

Chemically synthesized Ag/PPy-PVA polymer nanocomposite films as potential EMI shielding material in X-band

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Abstract

Silver/Polypyrrole/Polyvinylalcohol polymer nanocomposite films were prepared by *in-situ* polymerization of pyrrole with variable loading of silver nanoparticles from 0.5-10%. The conducting films prepared from the nanocomposite solution were flexible, light weight, thermally stable and showed high hydrophobicity/hydrophilicity ratio. X-Ray diffraction measurement showed formation of *fcc* silver nanoparticles with particle size in the range of about 20-40 nm. UV-visible spectroscopy revealed the characteristic bands of Ag nanoparticles and polypyrrole in the so obtained co-polymer nanocomposites. The SEM studies of the nanocomposite films showed that the filler material was well conjugated in the Polymer matrix. Vector Network Analyser showed Electromagnetic shielding efficiency (EMI) efficiency as high as -35 dB in the X band (8-12GHz). Copyright © 2016 VBRI Press.

Keywords: Conducting polymer, polypyrrole, nano-silver, nanocomposites, EMI shielding.

Introduction

As the modern applied technology is moving from mechanical to electronic systems and devices, there is an exponential increase in the number of electronic equipments, instrumentation etc. This meteoric rise in electronic gadgets has resulted in tremendous increase in the microwaves around us. These microwaves associated with various electronic circuitries may couple and interfere with the normal functioning of other electronic gadgets near to them. This coupling and interference is known as Electromagnetic Interference (EMI). In order to reduce Electromagnetic interference caused by electronic equipments, various kinds of shields are used. Previously metals have been widely used to provide shielding because of their superior electrical, magnetic, mechanical and thermal properties but the drawback associated with metals was that they are very bulky in nature and most of them are prone to corrosion [1-2]. Over the past three decades, polymers have emerged as highly useful material in all walks of life and indeed EMI shielding based on polymers have also therefore attracted the attention of the researchers around the globe. Polymers are light weight, have good mechanical properties, highly processable and are corrosion resistant thus making them suitable as a replacement for metals in many applications. In order to qualify them for EMI shielding application, these polymers should have good electrical i.e. conducting behaviour as well as good magnetic properties i.e. high permittivity (conducting polymers). But, one of the drawbacks associated with conducting polymers is their

restricted processability [3-4]. It is well documented that polymer nanocomposites based on metal nanoparticles can improve thermal and electrical properties leading to much more superior properties of the nanocomposites than the polymer alone. Due to their improved electrical and magnetic properties, such nanocomposites have found application as EMI shielding materials [5-6].

One of the major challenges that lie in transforming normal polymers with such desired properties is the insertion of metal or metal oxide nanoparticles (NPs) or related materials in them [7-8]. There are various strategies adopted by the researchers to handle these challenges chemically or physically. The nano-fillers enhance the thermal, mechanical, electrical, magnetic etc. properties of the polymer and make the polymer nanocomposite fit for EMI shielding application [9]. The nano-fillers can be incorporated or embedded in the polymer matrix *in-situ* or can be added *ex-situ*. The fillers grown *in-situ* are believed to have better conjugation in the polymer network through various functional groups responsible for possible chemical bonding or simple physically held within the network through Van der waal's forces or electrostatic forces. These alterations in the polymer nanocomposites make them suitable for EMI application. The filler added *ex-situ* may have problems like separation due to settling because of density differences in the filler and the polymer material. Increasing Hydrophobicity of the Polymer nanocomposite makes it more resistant to moisture attack and increases its stability in open.

