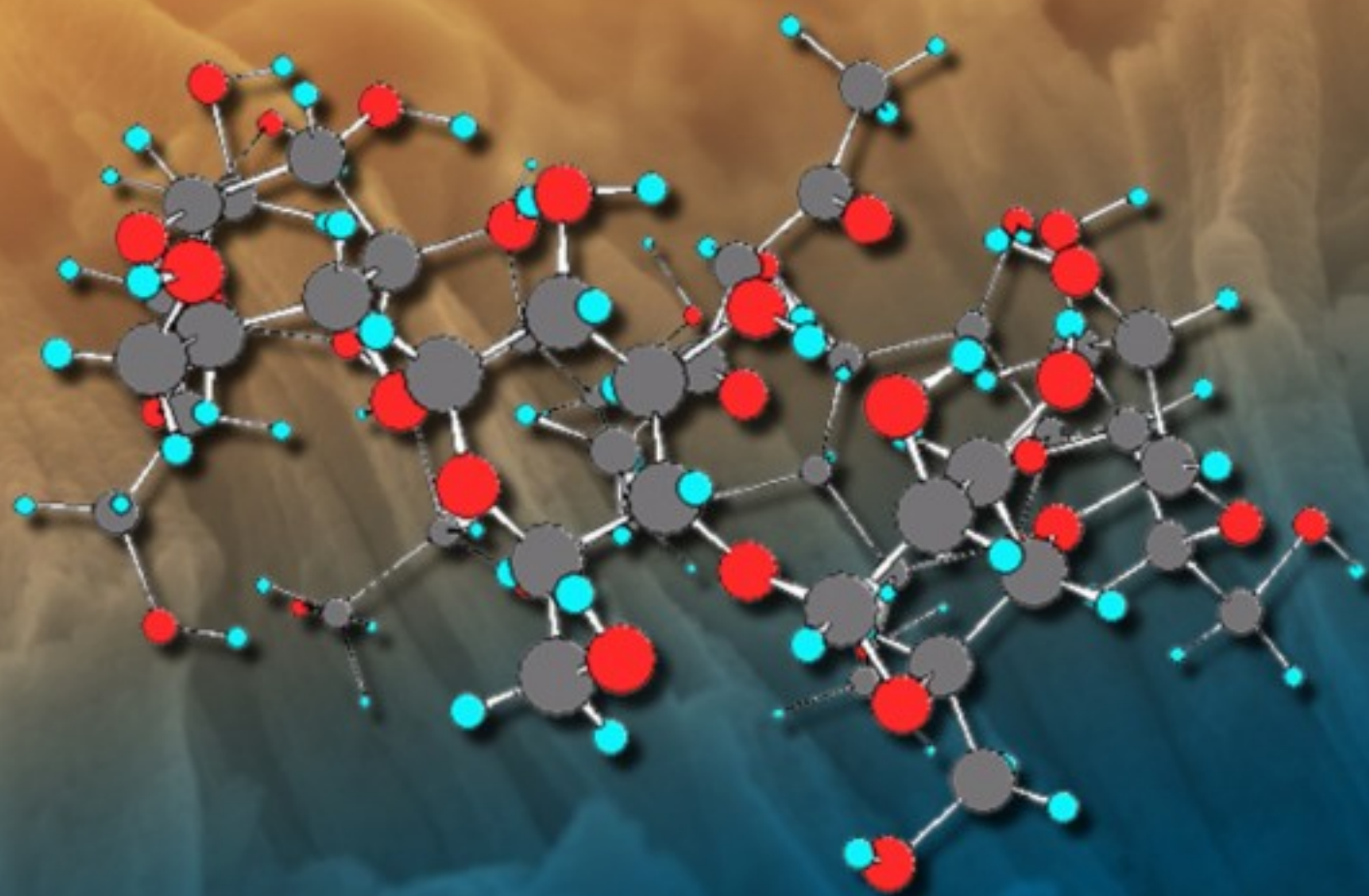


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Zinc and Pyrrole-added Akaganeite (β -FeOOH) Films by Ultrasonic Spray Pyrolysis Assessed as Propane Sensors

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Abstract: β -FeOOH films were grown by ultrasonic spray pyrolysis from a 0.05M methanolic solution of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ containing $(\text{CH}_3\text{COO})_2\text{Zn} \cdot 2\text{H}_2\text{O}$, added with distinct amounts of pyrrole monomer (0, 0.014 ml and 2.5 ml). Films were grown at low substrate temperatures to preserve the organic component in the film. They were characterized by using x-rays diffraction (XRD), infrared spectroscopy (FTIR), and atomic force microscopy (AFM). To assess their sensing capabilities, impedance spectroscopy (IS) was used. Results indicate that at low substrate temperature (approximately ≤ 150 °C) the films keep both, organic and akaganeite composition but the largest (2.5 ml) amount of pyrrole clearly produces a polypyrrole-akaganeite composite. Zinc acetate was not completely decomposed at the low deposition temperatures used. The electrical measurements were made under different atmospheres such as dry air, humid air and humid air-propane (500 ppm) at 30 °C and 50 °C. A better response towards humidity (~ 100 %) and propane (~ 1000 %) was obtained from akaganeite films at both temperatures. Lower but not negligible responses (10-40 % for humidity and 20-30 % for propane) were found from Ppy-akaganeite films. *Copyright* © 2012 IFSA.

Keywords: Polypyrrole (Ppy), Akaganeite, Sensors, Ultrasonic spray pyrolysis.

