



SYNTHESIS, CHARACTERIZATION AND ELECTRICAL PROPERTIES ON PANI/NiO COMPOSITE

Shailaja Mahadappa*

ABSTRACT

Executed by In situ chemically oxidative route has been prepared PANI and doped Polyaniline (PANI/NiO) with weight percent of doping oxide. Oxidant agent as an Ammonium persulfate & HCL as a catalyst. Further these composites were characterized by XRD & SEM methods to investigate their structural and morphological studies. As a part of electrical studies DC conductivity was studied as a function of temperature and AC conductivity was studied as a function of frequency.

Keyword: PANI, SEM, XRD, DC

*Government First Grade College, basavakalyan-585327, Email: shailajamallikarjun@gmail.com

***Corresponding Author:** - Shailaja Mahadappa

*Email: shailajamallikarjun@gmail.com

DOI: 10.53555/ecb/2022.11.11.157

Introduction

Conducting mixtures of materials built on polymers are designed by a appropriate compositions of conductive polymers such as polyaniline and polythiophene with metal oxides which increases the required properties. Current investigation shows that conducting polymer composites have been widely deliberate due to their uses in numerous fields [1–3]. Metal oxide nanomaterial's and their polymer Nano composite constituents have a lot of profitable demand in market due to their scientific and industrial importance [4]. It is informed in the works that the creation of metal oxide distributed polymer Nano composites without using any surfactant block copolymer patterns [5–9], where the nanomaterial's are presented in the polymer production. Recent reports show that no further doping is required after polymer synthesis and uniform dispersal of metal oxide within a polymer matrix without the aid of any surfactant [10]. The electrical stuffs of conducting polyaniline can be controlled suitably by charge transmission doping or proto- nation process. Inorganic nanomaterials that dispersed organic phase composite materials are important due to their extraordinary properties. These stuffs are arising from the synergism between the possessions of inorganic and organic components. One can find numerous synthetic routes for these materials, but the most prominent method is the incorporation of inorganic building blocks such as nanomaterials in organic polymers matrix. Inorganic–organic base nanocompos- its materials are attracted much due to their various properties such as electrical, mechanical, magnetic and thermal properties [11–16]. The current survey focusing the fabricates compositing polyaniline materials these composites have contains large property of the applications. The NiO may be an add-on advantage in the fields of polymer; in exacting, it may supply high-quality applications while dopes the PANI. The property of composites similar to higher stiffness and lofty melt points can be use the intractable agent. Little previous information explains the sensitivities and selectiveness in research of nanostructure PANI with the adding of dissimilar oxides of the metals

Materials and Method:

Materials and Methods

All chemicals used in the present experimentation are of AR grade evaluation. Polyaniline is used as a fuel for solid state self-propagating combustion reaction for union of nanosized metal oxide material. Double distilled water as a solvent for solution preparation and acetone is utilized as a solvent for the separation of the carbon particles.

- Preparation of 0.1M of ammonium persulphate in first volumetric flask.
- Preparation of 1N of hydrochloric acid (HCL) in second volumetric flask.
- Preparation of 0.1M of aniline in third volumetric flask.

Possible chemical reactions of metal and metal oxides

| | |
|-----|--|
| NiO | $\text{NiCl}_2 + (\text{COOH})_2 \rightarrow \text{Ni}(\text{C}_2\text{O}_4) + 2\text{HCl}$ $\text{Ni}(\text{C}_2\text{O}_4) + 1/2\text{O}_2 \rightarrow \text{NiO} + 2\text{CO}_2$ |
|-----|--|

Polyaniline-NiO oxide composite:

The prepared solution of 0.1M of aniline in volumetric flask is dissolved in a beaker along with prepared solution of 0.1N of hydrochloric acid and this mixer is stirred for one hour by magnetic stirrer at room temperature. After one hour of constant stirring, the prepared solution of 0.1M of ammonium persulphate was added drop wise in the solution of aniline hydrochloric acid using pipette. NiO oxide was added in the mass fraction to the above solution with vigorous stirring in order to keep the iron oxide homogeneously suspended in the solution. This mixer is stirred for 10 hours for the completion of reaction, and then it left for 24 hours to settle down. Later the prepared solution is filtered using vacuum pump and Buchner funnel. This precipitate was filtered, washed with deionised water, with acetone in order to remove the oligomers and excess ammonium persulphate, and with 1 N HCl solution to remove the Cl⁻ ions and unreacted aniline. Finally, the precipitate was dried in an air oven for 24 h at a temperature of room temperature to achieve a constant mass.

