



COMPARISON OF THE CONDUCTIVITY OF PPY/1,5NDSA AND PPY/NSA-APSNANOCOMPOSITE WITH INCREASING TEMPERATURE

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Abstract:

The effect of dopant concentration on structural morphology and yield of products were investigated using the dopants 1,5-Naphthalene disulfonic acid and Naphthalene sulfonic acid. A Keithley 6571B High Resistance Meter and Electrometer was used to evaluate the conductivity of different molar ratios of PPY/1,5NDSA and PPY/NSA-APS utilising a four- probe setup. The conductivity of polypyrrole films doped with different sulfonate derivatives of naphthalene decreases as the number of sulfonate groups in a doped molecule grows in the same aromatic ring (naphthalene). The polypyrrole films were characterised using Fourier transform infrared spectroscopy (FTIR), NMR spectroscopy, SEM examination, and XRD analysis.

Keywords - pyrrole, polypyrrole, Naphthalene Sulfonic Acid (NSA) and 1,5 Naphthalene disulfonic acid etc.

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1. Introduction

Conjugated polymers have gained a great deal of attention in recent years as cutting-edge materials with unique properties related to the delocalization of electrons in the pi-bond system. Polypyrrole has drawn the most interest among the conjugated polymers because to its higher environmental stability, straightforward manufacturing, and improved electrical conductivity compared to other conjugated polymers. [1] Electrical conductivity is influenced by electron concentration, charge, and mobility [2] an increase in electron mobility usually conducting polymer among all the coordinating polymers due to its effectively oxidizable nature, water dissolvability, high conductivity, highest environmental stability, and redox properties. [4] The method used to organize the polypyrrole as well as the potential dopant used determine nearly all of these properties. [5] by adding particles into the polymer's basic structure. The characteristics of PPY, such as solubility, electrical structure, and crystallinity, are changed by protonation with an organic acid [6]. It was suggested that other substituted monomers be used to minimize the intense intermolecular contact between conducting polymer chains in the doped state, which would improve PPY's solubility and processability. [7]. The planarity of the polymer structure is altered by the attachment of long-chain substituents to the monomer ring as a result of steric interference. This can be accomplished by combining polymers with organic acids. Bulk PPY's conductivity, form, and thermal stability are all dramatically impacted by the polymer's inclusion of organic sulfonic acid as a counter-ion. [8] The solvent [9], temperature, monomer concentration, supporting electrolyte [10], polymerization time, dopant concentration, and dopant type [11] are important polymerization parameters that have a significant impact on the polymerization of pyrrole. It has been demonstrated that the electrical conductivity of conducting polymers can be increased by adding a macromolecular proton acid as a dopant during the production process [12].

Currently, the effect of the doping concentration of various sulfuric acid on the nature and characteristics of polypyrrole nanoparticles has not been extensively studied. NSA and 1,5 NDSA -doped nanoparticles were prepared by in situ chemical polymerization

method. The effect of dopant concentration on morphological, thermal, structural, solubility and electrical properties of polypyrrole is investigated.

Material preparation and experimental techniques:

Chemicals: - Pyrrole (98% pure sigma Aldrich) Ammonium per sulphate (APS) as oxidant, 1-Naphthalene sulfonic acid (>50 % pure sigma Aldrich) (NSA) and 1,5Naphthalene disulfonic acid disodium salt (95 % pure sigma Aldrich) (1,5NDSA) used as a dopant, deionized water as neutral solvent are used for synthesis.

Preparation of undoped polypyrrole powder. [SPPY]

For synthesis of polypyrrole by chemical bath method, primarily all apparatus were washed with deionized water, rinse in acetone. Pyrrole is used as monomer for preparation of polypyrrole powder. Monomer is prepared by using 1M pyrrole in deionized water. Oxidant solution was prepared 1M concentration of APS in deionized water. The ratio of monomer to oxidant was kept 1:2. which means the polymerization proceeds in excess of oxidizing agent. Firstly, Monomer solution was added dropwise in an oxidant solution reaction being carry out at room temperature at 25⁰c. During precipitation heterogenous reaction occur. Polypyrrole produced was kept unagitated for 24 hours, so that polypyrrole powder settle down at bottom. The polypyrrole powder filter out under vacuum and washed with distilled water several times to remove any impurities present. Polypyrrole dried for 2 days at room temperature.

Preparation of doped polypyrrole using following dopant

1,5-Naphthalene disulfonic acid disodium salt [1,5NDSA]

The composite powder was prepared by same procedure mention above only dopant 1,5-Naphthalene disulfonic acid disodium salt sodium salt with 10%,20%,30%, 40%, 50%,60% w/v added in pyrrole solution before addition of oxidant solution. For example, synthesis A6, B6, C6, D6, E6, F6 in Table 1. Then doped powder were obtained. After 24hrs it was filtered and washed with distilled water several times to remove any impurities present in it. Doped polypyrrole powder dried for 2 days at room temperature.

