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RESEARCH ARTICLE - PHYSICS

Humidity Sensitive Flexible Microwave Absorbing Sheet Using Polyaniline–Polytetrafluoroethylene Composite

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Abstract

Pelletized or powdered polyaniline composite, a potential candidate for microwave absorbers, was synthesized in the sheet form for the first time, and its absorption characteristics along with structural, electrical and mechanical properties are presented. Enhanced microwave absorption behavior of this novel, thin, flexible, lightweight sheet in hydrous environment was analyzed for various humidity-dependent sensor and electromagnetic applications. The preparation method of protonated chlorine-doped polyaniline (PANI), and its synthesis in the sheet form using polytetrafluoroethylene (PTFE) are discussed. The surface and structural morphology were characterized by XRD and SEM, which reveal the granular, macro-porous and polycrystalline structure of the material. A transmission–reflection-based waveguide technique was used for obtaining the permittivity of sheets in the frequency range of 3–9 GHz by employing the Nicholson–Ross algorithm, and it was verified by cavity perturbation method. The temperature stability of the PANI–PTFE conducting sheet was checked using four-probe method. Conductivity enhancement of the sheet in hydrous environment was studied using a humidity chamber. The microwave absorption studies at various humidity conditions were carried out using waveguide method which also illustrated its potentiality as a humidity sensor. The mechanical strength of the proposed conducting polymer sheet was tested by standard load–extension procedure. To make this PANI–PTFE polymer material suitable for anechoic chamber-like applications, it was impregnated in polyurethane foam and its humidity-related microwave absorption studies were carried out using free space method.

Keywords Flexible composites · Polyaniline · Microwave absorber · Humidity sensor

1 Introduction

Microwave absorbers were of great importance even during the initial stages of research and development of electromagnetic devices and diversification of their applications. Nowadays, the relevance and the importance of these absorbers have increased manifold due to the exponential increase in the types and quality of electromagnetic gadgets. Owing to the high density of electromagnetic (EM) users added care has to be taken for avoiding the issues related to electromagnetic interference (EMI), which is of prime importance in communication systems and in various industrial, scientific, and medical (ISM) applications. Another major concern is health hazard confusions related to overexposure of microwave radiations which demand some sort of protective measures for the safe use of these advanced technologies. Such scenario demands intense research for the development of novel type of microwave absorbers having added advantages over conventional types in order to address the emerging issues related to state-of-the-art microwave technology.

Microwave absorbers are broadly classified based on the principle of electromagnetic absorption caused by conductivity and electric/magnetic losses owing to its hysteresis nature. The boundary mismatch, a major concern in connection with any type of microwave absorbers can be tailored

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by proper adjustment of permittivity (ε) and permeability (μ) and also by using various trigonometric shapes for the absorbing medium [1]. Most commonly used absorbents are carbonyl ions (e.g., carbon black, graphite powders, carbon nanoparticles) [2,3] and ferrites [4–6] usually mixed inside the polymers like plastic and rubber made in different variants like sheet and foam [1,7]. Polyurethane and polystyrene are widely used foam-type material as microwave absorber in 1–40 GHz region [8,9]. Carbon with neoprene binders is normally used nowadays to prepare microwave absorber foams for anechoic chambers [10–12]. Another group of cost-effective microwave absorbers for the range of 1–20 GHz are proposed by different researchers which uses agricultural wastes and rice husk in pyramidal and sheet forms, respectively. Though these absorbers are lightweight, they are rigid and require complicated synthesis process [13,14].

Another class of microwave absorbers use conducting polymers as the matrix in which different dielectric and magnetic fillers are added for enhancing the absorption characteristics. Some of the commonly used absorbents are barium titanate ($BaTiO_3$) [15–17], strontium ferrites [18], erium oxide (Er_2O_3) [19], iron-nickel alloy (NiFe) [20] and silicon carbides [21] which find wide range of applications in broadband microwave absorption and shielding. Development of these materials has opened up an entirely new dimension of polymer technology which finds uses in various fields like dynamic pH-sensing in micro-fluidic devices [22], amperometric cholesterol and alcohol biosensors [23,24], electrochemical microsensors [25] and super capacitor applications [26–28]. These polymers are also proposed in flexible forms like graphene sheet (GS)/polyaniline nanofiber composite [29] and free-standing graphene with polyaniline [30] which may find application in the development of low-cost electrode materials for storage devices. These conducting polymer composites are ideal for situations requiring high-degree microwave absorption levels. They have added advantages of selecting absorption levels and frequency ranges by suitable choice of fillers.

