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# Synthesis and Characterization of Polypyrrole/ Copper (II) Oxide Nanocomposite Electrolyte for Fuel Cell Application

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***Abstract:*** *Polyprrole-CuO nanocomposite is synthesized by chemical oxidation method using an anhydrous ferric chloride ($Fecl_3$) as an oxidizing agent. PPy/CuO nanocomposite prepare with varying amounts of Copper(II) Oxide (0.1g, 0.5g wt%). These composites are characterized such as structure, thermal stability, surface morphology and electrical conductivity. The surface morphology is studied by scanning electron microscopy (SEM) and structure by X-ray diffraction Technique. The SEM images of composite show agglomeration of particle. The XRD pattern of nanocomposite reveal the largely nanocrystalline. FTIR results show the broadening and shifts of peaks towards lower wave numbers in all composites suggesting better conjugation and some chemical interaction between PPy and CuO particles.*

***Keywords:*** *Polypyrrole, Nanocomposite, X-ray diffraction, SEM, Conductivity.*

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## I. Introduction

In recent years, the attention in the improvement of inorganic/organic nanocomposites has increased significantly because of a wide range of potential application of these materials [1,2]. Polypyrrole is a conducting polymer that has been widely used due to its good electrical conductivity and environmental stability. The composite of metal oxide/PPy core-shell structure shows better electronic properties compared to the pure substrate materials [3]. Among the conjugated polymers, Polypyrrole is one of the most studies intrinsically conducting polymer. PPy/CuO obtained high value in scientific community owing to its easy doping/depoing behavior. In metal-polymer composites conductivity depends on various factor suh as oxidant to monomer ratio, particle loading concentration, filler morphology, size, compactness and interfacile interactions between filler molecule and host matrix [4].Transition metal oxides (CuO) has received considerable interest in the fabrication of PPy hybrid materials because of its variety of applications in conductivity [5]. Polypyrrole/CuO is a most investigated conducting polymer because of its sizable electrical conductivity, ion-exchange capacity hydrophoric nature etc. PPy/CuO can be synthesized in the form of powder, films colloidal particles and composites in micro and nano sized as mentioned in literature [6-9]. It's one of the important green chemistry processes to prepare multifunctional polymers. This method has been widely used to prepare different types of conducting polymers and their different metal oxides composite[10, 11]. In the present paper, we have reported the preparation of polypyrrole in pure form and by using composite as CuO. The structure and morphology of these material has been investigated using the methods of FTIR, X-RD and SEM. The electrical conductivity has been measured by four-probe method.

## II. Experimental Studies

### *2.1. Materials used*

Pyrrole ($C_4H_5N$) monomer from AR, sigma Aldrich, India. Its stored in dark at 0°C. Iron (III) chloride hexahydrate-$FeCl_3.6H_2O$ (analytical grade) was used as oxidant reagent and Copper(II) Oxide was used as composite material for this work. All solutions were prepared in double distilled (DD) water.

### *2.2. Preparation of PPy/CuO Nanocomposite*

Preparationof Polypyrrole/CuO nanocomposite material by using In-situ chemical oxidative polymerization Method. The Stoichiometric amount CuO (0.5g and 0.1g) was mixed with Pyrrole monomer. The CuO and Pyrrole mixture solution was fully stirred by magnetic stirrer when complete dispersed CuO nanoparticles in to Pyrrole. Now the composite solution was kept at continues stirring conditions and addition of $FeCl_3$ oxidant slowly drop by drop. This process was taken for 30 minutes but polymerization started after one minute of first drop of addition of oxidant. The fully addition of oxidant reagent and the above homogeneous solution was stirrered for 6 hours to obtain a polymerization product. After that the above process, we got dark

greenish color of PPy/CuO nano composite precipitate. The precipitate of PPy/CuO nano composite was washed several times with acetone solution and rinsed with distilled water to remove the un-polymerized monomer and reagent. The composites so obtained are dried by keeping in oven at $60^0$C for one day. The synthesized Polypyrrole/CuO nano composite was finally grinded and the product was obtained in the form of fine black color of the nanoparticles.

