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INVESTIGATION OF ELECTROCHEMICAL PROPERTIES OF PANI DOPED NiO NANOCOMPOSITE FOR ENERGY STORAGE

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ABSTRACT:

Polyaniline-based inorganic nanocomposite have been receiving more curiosity as the connection of inorganic nanoparticles with conducting polymer results in greater capacitances. In this respect we have prepared nanocomposite based on metal oxide (Nickel oxide) coated with polyaniline (PANI) and studied their electrochemical performance. The transmission electron microscope (TEM) was used to perform the morphological characterizations.To characterize the prepared nanocomposite electrode, an electrochemical impedance test was conducted.

Keywords: *NiO, PANI, PANI/NiO, TEM, EIS.*

INTRODUCTION :

Polymer inorganic nanocomposites have attracted great attention as they offer advantages of both polymer and inorganic nanoparticles. In conducting polymer nanocomposites, filler in nanoscale plays the major role in strengthening the properties of the nanocomposites. The conducting polymer Polyaniline's (PANI) stability, electrical conductivity, and mechanical strength have made them a novel class of materials. PANI has a wide range of applications, including, solar cell applications [1–3], rechargeable batteries [4], active corrosion protection [5] electrochromic displays, electrochemical sensors, and capacitors. As a result, researchers are becoming more interested in creating nanocomposites based on PANI. The literature review on conducting polymer nanocomposites show that PANI has been successfully utilized for the preparation of nanocomposites [6-9]. In this paper we report the synthesis and effect of nickel oxide as nanosized filler on the electrochemical characterization of the conducting polyaniline nanocomposite.

EXPERIMENTAL:

MATERIALS AND METHODS:

Merck provided the sodium hydrochloride (99%), nickel carbonate hexahydrate (NiCo3.6H2O) (99%), and polyvinylidene fluoride (CH2CF2)n (99%).Ammonium persulphate $(NH_4)_2S_2O_8$) (APS (99%), N-methyl-2-Pyrrolidine (99%) and potassium hydroxide (KOH) (98%) were purchased from Qualigens Fine Chemicals. Before usage, aniline hydrochloride was distilled, and the other compounds used as supplements were AR grade.

Synthesis of NiO and PANI/NiO nanocomposite:

Using starch as a capping agent and nickel carbonate hexahydrate (NiCo3.6H2O) as a precursor, NiO nanoparticles were made using the straightforward chemical precipitation method [10].In-situ chemical oxidative polymerization of aniline using ammonium persulphate as an oxidant in the H_2SO_4 solution was employed for the synthesis of PANI/NiO nanocomposite [11].

Preparation of NiO and PANI/NiO electrode:

Three electrodes were used for the electrochemical measurements: a reference electrode made of Ag/AgCl, a working electrode made of the produced material, and a counter electrode made of platinum.The electrochemical reduction was carried out in 1M KOH aqueous solutions. NiO (active material), Polyvinylidene fluoride (PVDF) and carbon black in a weight ratio of 8:1:1 was mixed with a few drops of N-methyl-2-pyrrolidinone (NMP); specifically, the solvent dosage depends on its mixed powder counterpart [12]. The black mixture was ground until it became viscous. Then, the mixture was coated on a sonicated carbon rod and dried at 60^{o}C for 12h.Similarly, PANI/NiO electrode is prepared by using the same procedure in which PANI/NiO nanocompositewere used as an active material.

RESULTS AND DISCUSSION:

Transmission Electron Microscopy:

Analysis of Transmission Electron Microscopy (TEM)

Crucial details about both material systems—pristine NiO and its polyaniline-based nanocomposite—are revealed by the nanoscale structural insights provided by TEM imaging in Figure1.

1.A] NiO Nanoparticles (Left Image): In accordance with the scale bar (20 nm), the TEM image displays evenly distributed quasi-spherical NiO nanoparticles with a size distribution primarily in the 15–30 nm range. In comparison, the particles seem rather uniform, suggesting a homogeneous crystallinity. Because of their high surface energy, metal oxide nanoparticles frequently exhibit mild agglomeration. The lack of severe aggregation, however, indicates efficient control throughout synthesis, most likely made possible by the use of a starch capping agent.

