

# EXPERIMENTAL STUDY: THE ELECTRICAL CONDUCTIVITY OF POLYPYRROLE DOPED ORGANIC ACIDS THIN FILM

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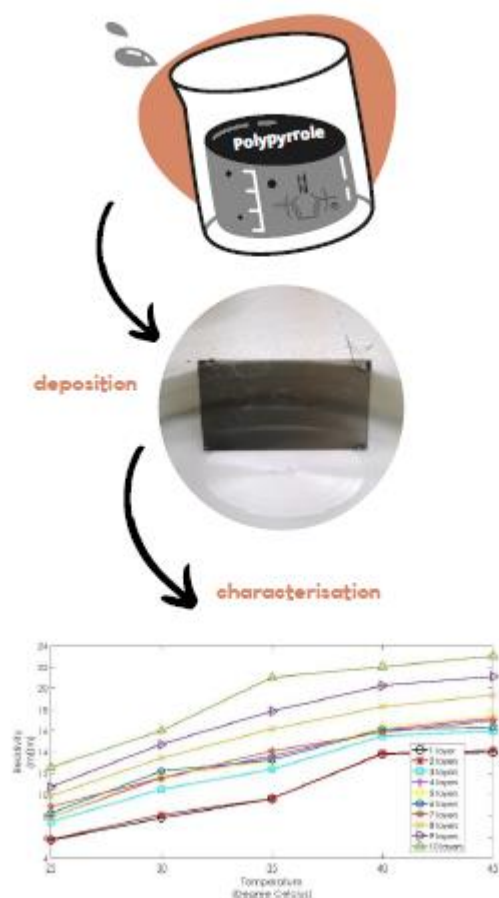
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## Graphical abstract



## Abstract

Properties of materials in microscale are different from those in bulk form. This article aims to presents the experimental studies of the electrical properties of Polypyrrole thin film doped in organic acid. Ten samples of Polypyrrole doped in organic acids thin films with different layers were prepared using the spin coating method applied on glass substrates. The electrical resistance of the thin film is measured as a function of film thickness and temperature. The resistance decreases as the thickness of the thin film increases. Resistivity and conductivity were also calculated. It was observed that the resistivity value of Polypyrrole doped in organic acids is relatively small which is in  $10E-3 \Omega m$ , thus making it a good conducting material. In terms of conductivity, the thinner is the film, the higher is its conductivity. Temperature starting from room temperature until  $45^\circ C$  with  $5^\circ C$  of increment was applied. The resistance increases when the temperature increases, thus exhibit a positive temperature coefficient type of thermistor characteristic. The resistivity on the other hand rise as the temperature rise. Whereas, the conductivity decrease with the increment of temperature. Advances in thin film technology will allow the fabrication of PPy doped in organic acids material for various applications including electronics and sensors.

Keywords: Polypyrrole, positive temperature coefficient (PTC), thermistor, thin film

## Abstrak

Sifat sifat bahan pada julat saiz mikro adalah berbeza dengan bahan bahan yang diuji dalam keadaan pukal. Artikel ini adalah bertujuan untuk membentangkan analisis kajian berdasarkan eksperimen mengenai nilai aktiviti elektrik lapisan nipis Polypyrrole/asid organik. Sepuluh sampel lapisan nipis Polypyrrole/asid organik telah disediakan dengan berlainan ketebalan meggunakan kaedah salutan putar (spin coating) di atas kepingn kaca. Kerintangan elektrik diuji bersandarkan pada saiz ketebalan lapisan filem dan suhu yang berbeza. Nilai kerintangan menurun apabila saiz lapisan filem menaik. Nilai ketahanan dan kekonduksian juga telah dikira. Pemerhatian telah menunjukkan bahawa nilai ketahanan Polypyrrole/asid organik adalah kecil, iaitu  $10E-3 \Omega m$ , menjadikannya bahan pengalir elektrik yang baik. Dari sudut pandang nilai kekonduksian pula, semakin nipis filem, semakin tinggi nilai kekonduksian bahan

tersebut. Suhu bermula daripada nilai suhu bilik, sehingga 45 °C dengan peningkatan 5 °C telah diaplikasikan sewaktu eksperimen dijalankan. Nilai kerintang yang bertambah apabila suhu meningkat telah menunjukkan ciri-ciri pekali suhu positif bagi perintang suhu. Nilai ketahanan pula menaik apabila suhu menaik. Manakala nilai kekonduksian menurun dengan peningkatan suhu. Kemajuan teknologi lapisan filem nipis akan membolehkan fabrikasi berdasarkan PPy/asid organik termasuk aplikasi elektronik dan sensor.

