

Optical and Electrical Characterization of Poly(3-Decylthiophene) Thin Films: Chloroform Sensor

André V. S. Simões^a , Nyara D. Ferreira^a , Maria E. R. S. Medina^a , Luiz A. R. Junior^a ,
Marcelo S. Borro^a, Clarissa A. Olivati^{a,*} 

^aUniversidade Estadual Paulista (Unesp), Faculdade de Ciências e Tecnologia, Presidente Prudente, SP, Brasil.

Received: April 27, 2024; Revised: June 30, 2024; Accepted: July 12, 2024

Conjugated polymers have attracted considerable attention to the organic electronics field, due to their properties and a variety of potential areas of application, such as solar cells, OLEDs, sensors, and transistors. In the sensor area, the study of chloroform gained prominence due to its toxicity and effects on the human body, such as damage to the liver and kidneys. This work aims to study thin films of P3DT in different concentrations, perform their optical, morphological, and electrical characterization, and verify their applicability as chloroform sensors. The thin films and devices are characterized using various techniques: optical characterization is performed through UV-vis spectroscopy, morphological characterization is performed through optical microscopy and electrical characterization is performed in direct current by studying the current vs. voltage curves. Electrical measurements of current vs. time are also performed in order to verify the electrical response of the active layer when exposed to a saturated chloroform atmosphere. Characterization shows expected results, similar to other P3ATs, while gas response initially shows a release in trapped charges, increasing the current, however, as the measure is repeated, the current started to decrease when exposed to the current, caused by a swelling in the polymer's chain.

Keywords: *drop casting, gas sensor, chloroform.*

1. Introduction

Through the discovery of polyacetylene by Heeger, MacDiarmid, and Shirakawa in the 70s, a new and emerging class of materials, known as π -conjugated conducting organic polymers has attracted attention due to their unique physical and chemical properties¹. In general, these conducting polymers are conjugated polymers with alternating single and double bonds in the structure and a delocalized π electron structure². Due to this π conjugation, the π electrons are effectively delocalized along the polymer chain, leading to one unpaired π electron per carbon atom³ that allows charge transport along the polymer chain and, as a consequence, exhibits conductive behavior or semiconductor^{4,5}.

These conjugated polymers have attracted considerable attention due to their unique properties such as ease of processing and a wide range of electrical properties, which make them technologically superior and more cost-effective than alternative materials⁶. It is noteworthy that these materials find applications in several areas, such as batteries⁷, transistors⁸, supercapacitors⁹, light emitting diodes (LED)¹⁰, sensors¹¹, solar cells¹² etc. Some studies aim to modify the outermost, non-conjugated segments, including engineering side chain asymmetry¹³, aiming to create free volume between polymer chains for energy dissipation under stretching to ensure efficient charge transport in stretched states¹⁴.

Highlight the polythiophene and its derivatives, which represent the simplest and most classic class of conjugated polymers and have been investigated in recent decades¹⁵. This class of conjugated polymers is of great interest in device applications¹⁶ due to the high stability of their undoped states, ease of structural modification, and solution processability¹⁷. Some studies have demonstrated that cyclic polythiophene oligomers can present unique properties, highlighting the interest in evaluating/proposing new polythiophene-based derivatives¹⁸. Although they have been studied for a while, by altering the morphology and therefore the geometry of the oligomer chains, the band gap and optical properties of polythiophene-based films can be drastically altered¹⁹.

In the last decade, there has been a significant increase in the study of conductive polymers aiming for application in gas sensors²⁰, however, there are records in the literature that use polypyrrole, polyaniline and polythiophene as active layers of gas sensors since before 1980²¹. The use of polythiophenes and derivatives is due to their relative stability in air and processability in solvents, their p-type semiconductor behavior and optical gap²², and have been used as sensing materials for different gases and vapors like NO_x²³ and volatile organic compounds (VOCs)^{24,25}, that include a variety of chemicals that significantly evaporate under normal conditions²⁶, and have been contributed to air pollution and they are suspected to cause cancer in humans²⁷.

*e-mail: clarissa.olivati@unesp.br

The study of chloroform (CHCl_3) sensors has gained prominence due to their effects and toxicity, such as damage to the liver and kidneys when breathing air, eating food or drinking water containing high levels of CHCl_3 for long periods of time, in addition to that large amounts can cause wounds when this compound comes into contact with the skin²⁸. In this work, we report the possibility of the use of poly(3-decylthiophene-2,5-diyl) (P3DT) as sensor active layer for CHCl_3 electrical detection. The sensing measurements were undertaken on an interdigitated electrode by measuring the current versus time with applied voltage at room temperature. This study investigates the impact of various concentrations on the electrical properties of P3DT and examines its electrical response to CHCl_3 , with the goal of applying this material as a chloroform sensor.

