

Research Article

Room Temperature Detection of Acetone by a PANI/Cellulose/WO₃ Electrochemical Sensor

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Chemical sensing based on semiconducting metal oxides has been largely proposed for acetone sensing, although some major technical challenges such as high operating temperature still remain unsolved. This work presents the development of an electrochemical sensor based on nanostructured PANI/cellulose/WO₃ composite for acetone detection at room temperature. The synthesized materials for sensor preparation were polyaniline (PANI) with a conductivity of 13.9 S/cm and tungsten trioxide (WO₃) in monoclinic phase doped with cellulose as carbon source. The synthesized materials were characterized by high resolution transmission electron microscopy (HRTEM), X-ray diffraction (XRD), cyclic voltammetry (CV), and Raman spectroscopy. The composite was applied for acetone detection in the range of 0 to 100 ppm vat room temperature with electrochemical impedance spectroscopy (EIS) for monitoring resistance changes proportional to acetone concentration. The developed sensor achieved a calculated limit of detection of 10 ppm and R^2 of 0.99415 with a RSD of 5% (n = 3) at room temperature. According to these results, the developed sensor is suitable for acetone sensing at room temperatures without the major shortcomings of larger systems required by high operating temperatures.

1. Introduction

Acetone is a gaseous organic compound widely used as solvent in academic and industrial settings with practical importance for fields such as occupational safety and clinical diagnosis. For example, since the exposure to acetone vapors has been associated with irritation in eyes, lungs, and nose, effective monitoring is required in order to guarantee occupational safety according to the current living standards [1–3]. Acetone derivatives have also been proposed as a potential biomarker present in human breath for clinical analysis of diseases such as *Diabetes Mellitus* (DM). The concentration of acetone in human breath has been reported within 300–900 ppbv for healthy subjects and 1800 ppbv for diabetic subjects [4, 5]. Even though detection of acetone is of practical relevance, the complexity of current techniques available for analysis of gaseous samples such as gas chromatography

and ion flow tube mass spectrometry avoids an effective monitoring of acetone. Therefore, the development of sensitive, user friendly, portable, and compact devices for acetone detection is highly desirable.

Chemical sensors based on semiconductor metallic oxides (SMO) have emerged as an active research area, due to changes in the resistance of these materials under the presence of reducing or oxidizing gases [6]. In SMO sensors the surface is modified by adsorption of gas species and space charge effects, affecting the conductivity. When SMO sensor is exposed to reducing conditions, the adsorbed oxygen is removed by reaction with the reducing gas and the reinjection of electrons reduces SMO resistance. Since acetone acts as a reducing gas, detection by sensors such as ZnO [7], In_2O_3 [8], and SnO_2 [9] has been previously studied but among SMO materials WO₃ has been proposed as the most suitable material for acetone sensing [10]. WO₃ is an

n-type semiconductor with a band gap between 2.6 and 3.0 eV [11]. Adsorbed oxygen species on WO₃ causes the transfer of electrons from WO₃ conduction band to form O^{2-} and O^{-} species. The interaction of a reducing gas, such as acetone with the chemisorbed oxygen, releases an electron to the conductance band of WO₃, which decrease its resistance. Even monoclinic phase of WO₃ which has been the most applied for acetone sensing, proper sensitivity, and selectivity is still unsolved. Thus, in order to improve the WO₃ sensitivity, doping with carbon sources such as glucose and cotton has been reported as an effective strategy to reduce the band gap value, improving WO₃ semiconductive characteristics [12].

A major technical disadvantage for the development of simple instruments based on SMO sensors is the operation temperature, which usually works within 200°C to 400°C. Thus, alternatives for room temperature operation have been studied, such as the preparation of SMO composites with conductive polymeric matrices of polyaniline (PANI). In this sense, room temperature sensors for detection of ammonia have been designed with nanocomposite thin film of PANI/Cu nanoparticles and PANI/ α -Fe₂O₃ [13, 14]. A room temperature sensor based on graphene/PANI nanocomposite was demonstrated for toluene at 50°C operation [15] and recently inclusion of WO₃ into PANI matrix has been reported to perform acetone and ETA detection at room temperature [16, 17].

In this work, a nanocomposite of PANI and WO₃ doped with carbon derived from cellulose (C–WO₃) was proposed for acetone detection at room temperature. The sensor was fabricated with two silver electrodes over an inert substrate with the PANI/C–WO₃ composite deposed in between. Sensibility of the fabricated device trough acetone was evaluated by EIS allowing detection at room temperature.

