JETIR.ORG ISSN: 2349-5162 | ESTD Year : 2014 | Monthly Issue
JOURNAL OF EMERGING TECHNOLOGIES AND INNOVATIVE RESEARCH (JETIR)
An International Scholarly Open Access, Peer-reviewed, Refereed Journal

SEM AND FTIR CHARACTERISATION OF NANOPOLYMER FILMS SYNTHESIZED BY SOLUTION CASTING TECHNIQUE

***M.Sreedevi*[1], *Dr.P.Thimmaiah*[*2], *S. Khaja Hussian*[*3]**

[*1,3] Research Scholar, Department of Physics, Sri Krishnadevaraya University, Anantapur, A.P, India-515003
[*2] Asst.Professor, Department of Physics, Sri Krishnadevaraya University, Anantapur, A.P, India-515003

Abstract: Composite based on polyvinyl alcohol (PVA)/Carboxy methyl cellulose (NaCMC) and MnO_2 manganese dioxide. This work fabricated NaCMC/PVA/MnO_2 Nanopolymer Films using a solution casting technique. The NaCMC/PVA/MnO_2 nanoparticle nanocomposites have been prepared with different blend concentrations by weight percentages of (0.2, 0.4, 0.6)wt%. the optical microscopic image indicates that the MnO_2 nanoparticle additive distribution was homogeneous in the PVA/NaCMC blend. FTIR suggests no change in the chemical structure between the NaCMC/PVA blends and manganese dioxide particles. Optical measurements showed that the absorbance of NaCMC/PVA/MnO_2 nanocomposite increases with the increase in the concentration of Mno2 NPs and the increase in the concentration of manganese dioxide nanoparticles

***Keywords**:* NaCMC-PVA MnO2, SEM and FTIR.

1. INTRODUCTION

The blending process of specific polar polymers is of significant interest for modifying and developing new polymeric materials with properties that differ from those of the parent polymers. This process effectively creates flexible polymer matrices with high miscibility, unlike synthesising new polymers. Among these materials, semicrystalline polyvinyl alcohol (PVA) and amorphous carboxymethyl cellulose (CMC) are non-toxic, hydrophilic, and biodegradable.

These polymers can form excellent flexible films through a solution casting technique. PVA, known for its high durability, sound clarity, and excellent thermal stability, has various applications, including flexible water-soluble packaging films, textile sizing, paper coatings, polarised sheets, and solid polymer electrolyte preparations. On the other hand, natural CMC retains several desirable properties such as low cost, water retention, suspension, emulsification, and excellent film-forming capabilities. Therefore, CMC is a promising material for industrial and biotechnological applications, including paper making, textiles, electrical components, personal care products, printing, and medicine.

The hydroxyl (OH) groups within the chain backbone of PVA and NaCMC are functional for inter- and intra-hydrogen bonding. Research indicates that NaCMC/PVA blends are miscible with each other, and a composition of 25/25 wt% in the PVA/NaCMC blend exhibits a lower absorption edge than other prepared samples. Saadiah et al. investigated the structural, morphological and electrical conductivity of Nacmc/PVA blends. They depicted the formation of intermolecular forces of H- bonding within the CMC/PVA hybrid polymer between the -OH- group of PVA and -COO- group of CMC. Polymer nanocomposite materials comprise the significant properties of the inorganic nanofiller and the flexible polymer matrix. Thus, these hybrid materials introduce multifunctional advanced polymeric materials for enormous technological uses, such as in the development of the next fabrication of optoelectronics devices, solar cells, and energy storage devices. the choice of Mno2 in the nanocomposite's preparation is due to its high chemical/mechanical stabilities and electrical conductivity bdelerazik et al. found the enhancement of optical and thermal properties of NaCMC/PVA matrix low content of MnO_2 NPs. Further, several authors reported that the electrical and thermal properties of various polymer nanocomposite films containing different concentrations of Mno2 were significantly improved.

