Microwave Characterisation of PANI treated Jute Using Suitable Solvent Methanol and DMSO

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Abstract. This work discusses the study on the dielectric properties of PANI(Polyaniline) treated jute fabric. The method involves usage of liquid dielectric measurement probe. Suitable solvents such as Methonal and DMSO(Dimethyl Sulfoxide) are used. The microwave properties such as dielectric constant, dielectric loss, absorption coefficient, and conductivity are studied in x-band(8-12 GHz) frequency band. In both the solvents, the PANI reacts in a similar manner. This exhibits similar nature of results with negligible deviations.

Keywords: PANI, Jute, X band, Solvent, Methonal, DMSO

1. Introduction

Human beings are exposed to electromagnetic fields from personal communication systems and radiation from electronics and electrical instruments. Hence a dielectric material is needed in antenna as well as in circuits for the reduction electromagnetic emission to the human body [1-4]. The materials with high polarizability are known as dielectrics. When a high frequency radiation field passes through a dielectric material, its electric dipoles align to the field. The properties of a dipole are collectively termed as dielectric properties. It includes complex permittivity, conductivity, skin depth, heating coefficient and attenuation constant etc., The X band ranges from 8GHz to 12 GHz which most of the common applications of microwaves exist [4].

Water is an important solvent used in daily life. Including this some important organic solvents are also used. These solvents are chosen based on their concentration and solubility level. Concentration is defined as the amount of compound that is dissolved in a certain volume of solvent. Solubility: Solubility is the amount of compound mixed that is soluble in a certain volume of solvent. There are two important types of solvents used, they are (1) Polar solvents (2) Non-polar solvents. Dipole-dipole bonding is possible in the polar solvents (i.e.) there is a separation between the positive and the negative charges.

The intermolecular forces act between one molecule and other (or) between large molecules having asymmetrical charge distribution. The most widely used solvent in chemistry is the alcohol. Alcohol is considered as the organic compound. This is represented by, hydroxyl group (-OH) bound with the carbon atom of any substituted alkyl group. Of these the important alcohols are methanol, ethanol, and propanol. There is a strong intermolecular H-bonding interaction between PANI-EB chains and alcohols. This interaction makes PANI to have novel chemical and physical properties [5, 6].

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PANI has attracted much interest worldwide because of its thermal and chemical stability. Its production cost is low and it can be easily doped with inorganic and organic acids to prepare the conductive form. Thermal stability of PANI is superior to other ICPs. Emeraldine base PANI has low conductivity in the range of 10^{-10} S/cm, while its salt created by modifying the base’s oxidative state has conductivity of 30S/cm. Shaolin et al. explained the special features of PANI related to good stability with respect to thermal properties and chemical properties [7]. In extended coil form, electrons are easier to delocalize, thus resulting in an increased conductivity. PANI has many sensing applications such as in ammonia sensors, EMI shielding, and precious metals recovery. PANI is investigated also for neural probes, controlled drug delivery, tissue engineering and shielding materials [8].

The PANI treated jute substrate has to be characterised for the suitability of either dielectric material or conductive material for antenna design and circuits as well as shielding purpose. Hence the aim of the work is finding one of the most convenient and also an inexpensive method for the characterisation. PANI treated jute fabric is dissolved in Methanol solvent for three days and DMSO solvent for seven days. The resulting products are characterized with Keysight technologies 85070E Dielectric probe.

2. Materials and Methods
The section consists of the fabrication process of the Jute composed of Polyaniline for the purpose of making a substrate material and also characterisation of the corresponding material characterisation in microwave regime.

2.1 In-situ synthesis of Polyaniline on Jute Fabric
Polyaniline coating is developed by in situ chemical polymerization of aniline on the jute fabrics. In this process, freshly distilled 100mM aniline is dissolved in the bath containing respective strength of hydrochloric acid solution. Dry jute fabric sample is placed in the above solution at 5°C and allowed for 2 hours to exhaust the aniline and dopant solution. 100ml ammonium per sulfate is separately dissolved in hydrochloric acid solution and it was then slowly added in to the diffusion bath to initiate
the polymerization reaction. The whole polymerization reaction is carried out at 5°C for 1 hour. After completing the polymerization process, the Polyaniline (PANI) coated fabric is taken out and washed in alcoholic water and dried at ambient condition as depicted in figure 2.

As indicated in figures 3-4, using test tube, a small quantity of 50 mg PANI treated jute was kept within the 100 ml Methanol solvent for five days with frequent shaking of the bottles. Similarly, the jute of same quantity was kept in 100 ml of DMSO solvent for eight days. The colour changes were observed inside the solution.
The liquid dielectric measurement (Keysight Technology Dielectric Probe 85070) was done. The above figure 5 shows the testing setup of dielectric value. The following formulae are used [16] to find the Loss tangent value, absorption and effective conductivity of the material.

\[
\text{Loss tangent } \tan\delta = \frac{\varepsilon_r'}{\varepsilon_r}, \quad \text{Absorption Coefficient } a_f = \frac{\varepsilon_r'}{c}.
\]

Effective Conductivity \(\sigma_{eff} = 2\pi\varepsilon_0\varepsilon_r'\) Where, \(\varepsilon_r'\) is Dielectric Constant, \(\varepsilon_r\) is the loss factor, \(f\) = Frequency of operation, \(c\) = Velocity of light, \(\varepsilon_0\) = Permittivity of free space.

