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Synthesis, characterization of Zinc oxide and assessment of electrical DC conductivity properties

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ABSTRACT

Polymer composites are widely used in electrical devices, sensor materials, electromagnetic interference (EMI) shielding, and other applications. Composites may be customized for any intended application by changing the ratios of the polymeric components. Polyaniline (PANI) is not as sensitive as metal oxides toward gasoline species, and its negative solubility in organic solvents limits its packages, however it's miles suitable as a matrix for instruction of engaging in polymer nanocomposites. Therefore, there has been increasing hobby of the researchers for the education of Nano composites based totally on PANI The fuel for the production of nanometal oxide comes from naturally occurring sources. The weight percentages of such generated metal oxides are altered during chemical polymerization with polyaniline. Composites of polyaniline cellulose and varying weight ratios of Zinc oxide (ZnO) are produced. In synthesised Polyaniline cellulose/ZnO composites, the formation of polymers and their interactions with metal oxides are investigated using Ultraviolet (UV)-vis, Fourier-transform infrared spectroscopy (FTIR), and X Ray Diffraction. The surface morphology of the composites is studied using Scanning electron Microscopy. The characteristics of the specified composites are investigated using electrical dc conductivity electrical conductivity.

1 Introduction

One of the maximum usual biopolymers and an average reinforcement material is fibre technology; cellulose is seemed as an infinite deliver of sustainable natural polymers in light of the growing want for biodegradable and environmentally friendly products [1-6]. Additionally, cellulose-based substances provide a ramification of blessings, together with being environmentally pleasant, biocompatible, and economically nice resources of carbon-based totally polymers and substrates for the introduction of nanocomposites with exclusive fabric properties. One of the maximum extensively used cellulose derivatives, cellulose acetate (CA), has a number of standout traits, which includes nontoxicity, renewability, fee efficiency,

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and biodegradability. Cellulose is a main, drastically dispersed, and low-price biopolymer of global economic significance [7]. A new enterprise has recently advanced to use this promising fabric in electronics programs together with sun cells, gasoline sensing, bio sensing, image detecting, strength gadgets, and electrodes [8]. Cellulose fibres emerge in nature as a nano fibril-community with diameters starting from 100nm to one micrometer [9]. Nano cellulose fiber (NCC) is decided by the synthesis method used. Because of its cylindrical floor/volume ratio, uneven structure, excessive electricity, low thermal enlargement, and stiffness, cellulose nanofibers (CNF) skinny movies are properly suitable to be used in sensing devices [10, 11].The usage of Nano composite materials as catalysts or adsorbents to take away risky compounds from wastewater has acquired a number of interests in the last decade. Mineral, organic, or organic adsorbents include clay, zeolites, activated carbons, silica beads, polymeric polymers, and metallic oxides [12-15] .Zinc oxide (ZnO) in its nanostructured shape is a promising semiconducting oxide to be used as a nanocomposite polymeric cloth due to its huge variety of low-dimensional nanostructures, efficient adsorption, excessive surface reactivity, adsorption capability, and unfavourable sorbent ion potential [16, 17]. ZnO nanostructures (together with nanoparticles, nanowires, nanoflowers, and nanosheets) had been often hired inside the polymeric membrane modification system as multifunctional inorganic nanomaterials [18]. Moreover, questions remain concerning the membranes' sturdiness, biocompatibility, and performance while utilized in real-global filtration or treatment devices. The resulting polymer/nanocomposite materials need show off exquisite electric, mechanical, thermal, and chemical houses, in addition to exact permeability, thermal balance, microbial resistance, and water selectivity.

2   Polymer Nano composites

The process of making polymers an essential component of polymer nanotechnology is nano composite. Nanometric inorganic compounds can be added to polymers to boost their properties and provide them a range of applications, depending on the inorganic material that is added. There are many different forms of polymer composites that have been described thus far, created using different methods, and characterised using different methods with distinct applications. Polymer nanocomposites are substances in which inorganic nanoparticles, typically 10-100 Ao in at least one dimension, are dispersed in an organic polymer matrix to significantly improve the performance and properties of the polymer. Nanocomposites are frequently transparent because of the nanoscale length scale, which reduces light scattering [19-21]. A new form of polymer that can take the place of conventionally filled polymers is polymer nanocomposites.

