

## POLYPYRROLE-SODIUM BENTONITE COMPOSITES AND ITS ELECTRICAL AND OPTICAL PROPERTIES

Aye Myat Maw<sup>1</sup>, Mya Theingi<sup>2</sup>, Khin Than Yee<sup>3</sup>

### Abstract

The application of bentonite to polymer industry is growing year by year. Bentonite is hydrophilic in nature and polymer is hydrophobic character. This makes the chemically incompatible. Therefore, NaCl treated bentonite as filler was used in the presence of sodium dodecyl sulphate as surfactant and pyrrole monomer to prepare polypyrrole-sodium bentonite composites by in-situ chemical oxidative polymerization method. In the preparation of polypyrrole-sodium bentonite composites (PPy-SDS-NaB), the various amounts of bentonite (4%, 8% and 12% w/v) were used. The resultant composites were designed by PPy-SDS-NaB 1, PPy-SDS-NaB 2 and PPy-SDS-NaB 3, respectively, where, PPy-SDS-NaB means polypyrrole-sodium bentonite composites in the presence of sodium dodecyl sulphate and the numbers refer to the amount of bentonite compositions. The prepared composites were characterized by FT IR, XRD, SEM and TG-DTA techniques. Their electrical properties were analyzed in the frequency range of 1 to 10 MHz by LCR measurement. From the analysis of LCR measurement, it was observed that PPy-SDS-NaB 1 has more electrical conductivity than other two composites. The optical property of the prepared composites were also analyzed in the wavelength range of 200 to 600 nm by using UV-Vis spectrophotometer and the optical band gaps were calculated by Taucs relation. From the analysis of absorption spectra, it was found that the band gaps of prepared composites are in the semiconductor wide band gap ranges of 4.0 eV to 3.8 eV and that shows great promise in sensors as well as electronic applications.

**Keywords:** Polypyrrole, Sodium-Bentonite, In-situ Chemical Oxidative Polymerization, Sodium Dodecyl Sulphate, Electrical Conductivity, Optical Property

### Introduction

Polymer composites can be defined as polymers that have been filled with natural or synthetic compounds to improve their properties. The polymer is called "matrix", if the filler is in the nanometer range, the composite is called "nanocomposite" (Hernandez-Hernandez *et al.*, 2016). Nanocomposite polymer materials filled with organo-bentonite are growing development. The addition of an organo-bentonite to the polymer matrix leads to increase of the mechanical strength, thermal stability, gas permeability and water-repellent properties of the polymers (Yoleva *et al.*, 2016). In the energy industry, the polymer nanocomposites positively affect the creation of forms of sustainable energy by offering new methods of energy extraction from benign and low cost resources. One example is the fuel cell membranes; other applications include solar panels, nuclear reactors and capacitors (Anadao, 2012).

Conductive polymers, also known as synthetic metals, have electric, magnetic and optical properties which can be compared to those of semiconductors. They are also called conjugated polymers because they present conjugated C=C links in their main chains, which allows for the creation of an electron flux under certain conduction. The conductivity of this kind of polymer depends on the polymer chain order at the moment of synthesizing the nanocomposite (Hernandez-Hernandez *et al.*, 2016).

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Conducting polymers such as Polyaniline (PAni), Polypyrrole (PPy), and Polythiophene (PTh), have found many industrial applications due to their unique characteristics, such as the dual function of ionic and electronic conductivity, large surface area and good mechanical properties (Utami *et al.*, 2016). The standard conducting polymers (CPs; i.e., Polypyrrole (PPy), Polythiophene (PTh), Polyaniline (PANI)) can be polymerized both chemically and electrochemically. Among the conducting polymers, PPy is one of the most frequently studied polymers because of high electric conductivity, ion exchange property, environmental stability as regards oxygen and water, and ease of synthesis (Wang *et al.*, 2001; Bouabdallah and Djelali, 2014). Therefore, it has been widely used in electrochemical applications (e.g., as an electrode material, in electrochemical energy storage, as a catalyst support in fuel cells, in supercapacitors and in solar cells (Utami *et al.*, 2016)

The present work reports the preparation of polypyrrole-sodium bentonite composites in the presence of sodium dodecyl sulphate on the local bentonite in Myanmar. Three methods are widely used in the polymer/clay nanocomposite preparation. They are in-situ polymerization, solution dispersion and fusion intercalation methods. In this work, it is proposed to use in-situ chemical oxidative polymerization method because it is ease of synthesis (Anadao, 2012).

The objective of this work is to study electrical conductivity and optical property for polypyrrole-sodium bentonite composites based on polypyrrole and organobentonite. Bentonite clay was used as filler in conductive composites because it is naturally abundant in Myanmar, low cost, ion exchange properties and has high thermal resistance and shell-shaped crystalline structure with nanometric thickness.

