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# Water Sensor for a Nonaqueous Solvent with Poly(1,5-diaminonaphthalene) Nanofibers

Md. Aminur Rahman, Mi-Sook Won, Nak-Hyun Kwon, Jang-Hee Yoon, Deog-Su Park, and Yoon-Bo Shim\*

Department of Chemistry and Center for Innovative BioPhysio Sensor Technology, Pusan National University, Busan 609-735, South Korea

A water sensor for a nonaqueous solvent was fabricated using poly(1,5-diaminonaphthalene (DAN) nanofibers, which were prepared through a catalytic chemical polymerization of the DAN monomer using Fe(III) salt as the catalyst. Poly(1,5-DAN) nanofibers were characterized by atomic force microscope (AFM), transmission electron microscope (TEM), scanning electron microscope (SEM), and UV–vis spectroscopy. The electrochemical properties of poly(1,5-DAN) nanofibers were investigated using cyclic voltammetry (CV). The electrochemical activity of poly(1,5-DAN) nanofibers was utilized for water sensing. The fabrication of water sensor was followed by placing one drop (about 2  $\mu$ L) of 0.01% poly(1,5-DAN) nanofibers solution in the gap between two split gold electrodes (PBSA) and completely dried. The response of the water sensor in an acetonitrile solution was evaluated under optimized conditions. The linear dynamic range was from 0.05 to 20%, and the detection limit was determined to be 0.01%. The response of this sensor was shown to be comparable to that obtained with the Karl Fischer titration method.

Among the methods for determining water content,<sup>1–5</sup> the Karl Fischer titration method has been most widely used in organic solutions.<sup>1</sup> Although this method is convenient for accurately measuring trace water (up to 100 mg) in materials, it has several disadvantages that include the following: it is a time-consuming process, it requires use of toxic and malodorous reagents, end-point determination is difficult, and chemical interference is problematic. To overcome the disadvantages of using reagents that contain pyridine, a modified titration method was developed.<sup>4</sup> In addition, there exists several methods for water detection, such as the solid-phase microextraction,<sup>5</sup> piezoelectric quartz crystal,<sup>6</sup> microwave interferometer,<sup>7</sup> Raman spectroscopic,<sup>8</sup> and electro-

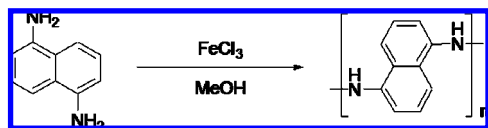
metric<sup>9</sup> methods, but these methods require using rather sophisticated equipment and require long measuring times. Thus, there is a need to develop a faster and more convenient method, such as a sensor, for water detection in nonaqueous solutions. Thus far only a few sensors that can detect water in nonaqueous solutions exist.<sup>10</sup> Optical sensors based on fluorescence<sup>11,12</sup> and absorbance<sup>13</sup> measurements have been developed for this purpose. These sensors require the use of a fluorescent dye or a metal-centered polypyridyl complex for the signal generation, and these compounds must be immobilized on the sensor surface. Electrochemical methods, such as impedance,<sup>14</sup> conductance,<sup>15</sup> and amperometric<sup>10,16</sup> measurements have also been used to detect water and humidity. However, these methods are still complicated and do not provide a simple and straightforward way to detect water. Moreover, many of these methods simply measure moisture and are not suitable for directly determining the water content in organic solutions. Thus, the development of an alternative method for determining water content has been in high demand for several decades. To our knowledge, there has been no development of a water sensor that directly determines water content in organic solvents.

On the other hand, conducting polymers (CPs) have attracted wide attention due to their use in optical devices, energy conversion devices, and sensors.<sup>17</sup> Polymers derived from the aromatic diamines, such as diaminonaphthalene (poly-(DAN)) through electrochemical methods, have been reported as another type of CP.<sup>18–21</sup> Although the electrochemical preparation of CPs is straightforward, the chemical method is much more suitable for obtaining CP nanostructures. In the present study, we describe

\* Corresponding author. E-mail: ybshim@pusan.ac.kr. Phone: (+82) 51 510 2244. Fax: (+82) 51 514 2430.