For moderate absorption purposes, conducting polymers can be used without any dielectric or magnetic fillers [31–33]. Polyaniline (PANI) and polypyrroles (PPy) are two such stable conducting polymers proposed to have applications in microwave shields and absorbers. Troung et al. [34] proposed a moderate conductivity tunable microwave absorber for use at 12–18 GHz where added care has to be taken to protect the original fiber shape of the polypyrrole. John et al. [35] proposed conducting polyaniline as a choice for microwave absorber and analyzed the environmental stability and absorption characteristics. The conductivity and the complex permittivity of polyaniline are analyzed in detail for pelletized and powdered form for selected frequencies [36].

In this paper, we present a self-standing, lightweight and flexible polyaniline-based microwave absorber in the form of a thin sheet. It shows enhanced microwave absorption properties in comparison with already reported powder and pellet forms of polyaniline. This novel polycrystalline sheet was prepared from polyaniline by embedding it in polytetrafluoroethylene (PTFE) matrix. It shows an added characteristic of high sensitivity to humidity-based microwave absorption. Absorption characteristic studies of this polyaniline-based foam-type microwave absorber are also presented.

2 Methods and Measurements

2.1 Material Preparation

Protonated chlorine-doped polyaniline was prepared by the standard chemical oxidation method [35,37,38]. Polyaniline in powder form was synthesized at room temperature from aniline in the presence of 1 M ammonium peroxydisulphate (APS) as oxidant, and 1 M HCl as dopant in the ratio 1:6:12 and polymerization continued for 5 h. The filtrate was then washed with acetone and diluted HCl solutions to remove the unreacted aniline and was air-dried to get polyaniline powder.

In order to prepare polyaniline-based microwave absorber sheet, this conducting polymer powder was used as filler in PTFE matrix. The lightweight PTFE adhesive was chosen as matrix to provide flexibility and to enhance the conductivity of PANI due to the closer arrangement of constituent particles. For this, the polyaniline powder (in gm) was dispersed in de-mineralized water (in ml) in the ratio 1:10 in a ultrasonicator. 7 wt% of polytetrafluoroethylene in aqueous solution was added to the above mixture and stirred for 1 h using a magnetic stirrer, and the suspension was filtered. The filtrate was air-dried at room temperature and ground to fine powder using a mortar. By adding a few drops of isopropyl alcohol, it was made into dough which was rolled into PANI–PTFE hybrid flexible sheets. During the synthesis of the sheet, it was observed that the increase or the decrease in the wt% of the PTFE in solution taken reduces the feasibility of transforming the composite into sheet form. Two such sheets of 0.4 and 0.9 mm were prepared by the above procedure. Figure 1 shows the photograph of PANI–PTFE hybrid sheet of thickness 0.9 mm.

Since many ISM applications prefer foam-type microwave absorbers, polyaniline-based foam sheets were also synthesized. Finely crushed 2 gm of polyaniline powder was dispersed in a mixture of 10 ml distilled water and 7 wt% PTFE solution. Then, it was continuously stirred for 1 h to get uniform PANI–PTFE dispersion which acts as a dielectric filler inside the foam. Polyurethane foam of 25 cm × 25 cm × 2.5 cm dimension was then impregnated with the above dispersion and air-dried at room temperature to obtain PANI–PTFE foam-type microwave absorber.

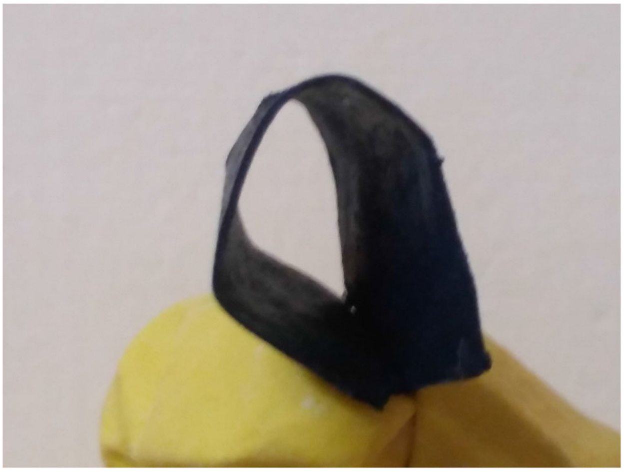

Fig. 1 Photograph of polyaniline–polytetrafluoroethylene (PANI–PTFE) hybrid sheet of thickness 0.9 mm