2. Methodology

2.1. Materials

In this work, a regioregular polythiophene poly(3-decylthiophene-2,5-diyl) (P3DT), acquired from Sigma-Aldrich, CAS number 110851-65-5, product number 495344, are used in the study of sensors to detect CHCl_3 . Figure 1 presents the condensed chemical structure of P3DT.

Glass substrates are used to perform optical and morphological characterizations. To electrical and gas sensing measurements, gold interdigitated electrodes (IDE), previously fabricated with photolithography on glass slides, are employed. IDEs consist of 50 digits with dimensions of 110 nm in height, 8 mm in length and 100 μm wide for each digit, and a spacing of 100 μm between each digit.

The use of IDE is essential for this work, because each pair of digits amplifies the total current measured²⁹, which facilitates the electrical characterization in materials with lower electrical conductivity. To prepare the solutions and perform the gas sensing measurements, the solvent CHCl_3 (99,9%), obtained from Synth, was selected.

2.2. Drop casting films

The drop casting deposition technique consists in one of the most simple deposition techniques from solution³⁰. The solution is dropped onto a horizontally stable substrate with an electronic pipette. After the solvent evaporation, only the material remains on the substrate, due to Van der Waals forces³¹. The technique, however, does not allow control over the film formation, facilitating the formation of aggregates in the structure, resulting in films with a heterogeneous surface.

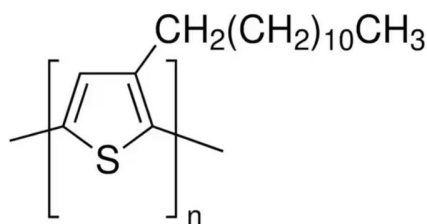


Figure 1. Chemical structure of P3DT.

This also limits the control over film thickness, although there are factors, such as the deposited volume and solution concentration, that can help to regulate the final film thickness and influence the film morphology, but with a low accuracy³⁰⁻³². Solvent evaporation is one of the most important parts of the film formation process, where we can choose to let it dry naturally or use a thermal process to accelerate the evaporation, however, disturbing the system during the evaporation can cause impacts on film morphology^{33,34}.

A volume of 200 μL is dipped onto the substrate, for both concentrations of 0.5 $\text{mg}\cdot\text{mL}^{-1}$ and 1.0 $\text{mg}\cdot\text{mL}^{-1}$. The films are left to dry naturally at room temperature ($\sim 30\%$ of humidity) for 24 hours to ensure a complete solvent evaporation and film stabilization.

2.3. UV-vis Spectroscopy

The study of the processes of electronic transitions that happen in the ultraviolet-visible range makes it possible to obtain important information about the materials, like the internal structural organization of a film³⁵. UV-vis spectroscopy measurements are performed using the Agilent Technologies model Cary 100 UV-Vis spectrophotometer, with the incidence of light in the range of 900 to 350 nm. The lower wavelengths are avoided due to the glass opacity in ultraviolet light. A detector located behind the samples is responsible for measuring the intensity of the light absorbed by the thin film.

2.4. Electrical characterization in direct current

Current versus voltage (*I versus V*) measurements are performed with a Keithley Instruments model 238 Source Measure Unit, in a range from -10 V to 10 V, with steps of 0.5 V, in a controlled temperature at 23°C. To calculate the electrical conductivity and resistance for each curve, Ohm's law equations are used with the geometric factor of the IDE (cell constant), with value of 5.1 m^{-1} , determined in previous works from the group, following Olthuis' method^{36,37}.

2.5. Gas sensing properties

In order to evaluate the performance of the films as CHCl_3 sensors, electrical characterization of the samples is carried out using current versus time (*I versus t*) curves, in a saturation regime of the CHCl_3 . The measures are performed in cycles of 5 min, alternating between an inert baseline of N_2 and CHCl_3 , carried out to the sample with the aid of the N_2 flow at 60NL/h, with a purity of > 99.999% (< 0.5 ppm of CO ; < 0.5 ppm of CO_2 ; < 1 ppm of O_2 ; < 0.1 ppm of THC; < 2 ppm of H_2O). It is important to note that detection can also be achieved with lower flow rates, between 15 and 20 NL/h, with minimal electrical pulse interference. The cycles are repeated in order to verify the reproducibility of the obtained results. Figure 2 presents a representation of the gas measurement system used.