Shielding of microwaves occur due to reflection, absorption and internal reflection. The total shielding efficiency is the sum of all the three components. Reflection losses or minimum reflectivity is the ratio of Conductivity of material and its magnetic permeability. For moderately conducting materials permittivity also plays a significant role in reflection losses. In general: Increases with increase in conductivity and decrease in permeability. Reflection of E field and plane wave increases with a decrease in frequency and distance. Likewise, reflection of H field increases with an increase in frequency and distance. Absorption loss can happen due to ohmic and heat losses attenuation of the wave when an electromagnetic wave enters a system and a current is induced in the medium. It is a square root of permeability and conductivity of the material. A good absorbing material should possess high conductivity and high permeability, and sufficient thickness to achieve the required number of skin depths even at the lower frequency. Thus, it can be said that the absorption losses increase with the increase in thickness, permeability and conductivity of the material. Multiple internal reflections is dependent on the absorption losses, if the value of absorption losses is high then multiple internal reflection does not have a significant role to play.

Plane Wave SE (dB) = $10 \log_{10} (P_1/P_2)$ Reflection Loss
 >> Absorption loss

E - Field SE (dB) = $20 \log_{10} (E_1/E_2)$ Reflection Loss
 >>> Absorption loss

H - Field SE (dB) = $20 \log_{10} (H_1/H_2)$ Absorption
 Loss >> Reflection loss [10-13]

Conducting polymers e.g. Polyaniline, Polypyrrole (PPy) can add an extra benefit when used for EMI shielding applications. These conducting polymers have shielding efficiency in the range of -26 dB to -30 dB and due to higher conductivity can efficiently act as a shielding material however; their limited processability and mechanical strength restrict their applicability. Shielding efficiencies of polypyrrole is around -25dB with absorption being the dominating mechanism of shielding. So, the thicker films show better SE than the thin films in case of polymer nanocomposites made of conducting polymers. Blending of such polymers with suitable resin can improve the film formation but may decrease its shielding efficiency [11]. The thin films with varied thickness can be cast by simple solvent evaporation method at ambient temperature and pressure or under vacuum but thick films are difficult to obtain. Often, films with certain thickness are desired for EMI shielding application and the thicker films show better EMI shielding properties [14-17] and techniques like hot pressing (at high temperature and pressure) can be used. Thus, we have presented a simple solvent evaporation technique coupled with hot-press technique to fabricate thin-films of Ag NPs/PPy/Polyvinyl alcohol with varied thickness for effective EMI shielding. The concentration of Ag nanoparticles is also varied in the films to study its effect of EMI shielding efficiency.

Experimental

Methods and materials used

Silver Nitrate and Pyrrole (density 0.9976g/ml) was obtained from Sigma Aldrich Co India, De-ionized water was used where ever required, Poly Vinyl Alcohol (MW 1,25,000, degree of polymerization 1700-1800) used was obtained from Spectrochem India Ltd.. N,N'-dimethylformamide (DMF), Methanol and Chloroform were purchased commercially and were used for recording various solution mode spectra. All reagents were used as received. Poly vinyl alcohol was used as a base material. It was preferred because of its solubility in water, easy availability, cost factor, bio-degradability, bio-compatibility and most important good film forming ability. X-ray diffraction (XRD) pattern was recorded using Cu $K\alpha$ radiation ($\lambda = 0.1546$ nm) at a generated voltage of 40 kV and current of 120 mA at room temperature at scanning of $2^\circ/\text{min}$ between 2θ 10-90°. Fourier transform infrared (FTIR) spectra were obtained using a Perkin Elmer Spectrum Two infrared spectrometer using in the range of 4000 to 400 cm^{-1} . Scanning electron microscope (SEM) measurement was carried out using Bruker Advanced operated at 300kV. Thermogravimetric analysis (TGA) was measured on a Perkin Elmer TGA7 under nitrogen from 25°C to 850°C at a constant heating rate of 10°C/min. Ultraviolet-visible absorption spectra were recorded on a UV 210 UV/Vis Spectrophotometers Speccord 210 in the wavelength range of 200 nm-800 nm.