2. CHARACTERIZATION TECHNIQUES

Polymer consists of chains of varying lengths, and each chain consists of monomer residues which affect its properties. A variety of instrumental techniques are used to determine the properties of polymers such as: FTIR, DSC, TGA, X-RD, EDS, SEM and FESEM.

In order to analyze the properties of the nanocomposite films, different characterizations have been employed on the fabricated nanocomposite films is shown in below Figure1.

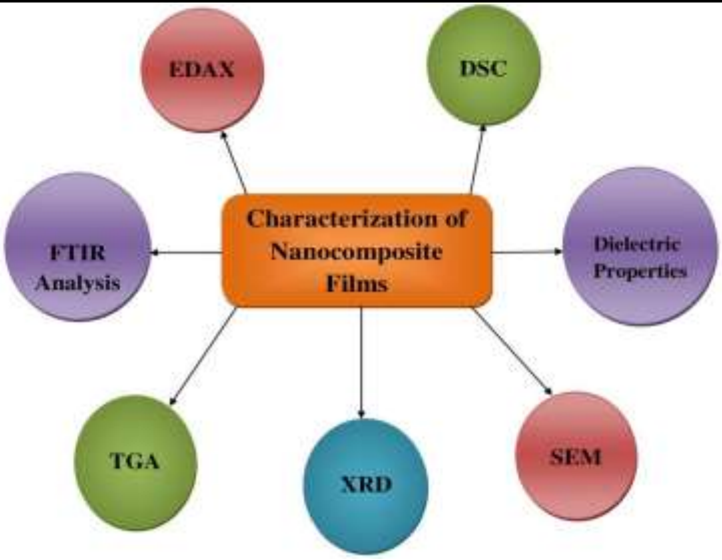

Figure 1: Various characterization techniques employed on fabricated nanocomposite films.

2.1. Fourier Transform Infrared (FTIR) Measurements

Infrared spectroscopy is the most common spectroscopic method used to determine the types of chemical bonds and functional groups in a polymer sample. FTIR is an important record which gives sufficient information about the structure of a compound or it can be employed to establish compounds or to determine the structure of a new compound.

The FTIR spectra result from energy changes arising due to different modes of characteristic stretching and bending vibrations. In the present thesis, FTIR analysis was done using Bomem MB-3000 (Make: Canada). The finely powdered and dried samples were mixed by crushing 3 mg of the polymer with 100 mg of KBr in a pestle and mortar. Pellets were prepared under a hydraulic pressure of 600 kg/cm^2.

These pellets were again crushed and repelleted. This step was repeated 2-3 times to get better reproducibility. Spectra were taken in the wavelength range 600-4000 cm^{-1}. The photo graph of instrument is given. **(See Fig: 2)**

Figure 2: FTIR Spectrophotometer (Bomem, Model: MB3000, Canada). (S.K.University)

2.2. Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) (Figure 3) is a type of electron microscope capable of producing high resolution images of a sample surface. Due to the manner in which the image is created, SEM images have a characteristic three-dimensional appearance and are useful for judging the surface structure of the sample.

Figure 3: Scanning Electron Microscope (SEM)

Scanning Electron Micrographs (SEM) of surface and cross section were taken for the pure and incorporated molecular sieves composite membranes, using software controlled digital scanning electron microscope JEOL JSM 5410 (Bangalore).

SEM micrographs of the membranes were obtained under high resolution (Mag. 300X5kv) Using Joel Model JSM 840A, scanning electron microscope (SEM), equipped with phoenix energy dispersive analysis. SEM micrographs were taken at Poorna pragna Institute of Scientific Research, Bangalore.

2.3. Field emission scanning electron microscopy (FESEM)

FESEM, CarlZeiss Ultra 55 with an image resolution of 0.8nm in high vaccumor variable pressure, an electron back scattered diffraction, an energy dispersion X-ray spectrometer, and a nano-pattern generation technology were used to obtain surface micrographs of the polymer films and nanomaterials deposited.