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**Figure 1.** The reaction scheme for polymerization of 1,5-diaminonaphthalene.

the catalytic chemical synthesis of conducting poly(1,5-DAN) nanofibers by using ferric salt and molecular oxygen as the catalyst and oxidant, respectively. The remarkable feature of this material is that it can be used to reliably determine water content in nonaqueous solvents. This unique property of the poly(1,5-DAN) nanostructures provides a novel approach for designing a water sensor that can detect water content in nonaqueous solvents. Thus, here, we have fabricated a water sensor for an organic solvent using poly(1,5-DAN) nanofibers. To maximize signal detection, experimental parameters that affect the sensor response (such as temperature, film thickness, applied potentials, etc.) were optimized. Finally, the ability of this sensor to detect water content in a nonaqueous solvent was demonstrated in an acetonitrile solution. In addition, the sensor response was compared with that of the more established Karl Fischer titration method.

## EXPERIMENTAL SECTION

**Apparatus.** The cyclic voltammetric and pulse chronoamperometry experiments were carried out with Kosentech Co. Model PT-2 and EG & G 273A potentiostat/galvanostats. The measuring cell was thermostatted with Polyscience Co. Model 80 T water bath. The Karl Fisher titration was carried out using a Metrohm Ltd. 682 Titrprocessor coupled with a 665 Dosimat. A JEOL JEM-2010 electron microscope (Jeol High-Tech. Co.) with an acceleration voltage of 200 kV was used to obtain TEM images. Atomic force microscopy (AFM) experiments were carried out on a multimode AFM system from Digital Instrument Inc. Commercial  $\text{Si}_3\text{N}_4$  tips (125 mm length, 300 kHz resonance frequency, and 5–10 nm radius) were attached to a triangular cantilever made of the same material. The force (spring) constant was 20–1000 N/m. A scanning electron microscope (SEM) image was obtained using a Cambridge Stereoscan 240.

**Preparation of Poly(1,5-DAN) Nanofibers.** 1,5-Diaminonaphthalene, purchased from Aldrich Co., was recrystallized twice in aqueous ethanol (50% v/v) under nitrogen and then dried at room temperature under vacuum over  $\text{P}_2\text{O}_5$  for 48 h. Poly(1,5-DAN) nanofibers were prepared using catalytic chemical polymerization by stirring a mixed solution of the corresponding monomer and different molar concentrations of ferric chloride ( $\text{FeCl}_3$ ) in different solvents. The typical reaction procedure was as follows: 2.0 g (0.0126 mol) of monomer was added to a 100 mL methanol solution of  $\text{FeCl}_3$  (3.40 g, 0.0126 mol), and the reaction mixture was stirred at 30 °C for 24 h. The reaction was performed in both nitrogen and oxygen atmospheres. The resulting deep blue precipitates were dried in a vacuum at 50 °C for 24 h followed by filtering and washing repeatedly with ethanol, 0.1 M HCl, and distilled water. The yield of poly(1,5-DAN) nanofibers was 98%. The reaction scheme for polymerization of 1,5-DAN is shown in Figure 1. All other reagents were used without further purification. The aqueous solutions were made using deionized water purified by a Milli-Q system (Millipore Co.).

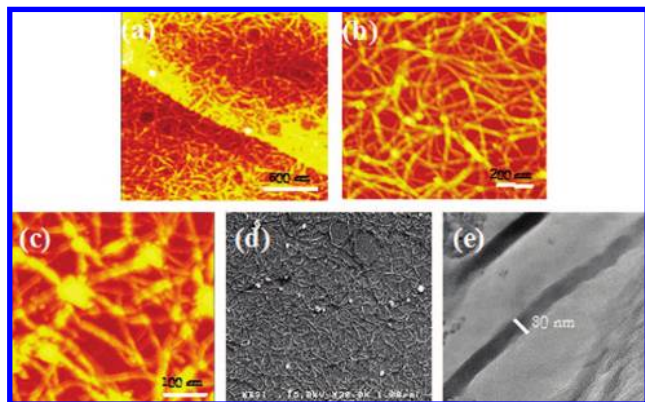
**Fabrication of the Electrode.** Au electrodes were fabricated on clean glass slides by vacuum deposition followed by treatment with (3-mercaptopropyl) trimethoxysilane (MPS). Prior to this treatment, the glass was pretreated with isopropyl alcohol. Twelve 3 in.  $\times$  1 in. glass microslides were placed in a glass dish and boiled in isopropanol for 10–15 min. After this, they were removed from the solvent, allowed to cool, and then dried in a stream of nitrogen. MPS (Sigma, 97% pure) was used as a molecular glue to attach Au to the slides. The solution was composed of 400 g of isopropanol, 10 g of water, and 10 g of MPS. The mixture solution was brought to a boil, and the slides were placed into the dish and covered with a watch glass for approximately 15–20 min. They were removed, cooled, and rinsed with isopropanol and water before being thoroughly dried in an argon gas stream until they appeared to be clear. Finally, the slides were placed in a dry oven at a temperature between 100 and 105 °C for 10 min. To obtain a uniform MPS coating on the glass surface, the entire procedure was repeated at least once. A thin plastic mask was used to define the electrode pattern. The contact pads were each 0.3 cm in diameter, and the lines connecting them were 0.1 or 0.3 cm across. The gold was vapor-deposited to a thickness between 800 and 1000 Å with a Hummer X Sputter Coater (Anatech Co.).

