

Research Article

Synthesis and Characterization of Conductive Polypyrrole: The Influence of the Oxidants and Monomer on the Electrical, Thermal, and Morphological Properties

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Conductive polymer, polypyrrole (PPy), was synthesized by chemical oxidative polymerization technique for a period of four hours at room temperature using pyrrole monomer (mPPy) in aqueous solution. Different oxidants such as ferric chloride (FeCl_3) and ammonium persulphate ($\text{N}_2\text{H}_8\text{S}_2\text{O}_8$) and surfactant sodium dodecyl sulphate ($\text{C}_{12}\text{H}_{25}\text{NaO}_4\text{S}$) were used. The produced PPy samples were characterized by using different techniques such as the electrical resistivity by four probe technique, thermogravimetry analysis (TGA), differential scanning calorimetry (DSC), Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), and scanning electron microscopy (SEM). The performance of the oxidants has been investigated and compared. It was found that both oxidants, FeCl_3 and $\text{N}_2\text{H}_8\text{S}_2\text{O}_8$, have decreased electrical resistivity as a function of temperature, which means increased conductivity. However, FeCl_3 has achieved better performance than $\text{N}_2\text{H}_8\text{S}_2\text{O}_8$, where it has achieved a lower resistivity of about 60 ohms at room temperature, which indicates higher conductivity of PPy samples with FeCl_3 as an oxidant. Similarly, further investigation of FeCl_3 oxidant has been conducted by varying its concentration, and its influence on the final properties was reported. It has been observed that the morphology of PPy samples has a significant influence on the conductivity. It was found that 0.1 M and 0.05 M concentrations of FeCl_3 oxidant and monomer, respectively, have achieved better thermal stability, which is $\text{FeCl}_3/\text{mPPy}$ ratio of 2 as an optimum value. FTIR and XRD results confirmed the structural formation of polypyrrole from pyrrole monomer during the synthesizing process.

1. Introduction

Recently conductive polymers, particularly polypyrrole (PPy), are gaining momentum for many commercial applications due to their excellent thermal stability, good electrical conductivity, relative ease of synthesis, and environmental stability [1–3]. Generally, conductive polymers are conductive in nature due to the existence of a conjugated electron or alternating single bond and double bond system in its chemical structure, because of this unique chemical property, the attention of many researchers and scientists from different research disciplines both in academics and industries around the world has been attracted [4, 5]. PPy has been actively used in many potential applications such as

electronic devices, sensors, batteries, microactuators, anti-electrostatic coatings, and biomedical [6–10], and it has been synthesized and prepared by using different techniques such as electrochemical [11, 12] or chemical oxidation of pyrrole monomer [13, 14] in various organic solvents and in aqueous media.

Due to the wide range of applications for the chemically prepared PPy, properties such as processability, thermal stability, and conductivity are the main properties that require further improvements for future advanced applications. Therefore, in a chemical oxidative polymerization of pyrrole monomer, many researchers have reported using different oxidants for synthesizing PPy. For instance, FeCl_3 has been used as an oxidant for synthesizing PPy films in order to

TABLE 1: All materials used and their concentrations.

Name	Designated	Concentration (M)
Pyrrole monomer	mPPy	0.025, 0.05, 0.1, 0.15
Ferric chloride	FeCl ₃	0.025, 0.05, 0.1, 0.15
Ammonium persulphate	(NH ₄) ₂ S ₂ O ₈	0.1
Sodium dodecyl sulphate	C ₁₂ H ₂₅ NaO ₄ S	0.0225

study the detection of gases such as ammonia [15, 16], and the use of ammonium persulphate as an oxidant focuses mainly on conductivity behavior. While Khamlich et al. [17] have used ferric chloride as an oxidant for preparing polypyrrole/graphene nanocomposites, investigating the influence of nanofiller on the final properties. Similarly, Hassanzadeh et al. [18] have utilized FeCl₃ as an oxidant for chemical synthesis of polypyrrole nanowire arrays for gas sensing applications. Also, many researchers reported PPy composites, for instance, Barrau et al. [19] have done work on carbon nanotube/epoxy resin with PPy, while Achour et al. [20] and Sangawar and Moharil [21] have reported using polymethyl methacrylate (PMMA) nanocomposite with PPy and polyvinylchloride (PVC) and PMMA-filled PPy composites, respectively. However, the use of different oxidants and pyrrole monomer for pure PPy and tailoring their performance on the final properties such as electrical, thermal, and morphological have not been fully exploited, and there are a few reports available in literature which use sodium dodecyl sulphate as a dopant and comparing the performances of ferric chloride and ammonium persulphate as an oxidizing agent in terms of thermal stability, conductivity, and morphology. Therefore, in this work, the influence of different oxidants and its concentrations on the thermal stability, conductivity, and morphology are investigated.