Synthesis

In-situ synthesis of silver/polypyrrole

Polypyrrole was *in-situ* synthesised using monomer pyrrole (density 0.9976 g/ml) in aqueous solution of polyvinyl alcohol (1%) prepared by dissolving 1.0g PVA into 100 ml pre-heated deionized water at 80°C and sonicated for 10 minutes. After sonication this solution was continuously stirred using a magnetic stirrer at about 70°C for an hour. A clear homogenous solution of PVA was obtained for further reaction as a matrix medium for synthesis of Ag/PPy which was typically synthesized in a one-step process as given below; Pyrrole (2.6 ml) was added to the PVA solution and AgNO_3 was added depending upon the concentration needed. Typically, for 1% loading of silver with respect to 1.0g of PVA 0.16 gm of silver nitrate was added to the solution. In-situ formed zero valent Ag (Ag^0) acted as a chain initiator for the polymerization of Pyrrole at a temperature of about 100°C. The formation of polypyrrole was marked by change in color of the reaction mixture from brownish to completely black. The reaction mixture was stirred for 2-3 hrs in order to ensure complete conversion of the pyrrole to Polypyrrole. The Ag^0 formed during the course of the reaction remain attached to the Polypyrrole chain leading to eventual formation of Ag/PPy-PVA nanocomposites

Thin film fabrication

Thin films were formed by simple solvent casting method. The reaction mixture was poured in to a mould of 0.5 mm thickness and allowed to dry for 24 hrs at room

temperature under vacuum. The dried film was peeled off from the mould.

Thick film fabrication

Thick films of 3mm thickness were fabricated by layer wise stacking of the thin films and hot pressing them using a hydraulic press at 100°C and 140 kg/cm² pressure for 20 minutes in a mould of fixed depth. The films so-obtained were thicker and less flexible than the films obtained by solvent casting (**Fig. 1**).



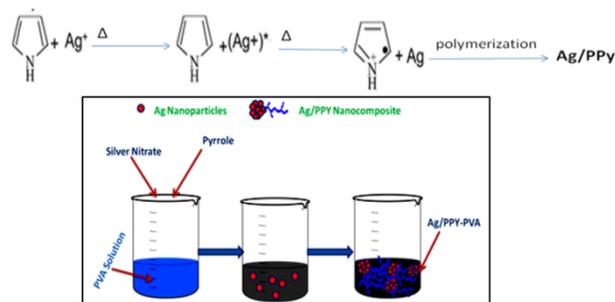
Fig. 1. Actual photographs of the samples (a) Thin film of Ag/PPy-PVA prepared in a glass petridish (b) Thin film prepared in a mould (c) Mould used for preparation of thin film (d) Thick film of Ag/PPy-PVA nanocomposites.

Results and discussion

Reaction of Pyrrole with silver nitrate has been earlier reported by our group [18]. Wherein, it has been described that Pyrrole acts as a reducing agent to silver nitrate thus leading to formation of silver nanoparticles which in presence of excess Pyrrole acts as a mild oxidizing agent to convert the Pyrrole monomer into Polypyrrole. Overall so-generated inorganic-polymer combination in presence of PVA solution leads to formation of Ag/PPy-PVA nanocomposite. The polymer blend along with presence of silver nanoparticles is a technologically effective material to block electromagnetic radiations. Since PVA itself is an effective polymer for EMI shielding application along with inorganic fillers [19], it prompted us to test its utility along with Ag/PPy for film making. Overall, formation of silver nanoparticles and polymerization of Pyrrole is depicted in **Scheme 1**. So-generated composition was monitored by UV-Visible spectroscopy to identify the formation of silver nanoparticles by recording surface plasmon resonance phenomenon.

The absorption spectrum of films with varying Silver loading 0.5 to 10% was recorded as solution (**Fig. 2**). Ag nanoparticles show surface plasmon resonance (SPR) in

the range of 380-500 nm depending on the morphology and size distribution of the particles [20].