2.2 Measurement Techniques

The microwave absorption characteristics of the PANI–PTFE hybrid sheet was analyzed at room temperature and room humidity by evaluating its permittivity from the reflection and the transmission coefficients which were obtained by waveguide method. Measurements were taken for polyaniline in powder form also. The absorption power level was normalized to 0 dB before taking measurements. For powdered sample, this procedure was done by keeping low loss polyvinylchloride (PVC) sample holders with a filling space of thickness 3 mm inside the rectangular waveguide. The same procedure was repeated for powdered sample with filling space of 5 mm also. In the case of PANI–PTFE hybrid sheet, two samples of thickness 0.4 and 0.9 mm were used. In both cases, the sample was placed inside the waveguide covering the entire cross section with the plane of absorber perpendicular to the propagation direction. The transmission (S_{21}) and reflection (S_{11}) coefficients of the samples were done in 3–9 GHz frequencies using suitable waveguide sections connected to a Vector Network Analyzer (VNA). Figure 2 represents a schematic diagram of the experimental setup.

Since polyaniline is a conducting non-magnetic material, its effective permeability (μ_r) is assumed to be 1. The real and imaginary parts of the permittivity were calculated from the hybrid parameters using Nicholson–Ross Algorithm [39–45] with the help of equations

$$\frac{\mu_r}{\varepsilon_r} = \left(\frac{1+\Gamma}{1-\Gamma}\right)^2 \tag{1}$$

and

$$\mu_r\varepsilon_r = -\left[\frac{c}{\omega d} ln\left(\frac{1}{P}\right)\right]^2 \tag{2}$$

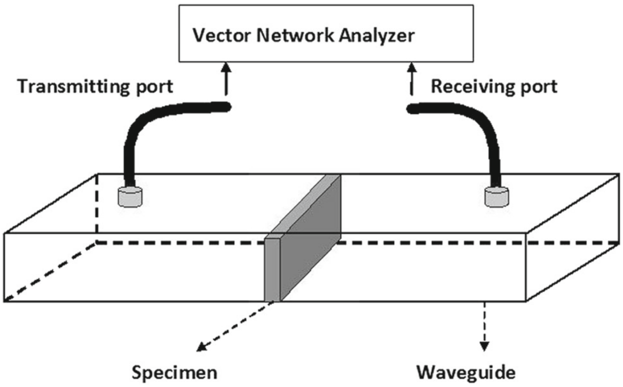

Fig. 2 Waveguide setup for measuring the transmission (S_{21}) and reflection (S_{11}) coefficients of polyaniline powder and PANI–PTFE hybrid sheets

where d is the thickness of the sample, ω is the angular frequency, P is the propagation factor, Γ is the reflection coefficient, and c is the velocity of light. The results obtained were verified using standard cavity perturbation technique [36].

The humidity sensitive microwave absorption properties of the PANI–PTFE were analyzed by taking the S_{21} curve for various humidity conditions. For this, a rectangular humidity chamber was constructed using perspex sheet and humidity was regulated using an atomizer-circulating fan setup. The possible changes in the D.C. conductivity in relation to various humidity conditions were measured by keeping the sheet inside a humidity chamber setup as shown in Fig. 3. For microwave absorption studies at different relative humidity (RH) values between 30 and 90%, the PANI–PTFE sheet was immediately shifted from the humidity chamber to the microwave setup and S_{21} curves were taken. To analyze the temperature stability of the newly fabricated sheet, conductivity–temperature study was carried out using standard four-probe method.

The surface morphology of the sheet was investigated using Scanning Electron Microscope (SEM, JSM-6390LA). Crystalline nature of the PANI powder and PANI–PTFE sheet samples was analyzed using XRD equipped with CuK_α ($\lambda = 1.54$ Å). Mechanical strength and the Young's modulus of the prepared PANI–PTFE were evaluated using the conventional load–extension technique. Microwave absorption characteristics of PANI–PTFE foam was analyzed using free space method by placing the foam in between two wide-band standard gain horn antennas connected to VNA [46]. Photograph of the experimental setup placed inside an anechoic test box is given in Fig. 4.