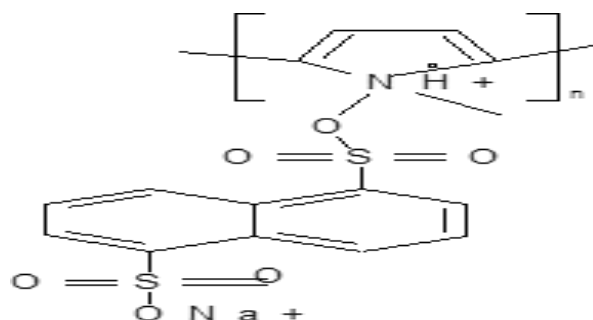


Fig: 1. PPY doped with 1,5 NDSA

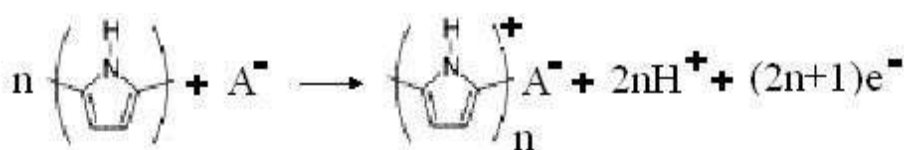


Fig.2 Chemical Reaction with anion

By using dopant Naphthalene Sulfonic Acid [NSA]

The polymerization was carried out either in neutral solution such as deionized water. An overview of synthesized materials is summarized in Table 1. Typical molar ratio of pyrrole: oxidizing agent: doping agent (when present) was 1:2: dopant Naphthalene Sulfonic Acid with 10%, 20%, 30%, 40%, 50%, 60% w/v, which means the polymerization proceeds in excess of oxidizing agent. For example, synthesis A7, B7, C7, D7, E7,

F7 in Table 1 proceeds as follows:

Firstly, 1M of pyrrole was added drop wise in dopant at different percent w/v stirred with magnetic stirrer until pyrrole and doping agent completely dissolved. and then polymerization take place on drop wise addition above mixture in 1M of oxidant APS and were added to 100 ml of distilled water at 25 °C.

The yield is given as follows in Table no. 1 (Monomer: oxidant =1:2 ratio)

Sr No.	Dopant (1,5NDSA)	Colour of product	Yieldin %	Sr No.	Dopant (NSA)	Colour of product	Yieldin %
A6	10 %	Black	30.59%	A7	10 %	Black	37.58%
B6	20%	Black	30.02%	B7	20%	Brown	37.92%
C6	30%	brown	32.92%	C7	30%	Brown	27.24%
D6	40%	brown	42.54%	D7	40%	Brown	34.19%
E6	50%	brown	49.19%	E7	50%	Brown	33.78%
F6	60%	brown	52.22%	F7	60%	Brown	15.51%

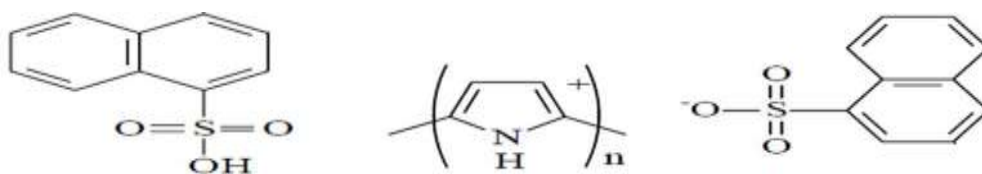
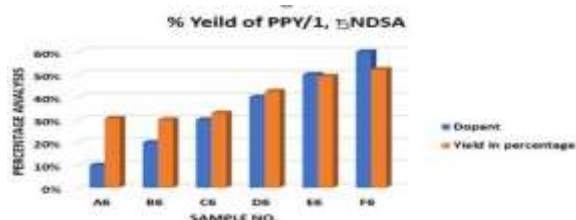


Fig:3 (Naphthalene sulfuric acid and Polypyrrole doped naphthalene sulfonic acid)PPY/NSA

This mixture was stirred continuously with the help of magnetic stirred for 4Hr until pyrrole and oxidant dopant mixture completely dissolved. Doped Polypyrrole produced was kept unagitated for 24 hours, so that polypyrrole powder settle

down at bottom. The polypyrrole powder filter out under vacuum and washed with distilled water several times to remove any impurities present. Polypyrrole dried for 2 days at room temperature.

a)



b)

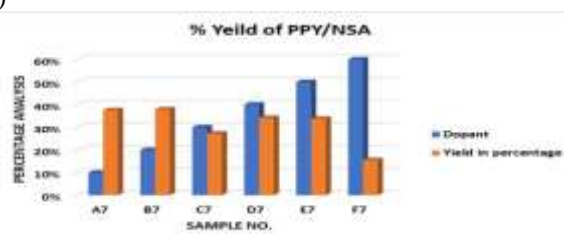


Fig.4: Effect of percentage of dopant (w/v) on yield of (a) PPY/1,5NDSA (b) PPY/NSA Table:-2 Overview of

Table:-2 Overview of synthesized PPY materials. The SO₄²⁻ dopants are residuals of APS.

Synthesis no.	Oxidizing agent	Doping agent	Dopants awaited in product
SPPY	APS	-----	SO ₄ ⁻²
A6 to F6	APS	1,5NDSA	Anion 1,5NDSA, SO ₄ ⁻²
A7 to F 7	APS	NSA	Anion NSA, SO ₄ ²⁻

Analysis and Measurements

FTIR spectra of all synthesized composite materials under investigation were recorded on Shimadzu FTIR-02634 (Serial no. A13749) Spectrophotometer between 400 and 4000cm⁻¹. NMR spectra of all synthesized composite materials under investigation were recorded on BRUKER ADVANCED II 400 NMR Spectrometer, SAIF, Punjab University, Chandigarh. The Polypyrrole was characterized by XRD using Analytical (Philips), model mini flex 600. The XRD patterns were recorded between 2θ = 10° to 40° The X-ray diffractometer uses CuKα radiation of θ = 1.5418 Å generated at 40 kV/20 ma. The morphology of Polypyrrole was examined using Scanning Electron Microscope (SEM) JEOL make JSM- IT200 model. The conductivity of different molar ratios of doped polypyrrole was performed by a Keithley 6571B High Resistance Meter and Electrometer using a four probe-setup

(DFP.2 model).