1. Introduction

The blend between organic and inorganic materials in the form of films is by now very common in exploration of materials properties. This idea is basically related to that when a semiconductor is intentionally doped with another constituent to change its properties. Consequently, much effort has been directed towards obtaining mixed materials with new properties, which allow them good performance in different applications. These materials are largely being used in multiple disciplines

such as biosensors, sensors, catalyst materials and corrosion process studies [1-3]. Unfortunately, so far the use of polymers has often been impeded by their delicate handling. For example, many polymers work at low temperature processes which place them in disadvantage with respect to classical semiconductor materials. Some other times, this fact becomes an advantage. A classical semiconductor employed as a sensor device needs to support the oxidation from air and other reactions that can change the original properties of semiconductor to another compound or produce a change in stoichiometry. Therefore, this is the great interest for trying to get the best combination properties between polymers and classical semiconductor materials. In order to reach the best mixing properties not only the methods and conditions involved during the material preparation but also the good selection of component materials become important. One of the most used polymers is polypyrrole due to the good stability of its properties and because it can be easily synthesized as a homopolymer and also as a composite. The success of polypyrrole applications depends on the improvement of the properties and the processability of this material. Therefore one of the main research goals is the correlation between the synthesis parameters and the molecular architecture of Ppy in order to obtain the required properties for specific application.

In this work we report some structural, optical, morphological and electrical properties of zinc-polypyrrole-iron oxyhydroxide (Ppy-FeOOH) films, studied by using XRD, FTIR spectroscopy, and AFM microscopy. In order to assess their potential as gas sensors, Impedance spectroscopy (IS) measurements of the films under different atmospheres were also carried out.

2. Experimental

2.1. Methanolic Solution Preparation

0.05 M methanolic solutions of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ which is considered by many authors as an oxidative agent for polymerization process of pyrrole monomer [2], were used. $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ was dissolved in methanol. The adequate amount of $(\text{CH}_3\text{COO})_2\text{Zn} \cdot 2\text{H}_2\text{O}$ was added to achieve 3 % at. Zn content in the solution, that was stirred during 10 min. 250 ml of solution were always prepared as a reference volume to add the monomer. Then the pyrrole monomer was poured into the same beaker. Finally, the whole solution is stirred continuously during 10 min. The color changed from clear yellow to black, when polymerization was taking place.

2.2. Substrate Preparation

Glass substrates were used to deposit the films. They were cut in pieces of 2 cm × 2 cm size. The cleaning was made leaving the glass substrates in an Extran-detergent solution to remove any content of grease. Then, they rinsed in deionized (DI) water with ultrasonic cleaning until detergent was eliminated. Afterwards, rinses in acetone and DI water were performed.

Silicon substrates were also used due to their transparency to the IR radiation. Substrates of 1.5 cm × 1.5 cm were cut. The cleaning process starts with Xylene for 5 min. Then ultrasonic rinses during 5 minutes in acetone and DI water follow. Finally, both glass and silicon substrates were kept in alcohol and prior to use they were dried with nitrogen gas.

2.3. Sample Preparation

In order to deposit the thin films, a variety of techniques can be employed. In our case, the precursor solution is atomized by means of an ultrasonic device and carried out by an inert gas (nitrogen) flux.

The substrate is supported upon a stainless steel heater at a desired temperature from 50 °C to 150 °C. 0 ml, 0.014 ml and 2.5 ml of pyrrole monomer (Py) were added to 250 ml of iron chloride solution. The deposition time was 5 min and a gas flow of 5.9 l/min for all samples was kept constant.

3. Results and Discussion

3.1. FTIR Spectroscopy Analysis

We can see in Fig. 1(a) and b) that both deposited temperatures have very similar spectra for 0, 0.014 and 2.5 ml of pyrrole monomer aggregated to each film. For the film without pyrrole monomer (spectra at the bottom of both figures (a) and (b)) we observe the existence of only an oxyhydroxide material identified by its IR bands localized in 874, 682 and 413 cm^{-1} for the film grown at 50 °C. For the one at 150 °C its IR bands are shifted as shown in Fig. 1(b) and for this case they are in 864, 676 and 413 cm^{-1} . This oxyhydroxide material is usually called akaganeite. The water contained in the films is revealed by the infrared bands in 1635 cm^{-1} and also by the very broad bands corresponding to the O-H hydroxyl region.

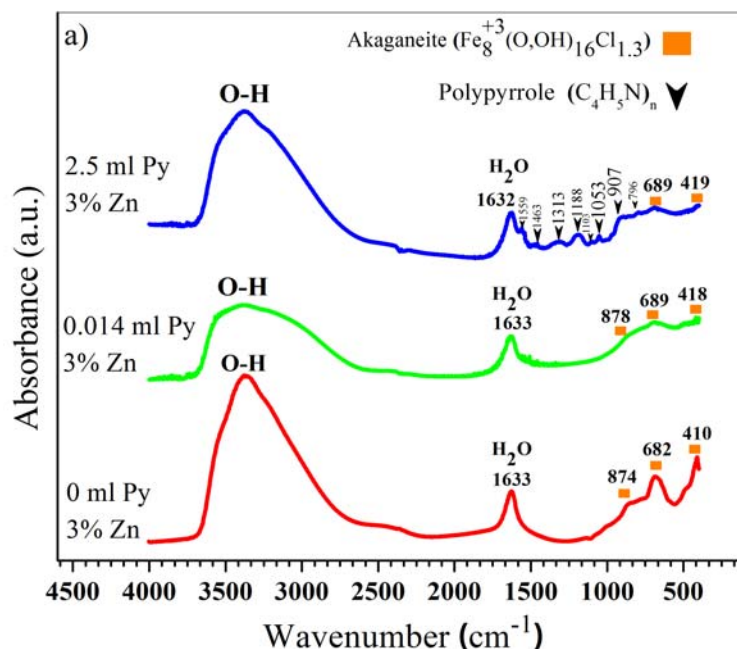


Fig. 1 (a). Illustrates the IR bands of Zinc-Oxyhydroxilic (akaganeite) films added with 0, 0.014 and 2.5 ml of pyrrole monomer (Py) for 50 °C.

