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Electromagnetic Interference Shielding Efficiency Enhancement of the PANI-CSA Films at Broad Band Frequencies

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Abstract—A material sample of Camphour Sulphonic Acid doped Polyaniline (PANI-CSA) is contemplated towards its conceivable use as a microwave shield. Shielding towards electromagnetic interferences (EMI) is measured over various frequency bands by the waveguide method. Plane wave electromagnetic theory is used to generalize the overall reflection and transmission coefficients of the polymer. EMI shielding of the polymer, in the form of Shielding Efficiency (SE), is analyzed over the microwave frequency range from 2 to 18 GHz, demonstrating the potential value of the polymer as an electromagnetic interference (EMI) shield for commercial purposes. The shielding film is fabricated using standard procedure with CSA as the dopant and m-cresol as the solvent. The shielding effectiveness as high as 45 dB for the sample of PANI doped with CSA is observed.

1. INTRODUCTION

The exponential growth of electronic industry has produced some unwanted offshoots in the form of electromagnetic interferences (EMI). This adversely affects the military and civil communication systems, and hence reduction of EMI is an intriguing research topic all around the world. Over the past few decades, extensive research has been performed in the development of EMI shielding materials which can reduce the impact of electromagnetic coupling with the incident EM wave.

Since the discovery of polyacetyle, the research world has been in a shift towards the world of conducting polymers [1] especially towards its tunable conductivity which is approximately 10^5 s/cm [2] while copper has 10^6 s/cm. The high conductivity of the polymer contributes to high shielding efficiency (SE) [3, 4]. The conductivity of the polymer can be tuned by adding appropriate dopants to the conducting polymer [5]. This can make the material somewhat ideal as a strong candidate against the metal [6–8] which is used as a shield in EMI atmosphere. Conventional EMI shielding techniques focused on metallic materials [3, 9, 10] and their composites, which can effectively reflect the electromagnetic waves. Metals have good mechanical strength but have the disadvantages of heavy weight, easy corrosion and uneconomic processability [11, 12]. Such a situation needs materials with finite conductivity and opaque nature to microwaves; conducting polymers can offer an attractive solution for this [7, 13, 14]. In this regard, PANI based composites have a vital role due to its tunable conductivity, permittivity, permeability, non-corrosiveness, nominal processing cost and on top of this its thermal and environmental stability [15] ensuing in technocommercial applications [6, 16].

Conducting polymers are intrinsically light weight, flexible and have tunable shielding effectiveness [17]. It is well known that the doping process affects the dispersion of the polymer. The composite of PANI-Emeraldine Base with functionalized protonic acids, such as camphor sulfonic

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acid (CSA), in a liquid medium, generally into organic solvents is now referred to as the conventional method of doping. These protonic acids oxidize the conducting polymer chain and the presence of larger counterions are creating more inter chain space in the bulk. Polyaniline became conductive by the protonation of nitrogen using these functionalized protonic acids. After the protonation, there will be polarons or bipolaron along the conjugated chain. High charge delocalization in the molecule, causes polaron conduction band to be half filled and thus an increase in the electrical conductivity. The hoping between the metallic islands or polaronic clusters are the reason behind the charge transport in Pani Films. Conducting polymers are usually classified as highly disordered systems [18].

In the earlier stages of the research, the solubility of polyaniline was a complex issue. During the recent years, this issue has been solved successfully through doping with protonic acids [19]. The hydroxyl group in m-cresol forms a hydrogen bond to the carbonyl group in camphor sulphonic acid. An enhanced Van der Waals interaction produced by the phenyl ring becomes coplanar with one of the PANI. This enhanced interaction may be the reason behind the increased solubility of CSA doped PANI in m-cresol. During the evaporation of m-cresol, stacking to crystal structure may twist the rings due to the planar m-cresol molecules on top of PANI rings. This also supports the high conductivity in the sample. When bulkier counterions are used, the solubility is observed to be increased [20]. M-cresol is small enough to diffuse into PANI cluster and has the ability to exchange a proton with camphour sulphonate ion and PANI, while CSA is relatively big enough to stay in the PANI crystalline region [21].