Fouries transform infrared (FTIR) investigation

The IR studied SPPY, PPY/1,5NDSA, PPY/NSA Doped (w/v) 20%,30%,40% synthesized in present research work are given in FTIR spectra Fig:6,7 respectively. The IR studied SPPY, PPY/1,5NDSA Doped (w/v) 20%,30%,40% synthesized in present research work are given in FTIR spectra Fig: 5. The C=C stretching vibration in the polypyrrole ring is represented by the band that occurs between 1541.12 and 1404.18 cm⁻¹. The C-N stretching vibration in the ring is represented by the band for in situ deposition occurring between 1342.46 cm⁻¹ and 1033.85 cm⁻¹. C-H or C-N in-plane deformation modes are attributed to the wide band between 1400 and 1200 cm⁻¹. The maximum frequency of the polypyrrole ring breathing vibration is at 1192.01 cm⁻¹, and it occurs between 1250 and 1100 cm⁻¹. The C-H ring

deformation is shown by the peak at 736.81 cm^{-1} . As it can be observed from figure 5, much sharper peaks are formed for longer deposition durations, suggesting a higher degree of polymerization. The

evidence of above IR stretching range is reported in elsewhere. [13] Some important peak ranges are summarized in following table no 3.

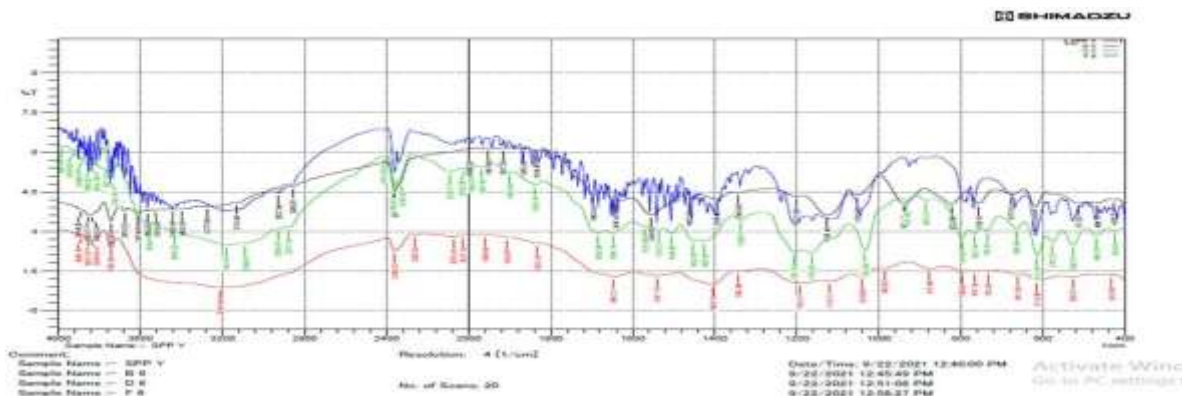


Fig - 5:- IR representation of SPPY and over lay of doped PPY/1,5NDSA (B6,D56,F6)

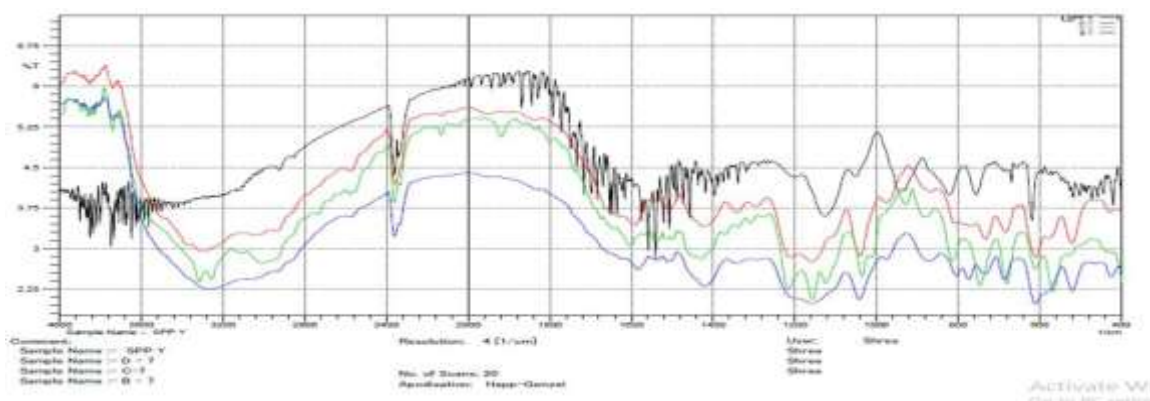


Fig - 6:- IR representation of SPPY and overlay of doped PPY/NSA (B7,D7,F7)

The IR studied SPPY, PPY/NSA Doped (w/v) 20%,30%,40% synthesized in present research work are given in FTIR spectra Fig: 6. FT-IR spectra of PPY in situ doped with NSA is similar to the spectra of PPY synthesized by a conventional method. The two bands at 1500 and 1700 cm^{-1} characteristic of the stretching vibration of $\text{C}=\text{C}$ and the different bands between 1475 and 1236 cm^{-1} due to the vibration modes of the pyrrole ring were observed as placed at 1051.20

the in-plane and out-of-plane vibration modes of N-H and C-H , respectively. Except for the in-plane and out-of-plane vibration in the range of $500\text{-}1000\text{ cm}^{-1}$, which may be caused by side reactions during polymerization, all of the typical absorption bands mentioned above were found. This indicates that the primary polymer chain's structure and that of the PPY doped with various sulfonic acids are highly similar

Above reference IR ranges were made a report somewhere. [14] Some important peakranges are summarized in following table no 3.