Because of their Nano scale dimension, filler dispersion nanocomposites offer significantly superior properties than pure polymers or traditional composites. Nonlinear optical properties, electronic conductivity, and luminescence, as well as increased modulus and strength, outstanding barrier properties, improved solvent and heat resistance, and decreased flammability, are all recommended for use in a wide range of applications, such as electrochemical sensors, electroluminescent devices, electro catalysis, batteries, smart windows, and memory devices [22-27].

To achieve a homogeneous dispersion and excellent contact between the polymer and additional particle surfaces, it is essential to comprehend and control the guest-host chemistry that takes place between the polymer and the layered compounds or nanoparticles.

Nano composite voltammetry sensors offer a distinctive combination of features, such as good electrical conductivity and wide surface area, making electrochemical investigations of various electro active chemicals at various manufactured polymer sensors using various electro analytical methods essential. Due to their complex formation, the innovative polymer Nano composite modified electrodes are anticipated to enhance current responsiveness with excellent stability and repeatability.

In present research, experiments had been performed for the development, synthesis and characterization of PANI/ ZnO Nano composites. Also in work is to assess how electrical conductivity of PANI/ZnO changes as ZnO content changes. UV-visible (UV) spectroscopy studies were used to evaluate the polymerization of PANI/ZnO. Through the use of X-ray diffraction, scanning electron microscopy (SEM), and Fourier-transform infrared (FT-IR) spectroscopy, the chemical and physical properties of the PANI/ZnO generated under the optimum circumstances were assessed.

3 Materials and Methods

3.1 Zinc Oxide

Zinc oxide is used inside the manufacture of rubber and cigarettes. Popularly regarded calamine lotion is produced from zinc oxide powder. It is also used in a number of different creams and ointments which can be used to deal with pores and skin illnesses. As an additive inside the manufacture of concrete. Ceramic enterprise has a number of makes use of for zinc oxide powder. It's also used as an additive in meals merchandise consisting of breakfast cereals. Various paints use zinc oxide as a coating agent. Business mantles are made up of a mesh hood of /thorium/ oxide surrounding a flame and are utilized in ornamental exterior lighting, gas lanterns for tenting.

3.2 Synthesis of ZnO nanoparticles

Precursor $Zn(NO3)2$ of about 2974mg is mixed with fuel 670mg Agaricus bisporus in the optimized concentration with minimum amount of nitric acid is added and stirred thoroughly to obtain homogeneous solution in a silica crucible. Similarly, In another reaction $Zn(NO3)2$ of about 2974mg is mixed with fuel 670mg Agaricus bisporus in the minimum amount of 0.5M NaOH (alkali) and stirred thoroughly to obtain homogeneous solution in a silica crucible, reaction is unfavourable to get expected product.

The mixture is heated in muffle furnace at 500oC for three hours. When the temperature of the solution goes beyond 350oC solution begin to boil with liberation of oxides of carbon, resulting in the reddish brown fumes shows the evolution of oxides of nitrogen, finally foamy white mass ZnO nanoparticles. Repeated heating and cooling yield constant mass of 0.691gm is optimized.