In scientific analysis, FESEM is a possible technique for analyzing comprehensive topographical images, morphological images, and solid sample compositional data on the basis of higher magnification, high resolution, and focus depth. This approach is used on the surface of materials to visualize a tiny segment of the sample. There should be a well-focused mono-energetic beam (50-150 kV) on a solid surface that has been placed in a vacuum. Consequently, secondary electrons would be released from the samples being bombarded. The distributed pattern then provides completed at a on the sample's scale, shape, and composition. A detector that captures and produces an electrical signal from a scattered electron. This signal are generated and translated into an image of a video scan or digital image that can normally be displayed upon on computer monitor. The benefits of the F E-SEM system include the ability to analyse smaller region contamination spots at electron acceleration voltages that are consistent with energy dispersion spectroscopy, decreased penetration of low-kinetic-energy electron probes nearer to just the material surface, and high-quality, low-voltage images obtained with negligible electrical charge of samples that are commonly used.

Figure 4: FESEM with EDS (Carl Zeiss/Ultra55instrument).

3. EXPERIMENTAL METHOD

Polyvinyl Alcohol (PVA) MW=1,25,000, AR Grade) This work used Merck India Ltd, India, and Carboxymethylcellulose (CMC) sodium salt purchased from Loba Chem, Mumbai, India. Films of PVA/NaCMC/MnO_2 nanocomposites were prepared using the solution casting technique.

3.1. Preparation of NaCMC solution

A total of 4 grams of the solute were carefully measured and added to 100 millilitres of deionized water. The mixture was then stirred continuously for approximately 24 hours to ensure thorough dissolution and to achieve a uniform, homogeneous solution. Throughout this stirring process, the solution was monitored to confirm that all the solutes had wholly integrated into the water, resulting in a clear and consistent preparation. This final solution was subsequently utilisedto conduct miscibility studies to evaluate its compatibility with other substances.

3.2. Preparation of PVA solution

I started by weighing out 4 grams of polyvinyl alcohol (PVA) and added it to 100 millilitres of deionised water. The mixture was stirred continuously for approximately 24 hours to ensure the PVA fully dissolved, and a homogeneous solution was achieved. Once the solution was uniform, it was diluted to match the NaCMC (sodium carboxymethyl cellulose) solution, maintaining the same volume and concentration for calculation purposes. This step was crucial for ensuring consistency in my experimental setups.

3.3. Preparation of NaCMC/PVA blend films

A homogeneous solution was created by mixing sodium carboxymethyl cellulose (NaCMC) and polyvinyl alcohol (PVA) in a 25/25 weight ratio, using a higher concentration than in previous experiments. The mixture was stirred until fully dissolved and transferred onto a ceramic plate at ambient temperature to allow gradual water evaporation.

After resting for 24 hours, the water evaporated completely, and the films were carefully peeled off the plate to prevent damage. The resulting high-quality blend films, infused with the properties of both NaCMC and PVA, were ready for further applications or testing.

3.4. Preparation of NaCMC/PVA/MnO_2 nanocomposite films:

NaCMC/PVA/MnO_2 nanocomposite films were successfully synthesised using the solution casting technique. Initially, a homogeneous solution of sodium carboxymethyl cellulose (NaCMC) and polyvinyl alcohol (PVA) in a 25:25 weight ratio was prepared. This mixture was subjected to continuous stirring to ensure complete dissolution and uniformity of the components.

To introduce manganese dioxide (MnO_2) into the composite, a series of suspensions with varying concentrations of MnO_2specifically (2%,4%,6%) were prepared. These suspensions underwent ultrasonication for one hour to promote effective dispersion of the MnO2 particles within the solution, breaking down agglomerates and enhancing the interaction between the components.

Once the MnO_2 suspensions were ready, they were gradually added to the NaCMC/PVA solution while maintaining constant stirring to achieve a homogeneous blend. This mixed solution was carefully poured into a ceramic plate to facilitate the casting process.

