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Synthesis, characterization and conductivity studies of Polypyrrole-Expanded Graphite–composites

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Abstract – *– In situ polymerization of pyrrole was executed in the presence of expanded graphite to synthesize polypyrrole–expanded graphite composites (PPy/EG) by chemical oxidation method. The PPy/EG composites have been prepared with various compositions (2%, 5%, 7.5%, 10% wt. %) of expanded graphite in pyrrole. Morphology of the composites has been examined by scanning electron microscopy (SEM). The polypyrrole-expanded graphite composites were also examined by X-ray diffractometry (XRD) and Attenuated total reflectance (ATR). The a.c. conductivity behavior has been analyzed by LCR-Q-Meter-SORTER. The presence of expanded graphite in the matrix have an excellent effect on the observed conductivity values. The results showed that these composites are of preferably technological and businesslike interest.*

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Key Words **:** Polypyrrole, Expanded graphite, composites; conductivity**.**

1.INTRODUCTION

Conducting polymers exhibit semiconductor or metal-like electrical and optical properties while at the same time they are lightweight, flexible, inexpensive, and easy to synthesize [1]. The understanding of electrical properties, morphology and crystal structure of polypyrrole composites may be useful in improving the stability characteristics of these materials which are the key factors in governing the device performance. The polymer/expanded graphite composite granules were obtained by in situ polymerization of the monomers it was found that composite with excellent electrical conductivity. [2] In situ polymerization of pyrrole was carried out to synthesize polypyrrole–fly ash composites (PPy/FA) in the presence of fly ash (FA) by chemical oxidation method. The results showed that fly ash have a greater influence on the observed conductivity values. [3]Fly ash is a waste product produced from coal fired thermal power stations during the combustion of coal. It is an alkaline grey powder with pH ranging from 9–9 9. Large number of coal fired power plants all over the world dispose a large quantity of fly ash. causing serious environmental problems [4]. In situ electro-polymerization of pyrrole onto carbon could improve the performance of carbon electrodes for use in supercapacitors. [5].PPy/Y2O3 composites were prepared by dispersing different amounts of Y2O3 particles in polypyrrole matrix. It was concluded that the increase in the a.c. conductivity of the PPy/Y2O3 composites over pure PPy was due to macroscopic conductivity. PPy/Y2O3 composites with higher weight percentage of Y2O3 showed higher conductivity than pure PPy due to increased orderness in the composites. [6] The thermal stability of PPy/A-FA composites was enhanced and this could be attributed to the retardation effect of amine-functionalized fly ashes as barriers for the degradation of polypyrrole. The composites possess high electrical conductivity at room temperature, weak temperature dependence of the conductivity and good magnetic properties. [7]. Graphene (GR) can be considered as nanosized-filler for PPy due to their high surface area and excellent conductivity. [8]. hydrate. The large surface area and high aspect ratio of the in-situ generated graphene played an important role in justifying the noticeable improvements in electrical conductivity of the prepared composites via chemical reduction. [9] The PPy/EG composites were fabricated by chemical oxidation polymerization showed that the EG sheet layer is loose with a lot of voids in it and the average dispersion of particle in the PPy matrix and the thermal stability of PPy/EG composites exhibited a beneficial effect comparing with pure PANI. [10]

In many methods of synthesis of PPy, chemical oxidation polymerization is the most easily to prepare PPy and minimum cost. In order to get a good conductivity, using EG as conducting fillers, we have successfully prepared PPy/EG composites by chemical oxidation polymerization. The morphology property, structure and electrical conductivity were also investigated base on scanning electron microscope (SEM), X-ray powder diffraction (XRD), Attenuated total reflectance (ATR) and LCR-Q-Meter-SORTER. Our way is adequate to obtain composites with appropriate EG dispersion on the PPy matrix with relatively high electrical conductivity.

2. EXPERIMENTAL DETAILS

2.1. Materials

The pyrrole (Py) was procured from Spectrochem Pvt.Ltd.Mumbai, India**.** Anhydrous Iron chloride procured from Merck Life Science Pvt.Ltd, Mumbai were used as

doping agent to prepare PPY/EG composites. Expandable graphite (PUCC50, H2O <1.0%) was purchased from Metachem Manufacturing Company Pvt.Ltd. Prior to use, the expandable graphite was calcinated (muffle furnace, 350°C, 10 min) to obtain Expanded Graphite.. All the experiments were carried out using distilled water.

