Synthesis and Analysis of Polyaniline Coated Natural/Synthetic Fiber Composites for Gas Sensor Application

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Abstract

In the present work polyaniline coated natural/synthetic fibers were synthesized using aniline as monomer, HCl as dopant and potassium dichromate as oxidant. The polyaniline composite with fibers was prepared by both in-situ polymerization method and solution process. The thin films of polyaniline and polyaniline coated fibers were obtained on glass slide using PMMA as binder to measure electrical conductivity. The properties of prepared polyaniline coated fibers were studied using various characterization techniques such as electrical conductivity studies, FTIR studies, thermal analysis and gas sensing behavior interms of voltage to understand the efficiency of the prepared composite material.

Keywords: Polyaniline, Natural/synthetic fibers, Thermal analysis, Composite, Gas sensor

1. INTRODUCTION

In the last two decades conducting polymer materials have gained importance since they are easy to process, have low density, high flexibility and have electrical properties similar to conductors and semiconductors. These properties make them ideal to be incorporated into electronic devices for various applications like super capacitors and super conductors, optoelectronic devices, plastic solar cells, electronic charge dissipaters. Conducting polymers has potential growth in the global market presently and is mostly expected to grow further in years to come.

Conducting polymers find a wide range of applications in various fields. Conducting polymers show a significant change in their thermal, electrical, optical properties when subjected to doping [1, 2]. There is a potential increase in usage of conducting polymers because of its high performance, light weight and cost effectiveness. Properties of conducting polymer such as flexibility, stability, chemical resistance and strength have accelerated the demand of conducting polymers globally[2]. Conducting polymers also find application in areas such as batteries, capacitors, solar cells, EMI protection, packaging, antistatic coating, and electrodes. The reversible redox behavior of conducting polymer composites can be used in the biological sensors, fabrication of chemicals and super capacitors [3]. The conducting polymers electrical conductivity can be manipulated by a process called doping which is similar to a process used in semiconductor technology. Upon application of electrical voltage to a conducting polymer system on the polymer chains, charge carriers are created [4].

Polyaniline (PANI) is one of the most commonly used conducting polymers due to its adjustable stability and conductivity over time. The conductivity of polyaniline is attributed to pi-conjugated electron system in doped or partially doped PANI; it also has different electronic states that increase the conductivity of the polyaniline. Polyaniline is a suitable conducting polymer for enhancement of thermal and electrical properties because of its unique oxidation state. It has gained more importance due to its improved electrical, optical, magnetic and thermal properties, good environmental stability, low cost and easy to prepare. Polyaniline as a conducting polymer has more scope due to the adjustability of its electrical properties from semiconducting to metallic region by using various dopants [5-7]. Polyaniline has wide variety of applications including biosensors, chemical sensors, electromagnetic interface shielding, corrosion protection, textiles, fuel cells, antistatic coatings, batteries and super capacitors [8, 9]. Polyaniline has been doped with a large number of dopants. A brief account of the different dopants, solvents and its effect on the properties of polyaniline is available in the literature[10-13].

Polyaniline possesses properties which has high potential to be used as material in the electronic devices. Polyaniline can be coated easily on various substrates/materials like glass, plastics, ceramic materials, textile materials. Polyaniline composites of natural and synthetic fibers have enhanced thermal and electrical properties making it more suitable for electronic devices. In the present work composites of polyaniline with natural/synthetic fibers were prepared and studies were conducted to find the enhancement of electrical conductivity and thermal properties. The electrical conductivity studies of the polyaniline and polyaniline coated fibers are analysed using four-point probe method. The functional group properties of the polymer are analysed using Fourier Transform Infrared spectroscopy (FTIR). The thermal behavior of materials under study are analysed by TGA analysis. The surface morphology of Polyaniline coated fibers are analysed using Scanning electron microscopy [SEM].

2. EXPERIMENTAL

In the present work polyaniline coated natural/synthetic fibers were synthesized using aniline as monomer, HCl as dopant and potassium dichromate as oxidant. The polyaniline composite with fibers was prepared by both in-situ polymerization method and solution process. The thin films of polyaniline and polyaniline coated fibers were obtained on glass slide using PMMA as binder to measure electrical conductivity. The polyaniline coated fibers with highest electrical conductivity were used for gas sensing application.