**Kata kunci:** Polypyrrole, pekali suhu positif, perintang haba, filem nipis

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## 1.0 INTRODUCTION

Conducting polymers (CPs) have received increasing attention over the past decades owing to their electrical properties similar to those of both metals and traditional semiconductor [1]. CPs is organic polymers that conduct electricity [2]. CPs are distinguished from other types of polymers due to their conjugated double bond that are responsible to allow electric flow. CPs show significant different physical behaviour because they can be switched between two or more redox states. Their conductivity can change from insulating to metallic upon doping. CPs were also been labelled as "synthetic metals" in the late 1970s [1, 3], other than being semiconducting and lightweight [4, 5].

One of the first discovered CPs is Polypyrrole (PPy). Since the discovery of PPy in 1979 [6], there has been extensive studies [7] on this material. PPy provides the advantages of chemical diversity, low density [1] and high conductivity [3, 8-12], biocompatibility [10] [13] and ease of processing [3, 12, 14] [15] [16, 17]. In addition, PPy also shows good environmental stability [3, 10-12, 14], and excellent thermal stability [18] [19] [17]. PPy is potentially useful as an electrical conductor [20], therefore making it an excellent material for sensors and electronic components [8]. Furthermore, PPy has the advantage of being flexible [1] and mouldable leading to maximum design freedom for constructing parts [21]. PPy also have lower potential for negative environment as it is an organic type of material [22].

PPy in general is an insulator, however it becomes electrically conductive with addition of dopants. Thus, been used in a wide range of sensor applications. PPy can be prepared using different methods and dopants in order to achieve desired electrical properties for various applications [13]. PPy with different dopant has a unique and different set of electrical characteristics such as resistivity and conductivity. These electronic properties are the basis for a growing world-wide interest in the use of PPy as advanced functional materials [1].

Organic acids are among the most common type of dopant available for PPy. Some of the available Organic acids dopant for PPy are such the Dodecyl

Benzene Sulphonic Acid (DBSA), Camphor Sulphonic Acids (CSA) and P-Toluene Sulphonic Acids (P-TSA) [23]. Organic acids belong to an organic compound group with acidic properties, such as carboxylic acids (-COOH) and sulfonic acids (-SO<sub>2</sub>OH). The Organic Acids are reported as a stronger acid than alcohol (-OH). One of the major advantages is that organic acids is soluble in water. In comparison to other solvent, water solvent possess a more polar character, thus giving a higher conductivity [24]. PPy doped in organic acids are also proven to enhance the CPs electrical properties and environmental stability [23]. Although one of the drawbacks of this material is, it is reported to be brittle, the brittleness however can be easily solved by casting it into thin films. PPy doped in organic acids also reported to be used as a novel cathode material [25].

Thin films technology is one of the most advance technology that is used worldwide where the term thin film is applied to samples that having thickness of less than 6 µm. Thin film material offers a lightweight solution with fast material response time.

Electrical characterization of PPy prepared in various organic acids dopant such as camphor sulfonic acid (CSA), dodecylbenzenesulfonic acid DBSA and β-Naphthalene Sulfonic Acid (NSA) has been done previously with various solvent and methods, but the electrical properties of a readily available PPy in organic acids has not yet to be discovered.

Bulk electrical characteristics of any material is one of the intrinsic properties of a material and it is dependent on the sample's dimension [26, 27]. However, as for the physical properties of a thin films, they are highly dependent on their thickness. The determination of the film thickness therefore is a fundamental task in thin film technology. Literature findings reported that the electrical resistivity of a material in thin film is not the same as in a bulk [26] [27]. CPs that is relatively small such as in nanostructures have superior properties over bulk Cps as it exhibit better electrical properties [5].