In this paper, we study the characteristics of Polyaniline-CSA and its use as an effective EMI shield. In this work, we analyze the characteristics of Polyaniline-CSA in various microwave bands S, X and Ku. Studies based on different samples of PANI CSA show that the shielding contribution is mainly by absorption [22]. Useful shielding effectiveness in the commercial application is 40 dB. But for military or near-field application it is 70–100 dB. The values ranging from 20 to 50 dB are reported in the microwave band [8, 23–28]. The polymer film presented here shows better performance in the broad band range than the existing PANI-CSA films.

2. SHIELDING EFFECTIVENESS: THEORY AND MEASUREMENT

The theory behind SE of a material is well reviewed by Schelkunoff [29]. Accordingly, the reflection and transmission characteristics are dependent on the thickness of the material. There are mainly three factors contributing towards shielding effectiveness; reflection, absorption, and multiple reflection inside the material sample [6] as in Fig. 1. In metal, the SE is mainly contributed by the reflection

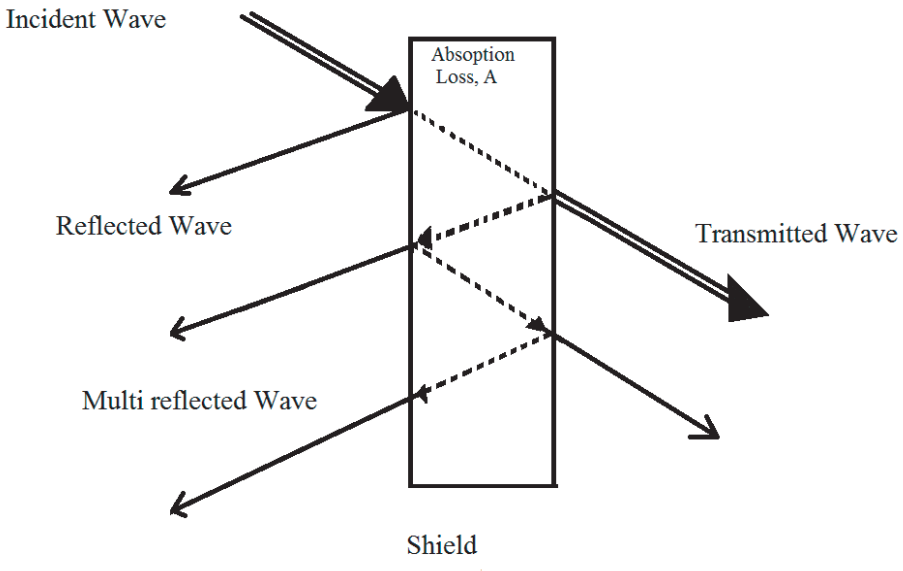


Figure 1. Shielding theory.

phenomenon due to the presence of a large number of free electrons [30]. In some materials, part of the incident wave is absorbed due to reactive heating of the material or ohmic losses which becomes the major contributor towards shielding by absorption. There is one more mechanism which is caused when incident wave undergoes multiple reflections inside the sample. This can be negligible when the Shielding Efficiency due to absorption exceeds approximately 10 dB [31], or the thickness of the sample used as shield is greater than the skin depth [32].

Shielding effectiveness is the ratio of the microwave power incident to the power transmitted as [33]

$$SE(\text{dB}) = 10\log_{10}(P_i/P_t) \tag{1}$$

where P_i and P_t are the power of the incident and transmitted EM waves, respectively. Total shielding is the sum of reflective, absorptive and multiple reflective components [33, 34]

$$SE = SE_A + SE_R + SE_M \tag{2}$$

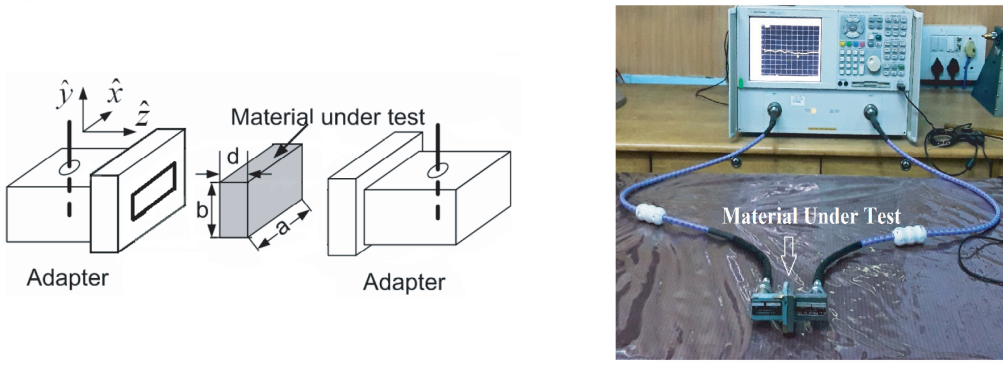
The fabricated sample of PANI-CSA, which effectively covers the cross section of the waveguide is placed between the two waveguide adaptors connected to the network analyzer as shown in Fig. 2(a). Photograph of the experimental set up of the waveguide method used for the measurement of the certain sample is also shown in Fig. 2(b). Full port TRL calibration technique is used here because of its high accuracy. From the measured reflection and transmission parameters the shielding effectiveness is calculated. The reflectance (R) and Transmittance (T) are represented from the scattering parameters as given below [35]

