Synthesis and Fabrication of Polyvinyl Alcohol Nanofibers Based Capacitive Relative Humidity Sensor

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Abstract

A capacitive humidity sensor based on Polyvinyl-alcohol (PVA) electrospun nanofibers were fabricated through a versatile electrospinning technique with a controlled diameter of nano-size ranging from 1 nm to 100 nm. The synthesized nanofibers were heat treated and characterized via Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM), and Thermo-Gravimetric Analyzer (TGA) for chemical, morphological and thermal properties. Fibers of the admirable morphological structure were selected and deposited over interdigitated alumina electrodes for the investigation of humidity detection characteristics. The sensor was reported capacitive type and was calculated 48 pf for relative humidity (RH %) 32–92 %. The dynamic response study confirmed the durability and stability of the sensor in a humidity chamber. The sensor exhibited quick response and recovery time and was observed 103.27 seconds to measure the maximum RH value (rise time) and takes 13 seconds (fall time) to regain its normal state. Also, it was noted that at low frequencies i.e. 500 Hz and 1 kHz the sensor shows maximum sensing ability. Furthermore, the sensor has a linear and repetitive response is cost-effective and is easy to fabricate.

Index Terms: Nanofibers, Fourier Transform Infrared Spectroscopy, Electrospinning, Capacitive Sensor, Polyvinyl-Alcohol.

I. INTRODUCTION

Nowadays, humidity sensors are gaining popularity in the international market because of their numerous applications in industrial processes, health monitoring, food storage and safety, agriculture, poultry farms, defense, smart homes, automobiles, intelligent systems, and wireless sensor monitoring for diagnosis of defects in infrastructures and civil engineering. However, there are several problems associated with humidity sensors, such as high cost, low perception, slow response and recovery, poor stability, and a small span of sensitivity [1-6].

Humidity sensing materials of various morphological properties such as nanowires [7], nanotubes [8], thin films [9], nanofibers [10], and one dimension [11] have been reported in the literature. One such important material is PVA. The PVA is a non-crystalline poisonous and biocompatible polymer with superior thermal and mechanical properties. Due to these characteristics, PVA has numerous applications [12] and [13]. PVA is widely used material in sensors and nanofibers synthesis [14-16]. There are two types of humidity sensing mechanisms that are generally used for RH sense, one is the resistive sensing mechanism and the other is capacitive. In resistive sensing mechanisms, variation in resistance is observed regarding changes in RH while in capacitive RH sensing mechanism change in capacitance concerning RH is observed. Resistive RH sensors are commonly used for conductive type materials such as silver nanowires while capacitive type RH sensors are used for non-conducting materials such as polymers. So, different transduction methods have been applied for the advancement of RH sensors like optical RH sensors [17-19], RH sensing through electromagnetic waves, and acoustic wave RH sensors [20].

On the basis of advantages and applications, many types of detecting mediums have been used in RH sensors like TiO₂, ZnO, Carbon Nano Tubes (CNT), silver nanowires, ceramics, and organic-inorganic composites [21].

In this research, PVA is used as a base material for the synthesis of composite nanofibers. PVA is also considered environmentally friendly and cost-effective [22].

Carbon Nano Tubes (CNTs) are special materials and ideal candidates for RH sensors by enhancing the mechanical and electrical properties of polymer/CNT composites [23].

Multi-Wall Carbon Nano Tubes (MWCNTs)/PVA composite thin film was investigated as a switching type RH sensor rather than a Single Wall Carbon Nano Tubes (SWCNTs) [24]. Where using CNTs and other materials as a polymer composite is an expensive option for RH sensors. A cost-effective and simple resistive RH based on PVA produce by the Sol-gel technique [25] and [26].



PVA nanofibers were deposited over interdigitated electrodes and used as RH sensing devices. The sensor has the advantages of high sensitivity, small size, and large surface area, however; some of the important parameters such as response and recovery time are missing [27-30].