2.1 Preparation of Polyaniline Coated Natural/Synthetic Fibers by In-Situ Polymerization

In the preparation of Polyaniline coated fibers by in-situ method 2g of Aniline was dissolved in 30ml of distilled water and 10ml of HCl solution, to this solution 2g of Potassium dichromate dissolved in 30ml of distilled water was added drop wise for 30mins under stirring condition for polymerization. 2g of Natural/Synthetic fiber was added to this solution and polymerization process was allowed for 2 hrs at room temperature under stirring condition. The final precipitate formed was then filtered using Whatman filter paper, washed with distilled water and ethanol to remove excess HCl. This was continued till the filtrate becomes colorless. The obtained polyaniline coated fiber was then dried at room temperature. Natural fibers can be employed for the preparation of Polyaniline composites as it is environmentally benign. In the present study used coir fibers and glass fiber are the natural fibers and synthetic respectively are the fibers used for the preparation of conducting polymers composites for the studies.

2.2 Preparation of Polyaniline Coated Natural/Synthetic Fibers by Solution Process

Polyaniline coated natural/synthetic fibers by solution process were prepared by using aniline monomer, oxidant potassium dichromate and concentrated hydrochloric acid. This process involves synthesis of polyaniline and coating of fibers using the synthesized polyaniline. In the synthesis of polyaniline by solution process 5g of aniline was dissolved in 50ml of distilled water and added to 15ml of HCl solution, to this solution 5g of Potassium dichromate dissolved in 50ml of distilled water was added drop wise for 2h under stirring condition for polymerization to take place. The thick dark green precipitate formed was then filtered using Whatman filter paper, washed with distilled water and ethanol to remove excess HCl. This was continued till the filtrate becomes colorless. The synthesized polyaniline was then dried at room temperature.

To coat Natural/Synthetic fibers by solution process emeraldine salt (ES) form of polyaniline has to be converted to emeraldine base form. Emeraldine salt form of Polyaniline was deprotonated using weak base to form emeraldine base (EB) form of polyaniline. 1g of ES form of polyaniline was dissolved in 20% K_2CO_3 and stirred using magnetic stirrer for 3h at room temperature. The blackish blue EB form of polyaniline obtained was filtered using Whatman filter paper, washed with distilled water and dried at room temperature. 1g of this EB form of Polyaniline was dissolved in 20ml of Tetrahydrofuran (THF), which is a solvent. 1g of natural/synthetic fiber was immersed in it for 2h. The polyaniline (EB) coated fiber was filtered using Whatman filter paper then washed with distilled water and dried at room temperature. After coating the fibers with emeraldine base form of Polyaniline it was doped with 10ml of HCl to increase the conductivity due to which it is again converted to green emeraldine salt form.

2.3 Preparation of Thin Films of Polyaniline and Polyaniline Composites for Conductivity Measurement

0.2g of Poly (methyl methacrylate) (PMMA) was dissolved in 4ml of Tetra Hydro Furan (THF) to form a thick paste. 0.1g of polyaniline was added to this PMMA-THF mixture. Thin film of Polyaniline was prepared by coating this mixture on glass slide. Similarly in the same way thin films of polyaniline coated fibers were obtained by adding 0.1 of polyaniline coated fibers to PMMA-THF mixture. The snapshots of thin film of polyaniline, polyaniline coated coir fiber and polyaniline coated glass fibers are shown in Fig. 1a, 1b and 1c.

Fig. 1a Thin Film of Polyaniline

Fig. 1b Thin Film of Polyaniline Coated Coir Fiber

Fig. 1c Thin Film of Polyaniline Coated Glass Fiber

The electrical conductivity of pure polyaniline and polyaniline coated fibers were measured using four-point probe method and the voltage of pure polyaniline and polyaniline coated fibers was measured using multimeter. The electrical conductivity of PANI and PANI composites is obtained from I-V curve obtained using four point probe method. The four point probe method is carried out using PM5 Cascade Probe Station and Semi Conductor device analyzer-B1500A. The equipments required for this