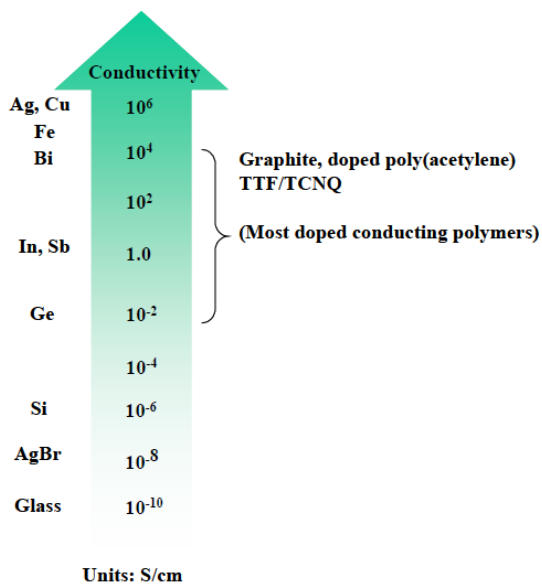


Figure 1 Comparison of conductivities of materials [28]

Also, it is interesting to know that the classification of materials such as insulators, semiconductors and metals, can be generated from comparison of conductivities. Thus, measurement of the stated properties can provide valuable information to ensure any intended application. Comparison of conductivities of various materials is shown in Figure 1.

Other than material type, length, and cross-sectional area, temperature is one of the factors that affecting the electrical value. Little attention has been given to how the PPy respond to changes in temperature [10]. It is reported that exposure to elevated temperature induced changes in the molecular structure of a CPs [15]. Hence, study of CPs conductivity profile with increased temperature is necessary for successful applications in the future.

This paper presents an electrical characterization of a thin film PPy doped in organic acids with 5 wt% dispersion in water from Sigma Aldrich, deposited on a glass substrate. The results from experiments are discussed to demonstrate the relationship between electrical resistance and film thickness, as well as temperature.

## 2.0 EXPERIMENTAL SETUP

### 2.1 Materials

- 1 Polypyrrole (doped in organic acids, 5 wt% dispersion in water) from Sigma Aldrich Inc. solution (482552-100 ml) is used without any further purification.
- 2 Ethanol (95% of purity, molecular weight: 46.07g/mol).
- 3 Deionized (DI) water.

### 2.2 Surface Treatment by Oxygen Plasma

Glass substrate of 1 mm thickness (dimension: 25.4 mm x 25.4 mm) was first consecutively rinsed with DI water. The rinsed substrate was then immersed in Ethanol solution for 6 minutes to remove any contaminant and impurities. Subsequently, the glass substrates were dried by an air gun.

After the drying process, the cleaned glass substrates were then exposed to oxygen plasma for approximately 6 minutes to promote oxygen group creating temporary hydrophilic charges on the glass and increase the spreading. Besides, plasma treatment is used to remove any air particle or moisture and incinerate all organic materials on the surface's samples. The glass samples were vacuumed in a chamber that contains UV light and oxygen gas. Table 1 below shows the parameter that had been used for plasma treatment.

Table 1 Plasma parameter settings

Parameters	Settings
Set power	250 W
Pressure	10 KPa
Flow	4 bar
Time	6 minutes

### 2.3 Deposition of PPy Thin Film

The thin films of PPy were deposited using a spin coating method. The spin coating process was performed at room temperature using a Laurell spin coater. 1 ml of PPy liquid was deposited on a glass substrate, which was spun in two stages as follows:

Stage 1:  
500 rpm (5 seconds), acceleration of 1000 rpm/s

Stage 2:  
1899 rpm (20 seconds), acceleration of 1000 rpm/s

A thin black film was obtained and dried on a hotplate at 70 °C for 2 minutes. Ten samples of PPy were prepared. The second layer and other consecutive layers were spin-coated after the first and previous layers were completely dried.

