Synthesis and Characterizations of Polyaniline/Nylone(6) Composites, Electrical Properties and Thermal Stability

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Abstract

Practical applications that are related to the conducting polymers including polyaniline (PAn) as membrane have been limited due to the fact that their mechanical properties are low. Yet, to mix the PAn with certain traditional insulating polymers like nylon as well as preparation composite films via blending or solvent casting might be proper approach for increasing the mechanical properties which are related to conducting polymer filmsPAn:nylon-6 composite films have been prepared through a process of diffusion as well as aniline's oxidative polymerization within nylon 6 as well as nylon-6,6matrixes. Composite films have been subjected to characterization through SEM, FTIR, in addition to their electrical properties. Surface electrical conductivity that is related to HCl of (1M) doped composite films increase and increment in film's PAn contents. Studying electrical properties within certain isothermal conditions at temperature in range (50-200Celsius) indicated that composite films have been stable in ambient conditions not more than a temperature of 90 Celsius with regard to DC electrical conductivity retention.

Key Word: Polyaniline/Nylone(6), Composites, Thermal Stability, Polyaniline, Membrane.

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Introduction

PAn is significant electrically conducting polymer, due to the inherent combination of high conductivity in addition to the high environmental stability. The high processibility of Polyaniline has stimulated the attention of several investigations in order to develop the role and applications of this polymer. Polyaniline conducting polymers were specified as excellent choice with regard to the commercial applications such batteries, sensors, separation and electro-chromic devices [1-3].0ther advantages are their ease of synthesis in both aqueous and non-aqueous solutions, low cost of monomer, relatively high conductivity (1-20 S/cm) and environmental stability. Regarding conducting polymers, one can find out that many researchers have been devoted to polyaniline during the last quarter of century [4-6]. PAn might be subjected to synthesizing through aniline's oxidation in the solution phase in Bronsted acids electro-chemically or through chemical oxidants [8-11]. PAn will change to base form which is referred to as emeraldine base (EB) through treatment process with the use of dilute aqueous alkali solutions.

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EB is considered to be insoluble in the water, yet it is soluble in certain non-aqueous and aqueous mixed solvents like 1-methyl 2-pyrrolidinone (NMP), dimethylsulfoxide (DMSO), aqueous acetic acid (80%), concentrated sulphuric acid, formic acid (88%), as well as dimethylformamide (DMF).
This is going to allow it to be considered as solution which is processed for producingfree standing, flexible, as well as large films related to EB polymer that might be protonated (doped) with non-oxidizing protonic acidslike HC1 to films which have conductivity of ~10 S/cm [12-13]. PAn's electrical conductivity depends on pH of polymer or solution. It changes into an insulator material by simple base treatment or at pH above 4. It has been reported [14] that organic acid dopants produce more thermal (in air) and environmental (water) stability than small sized pants. This stability can be due to immobility of the larger size counter ions. Therefore, the large size dopants anions contrast to Cl^- do not migrate or removed when the polymer immersed in water. In generally doped polyanilines are less thermally stable than undoped form. EB (undoped, oxidized base form) of PAn is the most thermally stable form of polyanilne (up to 500°C) with regard to structural integrity [15].There is no difficulty in controlling aniline's chemical polymerization in insulating polymer matrix for preparing conducting composites with regard to the needed electrical properties in the same time as retaining mechanical strength that is related to the insulating polymer matrix [16–18]. The presented study indicated straightforward chemical route for preparingPAn:nylon-6,6 as well as nylon6 composite films. One of the major materials used as thermo-plastics or fibers is the Nylon.

Experimental Materials

The major applied chemicals to prepare composite films have been potassium persulphate (98%)(CDH, India), nylon6 (from Research, Design, and Standard Organization), aniline (99%)(CDH, India), as well as hydrochloric acid (35%)(E. Merck, India).