$$Zn\ (NO3)2\ +\ Pani\ Cellulose\ \rightarrow\ ZnO$$

3.3 Preparation of PANI Cellulose/ZnO composites

0.6mol of Aniline is mixed with 50 ml, of 0.05mol of DBSA as a surfactant and 1 gm of Sigma cellulose are taken in a 500 ml beaker and kept at 0 0C with stirring. Above mixture is added drop wise with 0.2mole of FeCl3 for 5min. 5, 10, 15 and 20 weight percentages of nano ZnO added and the mixture is stirred for the 4-5 hours. The obtained composites are washed with alcohol and filtered using suction pump with what man filter paper No.40. The Yield obtained is 6598mg, 7886mg, 8786mg and 9356mg for Panicellulose/5ZnO, Panicellulose/10 ZnO, Panicellulose/15 ZnO and Panicellulose/20 ZnO respectively.
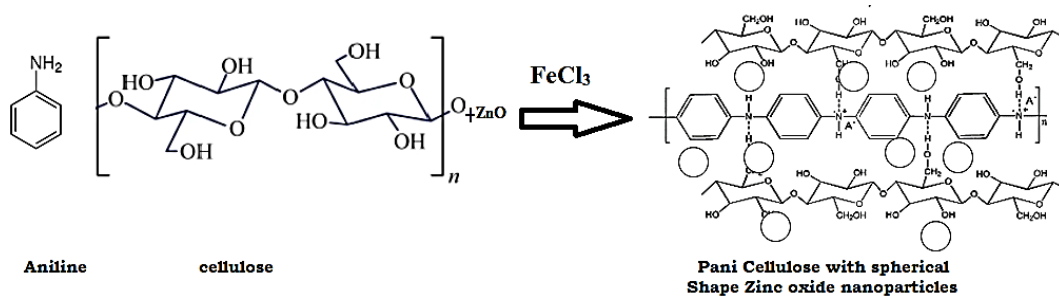

Fig. 1 – Reaction scheme for the synthesis of PANI cellulose/ZnO Nano polymer composites.

3.4 PANI sigma Cellulose/ZnO Nano composites

Figure 2. shows Pani Cellulose and Pani cellulose/ZnO composites. Pani sigma Cellulose shows peaks at sharp at 380-400 nm range. Are the characteristic peaks for the Pani. Except for the sample Pani sigma Cellulose/10% ZnO composte all other composites shows the shoulder at 360 nm indicate formation of the copolymerization of the Panisigma cellulose. π- π^* transitions for the Pani characteristic peak which slowly shifts towards the higher wavenumber side. Indicating the coating of Panicellulose on the ZnO nano particles.
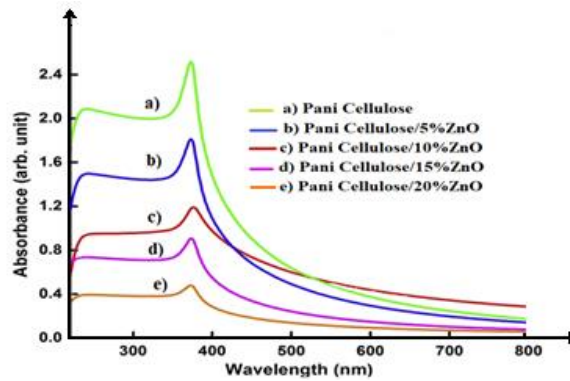
Fig. 2 – UV-visible spectra of a) Pani Cellulose b) Pani Cellulose/5%ZnO c) Pani Cellulose/10%ZnO d) Pani Cellulose/15%ZnO e) Pani Cellulose/20%ZnO
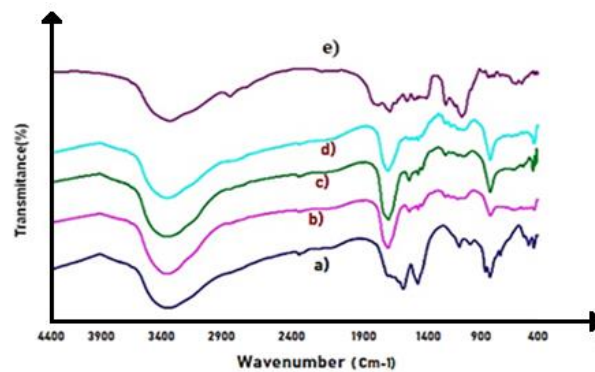
3.5 Panicellulose/ZnO Nano composites

