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PVDF-SPEEK Blend based resistive humidity sensors

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Summary:

We have investigated the effect of sulfonated poly Ether Ether Ketone (SPEEK) on the thermal stability, sensitivity, and hydrophilicity of polyvinylidene fluoride (PVDF) films based resistive humidity sensors. The spin coating technique has been used to deposit PVDF-SPEEK thin film on the ITO interdigited electrode. The surface morphology of the PVDF-SPEEK blend film was studied by Field Emission electron microscopy (FESEM) analysis. The impedance response of the PVDF-SPEEK blend film showed that the addition of SPEEK enhances the sensitivity of the sensing film at a lower humidity levels.

Keywords: Sensitivity, impedance, sensors, poly (Ether Ether Ketone), hydrophilicity

Background, Motivation an Objective

Monitoring and controlling the humidity level is an essential element in various industrial applications. For instance, in electronic and optical device fabrication monitoring of humidity levels are essential [1, 2]. To develop the polymeric humidity sensor which has shorter response and recovery time and exhibits small hysteresis. For this purpose, we investigated polyvinylidene fluoride (PVDF) piezoelectric polymer. PVDF is a polymer that has high thermal stability, excellent electrical properties and highly resistive to the chemicals. Owing to these unique properties of PVDF, many researchers are investigating the PVDF based sensing film for humidity sensing applications [3, 4]. Blending of polymers is a well-known method to enhance the hydrophilicity of the sensing film is to introduce the hydrophilic polar group (SO3H) within the polymer chain [5]. The sulfonation of PEEK occurs by introducing the hydrophilic sulfonic groups (SO3H) within the PEEK. This sulfonation of polymer significantly improves the absorptions of water molecules and proton conductivity which will increase the sensitivity of the humidity sensors [6].
we have investigated the effect of sulfonated poly (Ether Ether Ketone (SPEEK) on the humidity sensing properties of polyvinylidene fluoride (PVDF). The surface morphology of the sensing film was studied by FESEM analysis. The hydrophilicity of the sensing film was studied by the contact angle method.

Description of the New Method or System

The spin coating technique has been used to deposit the PVDF-SPEEK composite blend solution on the ITO/glass electrode (from Osilla). An optimization process for the rotation speed and the rotation time to form an even equilateral spread of the solution were done. The rotation speed and rotation time were optimized to 6000 rpm and 50 seconds. To analyze the effect of different concentration of SPEEK on PVDF-SPEEK blend morphology, hydrophilicity, and humidity sensing characteristics, we prepared 1 wt %, 3wt% and 5 wt% 7.5 wt%, 10 wt% and 15 wt% of SPEEK separately and kept the PVDF concentration constant at 2.5 wt%. The hydrophilicity of the new nanocomposite film was measured by the optical contact angle machine through the SCA software. The morphological analysis to determine the homogenous and surface defect of the blend film is done by the Field Emission Scanning Electron Microscope (FESEM). Whereas the to determine the thermal stability of the composite was done by the TGA analysis. The electrical characterization of humidity sensors was carried out by our previously reported method [7]. Figure1 shows the schematic diagram of preparation of the PVDF-SPEEK blend solution, its deposition via spin coating and the characterization of the sensing film.

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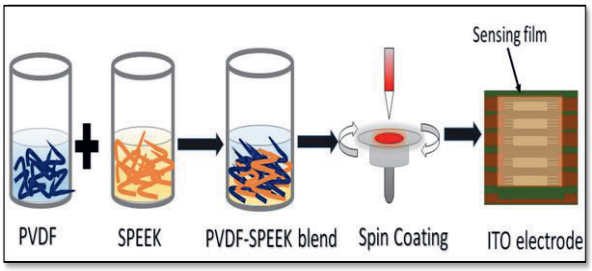


Fig.1. Schematic diagram of the spin coated PVDF-SPEEK resistive humidity sensors.

The electrical response of the fabricated PVDF-SPEEK humidity sensors was conducted in a sealed chamber with a humidifier connected with the chamber. The reference humidity meter was placed inside the sealed chamber. The capacitance was measured by the MS5308 LCR meter with it being clipped to the electrode with the sensing film. Figure 2 shows the set up used in in the electrical response with the inclusive components such as the humidity sensor, LCR meter, humidifier, and the nitrogen Derrite.

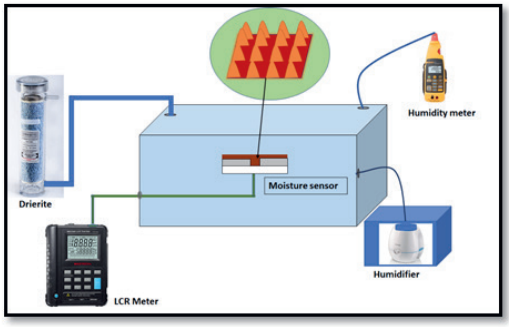


Fig.2. The electrical characterization setup for capacitive based humidity sensor.

Results

Morphological Analysis:

Morphology of the PVDF-SPEEK blend film determines the distribution of SPEEK within the blend film matrix. Morphological study of the PVDF-SPEEK blend film performed by FESEM analysis. Figures 2a and 2b show the FESEM analysis of PVDF-SPEEK blend film with a concentration of SPEEK is 5wt% and 10 wt%. The FESEM analysis reveals that PVDF-SPEEK (5 wt%) blend film has a uniform distribution of SPEEK with film surface is defect free. However As the concentration of SPEEK increases (10 wt%) within the blend membrane the spherical structures appear on the surface of the film. Due to the higher concentration of SPEEK higher amount of sulfonic group (SO3H) presents on the blend membrane which may form the spherical formation [8].