2. Experimental

2.1. Materials. Pyrrole monomer (mPPy) 98% was supplied by Sigma Aldrich. Anhydrous iron (III) chloride, also called ferric chloride (FeCl₃) 98%, and ammonium persulphate (N₂H₈S₂O₈) 98% were used as the oxidizing agent, and sodium dodecyl sulphate (C₁₂H₂₅NaO₄S), anionic surfactant, was used as the dopant/surfactant. All of them were supplied by Sigma Aldrich as well. All materials were used as received.

2.2. Synthesizing and Sample Preparation. Polypyrrole (PPy) was synthesized by chemical oxidative polymerization technique using pyrrole monomer and different oxidants. The chemical polymerization was carried out in a beaker with 100 ml of distilled water by mixing different molar ratios of pyrrole monomer, an oxidant, and a surfactant. Different concentrations of pyrrole monomer and oxidants were prepared, and as shown in Table 1, the polymerization solution was composed of various amounts of the pyrrole and oxidants and a fixed amount for the surfactant. Since this is an exothermic reaction, the addition of distilled water (cooled) was done slowly. A given volume of pyrrole monomer was quickly added to the distilled water with the required amount of oxidant and surfactant; vigorous magnetic stirring was

maintained to facilitate the dispersion of pyrrole. The polymerization reaction was carried out for a period of 4 hrs at a temperature of 25°C. The precipitation of fine black particles was nearly immediate. After the prescribed polymerization time, synthesized PPy was filtered from the solution with filter paper and thoroughly washed with distilled water and ethanol several times; PPy was dried in a vacuum oven at about 40°C overnight.

2.3. Electrical Property Evaluation. The resistivity of PPy samples was measured using a four-point probe method, Model 6220 DC, manufactured by Keithley Instruments Inc. The four-point probe method is the most common technique to measure a semiconductor material's resistivity due to its low demand on sample preparation and high accuracy. It consists of a probe station (four probe tips), an ampere meter, a DC current source, and a voltmeter. Two of the probes are used to source current, and the other two probes are used to measure voltage, which means as current flows between the outer probes, the voltage drop across the inner probe is measured. For the measurement procedure, a sample film with a thickness of about 0.4 cm was prepared from the PPy sample powder by using hydraulic press and applying a pressure of 70 MPa and finally dried and cleaned for testing. The ampere meter, DC current source, and the voltmeter were checked to ensure they were on. The sample was placed on the disk and was aligned so that the spot to be measured was under the probe head. After waiting for a few seconds for the voltage to stabilize, the measurements were taken. When sample testing was completed, old samples were removed and a new sample was inserted for testing. Three measurements were done from each batch of samples.

2.4. Fourier Transform Infrared (FTIR) Spectroscopy Analysis. Fourier transform infrared (FTIR) spectra of the samples were obtained with FTIR spectrometer in the wave number range 400 to 4000 cm⁻¹, and the IR spectra of powder were recorded in the KBr medium at room temperature at a 1 cm⁻¹ resolution averaging 15 scans. FTIR investigations were carried out to confirm the polymerization of the monomer during the synthesis process and existing functional groups in the produced polypyrrole conductive polymer.

2.5. Thermal Property Measurement. In order to analyze the thermal stability of the produced PPy samples, TGA was performed by using a DTG-60 instrument, manufactured by Shimadzu in Japan. A sample weight of around 10 mg was heated from 25 to 800°C at a heating rate of 10°C min⁻¹ under nitrogen atmosphere. The weight loss of the samples was estimated in accordance with ASTM D 3850-2000. DSC measurements were carried out on DSC-50, manufactured by Shimadzu in Japan. The starting temperature was 25°C and was raised at 10°C min⁻¹ to 300°C, then cooled down to room temperature.

