

# Effect of polymerization time on the humidity sensing properties of polypyrrole

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## Abstract

In this paper, we prepared polypyrrole (PPY) films using a chemical method at different polymerization times. Their structure and morphology were characterized through X-Ray powder diffraction (XRD), IR, and scanning electron microscope (SEM). The humidity sensors were made by a coating membrane method. We studied the humidity sensing properties of the PPY-*x* films (the letter 'x' denotes the polymerization time and it was 4, 24, or 96 h). It was found that the humidity sensing properties of PPY could be improved greatly by extending the polymerization time. According to our experiment results, we predict that the humidity sensing properties of conducting polymers could be improved by increasing their crystallinity degree.

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**Keywords:** Polypyrrole; Humidity sensitivity; Polymerization time

## 1. Introduction

In recent years, conducting polymers have been paid more attention due to their potential application values in microelectronics, microsystems, bio-medical applications, optical sensors, photoelectronic chemistry, and biosensors [1–5]. Synthesis and properties of various conducting polymers, such as polypyrrole (PPY) [6], polyamine [7] and polythiophene [8], have been reported largely. Compared with other conducting polymers, PPY has relative high conductivity and environmental stability. So it has become to be the focus of recent researches. The work to improve the conductivity of PPY by doping [9] or chemical modifying [10] has been made great progress. Some work has been made to study the humidity sensing properties (HSP) of the materials based on PPY [11,12]. However, as far as we know, the effect of polymerization time on the HSP of PPY has not been reported. Here, we synthesized PPY with different polymerization times using a chemical method, and studied the effect of polymerization time on the HSP of the PPY. It was found

that the HSP of PPY can be improved greatly by extending the polymerization time.

## 2. Experiment

### 2.1. Preparation of polypyrrole

A hydrated ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) was used without any purification as an oxidant for the chemical polymerization of pyrrole. Pyrrole was distilled under reduced pressure before use. Anhydrous ethanol was used as a solvent for the PPY synthesis. PPY was obtained by chemically synthesis. The process was as follows: 3.4 ml of pyrrole was dissolved in 25 ml of ethanol, and stirred for 10 min. Twenty five millilitres of a  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  ethanol solution ( $0.36 \text{ mol l}^{-1}$ ) was added dropwise to the ethanol solution of pyrrole. The mixed solution was magnetic stirred for different times at room temperature. The resulted solution was filtered and washed three times with ethanol, then dried in a vacuum box overnight. The resulting samples were designated as PPY-*x* (the letter 'x' denotes the polymerization time and it was equal to 4, 24, or 96 h).

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## 2.2. Methods of characterization

X-ray powder diffraction (XRD) patterns were collected on a Siemens D-5005 diffractometer (Siemens AG, Karlsruhe, Germany) using Cu  $K\alpha$  radiation at 40 kV and 30 mA. Infrared spectra were taken on a Perkin-Elmer series with a resolution of  $4\text{ cm}^{-1}$  and the samples were prepared in a form of KBr pellet with a thickness of about 1.3 mm. Each spectrum was collected at room temperature under atmospheric pressure. The morphology of the PPY products was characterized by a JEOL JSM-6700F field emission scanning electron microscope (SEM), and the samples were prepared by distributing the powder samples on a double-sided conducting adhesive tape. The variation curve of impedance as a function of relative humidity was measured on a ZL-5 model LCR analyzer at room temperature, 1 kHz, and 1.0 V. The preparation of humidity sensors was similar to the method in literature [13]. Briefly, the sample was mixed with a small amount of ethanol to form paste, and then the paste was coated on a ceramic plate ( $1 \times 0.5\text{ cm}^2$ ) on which interdigitated gold electrodes were printed and heated. A protecting film of cellulose was coated on the surface of the sensing film. The schematic image of this electrode was shown in Fig. 1. The controlled humidity environments were achieved using saturated aqueous solutions of different salts: LiCl,  $\text{MgCl}_2$ ,  $\text{Mg}(\text{NO}_3)_2$ , NaCl, KCl, and  $\text{KNO}_3$  in a closed glass vessel at ambient temperature, which yielded 11, 33, 54, 75, 85 and 95% relative humidity, respectively. The measurement of humidity sensing properties was carried out by placing a humidity sensor in a glass vessel with a special relative humidity and keeping it for 2 min; within this time, the value of impedance could obtain a stable state, that is, the impedance did not change basically any longer.