### 2.4 PPy Thin Film Thickness Measurement

The thickness of each deposited layer was determined using a profiler meter, a High Power Microscope (HPM) from DextarTAR. A diamond stylus (tip curvature approx. 10 μm) is pulled along the surface at constant velocity. The measurements were conducted on ten samples of PPy on the glass substrate of 10 different thicknesses. Three points of thickness readings were taken and the mean thickness for each sample was measured.

## 2.5 PPy Thin Film Electrical Characterization

LCR meter (GW INSTEK LCR-8101G) is used to measure the output resistance of the PPy conducting film, conducted at room temperature followed by set temperatures from 30°C to 45°C with 5°C increment. The input voltage of 1 V and a test frequency of 1 kHz were applied at each measurement. The separation between two probes was set at 1 cm. For each layer of PPy, 10 values of electrical resistance were recorded, and the mean resistance values were calculated. Whereas the temperature parameters were introduced using a hotplate.

## 2.6 PPy Thin Film Surface Characterization

The PPy thin film surface morphology examinations were done by Scanning Electron Microscope (SEM) analysis using a JEOL JSM6010LV with an electron beam of 15 keV. The samples were coated with platinum to obtain good electrical contact as well as to reduce charging effect during imaging. A copper tape is used to place the samples together in order to have a smooth electron flow during imaging. The stage for sample were tilted at 60 degrees for sample's image cross section.

## 3.0 RESULTS AND DISCUSSION: ELECTRICAL PROPERTIES

### 3.1 PPy Film Thickness at Room Temperature

In this section, the experimental results of PPy thin film as a function of its thickness is presented. The measurement was done in a condition of room temperature.

Table 2 shows the measurement of PPy film thickness for 10 different PPy samples. Each sample was measured at three different points and then the thickness mean value was determined. Based on Table 2, the thickness of the PPy film increases with the increase of deposition layer. The standard deviation (SD) for each layer of PPy are calculated to be less than 0.1, meaning that that spin coated PPy showed uniform distributions on the glass substrate even though the rate of increase in the thickness is not linear. This non linearity is due to the deposited material concentration different and solvent evaporation rate during the experimental procedure [29].

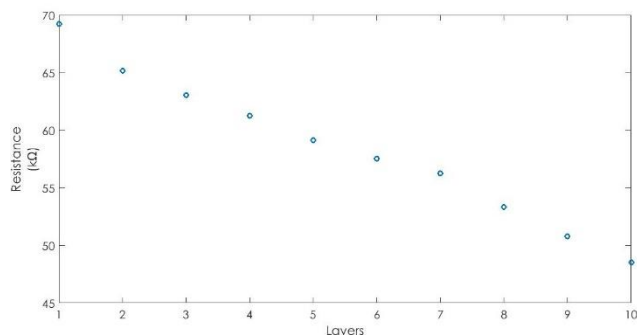
**Table 2** Thickness values of all 10 layers of PPy

PPy layers	Readings ( $\mu\text{m}$ )			mean	SD
	1 <sup>st</sup>	2 <sup>nd</sup>	3 <sup>rd</sup>		
1 layer	0.0838	0.0814	0.0818	<b>0.0823</b>	0.001
2 layers	0.0835	0.0931	0.0893	<b>0.0886</b>	0.005
3 layers	0.1169	0.1141	0.1167	<b>0.1159</b>	0.002
4 layers	0.1290	0.1313	0.1264	<b>0.1289</b>	0.002
5 layers	0.1346	0.1344	0.1328	<b>0.1339</b>	0.001
6 layers	0.1367	0.1363	0.1363	<b>0.1431</b>	0.000
7 layers	0.1637	0.1600	0.1484	<b>0.1574</b>	0.008
8 layers	0.1735	0.1931	0.1881	<b>0.1849</b>	0.010

PPy layers	Readings ( $\mu\text{m}$ )			mean	SD
	1 <sup>st</sup>	2 <sup>nd</sup>	3 <sup>rd</sup>		
9 layers	0.2051	0.2230	0.2139	<b>0.2017</b>	0.011
10 layers	0.2641	0.3076	0.2046	<b>0.2586</b>	0.052