2.6. Morphological Measurement. X-ray diffraction (XRD) studies were carried out by using a PAN Analytical, Rontgen Diffractometer System, Model PW3040/60, Netherland, with Cu-K α (1.5418 Å) radiation operating at 30 kV and 20 mA. The XRD patterns were recorded in the 2 θ range 10 to 80°

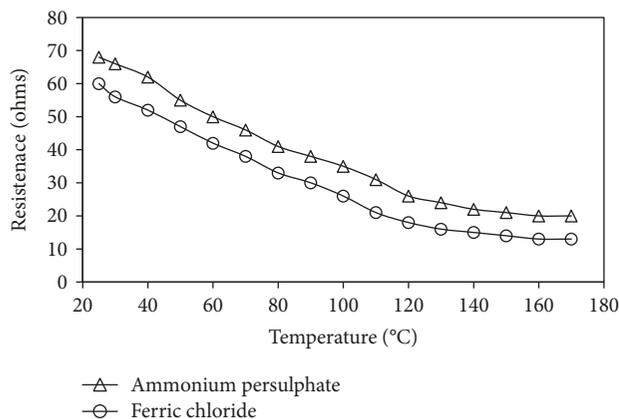


FIGURE 1: Resistance versus temperature for different oxidants (pyrrole 0.05 M and 0.1 M oxidants at 25°C, duration 4 hrs, and surfactant 0.025 M).

with step width 0.02° and step time 1.25 sec. The morphology of the polypyrrole sample was examined using scanning electron microscope (SEM), A TESCAN TS5135MM model.

3. Results and Discussion

3.1. Electrical Property Evaluation. Figure 1 shows the variation of electrical resistivity as a function of temperature of pure PPy samples with different oxidizing agents (ferric chloride and ammonium persulphate). It is observed that as temperature increases, the electrical resistivity decreases for both oxidants, which means increasing conductivity. It is generally expected that increasing temperature will cause higher energy for an electron in the valence band, which jumps to conduction band; hence, conductivity should increase with temperature. It is also possible that thermal curling affects PPy chain alignment, which could cause increasing conjugation length and decreases resistivity of the sample. However, it is observed that samples containing oxidizing agent, ferric chloride (FeCl_3), have achieved slightly lower resistance and higher conductivity compared to the other oxidizing agent ammonium persulphate ($\text{N}_2\text{H}_8\text{S}_2\text{O}_8$). Similar observation of increased conductivity by using ferric chloride was reported by Chitte et al. [15]. This phenomenon could be attributed due to structural difference between the two oxidizing agents and their interaction with pyrrole monomer during the chemical synthesis and their morphologies. The morphology of the samples has a significant influence on the conductivity. The highest conductivity was obtained for sample containing ferric chloride as an oxidizing agent as shown in Figure 2 with fibrillar morphology structure compared to the spherical morphology for sample containing ammonium persulphate as shown in Figure 3 (see Morphology Analysis). Similar observation was reported by Khadem et al. [22], where conductivity of fibrillar sample was higher than that of spherical samples. This behavior could be attributed to the better electron transfer within the polymer chain for fibrillar structure compared to the spherical-shaped morphology [22].

Other possible reason why ammonium persulphate has lower conductivity could be related to the addition of anionic surfactant and its more interaction with it, which as a result causes decrease in conductivity. Similar observation was reported by Kudoh [23], who found that the decrease in conductivity is due to the presence of surfactant anion adsorbed on the PPy surface. In order to observe the individual effects of the FeCl_3 and pyrrole monomer on the resistivity of the PPy samples, the concentration of the reactants was varied one at a time to evaluate their performance. As shown in Figure 4, with increasing FeCl_3 concentration, there are continuous decreases in resistivity occurring till 0.1 M. This could be attributed to the formation of carboxyl defects due to overoxidation by FeCl_3 . Generally, carbonyl defects could cause interruptions in conjugation and as a result act as impediments to charge transfer which eventually reduces conductivity; this is could be due to the overoxidation. Similar observation of overoxidation causing reduction in the conductivity has been reported in [24].

In the case of pyrrole monomer as shown in Figure 5, resistivity decreases with increase in concentration up to 0.05 M, beyond which resistivity increased with increasing pyrrole monomer concentrations. Hence, lower resistivity and higher conductivity were achieved at 0.05 M monomer concentration.