## 3. Result and discussion

### 3.1. IR spectroscopy

Fig. 2 shows the IR spectra of the PPY- $x$ . The polymerization times of curves  $a$ – $c$  were 4, 24, and 96 h, respectively. As can be seen from Fig. 2, the feature of the three curves is almost the same as each other, indicating that the structure of PPY was not changed by extending its polymerization time. The peak at  $786\text{ cm}^{-1}$  is characteristic of the C–H out-of-plane bend-

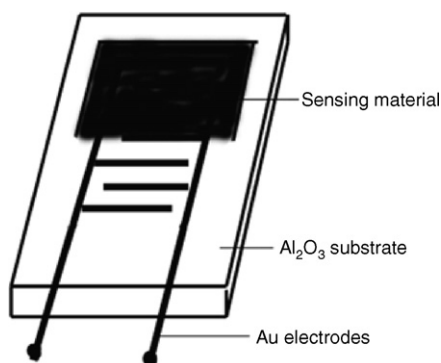


Fig. 1. Schematic drawing of the humidity sensor.

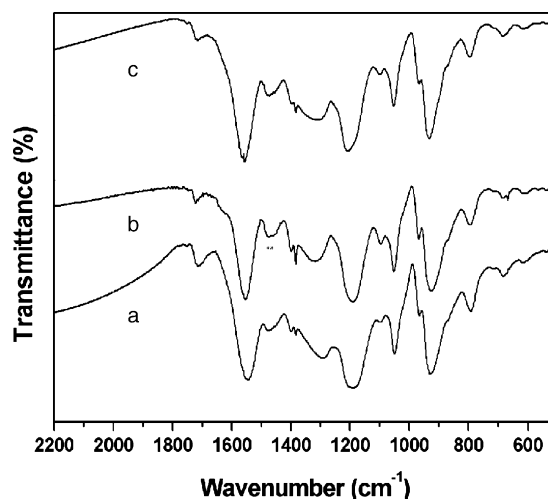


Fig. 2. IR spectra of (a) PPY-4, (b) PPY-24, and (c) PPY-96.

ing mode. The band assigned to the C–H in-plane deformation vibration of pyrrole rings appears at  $1050\text{ cm}^{-1}$  [14]. The bands observed at  $1474$  and  $1189\text{ cm}^{-1}$  represent the C–N stretching vibration, and this is in agreement with literature [15]. The peak appeared at  $1554\text{ cm}^{-1}$  comes from the C=C stretching vibration. The weak band around  $1705\text{ cm}^{-1}$  is normally attributed to the C=O stretching, which suggests that the pyrrole rings were slightly overoxidized [14,15]. The bands at  $1297$  and  $926\text{ cm}^{-1}$  may be assigned to the doped bands.

### 3.2. X-ray diffraction

Fig. 3 was the XRD spectrum of PPY- $x$ . The polymerization times of curves  $a$ – $c$  were 4, 24, and 96 h, respectively. As can be seen, there was a broad peak for every curve, which can be assigned to the interchain spacing of PPY [14]. In addition, we can see that the  $2\theta$  angle of the broad peak shifted to the higher angle side (from  $24.3^\circ$  to  $25.5^\circ$ ) with an increase in polymerization time, and the corresponding interchain spacing of PPY

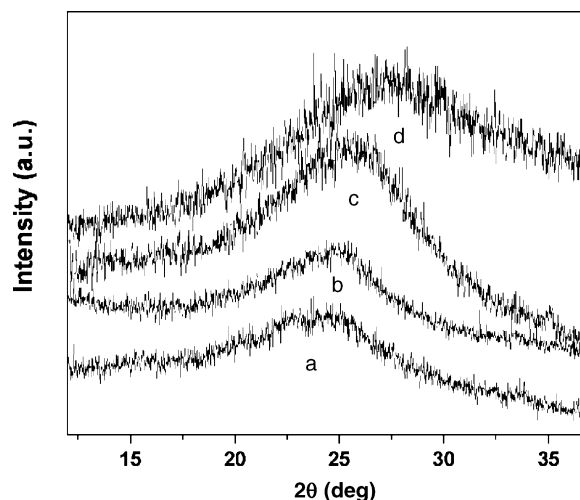


Fig. 3. XRD patterns of (a) PPY-4, (b) PPY-24, (c) PPY-96, and (d) PPY-96 exposed to high moisture.

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