$$T = |E_T|/|E_i| = |S_{12}|^2 = |S_{21}|^2 \tag{3}$$

$$R = |E_R|/|E_i| = |S_{11}|^2 = |S_{22}|^2 \tag{4}$$

where S_{11} and S_{21} are the reflection and transmission coefficients, respectively. The sum of terms T, R and A should be unity [35],

$$\text{i.e.,} \quad A + T + R = 1 \tag{5}$$



(a) (b)

Figure 2. (a) Schematic representation of the wave guide with the sample to be measured. (b) Photograph of the experimental set up of the Waveguide measurement method.

As T and R can be obtained from scattering parameters, A can be calculated from Equation (6). In the case of effective absorbance, A is related to T and R by following equation [35].

$$A_{eff} = \frac{1 - R - T}{1 - R} \tag{6}$$

SE_R, SE_A are the contribution of shielding effectiveness due to reflection and absorption towards the total shielding efficiency, defined as [36, 37].

$$SE_R = -10\log(1 - R) \tag{7}$$

$$SE_A = -10\log\left(1 - A_{eff}\right) = -10\log\frac{T}{1 - R} \tag{8}$$

$$SE_T = -10\log\frac{T}{1-R} - 10\log(1-R) \tag{9}$$

$$SE_{total} = 10\log\frac{1}{T} \tag{10}$$

where SE_{total} is the total shielding effectiveness which is the sum of the effects of shielding due to absorbance and reflection.

2.1. Material Preparation

HCl doped polyaniline was prepared by the chemical oxidative polymerization of aniline in the presence of HCl using Ammonium per sulphate (APS) as the initiator. Aniline was distilled first and 2 ml of aniline was stirred with 50 ml of 1 molar HCl. Then APS solution was added drop-wise to the solution while stirring. The mixture was ultrasonicated for 30 minutes and then the stirring was continued for 12 hours at 0–5°C using ice water bath. The polyaniline formed was filtered, washed with 1 molar HCl and water till the filterate becomes colourless, then the polymer was dried in an oven at 50°C and grounded well for further process.

In order to re-dope with camphor sulphonic acid (CSA) dopant, the HCl doped polyaniline was dedoped by stirring with 0.1 molar ammonia solution. Then it was filtered, washed with distilled water and dried at 60°C in an air oven for 24 hours. The dried polyaniline-emeraldine base was mixed with CSA in the molar ratio of 1 : 0.5. The CSA doped PANI was made into solution by stirring with m-cresol at room temperature for 2–3 days. The PANI solution was then casted onto a glass substrate and dried at 60°C in hot air oven. After drying, CSA doped PANI films were peeled off and the shielding properties were measured. PANI prepared was pelletized using a die. The film peeled off from the substrate is good enough to cover the cross section of the wave guide which is used in the measurement set up. A pictorial representation of the procedures used for the fabrication of the proposed film is shown in the Fig. 3. Due to screening of the interchain and the inter dopant coulomb repulsion, the dopants might be firmly stuffed as possible in polar solvent, e.g., m-cresol [38].