3.2. FTIR Analysis. FTIR investigations were carried out to confirm the polymerization of the monomer to polymer during the synthesis process. Figures 6(a) and 6(b) show the FTIR spectra of synthesized PPy samples from different oxidants such as ferric chloride and ammonium persulphate, respectively. The FTIR spectra of the PPy samples were recorded in the range of 4000 to 500 cm^{-1} to confirm polymerization. The occurrence of a wide peak at 3400 cm^{-1} is assigned to the presence of N-H stretching vibrations of pyrrole ring. The weak band at 2100 cm^{-1} is due to C-H stretching. The characteristic peak at 1600 cm^{-1} corresponds to the C=C stretching, whereas peaks at 1400 cm^{-1} and 1200 cm^{-1} represent C-N and C-H plane deformation bonds, respectively, and the peak at 1100 cm^{-1} is due to C-C stretching. The peaks observed in this study match well with those available in the literature [22, 25, 26] confirming the formation of polypyrrole polymer, as shown in Figure 6. The FTIR results have clearly shown the formation of PPy polymer and its composition structure details.

3.3. Thermal Stability Results

3.3.1. Differential Scanning Calorimetry (DSC) Results. Figure 7 shows DSC results for synthesized PPy for different oxidants, ferric chloride (FeCl_3) and ammonium persulphate ($(\text{NH}_4)_2\text{S}_2\text{O}_8$), while Figure 8 shows DSC results for different concentrations of ferric chloride oxidant. Table 2 presents the glass transition temperature (T_g) and melting temperature (T_m) obtained from the DSC analysis for polypyrrole samples of different oxidants used.

Figure 7 shows the comparison of the two oxidants, ferric chloride and ammonium persulphate. As shown in Figure 7, the PPy sample with ferric chloride as an oxidant had a

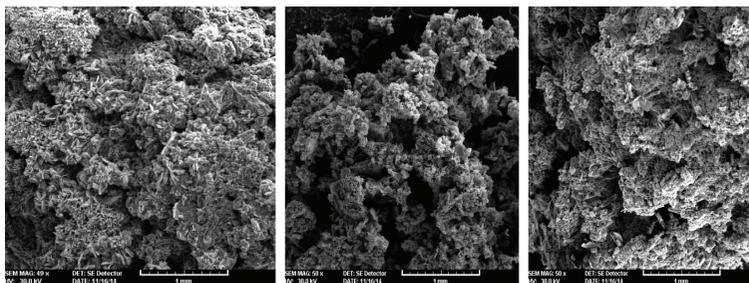


FIGURE 2: Microstructure of PPy samples where ferric chloride was used as an oxidizing agent (oxidant 0.1 M, pyrrole 0.05 M, and surfactant 0.025 M at 25°C and duration of 4 h).

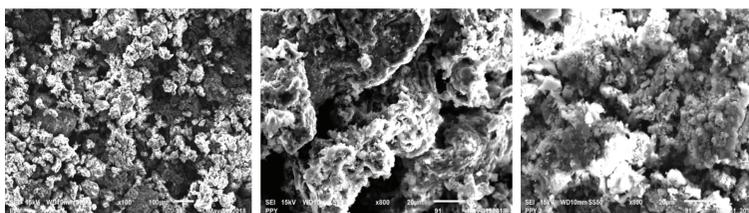


FIGURE 3: Microstructure of PPy samples where ammonium persulphate was used as an oxidizing agent (oxidant 0.1 M, pyrrole 0.05 M, and surfactant 0.025 M at 25°C and duration of 4 h).

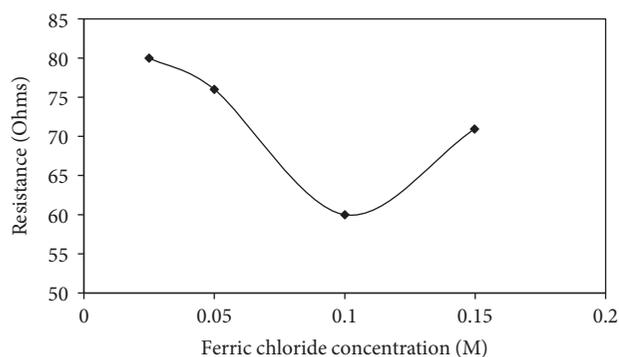


FIGURE 4: Resistance versus different ferric chloride concentrations (pyrrole 0.05 M at 25°C, duration 4 hrs, and surfactant 0.025 M).