There are not IR bands related to the organic material in the films with 0.014 ml of pyrrole. The intensity of the bands related to water for the film at 150 °C is decreased as the film starts losing water due to the higher deposition temperature. For the sample with 2.5 ml of pyrrole the film shows IR bands that correspond to a polypyrrole-akaganeite structure for both deposited temperatures (50 °C and 150 °C). Most of the polypyrrole IR bands are approximately in the 1630-670 cm^{-1} region. Fig. 1(a) depicts the polypyrrole IR bands that are localized in 1559, 1463, 1313, 1188, 1103, 1053, 907 and 753 cm^{-1} . In 1(b) they are in 1561, 1474, 1329, 1198, 1052, 921 and 798 cm^{-1} [3,4]. The akaganeite phase is evidenced by its bands in 689 and 419 cm^{-1} for 50 °C and 683 and 413 cm^{-1} for 150 °C [5]. In these spectra, no evidence of the Zn content is apparent. These results confirm the presence as separated phases of both kinds of materials (inorganic and organic materials) in the thin film deposited by ultrasonic spray pyrolysis.

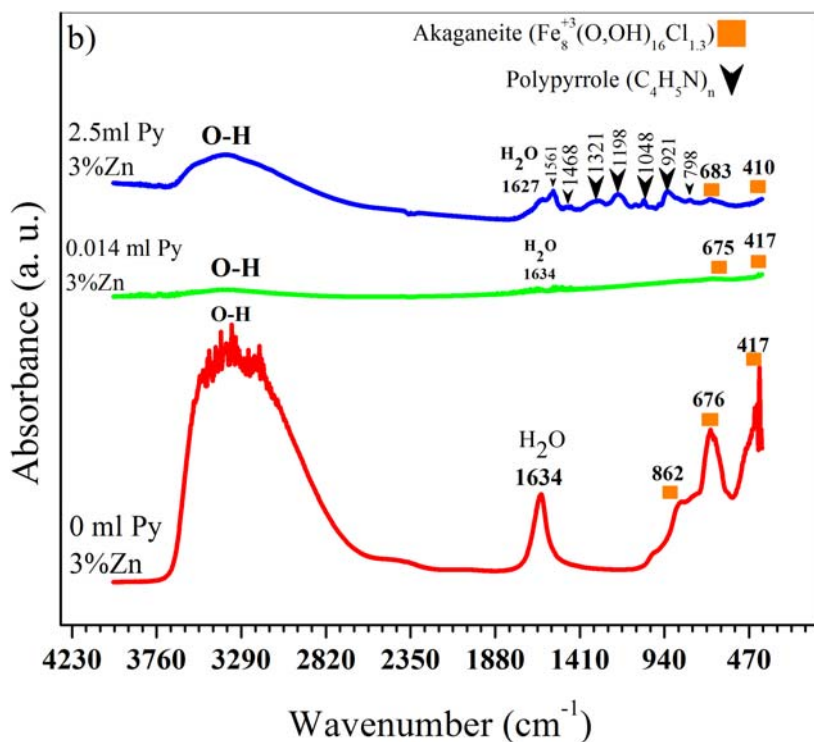


Fig. 1(b). Illustrates the IR bands of Zinc-Oxyhydroxide (akaganeite) films added with 0, 0.014 and 2.5 ml of pyrrole monomer (Py) for 150 °C.

3.2. XRD

Figs. 2(a) and (b) show the X-ray diffraction spectra of samples at the same two different deposition temperatures. Bands corresponding to the akaganeite phase dominate in the films added with 0, 0.014 and 2.5 ml amounts of pyrrole monomer. On both deposition temperatures we can see basically the same trend as the amount of pyrrole monomer increases. The free-pyrrole monoclinic akaganeite film at 50°C exhibits the main diffraction peaks in 11.8, 15.7, 26.6 and 34.3 degrees (00-042-1315, JCPDS).

That at 150 °C has the diffraction peaks in $2\theta = 11.9, 15.6, 26.5$ and 34.3 degrees. When the amount of pyrrole is 0.014 ml, a diffraction peak starts to appear at $2\theta = 23.3$ degrees. This peak makes evident the existence of some organic components in the akaganeite film. When pyrrole is 2.5 ml the peak in 23.5 degrees increases its intensity and width and the peaks corresponding to the akaganeite phase decrease their height. In the sample with 2.5 ml of pyrrole, the polypyrrole structure starts to appear together with the akaganeite oxyhydroxide phase, in accordance with IR results. Two small peaks corresponding to degraded Zn acetate are seen in the samples without pyrrole. In the other cases, one of these peaks is surpassed by that introduced by pyrrole. On the other hand, these results show clearly that an amorphous-like polypyrrole structure begins to dominate upon a polycrystalline akaganeite phase.

3.3. Surface Morphology

In this section we observe the morphology evolution of the akaganeite films containing different amounts of pyrrole monomer and distinct growth temperatures. The group of Figs. from 3 to 8 show the images upon areas of $5 \times 5 \mu\text{m}^2$. Clear differences among these images of the surface morphologies are illustrated.