### *2.3 Testing and Characterizations*

FTIR and XRD analysis were used to confirm the formation of PPy/CuO powder sample. The chemical bonding was analyzed by using FTIR- Agilent Cary 630 FTIR Spectroscopy. The X-ray diffraction (XRD) studies of the sample was done in Rigaka X-ray Diffractiometer with Cu-Kα radiation operating at 80 Kv. X-ray diffraction was carried out in the 2Θ range from 0 to 80° at the scan speed of 10° per minute. Scanning Electron Microscope (SEM) studied by SEM JEOL Model JED-2300 model using acceleration voltage of 5.0 Kv.

## III. Result And Discussions

### *3.1 X-Ray Diffraction Analysis*

The XRD patterns of the PPy/CuO nanocomposite are shown in figure 1.The average crystallite size of the PPy/CuO nanocomposite was calculated by using the Scherrer equation.

$$D=0.9\lambda/\beta \cos\theta$$

Where D is the crystalline size of particles, λ is the wavelength of X-ray, Θ is the half diffraction angle peak (in degree) and β is the true half peak width. The average crystallite size is 30 nm.

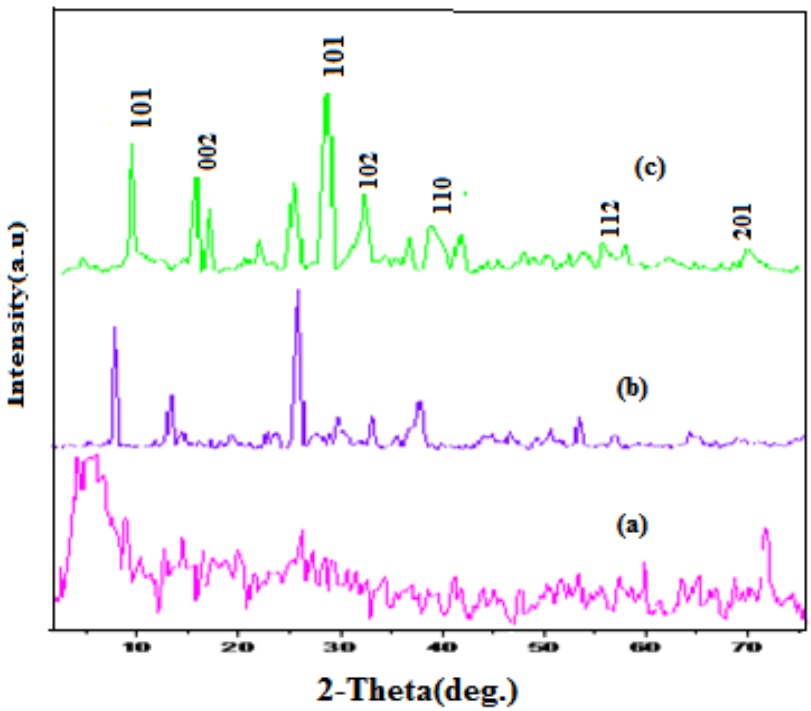


**Fig.1.** XRD Analysis of (a) Pure PPy(b) PPy/CuO(0.1g wt%) (c) PPy/CuO (0.5g wt%) nanocomposite

### *3.2 FTIR Spectroscopy*

The Polypyrrole powders were analyzed by FTIR. The FTIR spectra were recorded in the range of 4000$cm^{-1}$ to 400$cm^{-1}$, In the FTIR spectrum of PPy, peaks at 1562,1469, and 3410$cm^{-1}$ broad peak) corresponds to C-C,C-N and N-H stretching vibration in the Pyrrole ring respectively [12].Then the band at 1285$cm^{-1}$ which can be attributed to aromatic C-H stretching vibrations. The peaks at 1675 $cm^{-1}$ and 796$cm^{-1}$ represents C=N and C-N bonds, the band of C-H in plane deformation vibration is situated at 1010$cm^{-1}$ out of plane ring deformation of C-C and C-H at peak 685$cm^{-1}$ [13,14]. In our FTIR spectral wavelength and intensity of peaks also indicated that the peak shift of PPy-CuO nano compositen and CuO nano particles are diffusing to the PPy ring [15].