3 Results and Discussion

SEM images of hybrid sheet show macro-porous structure with clusters of PANI–PTFE granules. Figure 5 depicts the

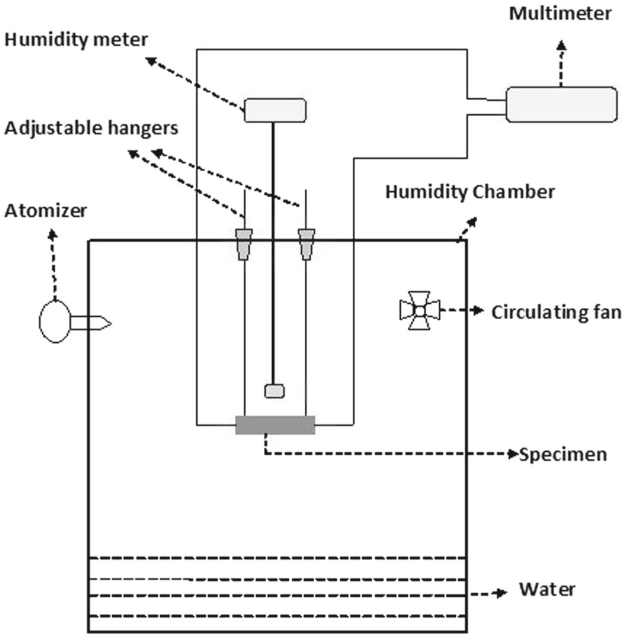

Fig. 3 Pictorial representation of atomizer-circulating fan setup for humidity varying measurements

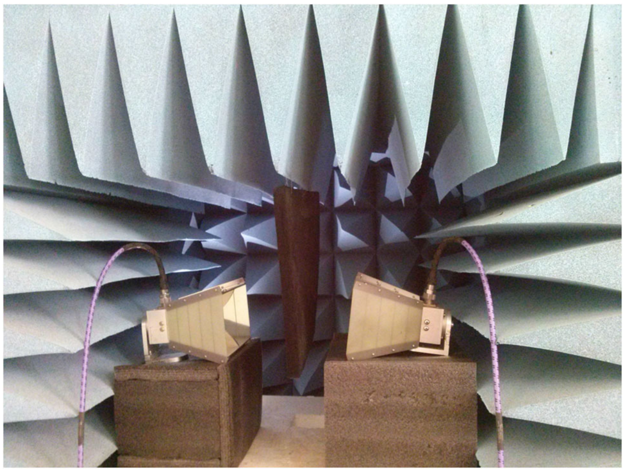

Fig. 4 Free space measurement setup of PANI–PTFE foam-type absorber

images obtained for a sample of thickness 0.9 mm. The cylinder-shaped inclusions seen scattered in Fig. 5c are the unglued molecules of PTFE.

The XRD pattern of polyaniline powder shows it as amorphous in nature [47–49], while PTFE shows neither a fully amorphous nor a fully crystalline structure [50–52]. The insertion of PANI into PTFE matrix was examined by XRD and Fig. 6 shows the spectra of PANI–PTFE conducting polymer sheet. The spectrum shows a polycrystalline structure due to the presence of peaks marked as ‘a’ at angles (2θ) 18.869°, 32.396°, 37.835° and 42.108° with spacing (d) 4.70055, 2.76133, 2.37595 and 2.14420 Å, respectively