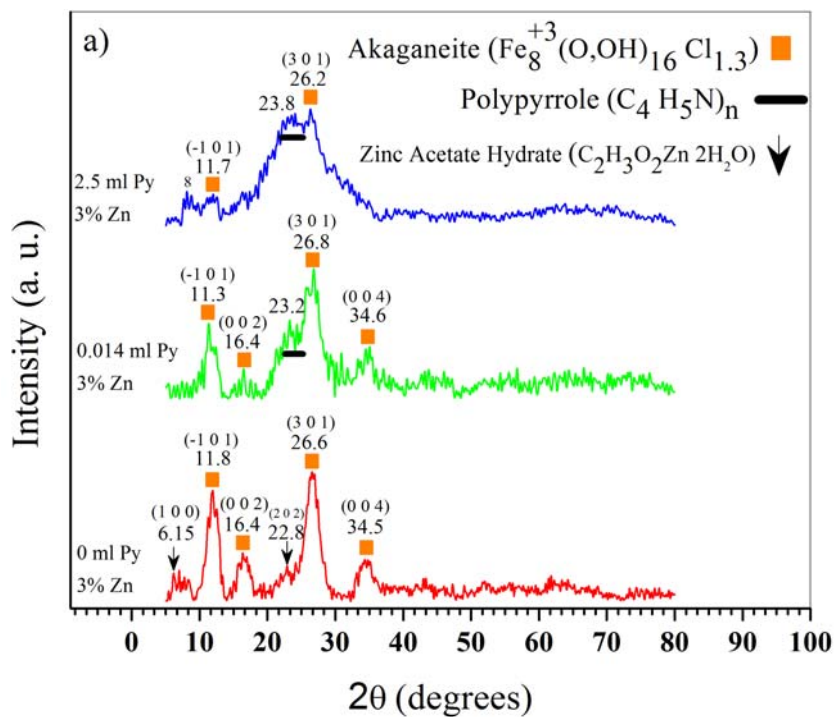


Fig. 2(a). Diffraction peaks of Zinc-Oxyhydroxide (akaganeite) films added with 0, 0.014 and 2.5 ml of pyrrole monomer (Py) for 50 °C.

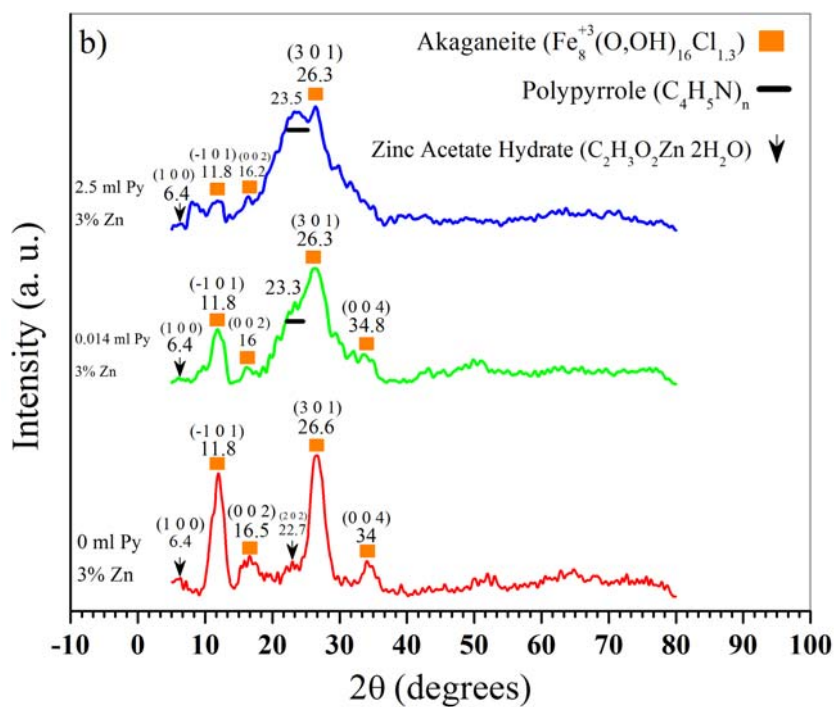


Fig. 2(b). Diffraction peaks of Zinc-Oxyhydroxide (akaganeite) films added with 0, 0.014 and 2.5 ml of pyrrole monomer (Py) for 150 °C.

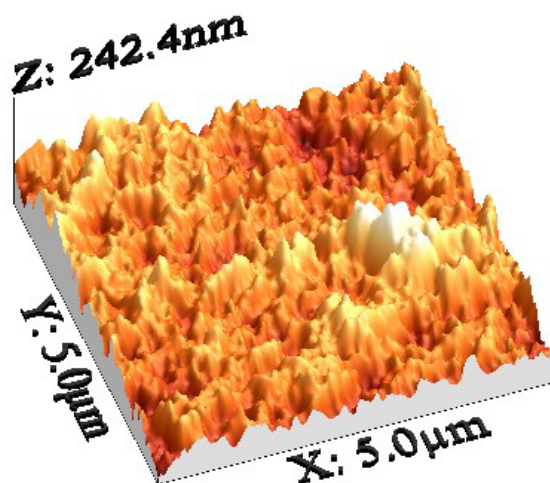


Fig. 3. Film of free-pyrrole akaganeite phase grown at 50 °C upon a glass substrate.