Different types of doping are used to enhance the conduction of electrons in PANI. Protonic acid doping is used here which gives enhanced conductivity. The selection of solvent is also important where the m-cresol will give a higher electron transport in the sample. HCl doped PANI salts have both crystalline domain and amorphous region. When this salt is dedoped it will become amorphous.In the case of m-cresol, delocalized π bonds on the phenyl group of m-cresol tempted π-π stacking interaction

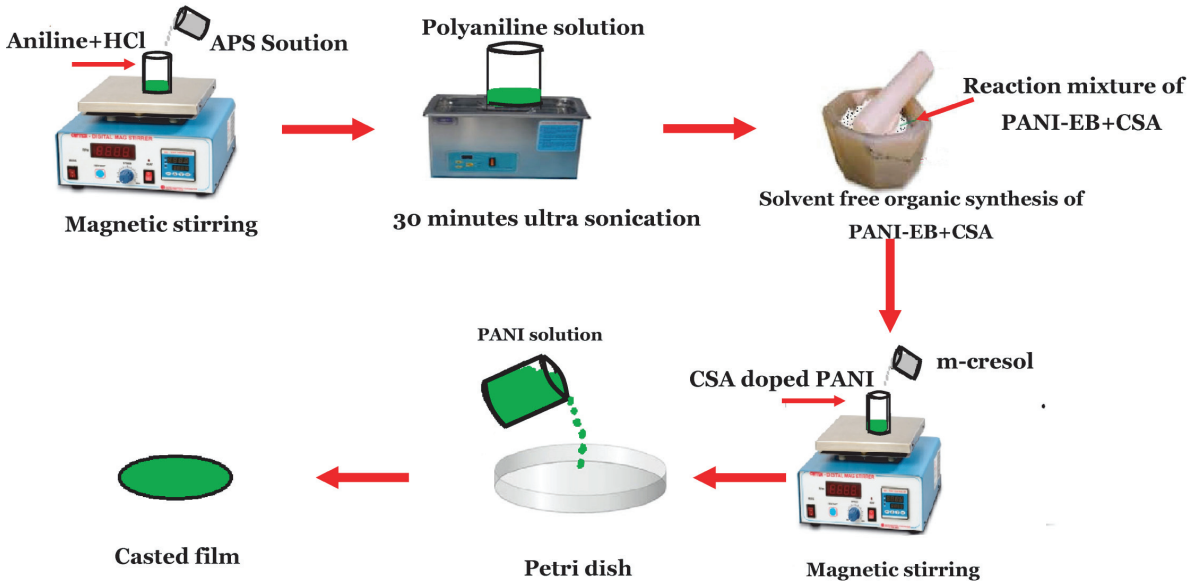


Figure 3. Film fabrication procedure.

with the phenyl groups in the back bone of PANI, and its hydroxyl group builds strong hydrogen bonding with amine group [21].

3. RESULTS AND DISCUSSIONS

The shielding effectiveness of the film is evaluated over various frequency bands and plotted in Figs. 4–6 for S, X and Ku bands. It is observed from Fig. 4 that the film made from PANI-CSA/m-cresol shows maximum SE of ~ 45 dB at X band. The sample have the thickness of 0.18 mm. The particular composite film is offering 45 dB of shielding on broad band frequency range in the X-band. The effect is probably due to the uniform dispersion, which causes homogeneous connectivity of the film composite. We have experimentally observed that the effect of secondary doping enhances the shielding ability of the sample as shown in the Fig. 4. The pelletized sample of PANI-CSA shows a very low SE of ~ 8 dB even though it has the highest thickness. Due to the loss of dopant molecules the pellatized samples always show a reduced conductivity, while applying pressure [39]. This leads to the low shielding efficiency of the sample. It is to be observed that PANI-CSA/m-cresol film which have a low thickness of ~ 0.05 mm provides SE of approximately 15 dB. When CSA doped Polyaniline is made into film using

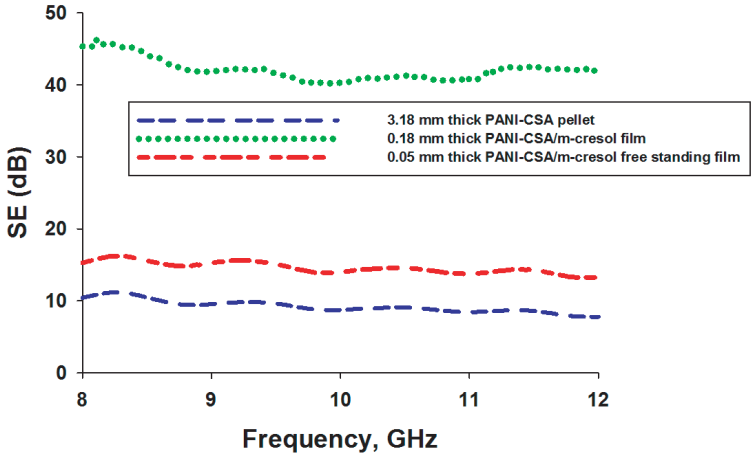


Figure 4. SE_{Total} of fabricated films and pellet of PANI-CSA/m-cresol at X band.