analysis is permanent magnet, or an electromagnet (500 to 5000 gauss), Constant-current source with currents ranging from 10μA to 100mA, High input impedance voltmeter covering 1 μV to 1 V. The surface morphology of pure polyaniline and polyaniline coated natural/synthetic fibers were studied using Scanning Electron Microscopy (SEM). The SEM analysis was carried out using FEI QUANTA 200F microscope. The infrared spectra of pure polyaniline and polyaniline coated natural/synthetic fibers were analyzed by Fourier transform infrared spectroscopy (FTIR) technique. The FTIR analysis was carried out using NICOLET IS5 spectrometer. The samples were analyzed in wave number range of 600-4000 cm^{-1}. The Thermal analysis of pure Polyaniline and selected Polyaniline coated natural /synthetic fiber composites were analyzed using thermo gravimetric analyzer (TGA). The TGA analysis was carried out using PerkinElmer make analyzer.

3. RESULT & DISCUSSION

3.1 Electrical Conductivity studies

Four point probe method was employed to analyse the electrical conductivity property of the thin films prepared and obtained I-V curve is used to obtain the required parameters of the materials used. The voltage drops in the thin films were measured using multimeter. The voltage drop in pure polyaniline obtained was 0.9mV. The voltage of PANI coated natural and synthetic fibers were measured and are given in the table 1.

Table 1 Voltage of PANI Coated Natural/Synthetic fiber

S. No	Polyaniline coated Composites	Voltage measured
1	PANI Coated Coir fiber by Insitu Process	2.3 mV
2	PANI Coated Coir fiber by Solution process	1.1 mV
3	PANI Coated Glass fiber by Insitu Process	1.6mV
4	PANI Coated Glass fiber by Solution Process	0.6 mV

The potential drop of polyaniline coated natural and synthetic fibers by insitu process and polyaniline coated coir fiber by solution process is more than that of pure polyaniline synthesized by solution process. The high potential drop of PANI coated natural/synthetic fibers were obtained as it has high surface area and thickness of the film. Thus by comparing   both   the   processes,   insitu   process   of polymerization is found to be better than solution process because of its homogeneous structure and low band gap. In case of solution process less potential drop was observed   due   to   acid/base   action   or   electro polymerization. Higher conductivity indicates low band gap.

The electrical conductivity of polyaniline and polyaniline coated fibers is obtained using four point probe method. The voltage (V) is measured by varying current (I) at room

temperature which follows a linear relationship to ensure that the samples follow ohmic behavior. The   electrical conductivity of polyaniline was estimated to be   1.01x10^{-5} S/m. The electrical conductivity of polyaniline coated fibers by insitu and solution process is given in Table 2.

Table 2 Electrical Conductivity of PANI Coated Natural/Synthetic fiber

S. No	Polyaniline coated Composites	Electrical Conductivity (S/m)
1	PANI Coated Coir fiber by Insitu Process	4.93×10^{-2}
2	PANI Coated Coir fiber by Solution process	3.18×10^{-2}
3	PANI Coated Glass fiber by Insitu Process	6.23×10^{-3}
4	PANI Coated Glass fiber by Solution Process	4.03×10^{-3}

The voltage measured by varying current at room temperature follows a linear relationship and ensures that the samples follow an ohmic behavior. The electrical conductivity of polyaniline is due to the presence of some conjugation defects like polaron and bipolaron in addition to the electrons and trapped ions. The electrical conductivity of polyaniline composites is high due to its high surface area than pure polyaniline. Thus it is evident that the polyaniline coated fibers/composites have higher electrical conductivity than pure polyaniline. Fig. 2 shows the current-voltage (I-V) curve of pure polyaniline and polyaniline coated fiber composites.

(a)

(b)

(b)