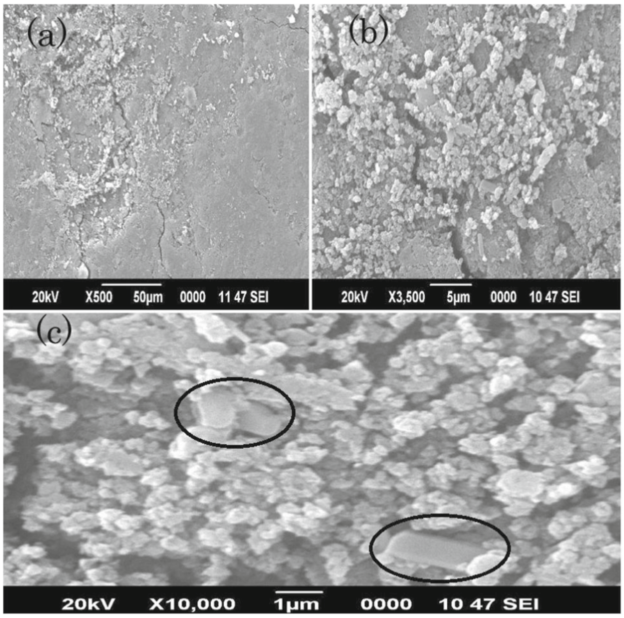

Fig. 5 SEM images of PANI–PTFE hybrid sheet of thickness 0.9 mm at different resolutions **(a, b, c)**, where unglued PTFE granules are shown within elliptical rings **(c)**

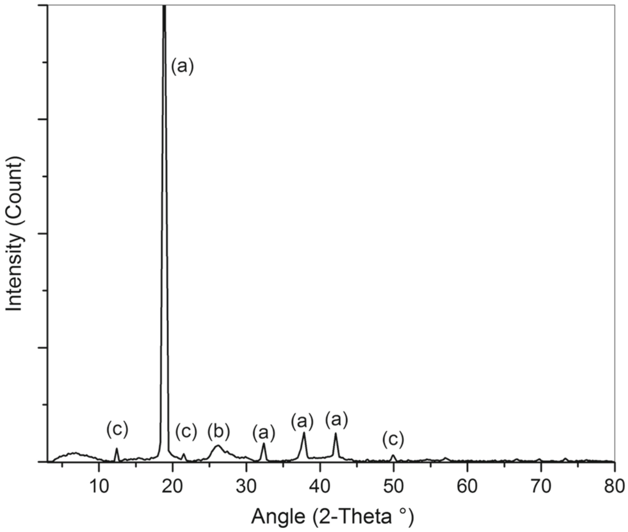

Fig. 6 X-ray diffraction pattern of PANI–PTFE hybrid sheet

which is due to the presence of PTFE [50,51]. The broad peak marked as ‘b’ at an angle 26.5° in the spectrum is due to the PANI [47,48]. The binding process of PANI with PTFE is also resulted in other peaks marked as ‘c’ at angles 12.401°, 21.509° and 49.914° with spacing 7.13174, 4.12805 and 1.82563 Å, respectively, depicts the enhanced crystalline nature.

Conductivity property of the prepared sheets was analyzed using four-probe method, and variation of conductivity with temperature is given in Fig. 7. The graph shows relatively steady conductance around room temperature (305 K)

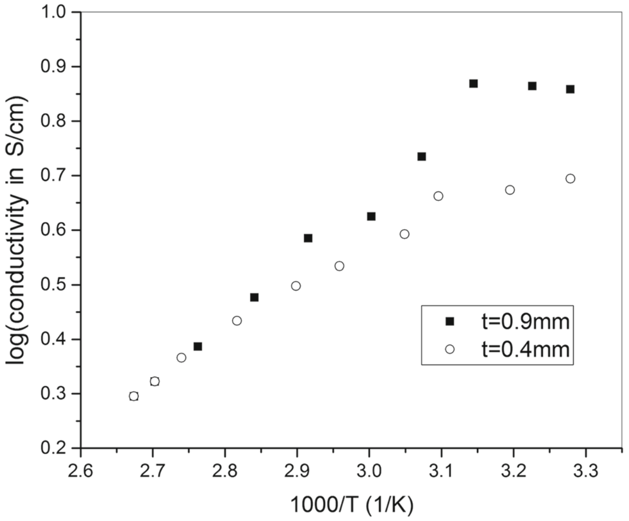

Fig. 7 Conductivity–temperature curve of PANI–PTFE sheet of 0.9 and 0.4 mm thickness measured using four-probe setup at 30% RH

