



# A dew sensor based on modified carbon black and polyvinyl alcohol composites



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## ABSTRACT

Polyvinylpyrrolidone-grafted carbon black (CB-PVP) was prepared by a free radical polymerization reaction. The resultant CB-PVP could disperse well in water. Dew sensors based on CB/PVA (polyvinyl alcohol) and CB-PVP/PVA were obtained respectively, and their humidity switching properties were researched. Thermal annealing was used to improve the stability of the sensitive films effectively. Both CB/PVA and CB-PVP/PVA sensors showed obvious humidity switching properties, and CB-PVP/PVA sensor performed properties better than CB/PVA sensor. In the case of the 6 wt% CB-PVP/PVA film, the resistance was about 10 k $\Omega$  below 80% relative humidity (RH) and increased sharply over 85% RH, with a resistance of 170 k $\Omega$  at 100% RH. The conductive mechanism of the CB-PVP/PVA materials was discussed through direct current reverse polarity method. The CB-PVP/PVA composite is a promising dew sensing material.

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## 1. Introduction

Resistive-type polymeric humidity sensors, which are mainly based on supporting polymers and conductive materials especially carbon materials, have become one of the most important humidity sensors with advantages of easy preparation, low cost, DC electrical conductivity and simple application circuit [1–4]. Recently, resistive-type polymeric humidity sensors have attracted the attention of many groups [5–9]. For example, Tang et al. have demonstrated resistive humidity sensitivity of polyimide/multiwall carbon nanotube composite films with linear and fast response [5]. Li et al. have reported a resistive-type polymeric humidity sensor based on poly(4-vinylpyridine) and CB with a wide sensing range of 0–97% relative humidity (RH) and a short response time for desiccation process [6]. Also Yu et al. have researched the humidity sensitive behavior of poly(ethyleneimine)/multiwall carbon nanotube composite films with layer-by-layer assembly [7]. What's more, Shim et al. reported that carbon black (CB) doped poly(1,5-diaminonaphthalene) to develop a sensor which showed a straight line over the entire measured humidity range [8]. Generally most of these sensors exhibited a linear response in a wide sensing range. Therefore, switch-type humidity sensors with a nonlinear response such as the dew sensor

need more researches and discussions on the basis of previous works.

Dew sensors made from conductive polymer composites could be used in high humidity environments for condensation watching check. The resistances of dew sensors exhibit drastic change when the materials are exposed to high humidity atmosphere. Composite films of the dew sensors consist of a hydrophilic swelling polymer and conductive particles, generally carbon material [10]. Polyvinyl alcohol (PVA) has a characteristic of hydrophilic swelling and humidity sensors based on PVA as supporting materials exhibit high humidity sensitivity, low hysteresis, good stability and repeatability [11,12]. Carbon black (CB) is a good conductor which has been used to form composites with a number of polymers [13–17]. Chen et al. reported a nonlinear sensing material based on hydroxyethyl cellulose (HEC) and CB by mechanical mixing [17]. However, CB could not disperse well in polymers owing to the agglomeration of CB particles. Hence CB should be modified to enhance the dispersibility of CB particles and decrease the interface action energy between carbon materials and polymer matrices [6].

In this research, hydrophilic polyvinylpyrrolidone (PVP) was grafted onto the CB to obtain water-dispersible conductive material and PVA was chosen as the supporting material to fabricate dew sensor. The grafted PVP on CB surface enhances its dispersibility in aqueous solution, hence mechanical mixing method can be replaced by a solution mixing method to prepare a humidity sensitive film. The humidity switching properties of the sensors based on CB and CB-PVP were investigated and compared, and the sensitive mechanism was also explored.

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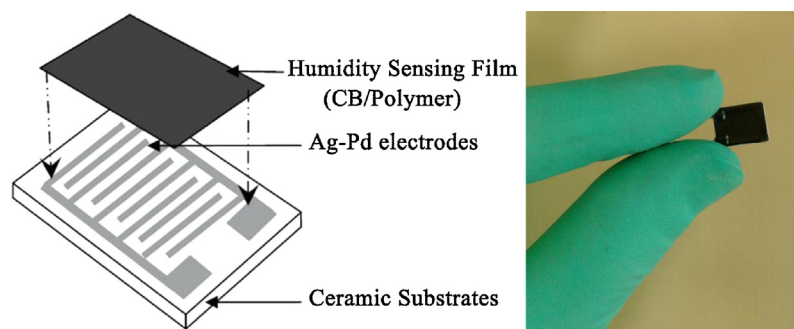


Fig. 1. A schematic diagram of the humidity sensor.