Fig. 3 – FTIR of the of the ZnO and Panicellulose /ZnO nanocomposites synthesized a) Pure ZnO b) Pani cellulose/10% ZnO nanocompositesC) Pani/20% ZnO nanocomposites

Prepared nano zinc and pani cellulose/ZnO samples are studied by FTIR are shown in figure 3 (a-c). Peaks at 643 and 717 cm-1 may be assigned to Zn-O stretching band originated from ZnO. Peak at 1731 cm-1 1790 cm-1 and 1517 cm-1 may be assigned to asymmetric stretching of C-O of CO2, C=N- of aniline and C-N respectively for Pani cellulose. Another notable peak of 3441 cm-1 may be due to the presence of N-H of aromatic aniline molecule. It is understood that the decrease in the particle size enhances the surface effects which in turn enlarge the absorption. Thus, the Zn-O absorption bands are widened. FTIR ensures the presence of Pani Cellulose and ZnO in the composites. There is a formation of OH peak at 3371 cm-1 which further indicate the formation of Zn(OH)2 it may be due to the transformation of the phase from ZnO to Zn(OH)2

3.6 Pani Cellulose/ZnO nanocomposites

Figure.4 shows the X-ray diffraction pattern of panicellulose and panicellulose-zinc oxide composites. The wide peak of paniculosic appears at 19 degrees, and pure paniculosic also shows a wide peak at about 12 degrees, but this peak does not appear at paniculosic / 5% ZnO, but only at paniculosic / 10%. increase. ZnO and Pani look like cellulose / 15% ZnO, indicating that the polymer is predominant. The theta values at peaks of 12 and 19 degrees for a composite of 2 panicellulose and 20 percent ZnO are clearly shown. A ZnO peak was also seen in the sample. Pancellulose (5 percent ZnO) and pancellulose (15 percent ZnO) have strong peaks at two values at 28 degrees and are found in all composite samples with two low intensity theta values. Other peaks were also detected in the Pani Cellulose / 5% ZnO and Pani Cellulose / 15% ZnO composites, but the strength was not very high. ZnO is used to polymerize aniline and cellulose in composite materials, but when the growth temperature is lowered (40 ° C), ZnO undergoes a phase transformation to Zn (OH) 2 and transforms to crystalline ZnO at high temperatures.
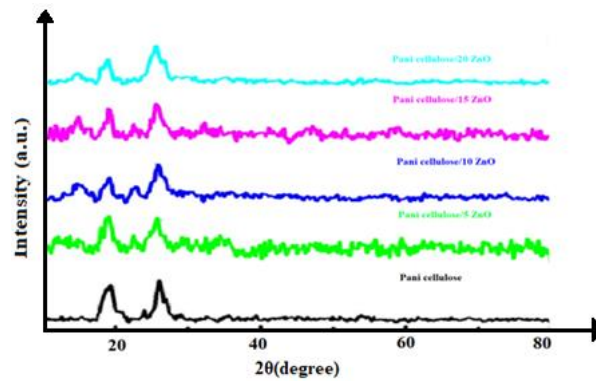
Fig.** Erreur ! Il n'y a pas de texte répondant à ce style dans ce document. **– XRD pattern of a) Pure Pani Sigma cellulose b) Pani Sigma cellulose/5% ZnO c) Pani Sigma cellulose /10% ZnO, d) Pani Sigma cellulose/15% ZnO and e) Pani Sigma cellulose /20% ZnO