(d)
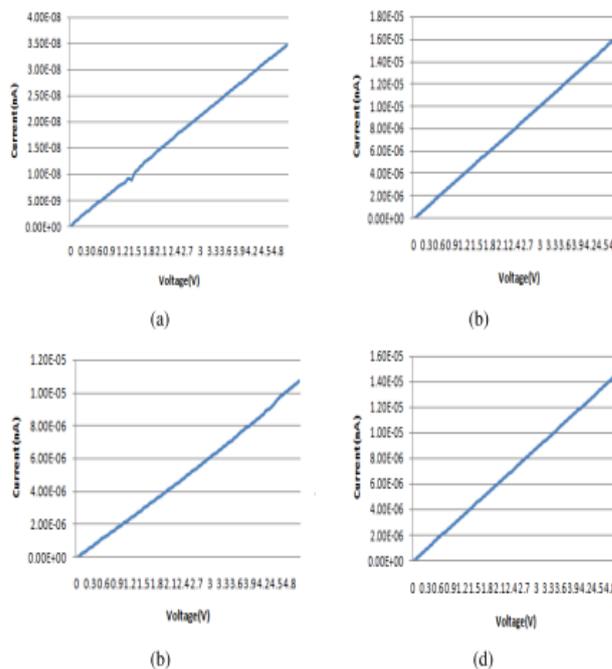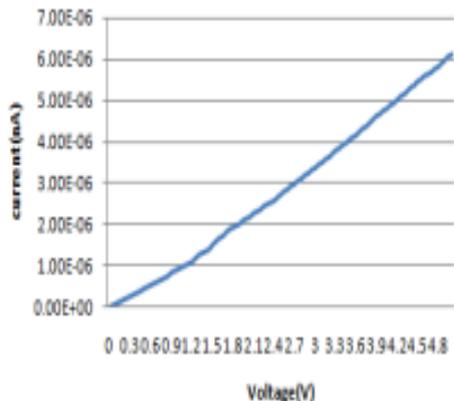
(e)

Fig. 2 IV Curve of (a) Polyaniline, (b) Polyaniline Coated Coir Fiber by InsituProcess, (c) Polyaniline Coated Coir Fiber by Solution Process, (d) Polyaniline Coated Glass Fiber by Insitu Process and (e) Polyaniline Coated Glass Fiber by Solution Process

3.2 Surface Morphology Studies

The surface morphology of PANI synthesized by oxidative polymerization technique using Insitu polymerization method was studied using scanning electron microscope [SEM]. The snapshot of SEM images of pure Polyaniline, Polyaniline coated natural fibers and Polyaniline coated synthetic fibers are shown in Fig 3a, 3b.and 3c respectively. The SEM images of synthesized pure polyaniline shows a clear evolution of morphology of the polyaniline. In fact, for high fractions of polyaniline, the roughness of surface appears. The SEM images of pure Polyaniline shown in Fig. 3a indicates the presence of pores of 3-5µm. The petal like formation indicates the use of potassium dichromate as oxidant. Mass of clews was observed and the average diameter is around 1 micrometer. The coating was uniform and pattern was similar to that of standard image of MERCK. This also shows that Polyaniline prepared is pure and can be further used in the preparation of polyaniline composites for making electronic devices.
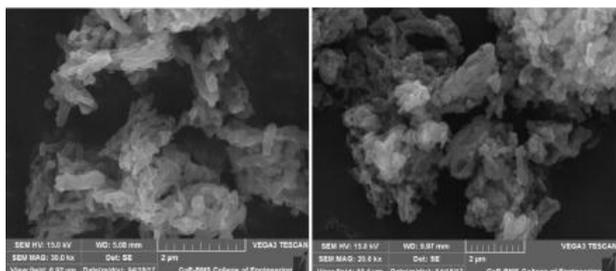

Figure. 3a SEM Images of Pure Polyaniline

The SEM images of polyaniline coated coir fiber shows uniform coating of polyaniline on the coir fiber. The SEM images of Polyaniline Coated coir fiber is shown in Fig.3b. A uniform coating of polyaniline on the coir fiber is observed. There were no clusters found and polyaniline was randomly scattered on fiber surface.
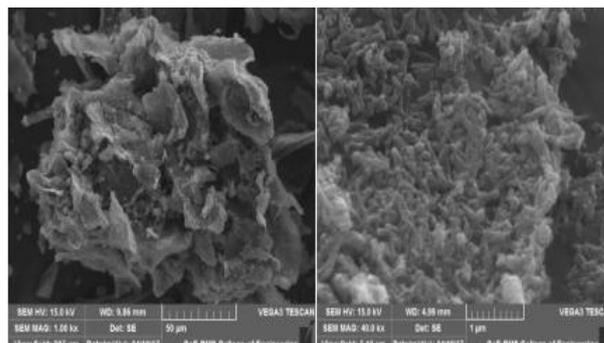