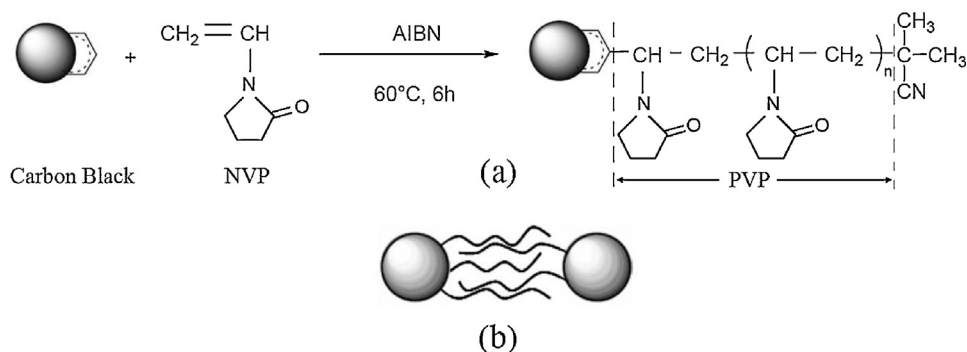


Fig. 2. The reaction equation of CB and NVP (a), and mode of stabilization of CB particles dispersions by grafting PVP (b).

## 2. Experimental

### 2.1. Preparation of sensing materials

BP-2000 CB from Cabot Limited Corporation of USA was selected as the conductive material because of its big specific surface area and small particle size (15–20 nm in diameter). PVA with a degree of polymerization of 1750 and an alcoholysis degree higher than 98% was from Sinopharm Chemical Reagent Limited Corporation, which is insoluble in water at room temperature. *N*-vinylpyrrolidone (NVP) and 2,2'-azobisisobutyronitrile (AIBN) were purchased from Aldrich and Acros and used directly. Tetrahydrofuran (THF) was purified using standard methods. The grafting of PVP onto CB was completed by the following process. A quantity of 1.3 g CB, 5.4 g of NVP, 0.03 g of AIBN and 18 mL of THF were added to a 100 mL flask, and the mixture was heated at 60 °C for 6 h. Then the reaction mixture was centrifuged several times to remove the ungrafted polymer and AIBN. The obtained solids were further purified by soxhlet extraction with THF for 48 h, and then dried under vacuum at 40 °C to afford the desired product.

### 2.2. Sensor preparation

In a typical procedure, appropriate amount of CB-PVP and PVA were added into the conical flask, and then distilled water was added. The mixtures were heated to 75 °C and kept stirring for 2 h to form a stable composite suspension. After being cooled to room temperature, the suspension was casted onto clean ceramic substrate (10 mm × 8 mm × 0.8 mm) with a thickness of about 30 μm, where an interdigitated array of Ag–Pd electrodes had been previously screen-printed as shown in Fig. 1. The ceramic substrate was then dried in air to obtain the humidity sensor. CB and CB-PVP sensors were prepared by similar method by casting the aqueous solution of CB and CB-PVP onto clean ceramic substrate.

### 2.3. Measurements

The microstructures of the films were checked by using a Hitachi H-800 transmission electron microscopy (TEM), with an accelerating voltage of 200 kV at the humidity of about 30% RH. Thermal gravimetric analysis (TGA) was undertaken on a Perkin–Elmer thermal analysis system at a heating rate of 10 °C/min and a nitrogen flow rate of 80 mL/min. Before the testing of humidity sensing behaviors, the sensors were put into a furnace for repeated thermal annealing to improve their stability. The furnace was heated up to 100 °C from room temperature within 10 min, and the temperature was kept for 1 h followed by cooling down naturally. The above thermal annealing was repeated until the resistance of the sensors remained almost unchanged at room temperature. The humidity switching properties of the sensors were investigated by recording the electrical response of the sensors at 1 V DC under different humidities at room temperature (25 °C). The atmosphere of humidities are produced by different saturated salt solutions in their equilibrium states including LiCl for 11% RH, MgCl<sub>2</sub> for 33% RH, Mg(NO<sub>3</sub>)<sub>2</sub> for 54% RH, NaCl for 75% RH, KCl for 85% RH, KNO<sub>3</sub> for 95% RH, and H<sub>2</sub>O for 100% RH at room temperature. The uncertainty of the RH values is about ±1%.

## 3. Results and discussions

In order to improve the dispersibility of CB, PVP was used to modify the CB surface. The chemical synthesis method was obtained from the literature [18]. The grafting of PVP onto CB was achieved by the trapping of PVP growing free radicals formed during the polymerization initiated by AIBN in the presence of CB. The reaction equation of CB and NVP was shown in Fig. 2a, and PVP was grafted onto the surface of CB particles. The resultant CB-PVP not only decreased the interface active energy between carbon materials and polymer matrices but also enhanced the stable dispersions of CB particles as shown in Fig. 2b.

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