Instrumental

SEM micrographs were taken by Mira3 Tescan instrument. The FT-IR spectra regarding PAn: nylon 6 as well as nylon-6,6 composites have been recorded through BRUKER-TENSUR 27.UV-Vishas been conducted with the use of Uv-1800 (Shimadzu).The DC electrical conductivity that is related to doped composites films has been determined with the increase in temperature with the use of Keithly model 2400 technique. The DC electrical conductivity (σ) has been determined with the use of Equations (1) and (2):

$$\rho_{D.C} = R_e \frac{\omega\ t}{L} \quad (1)$$

Where: R_e: represent sample's (thin film) resistance, ω: represent the electrodes' width, L: is distance between two electrodes and $\rho_{D.C}$: is electrical resistivity of the films.

$$\sigma_{D.C} = \frac{1}{\rho_{D.C}} \quad (2)$$

Where: $\sigma_{D.C}$: is the conductivity of the thin film.
Arrangement has been put in over controlled electrically. Current pass via outer probes, also floating potential across probes' inner pair has been evaluated [13]. Oven's power supply has been switched on;the temperature subjected to gradual increase. Voltage and current have been simultaneously recorded with increase in temperature. Composite sample's thermal stability with regard to DC electrical conductivity retention has been examined in certain isothermal conditions with the use of DC electrical conductivity measuring instrument. The presented research has been conducted on at temperatures 50, 70, 90, 110 in addition to 130 Celsius on required selected composite films for PAn and at temperatures of 130, 140, 150, 160, 170, 180, 190, and 200 Celsius with regard to polyamide. Electrical conductivity measurement has been achieved in intervals of five minutes. The Digital micrometer related to the film's measuring thickness as well as accurate digital voltmeter (Keithly) to measure potential throughout conductivity measurementshave been used.

Preparation of PAn: Nylon-6 Composite Films

Several weight from Nylon 6 (0.2g and 0.3g) has been soaked in doubly distilled aniline (30ml) for various time periods in air oven at a temperature of 80 Celsius for allowing the aniline to be diffused in nylon-6, 6 matrix. The aniline soaked films have been subjected to treatment with potassium persulphate (3g) in (1M)of the HCl solution, and after that kept for a period of twenty-four hours at ice temperature for the purpose of polymerizing the aniline in nylon-6,6 matrix. Therefore, the prepared PAn:nylon-6 composite films have been subjected to wash with the use of doubly distilled

water which is up to the acid neutralizations, after that un-doped through treatment with the use of excess regarding aqueous ammonia (1M) succeeded via wash with the use of distilled water till filtrate become neutral. Composite films have been dried at a temperature of 40 Celsius for twenty-four hours in dry box, after that stored in desiccators with regard to experiment purposes. PAn:nylon-6,6 composite films have been doped with the treatment of (1M) HCl solution at room temperature for a period of twenty-four hours.

Table 1. Preparation details of polyaniline (nylon-6)

Sample ID	Temp [°C]	Time [min]	Weight of nylon-6[g]	Amount of aniline soaked [ml]
PANI (1)	80	15	0.2	30
PANI (2)	80	20	0.2	30
PANI (3)	80	25	0.2	30
PANI (4)	80	30	0.2	30
PANI (5)	80	35	0.3	30
PANI (6)	80	40	0.3	30
PANI (7)	80	45	0.3	30
PANI (8)	80	50	0.3	30