Scheme 1. Pictorial processing and formation of Ag/PPy-PVA composite

In the present case, it is expected that SPR will be observed however, the same range is almost true for polypyrrole too. In view of this, natural overlap of the bands in UV-Visible spectrum is possible. Indeed, the sample a showed shallow band at around 435 nm and a clear band at about 460-63 nm. The characteristic absorption bands for Polypyrrole with the maxima at about 460 nm, 462 nm and 463 nm were found to vary only by about 1-2 nm with respect %Ag content in the matrix. The bands due to PPy match well with the characteristic peak of Ag/PPy [16]. The consistency in absorption value of the films indicates that the quality of composite is similar in all cases irrespective of concentration of the metal. The bipolaron band characteristic of polypyrrole is observed at about 435 nm which in the current sample seems to be overlapped with possible SPR of silver nanoparticles. Slight broadening in absorption pattern is probably due to wide-size particles distribution [21-22]. Size distribution was found to improve with higher silver content up to 2% and the film with least silver loading showed the most wide size distribution. However, 5% and 10%Ag containing films showed medium size distribution. Overall 2% silver content in PPy resulted in excellent absorption pattern.

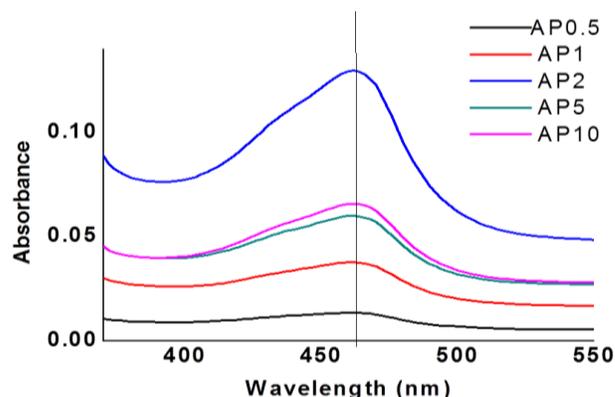


Fig. 2. UV-Visible spectra of Ag/PPy-PVA films dissolved in DMF

Fig. 3a shows X-Ray diffraction patterns of Ag/PPy-PVA Nanocomposites of different concentrations. The main diffraction peak for polypyrrole appeared at 2θ value of 19.59° which is matched with the value reported for polypyrrole in the literature [23-25]. The diffraction

peaks of silver are observed at 2θ values of 38.39, 44.65, 64.58, 77.11, and 81.38° correspond to (111), (200), (220) (311) and (222) crystallographic planes, respectively. These peaks match well with the standard value of XRD of face centred cubic crystal structure of silver metal. It is observed that the peak intensity is directly proportional to the % Ag loading in the polymer system. Size of the nanoparticles in the composite material was calculated using the Scherrer's formula taking FWHM at all planes and the average particle diameter was found to be in the range of 5.6, 7.89, 8.39 nm for 0.5 %, 5% and 10% respectively [26]. It is observed that diffraction peak at 19-23° is severely overlapped for PVA-PPy which closely matched with the reported values for PVA and PPy respectively [27].