2. Materials and Methods

2.1. Reagents. All reagents were from analytical grade or similar. Hydrochloric acid (HCl) and sulfuric acid (H_2SO_4) were purchased from J.T. Baker. Aniline monomer was obtained from Acros Organics. Ammonium persulfate, ethanol, and tungsten were purchased from Sigma-Aldrich. Acetic acid and acetone were obtained from Fermont. Hydrogen peroxide (H_2O_2) at 30% and sodium hypochlorite at 13% were from Golden Bell.

2.2. PANI Synthesis. PANI was synthesized according to the methodology of Rahy and Yang [18]. Briefly, 1.9 g of aniline was protonated with 100 mL of HCl (1 M), while 6.05 g of ammonium persulfate and 100 mL of HCl (1 M) were added to begin the polymerization. When a change in coloration was achieved 2 mL of sodium hypochlorite at 5% was added by fast dripping. After 30 min, successive washings with HCl (1 M), water, and acetone were performed. Finally, the solution was dried.

2.3. Tungsten Trioxide Synthesis Doped with Carbon. Cellulose was obtained from 5 g of cotton treated with H_2SO_4 at 64% *w/w*, in a ratio of 1g:8.75 mL of acid. Reaction was performed during 1 hour at 45°C with constant magnetic agitation. After this, cold distilled water was added to decrease acid concentration at 50% w/w. Finally, the solution was washed by centrifugation at 5000 rpm during 15 min and sonicated for 15 min. This procedure was repeated until the solution reached neutrality.

Separately, 16 mL of H_2O_2 (30%) was collected in a three-mouth flask placed in ice bath with vigorous magnetic agitation. Later, 4.76 g of metallic tungsten was slowly added to H_2O_2 until solution turned out gray. When the solution became transparent, a platinum mesh was placed during 10 min to eliminate the excess of H_2O_2 . Then the mesh was retired and 20 mL of ethanol was added along with 8 mL of acetic acid and 1.5 g of treated cellulose. Finally, in order to obtain WO₃, the mixture was sonicated for 15 min and calcined at 475°C for two hours.

The composite preparation was done as follows: synthesized PANI and cellulose doped WO₃ (C–WO₃) were mixed in a proportion 1:80 as reported by Hicks and Killard [16]. Two electrodes were prepared with colloidal silver over an inert glass substrate of 2×3 cm and $50 \,\mu$ L of the PANI/C–WO₃ was deposed in between to create a sensing layer. In order to evaluate the behavior of the fabricated sensor under acetone atmosphere, solutions within a range of 10 to 100 ppmv were tested in a closed camera. The sensor response to acetone concentration was evaluated by EIS applying a sinuous current of 10 mV in a frequency range from 0.1 to 1 MHz. The procedure for fabrication of the sensor is shown in Figure 1.

2.4. Characterization of Materials. Morphology and elemental diffraction analysis of PANI, treated cellulose, C-WO₃, and PANI/C-WO3 composite were obtained by HRTEM (JEM 2200 FS, JOEL). X-ray diffraction was performed with D8 ADVANCE (BRUKER) with a Cu–K α radiation source and a 2θ angle between 5° and 70° with a step size of 0.1° and a time step of 1s. The superficial area and the pore size were obtained by BET technique (Autosorb-1, Quantachrome). FT-IR was measured on the Perkin Elmer Spectrum system. Conductivity of PANI was measured with the four-point technique with a digital multimeter (34410A AGILENT). Reflectance was measured using Cary 5000 UV-Vis-NIR spectrophotometer within 300 and 900 nm. The CV and EIS were measured on the Potentiostat/Galvanostat SOLARTRON SI 1285/1260. For CV, HCl (1 M) was used as supporting electrolyte with a window of -0.5 to 1.2 V and a scan rate of 25 mV/s during 3 cycles against an Ag/AgCl reference electrode. Response of PANI/C-WO₃ sensor to acetone was evaluated with EIS measurements with a sine wave of 10 mV in range of 0.1 to 1 MHz and a two-electrode configuration. In order to prove sensor selectivity towards acetone, injections of 20 ppm of isopropanol and ethanol were tested and compared against acetone response with the electrochemical parameters for impedance measurements previously described.

3. Results and Discussion

3.1. Material Characterization. Cellulose treated with strong acids was added as a dopant to WO_3 in order to improve



FIGURE 1: Fabrication of the electrochemical PANI/C-WO₃ sensor.



FIGURE 2: (a) Morphology of treated cellulose used as dopant, (b) synthesized PANI, (c) lattice view of WO₃, and (d) view of PANI/C-WO₃ composite.

the sensitivity towards acetone. Morphological analysis of treated cellulose showed a fibrillar structure, with an average diameter and longitude of 22.5 +/-1 nm and 116.0 +/-1 nm, respectively. Treated cellulose fibers showed lengths from 44.0 +/-1 nm to 238.0 +/-1 nm throughout the sample as shown in Figure 2(a). The morphology of PANI showed

irregular granules with a mean diameter of 60 nm due to secondary chain grown during polymerization (Figure 2(b)).