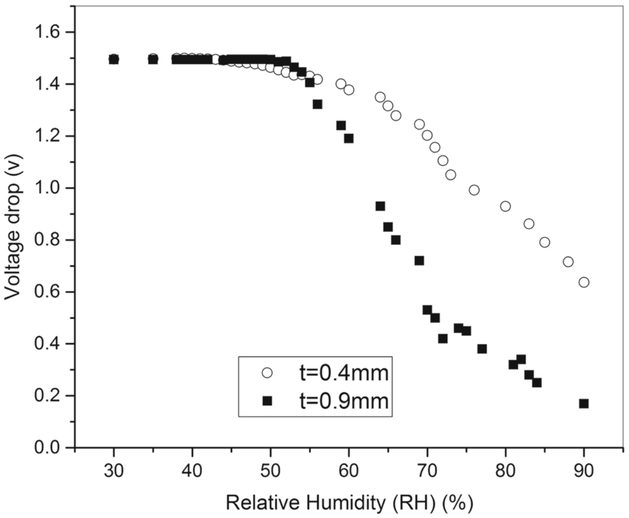

Fig. 8 Humidity response on the voltage drop measured between two points of 1 cm apart for the PANI–PTFE hybrid sheet of 0.9 and 0.4 mm thickness at 30 °C

for both samples and shows gradual decrease in conductivity with increase in temperature, which clearly indicates the conducting behavior for our PANI–PTFE sheets. However, the thick sheet ($t = 0.9$ mm) shows higher conductivity at low temperatures compared to thin sheet ($t = 0.4$ mm) as expected. The conductivity dependence on humidity in terms of voltage drop was measured for both sheets and is depicted in Fig. 8. It is quite evident from the graph that as relative humidity increases beyond 50% RH, the conductivity of both sheets shows marked enhancement. From the figure, it is clear that for the thick sheet voltage drop is lower than the thin sheet for higher humidity values owing to the lower resistance of the thick sample.

Table 1 Readings from cavity perturbation method

Parameters	Frequency (GHz) at	
	7.869	8.784
Real part of permittivity (ε')	3.449	3.2478
Imaginary part of permittivity (ε'')	1.4007	0.6659
Conductivity (σ), S/m	0.6129	0.3254
Skin depth (ρ), m	0.0073	0.0094
Dielectric heating coefficient, J	0.7139	1.5017
Absorption coefficient	136.9863	106.383

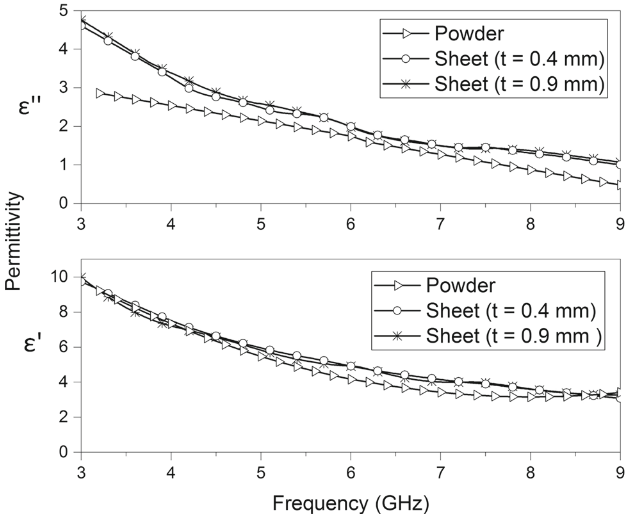

Fig. 9 Real (ε') and imaginary (ε'') parts of permittivity of polyaniline powder and PANI–PTFE hybrid sheets

The real and imaginary parts of permittivity of the polyaniline powder and PANI–PTFE sheets of thickness $t = 0.4$ mm and $t = 0.9$ mm were evaluated using Nicholson–Ross algorithm [Eqs. (1) and (2)] and are given in Fig. 9. Both parts of permittivity show gradual decrease with frequency. There is a difference for the permittivity between powder and sheet forms for both real and imaginary components. The difference due to the thickness of the sheet is observed to be negligible. The variation for imaginary part of the permittivity between powder and sheet form is noticeably high in the lower and higher frequencies of the experimental region, whereas for the real part this deviation is observed in the intermediate frequency regions. This markable variation in the imaginary part of permittivity is indicative of the prepared sheets ability to show more conductivity which may enhance its microwave absorption properties. The permittivity values were also verified for the PANI–PTFE hybrid sheet ($t = 0.9$ mm) using cavity perturbation method for selected frequencies and values obtained are given in Table 1. Table 1 also shows some other related parameters like conductivity, skin depth, dielectric heating and absorption coefficients which were also obtained by the cavity method.