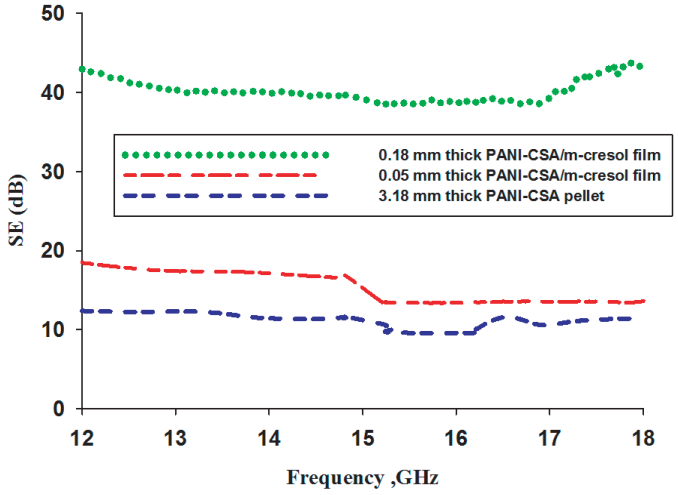


Figure 5. SE_{Total} of fabricated films and pellet of PANI-CSA/m-cresol at Ku band.

m-cresol, the flexibility of the polymer chain increases which inturn increases the dipolar polarization in PANI, which is contributed to the absorption of microwave. Also PANI becomes more conductive due to the protonation from CSA molecule which will lead to high charge delocalization in PANI, giving high conductivity and hence better shielding.

It can be clearly seen that in Fig. 5 PANI-CSA/m-cresol film with thickness of 0.18 mm gives $\sim$ 43 dB shielding at Ku band. The film of the same material with 0.12 mm thickness provides SE of $\sim$ 13 dB. The graph also shows that the effect of secondary doping is the same as that in other frequency bands.

It was difficult to fabricate a film suitable to cover the cross section of S band wave guide. Hence, we have coated the sample on a polyethylene sheet having a thickness of 0.05 mm. Fig. 6 shows that for a coating of PANI-CSA/m-cresol with thickness 0.07 mm, SE is $\sim$ 26 dB, while SE is $\sim$ 20 dB for a film with thickness of 0.04 mm. Table 1 gives a comparative study of the shielding efficiency of the proposed film with other PANI-CSA based films. Some reported materials of PANI-CSA which are used for EMI shielding applications, and their SE_T values are taken for comparison.

It is noticed in Fig. 7 that in PANI-CSA film the shielding due to absorbance is more than the shielding due to reflection, i.e., SE_A is dominant over SE_R. Probing in to the influence of SE against frequency, the SE_A is decreasing with increase in frequency while SE_R is increasing with frequency.

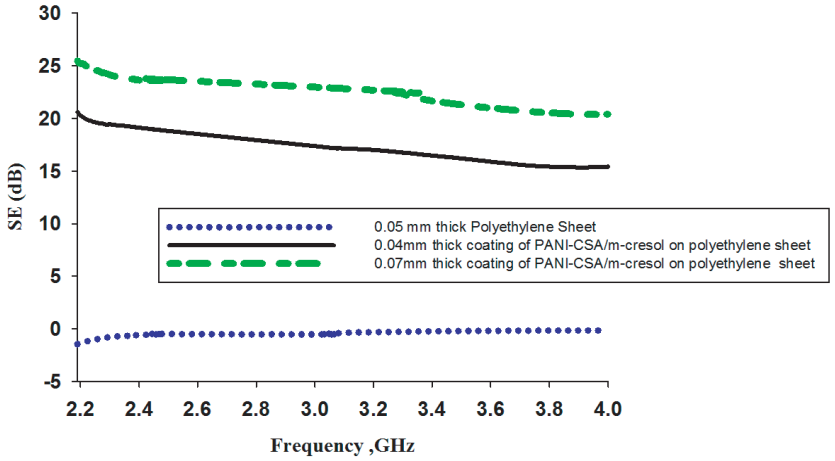


Figure 6. SE_{Total} of faricated films of PANI-CSA/m-cresol and polyethylene sheet at S band.