### 3.1.1 Resistance

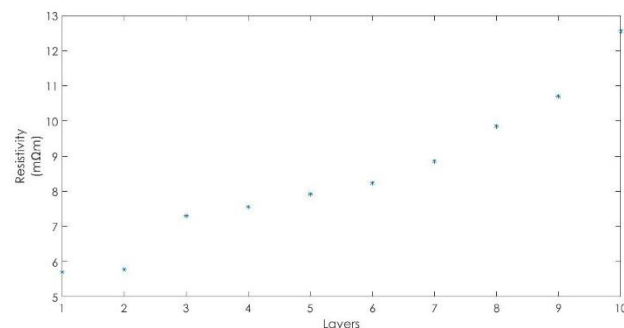
Resistance values of different thickness is shown in the Figure 2. The resistance decrease as the thickness increase. Thicker film having a lower resistance value is because the electrons will have a bigger area to flow since electrons have the ability to adjust itself according to whatever thickness a film or wire is.



**Figure 2** Resistance of all 10 layers of PPy at room temperature (25 °C)

### 3.1.2 Resistivity

The resistivity,  $\rho$  of a material is the characteristic that describes an ability to conduct electric currents. The electrical resistivity of a materials in thin film is not the same as in bulk form. This phenomena occurs due to the mean free path of the conduction is reduced thus increasing the scattering effects [27].



**Figure 3** Resistivity of all 10 layers of PPy at room temperature (25 °C)

Experimental evidence shows that the resistivity increase as thickness is increased such as in Figure 3. The minimum and maximum resistivity of obtained film was 5.6974E-3  $\Omega\text{m}$  for 1 layer and 12.5473 E-3  $\Omega\text{m}$  for 10 layers respectively. The calculated resistivity value of PPy shows that the PPy has enough free electrons to conduct electricity even at room temperature. The

value of resistivity,  $\rho$  with denoted unit of  $\Omega\text{m}$  is calculated by (1).

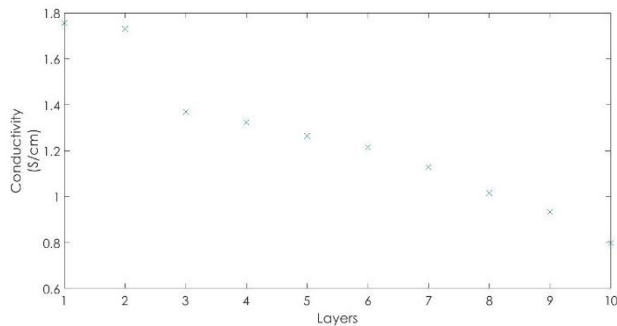
$$\rho = R_s t \quad (1)$$

Where  $R_s$  is the film sheet resistance and  $t$  is for the PPy film thickness (measured using profiler).

### 3.1.3 Conductivity

The electrical conductivity of PPy is determined by its electronic structure [1]. The electrical conductivity is the product of two number of carriers (electrons or holes) and charge carrier mobility [15]. In this paper, the conductivities are represented in Siemens/cm. The conductivity is the inverse of the resistivity [30] such as in (2).

$$\sigma = 1/\rho \quad (2)$$



**Figure 4** Conductivity of all 10 layers of PPy at room temperature (25 °C)

The highest calculated conductivity is ( $\sigma \sim 1.7542$  S/cm) for  $0.0823 \mu\text{m}$  thin for 1 deposition layer and lowest at ( $\sigma \sim 0.7969$  S/cm) for  $0.2586 \mu\text{m}$  at 10 deposition layers dried cast film, both at room temperature. Other conductivity studies shows that the composites exhibit conductivities at room temperature and in the range of  $10\text{E}-7$  and  $10\text{E}-6$  S/cm synthesized by a non-organic acid type of oxidizing agent such as Si-SBA-3 and Na-ALSBA-3 materials were synthesized [7], which is much lower value that our measured sample. The measured conductivity is also approximately as near to the bulk conductivity of PPy doped in Dodecylbenzene sulfonic acid (DBSA) which was about ( $\sigma \sim 1$  S/cm) [11]. However, the conductivity of bulk PPy(DBSA) is reduced to ( $\sigma \sim 10^{-3}$  to  $10^{-8}$  S/cm) after dissolution in a polar solvent when cast into film [11].