Table no. 3

Polymer composite	Position of absorption maxima (cm ⁻¹)						
	N-H stretching (Reference value 3500-3300cm ⁻¹)	Ar-H	Aromatic C=C stretching (Reference value 1700-1500cm ⁻¹)	C=N stretching (Reference value 1690-1640cm ⁻¹)	C-N stretching (Reference value 1350-1000cm ⁻¹)	C=C-H bending (Reference value 900-690cm ⁻¹)	S=O Stretching (Reference value 1375-1300cm ⁻¹ & 1350-1140 cm ⁻¹)
Pure PPY (SPPY)	Peak is observed in between range of 3743.83-3277.06 cm ⁻¹	2358.94 cm ⁻¹	1554.63, 1641.42 cm ⁻¹	1695.43 cm ⁻¹	1126.43 cm ⁻¹	821.68 cm ⁻¹	1342.46 & 1201.65 cm ⁻¹
Overlay of doped PPY/1,5NDSA (B6, D56, F6)	Peak is observed in between range of 3558.67-3182.55 cm ⁻¹	2360.87 cm ⁻¹	Peak is observed in between range of 1541.12, 1404.18 cm ⁻¹	Peak is observed in between range of 1695.43 - 1683.86 cm ⁻¹	Peak is observed in between range of 1342.46 - 1033.85 cm ⁻¹	Peak is observed in between range of 85.33 - 736.81 cm ⁻¹	1342.46 cm ⁻¹ & 1192.01 cm ⁻¹

			cm ⁻¹	cm ⁻¹	m ⁻¹		
Overlay of doped PPY/NSA (B7, D7, F7)	Peak is observed in between range of 3442.94-3277.06 cm ⁻¹	2358.94 cm ⁻¹	Peak is observed in between range of 1554.63-1695.43 cm ⁻¹	Peak is observed in between range of 1641.42-1690 cm ⁻¹	Peak is observed in between range of 1051.20-1342.46 cm ⁻¹	Peak is observed in between range of 821.68-756.10 cm ⁻¹	1342.46 cm ⁻¹ & 1201.65 cm ⁻¹

NMR spectra of SPPY/ doped PPY

The NMR spectroscopy is an important tool for structural determination of any chemical compound. It easily recognizes the aromaticity and conjugation in the substance. It also gives

information of heteroatomic chemical environment. The NMR spectra given in the (fig.7,8,9) which revealed that NMR spectra of SPPY, PPY/1,5 NDSA (E6) and PPY/NSA (E7) shows three distinct peaks.

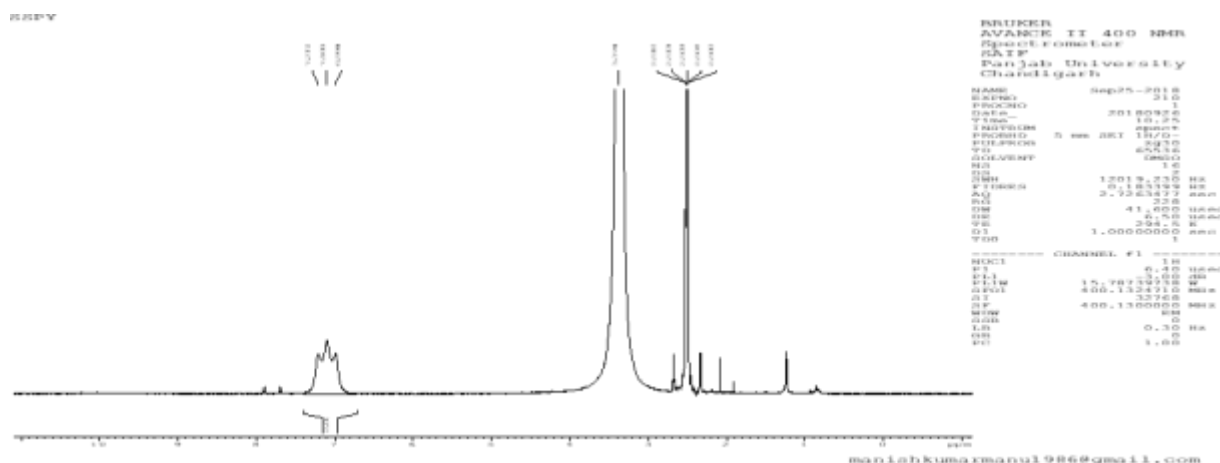


Fig: -7: NMR spectra of SPPY(undoped polypyrrole)