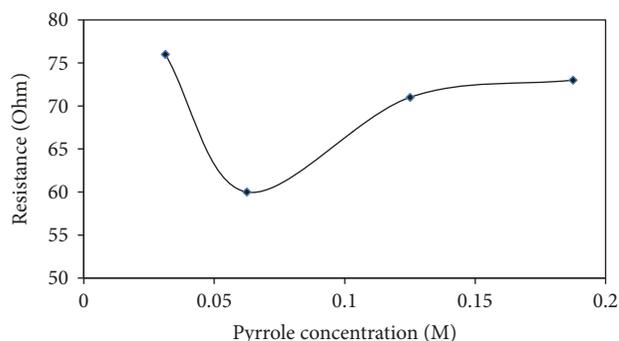


FIGURE 5: Resistance versus different pyrrole concentrations (ferric chloride 0.1 M at 25°C, duration 4 hrs, and surfactant 0.025 M).

higher melting point than the PPy sample with ammonium persulphate as an oxidant and also slightly had a higher glass transition temperature for FeCl_3 samples. This could mean ferric chloride as an oxidant has achieved a better and higher

thermal stability during synthesis compared to the other oxidant, ammonium persulphate. This could be attributed to the difference in the nature of chemical structure between the two oxidants. It is also believed that the possible reason for this occurrence was due to the low crystallization rate of PPy and its nature of being amorphous material. The DSC results for both oxidants are tabulated in Table 2.

Ferric chloride as an oxidant had a better performance than the other oxidant, ammonium persulphate. Hence, for further investigation, the influence of different concentrations of ferric chloride on the PPy samples in terms of resistivity and thermal stability was studied.

Figure 8 shows the influence of different ferric chloride concentrations on the melting and glass transition temperatures. As shown in Figure 8, the concentration of ferric chloride at 0.1 M achieved slightly a higher melting point of the PPy samples, while the lower concentration had a lower melting and lower glass transition temperatures. It is also important to note that increasing the concentration above 0.1 M had no significant influence on increasing the melting point temperature. All results are tabulated in Table 3.

3.3.2. Thermogravimetric Analysis (TGA). Figure 9 shows the TGA curves for PPy samples with different oxidants (ferric chloride and ammonium persulphate), while Figure 10 shows PPy samples with different concentrations of ferric chloride. The onset temperatures ($T_{20\%}$) of 20% weight loss and ($T_{60\%}$) of 60% weight loss deviation from the baseline T_{20} and T_{60} were used as the indicator of the compound's thermal stability and referred to as $T_{20\%}$ and $T_{60\%}$, respectively. During thermal degradation, the TGA curves display different degradation steps or processes for all of the samples. As shown in Figure 9, the first significant weight loss takes place approximately at temperature between 25 and 100°C due to water evaporation, while the second one starts at 100 and