Fig. 3b SEM Images of Polyaniline Coated Coir Fiber

The SEM images of Polyaniline Coated glass fiber shows uniform coating of polyaniline on the glass fiber. The SEM image of polyaniline coated glass fibers is shown in Fig. 3c.
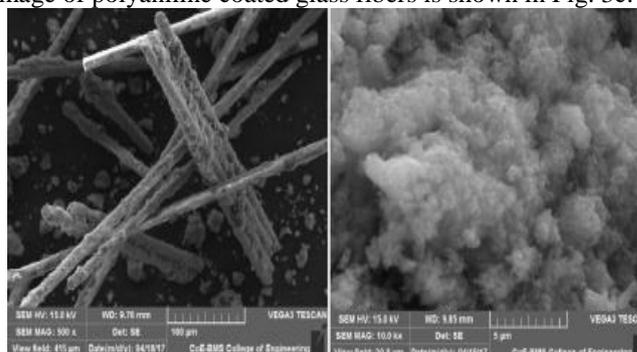

Fig .3c SEM Images of Polyaniline Coated Glass Fiber

The coating of polyaniline on the top of fibers is evidenced to be successful from the SEM images for the surface of PANI coated fibers. The polyaniline top coating is characterized by porous and rough surface morphology. The various preparation parameters which influenced the morphology of PANI are concentration of potassium dichromate, concentration of aniline, concentration of hydrochloric acid and reaction temperature.

3.3 FTIR Analysis

The IR analysis was done to confirm the formation of Polyaniline. The FTIR spectrum of pure Polyaniline is shown in Fig. 4a (plot of % transmittance as a function of wave number cm^{-1}). The peaks at 1491 and 1567cm^{-1} indicates the characteristic C=C stretching deformation of benzenoid rings and C=N stretching deformation of quinoid rings, respectively which is in accordance with the results reported in literature. The peaks at 1135 cm^{-1} is due to C-N stretching and vibration in the polar on structure of polyaniline. The peak at 2922 cm^{-1} is due to N-H stretching. The peaks at 1300 and 801 cm^{-1} can be assigned to C-N stretching of secondary aromatic amine and C-H deformation out-of-plane of the 1, 4-disubstituted benzene ring.
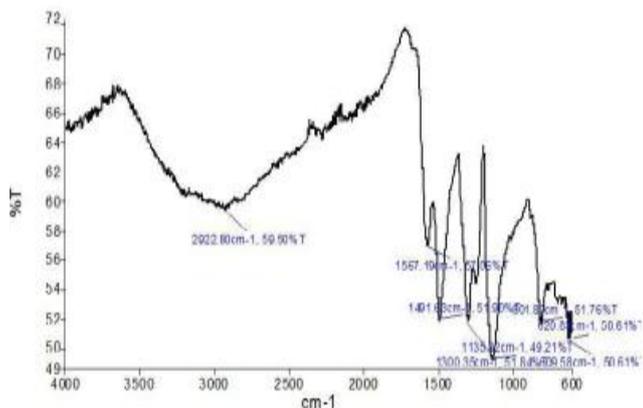
Fig.4a FTIR Transmittance Spectra of Pure Polyaniline

The FTIR spectra of Polyaniline coated Coir fiber by Insitu process is shown in Fig. 4b. The coir fiber mainly comprises of cellulose, lignin and hemicellulose. The peaks assigned to polyaniline were observed in the Polyaniline coated coir spectrum at 1580 and 1497 cm^{-1}, these are the characteristic of symmetric stretching vibration of the quinoid and benzenoid rings of Polyaniline.
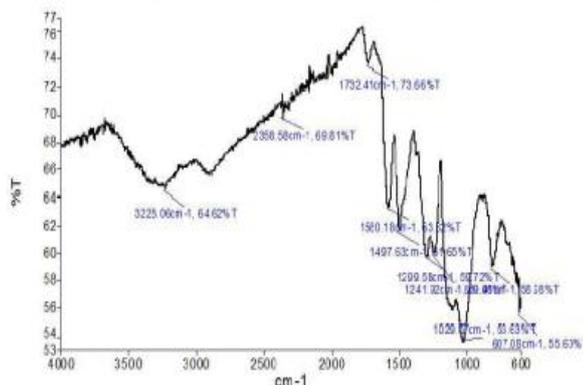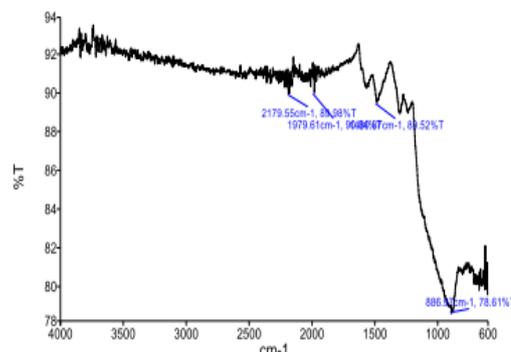