Results and Discussions

Prepare Conducting Polymer Composite Films

$$8NH_3 \longrightarrow 6NH_4^+ + 6e^- + N_2 \quad (1)$$

$$PTH^+–BF_4^- + NH_4^+ + e^- \longrightarrow PTH + NH_4BF_4 \quad (2)$$

$$6H_2O \longrightarrow 4H_3O^+ + 4e^- + O_2 \quad (3)$$

$$PTH^+–BF_4^- + H_3O^+ + e^- \longrightarrow PTH + HBF_4 + H_2O \quad (4)$$

$$HBF_4 \longrightarrow HF + BF_3 \quad (5)$$

Charge neutralization reaction is dependent on chemical reaction's rate between un-doping agent and the doped polymer, that is on the basis of polymer chain's reactivity as well as un-doping agent's basic strength [24]. There is extremely low basic strength related to the water, therefore, it is not acting as efficient effective undoping agent with regard to PAn, yet, analogous neutralization reaction related to the un-doping of PAn component of composites through ammonia solution might be indicated as can be seen in the equations below:

$$NH_3 + H_2O \longrightarrow NH_4OH \quad (6)$$

$$[(PANI–nH^+\ (Ny\text{-}6,6)][(nCl^-)] + nNH_4OH \longrightarrow PANI(Ny\text{-}6,6) + nNH_4Cl + nH_2O \quad (7)$$

FT-IR Studies

FT-IR spectra related to PAn:nylon-6,6 composites have been specified in the Figure. Band which correspond to the out plane bending vibration that is related to the C–H bond of p-disubstituted benzene rings appear at 824cm–1. Bands which correspond to the stretching vibration regarding N–B–N in addition to the N=Q=N structures appears at 1497cm–1 as well as 1587cm–1 respectively in which –B– in addition to =Q= represent benzenoid and the quinoid moieties in polymer. Bands which correspond to the vibration mode that is related to N=Q=N ring as well as the stretching mode related to C–N bond will appear at 1143 as well as 1302cm–1. Band which is close to 1130cm–1 has been specified as characteristic related to conducting polymer because of delocalization regarding the electrical charges resulted from deprotonation. FT-IR spectrum support the existence of benzenoid in addition to quinoid moieties in PAn. Characteristic bands at approximately 688, 1642, 3303 cm–1 due to nylon-6,6 exist in all composites. As-prepared PAn: nylon-6,6 composites indicated strong band of about 1650cm–1 that correspond to the carbonyl group regarding nylon-6,6. With regard to PAn, band correspond to the out regarding plane bending vibrations related to the C–H bond that is related to the p-disubstituted benzene ring appear about824cm–1. Bands correspond to the stretching vibrationsrelated to the N–B–N as well as N=Q=N structure appears about 1377cm–1 as well as 1500cm–1 respectively (in which –B– and =Q= represent the benznoid and quinoid moieties in polymer). Band correspond to the stretching mode that is related to C–N bond appear at around 1504cm–1 [25]. Slow increment in intensities regarding bands correspond to PAn and reduction in intensities of the bands correspond to nylon-6,6 support slow change in composition related to composite samples.

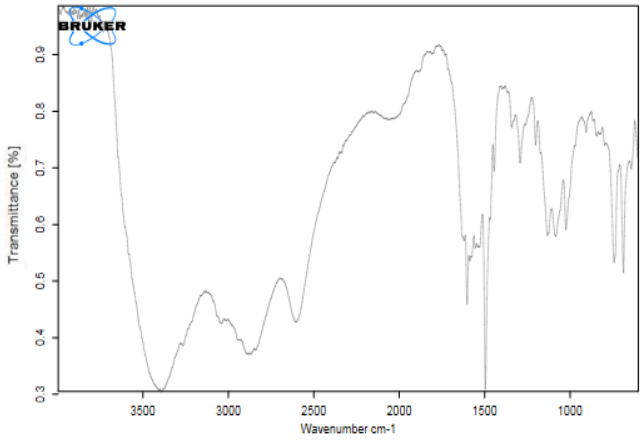

Figure 1. FT-IR spectra for PANI (Nylon 6).

SEM Studies

Figure indicate SEM photographs related to PAn nylon 6 composites at various magnifications. Obviously, homogeneous formation that is related to PAn's composites in nylon-6,6 matrix has been indicated. Surface morphology that is related to the PANI that is prepared chemically, indicated cloudy structure, whereas that related to nylon6 and nylon-6,6 is fairly granular, also composite film's morphology is very distinctive from the parent compounds, for example, PANI , nylon 6. Differences with regard to the composite's surface morphology are indicating that binding of parent component thus, composite's formation.