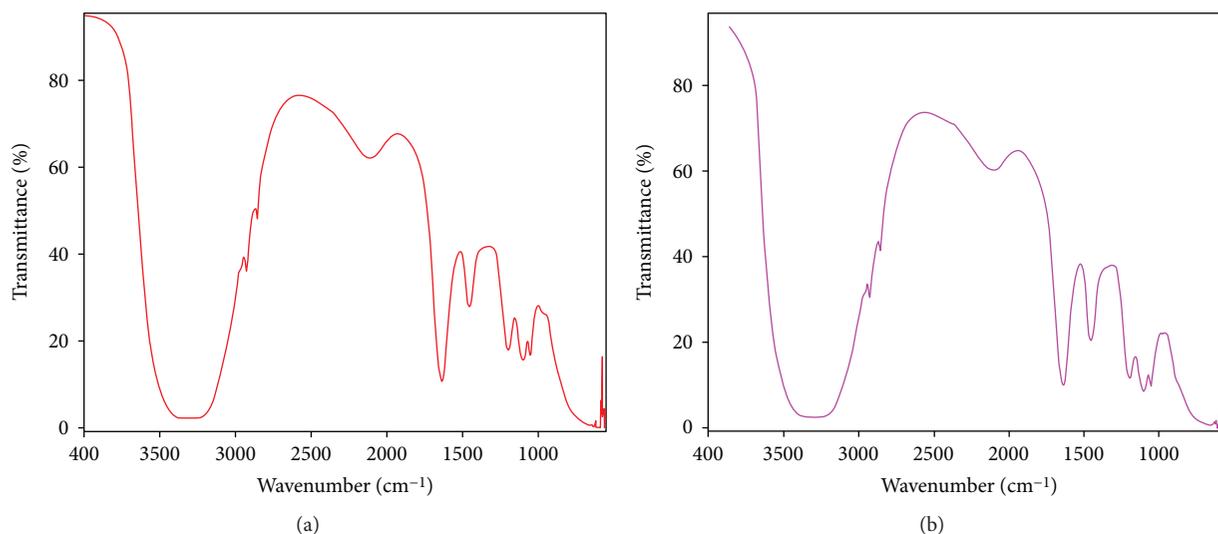


FIGURE 6: The FTIR spectrum of PPy samples with (a) ferric chloride and (b) ammonium persulphate.

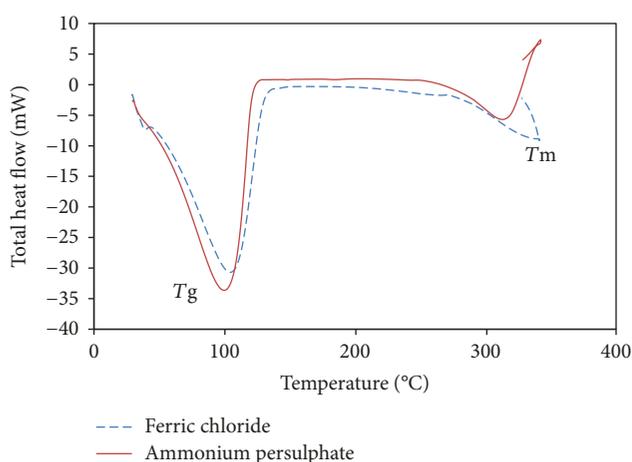


FIGURE 7: DSC results of PPy samples with different oxidants. Pyrrole 0.05 M and 0.1 M oxidants at 25°C, duration 4 hrs, and surfactant 0.025 M.

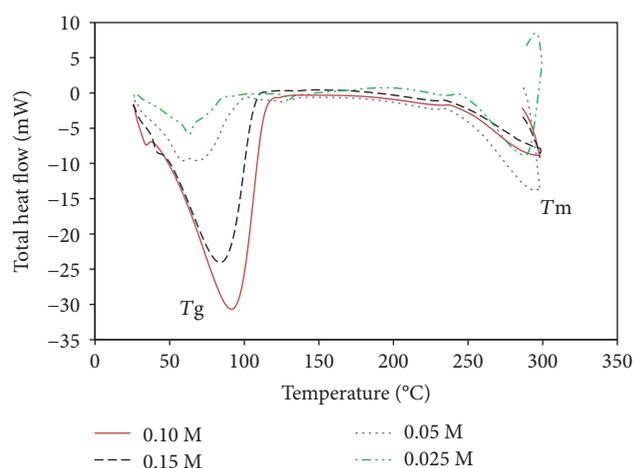


FIGURE 8: DSC results of PPy samples with different ferric chloride concentrations (pyrrole 0.05 M and surfactant 0.025 M at 25°C and duration 4 h).

ends at 180°C for possible impurities in the samples. The main and really mass loss for ferric chloride corresponds to polymer degradation which starts at about 227°C. However, for ammonium persulphate, the main degradation starts at 187°C, which means it decomposes at lower temperature compared to other sample. This could be attributed to chemical structure difference and interaction of monomer and surfactants during synthesis.

Therefore, as shown in Figure 9, PPy samples with ferric chloride oxidant show overall higher thermal stabilities compared to PPy samples with ammonium persulphate oxidant. The data for the variation in these temperatures are summarized in Table 4. As shown in Table 4, the $T_{20\%}$ of PPy sample with ferric chloride is 204°C, where the $T_{20\%}$ of PPy samples with ammonium persulphate is about 83°C. A similar trend was observed for the $T_{60\%}$ of PPy samples, which indicates higher thermal stability for PPy

TABLE 2: DSC glass transition and melting temperature results for two oxidants.