Table1.shows that the presence of peaks in pani cellose and Z(OH)2, Panicellulose and Panicellulose-ZnO nanocomposites have been analysed the use of XRD to determine the impact of the special ZnO concentrations on the Panicellulose structure. The xrd Diffraction styles of panicellulose and pani-ZnO nanocomposites are proven in Fig. four. due to the parallel and perpendicular periodicity of the polymer (Pani chain), big diffraction peaks in Panicellulose and its nanocomposites display between 10° and 30°. because of the repeated benzenoid and quinoid jewelry in Panicellulose chains, the Pani top diffracted at angles of 2=19.3° and a pair of=26° with d spacings of 4.fifty eight Å and three.42 Å, respectively, in the XRD sample, indicating low crystallinity of the conductive polymers [28]. in addition, Pani crystal size, D, is four.3 nm, as decided via Scherrer's equation [29] in the addition of ZnO nanorods, the crystal length of nanocomposites rose from four.four nm for Pani to five.three nm, five.five nm, and 8.2 nm for PaniCellulose-ZnO 5 wt percent, 10%, 15%, and 20% nanocomposite, respectively. The XRD patterns of nanocomposites may be proven to be similar to the ones of PANI. that is due to the low amount of ZnO nanoparticles, that is less than 20%, and it has no impact at the PANI diffraction sample. in keeping with Fig. four, two specific sharp peaks at 2=19.31° and 2=25.seventy two° with planes of (010) and (2 hundred), respectively, are displaced negligibly within the provided range pronounced for the extensive peak, but their electricity rises while the attention of ZnO nanorods in Pani-ZnO nanocomposite increases.

Table 1 – Diffraction planes with 2Ө values and interplanar spacing for the Pani and its ZnO composites

Polymer/composites	2Ө values for Pani Cellulose/ZnO with d spacing values				
	110	**200**	**010**	**102**	**200**
Pure PANICellulose			19	23	26
PANI Cellulose/5% ZnO	19.3 (4.58)	26 (3.41)	-	-	-
PANI Cellulose /10% ZnO	19.2 (4.81	25.9 3.42	-	-	-
PANI Cellulose /15% ZnO	19.28 4.8	25.83 3.47	-	-	-
PANI Cellulose /20% ZnO	19.04 4.86	25.6 3.4	-	-	-

The XRD results support recent findings that show the influence of ZnO nanorods in Pani-ZnO nanocomposites. As predicted, a modest proportion of ZnO nanorods has little influence on Pani's identity while improving the capabilities of this conductive polymer. The strength of the peaks was raised by increasing the number of ZnO nanorods, indicating that there is a contact between ZnO nanorods and Pani through hydrogen bonding between H-N and oxygen of ZnO [28].

4 Electrical conductivity

In the end, the composites were floor the usage of agate mortar in the presence of acetone medium. The usage of a hydraulic press, the powder is squeezed to create pellets with a thickness of 1-2 mm and a diameter of 10 mm. A micrometre

screw-gauge was used to measure the thickness of these pellets. On both aspects of the surfaces of the composite pellets, conductive silver paste changed into applied. to improve electric connections, copper wire of appropriate thickness is positioned on each surface.Panicelluose /ZnO, composites changed into used to degree the DC electric conductivity at a consistent temperature using the four-probe technique with a Keithley 224 steady modern source voltage carried out is 0.01-zero.05 V and a Keithly 617 virtual electrometer shown in figure 5. Resistance is measured and conductivity is calculated for the Pani/metal oxide composites.

Table 2 depicts temperature-dependent Panicellulose/ZnO composites. Panicellulose ZnO composites have much higher conductivity than Panicellulose composte. Pani cellulose/15% ZnO composite has the maximum conductivity. The conductivity behaviour of Panicellulose 5 percent ZnO and Panicellulose/10 percent ZnO composites is almost same, however conductivity values are lower than those of Panicellulose/15 percent ZnO composite.