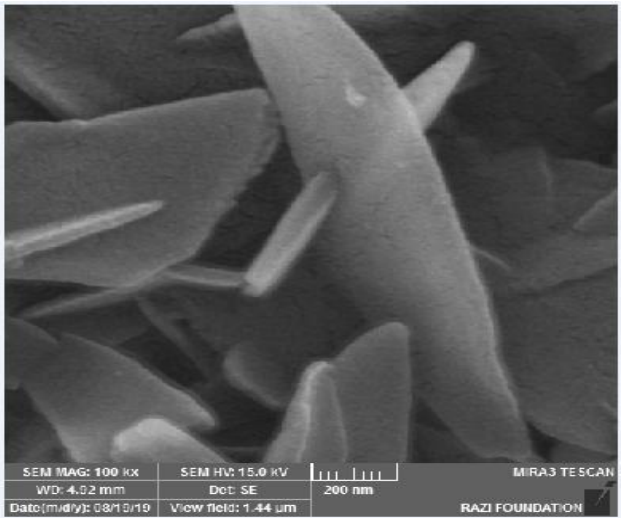

Figure 2. SEM for PAN (Nylon 6)

The Optical Characteristics

The optical absorption spectra are on the basis of film surface morphology, energy related to incident photon, crystal structure, chemical composition, as well as thickness of film. Figure (5) show absorbance spectra's variation with wave-length in range of (200-1200nm) of PANI as well as the PA-6 thin films. It might be indicated from the figure that film's absorbance has high values at wave-length in neighborhood related to fundamental absorption edge (287nm), after that absorbance will decrease with the increase in wave-length.

Figure (3) will show that film's absorbance has medium values in visible and near infrared region. Such behavior could be defined in the following way: with regard to large wave-length, incident photons do not have proper energy for interacting with the atoms, photon is going to be transmitted. In the case of decreasing the wave-length, interaction between material and the incident photon is going to happen, after that there will be an increase in absorbance [26]. Put differently, incident light will be absorbed through free electrons.

Furthermore, the figure indicated that film's absorbance has high values at wave-length in neighborhood that is related to fundamental absorption edge (200nm), after that there will be a decrease as the wave-length increase. Generally, film's absorbance has low values in visible and near infrared region. Such behavior could be defined in the following way: with regard to high wave-length, incident photons do not have sufficient energy for interacting with atoms, photon is going to transmit. In the case when decreasing the wave-length, interaction between material and the incident photon is going to happen and after that there will be an increase in absorbance.

UV-Vis absorption spectra that is related to films have been determined as can be seen in the Fig.(3)which indicated that PAn (EB form), single absorption band which is peaking at around (360nm)exist. Such optical absorption has been related to electronic transition from the highly occupied molecular orbital (HOMO) π-band to lowly unoccupied molecular orbital (LUMO) π*-band related to the electronic states. Absorbance at about (360nm) band is because of HCl that is created from AniHCl, indicating creating PANI. Therefore, more polymerization will happen. The results are in accordance with recent research on PANI synthesized through HCl doping dispersed in nylon 6 and nylon6,6, in which characteristic absorption bands have been(551-1013nm) wave-lengths .