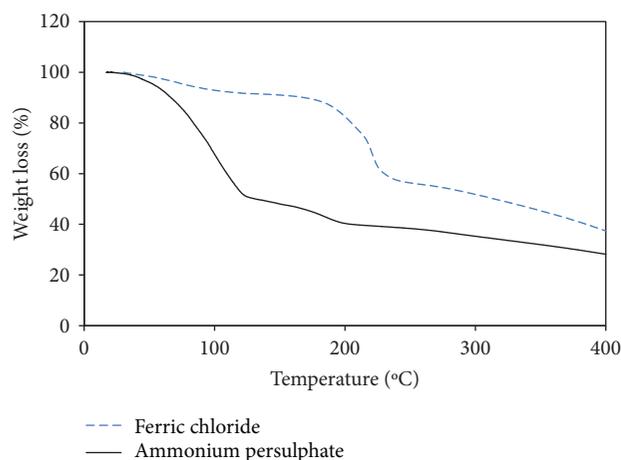
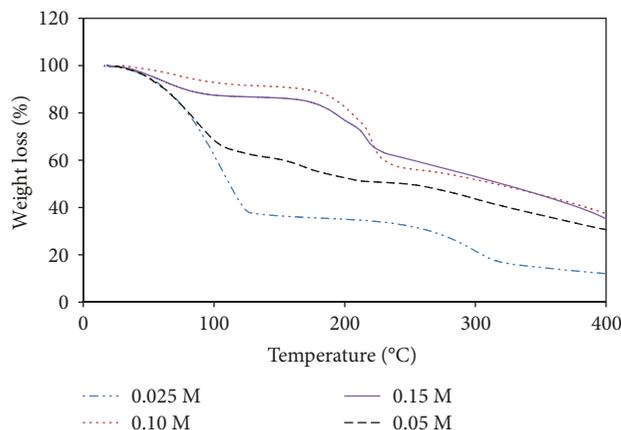
Oxidant	Glass transition temperature (T_g) (°C)	Melting point (T_m) (°C)
FeCl ₃	93	298
(NH ₄) ₂ S ₂ O ₈	87	270

samples with ferric chloride, which is 385°C compared to 200°C for ammonium persulphate.

Figure 10 shows the influence of different ferric chloride concentrations on the sample's degradation and thermal stability. As shown in Figure 10, the ferric chloride concentration at 0.1 M has achieved a better thermal stability for both samples at $T_{20\%}$ and $T_{60\%}$ weight losses compared to

TABLE 3: DSC results for different FeCl_3 concentrations.

FeCl_3 concentration (M)	Glass transition temperature (T_g) ($^{\circ}\text{C}$)	Melting point (T_m) ($^{\circ}\text{C}$)
0.15	85	295
0.1	93	298
0.05	67	289
0.025	63	287

FIGURE 9: TGA results for PPy samples with different oxidants (pyrrole 0.05 M, oxidant 0.1 M, and surfactant 0.025 M, at 25°C duration 4 h).FIGURE 10: TGA results with different ferric chloride concentrations (pyrrole 0.05 M and surfactant 0.025 M at 25°C and duration of 4 h).

other concentrations, particularly lower concentrations such as 0.05 M and 0.025 M. However, at a higher concentration of 0.15 M, no significant change of the thermal stability was observed. This indicates that 0.1 M concentration of ferric chloride could be the optimum value of achieving good thermal stability of the produced PPy samples.

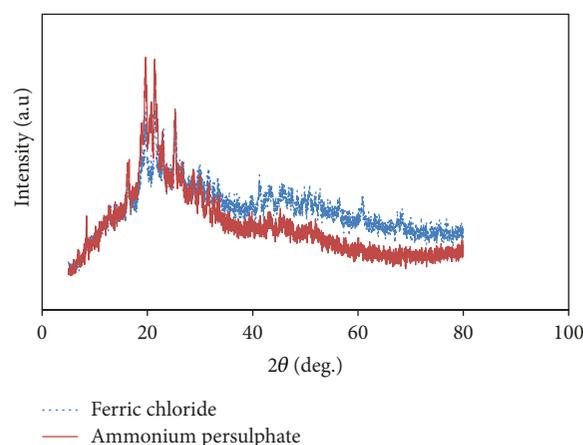
Table 5 shows $T_{20\%}$ and $T_{60\%}$ for different ferric chloride concentrations. From the TGA data, the thermal degradation

TABLE 4: TGA results for PPy samples with FeCl_3 and $(\text{NH}_4)_2\text{S}_2\text{O}_8$ as oxidants.