## Materials and Methods

### Sample Collection

Pyrrole monomer was purchased from Aladdin Industrial Corporation, Shanghai and used without further purification. Ferric chloride was purchased from Super Shell Chemical store at 27<sup>th</sup> street, Pabedan Township, Yangon and used as an oxidizing agent. Sodium chloride, supported by Physical Chemistry Laboratory, was used for the alkaline activation. Sodium dodecyl sulphate (SDS),  $\text{NaC}_{12}\text{H}_{25}\text{SO}_4$  (Mw 288  $\text{g mol}^{-1}$ ) was used as surfactant for the organic modification. Purified bentonite clay was collected from Saga Inn village, Ta Da U Township. Distilled water was used as the solvent in all analyses.

### Synthesis of the Activated Bentonite (NaB)

The pure bentonite was saturated with sodium ions by stirring for 10 hours in a 0.01 M sodium chloride solution. Then the filtered solid was washed with distilled water to remove the excess salt. Sodium-Bentonite (NaB) was dried at 100 °C, ground and sieved by 100 mesh (Motawie *et al.*, 2014).

### Synthesis of Polypyrrole and Polypyrrole-Bentonite Composites

First, 3.4 mL of pyrrole monomer was added to 25 mL of ethanol under constant stirring at 5-10 °C. Next, 25 mL of 0.1 M  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  solution was added to the monomer drop wise. The reaction mixture was stirred at 5-10 °C for 1 h. The resulting black precipitate was filtered

and washed with distilled water to remove unreacted monomer and excess ferric chloride. The black precipitate was dried at 60-70 °C in the incubator.

Polypyrrole-bentonite composites were prepared by adding different amounts of sodium-bentonite clay (1 g, 2 g and 3 g) with sodium dodecyl sulphate to pyrrole monomer. First, bentonite clay was dispersed in 25 mL of water under constant stirring. Sodium dodecyl sulphate and Pyrrole monomer was added drop by drop to sodium-bentonite clay suspension by constant stirring at 5-10 °C and then an oxidizing agent was mixed with clay-monomer-surfactant solution dropwise under constant stirring and they were dispersed in an ultrasonic bath for half past hour. The purification step was performed as described above. The resulting composites were denoted as PPy-SDS-NaB X, where PPy-SDS-NaB means Polypyrrole-Sodium Bentonite composite and X refers to the mass of sodium-bentonite clay. For example, PPy-SDS-NaB 1 means that the used mass of sodium-bentonite is 1 g. So, the composites were designated as PPy-SDS-NaB 1, PPy-SDS-NaB 2 and PPy-SDS-NaB 3, respectively.

### Preparation of Pellets

The obtained black powders (PPy and PPy-SDS-NaB composites) were pressed into pellets with diameter 1.5 cm and thickness 0.16 cm using MAEKAWA Testing machine.

### Characterization of the Prepared Samples

The structural characterization of polypyrrole-sodium bentonite composites was analysed using FT IR. The interaction of polymer-bentonite composites was investigated by XRD analysis. The morphological structure of the prepared composites was characterized by SEM. Thermal properties of the composites were analysed by TG-DTA. The frequency dependent electrical conductivity were also determined by LCR meter in the frequency range of 1-10 MHz and optical property were also studied in the wavelength range of 200 to 600 nm by UV-Vis spectrophotometer.

FT IR spectrum was recorded in the range of 4000-400  $\text{cm}^{-1}$  by using 8400 SHIMADZU, Japan FT IR spectrophotometer.

X-ray diffraction (XRD) analysis was carried out using Rigaku X-ray Diffractometer, RINI 2000/PC software, Cat. No 9240 J 101, Japan. Copper tube with nickel filter was used. The diffraction pattern was recorded in terms of  $2\theta$  in the range of 10-70 °.

The scanning electron microscopy (SEM) images were obtained using JSM-5610 Model SEM, JEOL-Ltd., Japan.

Thermogravimetric analyses of samples were performed using TG-DTA apparatus, (Hi-TGA 2950 model). The temperature ranged between 0 °C and 600 °C under nitrogen gas (at a rate of 50 mL/min).

For the electrical conductivity measurements, the obtained samples were pressed in the form of pellet using MAEKAWA Testing machine. The dielectric permittivities such as D, K and tangent loss of composites were determined using LCR-B110G meter (DC 20-10 MHz) in the frequency ranged of 1-10 MHz at ambient temperature. Frequency dependent electrical conductivity was evaluated by using dielectric equation;

$$C = \frac{\epsilon' \epsilon_0 A}{d}$$

$$\tan \delta = 2 \pi f R C$$

$$\omega = 2 \pi f$$

$$\sigma_{ac} = \omega \tan \delta \epsilon' \epsilon_0$$

where, C is capacitance (pF),  $\epsilon'$  is dielectric constant,  $\epsilon_0$  is electrical permittivity in vacuum ( $8.85 \times 10^{-14}$  F cm<sup>-1</sup>), d is sample thickness (cm),  $\omega$  is circular frequency (MHz),  $\tan \delta$  is dielectric loss tangent and  $\sigma_{ac}$  is electrical conductivity ( $\mu$ S cm<sup>-1</sup>)