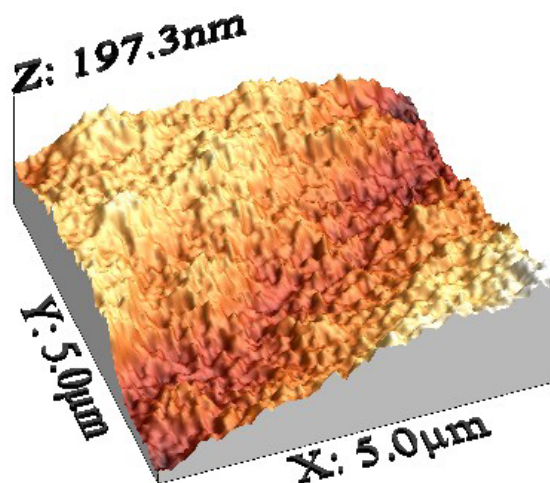


Fig. 4. Film of free-pyrrole akaganeite phase grown at 150 °C upon glass substrate.

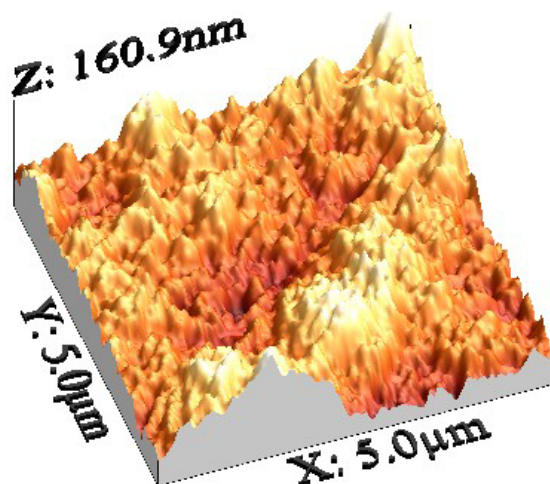


Fig. 5. Film of pyrrole-akaganeite phase added with 0.014 ml of pyrrole and grown at 50 °C upon glass substrate.

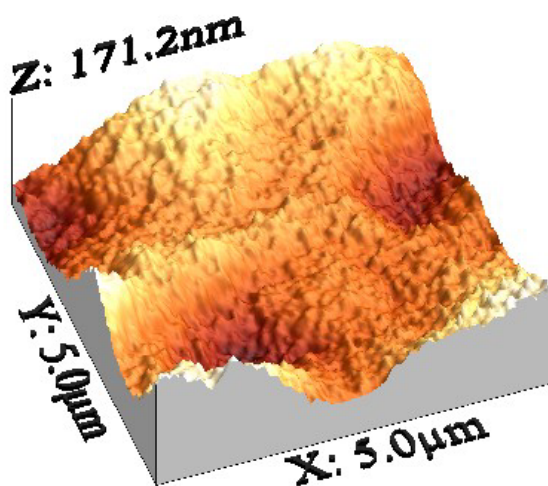


Fig. 6. Film of pyrrole-akaganeite phase added with 0.014 ml of pyrrole and grown at 150 °C upon glass substrate.

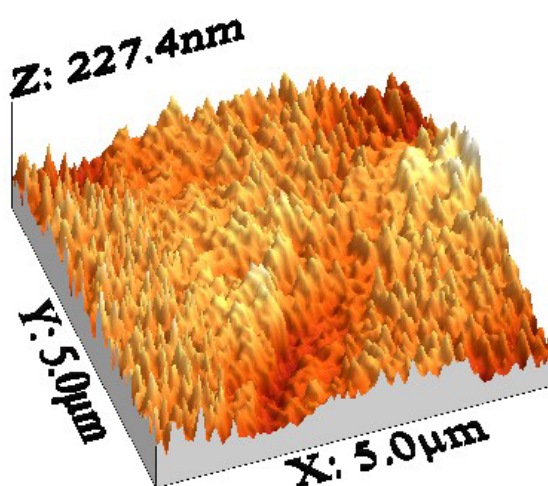


Fig. 7. Film of polypyrrole-akaganeite phase added with 2.5 ml of pyrrole and grown at 50 °C upon glass substrate.

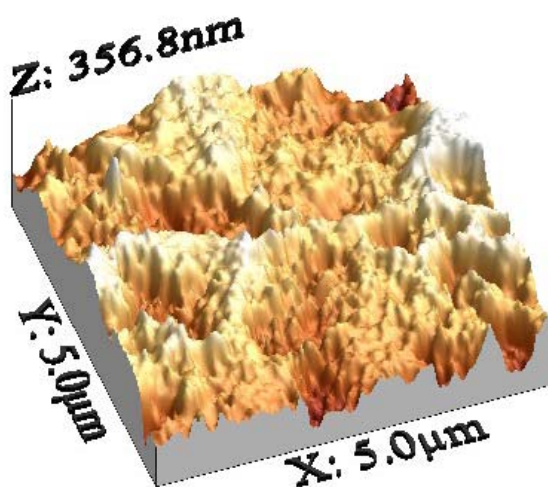


Fig. 8. Film of polypyrrole-akaganeite phase added with 2.5 ml of pyrrole and grown at 150 °C upon glass substrate.

The RMS roughness of the akaganeite films in the Figs. from 3 to 8 are shown in Table 1.

Table 1. RMS roughness of films in the Figs. from 3 to 8.

Film	RMS roughness (nm)	
	50 °C	150 °C
Akaganeite	32	37.3
0.014 ml Py-Akaganeite	22.5	36.7
2.5 ml Py-Akaganeite	27.5	56.7

The increase of the growth temperature increases the RMS roughness for the three kinds of material. For each temperature, addition of a small amount of pyrrole first decreases roughness and adding a larger amount increases it.

3.4. Impedance Spectroscopy (IS)

Electrical measurements were done under controlled atmospheres by impedance spectroscopy for the pure akaganeite and polypyrrole-akaganeite films for a comparison of their electrical responses towards dry air, humid air and humid air-propane (500 ppm) atmospheres. The experiment consisted of measuring the impedance of each sample under the above mentioned atmospheres by doing a frequency sweep between 1 Hz and 100 kHz.