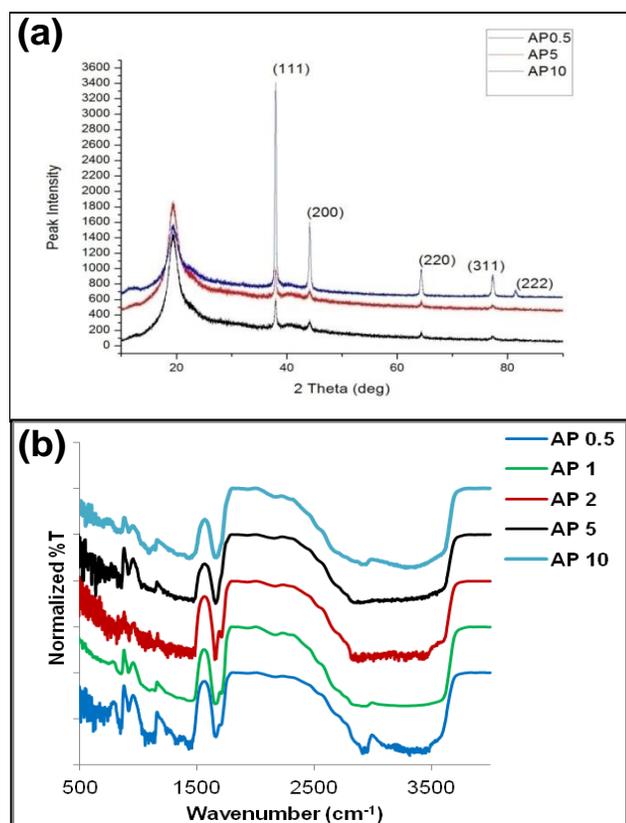


Fig. 3. (a) X-Ray diffraction studies of various Ag/PVA-PPy samples with different concentrations of Ag nanoparticles and (b) FTIR studies of Ag/PPy samples with different concentrations of Ag nanoparticles.

The crystalline nature of PVA was adversely affected with increase in silver content. Such a reduction in crystallinity of matrix may reduce brittleness in the film thereby making them more attractive for shielding application. Calculation of d spacing for PVA in the composite film (Ag/PVA-PPy) resulted in a value of 0.41 nm which is slightly lower than the reported value of 0.45 nm for crystalline PVA [27]. Such a reduction in the lattice spacing may be due to the loss of crystallinity and it hints towards the supporting role of this as a surrounding matrix for Ag/PPy which is also established by its physical appearance as a glossy film. Similarly, calculation of d spacing at 38° resulted in the value of 0.186 nm which matched well with the standard value normally reported for silver nanoparticles [28] (Table 1).

Table 1. Particle size and d -spacing of various Ag/PVA-PPy samples with different concentrations of Ag nanoparticles.

Sample	2 θ value/ d spacing		Particle size (nm)				
	PVA	Ag					
AP0.5	19.59/0.411	38.10/0.189	44.36	64.58	77.11	81.38	5.6
AP5	19.3068/0.4	38.39/0.186	44.65	64.58	77.00	81.2	7.89
AP10	19.59/0.411	38.39/0.186	44.36	64.58	77.11	81.38	8.39

The FTIR spectra of Ag/PPy-PVA films of concentrations 0.5 to 10% are presented in **Fig. 3b**. It was observed that all the peaks corresponding to the polymers are present and are found to be similar in all the composite composition. No significant variation with respect to silver content was observed. Peaks related to C-H bending and stretching are observed in the range of 1400-1600 cm^{-1} . Similarly, peaks at 2900 cm^{-1} region are for C-H stretching mode of vibrations. Further confirmation of PVA (contains O-H) and PPy (contain N-H group) was ascertained due to presence of peaks in the range of 3400-3500 cm^{-1} . The peak intensity in the spectra has reduced with increasing silver content which is only normal as the probability of bonding of functional groups with higher inorganic content is much lower due to availability of lesser number of functional groups thereby offering less coordinating sites for all the particles which will adversely affect the chemical interactions and thus intensity will reduce in addition to other observations. This situation will arise as the number of atoms of silver *vis-a-vis* availability of functional groups in combined polymers is not known in the present case [18, 29].