The casting plate was left at ambient temperature to allow for the complete evaporation of water from the solution. This process took approximately 24 hours, during which the excess moisture dissipated, forming a solid film. After this drying period, the resultant nanocomposite films were gently peeled off from the ceramic plate.

The final nanocomposite films exhibited a uniform thickness and were characterized by the incorporation of MnO_2, contributing to their potential applications in various fields, including electronics, sensors, and environmental remediation.

4. RESULTS AND DISCUSSION

4.1. FTIR Spectral Analysis

To prove possible intermolecular interactions between different components in the prepared nanocomposite systems. FTIR spectra of NaCMC/PVA blend, NaCMC/PVA/MnO_2 nanocomposites containing various concentrations of MnO_2 NPs (2%,4%,6%) were recorded in the range 500-4000cm-1 in the figure5.

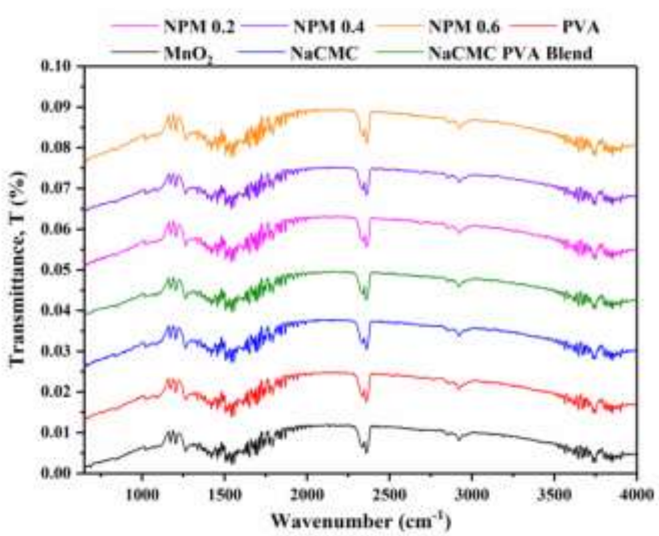

Figure 5: FTIR spectra for (NaCMC-PVA-MnO2)

FTIR spectra NaCMC/PVA/MnO_2 nanocomposites containing different concentrations of MnO_2 Nps (2%,4%,6%)comparing the IR spectra of the prepared Mno2, NaCMC and PVA as well as the fabricate NaCMC/PVA/MnO_2 nanocomposites films, form IR spectra it could be observed that the extra peaks of Mno2 Nps , NaCMC, PVA and NaCMC/PVA blend, all peaks seemed in the spectrum of NaCMC/PVA/Mno2 nanocomposites films, that suggests that there is no variation of the structure after compounding NaCMC/PVA shows FTIR absorption band at 3285 and 2928cm-1 associated to the hydroxyl (OH)group of (CH_2) asymmetric stretching related to NaCMC furthermore, Absorption bands concerning to Amide I and Amide II of C=O stretching vibrations and N-H bending vibrations of NH_2 as well as and CH2 flapping joined with OH group. this peaks at 1642cm^{-1} , 1581cm^{-1}, 1345cm^{-1} respectively.

The bands corresponding to OH groups shifted to 3310cm^{-1} and changed into weaker signals for NaCMC/PVA/MnO_2 nanocomposites films compared to NaCMC/PVA blend. The promising intermolecular interactions concerning these groups and nanoparticles influence the difference in the characteristic absorption bands.

4.2 SEM analysis

Figure 6 shows the SEM micrographs of the pure PVA/NaCMC blend & the filled samples had uniform surface morphology and a relatively smooth surface. After adding MnO_2 to the polymeric blend, as depicted in Figure 2b-d, bright spots and spots groups appeared on the surface of the films without any aggregation except in the highest concentration.