For their optical property, the absorbance and transmittance of PPy, PPy-B and PPy-CP-B composites were measured by UV spectrometer. Their optical absorption spectra have been recorded in the wavelength range from 200 nm to 600 nm. Optical band gap of them has been evaluated by using Taucs relation.

$$\alpha = 2.303 \left( \frac{A}{t} \right) \quad R = \left( \frac{n-1}{n+1} \right)^2 \quad \sigma = \frac{\alpha n c}{4\pi}$$

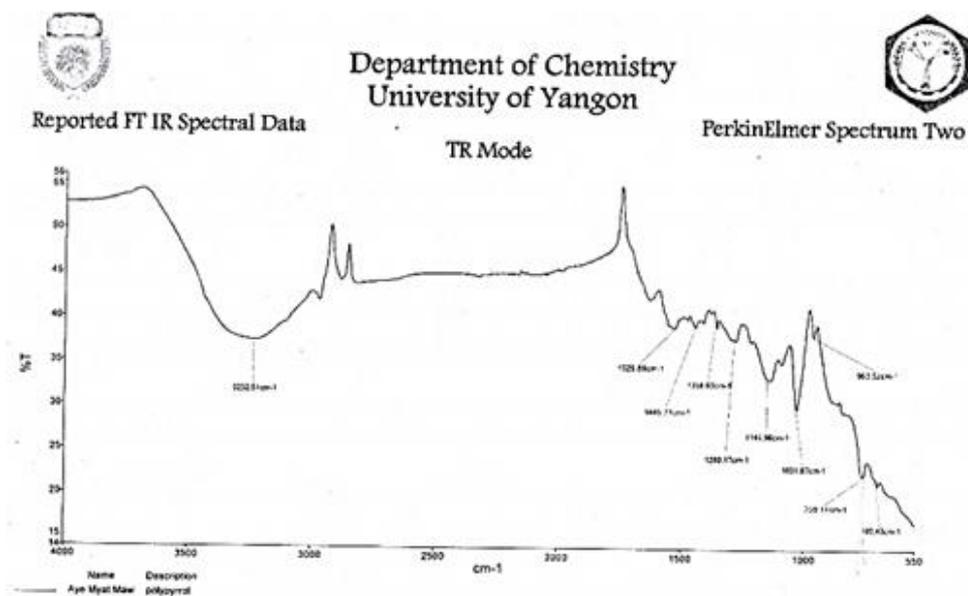
where,  $\alpha$  = molar absorption coefficient (cm<sup>-1</sup>), A = absorbance, t = thickness of matter, R=reflectance, n = refractive index, c = velocity of light in the space and  $\sigma$  = optical conductivity.

## Results and Discussion

Polypyrrole and polypyrrole-sodium bentonite composites were synthesized by the method described in materials and method section. The resultant polypyrrole and composites are black in color because they are fully oxidized. They were characterized by modern techniques (FT IR, XRD, SEM and TG-DTA). Moreover, their electrical and optical properties were investigated by LCR meter and UV-Vis spectrophotometer.

### FT IR Analysis

Figure 1 shows the FT IR spectrum of polypyrrole. The assignment data is summarized in Table 1. In the spectrum of polypyrrole, the peak observed at 3232 cm<sup>-1</sup> corresponds to N-H stretching vibration and the peak at 1529 cm<sup>-1</sup> corresponds to N-H bending vibration. The peak at 1031 cm<sup>-1</sup> can be attributed to C-H deformation of polypyrrole. The C-N stretching absorption band was detected at 1145 cm<sup>-1</sup>. In the spectrum of bentonite, the bands observed at 3696 cm<sup>-1</sup> and 1641 cm<sup>-1</sup> can be attributed to O-H stretching and O-H bending vibrations. The peaks observed at 1045 cm<sup>-1</sup> is due to Si-O bond stretching vibrations, while the band at around 918 cm<sup>-1</sup> corresponds to Al-O stretching vibration. The peaks at 1033 cm<sup>-1</sup> and 1383 cm<sup>-1</sup> in all the spectra of all composites can be attributed to Si-O stretching vibration and C-H deformation in polypyrrole (Figure 2 and Table 2). The characteristics peaks of PPy and bentonite in the composites indicate the interaction of PPy in bentonite clay (Silverstein *et al.*, 2003).