Figure 4 shows that as the thickness increase, the conductivity decrease. Hence, it concludes that the thinner the PPy film, the higher its conductivity. This is due to shorter ion transport distances [5]. The obtained values for conductivity is almost to a value of metallic material ( $\sigma \sim 5$  S/cm)[31]. Thin wire

of PPy ( $10\text{-}100 \mu\text{m}$ ) in a study done by C. B. Gorman has been found to have conductivities in a range of  $1\text{-}5$  S/cm, which is also stated as the expected range values for common PPy [20]. The conductivity of undoped polymer at room temperature is  $10\text{E}-6\text{-}10\text{E}-10$  S/cm, lies between the insulator and semiconductor properties [1].

## 3.2 Temperature Dependence

### 3.2.1 Resistance

In obtaining a successful material applications in the future, its electrical resistance is one of the key properties that need to be considered [15].

The measurement of resistance value for each sample was done for temperature ranging from  $25^\circ$  to  $45^\circ$  with  $5^\circ$  increment. As presented in Table 3, the measured resistance increase as temperature increase over a limited range of temperature for all ten PPy layers which indicates a conductive property [27]. This property is similar to a positive temperature coefficient (PTC) type of thermistor. Furthermore, the tested thin films for all layers shows a high sensitivity to temperature changes, which is a desirable property for future temperature sensing material.

**Table 3** The measured mean electrical resistance of PPy thin film for various thickness and temperature

Thickness ( $\mu\text{m}$ )	Resistance (k $\Omega$ )				
	RT	30 °C	35 °C	40 °C	45 °C
0.0823	69.227	94.655	116.599	167.655	169.545
0.0886	65.175	91.320	108.542	156.512	159.754
0.1159	64.061	90.502	106.542	133.791	137.560
0.1289	61.256	89.593	105.444	124.773	133.036
0.1339	59.112	87.479	98.4738	121.819	129.820
0.1431	57.547	85.188	92.552	112.634	113.720
0.1574	56.254	73.270	89.441	100.729	107.573
0.1849	53.304	72.563	87.382	98.683	104.567
0.2107	50.793	69.889	84.616	96.028	100.199
0.2586	48.525	51.609	82.509	84.428	88.942

It is also observed that as the thickness of the PPy thin films increases, the electrical resistance decreases for all measured temperature suggesting an increase of power output which shows a similar characteristic to any conductor or metallic material [27]. For metallic materials, the resistance increases linearly with temperature. Exposure of PPy to rising temperatures is known to induce changes in the molecular structure [15]. The resistance increases with increasing temperature is due to the collision between vibrating ions and electrons in the tested material. The ions inside vibrates as it acquires energy from the applied temperature, thus begins oscillating about their mean positions.

### 3.2.2 Resistivity

Figure 5 indicates the variation of  $\rho$  with temperature for PPy organic acids composite ranging from 25 °C to 45 °C. The values of resistivity increase with temperature. The resistivity increases with increasing temperature indicate a similar behaviour to conductor.

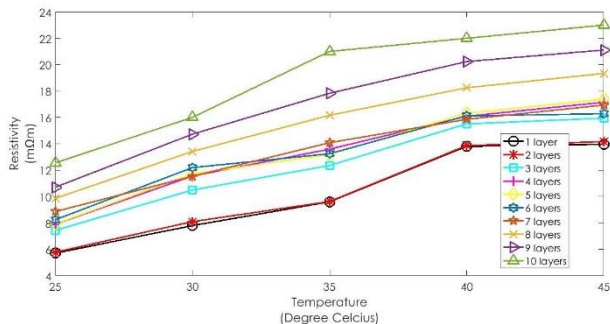


Figure 5 Electrical properties of PPy: Mean resistivity versus temperature

### 3.2.3 Conductivity

Figure 6 demonstrated the variation of electrical conductivity,  $\sigma$  with temperature for PPy organic acid composites obtained in a range of 25 °C to 45 °C temperature. The lowest conductivity value is ( $\sigma \sim 0.4348$  S/cm) which is measured at 45 °C for 10 layers film. Whereas the highest conductivity value recorded is ( $\sigma \sim 1.7552$  S/cm), measured at room temperature (25 °C) for 1 layer film deposition. The conductivity values lie in the semiconducting group, hence the PPy doped in organic acids is a suitable as a potential candidate for a resistive temperature sensor such as thermistor.