II. EXPERIMENTAL SETUP

A. Synthesis of Polyvinyl Alcohol (PVA) Nanofibers

The spinning solution of PVA was prepared by mixing 5 wt % PVA (2 gm) in de-ionized water (20 ml) and stirred gently for 30 minutes on a hot plate at 350 rpm and the temperature was kept 140 °C. Afterward, the solution was cooled at room temperature, and finally, a uniform and transparent solution were obtained. The PVA solution was pulled in a syringe of 1ml. The syringe was then placed in a laboratory-made microcomputer-controlled electrospinning unit equipped with a syringe pump and 20 kV DC power supply based on a fly-back transformer. The positive terminal was connected with the syringe's needle while the collector was grounded. Aluminum foil was wrapped around the collector side for the collection of fibers. When DC voltage of 20 kV was applied to a liquid droplet, liquid at the tip of the needle charged, and the droplet was stretched due to the electrostatic repulsion. At some critical point, a stream of liquid erupted towards the collector (aluminum foil). A jet of material in the form of fibers dried in flight and finally deposited on the collector. The needle tip and collector have been kept 12 centimeters apart. The rpm of the motor was kept as slow as possible to stop the solution from spoiling. The spinning process is shown in figure 1.



Figure 1: Schematic of the Electrospinning Setup

III. CHARACTERIZATION

A. Fourier Transform Infrared Spectroscopy (FTIR)

The PVA nanofibers characterize through Fourier Transform Infrared Spectroscopy (FTIR) for structural bond analysis. For FTIR analysis, 0.1 mg of the nanofiber treated at 100 0 C and pure PVA powder samples was placed over the universal, diamond ATR of the FTIR. Then, samples were scanned between 500 cm-1 and 4000 cm-1 with 2 cm-1 resolutions.

IR scans of PVA powder and nanofibers are shown in figure 2, The FTIR analysis was used to characterize

groups in PVA nanofibers. Peaks were observed at 3748 cm-1, 3305 cm-1 and 4043 cm-1 represent O-H vibrational molecules 2324 cm-1 corresponds to C-H stretching, 1418 cm-1 shows C-H₂ methylene group, 1092 cm-1 shows C=O stretching and 585 cm-1 corresponds to O-H aromatic compounds as shown in figure 2.



Figure 2: Fourier Transform Infrared Spectroscopy of PVA and PVA Fibers

B. Thermo-Gravimetric/Differential Thermal Analysis (TGA/DTA)

PerkinElmer SII analyzer, USA is used for TGA/DTA analysis. The nanofibers from the collector are separated after drying them in the oven at 40 °C for 10 hours. TGA/DTA analysis of the PVA nanofibers is then performed. TGA/DTA was performed at 100 c/min in static air up to 400 °C to find out the crystallization temperature and possible decomposition rate. The black line shows TGA while the blue line shows DTA. Due to moisture, in the first step, the weight loss is about 12.5 % from 30-75 °C which was due to loss of water molecules [31]. In the second step, weight loss is about 50 % at a temperature of 149 °C to 371 °C, which is the most intense weight loss corresponds to the side chains of polyvinyl alcohol nanofibers [32], the loss of H-bond (hydrogen) between polyvinyl alcohol molecules and Obond (oxygen) between C-O. In the entire process, the total weight loss is about 62.5 % as shown in figure 3.



C. Scanning Electronic Microscope (SEM) The structure and diameter of the AgNFs were

investigated through scanning electron microscopy (SEM), serial numbered JEOL 5910 SEM with a magnification range of 10X-300 K X, and tungsten electronic beam energies in the range 1-40 keV. The diameter of the fibers was calculated through image J software. The size and morphology of temperature-treated PVA nanofibers are investigated. The diameter of nanofibers is less than one micrometer and the sample treated at 100 $^{\circ}C$ shows excellent morphological structure and uniformity in length as shown in figure 4 (a), while nanowhiskers are formed when the nanofibers were treated at 200 $^{\circ}C$ as shown in figure 4 (b).