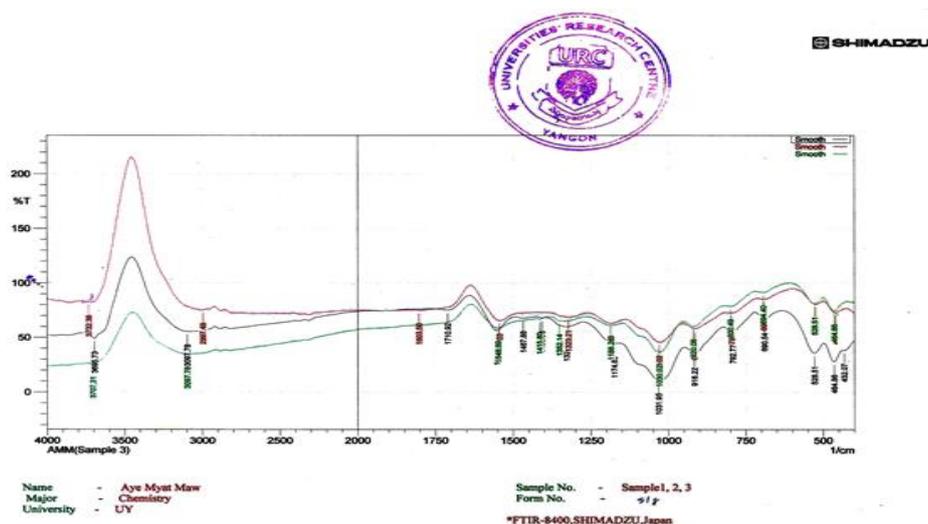


**Figure 1** FT IR spectrum of the prepared polypyrrole

**Table 1** FT IR Band Assignments of the Prepared Polypyrrole

| Observed wave number (cm <sup>-1</sup> ) | *Literature wave number (cm <sup>-1</sup> ) | Band Assignment                     |
|--|---|-------------------------------------|
| 3232                                     | 3460-3280                                   | stretching vibration of polypyrrole |
| 3107                                     | 3100-3000                                   | H stretching in aromatic ring       |
| 2970 }<br>2881 }                         | 2970-2850                                   | stretching (asym) and (sym)         |
| 1529                                     | 2565-1475                                   | bending                             |
| 1445                                     | 1460-1440                                   | ring stretching                     |
| 1280 }<br>1145 }                         | 1280-1180                                   | stretching                          |
| 1031                                     | 1050-1030                                   | deformation in polypyrrole          |
| 963                                      | 980-960                                     | bending                             |
| 759                                      | 770-690                                     | out of plane bending                |

\* Silverstein *et al.*, 2003



**Figure 2** FT IR spectra of (a) PPy-SDS-NaB 1, (b) PPy-SDS-NaB 2 and PPy-SDS-NaB 3 composites

**Table 2** FT IR Band Assignments of PPy-SDS-NaB Composites

| Observed wavenumber (cm <sup>-1</sup> ) |               |               | * Literature wavenumber (cm <sup>-1</sup> ) | Band Assignment                     |
|---|---------------|---------------|---|-------------------------------------|
| PPy-SDS-NaB 1                           | PPy-SDS-NaB 2 | PPy-SDS-NaB 3 |   |                                     |
| 3732                                    | 3707          | 3695          | 3700-3200                                   | O-H stretching (and) N-H stretching |
| 2997                                    | 3097          | 3097          | 3100-3000                                   | = C-H stretching in aromatic ring   |
| 1803                                    | -             | 1710          |   |                                     |
| 1545                                    | 1548          | 1552          | 1650-1550                                   | O-H bending (and) N-H bending       |
| -                                       | 1415          | 1467          | 1460-1440                                   | C-C ring stretching in polypyrrole  |
| 1323                                    | 1352          | 1329          | 1450-1200                                   | C-H deformation of polypyrrole      |
| 1186                                    | 1186          | 1174          |   |                                     |
| 1030                                    | 1030          | 1031          | 1110-830                                    | Si-O stretching                     |
| 916                                     | 920           | 916           | 950-900                                     | C-H bending (or) Al-OH bending      |
| 796                                     | 800           | 792           | 790-750                                     | Si-O bending                        |
| 692                                     | 694           | 690           | 721-681                                     | Si-O-Si stretching                  |
| 524                                     | 528           | 528           | 525   | Al-O stretching                     |
| 461                                     | 484           | 464           | 700-450                                     | Mg-OH stretching                    |