In Fig. 9 we see electrical impedance plots (Nyquist diagrams) of a pure akaganeite phase. We compare the electrical impedances of the akaganeite films under different atmospheres at two temperatures: 30 °C and 50 °C. These temperatures are expected not to change the films structure. The Fig. 9a) represents the electrical measurement at 30°. The electrical impedance has the shape of an incomplete semi-arc for the whole frequency region studied (1 Hz-0.1 MHz).

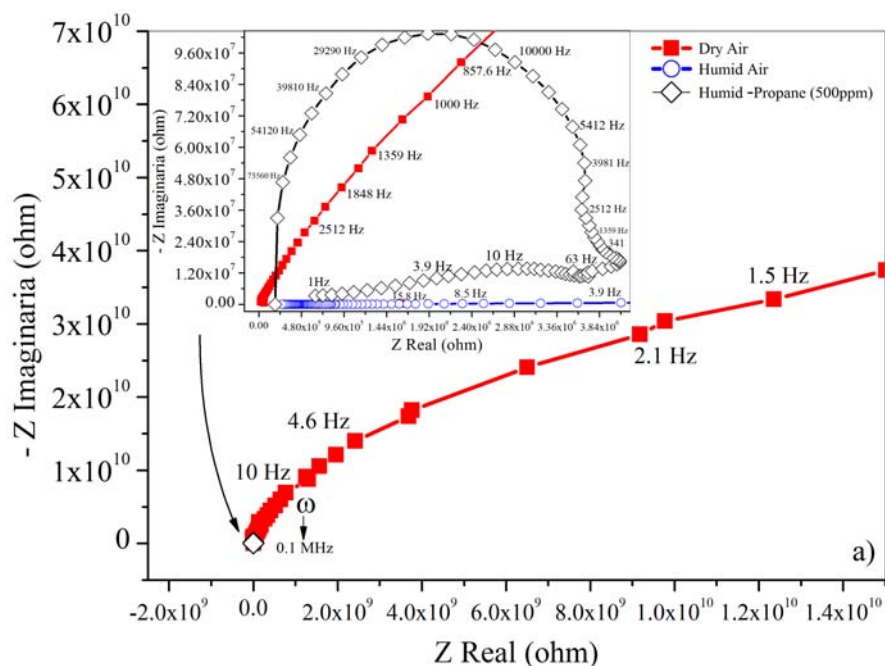


Fig. 9(a). Impedance Spectroscopy of pure akaganeite phase, $\text{Fe}_8^{+3}(\text{O},\text{OH})_{16}\text{Cl}_{1.3}$, in dry air, humid air and humid-propane (500 ppm) atmospheres at 30 °C.

At 1 Hz the electrical impedance acquired very high values, approximately $Z_{\text{Imag}}=3.8 \times 10^{10} \Omega$ and $Z_{\text{Real}}=1.5 \times 10^{10} \Omega$, the akaganeite film being a very resistive material. As the frequency increases towards 0.1 MHz, these impedance values decrease. Under humid air the impedance is reduced drastically in a manner that it is no longer visible in the scale corresponding to the dry air case. In the upper left of the Fig. 9(a) it is shown the impedance spectra corresponding to the next two atmospheres: humid air and humid-propane (500 ppm). In this figure a capacitive semi-arc in the region of high frequency (3.981 kHz to 0.1 MHz) occurs, while for the lower frequency region (1 Hz to 2.512 kHz), a depressed capacitive semi-arc in the 1 Hz to 63 Hz frequency range appeared. In Fig. 9(b) a similar behavior as in the former measurement can be observed.

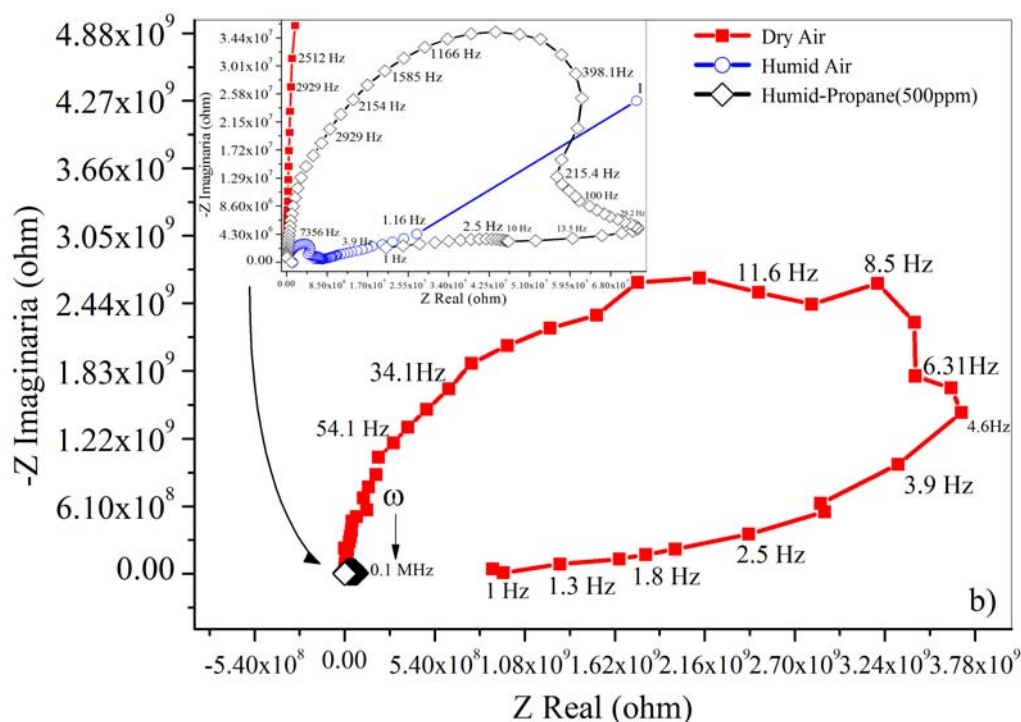


Fig. 9(b). Impedance Spectroscopy of pure akaganeite phase, $\text{Fe}_8^{+3}(\text{O,OH})_{16}\text{Cl}_{1.3}$, in dry air, humid air and humid-propane (500 ppm) atmospheres at 50 °C.