2.2. Synthesis of PPy/EG composites

PPy/EG composites were prepared using the following procedure. A certain amount of (2%, 5%, 7.5%and10% wt. %) EG were soaked in 6.7g Py for 1 hr. treated 100 ml distilled water were put in three bottle neck reactor stirred for 1 h. Then, aqueous solution of oxidant, 16.2 g FeCl3 anhydrous at appropriate concentration was added to the reaction media in drop wise fashion in 15 min using a dropping funnel. The reaction time was 4 h after FeCl3 of aqueous solution was dropped. Upon being filtrated, the composites were washed by 150ml of distilled water.

3. RESULTS AND DISCUSSION

3.1. Microstructures of PPy and PPy/EG composites

Fig -1: is the SEM microscopy images of PPy in a solution of FeCl3 doped, from Fig -1 shows, Oxide particles are covered by spherical nature of polypyrrole to form multiparticle aggregates, presumably because of weak interparticle interactions.

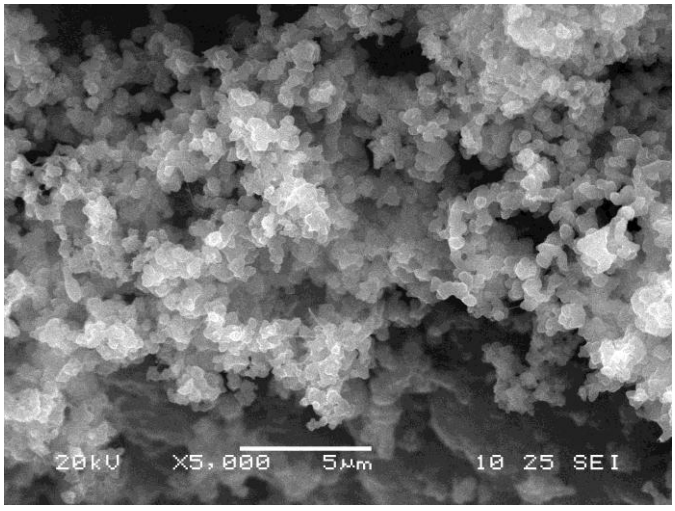

Fig -1: SEM microscopy images of PPy

Fig -2: The SEM micrograph of polypyrrole/EG composites reveals that uniform distribution of small particles in EG and PPy matrix, and we can view that EG dispersed in PPy and the PPy also encapsulated the EG. Additionally, EG is distributed quiet uniformly within the PPy matrix. Due to this structure, we predict that conduction property of PPy/EG composites may be controlled by the EG and reveal a soft value of percolation threshold, this explain was researched in the hereafter part

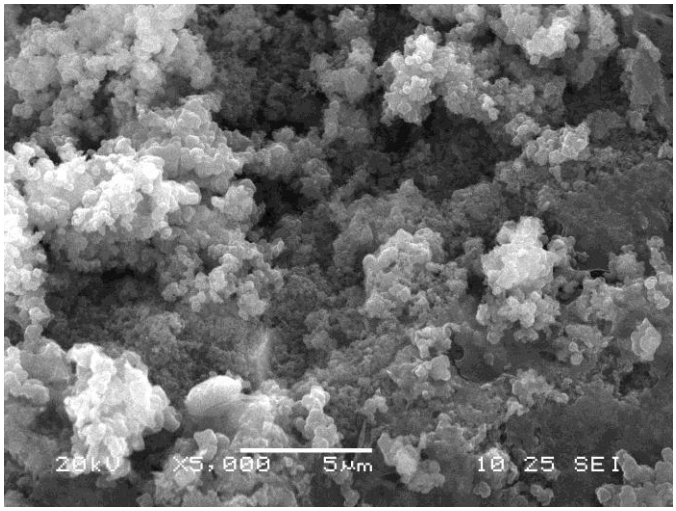

Fig -2: SEM microscopy images of PPy/5%EG.