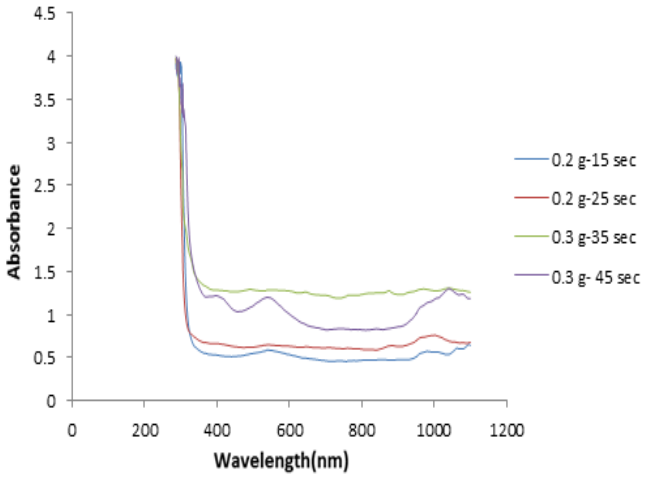

Figure 3. Absorbance spectrum as function of wave-length for PANI (Nylon 6) thin films.

Stability with Regard to DC Electrical Conductivity Retention

Four samples, PANI:Ny-6,6 have been chosen for studying the thermal stability with regard to the DC electrical conductivity retention. It has been indicated that electrical conductivity related to the PANI:Ny-6,6 composites elevated when exposed to HCl, because of doping related to the PAn component of composite films, as can be seen in the Equation:

$$\text{PANI(Ny-6,6)} + n\text{HCl} \longrightarrow [(\text{PANI}-n\text{H}^{+}(\text{Ny-6,6})][(n\text{Cl}^{-})] \quad (8)$$

Isothermal stability testing that is related to HCl doped films with regard to DC electrical conductivity retention has been conducted at a temperature of 130, 140, 150, 160, 170, 180, 190 as well as 200Celsius in air oven. Electrical conductivity measurements have been achieved at intervals of five minutes in accelerated ageing experiments. Electrical conductivity has been measured with regard to time regarding the accelerated ageing as can be seen in the Figures (4,5). Furthermore, thermal stability has been examined through continuously measuring the DC electrical conductivity with increase in temperature from 130 Celsius to 200Celsius for 5 times at intervals of forty minutes. Electrical conductivity that is related to PAn: nylon-6 films has been determined from a temperature of 50Celsius to 130Celsius and indicated to be in semi-conducting region. All composite films were in accordance with Arhenius equation for temperature dependence related to electrical conductivity at a temperature from 30Celsius to 115Celsius. Then, deviation in the electrical conductivity has been identified. Combination regarding some or all following factors might be accountable for the reduction in electrical conductivity beyond a temperature of 130 Celsius like loss of dopant in addition to degradation, dopant's chemical reaction with PAn or the nylon-6,6, semi-conductor to metal transition as well as approaching of *Tg*. Researches on electrical conductivity's stability within isothermal conditions at a temperature of 50, 70, 90, 110 and 130Celsius for PAn and 130, 140, 160, 150, 160, 170, 180, 190 200°C for the nylon-6,6 indicated that electrical conductivity has high stability at a temperatures of 50, 70, 130 and 140Celsiuswhich support the suggestion that all composite films have high stability with regard to DC electrical conductivity retention within ambient conditions not more than a temperature of 90Celsius and electrical conductivity will decrease in terms of time at a temperature of 110Celsiusas well as 130Celiusmight be because of dopant's loss. Stability with regard to DC electrical conductivity retention has been indicated as sufficient when examined through isothermal approaches. PAn:nylon-6,6 composite films have been indicated to have high suitability to be applied in electronic and electrical applications not more than 90Celsius within ambient conditions.