Figure 5 shows the Panicellulose/ZnO composites at room temperature shows highest for Panicellulose/15%ZnO composites follows the percolation behavious. The calculated activation energy for pure PANIcellulose and the PANIcellulose/ZnO composites. Obtained activation energy are as follows 14.54, 12.43, 10.77, 17.57 and 15.65 eV K-1 for pure pani, Pani/5 ZnO, Pani/10 ZnO, Pani/15 ZnO and Pani/20 ZnO respectively.
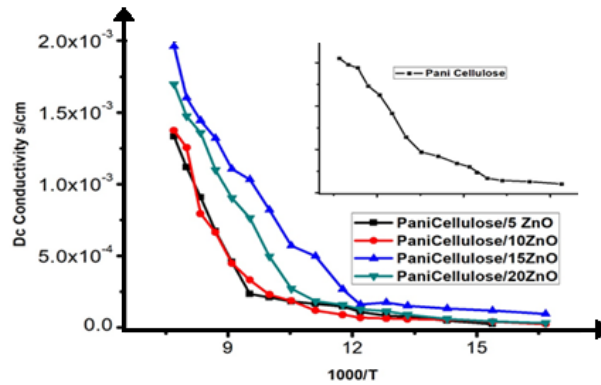

Fig. 5 – Temperature dependent dc Conductivity of PANI cellulose (inset figure) and PANI cellolose composites a) Pani /5Wt.% ZnO b) Pani / 10Wt.%ZnO c) Pani / 15Wt.% ZnO d) Pani / 20Wt.% ZnO composites.
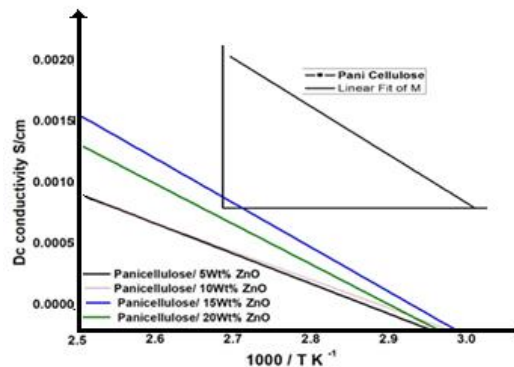

Fig. 6 – Arrhenius plot of Pani Cellulose (inset figure) and a) Pani /5 ZnO b) Pani /10 ZnO c) Pani /15 ZnO d) Pani /20 ZnO composites.

Figure 6 depicts temperature-sensitive Panicellulose/ZnO composites. Panicellulose ZnO composites have much higher conductivity than Panicellulose composite. The Pani cellulose/15% ZnO composite has the highest conductivity. The conductivity behaviour of Panicellulose 5Wt% ZnO and Panicellulose/10Wt% ZnO composites is comparable; nevertheless, conductivity values are lower than those of Panicellulose/15Wt% ZnO composite.

Exchange of electrons, the presence of a counter ion, and simultaneous modulation of the Fermi level or chemical potential are all involved in metal oxide of conducting polymers. Doping can be used to modify the electronic and optical properties of conducting polymers across a wide range. Mobile charge carriers supplied into the -electronic system by doping

influence the electrical conductivity of conducting polymers. At low doping levels, these charge carriers self-localize and form nonlinear topologies. Charge is expected to be carried mostly via conjugated chains due to the large inter chain transfer integrals. When a polymer is heavily doped, the wave functions are delocalized throughout the polymer chain over a significant number of lattice constants [30, 31].

Table 2 – DC conductivity of Panicellulose/ZnO nanocomposites at Room temperature.

