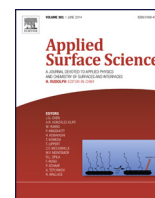




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# Studies on galvanostatically electropolymerised polypyrrole/polyaniline composite thin films on stainless steel

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## ABSTRACT

The polypyrrole/polyaniline composite thin films were prepared by a simple electrodeposition technique on stainless steel substrate. The FT-IR spectra confirm the formation of polypyrrole/polyaniline composite structure; the surface morphology showed that polypyrrole/polyaniline has cauliflower like structure in 90–10% composite whereas there is drastic change in morphology in the 50–50% composite. The microwave properties such as reflection, absorption, dielectric behavior of polypyrrole/polyaniline composite thin films with frequency were studied in the frequency range 13–18 GHz. The thickness, DC and microwave conductivity were also studied. The dielectric constant and microwave conductivity showed higher value in the 70–30% composite thin film.

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## 1. Introduction

In electronic industries there is a requirement of large area and smart surfaces at which the high frequency electromagnetic transmittance, reflectance can be controlled. This may be done by using the conducting polymers [1]. The conducting polymers have emerged as a new class of materials in the last few decades, because of their excellent electrical and mechanical properties such as high conductivity, electrical properties, ease of synthesis and ease of production etc.

Recently, conducting polymer composites have also received tremendous attention and it has become one of the most active and promising research areas [2]. These conducting polymer composites are attractive due to their potential applications in rechargeable batteries [3,4], shielding of electromagnetic interference [5], microwave and radar absorbing materials [6], sensors [7,8], electronic and bioelectronics components [9], membranes [10], electrochemical capacitors [11], electrochromic device [12], light-emitting devices [13], antistatic [14] and anticorrosion coatings [15], etc. Among the various conducting polymers, polypyrrole and polyaniline are the most stable polymers with good mechanical, electrical properties which can enhance the utility of stainless steel [16,17].

Stainless steel is a cost effective and useful material in the radio frequency and microwave region. It is used for the fabrication of

the antennas which is used in radio astronomy, remote sensing and communication purposes. The modification of the surface properties of stainless steel may lead to better performance of the antenna structure [18]. In this paper we report the properties of polypyrrole/polyaniline composite thin films deposited by electropolymerisation technique on stainless steel substrate.

## 2. Experimental

Pyrrrole monomer (Aldrich), aniline monomer (Aldrich), *p*-toluene sulfonic acid (PTSA) (Merck) was used as received, while water was distilled before use. Monomer solution was prepared by dissolving pyrrole (0.7 ml) and aniline (2.5 ml) in 100 ml distilled water and *p*-toluene sulfonic acid (1.735 gm) in 100 ml distilled water. Ratio of pyrrole and aniline was changed by (90–10)%, (70–30)% and (50–50)% with constant dopant concentration i.e. 5%.

The thin sheet of stainless steel (SS) of thickness ~0.5 mm was cut to the size of 1.1 × 2 cm. These dimensions were according to the size of the substrate holder of the microwave set up. Prior to the electrodeposition, the substrates were polished to a smooth surface finish using finer grades of polish papers, washed with soap solution and distilled water and dried under a hot air stream and wiped by lint less tissue paper. The electrodeposition setup consisted of working, counter and reference electrode attached to bakelite holder. A saturated calomel electrode was used as a reference electrode and high density and high surface area graphite rod was used as the counter electrode. The current density was 1.3 mA/cm. An adherent black colored composite film was deposited on the stainless steel (SS) substrate at the ratio (90–10)%. As aniline percentage

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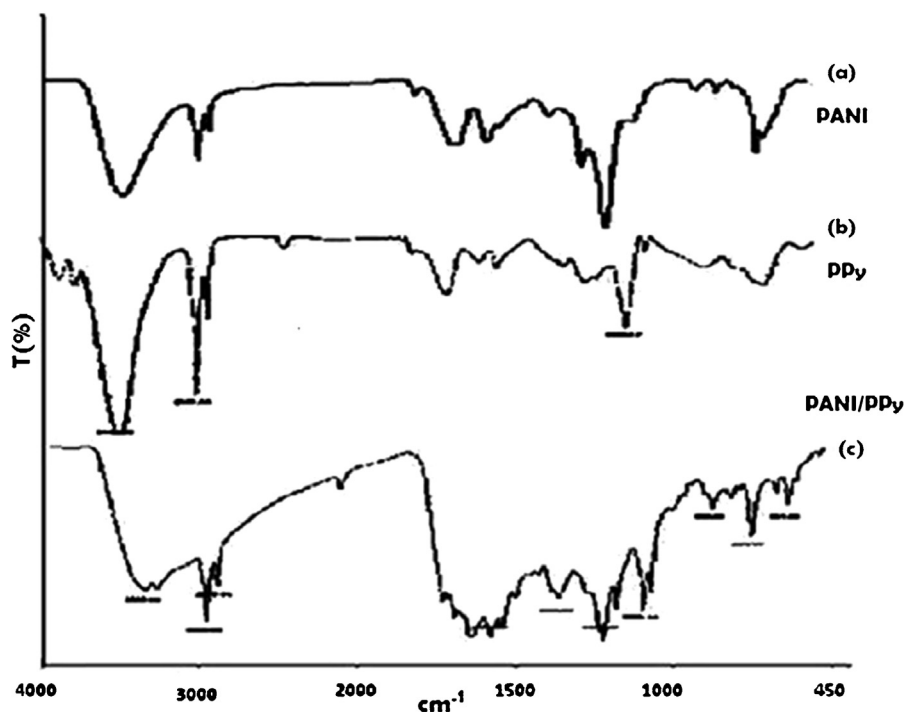