Figs. 10 (a) and (b) show the electrical impedance of polypyrrole-akaganeite films measured again at 30 °C and 50 °C respectively. Curves corresponding to the dry air, humid air and humid-propane (500ppm) atmospheres are shown. We observe that the aspect of their electrical impedances has changed for both temperatures and for all the atmospheres applied.

Clear effects from humidity and propane on the films impedance can be seen in both figures.

The response magnitude towards both, humidity and humid propane atmospheres is calculated with the usual expression:

$$S = \left| \frac{Z' - Z}{Z} \right| \quad (1)$$

where Z' is the impedance under the atmosphere of interest and Z corresponds to the reference one.

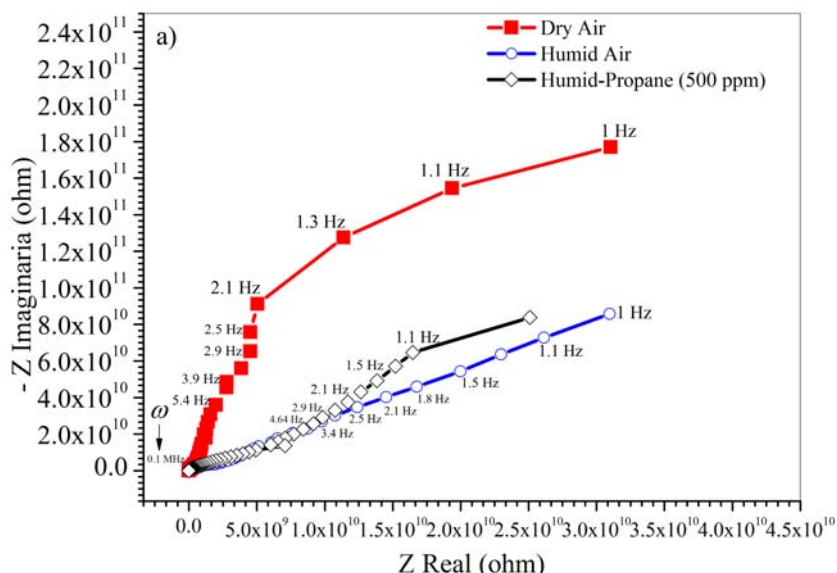


Fig. 10(a). Impedance spectroscopy of polypyrrole-akaganeite film in dry air, humid air, humid-propane (500 ppm) at 30 °C.

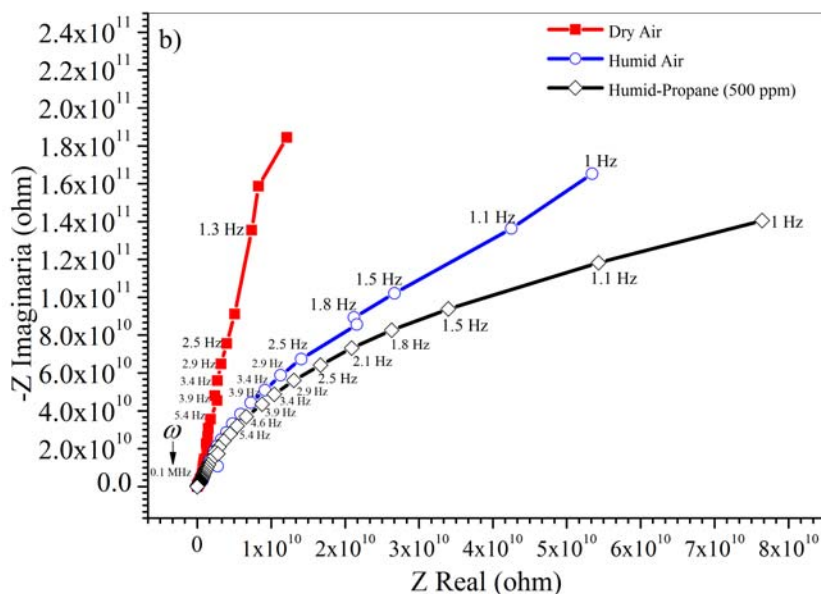


Fig. 10(b). Impedance spectroscopy of polypyrrole-akaganeite film in dry air, humid air, humid-propane (500 ppm) at 50 °C.

For the humid air calculation, we use dry-air as the reference and for propane we used the humid air atmosphere as the reference. After applying such conventions, the plots in Fig. 11 were obtained. In these plots the responses as functions of the measurement frequency are shown. Fig. 11(a) illustrates responses at 30 °C. Fig. 11(b) shows responses at 50 °C.