Result and discussion

XRD: PANI/NiO

XRD study was convinient to know the crystalline and phase structure of the synthesised nickel oxide sample shown in figure. The patterns show the presence of highly intensed Bragg's reflection indicates the crystalline nature of the sample. It is observed from the pattern that, most of the 2-theta values of sample acceptably match with the literature data of nickel oxide (JCPDS) this confirms the formation of the sample. The broadening and sharp peaks indicate the reduced particle size and high crystallinity of the sample. The XRD results are consistent with the results obtained by other research groups . The average crystalline size of the PANI are estimated to be approximately 2.86 nm are calculated by using Debye -Scherer formula, $(D=K\lambda/(\beta \cos\theta))$ Where D is crystalline size, λ is wavelength, K is crystallite shape factor a good approximation is 0.9, β if FWHM and 2θ is angle (deg.)[17-18].

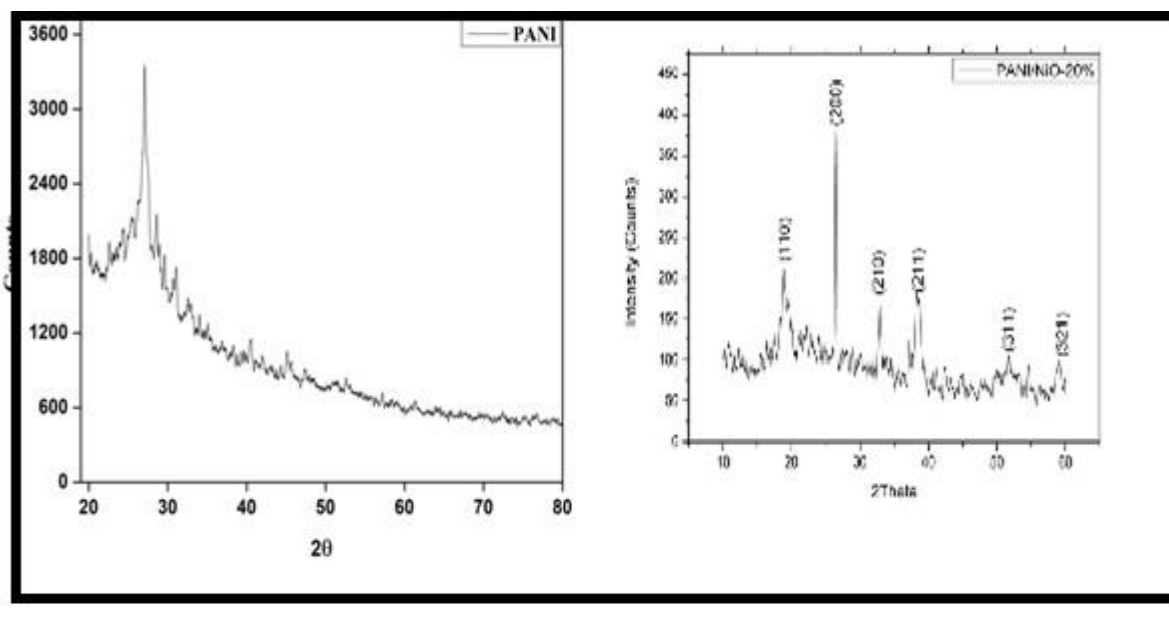


Fig explains the XRD pattern of PANI/NiO

SEM: PANI/NiO

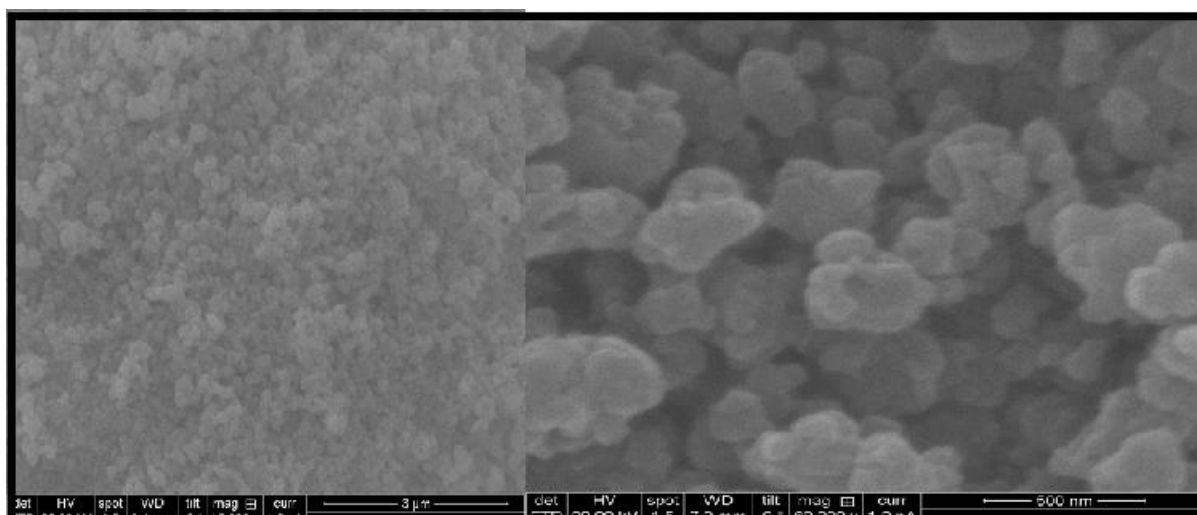
Figure shows the SEM image of nickel oxide sample respectively. It is observed from the figure nickel oxide particles shows variable size of the particles with different shape and size. Some of the particles shows close compact structure which indicates the crystalline behaviour. These results are in agreement with the XRD results. From SEM micrograph it clearly indicates that it has highly clustered structure. The presence of sulphuric acid has a brawny power on a variety of electrically parameter such as conductivities and dielectrics performance of this compound. The difference in the image is due to the difference in

dispersion from dissimilar exterior areas as a consequence of geometrically difference linking polyaniline.

The grain size is computed utilizing linear intercept technique and average grain size is figured by the accompanying condition.