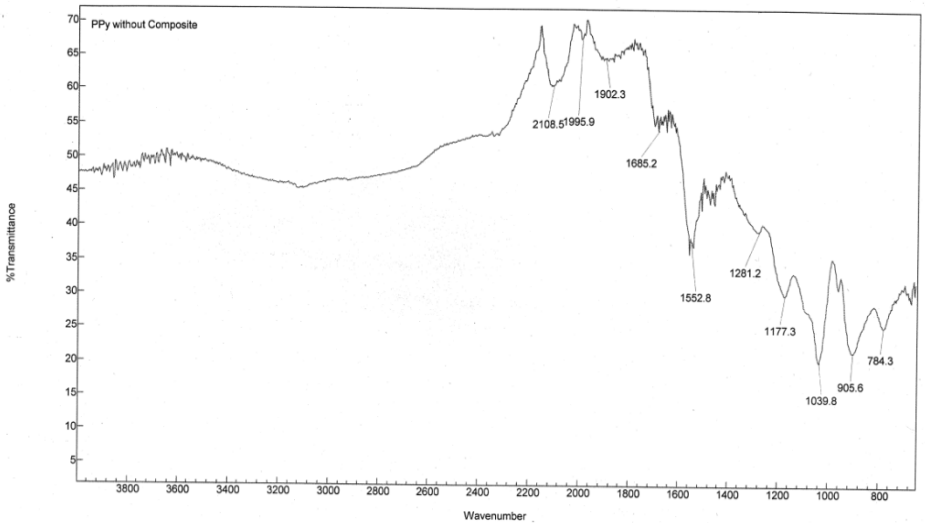


**Fig 2.**FTIR Spectral line of Polypyrrole

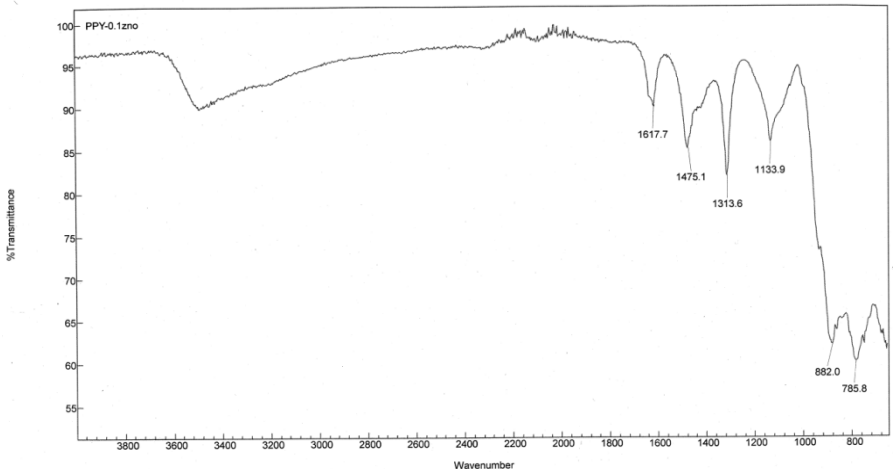


**Fig 3.**FTIR Spectral line of PPy/CuO (0.1g wt%) nanocomposite

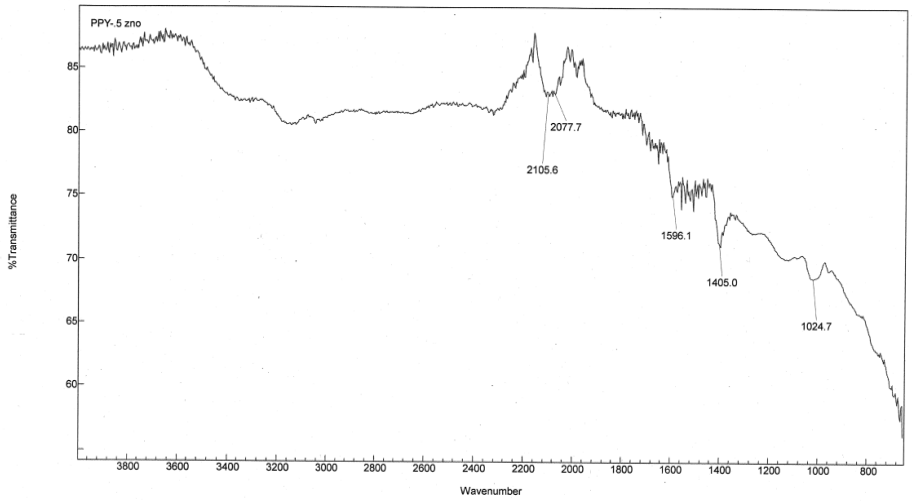


**Fig 4.**FTIR Spectral line of PPy/CuO (0.5g wt%) nanocomposite

### *3.3 Morphological Analysis*

Scanning Electron Microscopy (SEM) used an electron beam for surface imaging. The advantage of SEM over light microscopy is greater magnification and much larger depth of field. Different elements and surface topographies emit different quantity of electrons due to the contrast in a SEM micrograph which is representative of the surface topography and distribution of elemental composition on the surface [16].