**Polymer Nanofibers Coating Procedure.** The Au coated electrode was split in half by a sharp blade containing a gap width of approximately 5  $\mu\text{m}$ . Spin coating was used to coat the gap between the separated electrodes with poly(1,5-DAN) nanofibers. One drop (2  $\mu\text{L}$ ) of 0.01% poly(1,5-DAN) nanofibers in methanol was placed between the gap of the two split electrodes while being spun at 200 rpm to produce the sensor. The two sizes of the polymer-bridged split electrodes were used for CV and chronoamperometry, respectively. The polymer coating process was repeated three times. The area of the polymer-bridged split Au (PBSA) electrodes for CV was approximately 0.07  $\mu\text{m}^2$ . Sensors were activated, prior to use, by being dipped in a 1% water/acetonitrile solution for 5 min, followed by drying in a vacuum oven for 5 h.

**Analytical Procedure.** A double-step chronoamperometric measurement was performed after the PBSA electrode was activated and stabilized at an open circuit voltage for 2 min. Pulse-type double-step potentials were applied between +0.8 and −0.8 V with a pulse width of 10 s. The temperature of the measuring vessel was maintained at  $20 \pm 0.1$  °C. To avoid contaminating the trace water, a clean measuring vessel was dried at 160 °C in a vacuum oven for more than 6 h.

## RESULTS AND DISCUSSION

The concentration of  $\text{FeCl}_3$  and the solvent used for the preparation of poly(1,5-DAN) nanofibers was shown to have a significant effect on the reaction yield. Thus, the  $\text{FeCl}_3$  concentration was optimized to produce the highest yield of poly(1,5-DAN) nanofibers. In addition, we determined that methanol produced the highest reaction yield relative to the other solvents tested. The reaction atmosphere was also shown to have a significant effect on the yield of poly(1,5-DAN) nanofibers. The maximum yield of poly(1,5-DAN) nanofibers was obtained under an oxygen atmosphere (98%), which was more than 2 times higher than the yield obtained under a nitrogen atmosphere (41%). This is because  $\text{Fe(III)}$  ions acted as a catalyst in the presence of oxygen, and

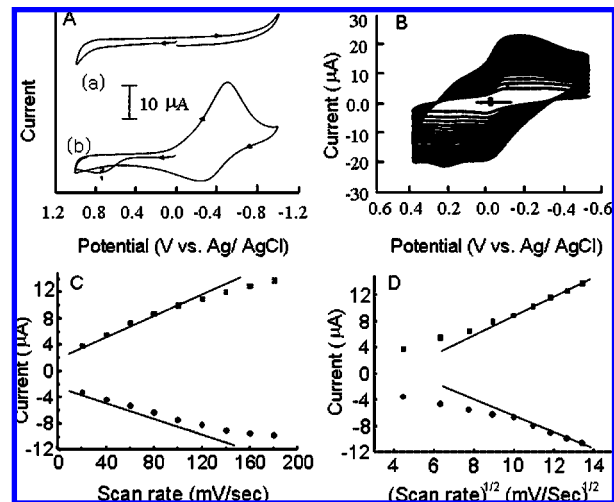


**Figure 2.** AFM (a–c), SEM (d), and TEM images (e) of the chemically prepared poly(1,5-DAN) nanofibers. Scale bars: (a) 500 nm, (b) 200 nm, (c) 100 nm, (d) 1  $\mu\text{m}$ , (e) 30 nm.

polymer reactants were continuously regenerated for subsequent reactions. The prepared poly(1,5-DAN) nanofibers were characterized by AFM, SEM, and TEM (Figure 2). The AFM images at low and high magnification clearly show the homogeneous formation of the poly(1,5-DAN) nanofibers. The nanofibers were approximately 10–30 nm in diameter and 400 nm in length. The SEM image in Figure 2 also confirmed successful nanofiber formation. Figure 2e shows the TEM image of a single poly(1,5-DAN) nanofiber having a diameter of 30 nm.