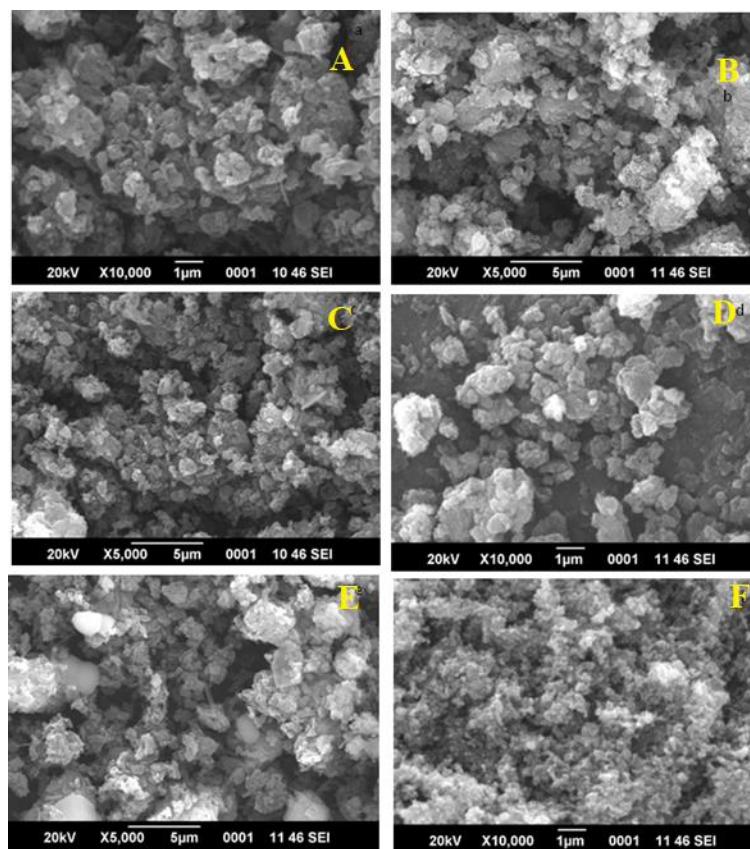| Percentage of Zno	DC conductivity s/cm
0	0.00125
5	0.00134
10	0.00137
15	0.00195
20	0.0017

Fig. 7 – SEM of a) Pure Pani cellulose b) Pani cellulose/5% ZnO c) Pani cellulose /10% ZnO, d) Pani cellulose/15% ZnO and e) Pani cellulose /20% ZnO f) ZnO nanocomposites

In the temperature range of 300–550 K, the DC conductivity of pure PANI and PANI/ ZnO composites was measured using a two-probe approach. By putting two steel electrodes within a specifically built metallic sample container, the bulk DC conductivity was determined. A calibrated copper-constantan thermocouple was put near the electrodes to detect the temperature. The DC conductivity Arrhenius figure reveals straight line behaviour. Pure PANI's DC conductivity increased exponentially with doping, indicating semiconductor properties. The relationship may be used to illustrate conductivity as a function of temperature.

$$\sigma DC = \sigma o \exp\{-\frac{\Delta E}{kT}\} \qquad (1)$$

where (ΔE) is the activation energy for the DC conduction mechanism, "k" is the Boltzmann constant, and "$\sigma 0$" is the pre exponential factor.

5   SEM of PANI cellulose/ZnO Nano composites

Figure 7 Show the SEM of PANI cellulose/ZnO Nano composites; all composites show the flower with small pebble like morphology. The figure depicts micrographs of a polyaniline/zinc oxide nanocomposite at various magnifications. Micro particle groups were formed by clumping the particles together. Figure 7b shows polyaniline SEM micrographs in a spherical shape. The SEM picture shows that zinc oxide is porous in nature, which increases the liquid-solid interfacial area, provides a conduit for ions to move, and ensures a high reaction rate. Figure 8c shows a combination of dark and light coloured particles, with the white zinc oxide particles wound around the black polyaniline.

6   Conclusion

ZnO doped polyaniline cellulose composites were created using in situ chemical oxidative polymerization at atmospheric pressure and visible light. XRD, FTIR, SEM, electrical conductivity, and UV-vis studies demonstrate that the PANI-ZnO nanocomposite was successfully synthesised in this work.

A viable process for the formation of PANI-ZnO nanocomposites has been proposed. According to XRD and FTIR results, the association between ZnO and PANI is based on hydrogen bonding and electrostatic contact. Experiments with electrical conductivity demonstrate that nanocomposites have lower conductivity than PANI and ZnO nanorods, and that the value of electrical conductivity declines as the concentration of ZnO rises. A SEM image reveals the uniform distribution of ZnO in the PANI-ZnO nanocomposite.

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