Fig. 4b IR Transmittance Spectra of PANI Coated Coir Fiber by In-situ Process

The FTIR spectra of Polyaniline coated Coir fiber by Insitu process is shown in Fig 4.c
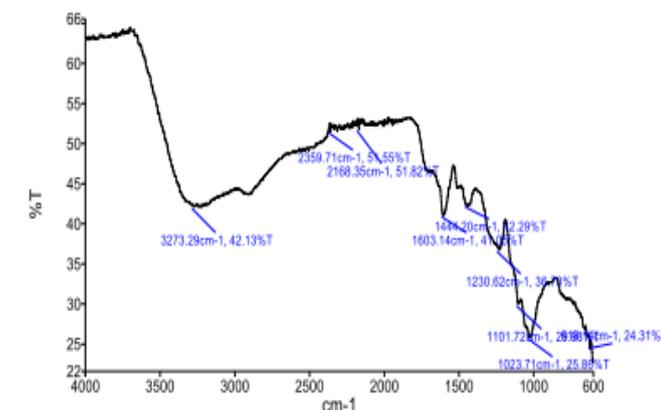.

Fig. 4c IR Transmittance Spectra of PANI Coated Coir Fibers by Solution Process

The FTIR spectrum of Polyaniline coated glass fiber is as shown in Fig. 4d. The peaks at 1093, 1272, 1460, 1572 can be assigned to N=Q=N stretching (Q is the quinoid ring), C-N

stretching vibration, C=C stretching of benzenoid ring and C=C stretching of quinoid ring, respectively.

Fig. 4d IR Transmittance Spectra of Polyaniline Coated Glass Fiber by Insitu Process

The FTIR spectra of Polyaniline coated Coir fiber by Insitu process is shown in Fig. 4e.

F   Fig.4e FTIR Transmittance Spectra of Polyaniline Coated Glass Fiber by Solution process

3.4 Thermal Analysis of Pure Polyaniline and Polyaniline Coated natural Fiber Composites

The Thermo gram of pure Polyaniline is shown in Fig 5a. The thermo gravimetric analysis was carried out by heating the test samples from 20-800°C. Both the thermograms shows 3 major stage of weight loss. The peak at 125°C relates to elimination of water and small adsorbed molecules. The next stage of weight loss at 250°C indicates removal of acids and at 500°C polymeric chain degradation occurs. At the temperature range of 187-700°C, the weight loss accounted to 90 % which is attributed to weight loss of the polymerized polyaniline and the thermal decomposition of polyaniline chains.
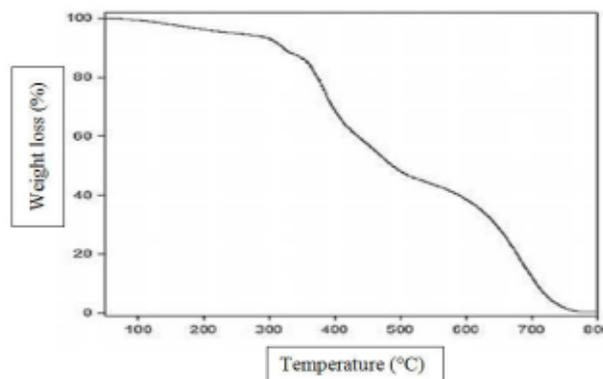

