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3/4-HYDROXY BENZOIC ACID DOPED POLYANILINE: SYNTHESIS AND CHARACTERIZATION

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ABSTRACT: -

Doping of polyaniline (PANi) by any inorganic and organic compounds increases or improves the conducting as well as gas sensing properties .In this work we have prepared the polyaniline composites by doping 3-hydroxy benzoic acid and 4-hydroxy benzoic acid. The composites were characterized by FTIR and XRD.

KEYWORDS: - *PANi, Doping, Composites, 3/4-hydroxy benzoic acid.*

INTRODUCTION: -

Polyaniline is one of the most important conducting polymers, which may be used as active component of organic light weight batteries, microelectronics, optical display, for anticorrosive protection, in bioanalysis, etc., due to its good electrical and optical properties as well as high environmental stability. Due to the fact that polyaniline (PANi) is one of the most extensively studied and described conjugated polymer [1-3]. Conjugated organic polymers are either electrical insulators or semiconductors. These polymers can become highly electrically conductive after carrying out a structural modification process called "doping". Doping can be simply regarded as theinsertion or ejection of electrons. Doping process results in dramatic changes in theelectronic, electrical, magnetic, optical and structural properties of the polymers.It is reported that the structural properties of conducting polymers can be modified conveniently by adding metals [4], metal oxides[5, 6], organic molecules [7] or carbon nanotubes [8], which in many case improve the specificity.Polyaniline is commonly synthesized by oxidizing aniline monomer under strongly acidic conditions (usually in 1M H_2SO_4 or1N HCl) at $\sim 0^0$ C using ammonium persulfate as an initiator of oxidative polymerization.

EXPERIMENTAL: -

Material: -The materials used in the present study aniline monomer (99.5% pure), ammonium peroxydisulphate (APS), and ortho and para-hydroxy benzoic acid from s.d.fine chem. Limited Bombay. Chloroform and hydrochloric acid were purchased from a chemical company and they were used as received without further purification process. Aniline used after distillation for synthesis of polyaniline**.**

STRUCTURAL CHARACTERIZATION

FTIR analysis

Fourier transform infrared (FTIR) spectroscopy (Model: Perkin Elmer 100) of PANi (EB) was studied in the frequency range of 400–4000 cm-1

XRD-analysis

X-ray diffraction (XRD) studies were carried out in 2_ range of 100 –70° usingan X-ray diffractometer (Model: PhilipsPW3710).

Synthesis of PANi:

In conventional synthesis method, with the help of transfer pipette 2 ml (20mmol) aniline and magnetic needle into 50 ml beaker (beaker A) was dissolved in 10 mL hydrochloric acid (1M) and stirred with magnetic stirrer for one hour. 4.57 gm (20mmol) ammonium persulfate and magnetic needle in 50 ml beaker (beaker B) was also dissolved in 10 mL hydrochloric acid (1M) and magnetically stirred for one hour. Then the ammonium persulfate solution (beaker) was added at the rate of one drop/second into the aniline solution (beaker A) at temperature of about 0 – 4^0C and was left at this temperature for 6 hours. Finally, the black green precipitate was stirred for 24 hours collected and washed several times by acetone and distilled water. The pure polyaniline was then dried at ambient temperature 40 ^{o} C for 24 hours.

Synthesis of 3-hydroxy benzoic acid doped polyaniline

2 gm of polyaniline was taken in a 10 ml deionized water in 100ml R.B.F. containing magnetic needle. Then 0.1 gm of 3-hydroxy benzoic acid added in it and stirred it well. The stirring was continued for 3 hours. The resulting solution was filtered by whatman paper no. 41 and washed with water and acetone. The resulting PANi-Acid composite was dried under UV lamp for 2 hours and then dried under vacuum for 4 hours. Same procedure was used for preparation of 4-hydroxy benzoic acid-polyaniline composite.

RESULT AND DISCUSSION:

Synthesis PANi

FTIR (Fig-1)

The bands at 1572 and 1489cm^{-1} are attributed to the C=N and C=Cstretching mode of vibration for the quinonoid and benzenoid units of polyaniline. The peaks at 1296 and 1239cm^{-1} are assigned to the C-N stretching mode of benzenoid ring. The peak at 1239cm^{-1} is characteristic of the conducting protonated form of polyaniline. The bands in the region 1000-1115cm^{-1} are due to in-plane bending vibration of C-H mode. The band at 797cm^{-1} originates from out-of-plane C-H bending vibration.

Synthesis of 3-hydroxy benzoic acid- polyaniline composite.