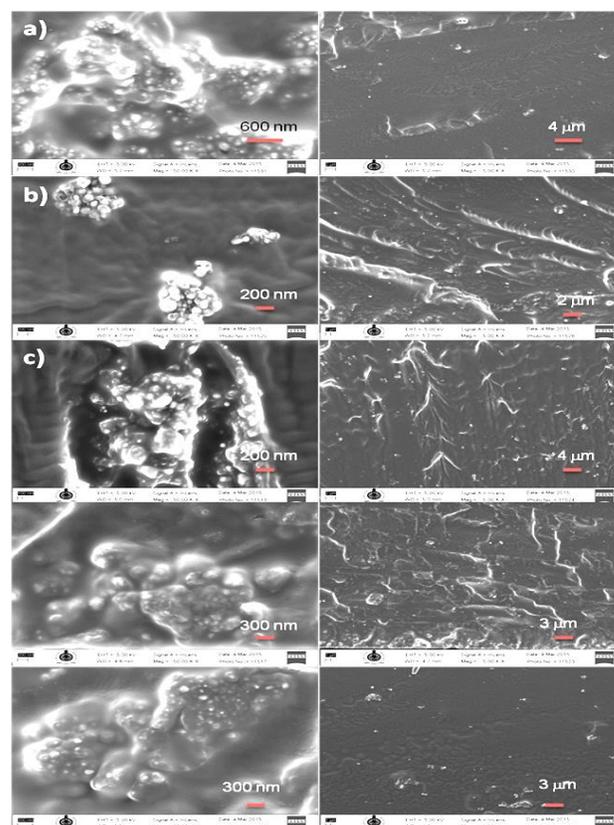


Fig. 4. SEM images of film containing various %Ag loading (a) 0.5%, scale bar 300nm and 2 micron (b) 1%, scale bar 100nm and 1 micron (c) 2%, scale bar 100nm and 2 micron (d) 5%, scale bar 100nm and 1 micron (e) 10%, scale bar 300nm and 1 micron.

Scanning Electron Microscopy results show the presence of nanoparticles and it also shows that as the agglomeration is increasing with Ag concentration (**Fig. 4**). From the SEM images one can see that at lower concentration of Ag (0.5%), the small particles are widely spread across the matrix. However, as the silver concentration is increased to 10%, agglomeration of particles was more prominent. Such an increase in agglomeration has been documented by several researchers [30] and is naturally quite obvious as the amount of matrix remain same which means holding capacity of the matrix remain similar to 0.5% loading of Ag. In other words, the increased concentration will not allow the particle to be properly embedded resulting in agglomeration. This could therefore be an obvious reason for inhomogeneous particle distribution and inaccurate measurement of electrical properties based on contact points. It is also observed that surface of each film under SEM shows random distribution with directional texture in film quality. In case of 2% silver loading this was seen as directional due to uniform particle distribution in same direction thus reducing the strain on the film. The cross section of the film during SEM was considered for the measurement of film thickness by measuring the width of the films and the same was found to be in the range of 0.1 to 0.18 mm depending on the area selected and % silver loading.

Fig. 5 shows the contact angle of water on the films. The contact angle studies give an idea of hydrophobicity to hydrophilicity of the substance. Increasing hydrophobicity is a desired property for prepared films. It was observed that with the increase in filler concentration the hydrophobicity of the films is increasing. The contact angle was observed to be in the range of 60 to 114.5° (**Table S1**).

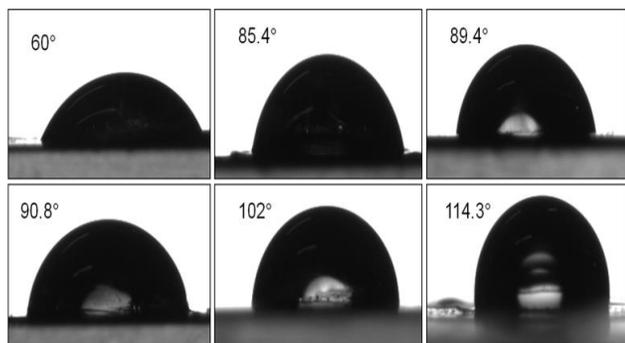


Fig. 5. Contact angle studies of the films at different %Ag (0.5%, 1%, 2%, 5%, 10%).

