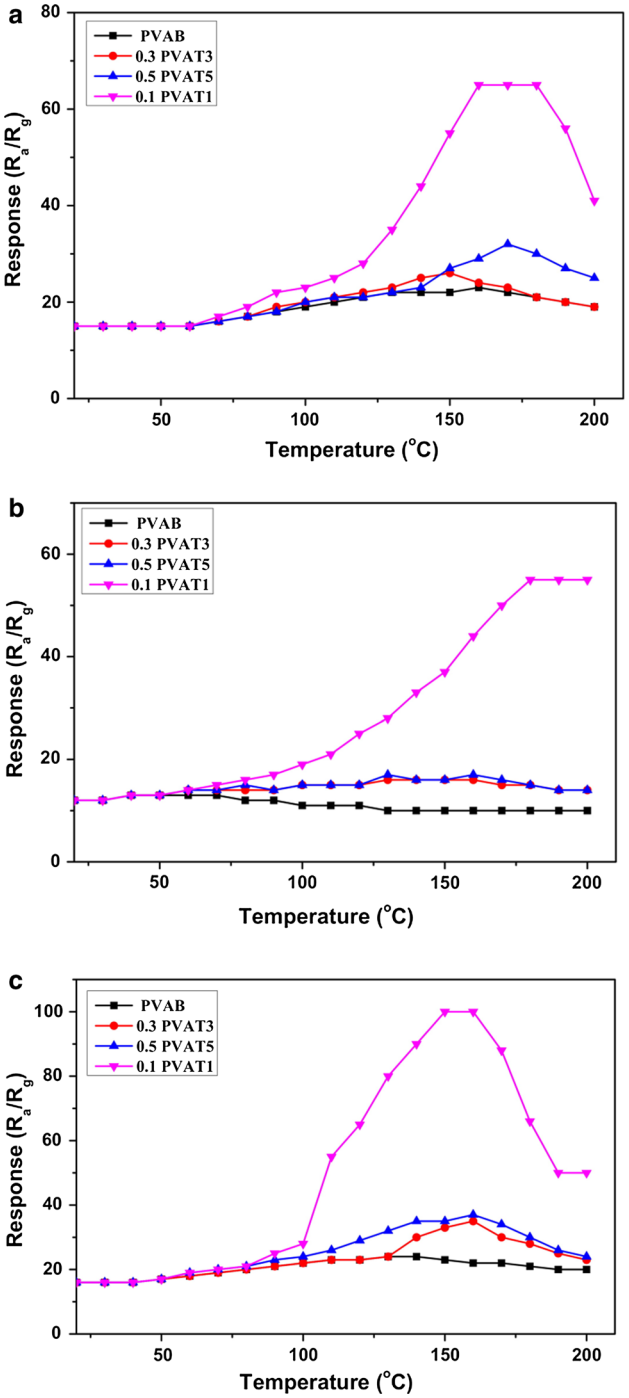

Fig. 4 Response of TiO_2-nanofiber gas sensors measured as a function of temperature for **a** CO_2, **b** O_2, and **c** LPG gases. (PVAT1, TiO_2 = 0.01%; PVAT3, TiO_2 = 0.03%; PVAT5, TiO_2 = 0.05%)

4 Conclusions

Polyvinyl alcohol and pluronic blended nanofibers containing different concentrations of titanium oxide were effectively prepared by electrospinning technique to be used as a sensitive gas sensor. The results confirmed that the addition of titanium oxide did not have a considerable effect on the nanofibers' properties like FT-IR and thermal gravimetric analysis. But the fiber diameters increased by increasing titanium oxide concentrations. The prepared nanofibers showed hopeful gas sensing features such as lower operation temperature (150–160 °C) and enough gas response (R_a/R_g = 100% for LPG) which is considered very high sensitivity. Those results show that the polyvinyl alcohol/pluronic blended nanofibers loaded with titanium oxide may be a hopeful material for gas sensor application.

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