3. Results and Discussion

The main work carried out in this paper is finding of the electrical characteristics such as permittivity, loss tangent, conductivity and absorption co-efficient of the conductive polymer immersed jute substrate. The following section gives the details about the above mentioned parameters.

3.1 Fourier Transform Infrared Spectrometry

FTIR spectra of control and dry heat-treated jute fibres is taken in the BRUKER FTIR analyser (Model ALPHA) using an attenuated total reflectance (ATR) mode. The data is recorded in the range from 4000 to 500 cm\(^{-1}\) wave numbers. The surface chemistry of the Polyaniline coated jute fibre is studied through FTIR spectra and presented in figure 6.
The FTR spectra of the Jute-PANI composite showed the peaks responsible for the functional groups of the PANI. Peaks at 1490 and 1600 cm\(^{-1}\) responsible for stretching vibrations of -N-B-N- and N=Q=N structures (where -B- and =Q= stand for benzenoid and quinoid moieties in the Polyaniline backbone); 1250 cm\(^{-1}\) responsible for polaronic structure of PANI; 1170 cm\(^{-1}\) responsible for -N=Q-N+=B-(protonated state) and it confirms the coating of PANI on the surface of the jute fibre. The peak at 3308 cm\(^{-1}\) is decreased and it revealed that PANI masks the free hydroxyl groups present in the jute polymer. The bands at 1600, 1490, 1170, 1300, 1250, 1030 and 806 cm\(^{-1}\) are corresponding to Polyaniline.

![Figure 6. FTIR of Control Jute and PANI treated Jute.](image)

3.2 **SEM Image** The surface morphology of control and Polyaniline coated jute fibre is captured in an environmental scanning electron microscope (Model FEI Quanta 200F with Oxford-EDS system IE 250 X Max 80) in different magnifications as in figure 7-8.
Figure 7. SEM Image of Control Jute.

Figure 8. SEM Image of PANI treated Jute.
3.3 Electrical Characteristics

From figures 9 – 11, at lower frequencies the permittivity value increases because of the large relaxation time. Dielectric loss increases with frequency. This is due to displacement current. Conductance component is equal with applied electric field, hence the loss tangent. The absorption coefficient and conductivity are related to dielectric loss as seen in figure 12 and 13 respectively.

![Figure 9. Permittivity Value](image1)

![Figure 10. Dielectric Loss](image2)
Figure 11. Loss Tangent

Figure 12. Absorption Value
4. Conclusions
The characteristic study of the PANI treated jute is performed with the help dielectric probe method. From the measurement results of permittivity and dielectric loss, loss tangent, absorption coefficient and conductivity are derived using formulae. The conductivity and absorption of the material is noticeable. The shielding characteristics further can be improved. The material can be used as a conductive material in antennas as well as in circuits. Comparing Methanol solvent, PANI treated jute exhibits maximum values as in DMSO solvent. Both loss tangent and absorption coefficient values were obtained in similar manner. However, two solvents Methanol and DMSO are considered as a suitable solvent in the electrical characteristics study of PANI treated jute since both results are differ by small value. In future the analytical validation will be performed. Using test fixtures of solid material, the jute material will be evaluated for dielectric characteristic study. In near future, the mechanical tensile test and temperature stability test will be performed for the suitability of the PANI treated jute for antenna and RF circuit design.

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References
[1] Ammayappan L and Chakraborty S 2017 Coating of Silver Nanoparticles on Jute Fibre by in situ Synthesis, Cellulose 24 (3), 1563-1577, 2017
[2] Ammayappan L Sekhardas, R Guruprasad, Deb P R and Prasanta K G 2016 Effect of Lac Treatment on Mechanical Properties of Jute Fabric/Polyester Resin Based Biocomposite Indian Journal of Fiber & Textile Research, 41, pp. 312-317, September 2016.
[3] Ammayappan L, Nayak L K, Ray Dp, Das S, Roy A K 2013 Functional Finishing of Jute Textiles- An Overview In India Journal Of Natural Fibers, 10 (4) p-390-413
[4] Siti Amira Othman 2013 Methanol as a Suitable Solvent for Polyaniline Emeraldine Base, In Proceedings of Progress of Physics Research in Malaysia ISBN No- 978-0-7354-0797-8 p-357-360
[5] Zhou H.H, Jiao S.Q, Chen J.H, Wei WZ and Kuang Y.F 2004 Relationship between preparation conditions, morphology and electrochemical properties of polyaniline prepared by pulse galvanostatic method (PGM), *Thin solid films* **450** (2) p-233-239

[6] Tan C K and Blackwood D J 2000 Interactions between polyaniline and Methanol vapour, *Sensor Actuactor*. **B71** p184-191

[7] Shaolin Spectral Characteristics of Polyaniline nano Structures synthesized by using Cyclic Voltammetry at different Scan rates *Journal of Physical Chemistry-B* **112** p11558-11563

[8] Mondal 2017 An effective strategy to enhance mechanical, electrical, and electromagnetic shielding effectiveness of chlorinated polyethylene-carbon nanofibernanocomposite*Composites Part B: Engineering* **109** p 155–169 DOI:10.1016/j.compositesb.2016.10.049

[9] Lakshmi 2008 Comparison of Microwave and Electrical Properties of Selected Conducting Polymers, *Microwave and Optical Technology Letters* **50**(2) p 504-508 DOI: 10.1002/mop