In a previous study, the UV–vis spectrum of the electrochemically prepared poly(1,5-DAN) film<sup>19</sup> showed two major absorption bands at 277 and 350 nm. The band at 277 nm was due to the  $\pi$ – $\pi^*$  transition of the benzoids and quinoids in the poly(1,5-DAN). The absorption band at 350 nm was due to the  $n$ – $\pi^*$  transition of the amine groups. The high-energy band observed from the  $\pi$ – $\pi^*$  transition indicated that the aromatic rings were isolated from each other and thus were not extensively conjugated. When the poly(1,5-DAN) film was progressively oxidized, the band at 350 nm showed a significant blue shift and decreased in oscillator strength, while a new broad absorption band appeared at 580 nm. Complete oxidation of the poly(1,5-DAN) film to the quinoid state resulted in the appearance of another absorbance band at 517 nm. The absorbance bands at 517 and 580 nm may be due to the presence of bipolarons and polarons, respectively, on the polymer molecules. It is plausible that the poly(1,5-DAN) nanofibers formation mechanism might be similar to that of an electrochemically prepared poly(1,5-DAN) film. However, the primary objective of this study was to utilize poly(1,5-DAN) nanofibers as a water detection material not to determine the exact mechanism of poly(1,5-DAN) nanofiber formation. Thus, further studies will be required to better understand this process.

The electrochemical properties of poly(1,5-DAN) nanofibers were investigated using cyclic voltammetry (CV). Figure 3A shows CVs recorded for a conventional bare gold disk electrode (a) and a gold-coated 0.01% poly(1,5-DAN) nanofiber electrode (b) in a 0.1 M  $\text{KNO}_3$  solution. As shown in Figure 3A, a pair of redox peaks of the polymer film appeared at  $-0.30$  V/ $-0.54$  V vs. Ag/AgCl. The presence of a quasi-reversible redox peak for the poly(1,5-DAN) nanofibers in an aqueous solution indicated that the polymer film had electrochemical activity. With the use of the four-point probe method, the conductivity of the polymer film was determined to be  $\sim 10^{-5}$  S/cm, indicating that the polymer



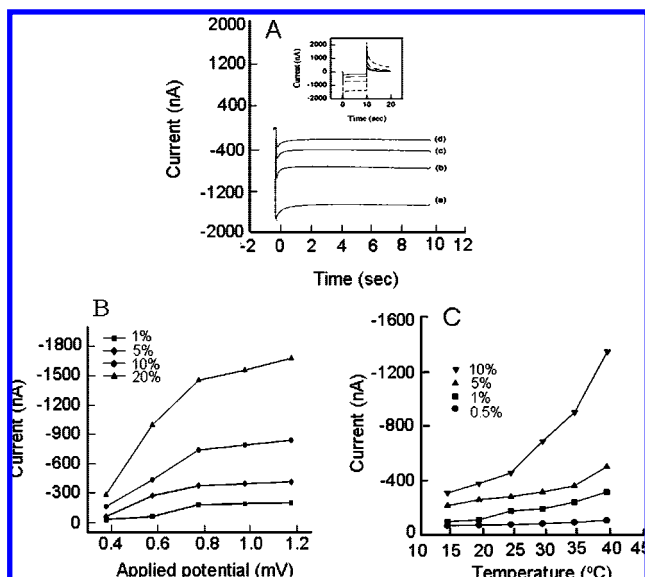
**Figure 3.** (A) Cyclic voltammograms of (a) a bare and (b) a 0.01% poly(1,5-DAN) coated gold disk electrode in a 0.1 M  $\text{KNO}_3$  solution, (B) CV recorded at different scan rates, (C) peak currents vs the scan rates dependency, and (D) peak current vs the square root of scan rates dependency.

