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Synthesis and Microwave Absorption Property of Cr Doped Poly-aniline

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Abstract: - Among the conducting polymer polyaniline (PANI) is an important polymer because of its high conductivity in doped state. It exhibits electronic, optical, magnetic properties which lead to its applications in a wide range of areas like photovoltaic cells, light emitting diode, biosensors, electronic sensors, electromagnetic radiation absorbers and electromagnetic shields. In this study Cr was doped in polyanline (PANI) by chemical oxidation method. Microwave absorption study showed above 80% absorption in 8.5 to 10.5 GHz frequency range due to doping of Cr in polyaninline (PANI).

Keywords: ***Polyaniline, Chemical oxidation, Microwave absorption***

I. INTRODUCTION

Microwave absorbers are in use both in civil and military applications on account of their ability to eliminate electromagnetic wave pollution and to reduce RADAR signatures. Radar absorbing materials or RAM is coated on the target that's electrical and magnetic properties have to be altered to give absorption of microwave energy at broadband or discrete frequency. The demands for various kinds of microwaves absorber has increased in the frequency range of 1-24 GHz because of their two fold use such as EMI shielding and counter measure to radar detection. Stealth technology for the defense in the area where these microwaves are employed for effective counter measures against radar surveillance. Application of microwave absorbing coatings on the exterior surface of military aircraft and vehicles also help in avoiding the detection by radar.

Absorbing materials should have dielectric, magnetic and conductive losses because the manner by which these properties change with frequency reflects the performance of the absorber. Ferrites are the only kind of material discovered so far, which shows some absorptive characteristics. These materials are semi-conducting ferromagnetic materials possessing high electrical resistivity with a good dielectric property and hence desirable absorbing materials for application at microwave frequencies.

Sharon etl [1] lately observed that even nanocarbon made out of camphor can absorb microwave about 90 % to 95 % (-24 dB). The ratio of reflected signals between aluminum plate and camphoric pellet was found to 1700/100 mV. They further studied the microwave absorption in the range of 8 GHz to 12 GHz and reported the absorption by carbon nanobeads to the extent of -20 dB [2]. Recently polyaniline (PANI) is a promising absorbing material due to its simple and easy way of synthesis, low density, controllable conductivity and multi-morphologies [3]. The study on higher wave absorbing performance of polyaniline and its composite materials are now one of the main research directions in the study of polyaniline. Polyaniline/graphite composites and found that the highest total electromagnetic interference shielding effectiveness of the composites in the X band (8.2-12.4 GHz) is -33.6 dB [4]. Core-shell PANI/carbon black (CB) nanocomposite and found that PANI / CB can possess a wide range of absorption frequency by adding different contents of CB in PANI [5]. Polyaniline (PANI) / montmorillonite (MMT) 10 % had a better microwave absorbing performance than PANI. The minimum reflection loss of PANI/MMT10%/paraffin composites with thickness of 8.0 mm were -37 dB at 14.8 GHz and the effective absorption band under -10 dB was from 13 to 16 GHz [6]. PANI / HA / TiO_2 / SWNT with 20 % of SWNT exhibits the best microwave absorption property (∼ 99.2 % absorption) with reflection loss of -21.7 dB at 6 GHz [7].

In view of this, we have investigated the microwave absorbing properties of Cr doped PANI. The results are reported herein.

II. EXPERIMENTAL

Materials and Methods

The chemicals required for synthesis *viz.*, aniline, ammonium per sulfate, $CrCl_3$ of analytical grade (AR) were purchased from commercial sources (S D Fine or SRL). Electronic spectra were recorded in methanol on a Jasco UV-Vis spectrometer model V-630 PC. IR spectra were recorded on a JASCO FT IR-6100 spectrometer.

Synthesis of materials

Synthesis of PANI [8, 9]

PANI was synthesized at lower temperatures (0-5 °C). Aniline (4.9 ml, 5.0 g, 0.054 M) was dissolved in 70 ml of HCl (1 M solution). This solution was kept in ice bath maintaining the temperature between 0-5 ºC. To this pre cooled (0-5 ºC) solution, ammonium peroxydisulfate [$(NH_4)_2S_2O_8$] (12.35 g, 0.054 M) dissolved in 70 ml of HCl (1 M solution) was added slowly with stirring. As the reaction proceeds within 2-3 min the intense blue green suspension appeared. The reaction mixture was stirred at room temperature for 2 h. After 2 h the precipitate was filtered over Buchner funnel and washed with deionized water until the colorless filtrate was observed. Later the precipitate was dried under suction remove the solvent water substantially to get cracked cake. The black precipitate obtained was dried *in vacuo* at 70 °C for 1 h to obtain black colored powder.