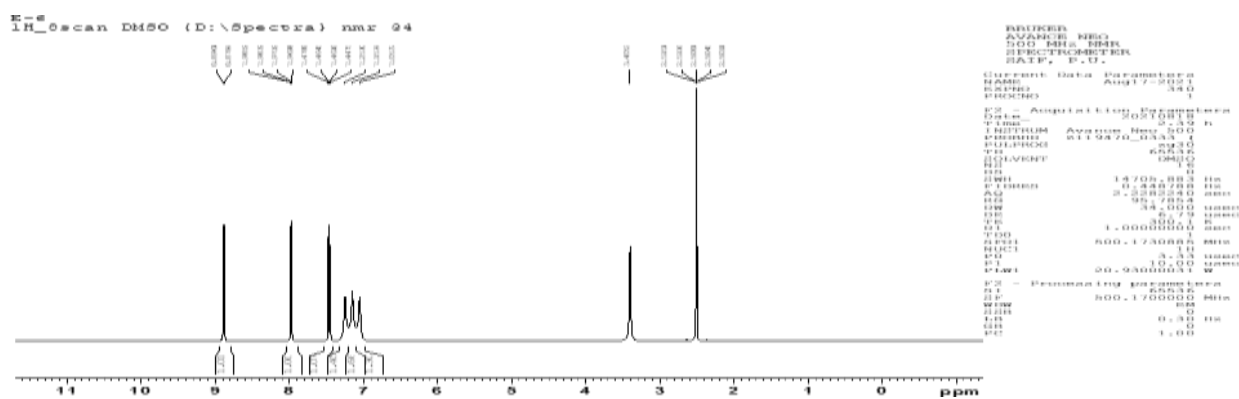


Fig:8-NMR spectra of PPY /1,5 NDSA (E6)

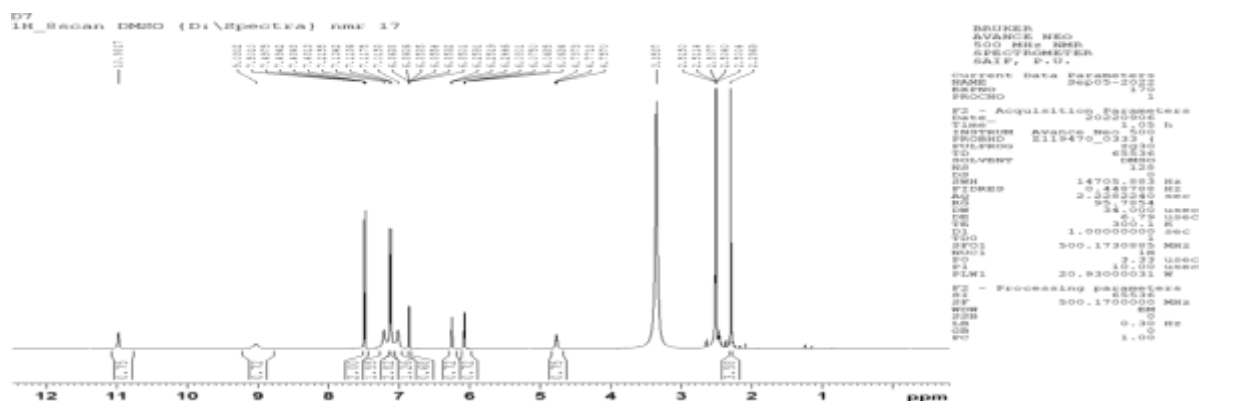


Fig: 9-NMR spectra of PPY /NSA (E6)

The first peak is obtained for C=C-H proton at δ 2.5093, 2.5091, 2.5081 in SPPY, PPY/1,2NQA, PPY/1,5 NDSA respectively. The N-H stretching δ is 3.3740, 3.4053 and 3.35 in SPPY, PPY/1,5 NDSA respectively. The peak corresponding to aromatic proton is observed that δ 7.1002 in SPPY-7.100 (t) and 6.9(dd), 7.0(t), 7.4(q), 8.8(d), indicate that aromatic compound such as 1,5 naphthalene disulfuric acid disodium salt used as dopant in it. The peak corresponding to aromatic proton is

observed that δ 6.00 ,6.600 (m), 6.9(dd), 7.0(d), 8.5(d) indicate that 7.4(dd),7.98 (dd) ,9.0(m), indicate that aromatic compound such as naphthalene sulfuric acid disodium salt used as dopant in it. The increase in δ value of aromatic proton may be due to presence of high concentration of anionic dopant ions in the structure chain after doping. The change in position of aromatic proton may be a consequence which highly affects the conductivity of the sample.