In these figures the heavy symbols correspond to the akaganeite sample and the empty ones to the ppy-akaganeite sample. The squares describe the response to humidity and the circles the response to propane. Responses of 100 % to humidity can be seen at both temperatures in most of the frequency range for the akaganeite film. The ppy-akaganeite film exhibits a lower response towards humidity in the whole frequency range at both temperatures of measurement but it is still larger than 30 % for 30 °C and larger than 10 % for 50 °C. Regarding propane, a large, about 800 % response is achieved by the akaganeite film near 5 kHz frequency at 30 °C and about 1000 % between 300 and 400 Hz at

50 °C. On the other hand, the ppy-akaganeite sample response towards humidity is ~ 30-40 % and 30 % towards propane at 30 °C while it is 10-20 % towards humidity and ~ 20 % (1 Hz) at 50 °C. Although these figures are lower than those for akaganeite, they are not negligible.

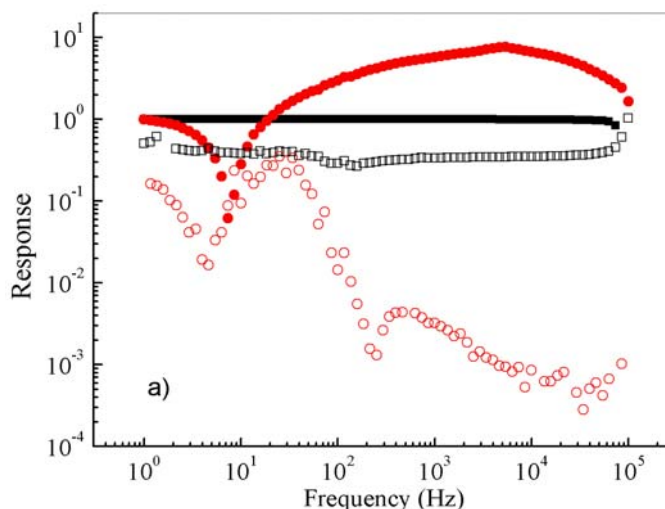


Fig. 11(a). Responses towards humidity and propane at 30 °C. The heavy symbols correspond to the akaganeite sample and the empty ones to the ppy-akaganeite sample. Squares indicate response towards humidity and circles towards propane.

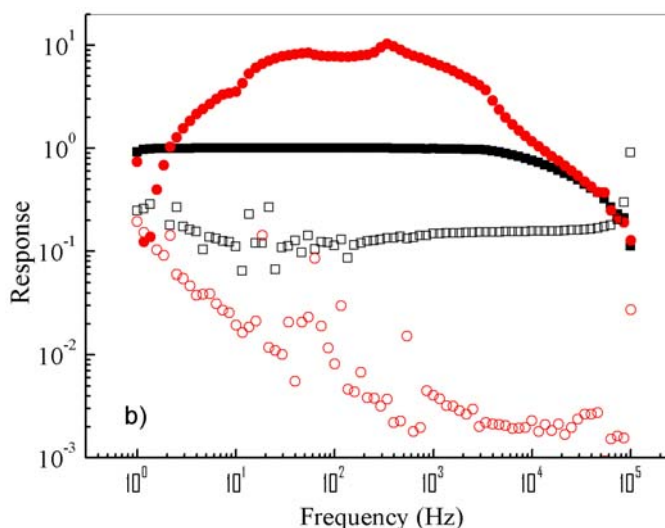


Fig. 11(b). Responses towards humidity and propane at 50 °C. The heavy symbols correspond to the akaganeite sample and the empty ones to the ppy-akaganeite sample. Squares indicate response towards humidity and circles towards propane.

4. Conclusions

Polypyrrole (Ppy) - iron oxyhydroxide (akaganeite) composite films could be prepared by a simple method like ultrasonic spray pyrolysis. $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ was used both as a source of the iron oxyhydroxide (akaganeite) and oxidant agent for the pyrrole monomer in methanolic solution. The polypyrrole-iron oxyhydroxide composite is obtained when the amount of pyrrole monomer is about 2.5 ml in 250 ml of salt solution, at low substrate temperatures (≤ 150 °C). Zinc acetate reagent added

to include zinc oxide/hydroxide to the film was not completely decomposed at these low temperatures. Results indicate a better response of akaganeite films towards humidity and propane at both 30 °C and 50 °C.

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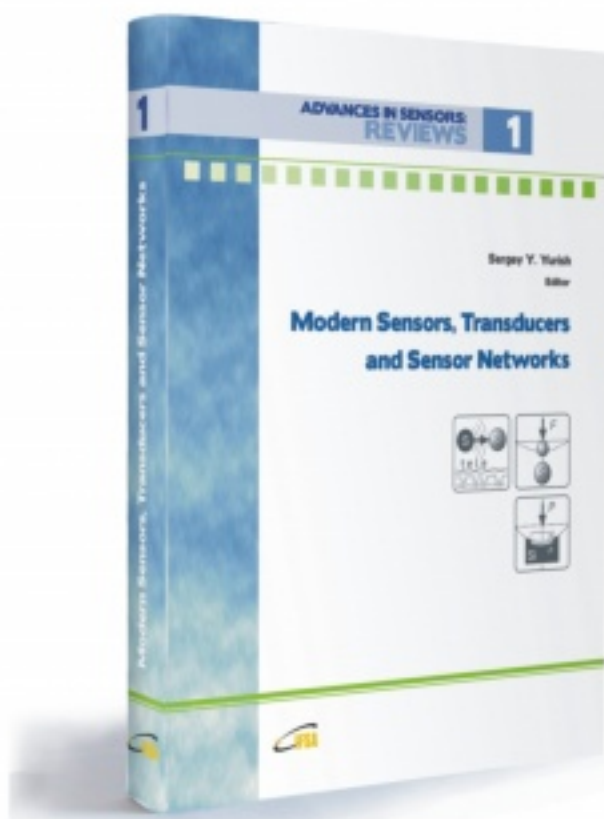
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