Figure 2(c) shows the typical HRTEM image for lattice structure of WO_3 . C– WO_3 was composed of small granules with a diameter between 10 and 50 nm grouped in clusters of 500 nm. TEM analysis also showed that, for PANI/C– WO_3



FIGURE 3: FT-IR of (a) treated cellulose, (b) PANI, and (c) PANI/C-WO₃ composite.

composite, the *p*-*n* heterojunctions were created with the C–WO₃ granules distributed around PANI as shown in Figure 2(d). PANI is a *p*-type semiconductor and was added as a supporting matrix to create synergistic effects with the *n*-type WO₃ semiconductor.

The FT-IR spectra of treated cellulose, PANI, and C-WO₃ are shown in Figure 3. The FT-IR of the treated cellulose presents a peak at 3335 cm⁻¹ corresponding to intermolecular and intramolecular hydrogen bridges (Figure 3(a)). The bands at 2899 cm⁻¹ correspond to the asymmetric stretching of the methylene (CH₂). The band at 1430 cm^{-1} can be assigned to the flexion of the C–6 CH₂ [19]. The analysis of PANI shows peaks at 1464 and 1551 cm⁻¹ due to stretching vibration of quinoid (N=Q=N) and benzenoid rings (N-B-N), respectively (Figure 3(b)). The band in 1287 cm⁻¹ belongs to the stretching of the bond carbonnitrogen with the secondary aromatic amine. The peak on 1230 cm⁻¹ is caused by the polaron formation on PANI structure. Finally, the peaks at 501 cm^{-1} and 511 cm^{-1} were caused by the vibration of simple bonds between carboncarbon atoms and the PANI quinoid structure [20-22]. In the C-WO3 spectrum, the O-W-O stretching mode appears as a broad band between 550 and 900 cm^{-1} (Figure 3(c)).

In Figure 4 the diffractograms of treated cellulose, WO3 and C–WO₃, are shown. The XRD pattern of the treated cellulose is shown in Figure 4(a). The most representative peaks are in the positions corresponding to the 2θ theta angle of 14.7°, 16.46°, and 22.60°; these correspond to the planes 110, 110, and 200, respectively. The intensity was increased by the removal of the hemicellulose through the treatment

with strong acids. The peaks that appear in these positions correspond to the structure of cellulose type I [23]. The peaks presented at 2θ angle for C–WO₃ and WO₃ samples correspond to a crystal formation of the monoclinic structure. It is also possible to identify the peaks at 23.06°, 23.53°, and 24.19° corresponding of the planes 002, 020, and 200 (Figure 4(b)). The crystalline structure of WO₃ was not affected by the doping with carbon atoms since there was no significant difference between the C–WO₃ sample and the blank (Figure 4(c)).

Figure 5(a) shows the Raman spectra obtained by $C-WO_3$ with two strong bands between 801-803 cm⁻¹ and 707–709 cm⁻¹ referred to the stretching ν (O–W–O). The bands between 264-262 cm⁻¹ and 315-326 cm⁻¹ were attributed to the stretching on δ (O–W–O), which include the band between 209 and 214 cm⁻¹. Finally, the band between 180 and 130 cm⁻¹ was referred to the framework of WO₃ atoms of crystalline monocyclic phase. Bands D and G referred to the presence of carbon in the material were observed with a closer look to the zone between 1200 and 2000 cm⁻¹ (Figure 5(b)). The first band was caused by the stretching movement of the C-C bond on graphitic compounds and is usually found in carbon compounds with sp² bonds. The D band is typical of graphite oxide and showed a disorder on their sp² bonds. The C–WO₃ sample showed a predominant G band, which suggest a preponderance of the crystalline structure of carbon.

PANI characterization was carried out by UV–Vis–NIR spectroscopy between 200 and 800 nm. The band observed between 325 and 365 nm corresponds to $n-\pi^*$ transitions of



FIGURE 4: XRD of (a) treated cellulose, (b) WO₃ without doping, and (c) PANI/C-WO₃ composite.



FIGURE 5: Raman specter for (a) $C-WO_3$ between 100 and 1200 cm⁻¹ and (b) augmented region between 1200 and 1700 cm⁻¹.

aniline, while the broad band at 600–620 nm correspond to $n-\pi^*$ transitions of quinine-imine groups (Figure 6(a)). WO₃ powder as well as C–WO₃ powder was analyzed between 200 and 900 nm (Figure 6(b)).