$$\text{Grain Size} = \frac{C}{MN}$$

Where, *C* is test line length, *N* is the intercepts and *M* is the magnification computed from the reference scale imprinted on the micrograph[19-20].



The SEM picture of PANI/NiO

DC CONDUCTIVITY: PANI/NiO PURE PANI

Fig shows the difference of DC conductivities as a role of hotness for PANI. The conductivities increasing with increases temp. Exhibits the

conductivities 3 phase in a warmth i.e. from 40 °C to 200 °C. the □ steady in 50 – 80 °C, and increase slowly in the hotness series 80 – 110 °C. In stage III, 110 – 150 °C, a linear increase in the conductivities value is experiential

Polyaniline – NiO composites

Figure shows the variation of dc conductivity as a function of temperature for NiO in polyaniline. It is observed that the value of dc conductivity of these composites increases exponentially with temperature. It remains nearly constant up to 100°C and there after it increases exponentially. The conductivity behaviour is the characteristic of amorphous materials. The initial increase in the

values of conductivity is due to the extended chain length of polyaniline due to which the charge carriers can hop between the favourable localized sites. The decrease in the values of conductivity at 25 wt % of NiO is due to partial blocking of charge carriers. Further, gradual increase in conductivity is noticed due to the variation in distribution of NiO particles in PANI[21-22].

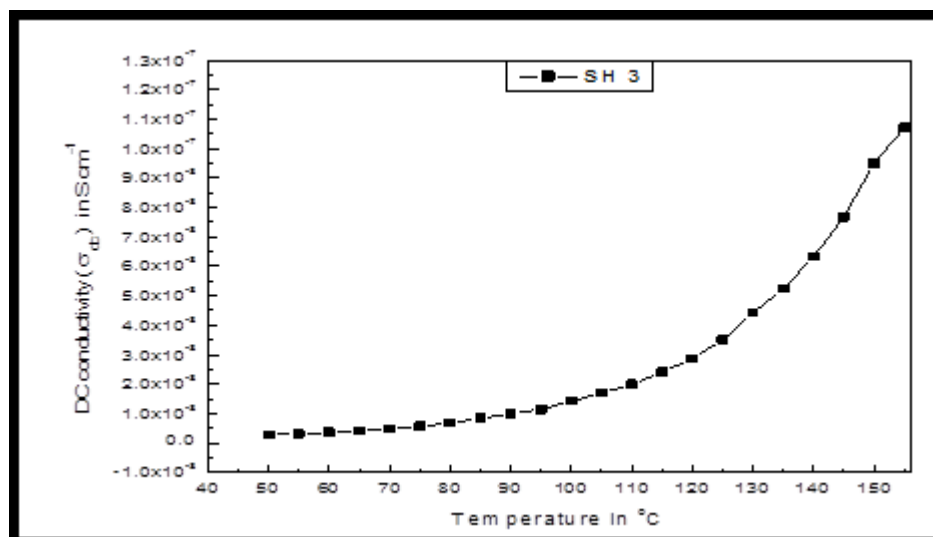


Figure shows the dc conductivity of polyaniline – NiO composites

AC CONDUCTIVITY: PANI/NiO PANI– NiO composites

From the figure shows that the variation of ac conductivity as a function of frequency for PANI–NiO composites (different wt %). The results revealed that, in all cases, σ_{ac} remains constant up to 10^5 Hz and maximum value of conductivity is