The increasing hydrophobicity shows that the film is becoming more moisture resistant and its stability in open environment has increased. This is a good property for any film to be used for protecting or shielding application as the environmental degradation can be effectively avoided and the films can last for longer period thus enhancing its commercial viability. The weight loss of the sample with increasing temperature was recorded with TGA analysis and is depicted in the **Fig. S2**. It was observed that there was sudden loss in weight at 310 °C and 338°C for the samples containing 10 and 1% Ag loading. The first stage decomposition was observed in

the range of 30-310°C which may be due to the loss of moisture content and decomposition of Poly vinyl alcohol along with initial degradation of polypyrrole. The second stage decomposition after 300 to 500°C is due to Polypyrrole in the presence of Silver. After 500°C the samples show a constant weight around 2.5% of its original weight which may be due to Ag and residual carbonaceous matter. TGA studies suggest that the polymer blend composite with silver as a filler has higher thermal stability than the PVA or PPy alone. It is document that PVA decomposes at about 100-150 and PPy (150-250) only slightly higher range of thermal degradation [31]. In the present finding it is observed that presence of 1% silver has lower thermal stability but still higher than the range document for these polymers. However, 10% loading has only marginally enhanced the thermal stability of the composite in comparison to 1%. Overall a four-stage decomposition profile is observed indicating degradation of various components in the composites.

Vector network analyzer studies of Ag/PPy films

For the studies of EMI shielding effectiveness the films were analysed using a vector network analyser. For this purpose, two experiments were performed i.e. 1) by increasing the loading of Ag and 2) by varying the thickness of the film from 1 to 3 mm. We believed that the absorption of the electromagnetic waves was the dominant mechanism of shielding in the various samples due to the large presence of PPy in the films. The Ag concentration in the films was varied between 0.5 to 10%. It was observed that increasing the silver concentration and the thickness from 1 to 3 mm, the shielding efficiency increased in all the samples. The values of EMI shielding effectiveness for various films are presented in **Table 2** and the plots are shown in **Fig. 6** (a & b; for 1 mm 3mm thick film with different silver concentration).

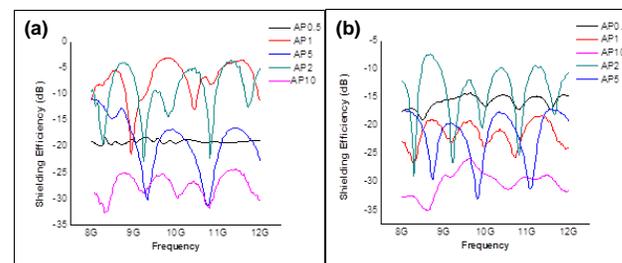


Fig. 6. Shielding efficiency of (a) 1mm thick films, (b) 3mm thick films with various concentrations of Ag nanoparticles.

For studies with 1 mm thick films of various concentration of silver, the films were prepared by stacking several (typically 10-12) solution cast thin films. However, films of 3 mm thickness were fabricated *via* compression moulding using hot pressing method. It was observed that the signals varied with method of film preparation without adversely affecting the values despite variation in method and thickness and was subject to EMI shielding vector analyser. The efficiency varied from -19 dB to -32dB depending upon the extent of silver loading in the films. The difference in dB values could be due to the difference in electrical properties and dielectric

constants of the films. However, irrespective of the particle size and distribution, final measurement of the film suggested an increasing trend of EMI shielding effectiveness with respect to %Ag loading. Similarly, 3 mm thick films were prepared by hot press compression moulding method which is considered a better technique for generation of uniform film with homogeneous particle distribution. The homogeneous Ag particle distribution will render more electrical conductivity in the films and EMI shielding is likely to be improved in comparison to solution cast film.

Table 2. Shielding Effectiveness of films.