\* Silverstein *et al.*, 2003

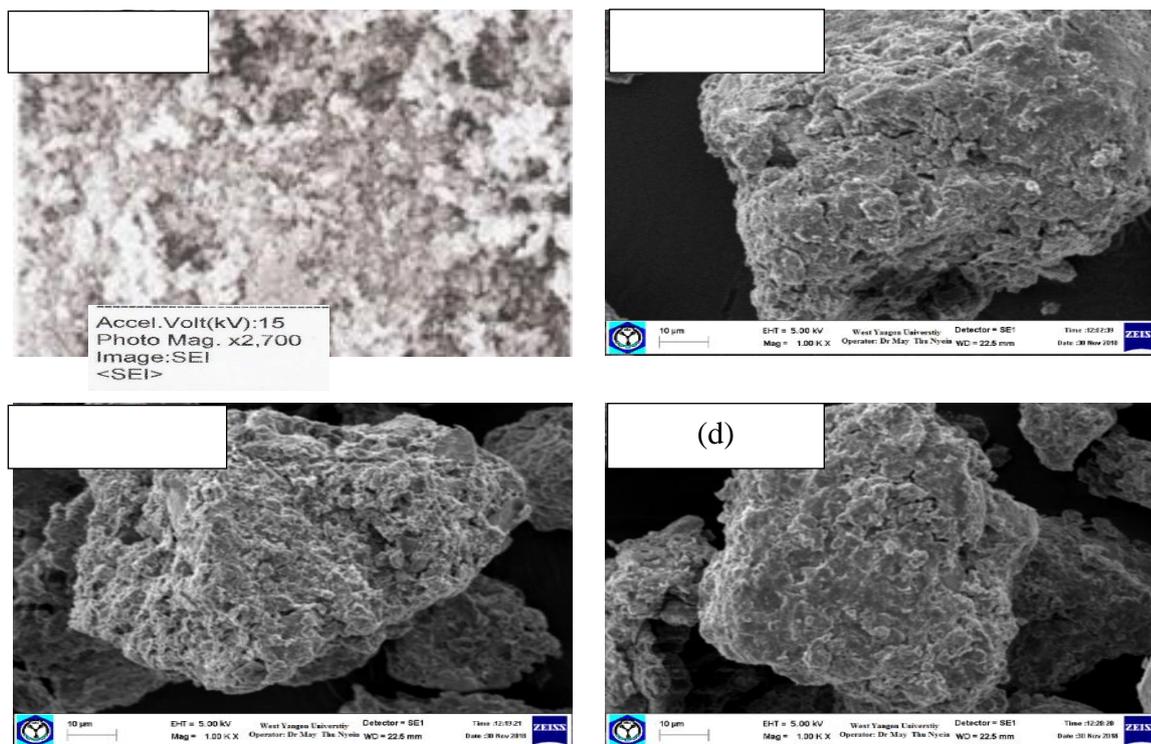


**Table 3 Average Crystallite Sizes of PPy-SDS-NaB Composites**

| Sample        | 2 $\theta$ | d (A $^\circ$ ) | h k l | F W H M | Average Crystallite Sizes (nm) |
|---------------|------------|-----------------|-------|---------|--------------------------------|
| PPy-SDS-NaB 1 | 26.633     | 3.3442          | 011   | 0.170   | 48                             |
|               | 36.538     | 2.4572          | 110   | 0.162   |                                |
| PPy-SDS-NaB 2 | 26.680     | 3.3384          | 011   | 0.151   | 53.5                           |
|               | 36.563     | 2.4556          | 110   | 0.147   |                                |
| PPy-SDS-NaB 3 | 26.649     | 3.3423          | 011   | 0.118   | 58                             |
|               | 36.559     | 2.4558          | 110   | 0.163   |                                |

### SEM Analysis

Figure 4 (a-d) shows the morphological differences between the polypyrrole and PPy-SDS-NaB composites. The surface morphology of polypyrrole is in the globular structure as shown in Figure 4 (a). After polymerization, the composites show significant changes in morphology as shown in Figures 4 (b-d). The surface morphology of PPy-SDS-NaB composites are as agglomerates. They are not much difference in their morphology. It may be due to homogeneous distribution of bentonite and polypyrrole. The structures of composites exhibit more ordered and denser structure with an increasing amount of bentonite clay.