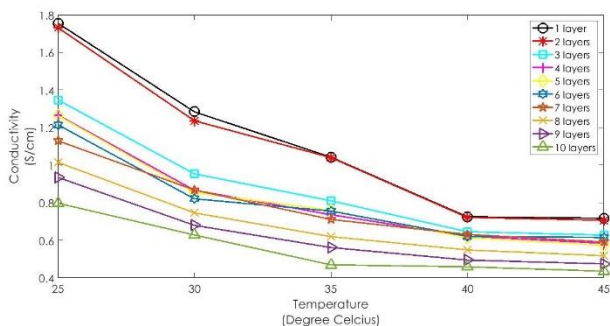


Figure 6 Electrical properties of PPy: Mean conductivity versus temperature

The conductivity decrease with temperature showing similar trend to metal or semiconducting behaviour's temperature profile. When the temperature increased, molecules inside the material will start to vibrate more due to thermal energy thus increasing in vibration. An increase in vibration will decrease the mean free path of molecules and

thereby its electrons. The amount of increasing number of free electrons, so when lattice vibration takes place all these electrons have their mean free path reduced in that way reducing conductivity.

Temperature dependent measurements have revealed that the films are *p*-doped. Generally, *p* doping is more popular in academic research and as well as for practical applications [1]. This is because the charge carriers in *n*-doping are not as stable as positively charged form. Representatively, conductivity in PPy is the outcome of positively charged (*p*-type) doping[1].

The tested PPy can be said as electrically conductive, thus makes it a suitable candidate for ceramics material replacement as well for heat-sensitive applications with a lower density [21], therefore much lighter than the equivalent components. Density of PPy is only  $1.47 \text{ g cm}^{-3}$  [32] whereas the density of ceramic is reported to be in a range of  $4.1$  to  $4.8 \text{ g cm}^{-3}$  [33].

### 3.3 Film Morphology

The surface morphology of the spin coated PPy doped in organic acids composite film at 400x magnification is shown in Figure 7. The SEM image shows a porous and granular surface morphology at the edge of the substrate but with good uniformity throughout. Noted that there also exist some lines. The lines might be due to a slight crack which is consistent to the material brittleness after dried. Overall, it can be said that the PPy doped in organic acids fully covered the area of interest on glass substrate for 1 layer of deposition.

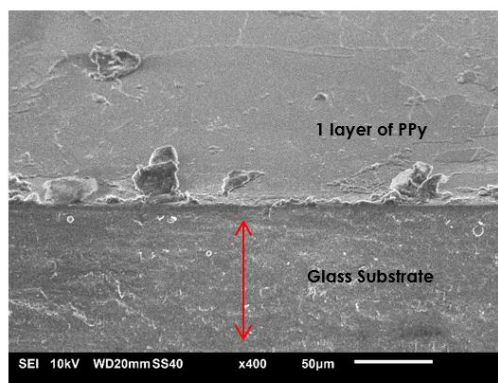


Figure 7 Surface morphology of PPy doped in Organic Acids at 400x magnification

DB Dupare et.al, 2011 has investigated PPy under different types of organic acids dopants. It was found that different organic acid produces different types of surface morphology.

## 4.0 CONCLUSION

PPy is a unique class of organic material that can conduct electricity. The electrical properties of PPy doped in organic acids is confirmed in this study, and it also affected by temperature as well as thickness. These findings provide insight into the underlying physics of PPy dope with organic acids thin film, and relates its electrical activity to temperature and thickness. Based on these findings, the PPy doped in organic acid material from Sigma Aldrich is suitable to be used as a temperature sensor material (resistance increase when temperature increase), specifically the PTC type of thermistor and it is electrically conductive with a uniform surface morphology.

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