observed for 25 wt% at 10^5 Hz. This type of behaviour attributed to addition of NiO-PANI in polymer matrix and all composites the conductivity increases at wt of NiO-PANI. This is due to extended chain length of PANI, which facilitated the polarization of charge carriers [23-25].

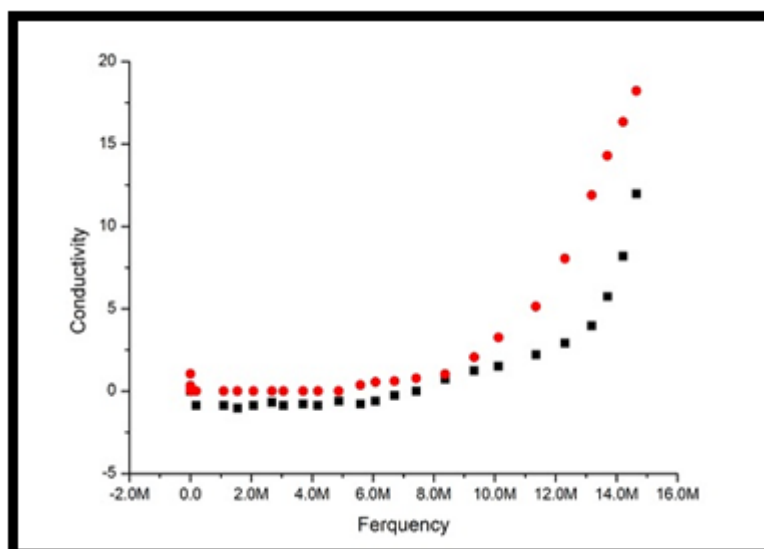


Figure shows the ac conductivity of polyaniline – NiO composites

CONCLUSION

The polyaniline-NiO composites were prepared by chemical oxidative polymerization method (COP) with ammonium persulfate as oxidant. The

wide peak in the XRD shape of the pure polyaniline shows the amorphous nature of the sample and has the prominent peak of pure polyaniline is in the range of 25-27°. The

representative XRD peaks at angle 2θ are peaks of iron oxide which is readily indexed and are consistent with the results. The average crystalline size of the composite is estimated to be approximately 20.21 nm. The SEM image of PANI/NiO reveals that the dopant particles are dispersed in PANI which also justifies the successful composite formation and mainly composed of irregularly arranged granular, nonporous, aggregated surface morphologies with diverse sizes. The conductivity increases with growth in frequency in single phase. It can also be seen that the ac conductivity of the PANI/NiO composite decreases with increase in the content of NIO in the PANI except composite 10%. The variation of dc conductivity as a function of temperature which recommends that as temperature increases conductivity also increases, hence thermally activated exponential behaviour of conductivity has been observed.

References:

1. B. Manjunatha & Arjun N. Shetty & S. Kaveri & Sundar S. Mety & K. C. Anjaneya & Ramakrishna Reddy & Sangshetty Kalyane 5 Chemical Mediated Synthesis of Polyaniline/Tungstenoxide (PANI/WO₃) Nanocomposites and Their Antibacterial Activity Against Clinical Pathogenic Bacteria **BioNanoScience** BioNanoScience 2020,, 10(1), 73-80
2. Morawetz H Encyclopaedia of Polymer Science and Engineering, 2nd edn. vol. 7, pp. 722-745 (Wiley, New York) (1987).
3. Angew.chem.Int. NO.19.3456 WEBSITE <http://www.ch.ic.ac.uk/local/organic/tutorial/steinke/4yrPolyConduct> (2003).
4. Bovey F. A., Academic Press, New York, (1982).
5. Jiang LH, Leu C MWei KH "Layered silicates/fluorinated polyamide nanocomposites for advanced dielectric materials applications" *Advanced Materials*, 14, (2002), 963.
6. Caruso F, "Nanoengineering of particle surface" *Advanced Materials*, 13, (2001),11.
7. Mallikarjuna NN, Venkataraman A and Aminabhavi TM, "A study on □-Fe₂O₃ loaded Poly (methyl methacrylate) Nanocomposites" *Journal of Applied Polymer Science*, 94, (2004), 2551
8. S. Radhakrishnan, N. Sonawane, C.R. Siju, Epoxy powder coatings containing polyaniline for enhanced corrosion protection, *Prog. Org. Coat.* 64 (2009) 383- 386.
9. M. Tiitu, A. Talo, O. Forsén, O. Ikkala, Aminic epoxy resin hardeners as reactive solvents for conjugated polymers: polyaniline base/epoxy composites for anticorrosion coatings, *Polymer* 46 (2005) 6855–6861.
10. The Nobel Prize in Chemistry, discovery and development of conductive polymers,2000. <https://www.nobelprize.org/prizes/chemistry/2000/summary/> (Accessed on: 14th January 2021)
11. N.N. Mallikarjuna, S.K. Manohar, P.V. Kulkarni, A. Venkataraman, T.M.Aminabhavi, Novel high dielectric constant nanocomposites of polyaniline dispersed with γ -Fe₂O₃ nanoparticles, *J. Appl. Polym. Sci.* 97 (2005) 1868-1874.
12. Muhammad Faisal and Syed Khasim, *Bull. Korean Chem. Soc.* 34(2013) 99-106
13. S. K. Shukla, N. B. Singh, R. P. Rastogi, *Indian Journal of Engineering & Materials Sciences*, 20(2013)319-324.
14. V. X. Moreir, F. G. Garcia, B. G. Soares, *JAppl.Polym.Sci.*,100(2006)4059-4065.
15. Patel,R.G.,Solanki,G.K.,Prajapati,S.M.,& Oza,A.T.(2005).Kuhnperiodicityinoligoaniline sandoligoaniline-I odinecomplexes.*MolecularCrystalsandLiquidCrystals*,44 2(1),167–180.
16. Manjunatha B1*, Nagajyoti2, Rahul Jabnoor3, Vinayak G K4, Arun Kumar Lakshetty5, Sangshetty Kalyane6 The Effect of Variation Sensitivity Against Time for Magnesium Oxide (MgO) Doped Polyaniline Nanocomposites Influenced by Ammonium Persulfate *Journal of Chemistry: Education Research and Practice* ISSN: 2578-7365, 6(2), 430-433,2022
17. Rahul Jabnoor¹, Manjunatha B², M V N AmbikaPrasad³, Arunkumar Lagashetty⁴, and Vijaykumar Basavarajappa⁵, Sangshetty Kalyane 6 Synthesis, characterisation and AC conductivity study of PANI/□-Fe₂O₃ nanocomposites *IOP Conf. Series: Materials Science and Engineering* 1221 (2022) 012001
18. Ternau J G, Report MRL –R – 1143 *Materials Research Laboratory*, Nov. 1988
19. Caruso F, "Nanoengineering of particle surface" *Advanced Materials*, 13, (2001), 11
20. Sinha R Outline s of polymer technology, New Delhi: Prentice Hall of India private Limited (2002).
21. *New Polymers for Special Applications*", book edited by Ailton De Souza Gomes, ISBN 978-953-51-0744-6, (2012)
22. Jiang J, Li L and Xu F- In Situ Synthesis and Characterization of LiNi_{0.5}La_{0.08}Fe_{1.92}O₄ - Polyaniline Core-Shell Nanocomposites, 2007, *Journal of Physics and Chemistry of solids*, Vol.68, 1656-1662.

23. Guo H, Zhu H, Lin H and Zhang J-
Polyaniline/Fe₃O₄ nanocomposites
synthesized under the direction of cationic
surfactant, 2008, *Materials Letters*, Vol.62,
2196-2199.
24. Mott N F and Davis E A (1979) *Electronic
Processes in Non-Crystal line Materials*
(Clarendon press Oxford) 28 50 81 205. 24.
Pollak M (1971) *Discuss Faraday Rev A* 133
564.
25. D. Y. Kim, Y. C. Chung, T. W. Kang and H.
C. Kim, "Dependence of microwave absorbing
property on ferrite volume fraction in MnZn
ferrite-rubber composite", *IEEE Trans.
Magnetics*, Vol. 32 (2), 1996, PP. 555-558