The microwave absorption studies of the PANI–PTFE hybrid sheets of thickness $t = 0.4$ mm and $t = 0.9$ mm were studied for dry and wet forms by measuring its reflection and transmission coefficients for frequency range 3–9 GHz. The dry sheet is at room humidity of 30% RH and wet one is at high humidity level of 90% RH. Figure 10a, b shows S_{11} and S_{21} obtained for thickness $t = 0.4$ mm, whereas Fig. 10c, d shows the results for $t = 0.9$ mm. It is evident from Fig. 10a, c that S_{11} values show a slight increase in reflection with increase in frequency. Both wet and dry sheets irrespective of thickness show the same results indicating that reflection mainly depends on the surface smoothness and not on the thickness and the moisture condition of the sheet. From Fig. 10b, d, following inferences can be drawn. As the frequency increases, the average absorption level decreases, but not in a linear manner. The absorption level is observed to be dependent on thickness of the sheet more prominently on the upper half of the experimental frequency band. In both cases, we find an enhanced absorption for the wet sheets which clearly indicate a higher microwave absorption property of the humid sheet. For the thin sheet ($t = 0.4$ mm), the difference between the absorption levels of the wet and dry sheets is around 3 dB at lower frequency region (3–6 GHz), whereas at higher frequency region (6–9 GHz) it is around 12 dB. But in the case of thick sheets, the difference between the wet and dry sheet is around 6 dB irrespective of the interacting frequency. The potentiality of using this PANI–PTFE hybrid polymer sheet as a good choice of microwave absorbing material is quite evident from the above figure. Increase in thickness and humidity of the PANI–PTFE sheets results to a good extent in the enhancement of microwave absorption which may be useful in various electromagnetic absorbing and shielding applications. The possibility of metallic inclusions during preparation of PANI–PTFE sheets can be explored for further enhancement of absorption levels.

For analyzing the flexibility of our newly formed PANI–PTFE sheet, the elasticity in terms of Young's modulus Y was calculated from its load–extension graph (Fig. 11) and the average value (loading–unloading) was found to be around 4.5 GN/m^2. This value is in the range of a typical nylon fiber which makes our hybrid sheet flexible as well as a durable one for different applications.

In order to use newly formulated PANI–PTFE material as a novel microwave absorber in applications like anechoic chamber, we examined the absorption characteristics of PANI–PTFE impregnated inside the polyurethane foam. The transmission coefficients obtained for the dry (30% RH) and humid (90% RH) conditions measured using free space method is presented in Fig. 12. A noticeable increase in