SEM Morphology

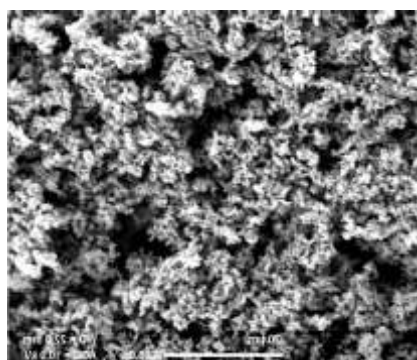


Fig. 10: SEM SPPY

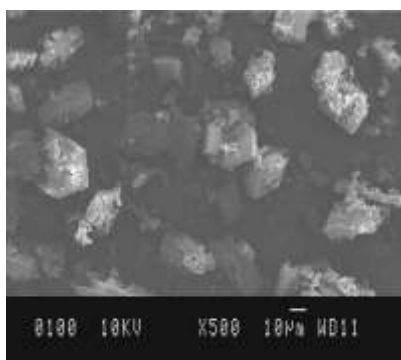


Fig.11: SEM of PPY/1,5 NDSA

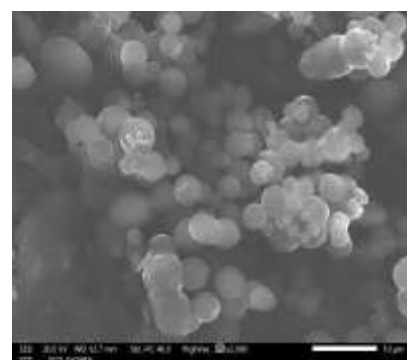


Fig.12: SEM of PPY/NSA

Figure 10,11,12 displays the comparison of the morphology of SPPY and doped PPY samples with different anionic surfactant like PPY/1,5NDSA and PPY/NSA. The PPY/1,5NDSA (fig.11) particles are clearly larger than the undoped PPY particles. The ratio of the surfactant and dopant, which changes the solution's viscosity and is likely to cause the surfactant and dopant to adsorb onto developing polypyrrole particles, caused the average particle size to change. Agglomerated PPY/1,5 NDSA microstructures have the appearance of flat rocks. Lower conductivity may result from this particle agglomeration when the molar ratio of 1,5 NDSA is raised. It was discovered that the surface of the polypyrrole doped with 1,5 NDSA is significantly affected by the increased molar ratio of the compound. The homogeneous distribution of holes and clusters of crystallites throughout the material in PPY/NSA (fig. 12) suggests that nanoparticles interact with

PPY. Different doping substances produce particles with varying sizes. It was shown that the shape of PPY samples was significantly impacted by the presence of the surfactant during polymerization.

X-Ray Diffraction Analysis

The sample's XRD patterns exhibit a broad peak in the range $15^{\circ} < 2\theta < 30^{\circ}$, indicating that the polypyrrole powder that results are amorphous in nature. [15] Similar range and extremely sharp peaks in the PPY/1,2NDSA composites, which are evenly dispersed in the polymer matrix (Fig. 13), indicate high crystalline in nature. As (PPY/NSA) fig.18 XRD pattern indicates, intermediate crystalline structure/semicrystalline structure of molecules is indicated by a less sharp peak compared to PPY/1,2NDSA. A typical X ray diffraction pattern for polypyrrole prepared using APS as oxidant is shown in Fig.16.

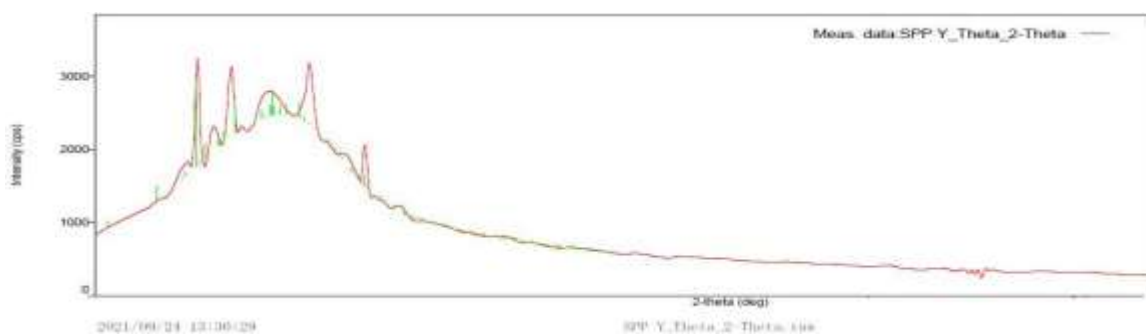


Fig.13: X-ray diffraction patterns of SPPY

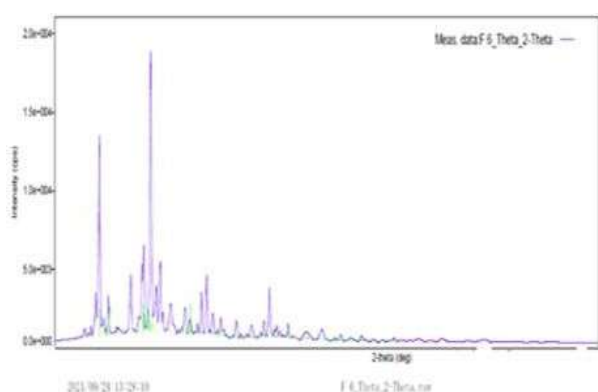


Fig.14: XRD Spectra of PPY/1,5NDSA

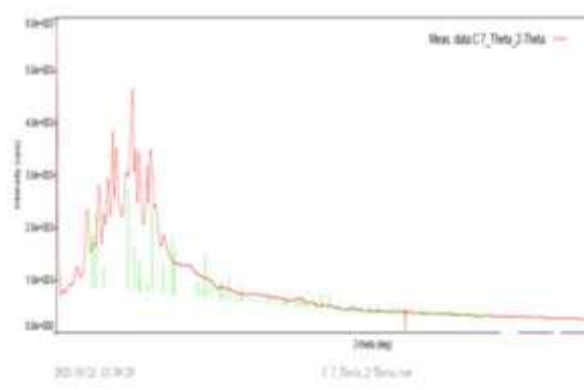


Fig.15: XRD Spectra of PPY/NSA

Conductivity measurements for various molar ratios of PPY/1,5NDSA and PPY/NSA

In this arrangement, pressure contact was used to sandwich the samples between the copper

electrodes [16]. The temperature was adjusted from room temperature (313 K) to 343 K while the voltage, V, kept constant.