The bright spots increase as nanofiller concentration increases, which denote that these spots perhaps are of MnO_2 nanoparticles and propose the occurrence of a homogeneous growth mechanism. The variations at the filled samples denote the complexation and interaction compatibility between MnO_2nanoparticles and pure PVA/NaCMC. It can be concluded that the nanocomposite films (0.02 & 0.04 wt %). They have relatively smooth surfaces with no aggregation of $MnO_2$and depict the suitability to use them as insulators and substrates for developing microelectronic devices.

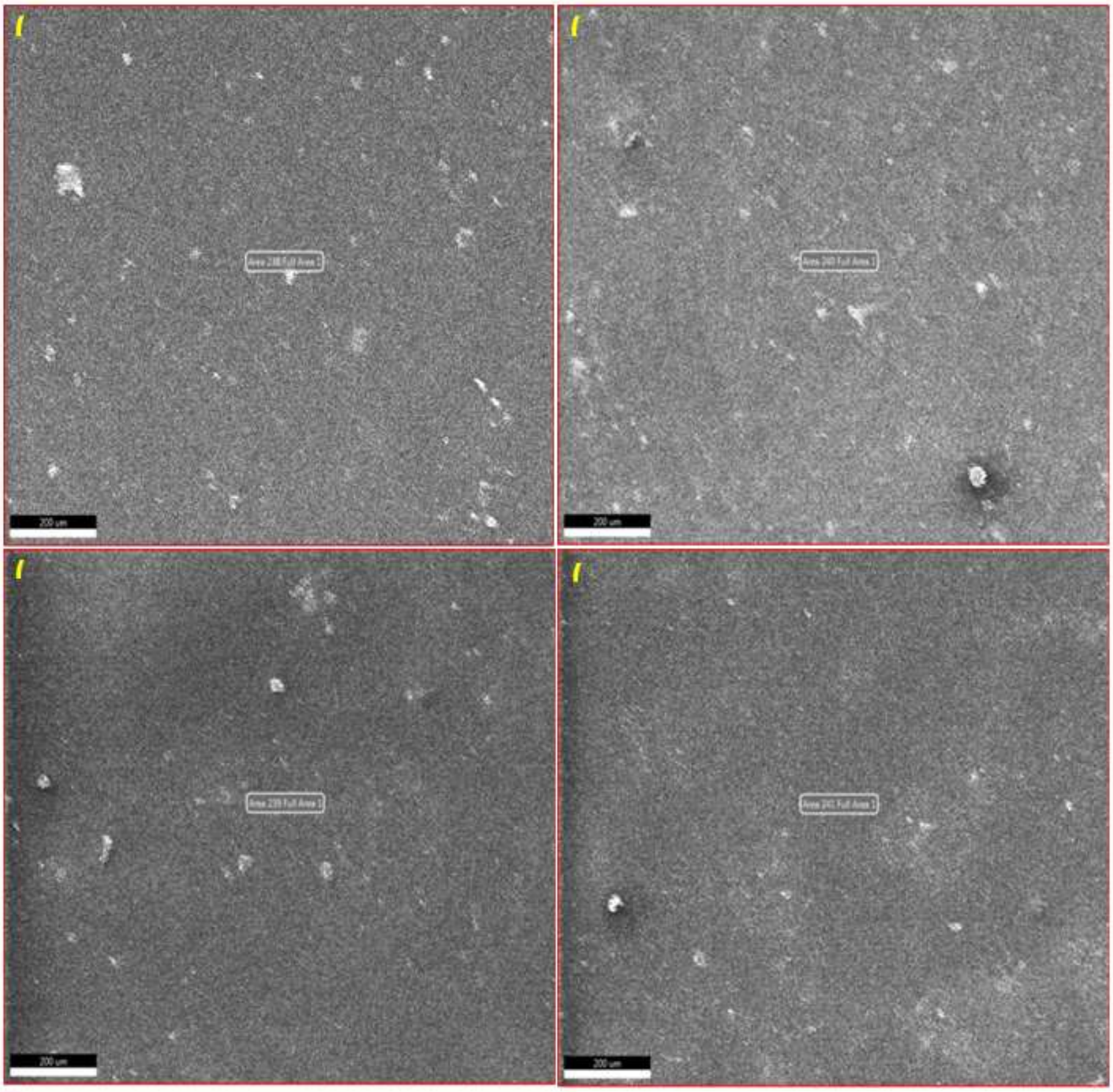

Figure 6: SEM photographs of Pure blend NaCMC/PVA (b) N/P/M0.2 (c) N/P/M0.4 (d) NPM0.6.