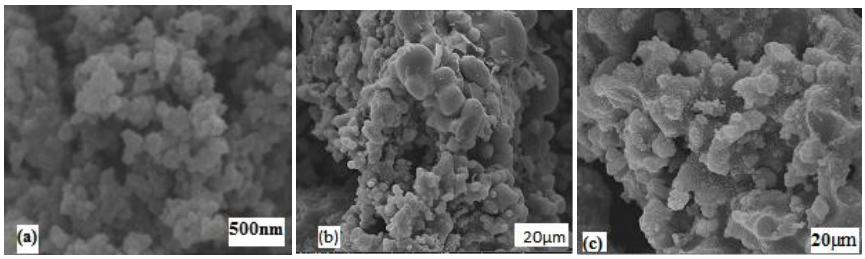


**Fig 5.**SEM Analysis of (a)Pure PPy (b) PPy/CuO(0.1g wt%) (c) PPy/CuO (0.5g wt%) nanocomposite

### *3.4 Electrical Conductivity Studies*

The fig.4 indicates that the conductivity of PPy/CuO nanocomposite was measured by four probe dc method. The conductivity increases with increasing temperature. This result indicates that the conductivity of polypyrrole / CuO (CuO -0.10%wt) dramatically increases upto 1.43Scm$^{-1}$ with temperature. But slight decreases to 1.42Scm$^{-1}$ and then increase with temperature. The conductivity of Ppy / CuO (CuO -0.5%wt) dramatically increases upto2.43 Scm$^{-1}$. But slight decrease to 2.42 Scm$^{-1}$and the increases with temperature.

