SYNTHESIS, CHARACTERIZATION, AND INVESTIGATION OF ELECTRICAL, MAGNETIC, SENSOR, AND MECHANICAL PROPERTIES OF POLYANILINE/ZINC DIOXIDE NANOCOMPOSITES

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ABSTRACT

Polyaniline/Nanocomposites were synthesized by the In-Situ Polymerization method. Characterization techniques are used to study the Crystalline nature, Particle size with X-Ray Diffraction, and Chemical composition and bonding by FTIR. Electrical properties were studied by using Keithley 6514 meter to know the conductivity of Polyaniline and Polyaniline / ZnO nanocomposites with respective the frequency Increase in Electrical conductivity found due to the addition of ZnO Particle, Magnetic properties were studied to know the behavior of samples like para magnetic, Ferromagnetic or diamagnetic nature obtained results are discussed. Mechanical properties were investigated to study the stress V/S strain and the results are discussed below.

Keywords: XRD, FTIR, SEM, Stress, Composite.

INTRODUCTION

Polymers are Insulating materials and their properties may be changed by doping metal. Recently many researchers are focusing on conducting polymers to change insulating polymer materials into conducting polymers or semiconductors, because of its unique properties like Electrical, Optical, and Optoelectrical properties, Polyaniline becomes one of the important conducting polymers, and also it is easy to synthesize, excellent environmental stability. It can be used in electrochromic devices, LEDs, Sensors Batteries, etc.¹⁻⁴ Cps are one of the interesting fields in research (CP’s) which is capable as the most major material for the Potential application. Therefore, the development of multifunctional conducting polymers plays a very excellent role and unique subject for researchers and academicians. Due to Particle size, shape, and distribution of particles, they can be adjusted to the desired application which they have many applications in electrical nanodevices.⁵⁻¹³ Among Metal oxide ZnO Nanoparticles shows excellent physical and chemical properties and also extensive application in diverse areas such as coating, solar cell, and photocatalysts.¹⁴⁻¹⁷

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EXPERIMENTAL

Preparation of Polyaniline
The chemical method was used to synthesize PANI. The synthesis was carried out at room temperature by mixing an aqueous solution of aniline, HCL, and ammonium persulphate. The reaction between aniline and HCL produces in a volumetric flask, aniline hydrochloride was dissolved in water to make a 100 ml solution. 100 mL of APS solution was added to the solution with continuous stirring for 3 hours. The following day, the Polyaniline sample was washed with 0.2 M Acetone and HCL. For one day, a PANI sample was dried in a vacuum at 60°C. Finally, Polyaniline is formed.

Synthesis of ZnO Particles
ZnO was synthesized by the sol-gel method. Zinc Nitrate solution and citric acid sol solution were prepared first with a specific percentage for the zinc nitrate solution we have to add citric acid solution drop at a continuous stirrer for 3 h at T=80 °C. To keep the PH stable, ammonia is added to the solution. The spongy gel forms after the complete evaporation of water molecules from solutions, and the above gel is heated. The gel was then heated in a hot oven at T=150°C for 1 hour to obtain the compound, which was then ground well and calcined for 3 hours to remove impurities, yielding ZnO nanoparticles.

Preparation of Polyaniline /ZnO Nano Composites
PANI/ZnO composites were synthesized by the In-Sute polymerization method. To make aniline hydrochloride, 1M HCL was mixed with 0.1 M aniline and agitated for 20 minutes. To keep the ZnO uniformly suspended in the solution, zinc oxide (ZnO Wt%) was added to the aforementioned solution and stirred on a regular basis. To completely polymerize, 0.1 M of APS was added to the solution with continuous stirring at 5 °C for 3 hours. The solution was filtered, rinsed with deionized Acetone, and finally dried in an oven for 24 hours to achieve consistent mass.

Characterization
XRD was Studied by Philips X–Ray Spectrometer with CuKα as the source. FTIR by ATR Nicolet Model. Morphology was studied by Model-EVO-18 (Special Edison Germany).

RESULTS AND DISCUSSION

X-Ray Diffraction Analysis
The XRD image of Polyaniline/ZnO shown in Fig.-1 may be due to scattering from the PANI chain at interlayer spacing, indicating the amorphous nature of polyaniline, and that peak corresponds to 200 diffraction planes of PANI, for pure ZnO the particle size is calculated by Scherrer equation the XRD (Fig.-1) shows the ZnO particles are good in agreement with the hexagonal formation with JCPDS Number -05-0664 the peaks for Polyaniline/ZnO nanocomposites are shown in Fig.-1, the intensity of Polyaniline/ZnO peaks are lesser then that of pure ZnO the amorphous nature of Polyaniline reduces the crystalline of ZnO/PANI composites. It was found that the particle size of ZnO is 34.63nm.

FTIR Analysis
FTIR Spectra of Pure ZnO shows in Fig.-2 that peak 24 is due to unsaturated amine, 1556 for C-N Stretching of quinoid rings, 1457 due to benzenoid ring, and the peaks at 1298 correspond to C-N starching which confirms the formation of Polyaniline the peaks at 1101 cm⁻¹ is for bending vibration,789
cm⁻¹ and 706 cm⁻¹ due to benzenoid group, 520 cm⁻¹ corresponds to stretching at out of the plane.
PANI/ZnO nanocomposites peaks appear at 3766 to 3397 due to water molecules, 2976 peaks due to C-H
stanch of composite the peak 2372 corresponds to unsaturated amine, 1565 corresponds to C-N Stretching
of quinoid rings, Benzenoid rings on 1498 peaks 1297 due to dopant anion, the peaks 582 due to
interaction of and peaks due to from 499 to 461 cm⁻¹ is due to metallic stretch.