3.2. FTIR Spectra

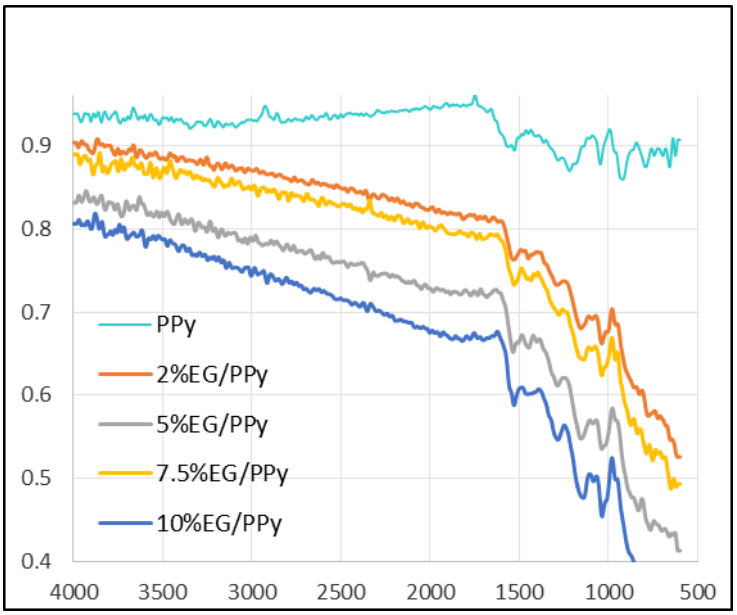

Fig -3: Infrared Spectroscopy of PPy and PPy/EG Composites (2%, 5%, 7.5%10% wt. %)

The IR spectra of polypyrrole–expanded graphite composite is shown in figure 3. From the graph it observed that frequency suppressed .This may be due to restrict movement of a molecules. During polymerization reaction, Aniline penetrate the expanded graphite structure and NH_2 attach to the bond. Since it is acidic nature some functional group generated on expanded graphite. It indicates that 730 cm^{-1}onwards chloro compound bond formed.

3.3 XRD

Figure 3 shows the X-ray diffraction patterns of polypyrrole composites (5 wt. % of EG in polypyrrole). A typical diffraction peak of graphite located at 2Θ= 25.3◦.This results suggested that Intensity and breadth of XRD peaks gives crystalline size.

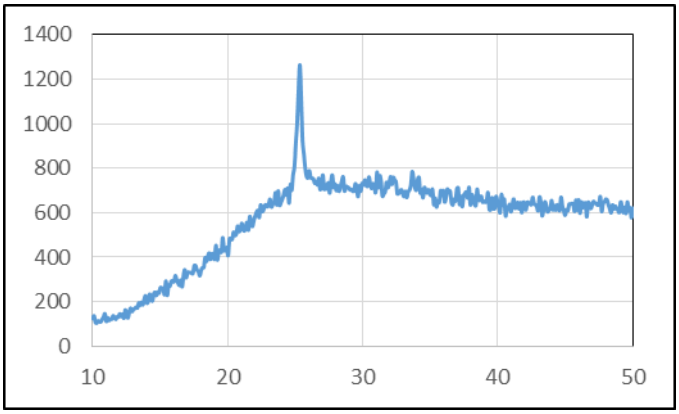

Fig -3: X-ray spectroscopy of polypyrrole–expanded graphite composites (5 wt. % of EG).

3.4. A.C. electrical conductivity

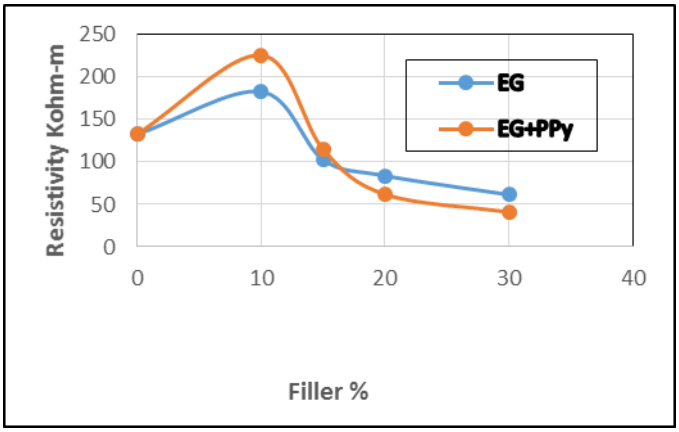

Fig -4a: Resistivity of EG and PPy/EG Composites.