3. Results and Discussions

Figure 3 exhibits the normalized absorbance spectra per wavelength obtained from the UV-vis measurements for both P3DT drop casting films (0.5 $\text{mg}\cdot\text{mL}^{-1}$ and 1.0 $\text{mg}\cdot\text{mL}^{-1}$) and P3DT solutions (0.5 $\text{mg}\cdot\text{mL}^{-1}$ and 1.0 $\text{mg}\cdot\text{mL}^{-1}$).

In both solution's curves, a peak at 450 nm is observed, usually related to $\pi-\pi^*$ interactions³⁸. However, the spectra are red-shifted on the drop casting films, due to a higher packing and molecular ordering when casted as thin films³⁹. This higher ordering results in the displacement containing distinct peaks observed in the film's spectra⁴⁰. It is also possible to see three distinct peaks at lower energies, approximately at 525 nm, 557 nm and 610 nm, assigned to vibronic absorptions⁴¹. The peaks at 525 nm and 557 nm



Figure 2. Representation of the gas measurement system.

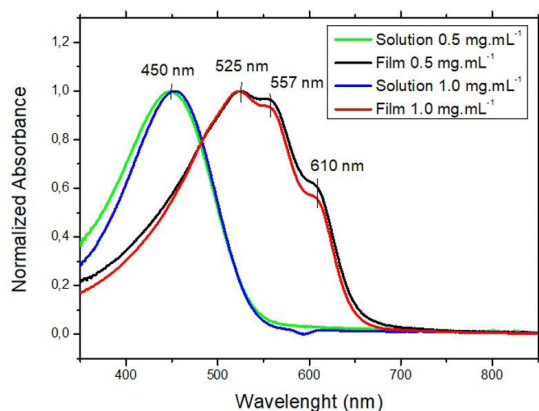
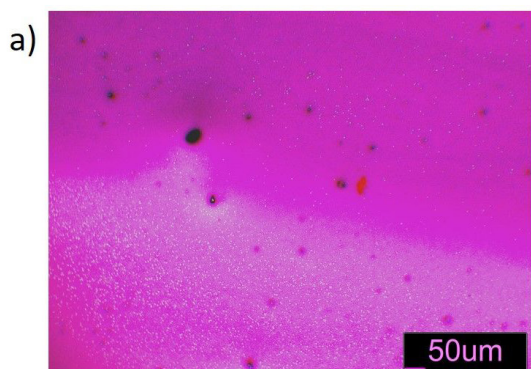


Figure 3. UV-vis spectra obtained from P3DT solutions and drop casting films.



are related to an intrachain exciton, while the peak at 610 is related to interchain adsorption⁴²⁻⁴⁴.

Figure 4 presents the surface morphologies of the P3DT drop casting thin films observed by optical microscopy, produced by the deposition of 200 μL of the thiophene solution with different concentrations.

In Figure 4b it is possible to observe the formation of multiple and random thiophene aggregates, an expected result, commonly obtained from drop casting films, since the drop casting deposition technique offers little to no control over the film morphology and structural organization³⁰⁻³². In Figure 4a however, the aggregates are smaller and lesser in number, a fact that can be a consequence of the lower solution concentration (0.5 mg.mL^{-1}) compared to the film shown in Figure 4b (1.0 mg.mL^{-1}), Thus not having enough material in the film to create aggregates in a similar size or number, even considering that both films were deposited with the same volume.

Figure 5 shows the electrical curve characteristic of P3DT films with different concentrations. The linear results obtained from the current *versus* voltage curves show that both films present an ohmic behavior, due to the electrical contacts, with a configuration of Au/Film/Au⁴⁵⁻⁴⁷. This behavior allows the use of Ohm's laws to calculate the electrical resistance and conductivity of the films. The values obtained are shown in Table 1.

The values obtained are consistent to the results found on the literature, to electrical conductivity found for others P3ATs^{41,48}, the most commonly found being P3HT and P3OT. There are, however, subtle differences between the results obtained from P3DT compared to other alkylated thiophenes, caused by different molecular conformations, influenced by the side chain size⁴⁹.

The difference in the conductivity obtained from the samples here can be explained by the different concentrations used in the deposition of the film. Studies show that thiophenes

Table 1. Electrical conductivity and resistance obtained to the drop casting P3DT films.