Oxidant	$T_{20\%}$ ($^{\circ}\text{C}$)	$T_{60\%}$ ($^{\circ}\text{C}$)
FeCl_3	202	384
$(\text{NH}_4)_2\text{S}_2\text{O}_8$	83	206

TABLE 5: TGA results for PPy samples with different concentrations of FeCl_3 .

FeCl_3 concentration (M)	$T_{20\%}$ ($^{\circ}\text{C}$)	$T_{60\%}$ ($^{\circ}\text{C}$)
0.15	192	381
0.1	202	384
0.05	80	325
0.025	79	122

FIGURE 11: X-ray diffraction of PPy samples (oxidant 0.1 M, pyrrole 0.05 M, and surfactant 0.025 M at 25°C and duration of 4 h).

temperature increases with increase in concentration of ferric chloride oxidant. For instance, at a concentration of 0.1 M, the highest thermal stability was achieved; however, a slight decrease of thermal stability beyond that concentration was observed. At lower concentrations, such as 0.05 M and 0.025 M, thermal stability decreased drastically, particularly at 0.025 M.

3.4. Morphological Results

3.4.1. X-Ray Diffraction (XRD) Analysis. Figure 11 shows the X-ray diffraction studies for PPy samples with different oxidants. As shown in Figure 11, the prominent observed peaks and the diffraction in PPy samples with ferric chloride and ammonium persulphate were registered at 2θ , equals 21° and 20° , respectively.

The broad peaks in the region $2\theta = 20^{\circ}$ - 21° indicate the amorphous nature of the polymer and intermolecular stacking structure. This is in agreement with the previously reported in the literature [15, 22].

3.4.2. Morphology Analysis. The microstructure of polypyrrole samples is shown in Figures 2 and 3 for ferric chloride and ammonium persulphate as oxidizing agents, respectively. The sample has a uniform granular morphology. The amorphous and partially crystalline nature seen in the SEM pictures is reflected in the XRD spectra previously shown.

It was observed that the PPy prepared using a PPy/ferric chloride showed fibrillar morphology in the micrograph, as shown in Figure 2. Similarly, as shown in Figure 3, PPy samples with ammonium persulphate as an oxidant show spherical morphology. Thus, the morphological change of the PPy was strongly dependent on the type of oxidant used during the chemical polymerization process. The morphological structure of the PPy samples has significant influence on the final properties particularly conductivity as shown previously in the section of electrical properties of the samples. It was found that the sample with fibrillar morphology has achieved higher conductivity compared to the sample with spherical morphology.

4. Conclusions

PPy samples were synthesized using different oxidants, and their performance in terms of electrical and thermal properties has been evaluated and compared. It was found that using ferric chloride (FeCl_3) as an oxidant had a significant influence on electrical and thermal properties of the produced PPy samples compared to the ammonium sulphate ($\text{N}_2\text{H}_8\text{S}_2\text{O}_8$). Similarly, the effects of FeCl_3 and pyrrole monomer concentrations on the electrical and thermal stability were examined, and results showed that increasing concentration of FeCl_3 up to 0.1 M decreases sample resistivity; similarly, increasing pyrrole monomer concentration decreases resistivity, however, beyond 0.05 M pyrrole concentration resistivity increases, which means at 0.05 M, pyrrole concentration could be optimum concentration to achieve lower resistivity. Hence, higher conductivity of the PPy samples was achieved at concentrations of 0.1 M and 0.05 M for ferric chloride and pyrrole monomer, respectively. It was found that morphological property of the sample plays a significant role in achieving higher conductivity. In terms of thermal property analysis, DSC results have shown that ferric chloride as an oxidant had achieved higher melting point than the PPy sample with ammonium persulphate as an oxidant; also, slightly higher glass transition temperature for samples with ferric chloride was observed. TGA observations showed that the PPy samples with ferric chloride oxidant have higher thermal stability than the samples with ammonium sulphate. It is also shown that TGA result concentration of ferric chloride at 0.1 M had achieved significant thermal stability. Overall result analysis of thermal property indicates that PPy samples with ferric chloride as an oxidant exhibited high thermal stability. FTIR, XRD, and SEM results have shown the formation of polypyrrole polymer during synthesis. All these results indicate the influence of different oxidants which could achieve different performance; therefore, it is possible to engineer the PPy properties according to the requirement and needs for particular applications.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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