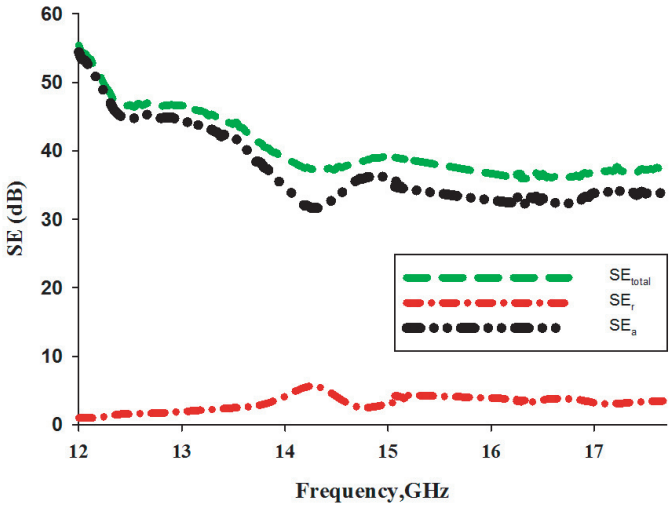


Figure 7. SE components of 0.18 mm thick PANI-CSA/m-cresol film at Ku band.

Table 1. Comparison of the proposed film with other PANI-CSA based films.

sl no.	Sample	Layer	Thickness (mm)	Frequency	Average Shielding (dB)	References
1	PANI-CSA /CMC film	single	0.3	1 GHz	14	[28]
2	PANI-CSA /GNR film	single	3.4	8–12	44	[40]
3	PVA/PANI-CSA /MWNT film	single	1	8.44 GHz	28(RL)*	[41]
4	PANI-CSA/PU	single	0.145	8–12 GHz	38	[42]
5	PANI-CSA /PU/Kapton	Multi	0.57	12–18 GHz	41	[42]
6	PANI/PU/CCo	Multi Multi	0.99 0.85	8–12 GHz 8–12 GHz	80 40	[43]
7	PANI-CSA /FeNi/PU	Multi Multi	0.97 0.93	12–18 GHz 12–18 GHz	80 40	[43]
8	PANI-CSA/PA	Single	0.1	10 GHz	23	[44]
9	PANI-CSA /Styrene acrylonitrile /fiber glass	Single	0.91	50 MHz–1 GHz	10	[45]
10	PANI-CSA/CMC /E-glass fabric	Single	0.15	1 GHz	10	[46]
11	PANI-CSA/PCP /E-glass fabric	Single	0.15	1 GHz	30	[46]
12	PANI-CSA/CMC	Single	0.6	1 GHz	19	[46]
13	Proposed film	Single	0.18	8–12 GHz	45	-

*Reflection loss

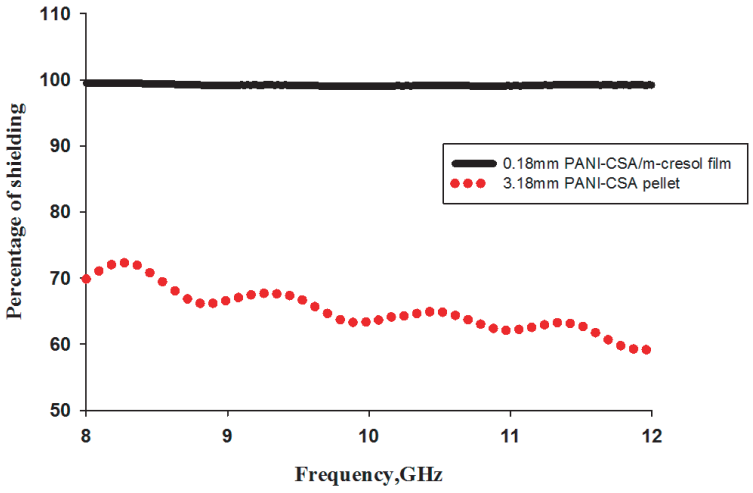


Figure 8. Percentage of shielding of PANI-CSA /m-cresol film at X band.