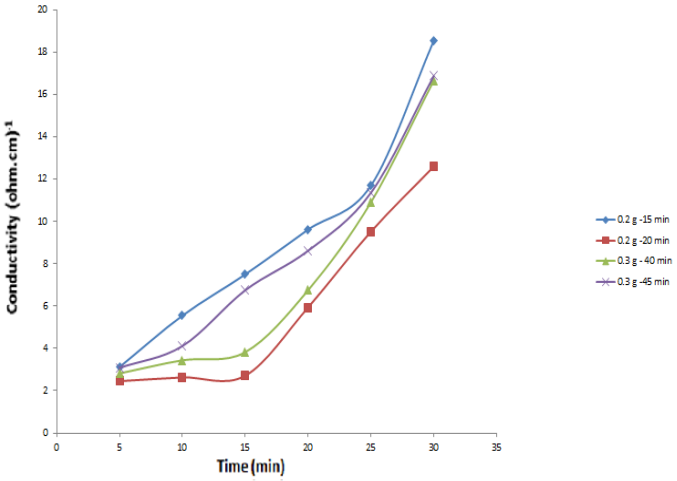

Figure 4. Variation of D.C electrical conductivity with time for PANI (Nylon 6).

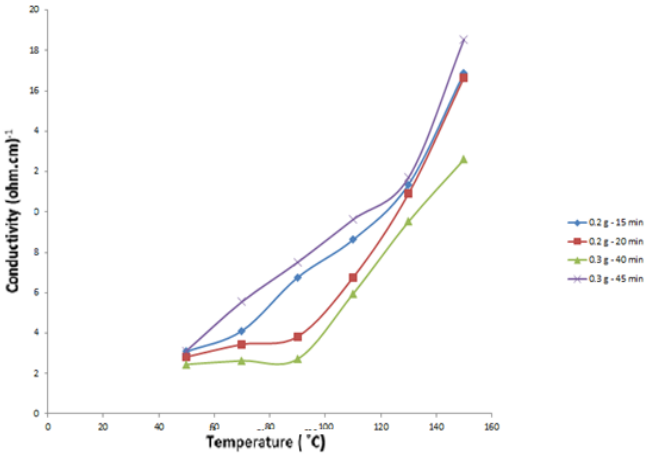

Figure 5. Variation of D.C electrical conductivity with temperature for PANI (Nylon 6).

Hall Effect Measurements

Charge carrier's type, resistivity (ρ), Hall mobility (μ_H), concentration (n_H), in addition to the conductivity (σ) were determined from the Hall measurements. Tables (3 and 4) indicated the major parameters which have been assessed from the Hall effect measurements for PANI(nylon 6) thin films with various temperatures and

concentrations. Hall coefficient (R_H) is important to know the electrical properties, (R_H) depends on the value of incident magnetic field on the film is located vertically in front that field. We can notice from this Tables (3 and 4) which films have p Hall coefficient. This is indicating that conducting type (P–type charge carriers), such result is in accordance with the results of [30].

From this Tables, the Hall coefficient and mobility increases, while conductivity and carrier concentration decrease with increasing of PANI this attributed to the greater charge carrier's concentration leads to shrinking region between Fermi level and the edge of the conductivity this agreement with increase the activation energy with increasing of the adding ratios. Value related to σ subjected to increase with the increase in temperature and PANI. The thin film's structure has been changed to reduce grain size as well as increasing internal grain's barrier potential, and that will make capture change carriers in grain bounding and after that increasing the scattering.

Table (2 and 3) shows the results of the measurements of the Hall effect that all films PA-6 prepared is the n-type through negative sign of the Hall coefficient. In films vestiges there was a clear increasing in the concentration of carriers and a decreasing in the values of mobility at increasing temperatures with increasing doping ratio, this was due largely to an increasing in the concentration of carriers formed near the conductive band, which in turn lead to an increasing the number of donor atoms of electrons capable of ionization within the thermal energy does not exceed the value of topical levels (k_BT), as for the decreasing in mobility shall be due to the increased concentration of carriers. These results are in agreement with[31].