exhibited semiconducting properties. The scan rate dependency of a set of quasi-reversible redox peaks for the polymer-coated electrode between 0.4 and  $-0.5$  V was investigated in a 0.1 M  $\text{KNO}_3$  solution. As the scan rate was increased from 20 to 180 mV/s, we observed a corresponding increase in the peak current of the polymer film (Figure 3B). As shown in Figure 3C, the anodic and cathodic peak currents were linearly proportional to the scan rate from 10 up to 90 mV/s. However, over 90 mV/s, the peak currents were proportional to the square root of the scan rate (Figure 3D). This observation suggests that a finite diffusion model dominates during the lower scan rates, whereas a semi-infinite model prevails at higher rates since the diffusion layer thickness is comparable with that of the polymer film layer. This can be explained by the fact that the charge is transported through the physical diffusion of the electroactive reactants themselves or of counterions.<sup>22</sup>

On the basis of these electrochemical properties, poly(1,5-DAN) nanofibers were utilized for water sensing. To fabricate the sensor, one drop (about 2  $\mu\text{L}$ ) of 0.01% poly(1,5-DAN) nanofiber solution was positioned in the gap (approximately 5  $\mu\text{m}$ ) between two split gold electrodes (PBSA) and completely dried. Figure 4A shows the response time of the PBSA electrode to changes in the water content of an acetonitrile solution under optimized conditions. Current changes were recorded over time using double potential-step chronoamperometry (inset in Figure 4A). The double chronoamperometric technique was used to attain a reproducible initial state of the sensing material. We obtained the initial state by reducing the polymer film at  $-0.8$  V at the forward potential step. Thus, in every measurement, the polymer layer was first reduced at the forward potential, and the anodic currents were measured during the reverse potential step. The response time was determined to be approximately 5 s. The anodic current during the reverse potential step was chosen as the analytical signal because it was larger and more reproducible than that obtained during the forward step. From these experiments, a linear

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**Figure 4.** (A) Response time dependency of the current recorded for the acetonitrile solution containing (a) 20%, (b) 10%, (c) 5%, and (d) 1% water (anodic pulse potential = +0.8 V, cathodic pulse potential = -0.8 V, delay time = 10 s, temperature = 30 °C, film thickness = 123 nm). (B) Applied potential and (C) temperature dependence of the response current of the PBSA sensor.

relationship between anodic current and the water content of an acetonitrile solution was observed. The results indicate that the PBSA electrode can function as a water sensor.

There are several potential mechanisms by which these polymer probe are detecting water. Based on the characteristics of conductive polymers, such as changes in electrical conductivity, two possible mechanisms are: i) the voltammetric current is related to the action of water acting as a nucleophile and generating radical cations in the polymer matrix (polaron or bipolaron states), which would cause an increase in conductivity, and ii) the diffusion of water into the polymer changes the effective dielectric constant of the polymer, thus altering the conductivity in proportional to water content.

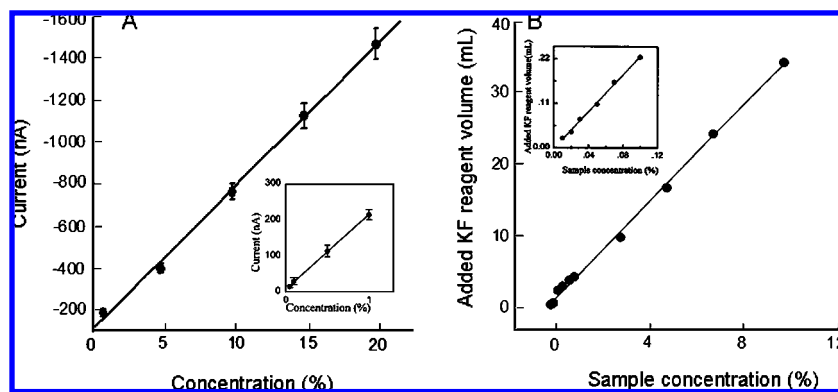
We also examined the temperature dependency of the current response of the PBSA sensor in acetonitrile solutions containing 0.5, 1, 1.5, and 10% water (Figure 4B). The temperature of the sample solutions varied from 15 to 40 °C. The response at different temperatures changed with the water content in the sample solution. The anodic current steeply increased as the temperature

of the measured solution increased from 15 to 40 °C. The increased conductivity of the polymer at increased temperatures is most likely due to the increasing diffusion rate of water. However, the current did not significantly increase with increasing temperature at 0.5% water content. At water contents of more than 1%, the current significantly increased due to a higher conductivity of the polymer film at increased temperatures. However, for practical purposes, the temperature was kept at 30 °C for all subsequent experiments.