![Fig.-2: FTIR for Pure ZnO](image)

![Fig.-3: FTIR for PANI/ZnO](image)

**SEM Analysis**

Scanning electron microscopy of PANI synthesized by the in-situ method is shown in Fig.-4. The SEM
image pure Zinc Oxide is highly mesoporous particles with granular in shape and they are well
interconnected with each other. Figure-5 shows that most of the particles are agglomerated and irregular
in shape & they are well interconnected to each other.

![Fig.-4: SEM Image of ZnO](image)

**AC conductivity**

Ac Conductivity of the prepared composite was studied at room temperature with an increase in
frequency. The increase in conductivity with increasing frequency is due to the universal power law, and
it is also observed that the higher frequency region has a sudden increase in conductivity, which attributes
to the characteristic properties of disordered materials. The highest conductivity is found in 50 wt. percent
of the PANI/ZnO composites may be due to dipole polarization.
Magnetic Properties
Pure Zinc oxide nanoparticles exhibit saturation Properties Msat a temperature of 81.45 emu/g Remanant induction Mr=78.89 and coercive field Hc// 1278.89 Oe, the measurement range H=11 KOe. The values of (Ms, Mr, Hc, and Oe) in the case of PANI – Zinc oxide nanocomposite are shown in Table-1. The loosely connected particles, but these particles doped in a PANI matrix show a considerable difference in terms of hysteresis opening up with coercivity. The magnetic properties of PANI/ZnO nanocomposite powder have to be determined at different wt%, as shown in Table-1.

<table>
<thead>
<tr>
<th>Wt % of Polyaniline –ZnO</th>
<th>Ms EMU/G</th>
<th>Mr EMU/G</th>
<th>HcOe</th>
</tr>
</thead>
<tbody>
<tr>
<td>10wt%</td>
<td>07</td>
<td>02</td>
<td>22</td>
</tr>
<tr>
<td>20wt%</td>
<td>14</td>
<td>05</td>
<td>30</td>
</tr>
<tr>
<td>30wt%</td>
<td>26</td>
<td>14</td>
<td>63</td>
</tr>
<tr>
<td>40wt%</td>
<td>40</td>
<td>22</td>
<td>74</td>
</tr>
<tr>
<td>50wt%</td>
<td>91</td>
<td>42</td>
<td>126</td>
</tr>
</tbody>
</table>

Mechanical Properties
Stress-Strain curve of PANI/ZnO composites for the above film obtained.
Table-2: Details of Mechanical Properties

<table>
<thead>
<tr>
<th>Sample Material</th>
<th>Youngs M</th>
<th>Break Point</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyaniline</td>
<td>1.92</td>
<td>89.6</td>
<td>5.89</td>
</tr>
<tr>
<td>PANI/ZnO (1%)</td>
<td>2.02</td>
<td>103</td>
<td>5.45</td>
</tr>
<tr>
<td>PANI/ZnO (3%)</td>
<td>2.34</td>
<td>117</td>
<td>5.05</td>
</tr>
<tr>
<td>PANI/ZnO (5%)</td>
<td>2.85</td>
<td>136</td>
<td>4.58</td>
</tr>
</tbody>
</table>

Sensor Properties

Polyaniline/ZnO Composites' sensor properties were investigated with increased sensitivity V/S Weight percentage. Compare to composites pure PANI Fig.-8 shows less sensitivity because of low surface area. Compare to all composites 50 wt.% of composites show maximum sensitivity. In the case of semiconducting metal oxide gas sensor sensitivity always depends on the chemisorbed oxygen ions and the interstitial ions.

CONCLUSION

Polyaniline/ZnO Composites were synthesized by the polymerization method. From XRD It confirms the amorphous nature of polyaniline the peak observed at 25 Degrees which is the characteristic peak of PANI. Crystalline peaks of a composite well-matched with JCPDS number. SEM micrograph of the Polyamine &ZnO/PANI composite. The SEM micrograph of pure Polyaniline clearly describes the surface morphology of the PANI semi-crystalline structure. FTIR spectroscopy is one of the different non-destructive methods and is well manufactured and invariably expands its manufacturing analytic method for analyzing diff range of materials. The different characteristic peaks of IR spectra of the polyaniline between the percentage transmittance (T%) and wave number are shown. AC electrical conductivity increases with increase as frequency increased from 1 kHz to 5 kHz. The AC electrical conductivity of the polyaniline was maximum when compared to all composites. Further, lessen in conductivity for PANI composite may be caused by the trapping of charge carrier hop. The magnetic and mechanical properties were also discussed.

CONFLICT OF INTERESTS

The authors declare that there are no conflicts of interest.

AUTHOR CONTRIBUTIONS

All of the authors made major contributions to this work, took part in its review and editing, and gave their final approval for publishing. The research profile of the authors can be verified from their ORCID ids, given below:

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