Figure 4: (a) SEM Images of PVA Nanofibers Treated at 100 ^oC (b) SEM Images of PVA Nanofibers Treated at 200 ^oC

IV. RESULTS AND DISCUSSIONS

A. Humidity Sensor Fabrication

A thin layer of copper is thermally deposited over a ceramic (Alumina) substrate and the device footprint is deposited over the copper layer using a screen printing technique. Screen printed device was etched in a FeCl3 solution for 5-10 minutes until all the unwanted copper is removed. The masking layer was removed by washing the substrate in Sodium Hydroxide (NaOH) solution. Finally, the humidity micro-sensor was fabricated by depositing the PVA nanofibers layer through electro-spinning over the interdigitated electrodes. After the deposition of nanofibers, it was kept in an oven at 100 °C. The distance between the comb fingers and the width of the comb fingers is 0.21 mm.

The length, width, and thickness of the substrate are 14, 7, and 1 mm respectively (0.5 mm Alumina & 0.5 mm Copper layer). The entire process of RH sensor fabrication and schematic of the device is given in figure 5 (a) and figure 5(b).



Figure 5: (a) Schematic of the Fabricated Device (b) Fabrication of the RH Sensor

B. RH Experimental Setup and Performance

The RH test was performed in a laboratory-made humidity test chamber. The RH experimental setup was composed of a DHT11 commercial-grade RH sensor, Arduino UNO ATmega 328 Microcontroller, humidity fire, and a dry nitrogen supply. LABVIEW software and Keysight E4980A LCR meter were used for the data acquisition during the experimental test of the RH sensor shown in figure 6.

The sensor was kept in the RH test chamber. LABVIEW was used to monitor variation in capacitance with respect to % change in RH using the LCR meter. During the experimental analysis, the temperature was 25 °C and the humidity varied from 32-92 % RH. The capacitance increases from 17-45 pf as humidity increases from 32-92 % RH, it is evident from the figures (figure 6 and figure 7) that the sensitivity at lower frequencies increases because the porous and hydrophilic nature of nanofibers facilitates the adsorption of vapor molecules on the surface, therefore, we get high sensitivity at lower frequencies [33]. In this research, PVA nanofibers are used as a sensing material deposited over interdigitated electrodes [34]. Used gold nanoparticles and PVA

composite for the synthesis of capacitive humidity sensor with a sensitivity of 0.05 nf /% RH as shown in figure 7.



Figure 6: Schematic of the Relative Humidity Experimental Setup



Figure 7: Capacitive Response of the Sensor towards % RH Change

In this article, the sensitivity of the sensor is 2.14 pF/% RH at 500 Hz, response and reversibility RH test of the sensor was performed at 500, 1 K, 10 K, and 100 kHz with a response time of 103 seconds and the recovery time is 13 seconds as shown in figure 8.



V. CONCLUSION

In this research, uniform and homogeneous PVA nanofibers were synthesized by a versatile electrospinning technique. For characterization, various

methods have been used for structural, morphological, and thermal behavior that proved the following conclusions.

The synthesized PVA nanofibers were dried at 90 0 C to minimize the diameter. SEM images reveal the diameter of nanofibers in nanometers. FTIR of PVA show peaks at 3748 cm-1, 3305 cm-1,4043 cm-1,2324 cm-1, 1418 cm-1, 1092 cm-1, and 585 cm-1 representing strong chemical bonds. The PVA nanofibers deposited IDE device was used for sensing relative humidity, and it was found to be of a capacitive type. At 48 pF the humidity was 32 % to 92 % RH and the average change in capacitance was 2.14 pF per 1 % change in RH. Response and recovery of the sensor were noted which was 13 seconds (fall time) and 103 seconds (rise time). It was reported that at low frequencies i.e. 500 Hz and 1 kHz the sensor shows excellent behavior towards Relative Humidity.

Finally: it was concluded that the device has a linear and repetitive response, is cost-effective, and is easy to fabricate. Also, it seems from the results that PVA nanofibers exhibit superior sensing properties towards RH.

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Authors Contribution

The authors contributed to this research in the given term; Haroon Ur Rashid did experimental work, data analysis, data compilation, and paper writing. Muhammad Ali supervising, programming, software designing, data analysis. Muhammad Kamran corresponding author, software reviewing, and editing. All authors have read and agreed to the published version of the manuscript.

Conflict of Interest

There is no conflict of interest between all the authors.

Data Availability Statement

The testing data is available in this paper.

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