P3DT	Conductivity (S.m^{-1})	Resistance (Ω)
0.5 mg.mL^{-1}	9.3×10^{-7}	5.5×10^6
1.0 mg.mL^{-1}	6.7×10^{-6}	7.6×10^5

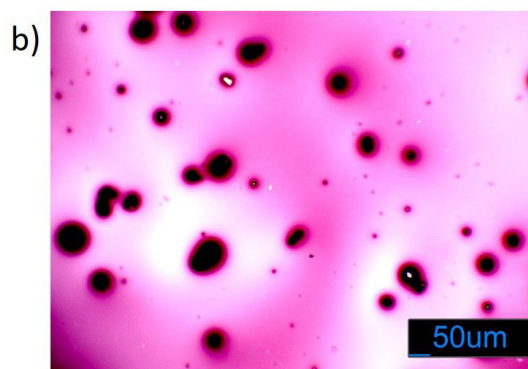


Figure 4. OM images of P3DT drop casting thin films a) from 0.5 mg.mL^{-1} solution; b) 1.0 mg.mL^{-1} solution.

in a higher concentration tend to have an increased charge mobility than thiophenes in a lower concentration⁵⁰. The same study also states that the influence of the morphology and the aggregates formed in the films has a less significant effect on the electrical conductivity compared to the charge mobility. The response of P3DT sensors to CHCl_3 for both concentrations are exhibited in Figure 6.

In general, both sensors show a pattern with a decrease of the current during the cycle of CHCl_3 , expected for the exposure to the compound. The first exposure to chloroform shows an increase in the current that can be explained due to an interaction between the CHCl_3 and the surface of the P3DT sensors that can release charges that were trapped during the film deposition^{51,52}.

In the subsequent cycles, however, both films exhibit the opposite behavior, with their currents lowering when exposed to CHCl_3 . This can be attributed to a swelling in the polymer chain, observed when poly(3-alkyl thiophenes) in general are exposed to organic solvents vapors, such as CHCl_3 , THF, and cyclohexanone⁵³. In previous works from the group, other alkylated thiophenes, like P3HT and P3OT also resulted in similar curves, with the samples decreasing its current when exposed to similar organic solvents^{36,41,48}.

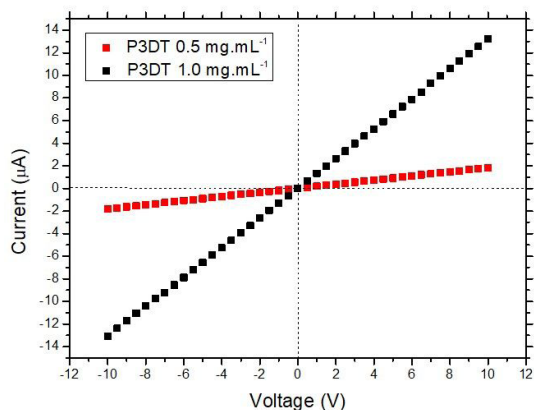


Figure 5. DC characterization of the P3DT drop casting films.

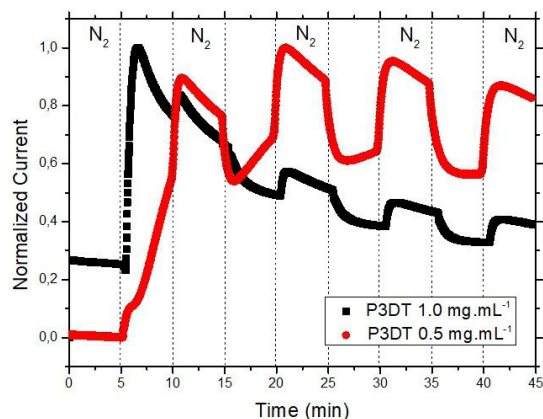


Figure 6. Response of P3DT thin films sensors to CHCl_3 .

4. Conclusions

This work presents the fabrication and characterization of P3DT drop casting thin films, in different concentrations, in order to perform their characterization and analyze their electrical responses when exposed to CHCl_3 vapors. UV-vis results in solution showed a peak at 450 nm, characteristic of P3ATs, which was red-shifted in the film due to a molecular ordering. The optical microscopy revealed that the film casted with a lower concentration resulted in the formation of lesser and smaller aggregates than the film with a higher concentration. The formation of such aggregates were expected due to the nature of the drop casting technique employed. The electrical conductivity observed in the film with a higher concentration was ~ 10 times higher than in the lower concentration film, a difference that occurs due to an increase of the carrier's mobility in P3ATs in higher concentration solutions. Testing the films responses to CHCl_3 revealed that the interaction material-solvent initially resulted in an increase of the current, probably related to the release of trapped charges, however, from the second cycle the current dropped when the film was exposed, caused by a swelling in the polymer's chain.

5. Acknowledgments

This research was funded by São Paulo Research Foundation (FAPESP), and National Council for Scientific and Technological Development (CNPq, INEO). The authors are grateful to the Brazilian Nanotechnology National Laboratory (LNNano/CNPq, LMF project 2023090) for supplying the gold interdigitated electrodes. This study was financed by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brazil (CAPES - Finance cod. 001)

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