Table 2. Hall parameters for PANI (nylon 6 – 0.2g) thin films

Sample	R_H (cm^3 / C)	n_H (1 / cm^3)	$\sigma_{R.T}$ $(\Omega.cm)^{-1}$	$\rho_{R.T}$ $(\Omega.cm)$	μ_H $(cm^2/ V.s)$
0.2g-15sec	9.712×10^{5}	6.427×10^{12}	2.029×10^{-8}	4.929×10^{7}	1.970×10^{-2}
0.2g-20sec	2.326×10^{8}	2.684×10^{10}	1.858×10^{-7}	5.383×10^{6}	4.321×10^{1}
0.2g-25sec	1.972×10^{7}	3.165×10^{11}	1.871×10^{-6}	5.345×10^{5}	3.690×10^{1}
0.2g-30sec	5.171×10^{6}	1.207×10^{12}	2.742×10^{-8}	3.647×10^{7}	1.418×10^{-1}

Table 3. Hall parameters for PANI (nylon 6 – 0.3g) thin films

Sample	R_H (cm^3 / C)	n_H (1 / cm^3)	$\sigma_{R.T}$ $(\Omega.cm)^{-1}$	$\rho_{R.T}$ $(\Omega.cm)$	μ_H $(cm^2/ V.s)$
0.3g-35sec	8.286×10^{7}	-7.534×10^{10}	1.565×10^{-6}	6.392×10^{5}	1.296×10^{2}
0.3g-40sec	3.967×10^{7}	-1.574×10^{11}	4.497×10^{-6}	2.224×10^{5}	1.784×10^{2}
0.3g-45sec	1.166×10^{7}	5.355×10^{11}	1.005×10^{-6}	9.952×10^{5}	1.171×10^{1}
0.3g-50sec	3.141×10^{8}	1.988×10^{10}	2.677×10^{-6}	3.735×10^{5}	8.408×10^{2}

Thermal Gravimetric Analysis (TGA) Study

Thermo gravimetric Analysis determine the change in mass as temperature's functions. It can be mainly applied to specify degradation temperature, material's absorbed content, the level of inorganic as well as the organic parts in the materials as well as residues of analysis solvent. It does use the sensitive electronic balance through which a sample will be suspended in furnace, which is controlled through temperature programmer. Thermal characteristics of samples of such polymers have been examined by TGA in Argon atmosphere at heating rate (10ºC per minute) [26-29]. With regard to this test, many values have been recognized including Ti, Top, Tf, T50%, Residue at 600 Celsius, and char yields at 500 Celsius as can be seen in (Table 4) .

Temperatures of fifty percent weight loss of (A1-A2) of polymers have been between (420-200) ºC, The char results of (PA6)is26 % at 500 ºC in Argon atmosphere, indicating that they could satisfy the requirements of temperature resistance that may be utilized in a variety of applications. Weight residue of (PA6) is25%, at 600 ºC.

Table 4. Some Thermal Stability Characteristics Curves Thermal Gravimetric Analysis (TGA) of polymers represents the temperature of decomposition.

Sample	T_i	T_{op1}	T_f	$T_{50\%}$	Residue at °C600	Char % At 500°C
PANI (Nylon 6)	480	170	680	200	25	26

Top represents optimal temperature of decomposition.
Ti represents the temperature of the initial decomposition.
T50% represents the temperature of 50% weight loss, which has been obtained from the TGA.
Tf represents the final temperature of decomposition. The final degree of dissociation temperature.
Char% at 400 ° C represents the residual weight percentage at 500°C in Argon by TGA.

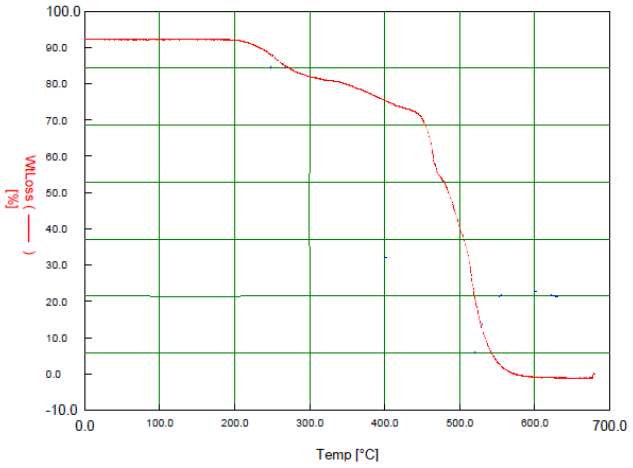

Figure 6. TGA of PAN (Nylon 6)