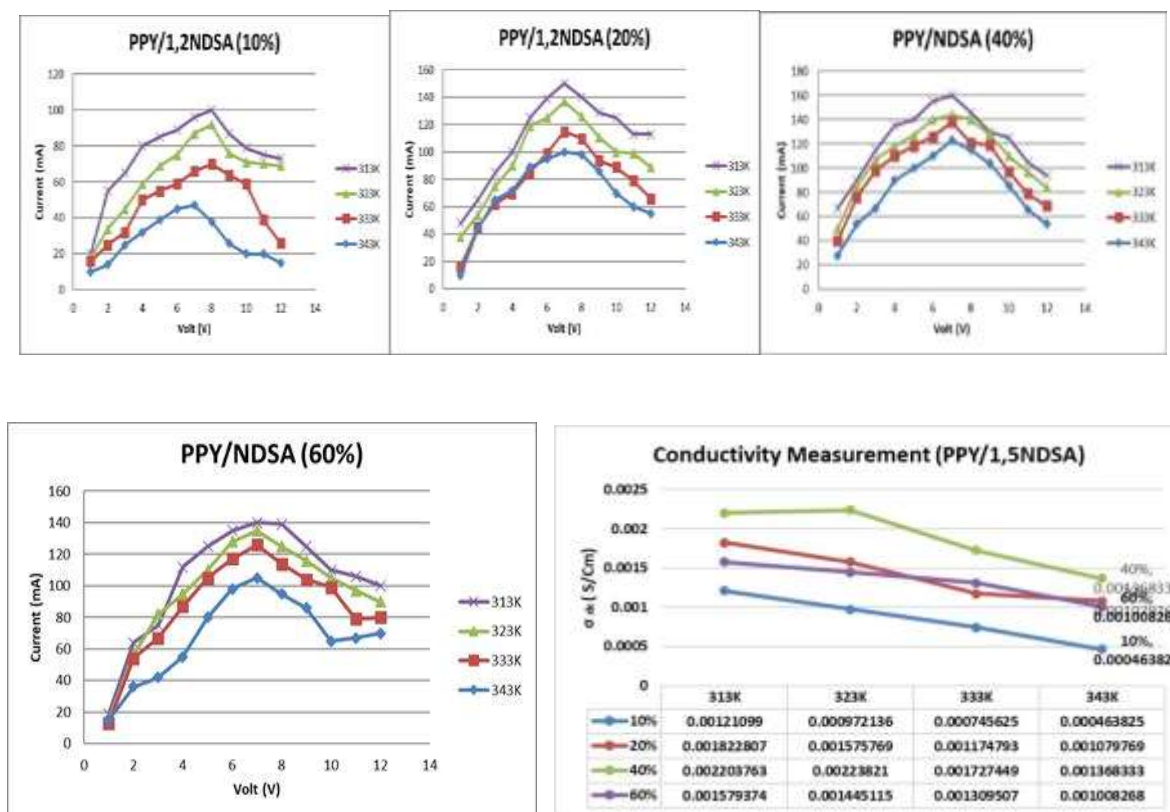


Fig.16.Current-voltage characteristics for various concentration of PPY/1,5NDSA

The current values were recorded with increase in temperature. The resistivity at different temperatures was calculated using the relation.

$$\rho = 4.5324 \times V \times d / I \times F$$

where, $F = d/s$ and I is current, V is voltage, d is thickness of the pellet (2.234 mm), F is correction factor and S is distance between two probes and distance of the probe which was 1.85 mm. The conductivity was calculated using the relation.

$$\sigma = 1/\rho$$

where ρ is resistivity; the correction factor depends on the sample geometry, thickness, borders and location of points in the sample. I-V characteristics for the PPy dopant-to-monomer ratio with varying NDSA and NSA concentrations for the synthesized polypyrrole powders (10–60%) are shown in Figs. 19 and 20 respectively. After NSA concentrations above 40%, the conductivity at room temperature

somewhat decreases with an increase in NSA concentration. It is observed that as dopant concentration increases from 10-40% conductivity increases but further increasing dopant concentration from 50-60% rather than it stable but fall down slightly. In PPY/NSA and PPY/1,5 NDSA pallet were used the initial part of rising current peaked at 4-8 volts respectively. After this peak when voltage was increased further, a decrease in current was observed. The reason for such departure from Ohm's law is caused by either the sample degrading at higher voltage or the current being primarily contributed by ions. As the I vs. V plot's temperature variation demonstrates, current decreases between 301K - 343K. We are looking at more evidence that ions contribute to the current. The conductivities were discovered to be in the 1.0×10^{-3} to 4.5×10^{-3} S/cm range.