% Ag	SE (dB) (maximum) 1mm thick film	SE (dB) (Average) 1mm thick film	SE (dB) (maximum) 3mm thick film	SE (dB) (Average) 3mm thick film
0.5	-19.04	-17.48	-19.94	-19.55
1.0	-21.68	-17.34	-26.56	-24.47
2.0	-22.83	-21.493	-28.86	-27.4
5.0	-32.49	-29.56	-33.027	-30.52
10.0	-30	-29.73	-35	-31.88

*EMI efficiency of neat PVA film was observed at -0.34dB

Indeed, it was observed that the trend remain same for solution cast film but the values are 10 % higher than 1 mm thick films. The higher values for 3 mm thick films are therefore considered due to two possible reasons, (i) homogeneous thickness as well as particle distribution within the films and (ii) due to thick non-porous surface of the film. Overall, it is observed that 2% Ag containing samples of 1mm or 3mm thickness show most consistent performance in terms of interaction with electromagnetic waves and their retaining excellent EMI shielding efficiency. The findings from the current experiments indicate that EMI shielding efficiency varies between -19 dB to -35 dB in case of 3 mm film however it was between -17 to -30 dB in case of 1mm thick film. The requirement in the X-band (8-12 GHz) region is normally reported to be in the range of -20 to -25 dB for commercial use. EMI shielding material has great demands not only in military application but also in communication technology. The as-prepared Ag/PPy/PVP films show good shielding effectiveness in the X-band which is required for Doppler, TV transmission, weather radar and telephone microwave relay systems [32]. Hence, we believe the as-prepared films may have good commercial utility due to their high SE and easy processing. Since PVA itself does not show any shielding against EMI, it is appropriate to conclude that Ag/PPy composite alone would be an option for extended utilization and our intention to use PVA stands vindicated for assisting formation of the Ag/PPy films. The variation in signals in various samples may be due to the change in degree of polymerization of Polypyrrole in the various samples as the silver loading was different in the samples.

Conclusion

In the present work, the EMI shielding properties of Silver-Polypyrrole Nanocomposite is prepared for its utility as EMI shielding material. Thin and thick films of the composite Ag/PPy along with Poly vinyl Alcohol (in

situ) were made and concentration variation was studied. Thin films were made using solvent casting method and thicker films were made by stacking and hot pressing the thin films. The UV-VIS, FTIR spectroscopy confirmed the formation of Ag/PPy composite which was grown in PVA matrix. The XRD studies confirmed the presence of silver in the films and the formation of Polypyrrole. Shielding effectiveness was measured using Vector Network Analyser which showed highly suitable range for EMI shielding application in X-band. The maximum shielding effectiveness observed was in the range of -30 dB to -35 dB for 5 and 10 % Ag loaded films while the average range for all the samples was 20 to 33 dB . The results obtained have shown that the material thus formulated is a potential EMI shielding material and can also be used for commercial applications due to ease of formation and processing.

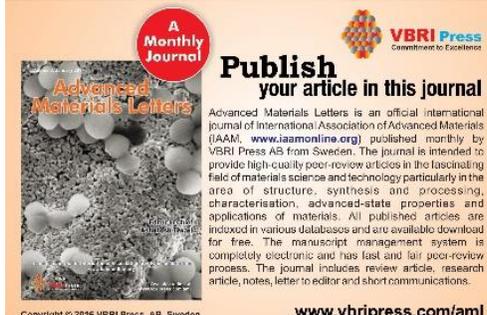
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Supporting Information

Table S1. Water drop contact angle of Ag/PPy-PVA composite film with varying % Ag.

% Loading of Silver	Contact Angle
0	60
0.5	85.4
1	89.4
2	90.8
5	102.4
10	114.3

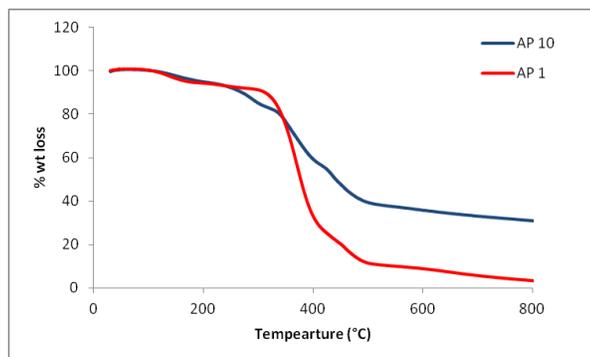


Fig. S2. TGA profile of Ag/PPy films with various Ag concentrations.