Figure 5a TGA curve of Polyaniline

The Thermo gram of Polyaniline coated natural fiber composites by insitu and solution process is shown in Fig. 5b. The thermo gravimetric analysis was carried out by heating the test samples from 20-800°C. The weight loss occurs in 3 stages. The first stage of weight loss occurs at 100-250°C where initial dehydration takes place that is elimination of water molecules and small absorbed molecules. The second stage of weight occurs at 280-350°C where hemicellulose, cellulose and lignin degradation of coir fiber takes place. The third stage of weight loss occurs at 250-500°C where C l ions and polymeric chain degradation occurs.
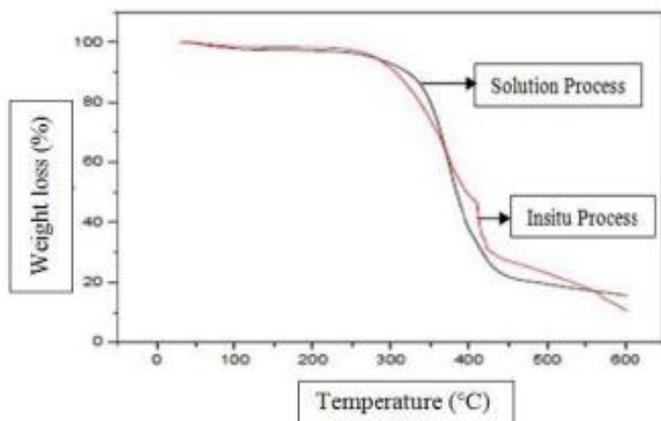

Fig. 5b TGA curve of Polyaniline Coated Coir Fiber

3.5 Gas Sensing studies - Effect on passing Ammonia gas on the thin film

Ammonia gas was passed on thin film of pure PANI and PANI coated natural/ Synthetic fibers in an evacuated chamber. The change in voltage was measured by varying the concentration of ammonia and the results are shown in Table 3. The plot of variation of voltage with concentration of ammonia is shown in Fig. 6. The potential drop was measured in terms of millivolt.

Table 3 Variation of voltage with concentration of ammonia

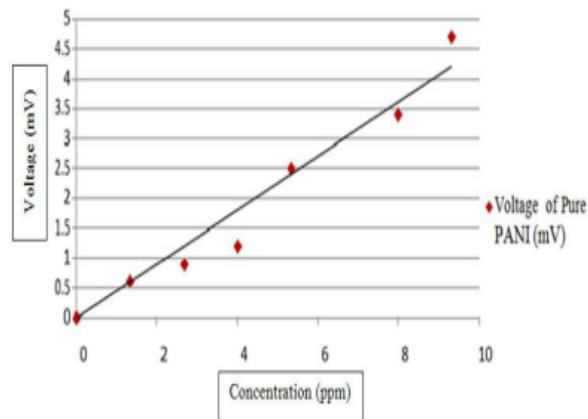| Concentration of ammonia (ppm)	Voltage of Pure PANI (mV)	Voltage of PANI coated coir fibers		Voltage of PANI coated glass fibers	
		Insitu process	Solution process	Insitu process	Solution process
0	0	0.45	0.20	0.50	0.16
1.33	0.6	0.68	0.26	0.68	0.20
2.67	0.9	0.92	0.33	0.95	0.34
4.00	1.2	1.50	0.70	1.50	0.45
5.33	2.5	2.70	0.85	3.00	0.60
8.00	3.4	4.20	1.00	4.20	0.80
9.33	4.7	5.50	2.00	6.00	1.50

Fig 6a Plot of Variation of Voltage v/s Concentration for pure PANI

It is observed that with increase in ammonia concentration there is a linear increase in potential drop.
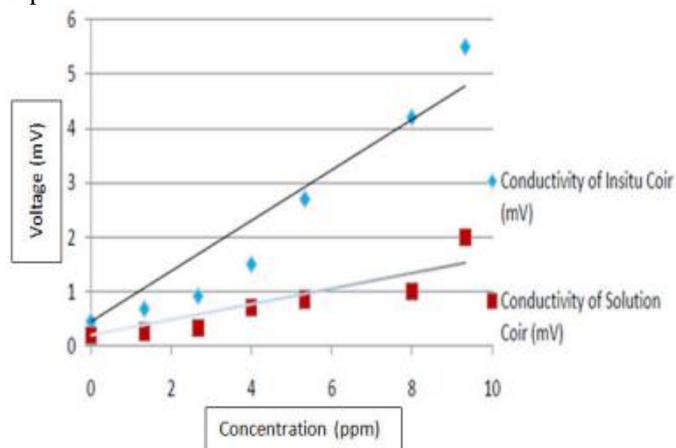