Synthesis of PANI doped with Cr

$CrCl_3$ (6.008 g, 0.15 mol) was dissolved in 150 ml of HCl (1 M) and cooled at 0 ºC which is treated as solution S1. 75 ml of solution S1 was mixed with pre cooled solution (at 0 ºC) of aniline (10 ml, 10.2 g, 0.109 M). This solution is treated as solution S2. Ammonium peroxydisulfate [$(NH_4)_2S_2O_8$] (24.69 g, 0.052 M) was added slowly with stirring to remaining solution S1 (75 ml). This solution is treated as solution S3. The solution S3 is added to solution S2. The reaction mixture was stirred for 2 h and kept for 24 h at 0 °C. It leads to formation of an intense blue green colored suspension. Later on the suspension was filtered by a Buchner funnel. The obtained precipitate was washed with demonized water till the filtrate become colorless. The precipitate was dried under suction to get the cracked cake. Then it was dried again completely *in vacuo* at 70 ºC for 1 h to give dark green compound.

III. RESULTS AND DISCUSSIONS

FT-IR analysis

The synthesized PANI based materials have poor solubility in water as well as organic solvents like chloroform, ethanol and even in dimethyl sulfoxide. Also they were amorphous as observed from for powder XRD patterns. Hence mainly FTIR is helpful for characterization of materials.

The bands around 1590 and 1510 cm^{-1} correspond to quinoid and benzenoid ring stretching vibrations respectively. The absorption band at 1310 cm^{-1} in the spectrum corresponds to the C-N stretching vibration of a secondary aromatic amine. The band at 1167 cm^{-1} is attributable to C-H bending vibrations and band at 832 cm^{-1} for C-H out-of-plane bending vibration in the 1,4-disubstituted ring. These absorption frequencies for the materials synthesized are in good agreements with the reported frequencies. The FTIR spectra are given below in Fig. 1. Similar bands were observed for Cr doped PANI with slight decrease in intensity, shifting and broadening of bands indicates the presence of Cr in PANI.

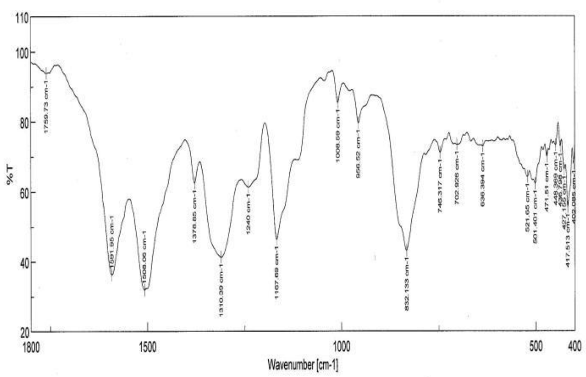

Fig. 1a FTIR spectrum of PANI

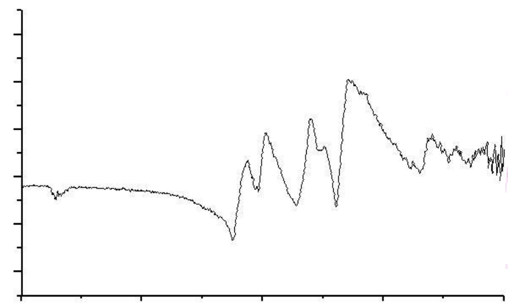

Fig. 1b FTIR Cr Doped PANI

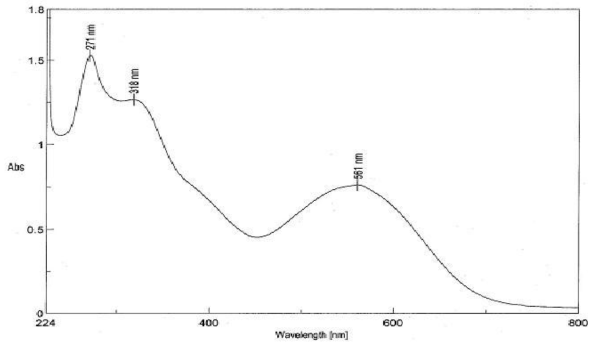

Fig. 2a UV-Vis spectra of PANI-BASE

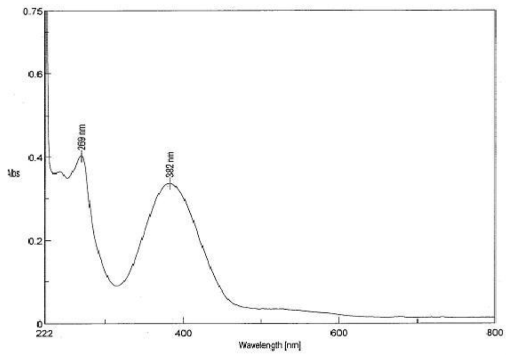

Fig. 2b UV-Vis spectra of Cr doped PANI in CH_2Cl_2

V-Vis absorption studies

It is in agreement of the report that PANI showed two strong absorptions at 320-340 and 600-660 nm. The PANI-doped materials exhibited strong absorption bands at ~ 269 and ~ 380 nm (Fig. 2b). The region of ~320 nm showed the π-π^* transition.