FTIR(Fig-2)

The band at 1687cm^{-1} corresponding acid carbonyl (C=O) and 1544cm^{-1}were attributed to C=C stretching in aromatic nuclei.The bands obtained at 1600-1500cm^{-1} corresponds to C-H stretching in aromatic compounds. The absorption bands at 1480, 1425 cm^{-1} are due to C=N stretching in aromatic compounds. The composite shows absorption band in the range 1300-1100 cm^{-1} which confirms the C-N stretching of primary aromatic amines. The absorption band at 1289 and 1253 cm^{-1} reveals the C-H bending vibrations. The absorption bands bellow 1248 cm^{-1} are characteristics of mono substituted benzene. The absorption band at 1532 cm^{-1} is assigned to qunonoid structure does not revealed any significant changes for composite.

Synthesis of 4-hydroxy benzoic acid- polyaniline composite.

FTIR (Fig-3)

The band at 1668 cm-1 corresponds to acid carbonyl (C=O) and 1557 cm^{-1} were attributed to C=C stretching in aromatic compounds. The absorption bands at 1444 and 1468 cm^{-1}shows C-N stretching in aromatic compounds. The absorption band at 1288 and 1234 cm^{-1}reveals the C-H bending vibrations. The absorption bands below 1000 cm^{-1} are characteristics of mono substituted benzene. The absorption band at 1557 cm^{-1} is assigned to qunonoid structure does not revealed any significant changes for composite.

Fig-1 FTIR-Polyaniline

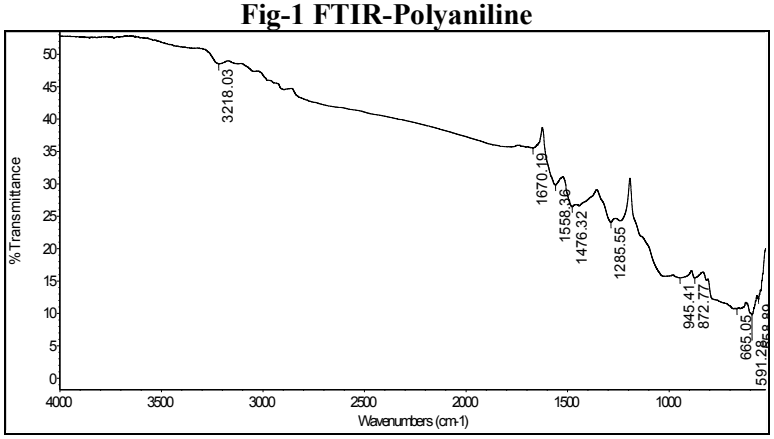


Fig-2-FTIR of 3-hydroxy benzoic acid doped polyaniline

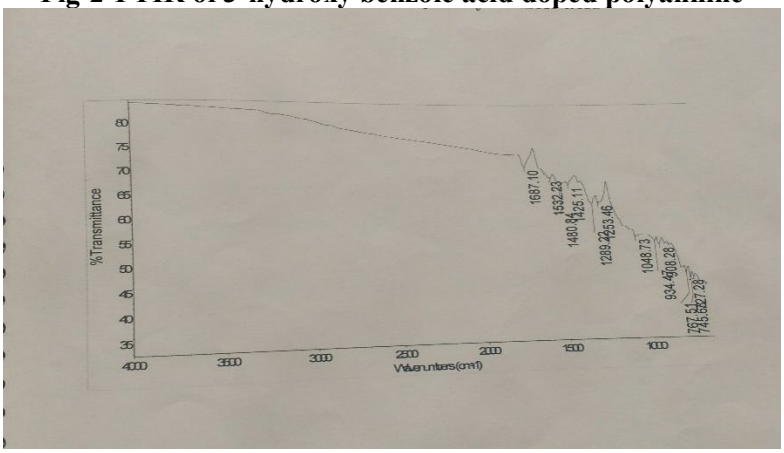
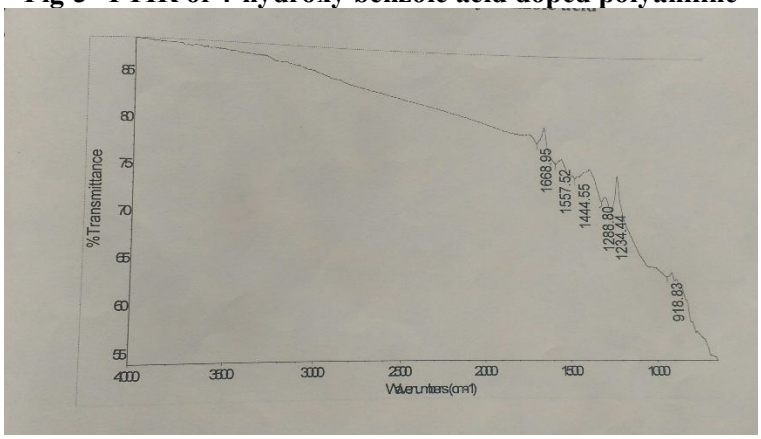


Fig-3--FTIR of 4-hydroxy benzoic acid doped polyaniline



X-ray diffraction analysis.