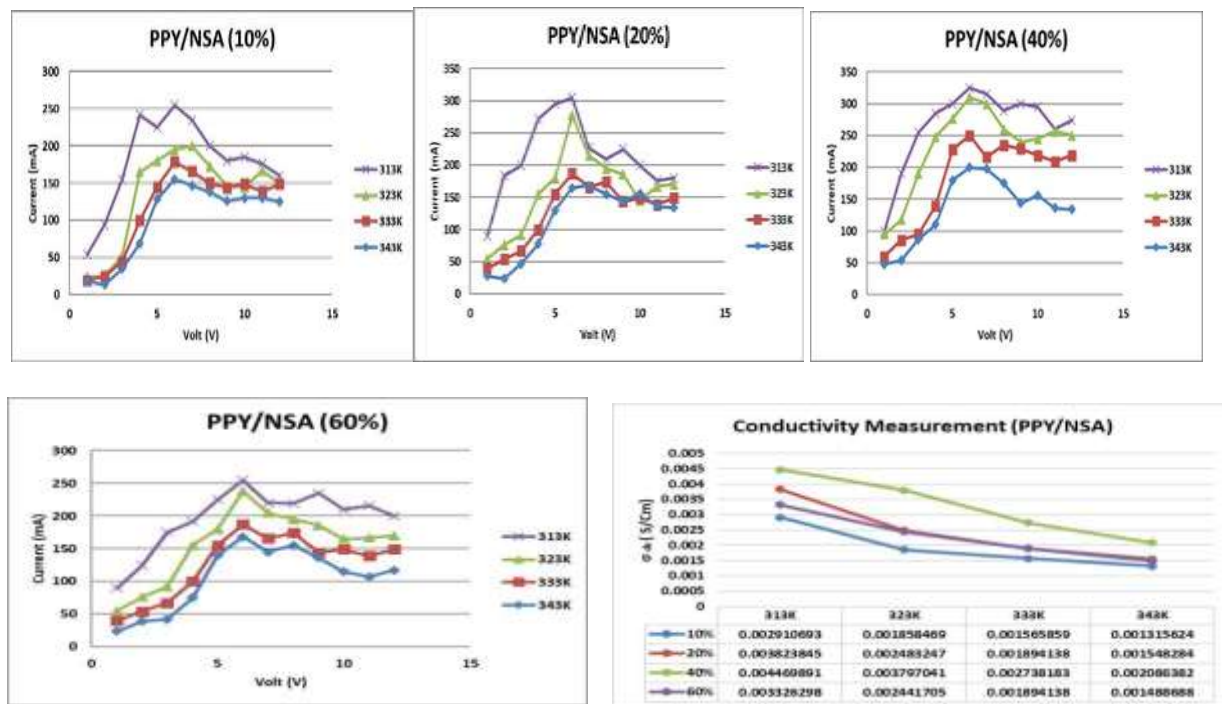


Fig.17.Current-voltage characteristics for various concentration of PPY/NSA

At various temperatures between 313K and 343K, the relationship between the electrical conductivity of polypyrrole synthesized with APS and their dopants (10%-60%) was examined. It was shown that the conductivity measurement increases up to a dopant concentration of 40% before gradually decreasing for samples with a dopant concentration of 50–60%. These actions resemble the behavior of metallic conductors. It should be noted that when pure pyrrole doped behaves more like a semiconductor but when undoped acts more like an insulator.

Comparison between conductivities of PPY/1,5NDSA and PPY/NSA composite

The conductivity is determined by the intrinsic conjugation length, and longer the conjugation length the easier mobility for carriers hopping by intra-chain and/or inter-chain. Since the conjugation length of PPY/NSA and PPY/NDSA in both composites is the same, the conductivity of the two composite materials differs. The difference between PPY/NDSA and PPY/NSA in the number of sulphonate groups per unit of polymerization could be the issue. The conductivities of the doped polymer PPY/NDSA, as compared to PPY/NSA, are found to decrease when the number of sulphonate groups increases on the same conjugation length of dopant molecules. It is mention that the increase in the number of sulfonate groups in a doped molecule causes localization of positive charge carriers in polypyrrole films, resulting in the observed

decrease of the film conductivity [17]. On the other hand, excess of dopant was absorbed onto the surface of pyrrole It formed protected envelope around the products at higher concentration, which prevented further polymerization. So, the conductivity of PPY/NSA and PPY/NDSA increased from molar ratio 10-40% (w/v) and then decreases from 50-60%(w/v) because of higher concentration of dopant. It has been noted that while SPPY reacts like an insulator at room temperature, polypyrrole conductivity is caused by dopant concentration and acts like a metallic conductor.

2. Conclusion

- SPPY, PPY/NSA and PPY/1,5NDSA composite were successfully synthesized by chemical oxidation. The % of yield increases with dopant concentration.
- FT-IR and NMR studies confirm that doping causes structural changes in the polymer chains supported by SEM Image.
- XRD Patterns demonstrated the amorphous, high crystalline in nature of the PPY/NSA. As (PPY/NSA) XRD pattern shows that less sharp peak as compared to PPY/1,2NDSA indicating moderate crystalline structure (fibrillar) of molecules. Supported by SEM image.
- As number of sulphonate groups increases on same conjugation length of dopant molecules then conductivities of doped polymer PPY/NDSA decreases as compared to PPY/NSA.As one more

sulphonates group present on PPY/NDSA.

- It is proposed that excess of dopant was absorbed onto the surface of pyrrole. It formed a protected envelope around the products at higher concentration, which prevented further polymerization. So, the conductivity of PPY/NSA and PPY/NDSA increased initially then decreases because of higher concentration of dopant.
- NSA, 1,2NDSA doping PPY provided both high conductivity and better solubility. Organic acids (sulfonic) were found to be suitable for use as dopants to produce more stable, more soluble (improved processability) and more conductive polypyrrole.
- As a result, conductive PPY/1,5NDSA and PPY/NSA polymers with advanced optical properties seem to have a good interface potential for the pro devices such as diodes and transistors.
- When bulky dopant molecular concentration increases, which prevented further polymerization. So, the conductivity of doped PPY decreased.

Acknowledgement

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