The applied potential dependency of the response current at various water concentrations (1–20%) was studied (Figure 4C). The pulse-type step potentials were separately applied at -0.8 V to +0.4, +0.6, +0.8, +1.0, and +1.2 V. The initial negative potential step was not varied because the cathodic current was not used. Instead we used the anodic current as the analytical signal, so only the anodic potential step was optimized. The current response gradually increased as the anodic potential shifted to a more positive potential between +0.4 and +1.2 V. However, current responses did not increase significantly above +0.8 V. Thus, the double-step potential was chosen as -0.8 and +0.8 V.

The film thickness dependency of the anodic current in 10% water in an acetonitrile solution was also studied. The film thickness was controlled by changing the number of 2  $\mu$ L drops of 0.01% poly(1,5-DAN) nanofiber solution from 3 to 13. The anodic currents were shown to increase linearly by increasing the polymer film thickness from 120 to 180 nm, where film thickness was measured in a completely dried state by tilting the electrode with the scanning electron microscope (SEM). These results indicate that the electrochemically active sites increased at increased film thickness. However, thicker polymer film can impede electron transfer; thus, a polymer film thickness of 120 nm was used in all subsequent experiments.

Figure 5A shows a calibration plot for determining water content in an acetonitrile solution using a PBSA sensor. Under the optimized condition, the water sensor showed a linear relationship between current and water content in the range of 0.05–20%. The relative standard deviation (RSD) at 1% water content was found to be  $\pm 2.98\%$ . This linear dependence between current and water content yielded equations of  $I$  (nA) = 106.8 (nA) + 68.4[% of water] with a correlation coefficient of 0.998, and  $I$  (nA) = 16 (nA) + 150[% of water] with a correlation coefficient of 0.997 at the low concentration range. On the basis of these results, the sensitivity at a low concentration range was



**Figure 5.** Calibration plots for determining water content in the acetonitrile solution with (A) a PBSA sensor and (B) the Karl Fischer titration method.

better than that at a high concentration range. The detection limit was determined from 3 times the standard deviation of the blank noise (95% confidence level,  $k = 3$ ,  $n = 5$ ). The average blank value was about 18 nA, and the standard deviation was  $\pm 0.55$ . The detection limit was determined to be 0.01% from the slope of the calibration plot (150 nA/% of water) at the low concentration range. The response of the present sensor was compared with that of the Karl Fischer titration method. Figure 5B shows the calibration plot obtained for an acetonitrile solution using the Karl Fischer method. The inset in Figure 5B shows a calibration plot obtained at a lower water concentration range. The linear range was between 0.01 and 10%, and the detection limit was determined to be 0.01%, which is comparable to that obtained with the water sensor. The detection limit of the present water sensor in acetonitrile was also comparable to that obtained from a fluorescence based water sensor<sup>12</sup> where the detection limit was determined in a batch system.

To examine the reproducibility of measuring water content with this sensor, five repeated measurements were made with newly fabricated sensors in acetonitrile solutions. The average value of the current response was 205.8 nA at 1% water content and the RSD was  $\pm 1.5\%$ . The high RSD value (about 3%) obtained through multiple tests using a single electrode relative to a fresh electrode used a single time is related to the surface poisoning of the poly(1,5-DAN) nanofibers modified electrodes. At 5% water content, the average value was 421.7 nA and RSD was  $\pm 6.0\%$ . The

long-term storage stability of this water sensor was tested every 5 days and was determined to be more than one month. Up to 40 days, the sensitivity retained 97% of the initial sensitivity. The above results clearly indicate that the proposed water sensor has a long shelf life.

## CONCLUSIONS

The present study describes the catalytic chemical synthesis of poly(1,5-DAN) nanofibers. Poly(1,5-DAN) nanofibers were characterized by SEM, AFM, TEM, and cyclic voltammetry. Poly(1,5-DAN) nanofibers have unique properties that may be useful in detecting water content in a nonaqueous solvent. On the basis of this, a water sensor, fabricated using poly(1,5-DAN) nanofibers, was shown to be highly sensitive in detecting water content in an acetonitrile solution. The detection limit of the water sensor was determined to be 0.01%, which was comparable to that obtained from the Karl Fisher titration method.

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