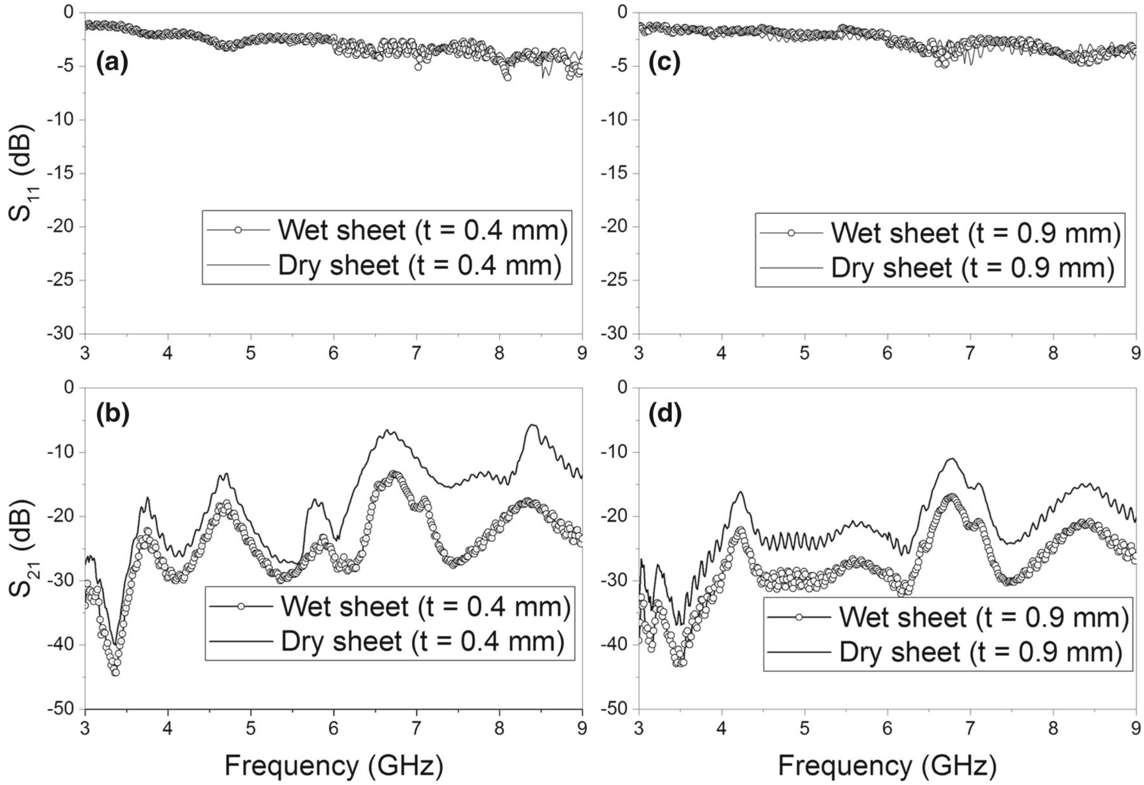

Fig. 10 Transmission (S_{21}) and reflection coefficients (S_{11}) of PANI–PTFE hybrid sheets of different thickness at different humid conditions in 3–9 GHz frequency range. **a**, **c** S_{11} for $t = 0.4$ and 0.9 mm, respectively, **b**, **d** S_{21} for $t = 0.4$ and 0.9 mm, respectively

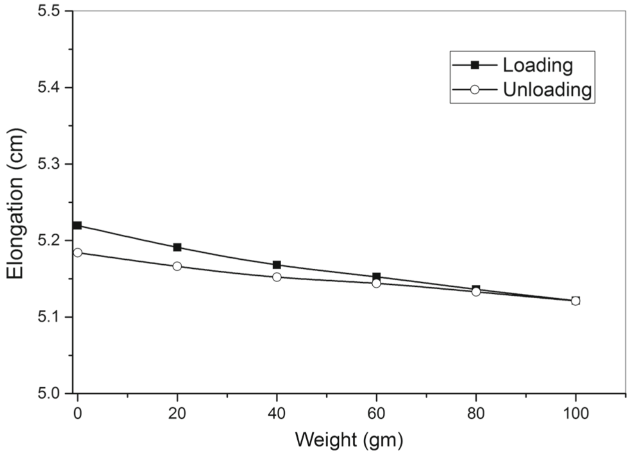

Fig. 11 Load–extension graph for PANI–PTFE hybrid sheet of dimension 6.8 cm × 1.5 cm × 0.13 cm

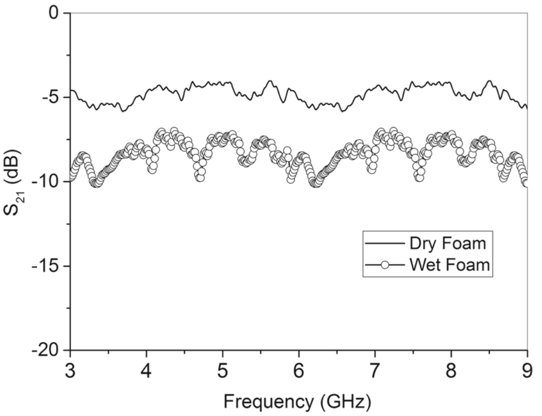

Fig. 12 Plot of transmission (S_{21}) coefficient of PANI–PTFE embedded foam in dry (30% RH) and humid (90% RH) conditions

absorption level of around 5 dB for the wet foam in the entire frequency range is observed.

4 Conclusions

Polyaniline–polytetrafluoroethylene (PANI–PTFE) hybrid sheet synthesized had a polycrystalline structure. This thin, flexible and lightweight sheet had excellent microwave absorption characteristics along with an added property of showing marked variation in absorption level with different humidity content, which makes this sheet an ideal candidate for humidity-based microwave sensors and other electromagnetic applications. The studies on mechanical strength and temperature stability of this PANI–PTFE sheet predict that it is suitable for state-of-the-art microwave technology. This cost-effective microwave absorber was also fabricated in thick foam form which makes it suitable for anechoic chamber-type applications.

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