Polyaniline (Fig-4)

XRD of polyaniline broader peaks between $2\theta = 19^0$-35^0 indicate that the synthesized polyaniline is semi-crystalline similar to results obtained elsewhere (9). The crystallinity of PANi can be ascribed to the repetition of benzenoid and quinoid rings in PANichains,

Synthesis of 3-hydroxy benzoic acid- polyaniline composite.(Fig-5)

Presence of sharper peaks between $2\theta = 19^0$-35^0indicate that the synthesized 3-hydroxy benzoic acid –PANI composite is a crystalline material.

Synthesis of 4-hydroxy benzoic acid- polyaniline composite.(Fig-6)

Presence of sharper peaks between $2\theta = 19^0$-35^0indicate that the synthesized 4-hydroxy benzoic acid –PANI composite is a crystalline material.

Fig-4- XRD –Polyaniline

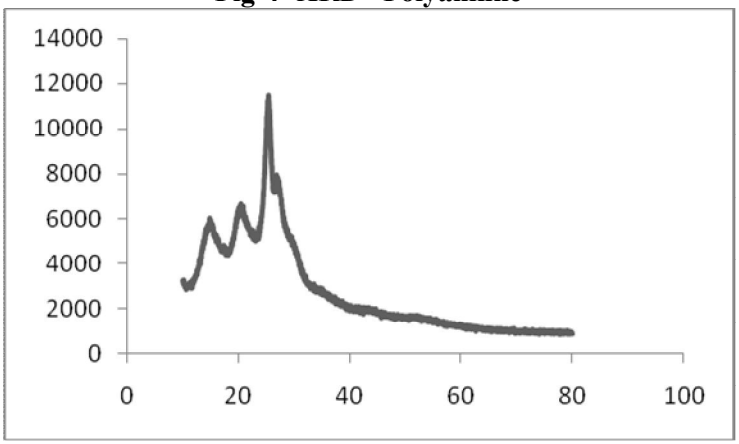


Fig-5-XRD of 3-hydroxy benzoic acid doped polyaniline

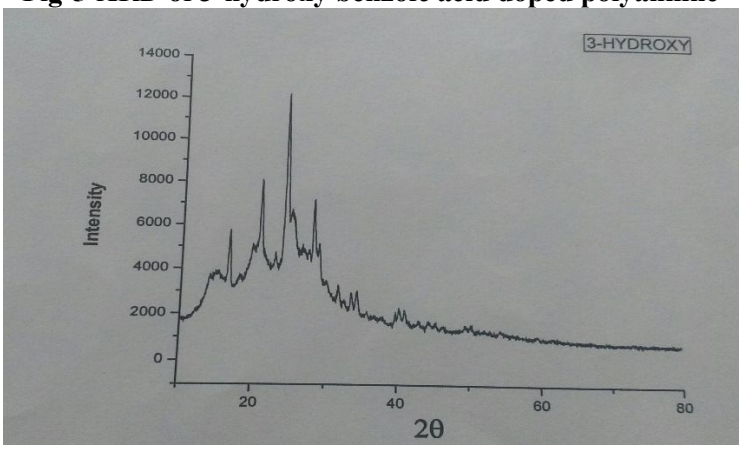
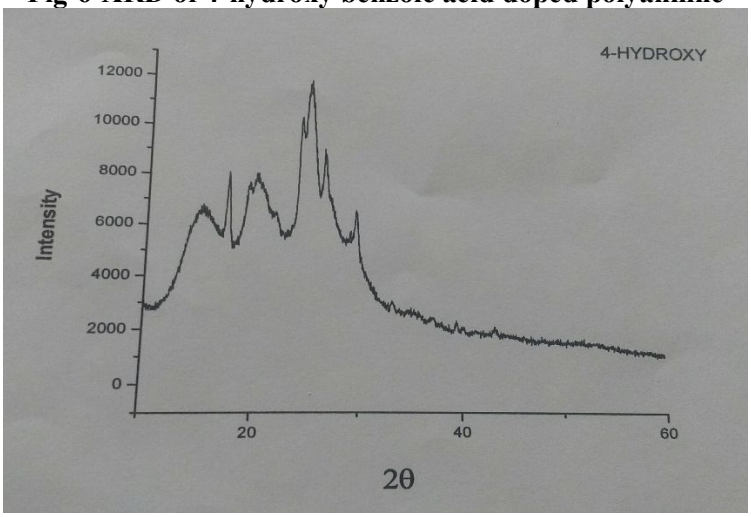


Fig-6-XRD of 4-hydroxy benzoic acid doped polyaniline



CONCLUSION:

We have successfully prepared 3-hydroxy benzoic acid and 4-hydroxy benzoic acid - polyaniline composites.These composites are characterized by FTIR and X-diffraction analysis. FTIR and XRD show that polyaniline is doped by 3-hydroxy benzoic acid and 4-hydroxy benzoic acid. Extent of doping is more for 3-hydroxy benzoic acid than 4-hydroxy benzoic acid. This may be due to more length of 4-hydroxy benzoic acid molecule than 3-hydroxy benzoic acid.

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