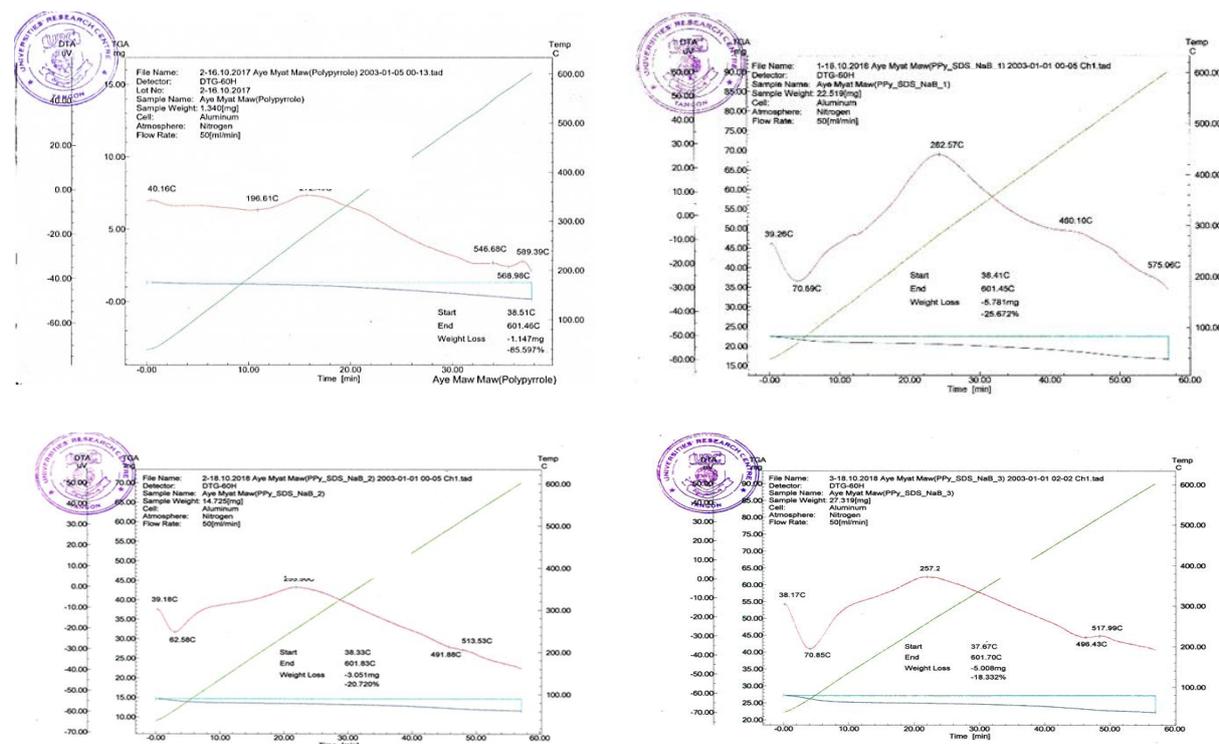


**Figure 4** SEM micrographs of (a) polypyrrole, (b) PPy-SDS-NaB 1, (c) PPy-SDS-NaB 2, and (d) PPy-SDS-NaB 3 composites

### Thermal Analysis

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) thermograms of prepared polypyrrole and prepared polypyrrole-bentonite composites are shown in Figure 5. Weight loss percent of PPy-SDS-NaB composites are listed in Table 4. TGA curve of prepared PPy indicates that there are two stages of decomposition in Figure 5 (a). Small exothermic peak observed at 40 °C with 10.08 % weight loss is due to the loss of volatile materials of PPy. Exothermic peaks observed at 272 °C and 589 °C with the 75.51 % weight loss is due to the thermal decomposition of polymer chain. It was observed that prepared PPy shows a poor thermal stability.

For PPy-SDS-NaB composites, three stages of weight loss are also observed. First weight loss in the temperature range between 38-100 °C is due to the dehydration of absorbed water and moisture. Second weight loss in the temperature range between 100-375°C may be due to the cleavage of CH<sub>3</sub>-, CH<sub>2</sub>- from surfactant molecule and the loss of volatile materials of polypyrrole and the final weight loss in the temperature range between 375-600 °C is due to dehydroxylation of bentonite samples. It can be seen that the incorporation of PPy with bentonite increases the thermal stability of PPy-SDS-NaB composites relative to that of PPy. This enhancement in thermal stability is due to the fact that the introduction of well-dispersed bentonite can prevent the heat to transmit quickly and then improve the thermal stability of composites. Therefore, polypyrrole-sodium bentonite composites exhibit higher thermal stability than pure polypyrrole. According to data in Table 6, it was found that more increase bentonite content in composites, higher thermal stability in them. When compare to other polymer composites (PPy-SDS-NaB 1 and PPy-SDS-NaB 2), PPy-SDS-NaB 3 has 18.33 % of weight loss.



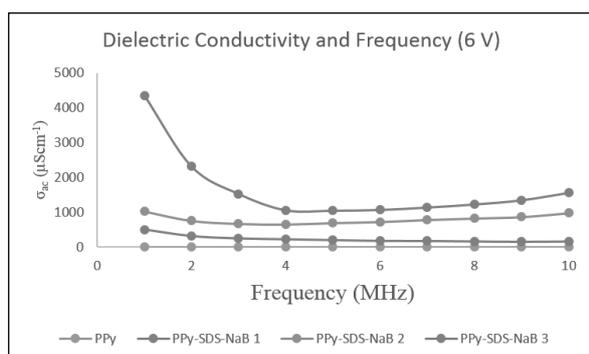
**Figure 5** TG-DTA thermograms of (a) prepared polypyrrole (b) PPy-SDS-NaB 1 composite (c) PPy-SDS-NaB 2 composite, and (d) PPy-SDS-NaB 3 composite