XRD studies

XRD pattern of Polyaniline is amorphous but Cr doped polyaniline is crystalline structure. XRD study confirms the presence of CrCl3 in polyaniline matrix.

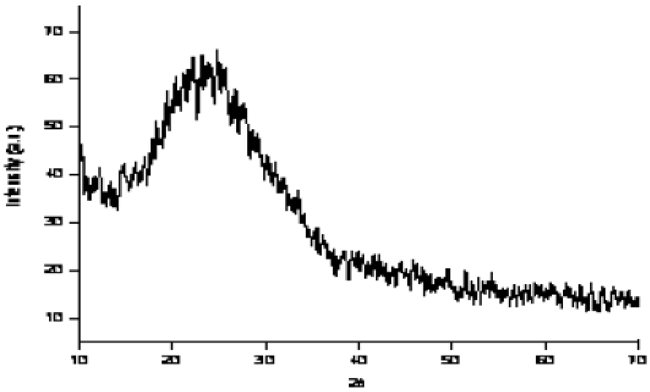

Fig. 3a XRD of PANI

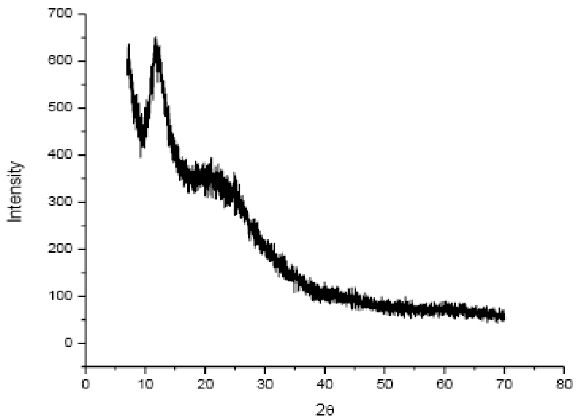

Fig. 3a XRD of Cr doped PANI

Microwave Absorbing Properties of PANI and Cr doped PANI

Microwave absorption properties of the PANI and Cr doped PANI pallet of thickness 0.2mm studied with microwave test bench in 8–12 GHz range, explored its facility as an absorber in X band of microwave. Microwave absorption study showed absorption in the range of~0.80–0.99 in 8.5 to 10.5 GHz frequency range due to doping of Cr in polyaninline (PANI). The exactly opposite trend for absorption between PANI and Cr doped PANI was observed in 8 to 11.5 GHz frequency range.

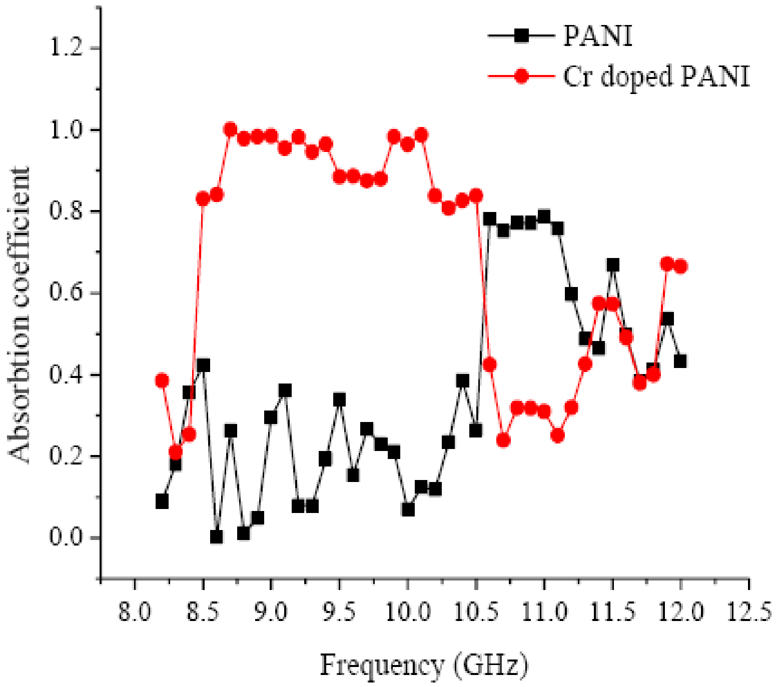

Fig. 4 Microwave absorbing properties of PANI and Cr doped PANI

IV. CONCLUSIONS

Microwave absorption study showed above 80% absorption in 8.5 to 10.5 GHz frequency range due to doping of Cr in polyaniline (PANI).

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