Fig -4a shows Resistivity decreases with filler concentration inceases.Graph reveals that PPy/EG more conductive than only EG.LCR-Q-Meter-SORTER Model 4912 used to calculate resistance and capacitance.

Fig -4b shows increase in Permitticity of PPy/EG composites which calculated from capacitance for different % filler.

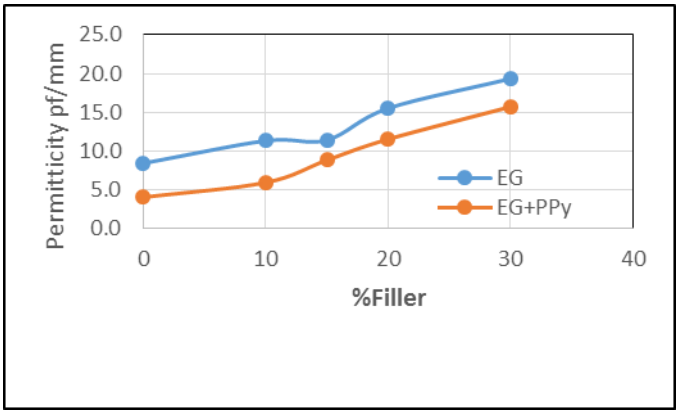

Fig -4b: Permitticity of EG and PPy/EG Composites.

4. CONCLUSIONS

PPy/EG composites are prepared by chemical oxidation method by using anhydrous $FeCl_3$. Composites characterization were successfully executed through XRD, SEM and IR. PPy/EG composites show better a.c conductivity than only expanded graphite and also dependence on the weight per cent of expanded Graphite (%filler) in polypyrrole.

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REFERENCES

[1] Yuvraj Singh Negi and P. V. Adhyapak, Polymer Research and Development Division, Centre for Materials for Electronics Technology, 42, 35-53

[2] Zheng, G., Wu, J., Wang, W., & Pan, C. (2004). Characterizations of expanded graphite/polymer composites prepared by in situ polymerization. *Carbon*, *42*(14), 2839–2847.

[3] Murugendrappa, M. V, Khasim, S., & Prasad, M. V. N. A. (2005). Synthesis , characterization and conductivity studies of polypyrrole – fly ash composites. October, 28(6), 565–569..

[4] Sahu K C 1994 Power plant pollution: Cost of coal combus- tion, survey of the Environment, The Hindu 47.

[5] Muthulakshmi, B., Kalpana, D., Pitchumani, S., & Renganathan, N. G. (2006). Electrochemical deposition of polypyrrole for symmetric supercapacitors. Journal of Power Sources, 158(2 SPEC. ISS.), 1533–1537.

[6] Vishnuvardhan, T. K., Kulkarni, V. R., Basavaraja, C., & Raghavendra, S. C. (2006). Synthesis , characterization and a . c . conductivity of polypyrrole / Y 2 O 3 composites, 29(1), 77–83.

[7] Yang, C., Wang, T., Liu, P., Shi, H., & Xue, D. (2009). Preparation of well-defined blackberry-like polypyrrole/fly ash composite microspheres and their electrical conductivity and magnetic properties. Current

Opinion in Solid State and Materials Science, 13(5-6), 112–118.

[8] Stankovich S, Dikin DA, Dommett GHB, Kohlhaas KM, Zimney EJ, Stach EA, et al. Nature 2006;442(7100):282-6.

[9] Bose, S., Kuila, T., Uddin, M. E., Kim, N. H., Lau, A. K. T., & Lee, J. H. (2010). In-situ synthesis and characterization of electrically conductive polypyrrole/graphene nanocomposites. Polymer, 51(25), 5921–5928.

[10] Wu, X., Liu, M., & Jia, M. (2013). Electrical conductivity of polypyrrole/expanded graphite composites prepared by chemical oxidation polymerization. Synthetic Metals, 177, 60–64.