**Table 4 Total Weight Loss Percent of Pure Bentonite and Polypyrrole-Bentonite Composites from TG-DTA Analyses**

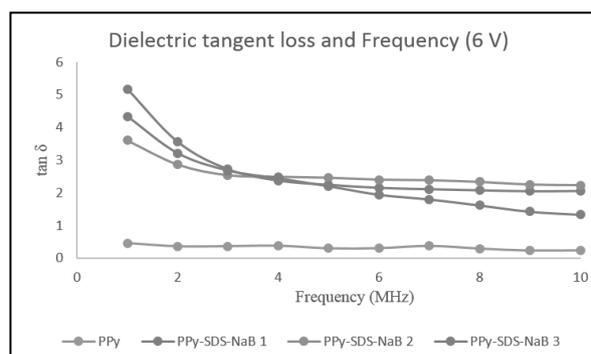
| Sample            | Temperature range (°C) | Peak's temperature (°C) | Nature of Weight Loss Peak | Weight Loss (%) | Total Weight Loss (%) |
|-------------------|------------------------|-------------------------|----------------------------|-----------------|-----------------------|
| (a) PPy           | 37 – 180               | 40                      | Exo                        | 10.08           | 85.59                 |
|                   | 380 -600               | 272 and 589             | Exo                        | 75.51           |                       |
| (b) PPy-SDS-NaB 1 | 38 – 100               | 70                      | Endo                       | 4.44            | 25.67                 |
|                   | 100 -375               | 282                     | Exo                        | 6.66            |                       |
| (c) PPy-SDS-NaB 2 | 375 - 600              | 460                     | Exo                        | 14.57           | 20.72                 |
|                   | 38 – 100               | 62                      | Endo                       | 5.07            |                       |
| (d) PPy-SDS-NaB 3 | 100 -375               | 255                     | Exo                        | 6.24            | 18.33                 |
|                   | 375 - 600              | 513                     | Exo                        | 9.41            |                       |
| (d) PPy-SDS-NaB 3 | 38 – 100               | 70                      | Endo                       | 7.49            | 18.33                 |
|                   | 100 -375               | 257                     | Exo                        | 4.99            |                       |
| (d) PPy-SDS-NaB 3 | 375 - 600              | 517                     | Exo                        | 5.85            | 18.33                 |

### Dielectric Properties

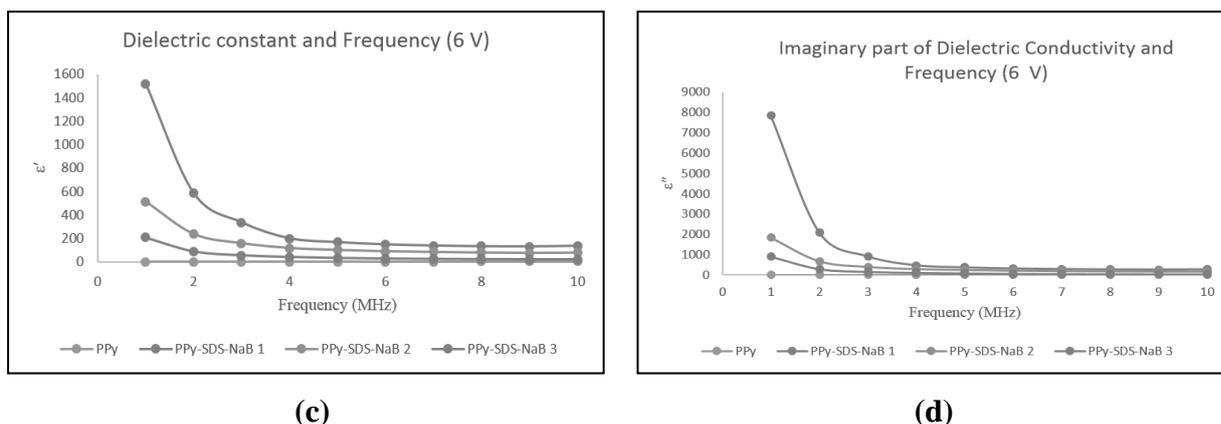
The frequency dependent dielectric permittivities such as  $\epsilon'$ ,  $\epsilon''$  and tangent loss ( $\tan \delta$ ) and dielectric conductivity ( $\sigma_{ac}$ ) of prepared PPy and PPy-SDS-NaB composites are presented in Figures 6 (a-d). As shown in Figures 6 (a, b) the dielectric permittivities decreases as the amount of bentonite in the composites increases. It is also seen that the relative permittivity decreases with increasing frequency. Both  $\epsilon'$  and  $\epsilon''$  exhibit typical dielectric behavior especially at high frequencies. This can be interpreted that the maximum interaction occurs at low frequency. The dielectric tangent loss ( $\tan \delta$ ), which is related to the imaginary part of dielectric conductivity ( $\epsilon''$ ), decreases with increasing frequencies. PPy-SDS-NaB 1 has a higher dielectric conductivity than PPy-SDS-NaB 2 and PPy-SDS-NaB 3. It may be due to the effect of both surfactant and sodium treated bentonite. According to Figure 6 (a), dielectric conductivity for PPy-SDS-NaB 3 can be negligible. It may be interpreted that as the amount of sodium treated bentonite increases, dielectric conductivity cannot be affected. The change in dielectric permittivity with respect to bentonite clay percentage and high dielectric permittivity values can be attributed to the absorbed moisture in interlayer of bentonite clay.