Fig.6b Plot of Variation of Voltage v/s Concentration for PANI coated Coir fibers
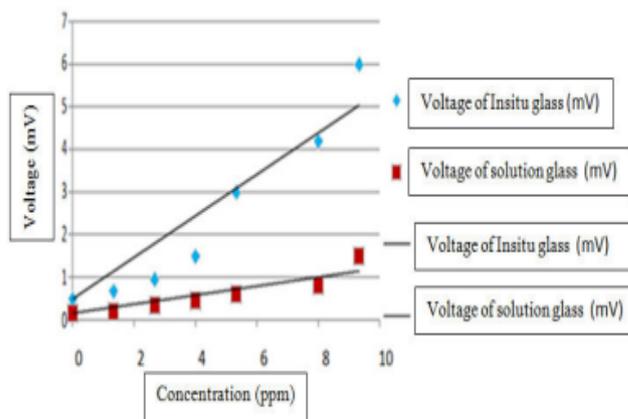

Fig 6c Plot of Variation of Voltage v/s Concentration of PANI coated glass fiber

It is evident from the above figures that good sensing ability of ammonia gas was evident by polyaniline composites than pure polyaniline due to large surface area of polyaniline composites. It was observed that with increase in ammonia concentration there is a linear increase in voltage. The gas

sensing ability of polyaniline composites prepared by insitu process is greater than polyaniline composites prepared by solution process. Hence, polyaniline coated Natural/Synthetic fiber composites prepared by insitu process can be used for similar gas sensing applications.

4. CONCLUSION

Polyaniline and polyaniline composites of natural/Synthetic fibers were synthesized successfully by both insitu polymerization process and solution process. The natural fibers can be commonly used as ideal carriers of conducting polymer than synthetic fibers as they are abundantly present in nature, low cost, sustainable, renewable and recyclable. The structural, thermal and functional properties were evaluated and predicted using scanning electron microscope, thermo gravimetric analysis and Fourier transform infrared spectroscopy respectively. The electrical measurement shows increase in electrical conductivity of composites as compared to pure PANI. The voltage of polyaniline and polyaniline coated fibers was measured using conductivity meter. The voltage of pure polyaniline was measured to be 0.9mV. The voltage of polyaniline coated natural and synthetic fibers was measured to be more than that of pure polyaniline. The electrical conductivity of polyaniline measured using four point probe method was estimated to be 1.01×10^{-5} S/m. The electrical conductivity of polyaniline composites prepared by both in-situ and solution process was estimated to be greater than that of pure polyaniline as it has high surface area and less band gap. The FTIR analysis confirms the formation of polyaniline. The FTIR spectra reveals a strong intermolecular interaction between -N-H groups in the polyaniline ring and the -OH functional group in the conducing polymer composites. Therefore, the FTIR spectroscopy proves the presence of polyaniline by confirming the presence of the functional groups and also the interaction between polymer chains and fibers Thermal analysis shows increase in thermal stability of PANI composite by about 300°C than that of pure polyaniline. Thermo grams of both polyaniline and polyaniline coated fibers show three major stages of weight loss from which the thermal stability of both polyaniline and polyaniline composites is observed. The scanning electron micrographs of polyaniline coated natural and synthetic fibers revealed that the coir fiber surface and glass fiber surface was completely coated with a uniform polyaniline layer which led to an electrical conductivity quite similar to that of the pure polyaniline. The petal like formation in the micrograph of polyaniline and polyaniline composites indicates the use of potassium dichromate as oxidant. The coating of pure polyaniline was observed to be uniform . The polyaniline composites were coated as thin film on glass slide and were tested for sensing ammonia gas. A good sensing ability of ammonia gas was evident due to large surface area of polyaniline composites. It was observed that with increase in ammonia concentration there is a linear increase in voltage. The insitu process provides a quick, simple, cheap and precise way to construct a gas sensor than solution process with controlled thickness and morphology of polyaniline coating. Hence, polyaniline

coated Natural/Synthetic fiber by insitu process can be used for similar gas sensing applications. The results observed in this study give new feasibility for use of conducting polymer and polymer composites as conductive filler in insulating polymer. The results obtained help in the development of conducting polymer composites for gas sensor applications.

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