The morphological changes indicate nanoparticle-polymer interaction, depicting good compatibility between the inorganic and organic constituents in the nanocomposite. The SEM micrographs were exemplary, and they approved the previous measurements.

5. CONCLUSION

The polymer nanocomposite samples of a PVA/NaCMC matrix with varying concentrations of MnO_2 nanocomposites were prepared using the solution casting technique. FT-IR analysis confirmed strong interactions between the OH/COO groups of the polymer/NaCMC and the MnO_2 nanoparticles. SEM images showed that adding MnO2 nanoparticles modified the surface morphology of the PVA/NaCMC blend from rough to smooth, particularly in samples with higher MnO_2 content. The samples exhibited semicrystalline characteristics. Optical and electrical measurements revealed a decrease in the band gap and a shift in displacement for the MnO_2/NaCMC/PVA nanoparticles.

6. REFERENCES

[1] Mohammed G, El Sayed AM, Morsi W. Spectroscopic, thermal, and electrical properties of MgO/polyvinyl pyrrolidone/polyvinyl alcohol nanocomposites. J Phys Chem Solids 2017.
[2] Morsi M, Abdelghani A. UV-irradiation assisted control of the structural, optical and thermal properties of PEO/PVP blended gold nanoparticles. Mater Chem Phys 2017; 201:100–12.
[3] Saadiah M, Zhang D, Nagao Y, Muzakir S, Samsudin A. Reducing crystallinity on thin film-based CMC/PVA hybrid polymer for application as a host in polymer electrolytes. J Non-Cryst Solids 2019; 511:201–11.
[4] ElFewaty NH, El Sayed A, Hafez R. Synthesis, structural and optical properties of tin oxide nanoparticles and its CMC/PEG–PVA nanocomposite films. PolymSciSer A 2016; 58:1004–16.
[5] Mohammed G, El Sayed AM. Structural, morphological, optical and dielectric properties of M3+/PVA/PEG SPE films (m = La, Y, Fe or Ir). PolymAdvTechnol2019, http://dx.doi.org/10.1002/pat.4508.
[6] Choudhary S. Structural, morphological, thermal, dielectric, and electrical properties of alumina nanoparticles filled PVA-PVP blend matrix-based polymer nanocomposites. Polym Compos 2018;39: E1788–99.
[7] Choudhary S. Characterization of amorphous silica nanofiller effect on the structural, morphological, optical, thermal, dielectric and electrical properties of PVA–PVP blend based polymer nanocomposites for their flexible nanodielectric applications. J Mater Sci Mater Electron 2018; 29:10517–34.
[8] Yassin AY, Mohamed A-R, Abdelrazek EM, Morsi MA, Abdelghany AM. Structural investigation and enhancement of optical, electrical and thermal properties of poly (vinyl chloride-co-vinyl acetate-co-2-hydroxypropyl acrylate)/graphene oxide nanocomposites. J Mater Res Technol 2019; 8:1111–20.
[9] Abdelrazek EM, Abdelghany AM, Badr SI, Morsi MA. Structural, optical, morphological and thermal properties of PEO/PVP blend contain different concentrations of bio synthesized Au nanoparticles. J Mater Res Technol 2018; 7:419–31.
[10] Elashmawi I, Elsayed NH. The role of gold nanoparticles in the structural and electrical properties of Cs/PVP blend. Polym Bull 2019:1–14.