The majority of the particles appear as individual crystallites rather than sintered masses, and the particle boundaries are clear and sharp. These characteristics suggest a high degree of surface accessibility, which is highly advantageous for applications such as electrochemical energy storage, where open, accessible sites are necessary for quick redox reactions.

NiO has a high surface-to-volume ratio and quick ion diffusion capabilities due to its visible porosity and nanoscale homogeneity, making it perfect for supercapacitor electrodes.

1.B] PANI/NiO Nanocomposite(Right picture): TEM picture displays a notably altered shape when seen at a greater scale (50 nm bar). PANI chains are now responsible for the NiO nanoparticles' appearance immersed within a diffuse, semi-transparent matrix[13-14]. This coating, which was most likely created by in-situ oxidative polymerization reflects the concept of strong interfacial contact or potential partial encapsulation which is supported by the smooth and continuous appearance of the interface between NiO and the PANI matrix in multiple areas. Increased agglomeration is seen in the image, however this is more likely the result of the polymer's ability to function as a binder and pull nanoparticles together than of unchecked clustering.

Individual NiO grains can still be seen in the composite, but they are less clear than in the pristine sample, which is proof that the polymer phase has successfully integrated them. The conducting PANI creates electrical percolation channels, which are necessary for hybrid

capacitive behavior, as indicated by the fuzzy zones between particles.

The development of a PANI-wrapped NiO network produces a hybrid interface that facilitates the traditional dual-charge-storage mechanism of pseudocapacitive behavior from PANI as well as Faradaic redox activity from NiO.

Electrochemical Impedance Spectroscopy:

Nyquist plots of NiO and PANI/NiO electrode are shown in figure 2 in order to study electrochemical behavior of NiO and PANI/NiO electrode. **Small semicircle at high frequency,** beginning near zero on real axis ($Z' \approx 0\,\Omega$) and ending around 100 Ω. This arc represents combined electrolyte and charge-transfer resistance (Rs + Rct), typical of a Randle's circuit. Minimal **vertical tail,** instead showing a gradual slope in the low-frequency region indicates noticeable Warburg impedance due to slow ion diffusion within the NiO electrode. NiO has moderate internal resistance indicates electrons transfer with some hindrance and ion movement is diffusion-limited, which is typical for bare oxide electrodes [15]. Hence, NiO exhibits moderate charge-transfer resistance with visible Warburg diffusion, the gradual slope confirms ion transport is limited.

Very large semicircle spanning from~0 to ~3.5 kΩ indicates higher apparent resistance, but this could be largely due to the enlarged electrochemical interface rather than poor conductivity. Nearly vertical tail at low frequency, approaching an almost pure imaginary component. Signifies strong capacitive behaviour and minimal diffusion resistance consistent with high pseudo capacitance from PANI. The PANI layer enhances capacitive storage (vertical low-frequency response), but the large Rct semicircle suggests complex interfacial effects likely modelling the coating as a resistor-CPE network [16]. This aligns with literature noting low apparent Rct in PANI/NiO composite along with vertical capacitive lines. **PANI/NiO** Displays strong capacitive storage due to PANI, with apparent high resistance potentially due to composite interfacial complexity [17]. The vertical response indicates efficient capacitor-like storage with minimal diffusion limitations.

From the nyquist plot of the NiO electrode, low charge transfer resistance shows high specific capacitance as compared to PANI/NiO electrode due to higher surface area and smaller grain size (23.54nm) [17]. These remarkable properties of the NiO electrode material make it better electrode material as compared to the PANI/NiO electrode. Hence NiO electrode could be used for practical application of Supercapacitor for energy storage applications.