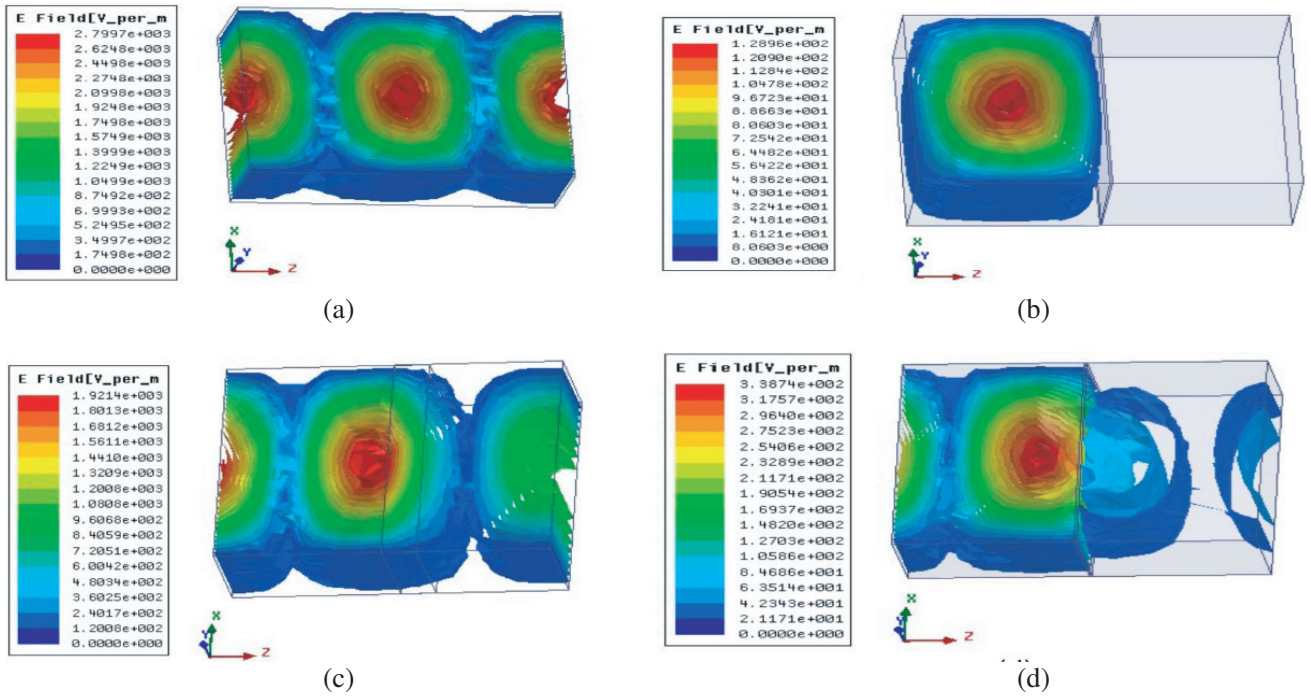


Figure 9. Electric field distributions ((a) Inside a standard wave guide, (b) copper film inserted between the waveguide adaptors, (c) PANI-CSA pellet inserted between the waveguide adaptors, (d) PANI-CSA/m-cresol film inserted between the waveguide adaptors).

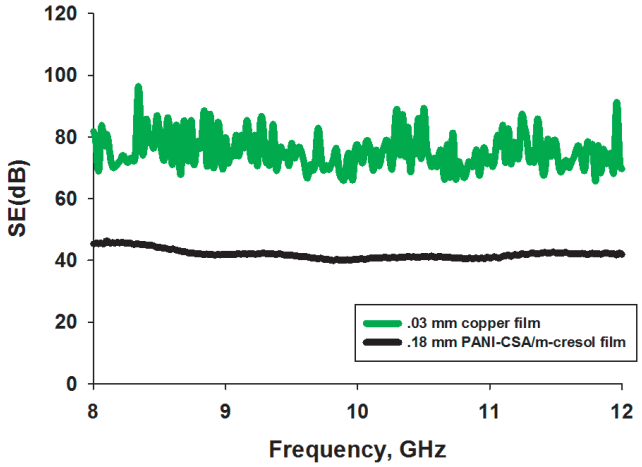


Figure 10. SE comparison with copper and PANI-CSA/m-cresol film.

In Fig. 8 it is noted that the pellet of PANI-CSA offers 70% of shielding with thickness 3.18 mm while the film made from m-cresol will give a constant shielding of approximately 99.9% with thickness 0.18 mm.