Tauc's plot was applied to calculate the bang gap of WO_3 and C-WO₃ with obtained band gap of about 2.82 and 2.57 eV, respectively. The slight difference was attributed to the doping effect of cellulose as reported previously by Xiao et al. [4].

Cyclic voltammetry of PANI was carried out in order to probe the stability and electroactivity of the material. PANI showed two redox pairs around 0.125 and 0.7 corresponding to changes in the oxidation state of the PANI movement of the C–C bond on graphitic compounds and is usually found in carbon compounds with sp² bonds (Figure 7(a)). The material showed a high stability after 3 cycles. PANI/C–WO₃ composite in a relation 1:80 was also tested by CV. The voltamperogram showed negative values for oxidation peak as well as oxidation and reduction peak for PANI and WO₃. The low electroactivity of composite was attributed to the predominant concentration of WO₃ in the sample (Figure 7(b)).

Additionally, conductivity by the four-point technique was performed for PANI sample. The conductivity of PANI has been reported within the range of 2–10 S/cm and can be influenced by various factors such as electrochemical redox state, pH, humidity, and temperature. The final conductivity for PANI was 13.9 S/cm. Finally, the maximum superficial area for C–WO₃ sample was 245.85 m²/g and the obtained pore size was 169.4 Å.

3.2. Sensor Operation. Initially the sensor was continuously measured at room temperature atmosphere to evaluate the stability of the composite after successive measurements of acetone. As shown in Figure 8(a) no significant changes in the resistance of materials caused by room temperature operation and the presence of air components were observed. Then, the sensor was evaluated with concentrations between 0 and 100 ppmv of acetone, with increments of 10 ppmv after each measurement. In Figure 8(b), Nyquist diagram showed that the real part of impedance decreased when acetone increased. A calibration curve relating the absolute impedance with acetone concentration was constructed for 0 to 100 ppmv (Figure 8(c)). A correlation coefficient (R^2) of 0.99415 was obtained for the relation between sensor impedance and acetone concentration at room temperature with a calculated



FIGURE 6: UV-VIS specters for (a) PANI and (b) WO₃ and C-WO₃.



FIGURE 7: CV for (a) PANI and (b) PANI/C-WO₃ composite.

detection limit of 10 ppmv at room temperature with a RSD of 5%. The same procedure was applied to PANI sensor in order to prove the superiority of the developed composite for sensing, and, as shown in Figure 8(c), response of PANI sensor to acetone concentrations was negligible when compared with PANI/C–WO₃ composite. Selectivity of developed PANI/C–WO₃ sensor was evaluated with injections of acetone, ethanol, and isopropanol under the same conditions described for acetone detection. Figure 8(d) shows the recorded response toward each gas after 40 ppmv injections of each gas at room temperature. It can be observed that the developed sensor exhibited a higher response to acetone compared to the other gases. This can be attributed to the selectivity exposed by doped WO₃, while the room

temperature operation can be attributed to synergistic effects of PANI and C–WO₃.

3.3. Sensing Mechanism. The acetone vapor sensing mechanism of the PANI/C–WO₃ based sensor can be described by the processes of electron transfer and chemisorption. The absorption of oxygen forms ionic species as O^{2-} , O_2^- , and O^- , which can react with acetone vapor at high temperatures [24]. At lower temperature, the species cover the sensor surface, which prevents reactions of acetone vapor and chemisorbed oxygen and therefore resistance of the sensor decreases considerably [25]. However, the use of PANI in the composite induces the formation of *p*-*n* heterojunctions between *p*-type PANI and *n*-type WO₃ and lead to a redistribution of charge



FIGURE 8: Sensor operation at room temperature for (a) air atmosphere, (b) acetone concentration, and (c) obtained calibration curve for PANI/C–WO₃ composite and only PANI under acetone concentrations.

carriers at the interface of these material, decreasing the activation energy and enthalpy required for the physisorption of acetone, allowing the detection at room temperature [20, 26, 27]. The inclusion of cellulose as the carbon source in the composite increases the sensitivity of the sensor, which might be induced by the formation of pores on the surface of composite, as reported by Sakai et al. [28].

4. Conclusions

A nanostructured material composed by a conductive polymeric matrix of PANI with WO_3 doped with cellulose was applied to the detection of acetone at room temperature and low concentrations. The developed sensor showed a limit of detection of 10 ppm after repeated exposure of acetone, which proves the suitability of the developed material for room temperature sensing of acetone without the major shortcomings of larger systems required by high operating temperatures.

Conflicts of Interest

The authors declare no conflicts of interest.

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