Fig. 1. Infrared spectrum of (a) PANI (b) PPy and (c) PPy/PANI composite thin films.

increased (70–30)% and (50–50)%, the color of thin film changed from black to bluish black. The thickness of polypyrrole thin films measured by using gravimetric weight difference method was varied by changing the time of deposition as 4, 8, 12 and 16 min, The Ku band (13–18 GHz) waveguide reflectometer and VSWR slotted sections methods was used for the measurements of absorption and dielectric constant [19].

### 3. Results and conclusions

#### 3.1. FT-IR spectroscopy

The FTIR spectrum of polypyrrole (PPy), polyaniline (PANI) and polypyrrole–polyaniline (PPy/PANI) composite thin film was recorded between 4000 and 450  $\text{cm}^{-1}$  at a spectral resolution of 2  $\text{cm}^{-1}$  on a Perkin–Elmer 1710 spectrophotometer which is shown in Fig. 1(a–c), respectively. All the major peaks of polyaniline are seen in Fig. 1(a). In PANI the strong characteristic peaks of benzoid ring, quinoid ring and N–H group are attributed at 1495  $\text{cm}^{-1}$ , 1528.23  $\text{cm}^{-1}$  and 3323.9  $\text{cm}^{-1}$ , respectively. N–H bending is located at 1308.83  $\text{cm}^{-1}$  while C=C stretching vibration is observed at 1482  $\text{cm}^{-1}$  and C–N stretching is occurred at 1460  $\text{cm}^{-1}$ . This confirms the formation of polyaniline structure. The characteristic peaks of N–H stretching, C–H stretching, C=C stretching are observed at 3323.9  $\text{cm}^{-1}$ , 2923.88  $\text{cm}^{-1}$ , and 1612  $\text{cm}^{-1}$ , respectively, of polypyrrole are observed in Fig. 1(b). The C–H vibrations occur at 1374  $\text{cm}^{-1}$  and at 802  $\text{cm}^{-1}$  C–H out of plane bending vibrations is occurred. The plane deformation is occurred at 1033.44  $\text{cm}^{-1}$ . The peaks at 1160.12  $\text{cm}^{-1}$  corresponds to C–C stretching [20–22]. The presence of these absorption peaks confirms the formation of polypyrrole structure.

The IR spectrum of PPy/PANI composite thin film is shown in Fig. 1(c). However, there is slight difference in the intensity and position of the peaks could be observed in the composite. In comparison with the polypyrrole (PPy) the characteristic peak of C–H out of bending vibration is shifted to 814.56  $\text{cm}^{-1}$  at higher

frequency region in composite whereas it is observed at observed at 802.14  $\text{cm}^{-1}$  in PPy. The shifting of peak is the result of the changes of chemical environment and this is due to the existence of PANI in the composite. The characteristics bands at 1482  $\text{cm}^{-1}$  and 1308.83  $\text{cm}^{-1}$  corresponding to the peaks of PANI with C=C stretching and N–H bending vibration, respectively [23]. It should be pointed out that from IR spectrum of PPy and PANI; the brought band at 3323.90  $\text{cm}^{-1}$  of amine groups corresponding to N–H stretching mode of PANI were overlapped with PPy. The peak observed in the region 2000–2100  $\text{cm}^{-1}$  is of typically the conducting form of the PANI film. It is suggested that application of PANI fibers would allow the PPy monomers to have more site selective interaction with PANI increasing the effective degree of electron delocalization and enhancing the conductivity of polymer chains. The quinone and benzene ring stretching deformation are observed at peak at 1528.20  $\text{cm}^{-1}$  and 1495  $\text{cm}^{-1}$ , respectively. Compared with PPy and PANI the intensity of the peaks in the composite IR spectrum decreases this is due to the interaction between  $\pi$  bond in the aromatic rings of polyaniline and polypyrrole would occur [28]. The band at 1460  $\text{cm}^{-1}$  and 1160.92  $\text{cm}^{-1}$  is corresponds to C–N stretching of PPy ring and C–C PPy stretching. The in plane deformation is found at 1033.44  $\text{cm}^{-1}$ . The formation of composite made an effective change in the molecular structure. From Table 2 it is clear that almost all the molecular groups seen in PANI and PPy are also present in the composite structure.

#### 3.2. Scanning electron microscopy

The JSM-6360 JEOL, Japan scanning electron microscope was used to study surface morphology of the samples. The surface morphology of pure PPy, pure PANI and PPy/PANI composite thin films with different concentrations is as shown in Fig. 2(a–e). From the figure it is seen that, the pure PANI has fibril like morphology and the pure PPy has cauliflower like morphology, as the concentration or the percentage of PANI in the in the PPy increases the surface morphology changes from cauliflower to fibril. This may

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