Figure 9 shows the simulation results of the wave guide measurement setup with metallic film, PANI film and PANI pellet at 12 GHz. Fig. 8(a) and Fig. 8(b) show the electric field distribution inside a standard wave guide and when a copper film is inserted between the wave guide adapters. When metal is used as a shield, 100% of the EM wave is shielded. Fig. 8(c) and Fig. 8(d) show good agreement with measurement results of PANI film and PANI pellet as shown in Fig. 8. PANI film can shield almost 99% of EM wave. It is observed from the Fig. 8 the pellet is not an effective EMI shield as that of film.

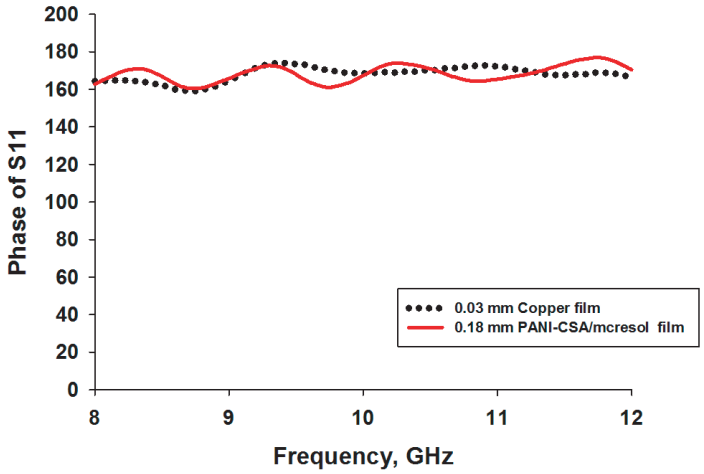


Figure 11. Phase comparison of PANI-CSA/m-cresol film with metal at X band.

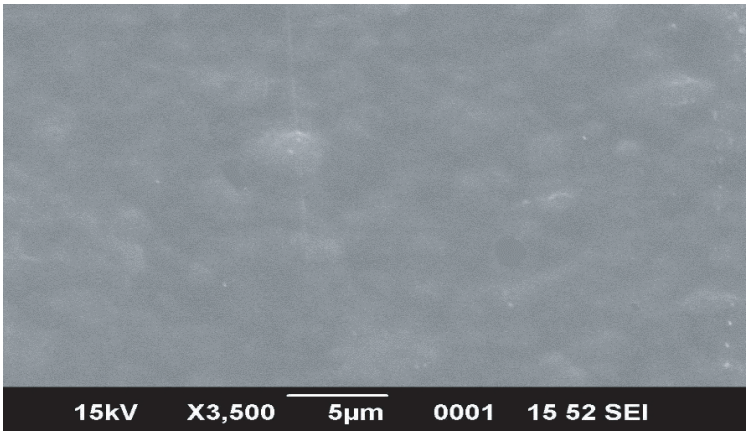


Figure 12. SEM image of PANI-CSA/m-cresol film.

In Fig. 10, the SE of PANI-CSA/m-cresol film is compared with the SE of a thin metal sheet of same cross sectional dimensions at X band. The phase of the reflection coefficient (S_{11}) plotted in Fig. 11 shows a value of approximately 160° over the entire range in X-band. This clearly indicates the suitability of the PANI-CSA/m-cresol sample as an alternate to ordinary metal plates.

The SEM image (Fig. 12) of the composite shows the uniform coating of PANI-CSA/m-cresol film over the substrate of a glass slide. The coating is done by drop casting method and the thickness of the film is approximately 20 µm measured by stylus profile meter.

4. CONCLUSION

In the present work, we fabricate a PANI-CSA/m-cresol thin film and evaluate its Electro Magnetic Interference Shielding Efficiency (EMI SE) in S, X and Ku Microwave frequency bands. From the detailed studies, we evaluate EMI SE of the film which gives 99.9% attenuation while the pellet of PANI-CSA offers a shielding of only 70%. The shielding of the composite is increased by the application of m-cresol; not only as a solvent but also as a secondary dopant. This composite provides nearly 45 dB of shielding in a broad band frequency range which is favorable for its use in commercial applications. It can be considered as a substitute to metal based techniques, which have drawbacks of corrosion, larger weight, etc. It is worth noticing that the low profile film (< 0.18 mm) can provide a uniform shielding against EM waves.

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