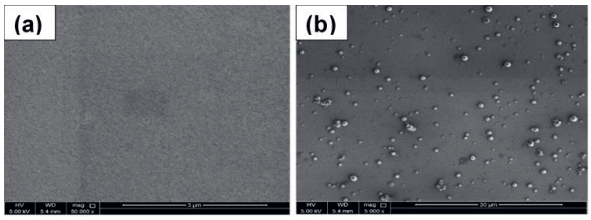


Figure 1. FESEM micro graph of (a) PVDF- SPEEK blend (5 wt%) (b) PVDF-SPEEK (10 wt%) blend film

Thermal Stability Analysis:

The thermal stability of the PVDF-SPEEK blended film was investigated by Thermal gravimetric analysis (TGA).Figure 3 plots the thermal behavior of PVDF-SPEEK blend with different concnetration of SPEEK (1 wt%, 3wt%, 5 wt%, 7.5 wt%, 10 wt% and 15 wt%). TGA analysis reveals that the PVDF-SPEEK blend with lower concentrations of SPEEK is more stable.

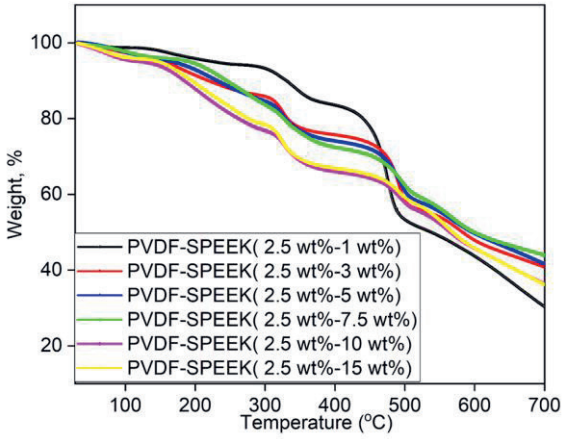
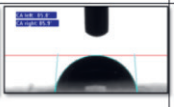
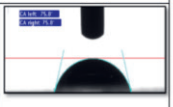


Fig3.Shows the TGA analysis of PVDF-SPEEK composites with the concentration of SPEEK varied from 1wt% to 15 wt%.

Hydrophilicity of PVDF-SPEEK blend

The hydrophilcicity of the sensing film measured by the contact angle method. As the concentration of SPEEK increases in the PVDF-SPEEK blended film the contact angle decreases which is mainly due to the sulfonic acid group present in SPEEK. This increase in hydrophilicity of blended film enhances the sensitivity of the film.Table. 1: Contact angle measurements of PVDF, PVDF-SPEEK (2.5 wt%-1 wt%) composite, and PVDF-SPEEK (2.5 wt%-5 wt%) composite films.

Sample Type	PVDF Film	PVDF- SPEEK (2.5 wt%-1wt%) composite film	PVDF-SPEEK (2.5 wt% - 5 wt%)
Contact angle image			
Contact angle	92.25°	85°	75.8°

Electrical Response:

The PVDF-SPEEK (2.5 wt%-5 wt%) blended film based resistive sensors show high sensitivity, stable response and low hysteresis as compared to pure PVDF film. The developed PVDF-SPEEK (2.5 wt%- 5 wt%) resistive sensors are excellent for sensing low humidity levels. The inset in figure 4 shows the stable and repeatable response and recovery cycle of the PVDF-SPEEK (2.5 wt%- 5 wt%) resistive sensor. The calculated responsr and recovery time are found to be 25s and 40s respectively.

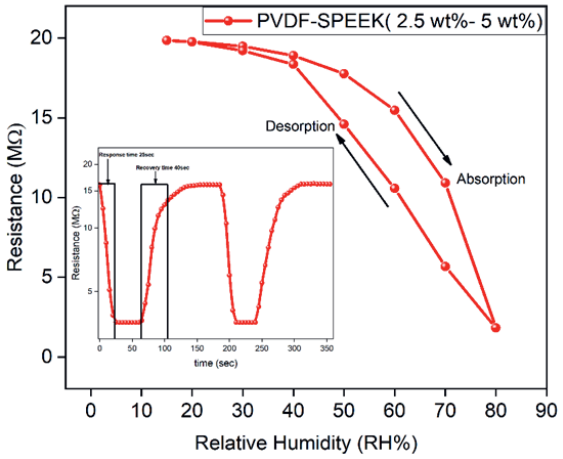


Fig4. Represent the absorption and desorption response of the PVDF-SPEEK (2.5 wt%- 5 wt%) based resistive humidity sensor. Inset shows the response and recovery cycle (40–85% RH) of PVDF-SPEEK (2.5 wt%-5 wt%) based sensor.

Conclusion:

The effect of different concentration of SPEEK on the morphology, thermal stability and electrical response of the PVDF-SPEEK blend was studied. The morphological analysis was achieved by FESEM analysis which established that higher concentration of SPEEK not uniformly blend within the PVDF polymer chain. It can be also observed that as the concentration of SPEEK increases the hydrophilicity of the PVDF-SPEEK blend increase as well. This increase in hydrophilicity of blended film enhances the sensitivity of the film. The PVDF-SPEEK (2.5 wt%-5 wt%) blended film based resistive sensors show high sensitivity, stable response and low hysteresis as compared to pure PVDF film. The response and recovery times of the PVDF-SPEEK (2.5 wt%- 5 wt%) resistive sensor are found to be 25s and 40s respectively. The developed PVDF-SPEEK (2.5 wt%- 5 wt%) resistive sensors are excellent for sensing low humidity levels.

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