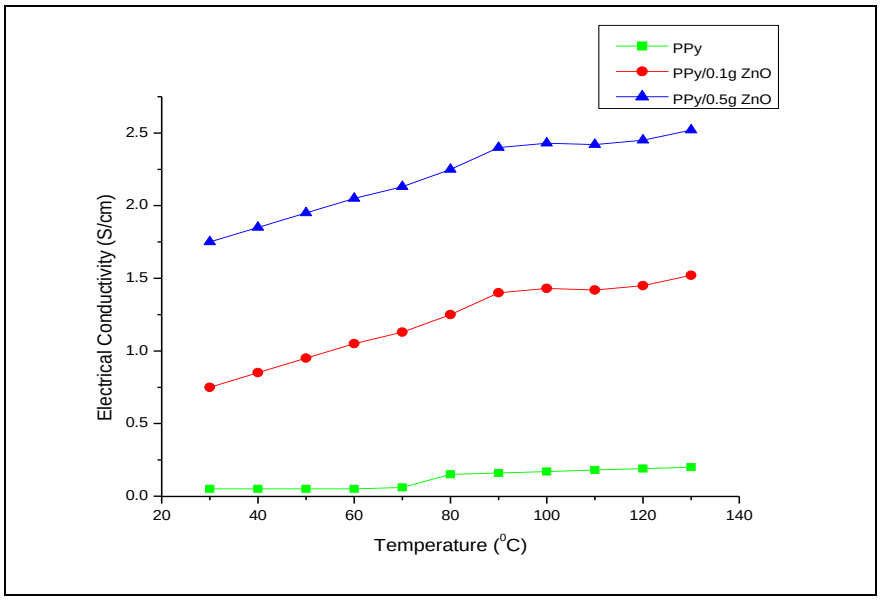


**Fig 6.**Conductivity of PPy/ CuO NanoComposite

## IV. Conclusion

**T**he PPy/CuO composite have prepared by using ferric chloride as an oxidant. The nanocomposite of PPy/CuO material has been successfully synthesized by novel in-situ chemical polymerization method. The XRD confirmed the structure of PPy/CuO nanocomposite and FTIR confirmed the functional group The surface morphological was achieved by SEM and Conductivity study by d.c four probe method. All the characterizations are clearly indicated that the formation of PPy/CuO nanocomposite and it is a novel electrolyte material for fuel cell application.

## References

[1] Jing,S.,et al: Synthesis and characterization of Ag/polypyrrole nanocomposite based on silver nanoparticles collid.Mater.Lett 61(23-24),4528-4530 (2007).

[2] Chen S., Zhitomirsky, I.: Influence of dopants and Carbon nanotubes on polypyrrole electropolymerization and capacitive behavior.Mater.Lett.98, 67-70(2013).

[3] J.Zang, X.Li, In Situ Synthesis of Ultrafine β-$MnO_2$ /PPy nanorod Composite for high-performance Super Capacitors, J.Mater.Chem.21(2011)10965-10969.

[4] H.C.Pant, M.K.Pata, S.C.Negi, A.Bhatiya, S.R.Vadera, N.Kumar,Bull,Mat

[5] Y.He, A novel emulsion route to sub-micro meter Polyaniline/nano-ZnO Composite fiber' Applied Surface science, Vol.249, pp 7-18,2010.

[6] Huiling Tai, Yadong Jiang,Guang Zhong Xie, Jun Shung Yu and Xuan Chen, Fabrication and gas sensitivity of Polyaniline-$TiO_2$ nanocomposite thin film, Sensors and Actuators B25,644-650(2007)

[7] Nguyen Thi Le H., Garcia B.,Deslouis C. and Le Xuan Q., Corrosion Protection and Conducting Polymers; Polypyrrole films on iron , Electro Chemical Acta,46,4259-4272(2001)

[8] Seandeera G.K.R., Kitamura T., Wada Y. and Yanagida S., Deposition of polyaniline via molecular Self-assembly on $TiO_2$ and its uses as sentiser in solid –state Solar Cells ,Jour of Photochem and hotobiol A: Chem., 164, 61-66(2004).

[9] Au Ji Ru Son, Hoosung Lee and Bongjin Moon, Morphology and Photoluminescence of Colloidal Polypyrrole and nanoparticles, Synth.Mat., 157,597-602(2007).
[10] Peng,Y., et al.: Facile Preparation of water dispersible polypyrrole nanotubes-Supported silver nanoparticles for hydrogen peroxide reduction and surface-enhanced Raman scattering Electrochimica Acta 75, 399-405(2012)
[11] Sahoo.S, Karthikeyan.G, Nayak .G.C, Das C.K. Synth Met 2011; 161(15-16): 1713-9.
[12] Bora C, Doului SK, polymer 2012;53(4): 923-32.
[13] R.Ansari, Polypyrrole Conducting Electroactive Polymers: Synthesis and Stability studies ,E-Journal Chemistry, Vol-3, pp.186-201(2006)
[14] H.Kato,O.Nishikwa,T.Matsui, S.Honma and H.Ko-kado,mThe journal of physical chemistry,Vol.95,No.15, pp.6014-6016(2008).
[15] K.Cheah, M.Forsy and V.T.Truong, Ordering and Stability in Conducting polypyrrole ,Synthesis Metals,94(2),215-219(1998)
[16] Lei J, Leang W, Martin C.R.Infrared investigating of Pristine, doped and partilly doped polypyrrole,Synthetic Metals,48(3),301-312(2002).

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