CONCLUSION:

PANI/NiO nanocomposite were successfully synthesized by in-situ chemical oxidative polymerization of aniline using ammonium persulphate as an oxidant in the H_2SO_4 solution.TEM image of PANI/NiO nanocomposite shows predominant formation of spherical NiO nanoparticles within PANI matrix in the range of 50nm.Theelectrochemical datademonstrate that the PANI/NiO electrode exhibits good pseudocapacitive behavior due to smaller crystallitesize with a spherical morphology which enhances fast ion and electron transport and also provides a large surface area. These excellent properties make the PANI/NiO electrode suitable and more efficient for Supercapacitor application.Overall Electrochemical Performance of PANI/NiO composites outperform pure NiO in energy storage applications by merging fast electron kinetics and capacitive behaviour though proper circuit modelling is essential to extract physically meaningful resistances.

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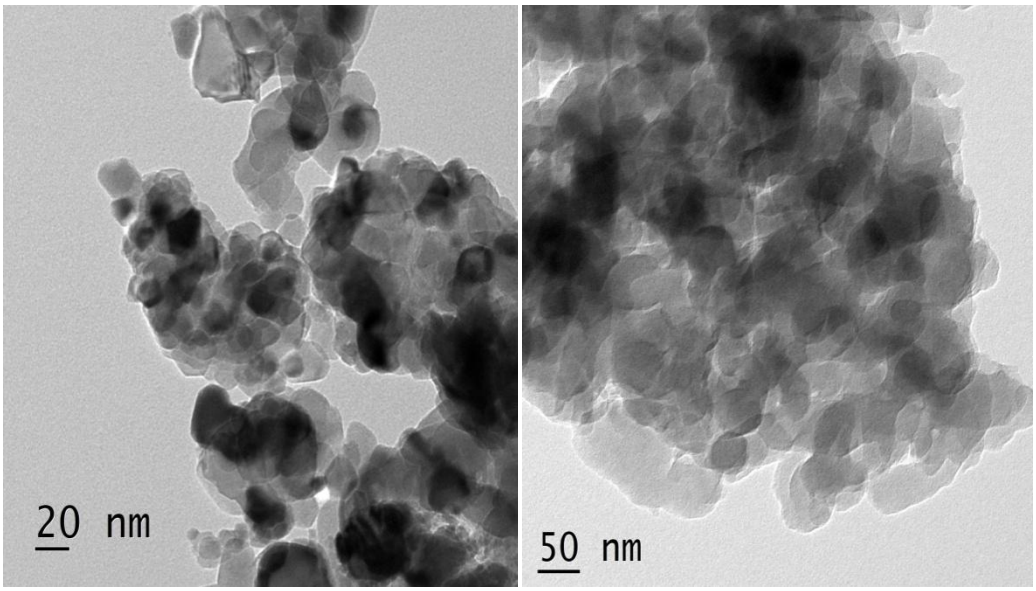

Fig.1: A]TEM image of NiO **B]TEM image of PANI/NiO nanocomposite**

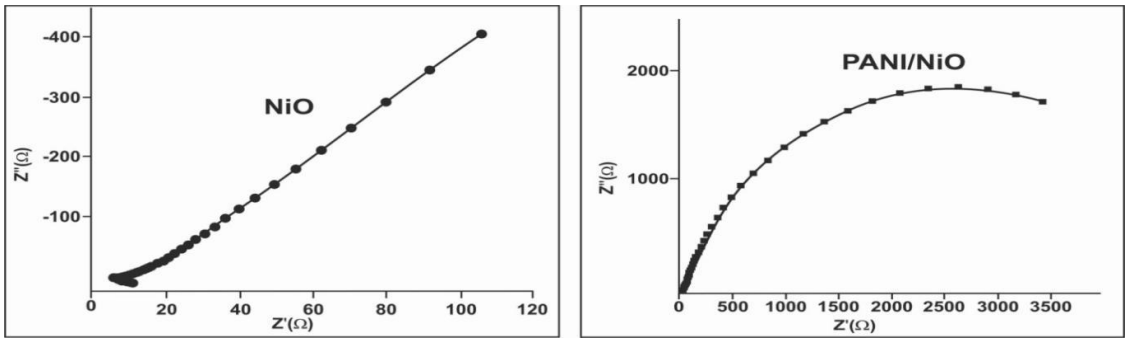

Fig.2: A]Nyquist plot of NiO **B] Nyquist plot of PANI/NiO electrode**