(a)



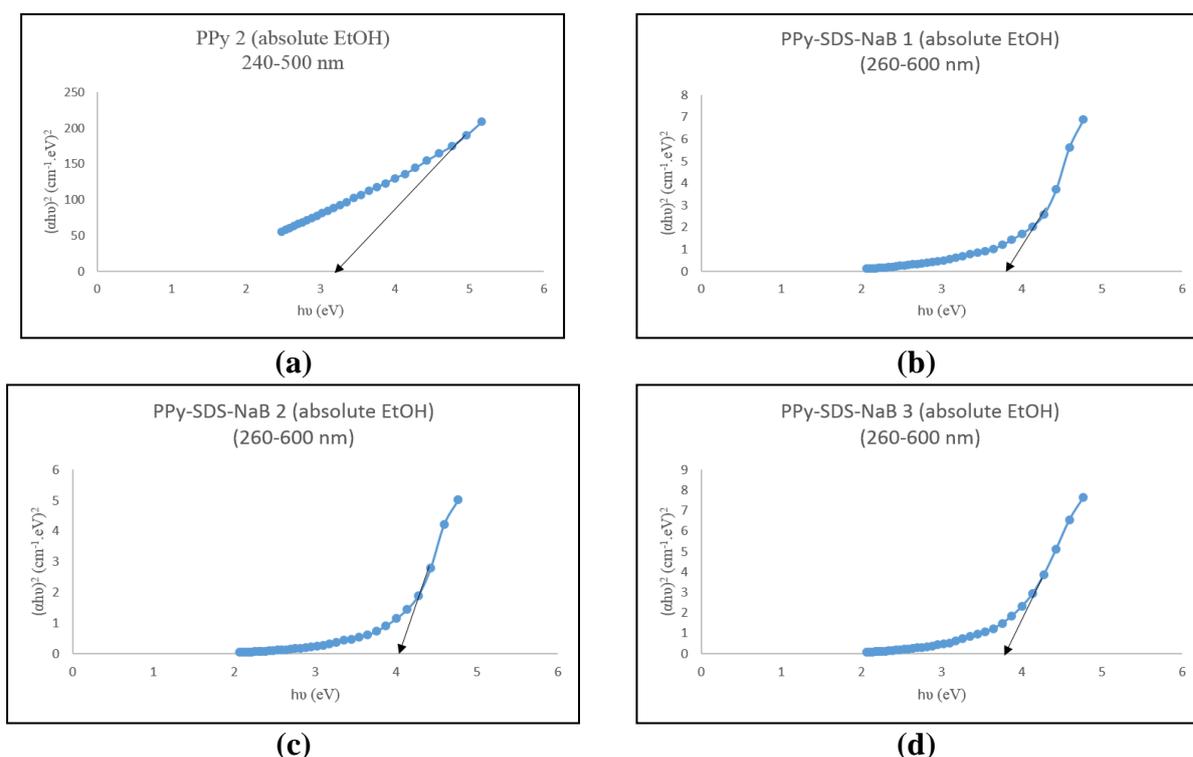
(b)



**Figure 6** Plots of relationship between frequency and (a) dielectric conductivity (b) dielectric tangent loss (c) dielectric constant and (d) imaginary part of dielectric conductivity of PPy and PPy-SDS-NaB composites at 6 V potential

**Optical Properties**

The optical properties of prepared PPy and PPy-SDS-NaB composites were studied by UV-vis spectrophotometer. The absorption coefficient ( $\alpha$ ) was calculated from the absorption spectra. The optical band gap values were evaluated using Taucs' realation. The electronic transition is presented in Figure 7. In general, the band gap values depend on the crystal structure of the composites and the arrangement and distribution way of atoms in the crystal lattice. From the curves shown in Figure 7, it was found to be the band gap values of 3.2 eV, 3.8 eV, 4 eV and 3.8 eV for PPy, PPy-SDS-NaB 1, PPy-SDS-NaB 2 and PPy-SDS-NaB 3 composites, respectively.



**Figure 7** Tauc's plot of  $(\alpha h\nu)^2$  against  $(h\nu)$  for (a) polypyrrole (PPy) (b) PPy-SDS-NaB 1 composite (c) PPy-SDS-NaB 2 composite (d) PPy-SDS-NaB 3 composite

## Conclusion

In this study, polypyrrole and PPy-SDS-NaB composites were performed by chemical oxidative polymerization in aqueous medium. The polymerization mechanism and chemical structures of composite materials are investigated by means of XRD and FT IR. XRD results confirmed the insertion of PPy between the interlayers of bentonite clays. The morphological study showed that polypyrrole polymerization occurred within bentonite interlayers. It is shown that the denser and more compact morphology of PPy-B composites with the increase in amount of bentonite clay. Intercalated composites are found to be more thermally stable than pure polypyrrole. The overall thermal stability trends are PPy-SDS-NaB 3 > PPy-SDS-NaB 2 > PPy-SDS-NaB 1 > PPy in the TG-DTA thermograms. FT IR analysis shows the incorporation of PPy in the clay structure. The prepared PPy-SDS-NaB 1 can be used as a promising material for the variety of electrical applications within these frequency range of 1-10 MHz at 6 V potential because it exhibits improved thermal properties, reduced flammability and better dielectric barrier properties than unfilled polymer. Therefore, cheap filler-like sodium treated bentonite can be effectively used as a good reinforcement for PPy matrix. From the analysis of absorption spectra, it was found that the optical band gaps of prepared composites with the various amounts of sodium bentonite are in the semiconductor wide band gap ranges of 4.0 eV to 3.8 eV. Thus it shows great promise in sensors as well as electrical applications.

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