Differential Scanning Calorimeter Analysis (DSC) Study

DSC can be defined as an approach related to thermal analysis that examined how the heat capacity (Cp) of materials will be transformed through temperature. Recognized mass sample will be cooled or heated and variations in the heat capacity will be indicated as alterations in heat flow. This will allow revealing transitions like crystallization degree (Tc) (20), melting point (Tm), as well as melts glass transitions (Tg). Results related to the (PA6) polymer indicated in figure (7) value of (Tg) of mixture (20.5 Celsius), showing elevation in flow of temperature and after that elevation in rate of absorption of sample to temperature till (Tm) at (510 Celsius) in the case when totally melted and after that the rate of absorption related to sample to heat and through curve has been set Tc of mixture (260 Celsius) [30].

Table 5. Indicate Degree of Tg, Tm, and Tc inDifferential Thermal Analysis:

Simple	Tg (cº)	TM (cº)	TC (cº)
PANI (Nylon 6)	20.5	510	260

Tg: Degree glass transition.
Tm: Melting Point.

Tc: Degree of crystallization

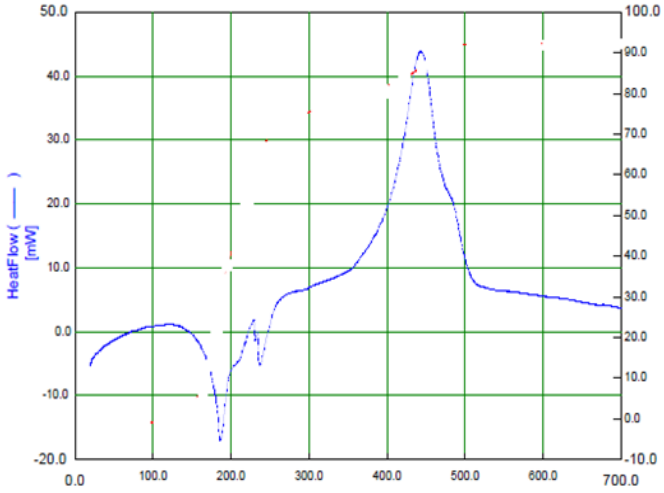

Figure 7. DSC of PAN (Nylon 6)

Conclusions

This study indicated that there is possibility for synthesizing PAn composites with certain traditional thermoplastic polymers like nylons with enhanced mechanical properties, with excellent thermal stability as free standing films or membranes.
The molar ratio regarding PAn to nylon is of maximum impact on the composite material's mechanical properties. A lot of applications related to the PAn conducting polymers might require that is must be prepared as membranes. Preparation related to the conductive PAn:nylon-6,6 composite films is excellently specified via aniline monomer's diffusion into nylon-6,6 matrix succeeded via aniline's oxidative polymerization in nylon-6 matrix method. Therefore, prepared PAn:nylon-6,6 composite films have high electrical conductivity at high doping level. Also, it is indicated that electrical conductivity related to PAn:nylon-6,6films increase with the increase in PAn content of composite. Composite material has been effectively characterized with regard to the electrical properties, SEM and FTIR. The thermal stability with regard to DC electrical conductivity retention is sufficient as examined through a few of the

experimental approaches. The conductivity related to the PAn/Nylon composites following doping with HCl is extremely low in comparison to pure PAn, yet it is extremely high in comparison to Nylon (insulator). PAn/Ny composites change into 7hlatorwhen in the case of treatment with dilute ammonia solution. Conductivity recover totally in the case of re-doping with HCI solution. The majority of formulations related to composites prepared are utilized in electrical as well as electronic applications not more than 90 Celsius within ambient conditions.

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